International Research Journal of

SCIENCE & ENGINEERING

An International Peer Reviewed Open Access Refereed Journal

Special Issue A3 January 2018

State level Conference on

Recent Approaches in Environmental Protection and Sustainable Development

(RAEPSD -2018)
19-20 January, 2018

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Organised by
Department of Chemistry
Dnyaneshwar Gramonnati Mandal's

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ARTS, COMMERCE AND SCIENCE COLLEGE
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Savitribai Phule Pune University, Pune, BCUD
Sponsored

State Level Conference
On

"RECENT APPROACHES IN ENVIRONMENTAL PROTECTION AND SUSTAINABLE DEVELOPMENT"
[RAEPSD-2018]

19th and 20th January 2018

Principal
Dr. Subhash K. Wadekar

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Message

It gives me a great pleasure to know that Department of Chemistry, Dnyaneshwar Gramonnati Mandal Ale's Hon. Balasaheb Jadhav Arts,Commerce and Science College, Ale is organizing a State Level Conference on "Recent Approaches in environmental Protection and Sustainable Development." sponsored by BCUD of Savitribai Phule Pune University on 19th and 20th January 2018.

The theme of this conference, recent approaches in environmental protection and sustainable development is very important in present situation. Industry is a major cause of air pollution, since the operation of factories results in the emission of pollutants, including organic solvents, respirable particles, sulfur dioxide and nitrogen oxides These pollutants can both harm public health and damage the environment by contributing to global phenomena such as climate change, the greenhouse effect, ozone hole depletion, acid rain and increasing desertification. I hope that this conference will help the students, teachers and delegates to discussion on application of green chemistry principles to achieve sustainable development.

I congratulate all the trustees of Dnyaneshwar Gramonnati Mandal, Ale, Principal Dr. Subhash Wadekar, Vice Principal Prof. Sanjay Wakchaure, Convener Prof. Jaising Gadekar, organizing secretary Dr. Bhujbal R.C. and the whole team of RAEPSD-2018 for organizing this conference and wish the State Level Conference a grand success.

Dr. V.B. Gaikwad,
Ex. Director, BCUD, SPPU, Pune
Principal,
KTHM College, Nashik.

From Principal's Desk....

Our College is one of the reputed College in Savitribai Phule Pune University. I have pride of our work culture and team spirit of Chemistry Department for successful planning, organizing conference and publishing articles in UGC approved International Research Journal. My best compliments to our best team of department of Chemistry. The department of chemistry of our College is organizing two days state level conference sponsored by BCUD. It gives me great pleasure to welcome eminent dignitaries, invitees, researchers, participants and students, to conference on "Recent Approaches in Environmental Protection and Sustainable Development" (RAEPSD-2018). I also congratulate the participants who attend and present their papers in this conference with best wishes.

We not only empower the students in this ever changing world but also aim to achieve the development of our society. New ideas, trends, technologies and methods are emerging for addressing new issues and problems. Thus strategies are to be identified to face these opportunities.

Interaction and exchanges of ideas through this conference will certainly contribute towards generation of knowledge which is extremely essential in the present context. It would create a platform for discussion, and innovative ideas.

The purpose of this conference is to provide a forum for exchange of ideas among faculty members, Research Scholars and students. We fulfill this mission through holding annual conferences, Seminars and publishing the edited proceeding and publishing the refereed journals.

Best complements to all participants and delegates with warm regards.
Thanking You,

Dr. Subhash K. Wadekar
(M.Com.,M.Phil.,M.B.A.,Ph.D.,D. Litt.)
Principal,
Hon.B.J.A.C.S.College,Ale.

I am very much glad to learn that Hon. Balasaheb Jadhav Arts, Commerce.& Science college, Ale, Junnar Dist. Pune, Department of Chemistry, organizing a **State Level Conference on Recent Approaches in Environmental Protection and Sustainable Development (RAEPSD-2018).**

It is praiseworthy to know that this Conference will provide the great opportunity to young researchers to share their ideas and views and learn from experienced experts.

I congratulate to Principal. Convener, Organizing Secretary and all local organizing committee members for conducting and organizing such conference at college level.

I am confident that this conference will be useful in expanding the knowledge of young researchers and also remain beneficial to student and society too.

With all my Best Wishes.

Dr. N. B. Patel

Professor & Head

Department of Chemistry

Convener's Message

It gives me an immense pleasure to welcome all the resource persons, delegates ,teachers and students from various academic and research institutes as well as industries on the occasion of State Level Conference on ,"Recent Approaches in environmental Protection and Sustainable Development" organized by Department of Chemistry, Dnyaneshwar Gramonnati Mandal Ale's Hon. Balasaheb Jadhav Arts,Commerce and Science College, Ale. It is a thing of great pleasure for me and my colleagues to present the special issue of 'International Research Journal of Science and Engineering' which contains the research papers of the participants of this State Level Conference.

The basic aim of this conference is to provide a common platform to students, teachers, researchers and industrialists to share a knowledge, experience and expertise regarding recent approaches in environmental protection and sustainable development. Today the earth and the life on the earth are under a threat of pollution. Every chemical industry is responsible for air, water and soil pollution. Population explosion, overexploitation of natural resources, global warming, climate change, extreme weather are the challenges before science and scientists. Such burning topics will be discussed in this conference. This will certainly create awareness about environmental degradation and the effects on life on the earth. What we need today is sustainable development and this conference will throw a light on the aspects of sustainable development.

I would like to acknowledge BCUD and Savitribai Phule Pune University for funding this conference. I am thankful to all the trustees of Dnyaneshwar Gramonnati Mandal Ale, Principal Dr. Subhash Wadekar and Vice principal Prof. Sanjay Wakchaure for their encouragement us. It's my privilege to be associated with a team of colleagues who always work with enthusiasm to make any event successful.

Prof. Jaising H. Gadekar
Convener, RAEPSD -2018 &
Head, Department of Chemistry.

Editorial....

Dnyaneshwar Gramonnati Mandala's, Hon. Balasaheb Jadhav Art's, Commerce and Science College, Ale, Tal-Junnar, Dist- Pune, Department of Chemistry, in association with Board of College and University Development (BCUD), SPPU, Pune, organized two days state level conference. It gives me an immense pleasure to welcome you all for **"Recent Approaches in Environmental Protection and Sustainable Development" (RAEPSD-2018)** hold on 19th and 20th Jan 2018.

I therefore, as Organizing Secretory, welcome Dr. V. B. Gaikawad (Ex. Director, BCUD, SPPU, Pune, Principal, KTHM College, Nashik) our chief guest for this conference. The conference is organized for discussion and sharing of various issues of environment and Chemistry among researcher and students. The conference will bring new innovative ideas and become helpful to researchers in various fields of science to carry out quality research. We would be greatly honored if you could attend the Conference and also look forward to your co-operation to motivate the faculty members, students and colleague's for their enthusiastic and encouraging participation along with the research papers/articles as we are publishing the conference proceeding which is in your hands.

I would like to acknowledge Board of College and University Development (BCUD), Savitribai Phule Pune University.

At this stage, I would like to express our sincere gratitude to Management of institution, Principal Dr. Wadekar Subhash.K., Vice Principal Prof. Wackchaure S.D., Head, Dept of Chemistry, Prof Gadekar Jaysing, for encouraging and guiding us. I would like to thanks also to all the Heads of various Departments and their faculty, my colleagues, nonteaching staff, students for their assistance and cooperation in organizing this conference. I would like to thank Mr. Sunil Bhujbal, Mr. Sachin Jatar for giving freely of their time and helping in many ways and all those who have helped in organizing this conference.

I, therefore, once again welcome one and all present here for the two day conference.

Dr.Ravindra. C. Bhujbal
Organizing Secretory,
RAEPSD -2018
Department of Chemistry.

Editorial Board

Recent Approaches in
Environmental Protection and Sustainable Development

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Pharmacological Review of *"Luffa acutangula* (L) Roxb

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Manuscript Details

Editor: Dr. Arvind Chavhan

Cite this article as:

ABSTRACT

Ourancestors looked for the drugs to rescue from diseases. Many synthetic drugs were discovered but they have fewer efficacies and may contradictions. Now a day's most of the herbal drugs were evolved with the great potentials. The fruit of one of such herb *Luffa acutangula* (L) roxb, belongs to family Cucurbitaceae is used as common vegetable. The fruits, seeds and plant extracts have been used as antidiabetic, hepato protective, antioxidant, antimicrobial, anticancer, antiulcer, CNS depressant etc. The intention of this article is to focus on variety of pharmacological uses of *Luffa acutangula*. The present review is based on the areas which are needed to have immediate attention for preparation of herbal drugs based on the pharmacological evidences.

Keywords: *Luffa acutangula,* Hepato-protective, Antidiabetic, Antioxidant, Antimicrobial, Antiulcer, CNS depressant.

INTRODUCTION

Plant plays an important role from ancient times where man used plant as drug to cure various diseases. About 2000 species of medicinal plantsin India have high potential values unfortunately; veryfew of them were studied chemically and pharmacologically for their medicinal benefit. *Luffaacutangula* (L) Roxb called as ridge gourd, angled gourd or angled Luffa is one of the potential herbs found in Asia. India is considered as a centre of its origin

This is common fruit vegetable cultivated in India, China, Southeast Asia, Egypt, Japan and other parts of Africa. The various pharmacological activities includes antidiabetic activity [4, 5], antioxidant activity, hepato-protectiveactivity [1], CNS depressant activity [3], anticancer [24, 25] etc. *Luffa acutangula (LA)*as a whole is the source of the secondary metabolites i.e. amino acids, carbohydrates, proteins, flavonoids, anthraquinones, terpenoids, tannins, and saponins[6]. Seeds of this plant shows presence of saturated and unsaturated fatty acids, linoleic, palmatic, stearic, oleic and traces of lignoceric acid while fruits contain cucurbitacin B, E and oleanolicacid [7]. Although many activities of *L. acutangula*were reported butit's potential as a pharmacological aid has to be studied for more applications.

PHARMACOGNOSY

Scientific Classification
Kingdom: Plantae

   Division: Magnoliophyta

       Class : Magnoliopsida

           Order : Cucurbitales

               Family:Cucurbitaceae

                   Genus : Luffa

                       Species: *L. acutangula*

Other Indigenous Names:
Assamese: Zika.
Bengali   **:** Jhinga, Ghoshalata, Titotorai, Titojhinga, Titodhundal.
Chinese   **:**Ling Jiao Si Gua, You Lin Si Gua, Sze Gwa, Sigw,sin qua or sing kwa.
English: **Angled** loofah, Chinese okra, Dish-clothgourd, Ridge gourd, Ribbed loofah, Silk gourd, Silky gourd, Sinkwatowelsponge, Sinquamelon,Vegetable sponge.
Gujarati   **:**Turiya.
Hindi   **:**Jhimani,Karvitarui, antorai,Sankirah.
Japanese   **:**itouri, tokadohechima.
Kannada   **:**Heere, Kahire, Kahi, Naagadaaliballi.
Malayalam: Athanga.

Marathi   **:**Divali,   Kadudodaki,   Kadudod-ka, Kadushirali, Kaduturai, Ranturai.
Odia   **:** Janhi.
Punjabi **:** Jhinga,Shirola
Sanskrit : Gantali,Kosataki,Ksweda, Mridangaphalika, Sutikta.
 Tamil **:** Itukari, Itukarikkoti, Kacappi, Kacappuppirkku, Kaccam,Karniti, Karnitikkoti,Katiyavisava, Kirutacittirar, Kirutavetanai, Peerkangai.
 Telugu : Adavibeera,Chedubira,Chathibeera, Sendubirai, Verribeera

DESCRIPTION

*Luffa acutangula*is a climber.
Roots: Roots of LA plants are yellowish- Brown in colour, almost cylindrical in shape, having 8-12cmlength and 0.7cm thickness. They are rough in touch because of longitudinal wrinkles and also showed new adventitious roots.
Stems:It is with 5 angled glabrous stem and 3-fid tendril.
Leaves : Leaves of LA are orbicular in outline with the length 15-20 cm long, palmately 5-7 angled or sublobate, scabrid. Veins and vein lets are prominent.
Fruits : The fruits of *LA are* obovate, pale yellowish brown in colorwith 4-10 cm long, 2-4cm broad and outer surface being covered with 8 -10 prominent longitudinal ribs. It is tapering towards the base and longitudinally ribbed. The fruits are divided into 3 chambers. The inner part is fibrous in nature and easily detachable as a whole from the outer part. Taste is bitter. Transverse section of fruit through a rib shows a single layer of papillose epidermis covered with thick striated cuticle, followed by 4-6 layers of parenchymatous cells.
Seeds **:** Seeds are black in colored with bitter taste and having ovoid- oblong shape. The length is generally 0.6-0.8cm with width of 0.5-0.6cm.
Distribution : *Luffa acutangula* cultivated throughout India as it is pan tropic.
Habit: Herb.
Propagation: Propagation of *L.acutangula*is byseeds.
Native range: India and Naturalized throughout the moister Tropics.
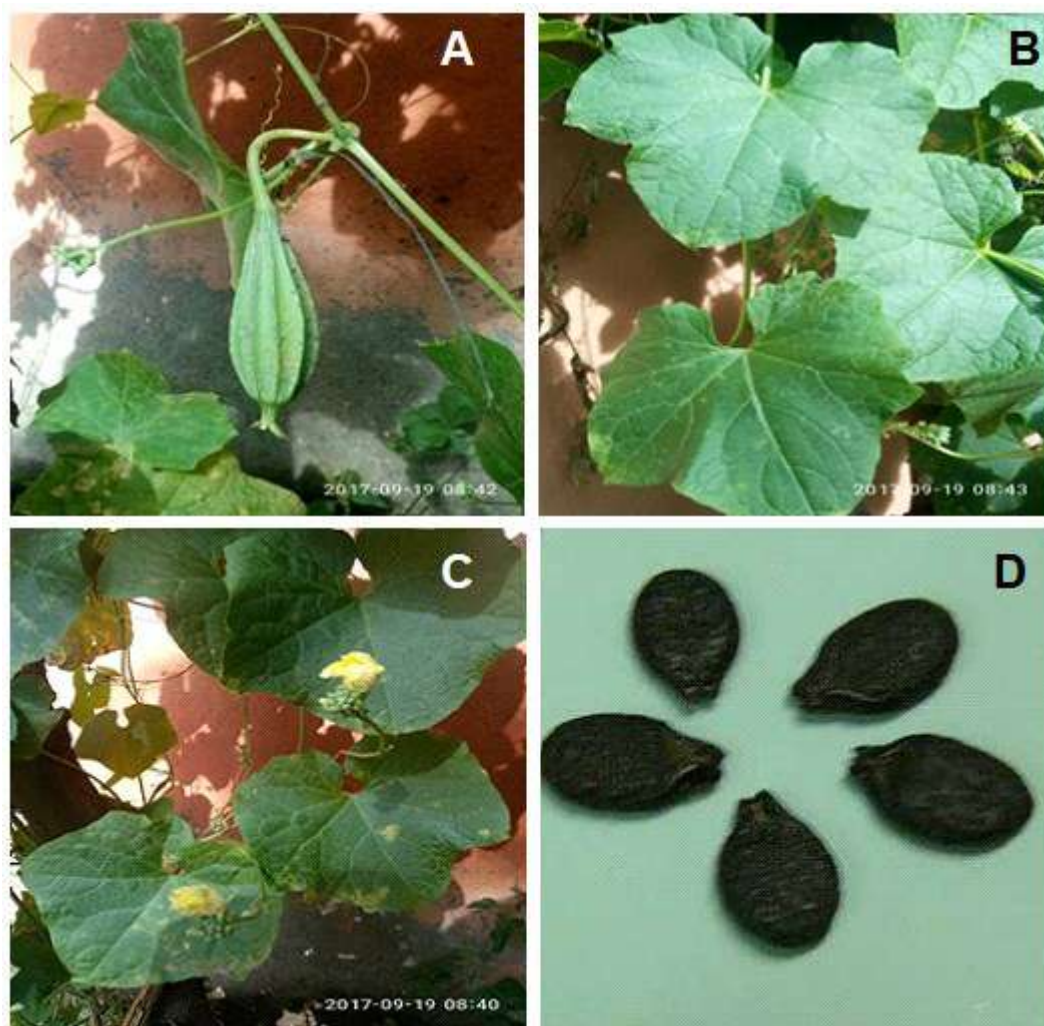
Fig. 1 A: Fruit of *Luffa acutangular*   **B**: Leaves of *Luffa acutangular*
C: Flowers of *Luffa acutangular* **D:** Seeds of *Luffa acutangula*

Phytochemical Review

The quality controlled studies on fruit of *Luffa acutangula*shows that water has maximum extractive power as compared to other solvents such as hexane, ethanol, chloroform, ethyl acetate. The LA extracts when examined for the following test for purity e.g. acid insoluble ash, loss on dryingwater soluble and sulphated ash shows that sample is rich in drug having large polar compounds. Simple preliminary analysis reveals that these extracts contain carbohydrates, reducing sugars, alkaloids, saponins, steroids, glycosides, phenolic compounds, flavonoids, quinines, tannins and lignin's. The flavonoids and tannins are important in treating the cancers and heart related problems, so it is suggested that it is included in dietary supplement for such patients.

The XRF and FPM study indicates that the plant is good source of Ca, Mg, K and P. It also contains moderate concentration of Si and S. The entire elements play an important role in efficient and smooth biological functioning. LA hexane extract on GC-MS analysis indicate that it contain forty-five different class of compounds viz.Hexadecanoic acid ethyl ester, 9, 12, 15-octadecatrienoic acid, 3, 7, 11, 15-Tetramethyl – 2 – hexadecenl-ol, 9, 12 octadecanoic acid ethyl ester, Hentriacontane and Hexadecanoic acid methyl ester while GCMS of chloroform extract indicatethat it contain 35 different compounds such as n-Hexadecanoic acid, Triacontane,Tetratriacontane, 9, 12, 15-octadecatrienoic acid methyl ester, (z, z, z), citronellyltiglate. Different spot under HPTLC at 366nm with different Rf values differentiate the drugs [8].

Nutritional Composition

The seeds and fruits of *Luffa acutangular* were studied for potential nutritional and oil characteristics. The fatty acid profile of seeds indicates that the glycerides of oleic and linoleic acid constitute 68% of the total kernel oil. The seeds were also found to be a good source of amino acids, phosphorous, iron and magnesium. The fruit is a major source of moisture, crude fiber, vitamin B, calcium, iron and magnesium [9].

Ethno pharmacology Review

The ethno pharmacological survey in Rajasthan, Katewa et al., (2008) collected the information on medicinal uses of plants. They found that half tea spoon of LA seeds powder plays an important role if given orally with water for 3–4 days for urinary bladder stone [10].

The tribal's of Western Madhya Pradesh of India uses this plant for the treatment of jaundice. They enlisted 13 plants as drug in common practice as herbal drug. Among these the extensively used herb was found to be LA. The LA commonly known as Kadwi Turai, and their leaves and fruits were used for treatment of jaundice. The 2-3 drops of leaf or green fruit juice without using water are administered into one of the nostrils for 4successive days [11].

Kanaka et al, (2013), during their ethno botanical survey in Reserve Forests of Mahadevpur Karimnagar, AP they noticed that LA fruit were widely used by local inhabitants for treatment of Diabetes [12] also.

Pharmacological review on LA Hepato-protective activity

The aqueous ethanolic extract (Hydro alcoholic extract) of LA was tested for hepatoprotective activity by using silymarin as a standard reference drug for comparison. The Hepatoprotective action of this extract was due to thedecreased levels of serum marker enzymes (AST, ALT, ALP and LDH) and increased total protein including the improvement in histo-architecture of liver cells of the treated groups as compared tothe control group. LA extract showed mark decrease in malondialdehyde (MDA) formation, increased activity of non-enzymatic intracellular antioxidant, glutathione and enzymatic antioxidants,

catalase and superoxide dismutase. The result of this study indicates that hepatoprotective activity of hydro alcoholic extract of LA was due to contribution of endogenous antioxidants and inhibition of lipid per oxidation of membrane. Investigations were made to study hepatoprotective activity of saponin fraction of *LA* seeds in liver fibrocytic rat-induced with CCl4male.Wistar rats were divided into six groups consisting of normal group, control group, comparator group and three test groups (given saponin fraction of *Luffa acutangula*). The study concluded that administration of saponin fraction at dose 10 mg/kg b.w twice a week, 20 mg/kg b.w twice a week and 20 mg/kg b.w once daily showed hepatoprotective activity and the highest effect was showed at a dose of 20 mg/kg b.w once daily. Thus, saponins present in LA extract plays an important role in prevention of oxidative hepatic damage. The reactive oxygen species is stabilized by the flavonoids and saponins which are present in the LA extract [1].

CNS depressant activity and cerebro-protective effects

The study of evaluation of protective effect of *Luffa acutangular* extract against bilateral carotid artery occlusion (BCAO) induced stroke in rats by Sathianarayanan. S (oct 2012) confirmed that ELA protects rats from ischemia induced brain injury. This protection was manifest from in-vivo behavioral tests. Petroleum ether extracts of Whole plant of *Luffa acutangular* produced cerebroprotective effects in global cerebral ischemia as evident from reduction in behavior pattern, hyper locomotion and neuronal damage [2].

The allied species of *Luffa echinate* Roxb has reported to possess CNS depressant activity and alkaloid isolated from it acts as important local anesthetic and antispasmodic action. On this basis A.V. Misar (2004) studied the CNS depressant activity of the ethanolic extract of LA fruit in mice. The extract shows the effect on behavioral changes, exploratory activity, and barbiturate sleeping time in mice. The extract exhibited dose-dependent CNS depressant activity [3].

Antibacterial and Antifungal Activity

It is found that LA fruit methanolic extract prevent the growth of *B. Subtilis, S. Aureus, P. aeruginosa* and *E.*

coli, but did not prevent the growth *of E. Aerogenes* and *S. Thypi*. The other extracts of LA fruits which had the highest antibacterial activity were ethyl acetate extract followed by chloroform, butanol and hexane extracts. Chemical screening indicates that extract does not contain a class of alkaloid, saponin and terpenoid compounds except hexane extracts contained only terpenoid compounds. Antibacterial activity of ethyl acetate extract of LA fruits is less when it is compared with Ampicillin [13].

The comparative study of display aqueous fruit extract of LA shows more better antibacterial activity as compared to the leaf extract of LA. Among fungi *Curvularialunata* was found highly sensitive to leaf and fruit extract of LA while to same extract *Phomasorghina* showed poor sensitivity [14].

The silver nano-particles prepared using leaves extract of LA shows the antibacterial activity against the human pathogenic bacteria such as Escherichia coli, Klebsiella pneumonia, Proteus vulgaris, Pseudomonas aeruginosa, Salmonella paratyphi and Staphylococcus aureus. The zone of inhibition was higher in Klebsiella pneumonia and zone of inhibition lower in *Staphylococcus aureus* [15].

Antidiabetic Activity
The antidiabetic activity of *Luffa acutangular* fruit extracts in streptozotocin induced NIDDM rats**.** The study reveals that both the methanolic extract (LAM) and aqueous extract (LAW) shows activity. The LAM extract was found to be superior to LAW extract in management of diabetes and its associated lipid imbalance. The methanolic extract shows the significant activity (p<0.01) at a dose of 200 and 400 mg/kg. Both LAM and LAW appeared to be beneficial in treatment of type-II diabetes mellitus. The extracts act as inhibitors for suppressing postprandial hyperglycemia in the management of Type II diabetes [4].

Multicomponent formulation of herbal drugs for evaluation of antidiabetic activity was studied by Harpreet Singh (2014). The formulation of hydro-alcoholic extract of *Luffa acutangula* with *Madhuca longifolia* shows the antidiabetic activity. The formulation was design in solid dose form is used as

tablets. Significant (p < 0.05) reduction in fasting blood glucose levels were observed in the normal as well as in the treated diabetic animal. The activity was evaluated in alloxan monohydrate induced diabetic model [5].

Ameliorative Effect
The ethanolic extract suspended in the carboxy methyl cellulose called HAELA of *Luffa acutangula*shows ameliorative effect against doxorubicin induced cardiac and nephrotoxicity in mice. There is major decrease in certain enzymes such as serum biomarkers, lactate dehydogenase, alanine amino transferase and creatinine phosphokinase in kidney and heart in doxorubicin treated mice. It also plays an important role in restoring the low levels of catalase, glutathione and superoxide dismutase in the tissues of kidney and heart. The HAELA plays an important role in membrane stabilization in doxorubicin induced cardiac and nephrotoxicity in mice [16].

Antioxidant Activity
Antioxidants are compounds which protect cells from the damage caused by unstable molecules known as free radicals. The antioxidant activity of methanolic extract and its derived fractions of LA fruit by β-carotene bleaching method. Statistical analysis shows that antioxidant activity decreases in the order: Residual aqueous fraction (LAA)> n-Butanol fraction (LAB)> Methanol Extract (LAM)> Ethyl acetate fraction (LAE) >Chloroform fraction (LAC) >n-Hexane fraction (LAH)

When all evaluated with β-carotene bleaching method. This study shows that the antioxidant activity is not totally related to the phenolic content but other fractions also i.e. presence of carotenoids in those fractions [17]. In Vitro antioxygenic activity of Ridge Gourd (*Luffa acutangula*) Pulp, Peel and their extracts on per-oxidation Models.LA peel powder and its extracts showed slightly higher antioxygenic activity than LA pulp powder and its extracts. This may be due to higher amounts of phenolics and flavonoids contain [18].

DPPH assay studied with the five different extracts of LA. The vegetable peel aqueous extract showed comparatively more radical scavenging activity (24.71

%) followed by ethanol (18.87), acetone (13.05), methanol (11.13) and ethyl acetate extracts (7.14) respectively. Aqueous extract was found to be a potent free radical scavenger with IC50 value 0.05 µg mL-1 followed by ethanol (0.07 µg mL-1) in another study of DPPH radical-scavenging assay. The radical-scavenging activity LA fruit pericarp was estimated by comparing IC50 of the extracts and those of ascorbic acid. The radical scavenging ability was found to be in descending order as: Ethanol extract>Ethyl acetate extract>Aqueous extract>Petroleum ether extract

The ethanolic extract of this plant shows the greater antioxidant activity than others but less than ascorbic acid [19]. The photochemical study of LA seeds shows that it contains carbohydrates, triterpenoid, flavonoids coumarin glycoside and phenolic compounds. Naresh sing gill and his colleague (2011) evaluated the antioxidant activity of ethanolic extract of seed of LA by DPPH and hydrogen peroxide method. The ethanolic extract at concentration 200 µg mL-1 shows 75.33 ±0.592 by DPPH model and 76.50 ± 0.281 by Hydrogen peroxide method as compared to ascorbic acid method. The reduction in free radical scavenging is measured by determination of their absorbance at 517 and 230 nm wavelengths. Antioxidant activity increases with increasing concentration of extract [20]. The oil extracted from seeds of LA by petroleum ether shows antioxidant activity by DPPH method. The radical scavenging activity was compared with the standard butylated hydroxytoluene (BHT) [21]. In another study carried out in Bangladesh by Israt Jahan Bulbul LA leaves extracts of solvent in n-hexane, chloroform, ethyl acetate shows greater antioxidant activity with the concentration range 50.95 µg mL-1 ,57.81 µg mL-1, 51.77 µg mL-1 as compared with the standard ascorbic acid concentration 43.22µg mL-1 by DPPH radical scavenging activity method[22]. Out ofhexane, methanolic and aqueous extract of LA fruit, the methanolic extract shows higher antioxidant activity by DPPH model. Further partially purified methanolic extract antioxidant activity was evaluated in lipid per-oxidation, superoxide dismutase, catalase and glutathione in t-butyl hydro peroxide (t-BHP) treated-erythrocytes [23].

Anticancer Activity

Luffa acutangular have been reputed to possess an anticancer activity. In vitro anticancer effect of LA leaves extract in human lung cancer cell line (NCI-H460) was studied. The anticancer activity was compared with that of paclitaxel as positive control and the effect was assessed by MTT assay, mitochondrial membrane potential (MMP) alteration, intra-cellular reactive oxygen species (ROS) measurement and nuclear morphology with the appearance of apoptosis. This extract displays high anti proliferative activity against cell line tested, as determined with MTT assay. The concentrations of growth inhibition at 50% (IC50) ranges from 1 to 50 µg/mL after 24 h treatment and the IC50 was found to be 20 µg/mL for LA. Further study is needed to formulate natural compounds with anticanceractivities [24]. Anticancer activity of ethanolic and aqueous extracts of *Luffa acutangular* was evaluated in Ehrlich Ascites Carcinoma (EAC) Swiss albino mice at the doses of 200 and 400 mg/kg body weight orally. Antitumor effect of both extracts was determined by evaluating tumor volume, viable and nonviable tumor cell count, tumor weight and hematological parameters of EAC bearing host. Ethanolic and aqueous extracts showed significant decrease in (p<0.0001) tumor volume, viable cell count, tumor weight and elevated the life span of EAC tumor bearing mice. The investigation of Blood reveals that red blood cell (RBC), hemoglobin, and white blood cell (WBC) count reverted to normal level in treated mice. The study manifest that the extract has potent dose dependent anticancer activity compare to cisplatin. Aqueous extract at 200mg/kg and 400 mg/kg dose and ethanolic extract at 400 mg/kg dose showed potent anticancer activity [25].

Analgesic Activity

The analgesic activity of *Luffa acutangular* was studied with the help of ethanolic extract on adult albino rats using tail flick method and tail immersion method. This extract showed significant activity with the reaction time of 6.25±0.52 in tail flick method and 5.80±0.50 in tail immersion method at the dose of 400 mg/ml as compared to pentazocin (standard). The analgesic activity is may be due to its free radicals scavenging activity and central modulation of pain by dopaminergic, noadrenergic and seratonergic systems [26].

Antiulcer Activity

The gastro protective effect of *Luffa acutangular* methanolic and aqueous extracts on gastric ulceration in NIDDM rats was studied by inducing type II diabetes with help of *Streptozotocin* (65mg/kg, i.p.) along with nicotinamide (120 mg/kg, i.p.) and gastric ulceration to diabetic rats was induced by aspirin. LA methanolic extract shows significantly ($P<0.01$) increased mucosal glycoprotein and antioxidant enzyme level in gastric mucosa of diabetic rats than LA aqueous extract ($P <0.05$). Methanolic extract of LA was found to be efficient in reversing delayed healing of gastric ulcer in diabetic rats close to the normal level. LA methanolic extracts exhibit both anti-hyperglycemic and mucosal defensive actions it is better in ulcer healing than glibenclamide and LA aqueous extract. This shows that methanolic extract of LA is a better alternative for treating gastric ulcers co-occurring with diabetes [27].

Cytotoxic and Anthelmintic activity

The LA plant methanolic and petroleum ether extract of aerial parts shows cytotoxic and anthelmintic activity. The activity was evaluated by Brine Shrimp lethality bioassay and anthelmintic activity by in-vitro test using earth worm *Pheretimaposthuma* (Annelida) as test animals. The methanolic and petroleum extract shows the moderate cytotoxic and anthelmintic activity [28].

CONCLUSION

The detailedpharmacologicalreview it is revels that the plant is apotential source of drugs that may be used as a dietary supplement in the treatment of liver related diseases. Apart from this, the plant has to be standardized forpharmacological applications. The most of the research work reported on fruits of LA and less on other plant parts such as roots, Leaves, flowers and seeds. LA hashepato-protective, CNS depressant, antibacterial, antifungal, antidiabetic, ameliorative effect, antioxidant, analgesic, antiulcer activity, anticancer, cytotoxic and anthelmintic activity. Thus, indicates the need of pharmacological evaluation with phytochemical correlation. It further reflects a hope for the development of bioactive molecules that may be used as an alternative medicine for various diseases.

Conflicts of interest: The authors stated that no conflicts of interest.

REFERENCES

1. Jadhav VB, Thakare VN, SuralkarAA, Deshpande A D, Naik S R,Hepato-protective activity against CCl4 and Rifampicin induced liver toxicity in rats. Indian Journal of Experimental Biology, 2010;48:822-829.
2. Sathianarayanan. S, Asha Jose, Rajasekaran.A, Rijo Mary George, Amrutha.B.Chittethu, Evaluation of protective effect of *Luffa acutangula*extractagainst bilateral carotid artery occlusion (BCAO) induced stroke in rats. Indian Journal of Pharmaceutical Science & Research, 2012; 2(1):1-6.
3. Misar AV, CNS depressant activity of ethanol extract of *Luffa acutangula* CB Clarke fruits in mice. Indian journal of pharmaceutical sciences, 2004; 66(4):463-5.
4. Pimple BP, Kadam PV, Patil MJ. Antidiabetic and antihyperlipidemic activity of *Luffa acutangula*fruit extracts in streptozotocin induced NIDDM rats. Asian Journal of Pharmaceutical and Clinical Research, 2011; 4(2):156-63.
5. Harpreet Singh, Sudhanshu Arora, Munish Mani, Kamal Kr Mahaur, Phool Chandra. Development of multi-component formulation of herbal drugs for evaluation of Antidiabetic activity, Der Pharmacia Lettre, 2014; 6 (1):219-223.
6. Ananthalakshmi R, Xavier SR Rajarathinam, and Mohammed Sadiq A. Preliminary Phytochemical Screening of Aqueous Extract of Various Parts of *Luffa acutangula* (Ridge Gourd). *International Journal of Latest Engineering and Management Research (IJLEMR)* 2017; 02(07): 01-04.
7. Rastogi RP and Mehrotra BN, Ed. *Compendium of Indian medicinal plant, 5thEdn. CSIR,* Luckhnow: 2001:503-504.
8. Manikandesalvi S, Brindha P, Quality Control Studies on of *Luffa acutangula. International Journal of Pharmacy and Pharmaceutical Sciences* ISSN- 0975-1491 2014;6.
9. Jadhav Santosh Jaysingrao, Chavan Niranjana Sunil, Nutritional assessment of fruits of *var.* amara., *International Journal of Science and Research (IJSR)* Volume 3 Issue 10, October 2014 (2012).

10. Katewa SS. Chaudhary BL. Jain A, Folk herbal medicines from tribal area of Rajasthan, India, *Journal of Ethnopharmacology*. 2004 May 31; 92(1):41-6.

11. Samvatsar S, Diwanji VB. Plant sources for the treatment of jaundice in the tribal's of Western Madhya Pradesh of India, *Journal of Ethnopharmacology*.2000 Nov 30; 73(1):313-6.

12. Kanaka RC. Narasinga RN. Venkateshwarlu M. Sammaiah D. Anitha U. UgandharT. Studies on the medicinal plant biodiversity in forest ecosystem of Mahadevpur forest of Karimnagar (A.P.) India, Bioscience Discovery, 2013;4(1), 82.

13. Resmi Mustarichie, Linar Zalinar Udin, Muchtaridi, Supriyatna. Identification and antibacterial activity of methanol extract of *Luffa acutangula*roxb, Medical and Health Science Journal, MHSJ, 2012;12:70-77.

14. Dandge, VS. Rothe, SP. Pethe, AS., Antimicrobial activity and pharmacognostic study of *Luffa acutangula* (l) Roxbvar*amara*on some deuteromycetes fungi. *International Journal of Science Innovations and Discoveries,* 2012; 2(1): 191-195.

15. Syed Moideen R and Lakshmi Prabha A, Green synthesis of silver nano particles using *Luffa acutangula*roxb. Var. amara. Lin and its antibacterial activity 2014; 5(4): 1051-1061.

16. Jadhav, VB, Thakare VN, Suralkar AA, and Naik SR. Ameliorative effect of *Luffa acutangula* Roxb. on doxorubicin induced cardiac andnephrotoxicity in mice. *Indian journal of experimental biology,* 2013;*51:* 149-156.

17. VentyS uryanti, Soerya Dewi Marliyana and TikaWulandari. Antioxidant activity, total phenolics and flavonoids contents of *Luffa acutangula* (L.) Roxb fruit *Journal of Chemical and Pharmaceutical Research*, 2015; 7(1):220-226.

18. Ananthan Padmashree, Gopal Kumar Sharma, Anil DuttSemwal, Amarinder Singh Bawa, In Vitro Antioxygenic Activity of Ridge Gourd (*Luffa acutangula*) Pulp, Peel and Their Extracts on Peroxidation Models *American Journal of Plant Sciences* 2012;3(10):24127,4.

19. Swetha MP, Muthukumar SP, Characterization of nutrients, amino acids, polyphenolsand antioxidant activity of Ridge gourd (*Luffa acutangula*) peel J Food SciTechnol, July 2016; 53(7):3122–3128.

20. Gill NS. Arora R., Evaluation of Antioxidant, Anti-inflammatory and Analgesic Potential of the *Luffa acutangula*Roxb. var. *amara*, *Research Journal of Phytochemistry*. 2011; 5(4):201-8.

21. Kalyani GA, Ramesh CK, Krishna V, Extraction and characterization of *Luffa acutangula*var*amara*seed oil for antioxidant activity, *International Journal of Research in Ayurveda & Pharmacy*.2011 Sep 1; 2(5).

22. Israt Jahan Bulbul, Abu Hasanat Md. Zulfiker, Kaiser Hamid, Mst. HajeraKhatun, and Yesmin Begum, Comparative Study of In vitro Antioxidant, Antibacterial and Cytotoxic Activity of Two Bangladeshi Medicinal Plants-Luffa cylindrica L. and *Luffa acutangula*.Pharmacognosy Journal July 2011;3(23): 59-66.

23. Boreddy Purushotham Reddy, S. Venkata Mohan and P. N. Sarma, The Protective role of *Luffa acutangula*fruitMethanolic Fraction against *t*-BHP Induced Oxidative damage in Human Erythrocytes Current Trends in Biotechnology and Pharmacy 2011;5 (1):1073-1082.

24. Vanajothia R, Sudhaa A, Manikandanb R, Rameshthangam P and Srinivasana P, *Luffa acutangula*and *Lippianodiflora* leaf extract induces growth inhibitory effect through induction of apoptosis on human lung cancer cell line Biomedicine & Preventive Nutrition, Volume 2, Issue 4, October–December 2012: 287-293.

25. Dashora N, Chauhan LS. In vitro antioxidant and in vivo anti-tumor activity of *Luffa acutangula*against Dalton's Lymphoma Ascites (DLA) cells bearing mice Journal of Chemical and Pharmaceutical Research, 2015; 7(6):940-945.

26. Fernandes L C B and Corderio L A V, Evaluation of abortifacient effect of *Luffaacutangula*Roxb. in rats. Journal of Animal and Veterinary Advances, 2010;9(8):1255-1258.

27. Pimple BP. Kadam PV. Patil MJ., Protective effect of *Luffa acutangula*extracts on gastric ulceration in NIDDM rats: role of gastric mucosal glycol-proteins and antioxidants, *Asian Pacific Journal of Tropical Medicine*. 2012 Aug 31; 5(8):610-5.

28. Md. Mosiqur Rahman, Ashik Ahmed, Saibal Saha Sunny, Md. Samiul Huq Atanu, Abdullah Faruque and Md. Sohel Rana., In-vitro Evaluation of Cytotoxic and Anthelmintic Activity of *Luffa acutangula*, *Luffa aegyptiaca* and *Momordicacochinchinensis*.British *Journal of Pharmaceutical Research* 2014;4(2): 267-277.

RESEARCH ARTICLE   **OPEN ACCESS**

Apparent molar volumes of Aspirin in water at temperatures from 298.15 to 313.15 K

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ABSTRACT

Apparent molar volumes (ϕ_v) and viscosity B-coefficients for aspirin solutions in pure water system have been determined from density (ρ) and viscosity (η) measurements at 298.15 to 313.15 K using a pycnometer and Ubbelohde viscometer respectively. Eight different concentrations ranging from 0.0040 to 0.0145 M were prepared. The apparent molar volumes were calculated from the density data. In addition, the concentration dependence of the apparent molar volumes was examined using Masson's equation. The Jones-Dole equation was used to analyze viscosity data to obtain viscosity 'A' and 'B' coefficients. The drug interacts with various ions or molecules or biological membranes present in the biological system are an important phenomenon. The parameters derived from these equations have been interpreted in terms of solute-solute and solute-solvent interactions.

Keywords: Aspirin, density, viscosity, B-coefficient.

INTRODUCTION

The partial molar volume and the related volumetric parameters of drug compounds in dilute aqueous solutions at different temperatures and pressures have been investigated by several authors [1-4]. It is well known that physicochemical characterization of drugs plays a crucial role in all the stages associated to design and development of pharmaceutical dosage forms, especially those intended to parenteral administration [5].

In this context, as a contribution to generation and systematization of physicochemical information about drugs' aqueous behavior, the main goal of this study was to evaluate the effect of concentration and temperature on the apparent molar volume of drugs in water. With that purpose, an interpretation in terms of solute-solvent interactions based on the corresponding volumetric behavior was developed.

Drugs of the analgesics, antipyretics, and anti-inflammatory class include a heterogeneous group of compounds. Many of these agents affect pain, fever and inflammation and are referred to as the non-steroidal anti-inflammatory drugs (NSAIDS).

Non-narcotic analgesics have three important properties namely analgesics, antipyretics and anti-inflammatory (e.g. Aspirin and paracetamol). The non-narcotics (salicylates) are called aspirin like or Non-steroidal Anti-inflammatory Drugs (NSAIDS). These drugs have common mechanism of inhibiting the cyclooxygenase (COX), the key enzyme responsible for biosynthesis of Prostaglandins (PG).

Bio-pharmaceutics is the study of factors influencing the extent and rate of absorption. The rate and amount of drug absorption depends on biological and physicochemical factors. During their way to site of action, drug molecules have to cross one or more membranous barrier, which are lipoidal in nature and have different size of pores.

Physicochemical factors include lipid solubility, salt complexation, dissolution rate, Viscosity and drug stability in GIT. Lipid soluble drugs more unionized and easily absorbed Na and K salts of weak acid have higher absorption rate than acids.

All the drugs in any solid dosage form or suspension when administered will first change into drug solution in body fluids. So, dissolution rate is important factor affecting the rate of absorption. When a drug is more rapidly or completely absorbed from solution, it is very likely that its absorption will be dissolution limited.

Viscosity limits the dissolution rate and there by affect the rapid absorption. Eg. Aqueous Solution of Na-Salicylate showed its rapid appearance in plasma while the same drug in suspension form failed to reach the target as quickly as with aqueous solution [6].

The study of the volumetric behavior of electrolytes in solution provides information useful to elucidate ion–ion, ion–solvent, and solvent–solvent interactions. The concentration dependence of the apparent and partial molar volumes can be used to study ion–ion interactions, whereas the partial molar volumes at infinite dilution provide information on ion–solvent interactions. The data reported here were obtained by performing density measurements on aqueous solutions of aspirin.

METHODOLOGY

Materials:
Aspirin of high purity was recrystallized and then used. Deionized water with a specific conductance of $< 10^{-6}$ S.cm^{-1} was used for the preparation of solutions at room temperature in a molarity range $(4.0 \times 10^{-3}$ to $1.45 \times 10^{-2})$ mol.L^{-1}. The precision of balance used was $\pm 1 \times 10^{-5}$g.

Density measurements:
The pycnometer was calibrated by measuring the densities of triple distilled water. The densities of distilled organic liquids like acetone, alcohol, benzene, carbon tetra chloride, aniline, and nitrobenzene were evaluated with respect to density of water. The density was measured with an uncertainty of $\pm 1.48 \times 10^{-4}$g.cm^{-3}.

Viscosity measurements:
The solution viscosities were measured with an uncertainty of $\pm 2.4 \times 10^{-4}$ mPa.s by using Ubbelohde viscometer. The viscosity measurements were performed at 25, 30, 35, 37 and 40°C. The temperature of thermostat is maintained to desired temperature, by using demerstat with an accuracy of ± 0.1 K. The flow time will be measured at the accuracy of ± 0.01 s.

The different compositions (0.0145M to 0.0040M) of solutions of aspirin were prepared in pure water. The

viscosities were measured at 25, 30, 35, 37 and 40°C for seven different concentrations.

DATA EVALUATION

The apparent molar volumes, ϕ_v, were obtained from the density results using the following equation[7-10]

$$\phi_v = \frac{1000\,(\rho_0 - \rho)}{C\rho_0} + \frac{M_2}{\rho} \qquad (1)$$

where M_2, C, ρ and ρ_0 are the molar mass of the aspirin, concentration (mol.L^{-1}), and the densities of the solution and the solvent, respectively.

The apparent molar volumes (ϕ_v) were plotted against the square root of concentration ($C^{1/2}$) in accordance with the Masson's equation[11]

$$\phi_v = \phi_v^0 + S_v \cdot C^{1/2} \qquad (2)$$

Where ϕ_v^0 is the limiting apparent molar volume and S_v a semi-empirical parameter which depends on the nature of solvent, solute and temperature.

The viscosity results for the aqueous solutions of drugs were plotted in accordance with Jones-Dole equation [12]

$$\frac{\eta_r - 1}{C^{1/2}} = A + BC^{1/2} \qquad (3)$$

Where $\eta_r = (\eta/\eta_o)$ and η, η_o are viscosities of the solution and solvent respectively, C is the molar concentration. The linear plots for $(\eta_r - 1)/C^{1/2}$ versus $C^{1/2}$ were obtained for the aspirin. The B-coefficients were obtained from the linear plots using the least-square fitting method. The A- coefficient reflects solute-solute interaction[13] and the B-coefficient reflect the solute-solvent interactions.

RESULTS AND DISCUSSION

The values of the densities (ρ) and apparent molar volumes (ϕ_v) of aspirin solution in pure water at 298.15, 303.15, 308.15, 310.15 and 313.15K temperature

are shown in Table 1. In all sets the densities and ϕ_v values of solutions increases with increase in concentration of solution. Figure 1 shows the linear plots of ϕ_v vs $C^{1/2}$ for aspirin solutions in pure water at different temperatures. Masson's parameters Φ_v^0 and S_v were obtained from linear plots are reported in table 2. The Φ_v^0 values are negative and S_v values obtained are positive for the systems studied furnish important information regarding the solute-solvent and solute-solute interactions.
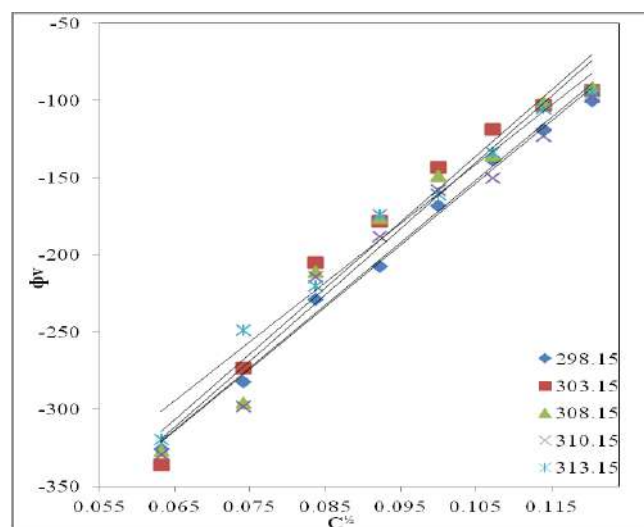
Fig 1: Plot of Φ_v (cm^3.mol^{-1}) Vs $C^{1/2}$ (mol$^{1/2}$.dm$^{-3/2}$) for aspirin solutions in pure water at temperature range T= (298.15 to 313.15K).
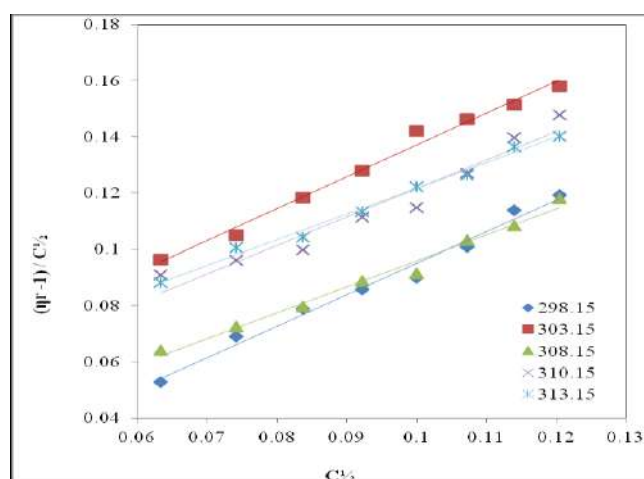
Fig2: Plot of $(\eta_r - 1)/C^{1/2}$ versus $C^{1/2}$ for aspirin solutions in pure water at different temperatures.

The values of the viscosity and relative viscosities of aspirin in pure distilled water at 298.15, 303.15, 308.15, 310.15 and 313.15K temperature are given in Table 3.

In all sets the viscosities of solutions increases with increase in concentration of solution, while viscosity decreases with increase in temperature. Figure 2 shows the variation of $(\eta_r-1)/C^{1/2}$ against square root of concentration at different temperatures in pure water.

'A' is constant independent of concentration and 'B' is Jones-Dole coefficient represents measure of order and disorder introduced by solute into the solution; positive 'B' coefficient shows strong alignment of solvent towards solute and is related to the effect of the ions on the structure of water[14]. The Jones-Dole parameters are given in Table 4. The positive values of 'B' at all temperatures indicate water structuring[15]. The values of B-coefficient decreases with increase in temperature as shown in fig.3 to indicate structure promoting tendency of the compound[16].
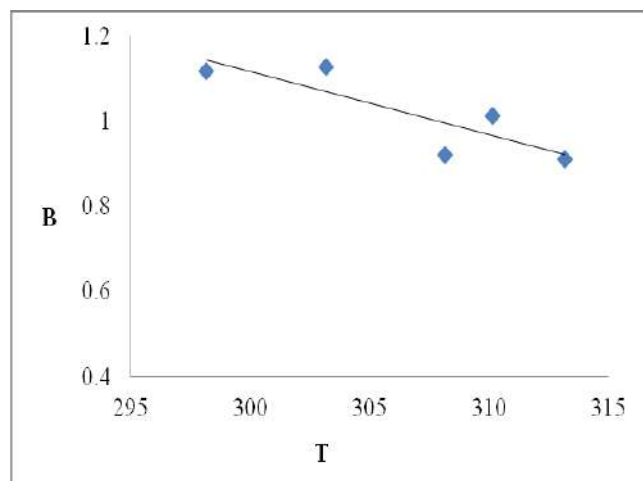
Fig 3: Plot of B-coefficient versus temperature for aspirin solutions in pure water at different temperatures.

Table 1: Densities and apparent molar volumes of aspirin solution in pure water at different temperatures.

Molar Conc. of aspirin (C) mol/dm^3	Temperatures				
	298.15K	303.15K	308.15K	310.15K	313.15K
Densities (ρ), (g.cm^{-3})					
0.0040	0.99906	0.99776	0.99609	0.99536	0.99422
0.0055	0.99958	0.99819	0.99668	0.99595	0.99458
0.0070	0.99990	0.99839	0.99680	0.99608	0.99502
0.0085	1.00033	0.99874	0.99709	0.99645	0.99523
0.0100	1.00052	0.99893	0.99735	0.99670	0.99563
0.0115	1.00070	0.99913	0.99769	0.99712	0.99583
0.0130	1.00092	0.99938	0.99772	0.99726	0.99593
0.0145	1.00110	0.99966	0.99800	0.99735	0.99620
Apparent molar volumes (Φ_v) (cm^3.mol^{-1})					
0.0040	-325.80	-336.29	-326.78	-329.54	-319.82
0.0055	-282.49	-273.74	-296.14	-298.19	-249.05
0.0070	-229.09	-205.01	-211.09	-214.12	-220.12
0.0085	-207.51	-178.25	-176.18	-188.15	-174.13
0.0100	-168.34	-143.46	-148.72	-157.89	-161.09
0.0115	-138.51	-118.61	-135.43	-150.41	-133.92
0.0130	-118.65	-103.36	-101.21	-122.97	-105.27
0.0145	-100.14	-93.345	-91.417	-97.733	-94.366

Table 2: Masson's parameters Φ_v^0 (cm³.mol⁻¹) and S_v (cm³ .mol⁻³/² .L¹/²) of aspirin solutions in pure water solvent system at different temperatures.

Temperature (K) →	298.15	303.15	308.15	310.15	313.15
Φ_v^0 (cm³.mol⁻¹)	-575.3	-584.5	-591.1	-577.8	-543.6
S_v (cm³ .mol⁻³/² .L¹/²)	4017	4271	4394	4058	3829

Table 3 : Viscosities and relative viscosities of aspirin solution in pure water at different temperatures.

Molar Conc. of aspirin (C) mol/dm³	Temperatures				
	298.15K	303.15K	308.15K	310.15K	313.15K
Viscosities (η) (Nm⁻³.s)					
0.0040	0.89665	0.80560	0.72544	0.69868	0.65966
0.0055	0.89824	0.80696	0.72640	0.69963	0.66089
0.0070	0.89957	0.80865	0.72732	0.70048	0.66172
0.0085	0.90074	0.81016	0.72842	0.70181	0.66286
0.0100	0.90172	0.81211	0.72912	0.70265	0.66402
0.0115	0.90334	0.81329	0.73051	0.70415	0.66490
0.0130	0.90526	0.81456	0.73144	0.70575	0.66621
0.0145	0.90651	0.81595	0.73277	0.70705	0.66708
Relative viscosities (η_r)					
0.0040	1.00333	1.00609	1.00405	1.00576	1.00558
0.0055	1.00511	1.00779	1.00538	1.00713	1.00745
0.0070	1.00660	1.00990	1.00666	1.00835	1.00872
0.0085	1.00791	1.01179	1.00818	1.01026	1.01046
0.0100	1.00901	1.01422	1.00915	1.01147	1.01223
0.0115	1.01082	1.01570	1.01107	1.01363	1.01357
0.0130	1.01297	1.01728	1.01236	1.01594	1.01556
0.0145	1.01437	1.01902	1.01420	1.01781	1.01689

Table 4 : Jones-Dole parameters of aspirin solutions in pure water.

Parameters of Jones-Dole equation					
T (K)	298.15	303.15	308.15	310.15	313.15
A (dm³/²mol⁻¹/²)	-0.016	0.024	0.003	0.020	0.030
B (dm³.mol⁻¹)	1.118	1.128	0.920	1.013	0.911

CONCLUSION

In the present report, from volumetric and viscometric study of aqueous solutions of aspirin at different temperatures are systematically presented. The densities of pain killer solutions under investigation decrease with increase in temperature and increases with increase in concentration. It has been observed that there exist strong solute–solvent interactions in these systems, which increases with increase in pain killer concentration.

The negative Φ_v^0 and positive S_v values shows that aspirin will be considerably associated in presence of ions. Since S_v is measure of solute-solute interactions. S_v values do not change systematically with change in temperature, and hence it suggests that the solute-solute interactions are insensitive to change in temperature. The positive values of Jones-Dole coefficient 'B' indicates structure promoting tendency and strong interactions between solute and solvent. Positive values of 'B' suggesting strongly hydrated solute indicating structure promoting tendency i.e. kosmotropes (structure makers). The Jones-Dole and Masson's equations are found to obey the aspirin in pure water system.

Conflicts of interest: The authors stated that no conflicts of interest.

REFERENCES

1. Shahidi F, *Can. J. Chem*., 1987,65 : 1924–1928.
2. Iqbal M, Verrall RE, *J. Phys. Chem.*,1987,91(4) :967–971.
3. Mendonca AFSS, Barbas MJA, Freitas JM, Lampreia IMS, *J. Chem. Thermodyn*.,2004, 36 :965–969.
4. Lu X-M, Xu W-G, Gui, J-S, Li H-WYang, J-Z, *J. Chem.Thermodyn*., 2005, 37:13–19.
5. Martin A, Bustamante P, Chun AHC, "Physical Chemical Principles in the Pharmaceutical Sciences", *4th edition, Lea & Febiger, Philadelphia,* 1993.
6. Essentials of Pharmacology, Surender Singh, Academa publishers, *2004, 1st edition,General Pharmacology,* Section-1, Chapter 1.2, Page No.8-10.
7. Nikam, PS, Ansari HR and Hasan M, *J. Indian Chem. Soc*., 1999, **76**: 344-346.
8. Nikumbh Arun B, Kulkarni Ganesh K and Bhujbal Ravindra C**,** **"**Study of flow and interaction parameters of Aspirin in NS, DNS, RL, and pure water system at 298.15K, 303.15K, 308.15K, 310.15K and 313.15K"**.** *International Journal of Innovative Research in Science, Engineering and Technology*, ISSN: 2319-8753, 2013,Volume 2, Issue 9.
9. Md. Monimul Huque, A.N.M. Hamidul Kabir, Md. Nurul Huda, Shaila Kabir Bangladesh *Pharmaceutical Journal*,2010,Vol.13(2).
10. Muhammad Javed Iqbal, Mansoora Ahmed Chaudhry, Thermodynamic study of three pharmacologically significant drugs: Density, viscosity, and refractive index measurements at different temperatures, *J. Chem. Thermodynamics*, 2009, 41: 221–226.
11. Chauhan S, Syal VK, Chauhan MS, Poonam Sharma, Viscosity studies of some narcotic–analgesic drugs in aqueous–alcoholic mixtures at 25 °C, *J. Mol. Liqs.,*2007,136: 161- 164.
12. Jones G, Dole M, The viscosity of aqueous solutions of strong electrolytes with special reference to barium chloride, *J. Am. Chem. Soc.*,1929, 2950-2964.
13. Falkenhagen H, Dole M, Viscosity of electrolyte solutions and its significance to the Debye theory. Zeitschrift Für Physik 30 (1929)611- 616.
14. Robinson RA, Stokes RH, Electrolytic solutions, *Butterworth scientific publications, London*,1959.
15. Haribar B, Southall NT, Vlachy V, Dill KA, *J. Am. Chem. Soc.,*2002, 124:12302.
16. Iqbal MJ and Choudhry MA, volumetric and viscometric studies of antidepressant drugs in aqueous medium at different temperatures, *J. Chem. Eng. Data,* 2009, 54:2772- 2776.

RESEARCH ARTICLE   **OPEN ACCESS**

Size dependent variation in the rate of oxygen consumption, ammonia excretion and O:N ratio of freshwater bivalve, *Indonaia caeruleus* from Yedgaon Dam during monsoon season (M.S.) India

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ABSTRACT

Study O: N ratio in freshwater bivalve molluscs, *Indonaia Caeruleus* is reported here by considering the size dependent variation. Freshwater bivalve molluscs were collected from the banks of Kukadi River at Yedgaon dam, near Junnar (Pune). The freshwater bivalves with two different size i.e. small (46-50 mm shell length) and large (56-60mm in shell-length) were collected for determination of changes in the rate of oxygen consumption, rate of ammonia excretion and O:N (oxygen : nitrogen) ratio, during monsoon on July and August . The small size bivalve showed high values of O:N ratio than that of large sized one. The results are discussed in the light of metabolic processes in fresh-water bivalve molluscs.

Key words - Ammonia excretion, *Indonaia Caeruleus*, oxygen consumption.

INTRODUCTION

The O:N ratio is an index of protein utilization in energy metabolism. This ratio is also useful for assessing the relative contribution of protein catabolism [1]. The respiration rates could be used to evaluate mussel stress and over all fitness of animal for survival and reproduction.

The body size of the bivalve mollusc is an important parameter for influencing the pattern of metabolic responses. The relationship between the rate of ammonia excretion and the body size can be variable due to a disproportionate reliance of protein catabolism for energy production in bivalve molluscs. Regulation of chemical composition of the body fluid is an important function of the ionic and somatic regulation and of excretion. This helps in the elimination of waste and conservation of useful metabolites for growth, maintenance and reproduction. Bayne and Scullard [3] reported that amount of nitrogen loss as amino acids relative to ammonia varied with season as well as location site of collection. In bivalve molluscs, several workers have studied nitrogenous excretory products and their reports reveled that, ammonia is the dominant products and large amount of amino- nitrogen are lost in excretion. Segawa [4] observed that, increased oxygen consumption and ammonia excretion is linear with increase in weight and decreases with period of starvation in abalone sulculus diversicular. Ganzalo and Cancino [5] reported that, oxygen conception and ammonia excretion of bivalve Guimordia bahamondei is a function of body weight.

According to Barkai and Griffths [6], 63% of energy content of the food consumed was lost as faces and 32% expended on respiration in abalone. Navarro and Torrijos [7] reported that, energy utilized in oxygen uptake and ammonia excretion was depending on the season and temperature of the water body. A number of investigator have studied oxygen consumption and ammonia excretion according to envirmental factors, turbidity [8], size [9][9], time [10] and growth [11]. Howkins et al. [12] reported O:N ratio on Perna Viridis and Perna indica from Cochin backwaters. Mathew and Menon [13] reported heavy metal stress induced variation in O:N ratio in Perna indica and Donax incarnates.

Review of literature reveled that, very little information was available on fresh water bivalve molluscs from India. By considering the abundant distribution of bivalve molluscs along the banks of kukadi river and paucity of information on O:N in fresh water bivalves, the present study was undertaken on *Indonaia caeruleus.*

METHODOLOGY

During monsoon (July - August) freshwater bivalve molluscs, *Indonaia caeruleus* were collected from banks of Kukadi river at Yedgaon. Animals with two different shell length i.e. small size (46-50mm shell length) and large size (56-60 mm shell length) were selected. After collection, bivalves were brought to the laboratory immediately. In order to remove the algal biomass, mud and other waste materials, the shells of the animals were brushed and washed with freshwater. The cleaned animals were divided into two size group's viz. small size (45-52) and large size (57-64 mm) according to their shell length. Each group comprises 15 animals. Then, they were allowed to defecation and depuration for 12-13 hrs (not acclimatization) in laboratory conditions, under constant aeration.

The physico-chemical parameters of water i.e. Temperature, pH, hardness and dissolved oxygen contents were also measured. The rate of oxygen consumption of individual animal was determined according to Wrinkler's modified method [14]. For determination of oxygen consumption of individual bivalve ,four closed respiratory jars 1.0 litre capacity each with an inlet and outlet were used . In order to open their valves, they were kept in continuous circulation of water inside the chamber. After opening their valves, the flow of water was cut off. A sample of water from it was drawn for determination of oxygen consumption and ammonia excretion. After one hour, 50 ml of sample water from the chamber was drawn to find out the oxygen content. At the same time 10 ml of the sample water from the chamber was also drawn and processed for analysis of ammonia according to phenol-hypochloride method suggested by Solorzano, [14]. Data on oxygen consumption, ammonia excretion and 0:N ratios were calculated for each individual bivalve used in this experiments, by dividing its oxygen consumption rate in moles O and by its ammonia excretion rate in moles N [15-16]. The mean values of four individual bivalves from each group were used for statistical analysis. Rate of oxygen consumption of individual bivalve represented mg O_2/l/h/gm body weight and rate of ammonia excreted represented mg NH_3-N/l/h body weight.

RESULTS AND DISCUSSION

The physico-chemical characteristics of the habitat water were temperature 26.5˚C - 29.5˚C on July and 25.5˚C - 28.0˚C on August, pH 8.08 - 9.27 on July and 8.78 - 8.83 on August. Hardness of water is 104 - 115 ppm on July and 97 - 111 ppm on August and dissolved oxygen 6.870 - 7.300 mg /l/h on July and 6.980 - 7.280 mg /l/h on August during monsoon season.

The rate of oxygen consumption during monsoon was found maximum in small sized animal as compared to large sized animal. It was found to be to be (0.7275 ± 0.0866 mg/l/h) on July and on August it was (0.7525± 0.0486 mg/l/h) in small animals. While in large sized animal oxygen consumption was found to be (0.5625 ± 0.0117 mg/l/h) on July and on August it was (0.5628 ± 0.0063 mg/l/h).

The rate of ammonia excretion was found maximum in large sized animal as compared to small sized animal, during monsoon .On July it was found to be (0.0119 ± 0.0006 µg NH_4-N/l/h) and on August it was (0. 0140 ± 0.0003 µg NH_4-N/l/h) in small sized animal. While in large sized animal it was (0.0186 ± 0.0010 µg NH_4-N/l/h) on July and on August it was (0. 0158 ± 0.0007 µg NH_4-N/l/h).

The calculation of O: N ratio revealed high values in small sized animals than large sized one during monsoon on July and on August. On July, the O:N ratio was found to be (53.5740 ± 4.1653) and on August it was (48.3229 ± 2.9219) in small sized animals. And in large sized animals the O: N ratio was (26.5243±1.7791) on July and on August O: N ratio was (31.2318 ± 1.6214).

Table-1 Size dependent variation in the rate of oxygen consumption, rate of ammonia excretion and O:N ratio of freshwater bivalves, *Indonaia caeruleus*, during monsoon season

Month	Size Specific Group	Animal No.	Body Size (mm)	Body weight (gms)	Oxygen consumption ml/lit/hr	Oxygen consumption mg/lit/hr	Ammonia excretion mg- NH_4-N/L	Atomic equivalent of oxygen	Atomic equivalent of ammonia	O: N ratio
July	Small size	I	50	7.821	0.5795	0.8275	0.0125	0.0517	0.00089	58.0898
		II	47	7.228	0.4738	0.6765	0.0113	0.0422	0.00080	52.7500
		III	46	7.204	0.4752	0.6785	0.0120	0.0424	0.00085	49.8823
						0.7275 ± 0.0866	0.0119 ± .0006			53.5740 ±4.1653
	Large size	I	56	8.418	0.4021	0.5741	0.0189	0.0358	0.00135	26.5185
		II	59	9.815	0.3856	0.5506	0.0195	0.0344	0.00139	24.7482
		III	60	10.194	0.3942	0.5629	0.0174	0.0351	0.00124	28.3064
						0.5625 ± 0.0117	0.0186 ± 0.0010			26.5243 ± 1.7791
August	Small size	I	47	7.227	0.5640	0.8053	0.0138	0.0503	0.00098	51.3265
		II	49	7.702	0.5202	0.7428	0.0144	0.0464	0.00102	45.4901
		III	50	7.805	0.4968	0.7094	0.0138	0.0443	0.00092	48.1521
						0.7525 ± 0.0486	0.0140 ± 0.0003			48.3229 ± 2.9219
	Large size	I	58	9.560	0.3951	0.5642	0.0150	0.0352	0.00107	32.8971
		II	59	9.803	0.3894	0.5560	0.0165	0.0347	0.00117	29.6581
		III	57	8.491	0.3981	0.5684	0.0160	0.0355	0.00114	31.1403
						0.5628 ± 0.0063	0.0158 ± 0.0007			31.2318 ± 1.6214

Hardness of water is 104 - 115 ppm on July and 97 - 111 ppm on August and dissolved oxygen 6.870 - 7.300 mg /l/h on July and 6.980 - 7.280 mg /l/h on August during monsoon season.

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The results of the experiments were shown in Table-1.

DISCUSSION

The rate of oxygen uptake was increased in small sized bivalves, as compared to large sized ones during monsoon season. While the ammonia excretion rate was found more increased in large sized bivalves as compared to small sized ones on July and August during monsoon seasons. Increased in the rate of oxygen uptake in small sized animals is due to small size relatively with small glycogen reserves. This increases considerably their protein catabolism, whereas larger ones with large size having large glycogen storage [17]. The metabolic rate is strongly dependent on body size. It is known that weight specific rate of oxygen consumption is lower in larger organisms than in smaller ones. This generalization

applies in both intra- species as well as inter-species bivalve molluscs of different sizes. In the present study on *Indonaia caeruleus*, the size specific oxygen consumption followed a general trend of acceptance i.e. smaller sized bivalve shows higher values of oxygen consumption than larger one. Mane [19] and Bayne [20] revealed that, body size in bivalves is important factor. Hence older and large bivalves have a lowest value than those of small individuals. It is known the oxygen uptake was mainly dependent on reproductive condition of bivalves during monsoon season, hence, the rate of oxygen consumption showed significant increase in smaller sized bivalve particularly during monsoon.

The energy utilization in oxygen consumption and ammonia excretion was significantly different which depending on size, season and temperature, but season being important factor which affect the overall fitness of the animal [6]. Many authors have shown that, in bivalve major nitrogenous excretory product is ammonia. A profound difference occurs in loss of nitrogen between different sizes and seasons [20,21]. In the present study on *Indonaia caeruleus*, the rate of ammonia excretion is more in large sized bivalves on July and August during monsoon seasons, because small size bivalves catabolise different biochemical substrates to varying degrees, according to season [22] The O:N ratios can provide indices of balance in animal tissues between the rate of catabolism of protein, carbohydrate, and lipid substrates. This ratio may be used to indicate the proportion of protein catabolised to carbohydrates and lipids. Higher value of O:N ratio indicates increased catabolism of carbohydrates or lipids. The increase or decrease of O:N ratio in bivalves of different sizes, could be due to the state of a gonadal development and level of metabolic activity of the bivalve molluscs. .

CONCLUSION

From this study it was concluded that, the rate of ammonia excretion in *Indonaia caeruleus* was found to be depleted in small sized bivalves as compared to large sized bivalves particularly on July and August of monsoon seasons. It was found to be weight specific and also dependent upon physiological state of the

organism. The rate of oxygen consumption of *Indonaia caeruleus* showed seasonal patterns as well as the reproductive activity of the animal. Low rate of oxygen consumption was observed during monsoon in small and large sized bivalves. The rate of ammonia excretion in *Indonaia caeruleus* was found to be depleted in small sized bivalves as compared to large sized bivalves particularly on July and August of monsoon seasons. It was found to be weight specific and also dependent upon physiological state of the organism. The O:N ratio shows higher value in small sized bivalve than that of larger ones.

REFERENCES

1. Bayne BL and Widdows J. The physiological ecology of two populations of Mytilus edulis J. Oecolosia.1978; 37: 137-162.

2. Bayne BL. In Marine mussels: Their ecology and physiology. *International Biological programme to Cambridge University Press. Cambridge, London, New York, Melbourne*, 1976:159.

3. Bayne BL and Scullard CS. An apparent specific dynamic action in Mytilus edulis, in the Laboratory, Helgol. Wiss. Meersunter. 1977;20: 526-552.

4. Segawa S. Oxygen consumption and ammonia excretion by the balone Sulcus diversicolor in starved condition. *Bull. Jap. Soc.Sci. Fish.* 1991;57: 2001-2006

5. Ganzalo BA and Cancino JM. Physiological characteristics of the brooding bivalve, Galmardia. bahamondei, Madio Ambiente. 1988;9: 13-20.

6. Barkai R. Gnffiths CL. An energy budget for the South African abalone Haliotis midae Linnaeus. *J mollusc Stud* 1988; 54:43-51

7. Navarro JM and Torrijos R. Seasonal variation in oxygen uptake and ammonia excretion in the predatory gastropod Concholepas concholepas. Comp. *Biochem. Physiol*. 1994;108A: 39-49.

8. Grants J. and Thorpe B. Effects of suspended sediments on growth, respiration and excretion of soft- shell clam, Mya arenaria *Can J. fish. Agnat. Sci.* 1991;48: 1285-1292.

9. Bhagde RV and Mane UH. A study on the metabolism in Green mussel, Perna viridis *J. mar. Biol. Ass. India*. 2005;47 (1): 106-110.

10. Vitale MA and Friedl FE. Ammonia production by freshwater bivalve, Ellipto buckley (Lea). Intact and Monovalve preparations Comp. *Biochem. Physiol*. 1984;77(A):113-116.

11. Bacon CS and Mac Donald BA. Food quality, feeding activity and energy balance in the sea scallop, Placopecten megelanicus *J. Shell-fish. Res.* 1991; 10: 1-37.

12. Hawkins AJS, Bayne BL, Menon NR and Damodaran R. The mussels Perna viridis and P. indica as transplantable indicators of pollution: comparison of their metabolic responses to reductions of both oxygen tension and salinity. National Seminar on mussel watch, Cochin Univ. Sci. Technol., Cochin, 1986:51-64.

13. Mathew P and Menon NR. Heavy metal stress induced variation in O.N. ratio of two tropical bivalves Perna indica and Donax incarnatus. *Indain J. Expt. Biol.* 1993;31: 275-292.

14. Golterman HL, Clymo RS and Ohnstad MAM. Physical and chemical analysis of freshwaters, IBP, Handbook, No- 08. Blackwell Scientific Publication, Oxford, London, Edinburgh, Melbourne, 1978;2nd Edn. 172-178

15. Solorzano L. Determination of ammonia in natural waters by phenol-hypochlorite method. *Limnology and oceanography* 1969;14: 799-801.

16. Widdows J. Physiological indices of stress in Mytilus edulis. *J. Mar. Biol. Ass*. U.K. 1978; 58:125-142

17. Bayne BL and Newell RC. Physiological energetics of Marine Molluscs. In: The Mollusca Physiology, (Wilbur. K. M.) Part-I Vo1. 4, Academic press, New York, 1983:405-515.

18. Bayne BL. Aspects of metabolism of Mytilus edulis during starvation, *Neth. J. Sea. Rev*. 1973; 7:399 - 410

19. Mane UH. Oxygen consumption of clam, Katelysia opima in relation to environmental conditions. *Broteria*. 1975;64(1-2): 33-58.

20. Nagwanshi M. Reproductive physiology & freshwater bivalve molluscs from Aurangabad, Maharashtra State, Ph.D. Thesis, Dr.B.A.

Marathwada University, Aurangabad, India, 1996:364.

21. Salve SK. Changes in biochemical constituents of *Indonaia caeruleus* (Prashad, 1918) Mollusca: Bivalvia, from Godavari river at Paithan. Ph. D. Thesis Dr.Babasaheb Ambedkar Marathwada University, Aurangabad.(M.S.). 2007:1 – 245.

22. Gabbott PA. Developmental and seasonal metabolic activities in marine molluscs, In: Hochachka P. W (ed). The Mollusca. Vol. 2. Academic Press, New York, 1983: 165-217.

23. Jadhav Mangesh, Bhavane Vasant & Gulave Arun. Size dependent variation in the rate of oxygen consumption, ammonia and O:N ratio of freshwater bivalve, Lamellidens marginalis from Godavari river during monsoon (m.s) india, *J. Trends in Fishery research*,2012;1(2):22-26

24. Gulave AR. Size specific physiological fitness of fresh water bivalve, indonaia caeruleus (prashad 1918) from Gajanan sagar Yedgaon, Tal – Junnar , Dist – Pune ., Ph. D. Thesis Dr. Babasaheb Ambedkar Marathwada University, Aurangabad. (M.S.). 2012:1 – 237.

RESEARCH ARTICLE   **OPEN ACCESS**

Synthesis and evaluation of some 1,2,3,4-tetra-hydro pyrimidine derivatives as antitumor agents.

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Manuscript Details

Editor: Dr. Arvind Chavhan

Cite this article as:

ABSTRACT

Sixteen [3, 6 (4- substituted aryl)1-(1,2 hydrazino-2-oxo-ethyl)-2oxo-1, 2, 3, 4-tetrahydropyrimidine-5-yl] propionic acid derivatives have been synthesized in a four-step reaction. In the first step benzene or substituted benzene react with glutaric anhydride in presence of aluminum trichloride (Friedel Craft reaction) to obtain 5-(substituted phenyl)-5-oxo pentanoic acid. Second step involves synthesis of [4, 6 (4-substituted aryl)-2-oxo-1, 2, 3, 4- tetra-hydro-pyrimine-5 yl propionic acid by reaction between 5-(substituted phenyl)-5-oxo pentanoic acid, urea and substituted aldehydes (Biginelli reaction).Third step involves reaction of [4, 6 (4-substituted aryl)-2-oxo-1, 2, 3, 4- tetra-hydropyrimine-5 yl propionic acid was dissolved in a solution prepared by reacting Na (0.1mol) with 200ml of absolute ethanol and then solution is refluxed with ethyl chloroacetate to produce. 3-[4-chloro-6-(4-chlorophenyl)-1-(2-ethoxy-2-oxoethyl)-2-oxo-1,2-dihydropyrimidin-5-yl] propanoic acid. In forth step Compound (3) was treated with hydrazine hydrate to form[3, 6 (4- substituted aryl)1-(1,2hydrazino-2-oxoethyl)- 2oxo- 1,2,3, 4tetrahydro pyrimidine -5-yl] propionic acid derivatives. Their structures are confirmed by IR, ^{1}H NMR. TLC of synthesized compounds performed in chloroform: ethanol (3:1) solvent system. All compounds were screened for their antitumor activity by trypan blue assay at. (1 µg /mL, 10 µg/mL, 100 µg /mL, 1000 µg /mL). And their results were compared with standard drug Cyclophosphamide.

Key words: Pyrimidine, Antitumor activity, aryl alkanoic acid.

INTRODUCTION

In medicinal chemistry pyrimidine derivatives have been very well known for their therapeutic applications. [1] The presence of a pyrimidine base in thymine, cytosine and uracil, which are the essential binding blocks of nucleic acids, DNA and RNA is one possible reason for their activity. [2,3,4] The literature indicated that compounds having pyrimidine nucleus possess broad range of biological activities, like 5-fluorouracil as anticancer; [5] idoxuridine and trifluoridine as antiviral; [6] zidovudine and stavudine as antiHIV; [7] trimethoprim, sulphamethiazine and sulphadiazine as antibacterial; [8] sulphadoxin as antimalarial and antibacterial; [9] minoxidil and prazosin as antihypertensive; [10] barbiturates e.g. phenobarbitone as sedative, [11] hypnotics and anticonvulsant; [12] propylthiouracil as antithyroid; [13] thionzylamine as H1antihistamine; [14] and toxoflavin as antibiotics.[15] Pyrimidines derivatives are undertaken with the aim of improving safety profile and reduction of GI side effects by increasing aryl chain length of acid and addition of amide group. [16,17,18]

METHODOLOGY

The chemicals used are benzene, glutaric anhydride, sulphuric acid, aluminium chloride, aluminium hydroxide, methanol, urea, chlorobenzaldehyde, ferrous chloride, ethanol, potassium dichromate, hydrazine hydrate, ethyl chloroacetate were of LR grade and purchased from Sigma Aldrich, Mumbai, India.IR Spectra taken by using Perkin Elmer 65 FT-IR Spectrophotometer using KBr disc.1H NMR Spectra is taken by using Brucker Spectrophotometer (400 MHz) in DMSO from University of Pune Maharashtra, India. All melting points were determined in open capillaries and are uncorrected. Purity of compounds are checked by Thin layer chromatography using prepared silica gel G slides as a stationary phase and chloroform: ethanol (3:1) solvent system as mobile phase. The spots resolved were checked by UV-chamber and iodine chamber.

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Step 1: Synthesis of 5-(substituted phenyl)-5-oxo-pentanoic acid [19]

Placed (0.00225 mol) of substituted benzene and (0.0034 mol) of glutaric anhydride in a 1 liter three-necked flask, Stirred the mixture and added (0.0075 mol) of powdered aluminium chloride all at once. The reaction usually start immediately, HCl is generated, evolved and reaction mixture become hot. If there is no apparent reaction warm gently. Heated in an oil bath to gentle refluxing with continued stirring for a half hour. Allowed to cool, immerse flask in a bath of cold water and slowly added 15 ml of water from a separating funnel. Introduced 5 ml of conc. HCl and separated benzene layer. Transferred the hot mixture to a 60 ml beaker. The 5-(substituted phenyl) -5-oxo-pentanoic acid separated as colourless oil, which soon solidified. Cooled in ice, filtered off acid at the pump and washed with 10 ml of cold water. Dissolved the crude acid in solution of 4g of anhydrous sodium carbonate in 25 ml water by boiling for 10-15 min. filtered the solution, suctioned to remove the small amount of aluminium hydroxide and washed with 2.5 ml portion of hot water. Treated the hot filtrate with 2g of decolourising carbon. Stirred for 5 min and filtered at the pump through a preheated buchner funnel. Transfer the filtrate to 1-liter beaker, cooled to about 50 0C and cautiously acidified with 6-7 ml of conc. HCl. Cooled to 0 0C in a freezing mixture of ice and salt. Filtered, washed thoroughly with cold water, dried for 12 hours upon filter paper and then weighed the compound.

Step–2: Synthesis of [4, 6-(4-substituted aryl)-2-thioxo- 1, 2, 3, 4-tetrahydro-pyrimidin-5-yl]-propanoic acid [20]

An equimolar reaction mixture of 5-(substituted phenyl) -5-oxo- pentanoic acid (0.0022 mol) urea (0.002 mol), substituted aldehyde (0.0022 mol) and K2CO3 (0.0022 mol) in 7 ml ethanol were refluxed in oil bath for 7 hrs. The reaction mixture was cooled and the solid obtained by filtered was dissolved in hot water and filtered. The filtrate was neutralized with acetic acid. The product thus obtained was recrystallized from methanol.

Step-3 : Synthesis of 3-[4-chloro-6-(4-chlorophenyl)-1-(2-ethoxy-2-oxoethyl)-2-oxo-1,2-dihydropyrimidin-5-yl]propanoic acid [21]

The compound 2 (0.1mol) was dissolved in a solution prepared by reacting Na (0.1mol) with 200 ml of absolute ethanol. The solution was refluxed with stirring and ethyl chloroacetate (0.1mol) was added in three portions over a period of 0.5 hr. After heating under reflux for 16 hr, the reaction mixture was filtered while hot to remove precipitated sodium chloride, the solvent was removed on a rotary vaccum evaporator. The crude product was collected and recrystallised from ethanol.

Step-4 : Synthesis of compounds [3, 6 (4- substituted aryl)1-(1,2 hydrazino-2-oxoethyl)-2oxo-1, 2, 3, 4-tetrahydropyrimidine-5-yl] propionic acid derivatives [22]

Mixture of compound 3 (0.01 mole), FeCl3.6H2O (0.02 mole) & 0.01 mole Hydrazine hydrate of was ground by pestle & mortar at room temp. The reaction mixture was digested with water. The resultant solid was filtered, washed with water & the crude material is purified by recrystallization from methanol to afford compound 4.

ANTI TUMOR ACTIVITY:
Trypan Blue Assay

Viable Cell Counts Using Trypan Blue

Trypan Blue is a vital dye. The reactivity of trypan blue is based on the fact that the chromophore is negatively charged and does not interact with the cell unless the membrane is damaged. Therefore, all the cells which exclude the dye are viable. HL-60 cell line is taken for antitumor studies
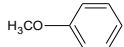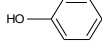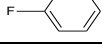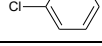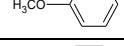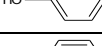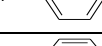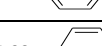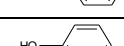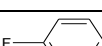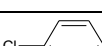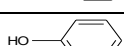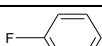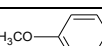
Scheme 1: Synthesis of 1, 2, 3, 4-Tetrahydro-pyrimidine derivatives
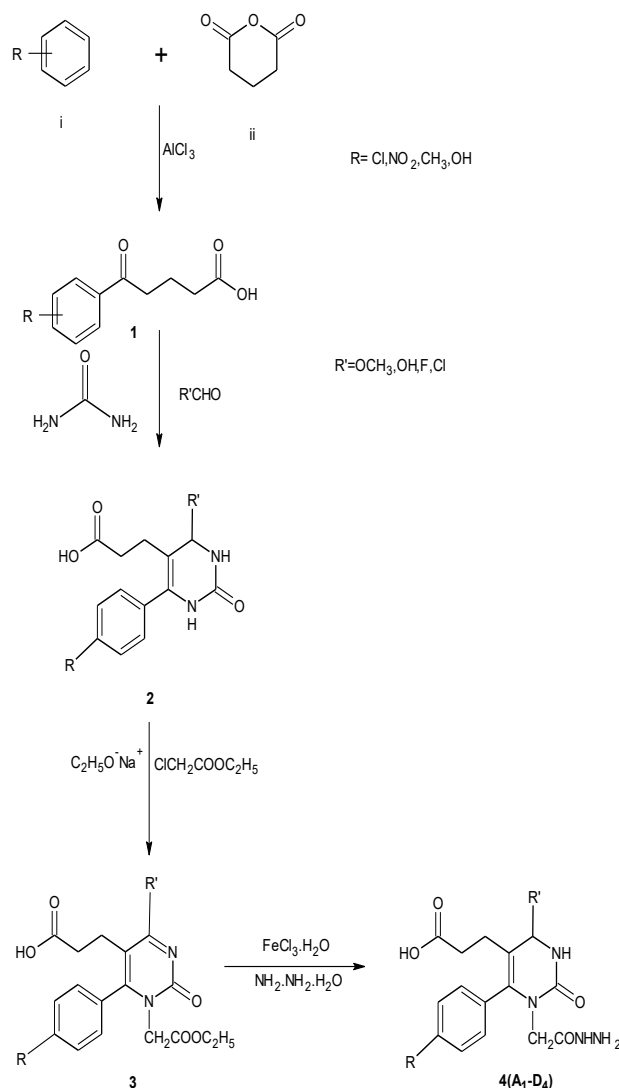
Procedure:

Trypan Blue Staining of Cells Place 0.5 ml of a suitable cell suspension (dilute cells in complete medium without serum to an approximate concentration of 1 x 105 to 2 x 105 cells per ml in a screw cap test tube. Add 0.1 ml of 0.4% Trypan Blue Stain. Mix thoroughly. Allow to stand 5 min at 15 to 30°C (room temperature). Fill a hemocytometer as for cell counting. Under a microscope, observe if non-viable are stained and viable cells excluded the stain.

Table 1. Synthesized compounds

Compound	R	R1	Mol. formula	Mol. wt	M.P	% yeild	R.f value (Chloroform: Ethanol)
A1	Cl	H₃CO—⬡	$C_{22}H_{23}ClN_4O_5$	458.89	138.5	73.25	0.473
A2	Cl	HO—⬡	$C_{21}H_{21}ClN_4O_5$	444.86	134.2	75.23	0.356
A3	Cl	F—⬡	$C_{21}H_{20}FN_4O_4$	446.85	136.9	71.58	0.436
A4	Cl	Cl—⬡	$C_{21}H_{20}Cl_2N_4O_4$	463.31	142.3	68.22	0.388
B1	OH	H₃CO—⬡	$C_{22}H_{24}N_4O_6$	440.44	133.0	76.59	0.412
B2	OH	HO—⬡	$C_{21}H_{22}N_4O_6$	426.44	125.8	74.32	0.362
B3	OH	F—⬡	$C_{21}H_{21}FN_4O_5$	428.41	127.4	69.33	0.268
B4	OH	Cl—⬡	$C_{21}H_{21}ClN_4O_5$	444.86	134.8	75.66	0.387
C1	CH3	H₃CO—⬡	$C_{23}H_{26}N_4O_5$	438.36	131.5	81.57	0.425
C2	CH3	HO—⬡	$C_{22}H_{24}N_4O_5$	424.44	123.6	69.56	0.389
C3	CH3	F—⬡	$C_{22}H_{23}FN_4O_4$	426.44	125.7	73.66	0.296
C4	CH3	Cl—⬡	$C_{22}H_{23}ClN_4O_4$	442.89	134.6	78.32	0.432
D1	NO2	H₃CO—⬡	$C_{22}H_{23}N_5O_7$	469.44	144.5	68.72	0.459
D2	NO2	HO—⬡	$C_{21}H_{21}N_5O_7$	455.22	141.2	76.52	0.342
D3	NO2	F—⬡	$C_{21}H_{21}FN_5O_6$	457.41	142.6	77.25	0.267
D4	NO2	H₃CO—⬡	$C_{21}H_{20}ClN_5O_6$	473.86	148.3	68.74	0.496

SPECTRAL DATA

A1-IR (KBr,cm-1) :3430 (O-H)str., 3230 (N-H str.)3068 (Ar-C-H)str,2966 (C-H)str,1697(C=O)str,1650(C=O amide) str,1574 (C=C)str, 1280 (C-O)str.,1109(C-N)str. 667 (C-Cl)str.,

1H NMR (CDCl3) :7.2-8 (8H (s) of Ar-H), 6.9 (2 H (s) of pyrimidine), 10.8 (1 H (s) of –COOH), 3.2(3 H (s) of OCH3), 1.42 (2H (t) of CH2), 2.82 (2H (t) of CH2).3.8(1H(S) of –CONH),3.6(2H(S) of –NH2).2.1(2H (s) of CH2.

A2-IR (KBr,cm-1)   :3450 (O-H)str., 3280 (N-H str.)3030 (Ar-C-H)str,2940 (CH)str,1720(C=O)str, 1650(C=O amide) str,1550(C=C)str, 1278 (C-O)str.,1115(C-N)str. 662 (C-Cl)str.

A3-IR (KBr,cm-1) :3480 (O-H)str., 3250 (N-H str.)3047 (Ar-C-H)str,2957 (CH)str,1715(C=O)str, 1650(C=O amide) str,1558(C=C)str, 1275 (C-O)str.,1120(C-N)str. 662 (C-Cl)str. 710(C-F)str.

A4-IR (KBr,cm-1) :3465 (O-H)str., 3278 (N-H str.)3056 (Ar-C-H)str,2950 (CH)str,1715(C=O)str, 1650(C=O amide) str,1556(C=C)str, 1270 (C-O)str.,1110(C-N)str. 662 (C-Cl)str.

B1-IR (KBr,cm-1) :3460(O-H)str., 3245 (N-H str.)3030 (Ar-C-H)str,2960 (C-H)str,1715(C=O)str, 1650(C=O amide) str,1574 (C=C)str, 1280 (C-O)str.,1109(C-N)str. 667 (C-Cl)str.,

1H NMR (CDCl3) :7.2-8 (8H (s) of Ar-H), 6.9 (3 H (s) of pyrimidine), 10.8 (1 H (s) of –COOH), 3.2(3 H (s) of OCH3), 1.42 (2H (t) of CH2), 2.82 (2H (t) of CH2).3.8(1H(S) of –CONH),3.6(2H(S) of –NH2).3.9(1H(S) of –OH).

B2-IR (KBr,cm-1) :3450 (O-H)str., 3280 (N-H str.)3030 (Ar-C-H)str,2940 (CH)str,1720(C=O)str, 1650(C=O amide) str,1550(C=C)str, 1278 (C-O)str.,1115(C-N)str.

B3-IR (KBr,cm-1) :3476 (O-H)str., 3264 (N-H str.)3047 (Ar-C-H)str,2957 (CH)str,1715(C=O)str, 1650(C=O amide) str,1558(C=C)str, 1275 (C-O)str.,1115(C-N)str, 710(C-F)str.

B4-IR (KBr,cm-1) :3465 (O-H)str., 3278 (N-H str.)3056 (Ar-C-H)str,2950 (CH)str,1715(C=O)str, 1650(C=O amide) str,1556(C=C)str, 1270 (C-O)str.,1110(C-N)str. 660 (C-Cl)str.

C1-IR(KBr,cm-1):3430 (O-H)str., 3230 (N-H str.)3068 (Ar-C-H)str,2966 (C-H)str,1697(C=O)str,1650(C=O amide) str,1574 (C=C)str, 1280 (C-O)str.,1109(C-N)str.

1H NMR (CDCl3) :7.2-8 (8H (s) of Ar-H), 6.9 (3 H (s) of pyrimidine), 10.8 (1 H (s) of –COOH), 3.2(3 H (s) of OCH3), 1.42 (2H (t) of CH2), 2.82 (2H (t) of CH2), 3.8(1H(S) of –CONH),3.6(2H(S) of –NH2).

C2-IR (KBr,cm-1) :3450 (O-H)str., 3280 (N-H str.)3030 (Ar-C-H)str,2940 (CH)str,1720(C=O)str, 1650(C=O amide) str,1550(C=C)str, 1278 (C-O)str.,1115(C-N)str.

C3-IR (KBr,cm-1) :3476 (O-H)str., 3264 (N-H str.)3047 (Ar-C-H)str,2957 (CH)str,1715(C=O)str, 1650(C=O amide) str,1558(C=C)str, 1275 (C-O)str.,1115(C-N)str, 710(C-F)str.

C4-IR (KBr,cm-1) :3465 (O-H)str., 3278 (N-H str.)3056 (Ar-C-H)str,2950 (CH)str,1715(C=O)str, 1650(C=O amide) str,1556(C=C)str, 1270 (C-O)str.,1110(C-N)str. 660 (C-CI)str.

D1-IR(KBr,cm-1):3430(O-H)str., 3230 (N-H str.)3068 (Ar-C-H)str,2966 (C-H)str,1697(C=O)str,1650(C=O amide) str,1574 (C=C)str,1344(NO2)str, 1280 (C-O)str.,1109(C-N)str.

1H NMR (CDCl3) :7.2-8 (8H (s) of Ar-H), 6.9 (3 H (s) of pyrimidine), 10.8 (1 H (s) of –COOH), 3.2(3 H (s) of OCH3), 1.42 (2H (t) of CH2), 2.82 (2H (t) of CH2),1.9(3H (S)of-CH3),3.8(1H(S) of –CONH),3.6(2H(S) of –NH2).

D2-IR (KBr,cm-1) :3450 (O-H)str., 3280 (N-H str.)3030 (Ar-C-H)str,2940 (CH)str,1720(C=O)str, 1650(C=O amide) str,1550(C=C)str, 1325(NO2)str, 1278 (C-O)str.,1115(C-N)str.

D3-IR (KBr,cm-1) :3476 (O-H)str., 3264 (N-H str.)3047 (Ar-C-H)str,2957 (CH)str,1715(C=O)str, 1650(C=O amide) str,1558(C=C)str, 1328(NO2)str, 1275 (C-O)str.,1115(C-N)str, 710(C-F)str.

D4-IR (KBr,cm-1) :3465 (O-H)str., 3278 (N-H str.)3056 (Ar-C-H)str,2950 (CH)str,1715(C=O)str, 1650(C=O amide) str,1556(C=C)str, 1344(NO2)str, 1270 (C-O)str.,1110(C-N)str. 660 (C-CI)str.

Table 2:Antitumor activity of [4, 6 (4- substituted aryl)-2-oxo-1, 2, 3, 4-tetrahydropyrimidine- 5-yl] propionic acid derivatives

Sample	Conc. (ug/ml)	Observed Viable Cell	Total cell count	% Viability	Mean ±SEM
Control	-	89	113	78.76	--
A₁	1000	33	117	28.20	28.62 ± 0.4250*
	100	43	103	41.74	45.62 ± 3.8850
	10	48	99	48.48	47.99 ± 0.4850
	1	55	92	59.78	65.52 ± 5.740
A₂	1000	28	119	23.52	23.42 ± 0.0953*
	100	36	108	33.33	32.80 ± 0.5299
	10	47	104	45.19	45.24 ± 0.0432
	1	59	97	60.82	59.79 ± 1.0300
A₃	1000	26	113	24.52	24.42 ± 0.0832*
	100	33	102	34.33	35.80 ± 0.466
	10	56	135	55.12	48.56 ± 0.0657
	1	59	97	60.82	59.79 ± 1.0300
A₄	1000	18	112	16.07	16.44 ± 0.370*
	100	21	126	16.66	17.20 ± 0.540*
	10	27	123	21.95	21.38 ± 0.574*
	1	22	112	19.64	20.18 ± 0.540*
B₁	1000	25	112	22.32	22.97 ± 0.6550*
	100	43	108	39.81	41.33 ± 1.5200
	10	54	96	56.96	56.69 ± 0.2647
	1	60	89	67.41	67.04 ± 0.3746
B₂	1000	45	116	32.56	32.09± 0.4250
	100	47	112	34.45	34.12 ± 2.8850
	10	44	95	42.76	42.65 ± 0.5640
	1	45	82	59.45	58.12 ± 3.687
B₃	1000	33	103	32.03	32.85 ± 0.8149
	100	46	95	48.42	49.21± 0.7900
	10	59	93	63.44	63.82 ± 0.3855
	1	63	85	74.41	71.27 ± 3.1400

	1000	23	108	23.42	23.32 ± 0.0562
B₄	100	34	105	33.32	32.80 ± 0.345
	10	54	137	45.42	44.43± 0.0873
	1	43	98	52.82	51.79 ± 1.0546
	1000	25	110	22.72	23.97 ± 0.5640*
C₁	100	42	105	40.00	40.33 ± 1.5620
	10	58	98	59.18	59.69 ± 0.1345
	1	56	89	62.91	64.04 ± 0.3987
	1000	45	116	32.56	32.09± 0.4250
C₂	100	47	112	34.45	34.12 ± 2.8850
	10	44	95	42.76	42.65 ± 0.5640
	1	45	82	59.45	58.12 ± 3.687
	1000	18	112	16.07	16.44 ± 0.370*
C₃	100	21	126	16.66	17.20 ± 0.540*
	10	27	123	21.95	21.38 ± 0.574*
	1	22	112	19.64	20.18 ± 0.540*
	1000	25	110	22.72	23.97 ± 0.5640*
C₄	100	42	105	40.00	40.33 ± 1.5620
	10	58	98	59.18	59.69 ± 0.1345
	1	56	89	62.91	64.04 ± 0.3987
	1000	18	113	16.54	16.13 ± 0.845*
D₁	100	27	132	20.23	20.43 ± 0.675*
	10	30	139	23.48	24.24 ± 0.459*
	1	48	125	38.58	38.64 ± 0.794*
	1000	23	108	23.42	23.32 ± 0.0562
D₂	100	34	105	33.32	32.80 ± 0.345
	10	54	137	45.42	44.43± 0.0873
	1	43	98	52.82	51.79 ± 1.0546
	1000	25	110	22.72	23.97 ± 0.5640*
D₃	100	42	105	40.00	40.33 ± 1.5620
	10	58	98	59.18	59.69 ± 0.1345
	1	56	89	62.91	64.04 ± 0.3987
	1000	16	110	14.54	14.13 ± 0.845*
D₄	100	25	130	19.23	19.43 ± 0.675*
	10	29	135	21.48	22.24 ± 0.459*
	1	45	123	36.58	36.64 ± 0.794*
	1000	16	102	15.68	16.42 ± 0.324*
Cyclophosphamide	100	19	113	16.81	17. 32± 0.583*
	10	25	120	20.83	21.48 ± 0.276*
	1	21	108	19.44	20.08 ± 0.470*

RESULTS AND DISCUSSION

In scheme all compounds have shown moderate anticancer activity at both the concentration. (100 µg /mL, 1000 µg /mL). Compounds **A₄**, **C₃**, **D₁**, **D₄** have shown excellent anticancer activity at all concentrations (1 µg /mL, 10µg/mL, 100 µg/mL, 1000 µg/mL). Cyclophosphamide was used as standard drug.

Increased antitumor activity possessed by derivatives may be because of presence of tetrahydropyrimidine ring. Cl, F derivatives shows increased antitumor activity.

CONCLUSION

Sixteen [3, 6 (4- substituted aryl)1-(1,2 hydrazino-2-oxoethyl)-2oxo-1, 2, 3, 4-tetrahydropyrimidine-5-yl]

propionic acid derivatives have been synthesized in a four-step reaction. Their structures are confirmed by IR, ^{1}H NMR and TLC. The antitumor activity of all compounds has been recorded on the basis of reference standard Cyclophosphamide. All the compounds showed antitumor activity. The antitumor data shows that use of cloro and fluoro group in product shows increase in activity. The other compounds shows moderate antitumor activity.

REFERENCES

1. Holmes JT. Prostaglandins, Leukotrienes and other Eicosanoids in Wilson and Giswold's Textbook of Organic Medicinal and Pharmaceutical Chemistry. Lippincott Williams and Wilkins, PA, 2004;11:818-829.

2. Huskisson EC. Antirheumatic drugs. Clinical pharmacology and therapeutic series. Praeger Publishers, California, 1983;3:117- 125.

3. Borne RF. Nonsteroidal Anti-inflammatory agents, Foye's Principles of Medicinal Chemistry, New York, 2002;5:751-793.

4. Kadam SS, Mahadik KR, Bothara KG. Nonsteroidal Antiinflammatory agents, Principles of Medicinal Chemistry, 15th edition, Nirali Prakashan, Pune, 2002; 2:138-139.

5. Wallace JL, Bure AG. GI-Sparing Anti-inflammatory Drugs: A Promising Future. Inflammation Research Network, University of Calgary, Calgary, Alberta, Canada 1993;3:154- 158.

6. Bhosale D, Bharambe S, Gairola N, Dhaneshwar SS., Mutual prodrug concept: Fundamentals and applications, *Ind.J.Pharm.Sci*, 2006;68, 3: 286-294.

7. Bartsch H, Nair J. Chronic inflammation and oxidative Stress I the genesis and perpetuation of cancer: role of lipid peroxidation, DNA damage and repair. Langenbecks Arch Surg, 2006;391:499–510.

8. Lu H, Ouyang W, Huang C. Inflammation, a key event in cancer development. Mol Cancer Res, 2006;4:221 -233.

9. Winter C A. GI-Sparing Anti-inflammatory Drugs. Proc Soc Exp Bio Med, 1962;1:544.

10. Pirsino R, Bainchini F, Banchelli G. 2-phenylpyrazolo- 4-ethyl- 4, 7-dihydro [1, 5-a] pyrimidine-7-one for its analgesic, antipyretic and anti-inflammatory activities. Pharmacol Res Comm, 1996;18:241.

11. Modica M, Santagati M, Santagat A. Some new thiadiazolothieno-pyrimidinones for anti-inflammatory and analgesic activities. Pharmazie, 2000;55:500.

12. Cenicola ML, Donnoli D, Stella L. Someimidazolo [1, 2]- pyrimidines for anti-inflammatory, analgesic and antipyretic activities. Pharmacol Res,1990;22:80.

13. Nargund LVG, Badiger VV and Yarnal SM. Substituted 2- mercapto-3-(N-alkyl) pyrimido [5, 4-c] cinnolin-4-(3H)-ones for anti-inflammatory and antimicrobial activities. *J Pharm Sci,* 1992;81:365.

14. Cottam HB, Wasson DB, Shikh HC. Pyrazolo [3, 4-d] pyrimidine derivatives as potential inhibitor of adenosine kinase. *J Med Chem*, 1993;36.

15. Bruni F, Costanzo A, Selleri S. Series of pyrazolo [1, 5- a] pyrimidin-7-one for anti-inflammatory activity. *J Pharm Sci,* 1993; 82:480.

16. Tozkoparan B, Ertan M, Kelicen P and Demirdamar. New 2- benzylidene -7 –methy l- 3- oxo - 5-(substitutedphenyl)-2,3-dihydro-5H thiazolo [3,2] pyrimidine-6-carboxylic acid esters for anti-inflammatory activity R. I L Farmaco, 1999;54:588.

17. Lee C H, Jiang M, Cowart M. Some 6-substituted pyridopyrimidine analogues as potential AK inhibitors. *J Med Chem,* 2001;44:2133, 2001.

18. Boyle DL, Kowluk EA, Jarvis MF. Pyrazolo-pyrimidine derivatives as potential inhibitor of adenosine kinase. *J Pharmacol Exp Ther*, 2001;296:495.

19. Molina P, Aller E, Lorengo A. Pyrido [1, 2] pyrimidines on solid phase using the iminophosphorane methodology and tested for effects on leukocyte function *in-vitro* and anti-inflammatory activity. *J Med Chem*, 2001;44:1011.

20. Vidal A, Ferrandiz ML, Ubeda A. Hexahydroimidazo [1, 2] pyrimidine derivatives on leukocyte functions. *J Pharm Pharmacol*, 2001;53:1379.

21. Furniss BS, Hannaford AJ, Smith PWG, Tatchel AR. Vogel's textbook of practical organic chemistry, 2004;5:1015-1016.

22. Sodhi SM, Dinodia M, Rani R, Shukla R. Synthesis, anti-inflammatory and analgesic activity of some pyrimidine derivatives. *Indian J of Chemistry*, 2009;49B:273-281.

23. Pattan SR, Hole MB. Synthesis and evaluation of some novel [1,2,4] triazolo[1,5-α] pyrimidine derivatives for anticancer activity. *Indian J of Chemistry,* 2012;51B:774-779.

Comparative Study of Surface Activation of Nanocrystalline MgO Under Thermal and Microwave Heating in Wittig Reaction

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ABSTRACT

The comparative study of surface activation of MgO under thermal and microwave heating was performed for Wittig reaction under grindstone technique. The nanocrystalline MgO obtained by thermal decomposition of alkali leached $Mg(OH)_2$ under hydrothermal condition and characterized by XRD, UV, IR and Hammett indicator method. Nanocrystalline MgO is activated under thermal and microwave heating for different scale of time and surface activated MgO used as solid base in Wittig reaction. Surface basicity's are compared for both techniques. Activation of surface is studies by IR techniques and phenolphthalein Hammett indicator method. All results viz. Surface basic activity, IR techniques and Hammett indicator methods suggested that thermal activation of MgO is superior over microwave activation.

Key Words: Surface Activation, Thermal Heating, Microwave Heating, Wittig Reaction.

INTRODUCTION

Surface activation of heterogeneous catalyst is an important task in order to carry out chemical reactions in an economic way according to Pushpaletha et al [1]. The reactivity of catalysts depends on activity of surface. For this purpose, different physical and chemical methods have been developed for activation of catalysts.

Hideshi [2] reported alkaline earth metal oxides are activated by conventional thermal treatments while Xinhuan et al [3] reported that transition metal is activated by microwave, sonochemical and irradiation techniques. Bio-activation methods are also used for some metal oxide activation. Zeolites are activated by acid treatments as reported by Xie [4]. Magnesium oxide is a solid base in different organic reactions such as Aldol condensation, Clainsen-Schimdt Condensation, Knoenvengal condensation, Coupling reactions, Wittig reaction, double bond isomerization etc [5-7]. Wittig reaction is base assisted reaction of phosponium salt with aldehyde or ketones through phosphorus ylides formation. It is known for selective synthesis of trans-alkene, important in perfumes, pharmaceutical drugs and vitamins as reviewed by Horst Pommer [8]. This reaction can proceed in presence of heterogeneous solid base MgO. MgO is required for generation of phosphorus ylide from phosponium salt by abstraction of acidic proton. This heterogeneous surface basic nature of MgO makes it a superior solid base. MgO exhibit surface basic activity due to its surface low coordinated oxygen's as reported by Ferretti and et al [9]. Low coordinated oxygen's such as 5 coordinated, 4 coordinated and 3 coordinated oxygen's are present on surface, terrace and corners on crystal lattice. Corma and Iborra [10] observed that 3 coordinated oxygen's are more basic over 4 and 5 coordinated oxygen. But these low coordinated basic sites of MgO is masked by chemisorption of CO_2 and water vapors at ordinary temperature. CO_2 and water responsible for carbonation and hydroxylation of magnesium oxide surface and hence basic activity of surface is destroyed. Tanabe and Yamaguch [11] showed that at very high temperature CO_2 and water are eliminated for preservation of surface basic activity. For this purpose, MgO solid base is activated at very high temperature prior to use in catalytic applications. High energy consumption by conventional thermal heating limits use of MgO in catalytic applications.

Microwaves are electromagnetic radiations ranging from 300 MHz to 300 GHz wavelength suggesting its less energetic nature. However, Raghubar [12] reported that microwaves are responsible for heating materials through dielectric polarization, dipolar polarization and conduction effect. Industrially, microwave heating is employed for ore grinding, pretreatment of gold, reduction of metal oxides, drying and anhydration, leaching of minerals and waste management [13-18]. Such multipurpose applicability of microwave heating required its use in catalytic applications too [19]. Many reports suggest microwave can be successfully used for activation of catalysts for chemical reactions. With this view, proposed research work uses microwave heating for surface activation nanocrystalline MgO as a solid base catalyst in Wittig reaction and results are compared with conventional thermal activation.

METHODOLOGY

Commercial MgO, NaOH flakes, benzaldehyde, ethyl bromoacetate triphenyl phospine, phenolphthalein benzoic acid and organic solvents were purchased from Loba Chemicals and used without further purification. The nanocrystalline MgO catalysts are characterized by XRD, FESEM, UV-DRS, FT-IR techniques. X-ray diffraction (XRD) analysis of the synthesized material was carried out using Bruker AXS model D-8, (10 to 80° range, scan rate =1° min^{-1}) equipped with a monochromatic and Ni-filtered Cu Kα radiation. SEM analysis was used to determine particle morphology of the desired powder catalysts using HITACHI S-4800 model. The UV-DRS spectra are determined using Shimadzu UV-3600 instrument. For characterization of Wittig reaction product ethyl cinnamate, HR-MS is taken on Bruker Compass Data Analysis 4.2. ^{1}H-NMR analysis of products was carried out using Bruker model.

Preparation of nanocrystalline MgO: For preparation of nanocrystalline MgO, 5 g commercial MgO powder is added with stirring to 100 ml 10 M NaOH in 200 ml capacity teflon reactor with stainless still outer jacket. It is subjected for alkali leached hydrothermal reaction at 180 °C for 24 hrs. After cooling, the reaction content is transferred to 500 ml water in a beaker. It is diluted and washed with 20 liter deionized water and excess alkali is neutralized with very dilute HCl. This content is filtered on suction pump using whatmann filter paper no 41 and dried at 60°C for 6 hrs. The resulting magnesium hydroxide is calcined at 450 °C according to procedure of Ding and et al [20] to obtain nanocrystalline MgO catalyst.

Activation of nanocrystalline MgO under thermal heating: For measurement of catalytic activity of nanocrystalline MgO, it is activated thermally at 700 0C in a muffle furnace for different scale of time in minutes and immediately used in Wittig reaction under grindstone techniques.

Activation of nanocrystalline MgO under Microwave heating: For measurement of catalytic activity of nanocrystalline MgO, it is heated in commercial microwave oven at 500 Watt for different scale of time in minutes and immediately used in Wittig reaction under grindstone technique.

Preparation of phosponium salt from ethyl bromoacetate and triphenyl phospine: Ethyl bromoacete and triphenyl phspine are stirred in toluene solvent in one neck R. B overnight. The resulting product was filtered over Buchner funnel on vacuum pump and washed with excess of toluene solvent. The product was vacuum dried.

Measurement of catalytic activity of nanocrystalline MgO in Wittig reaction: (6.7 mmoles) benzaldehyde, (4.7 mmoles) phosponium salt are crushed with 0.100g activated MgO in a mortar using pestle for about one hour. Initially reaction mixture liquefies but after this it solidified indicating the completion of reaction. Reaction is monitered by TLC. After this DMF solvent is added in reaction mixture and catalysts is separated by centrifugal separation at 5000 rpm. Reaction mixture is subjected for work up with water and ethyl acetate to remove excess of DMF. Products olefin and triphenyl phospine oxide are purified and separated on silica loaded column using pure hexane as eluent. The product of Wittig reaction is characterized by HR-MS, ^{1}H-NMR techniques.

RESULTS AND DISCUSSION

Preparation of nanocrystalline MgO is carried out using alkali leached hydrothermal method as given in experimental section. It is activated under both conventional heating as well as microwave heating. As synthesized product materials were characterized by using XRD, UV-DRS, FT-IR and Hammett indicator method.

XRD Analysis of nanocrystalline $Mg(OH)_2$ and nanocrystalline MgO:

Figure 1 shows the XRD patterns of nanocrystalline magnesium hydroxide (**fig. 1a**) and nanocrystalline MgO (**fig. 1b**). The XRD analysis of nanocrystalline MgO shows the XRD peaks are matching with $Mg(OH)_2$ the corresponding 2θ values at 18.52, 32.89, 37.99, 50.86, 58.63, 62.05, 68.32 and 72.04 degrees which corresponds to 001, 100, 101, 102, 110, 111, 103, 201 planes respectively. This 2θ values with corresponding planes indicates the hexagonal crystal system with brucite phase (JCPDS file no 44-1482). As synthesized $Mg(OH)_2$ is further calcined at 450 ^{O}C. At 450 ^{O}C the hexagonal $Mg(OH)_2$ is completely converted to cubic MgO **(Fig. 1b).** The corresponding 2θ values of XRD patterns at 36.91, 42.88, 62.29, 74.68, and 78.61 with planes 111, 200, 220, 311, 222 respectively are well matched with cubic phase of MgO (JCPDS file no 45-0946).
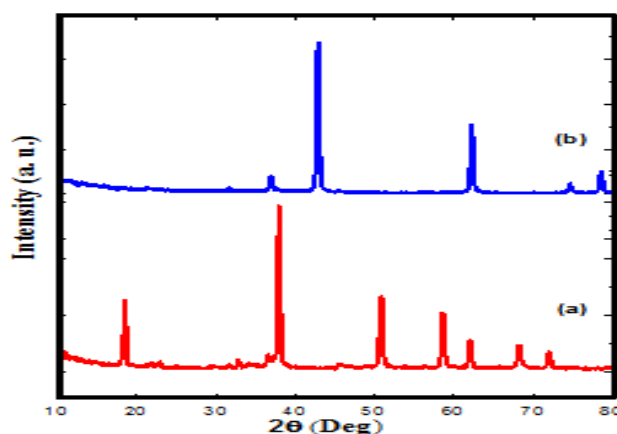
Figure 1. XRD Analysis of nanocrystalline $Mg(OH)_2$ and nanocrystalline MgO

UV-DRS Analysis of nanocrystalline $Mg(OH)_2$ and nanocrystalline MgO.:

Figure 4 shows the UV diffused absorption spectra of nanocrystalline $Mg(OH)_2$ (**fig. 2a**) and nanocrystalline MgO (**fig. 2b**) shows formation of pure phase. For nanocrystalline $Mg(OH)_2$ shows absorption band at 271 nm with red shift at 347 nm as observed by [21]. The sharp absorption band at 271 nm confirms the presence of Mg^{+2} ion. These multiple sharp absorption band of UV-DRS analysis is an important tool for gaining the idea about surface basicity of MgO. In case of nanocrystalline MgO absorption band at 230 nm and 280 nm are responsible for presence of 4 coordinated and 3 coordinated surface oxygen's [2]. In

case nanocrystalline MgO, the absorption band at 281 nm with red shift at 369 nm suggests the presence of only 3 coordinated surface oxygen's. The band at 230 nm is not appeared confirming the absence of 4 coordinated oxygen's on surface. This indicate the hydrothermal alkali leached methodology is useful enhance the more reactive 3 coordinated on surface of MgO.
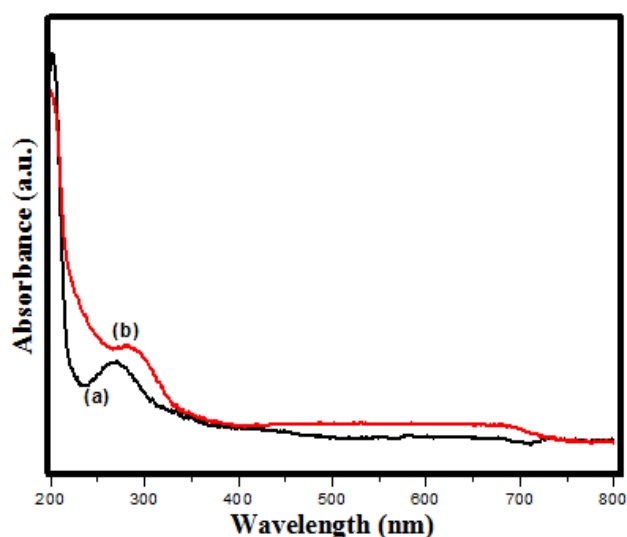
Figure 2. UV-DRS Analysis of (a) nanocrystalline Mg(OH)$_2$ and (b) nanocrystalline MgO

FT-IR Analysis of nanocrystalline Mg(OH)$_2$ and nanocrystalline MgO.:

Figure 5 shows the FT-IR spectrums nanocrystalline Mg(OH)$_2$ and nanocrystalline MgO. Two main regions in range of 400-850 cm^{-1} are observed in FT-IR spectrum. In first region, the bands due to different fundamental Mg-O vibrations are observed between 400-500 cm^{-1} as observed by Raman [23] In second region combination bands of fundamental vibrations appeared between 550-850 cm^{-1} [24]. Different types of IR bands in this two regions suggests the Mg-O vibrations and also.

Two bands appeared at between 1400-1500 cm^{-1} indicates the adsorption of CO$_2$ on MgO surface which is also supported with a band near small band at 2300 cm^{-1}[25]. The sharp peak at 3693 cm^{-1} indicates the O-H stretching vibrations in Mg(OH)$_2$. No significant bands are appeared between 3400-3600 cm^{-1} for nanocrystalline MgO confirms the calcination of Mg(OH)$_2$ to cubic MgO.
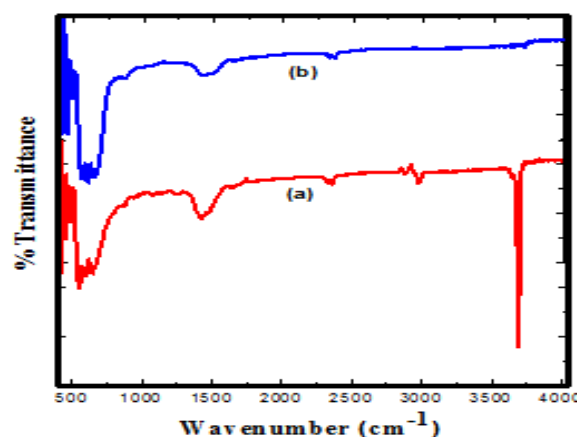
Figure 3. FT-IR Analysis of (a)nanocrystalline Mg(OH)$_2$ and (b)nanocrystalline MgO
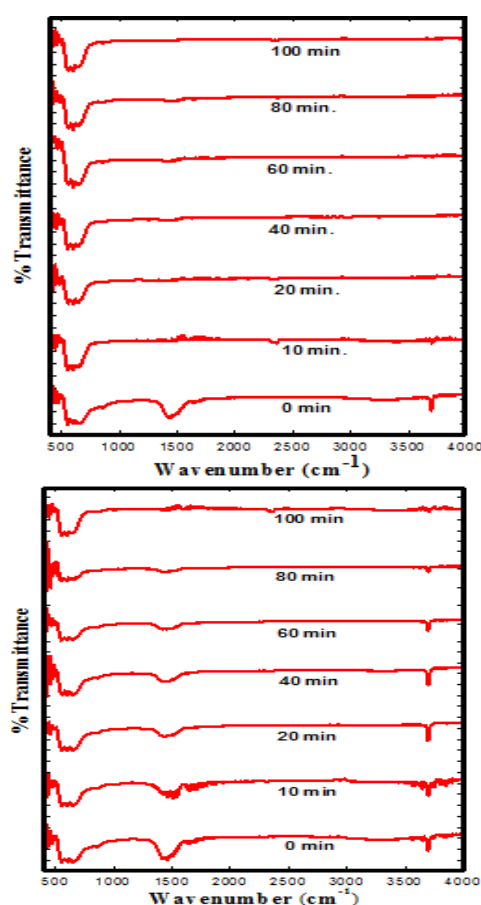
Figure 4. FT-IR Analysis for (a) Thermal activation and (b) Microwave activation of nano. MgO for different scale of time

FT-IR studies gives clue about the surface and corner oxygen which adsorb CO$_2$ and water vapors at ordinary temperature. Very high temperature is required for elimination of CO$_2$ and water vapors hence for surface activation. For this purpose, nanocrystalline MgO is activated under thermal

heating at 700 ^{0}C (**fig. 3a**) and microwave heating at 500 watts (**fig. 3b**) for different scales of time for 0, 10, 20, 40, 60 80 and 100 minutes for surface activation. IR spectra for each time are compared for surface carbonates and hydroxyl groups for both heating approaches. For thermally activated nanocrystalline MgO, the IR absorption peak at 1400-1600 cm^{-1} and 3600 cm^{-1} for carbonates and hydroxyls respectively are completely vanishing confirming activation of surface within 20 minutes. But for microwave activated nanocrystalline MgO, even after 100 min heating, persistence of carbonates and hydroxyls peaks confirming the MgO surface is still covered with CO_2 and water vapors. Careful observation of microwave heated FT-IR spectra for carbonate and hydroxyl peaks shows that MgO is not good microwave absorber as observed by Aguilar and Pearce [26] by thermocouple study. FT-IR study shows that thermal activation method is superior over the microwave activation method.

FESEM Analysis of nanocrystalline Mg(OH)$_2$ and nanocrystalline MgO:

Figure 5 shows FE-SEM photographs of nanocrystalline Mg(OH)$_2$ and nanocrystalline MgO. The FESEM photograph of Nano sized Mg(OH)$_2$ shows formation of nano sheets with 20 nm thickness under alkali leached hydrothermal condition. This nano sheets during the calcination of Mg(OH)$_2$ breaks down into nano sized MgO hexagonal sheets with same thickness. This hexagonal plates are stacked on above each other. FESEM photographs shows, at nano size large number of low coordinated oxygen's on surface of MgO increases. MgO at nano size exhibit maximum basic catalytic activity.
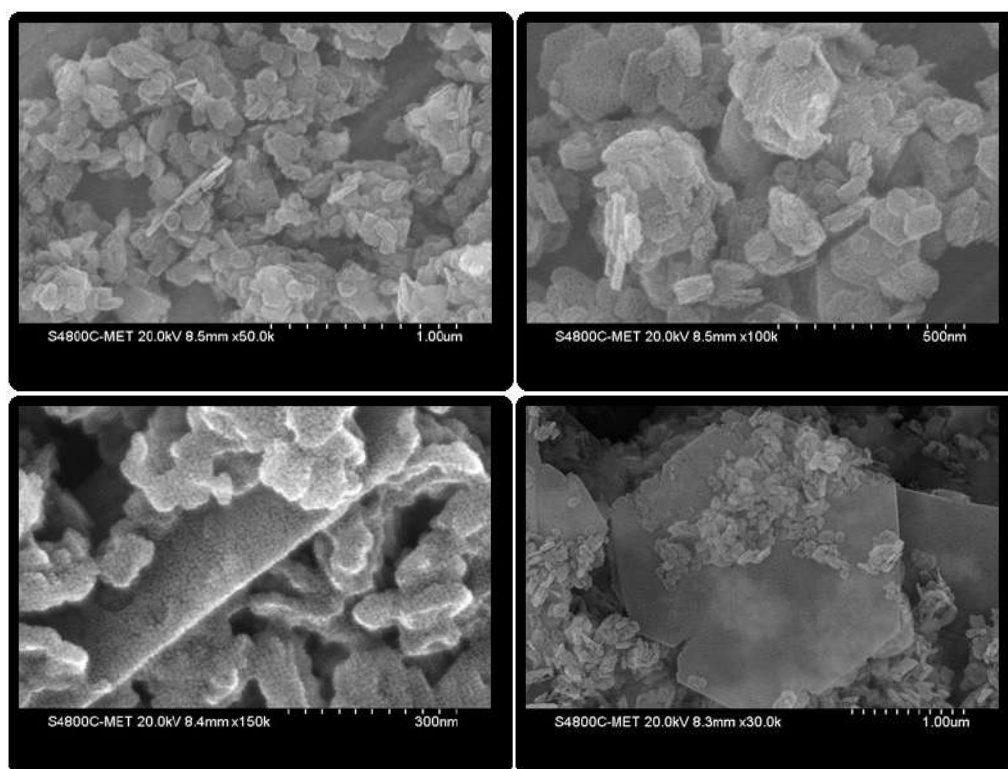
Figure 5. FESEM-Photographs of (a-b) nanocrystalline Mg(OH)2 and (c-d) nanocrystalline MgO

Scheme 1. Wittig reaction of benzaldehyde with phosponium salt in presence of nanocrystalline MgO using Grindstone technique.
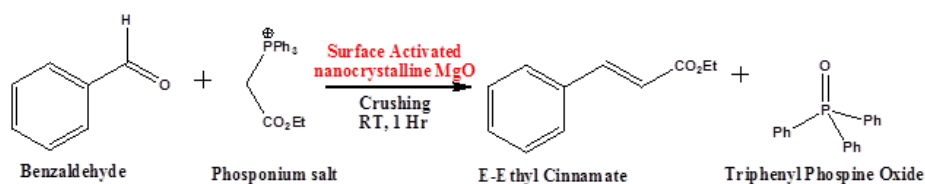
Table 1. Effect of thermal and microwave activation on catalytic activity of nanocrystalline MgO In Wittig reaction

No	Time of Heating/Min	Surface basicity/ mmoles/g	
		Thermal heating (700 °C) (a)	**Microwave heating (500 W) (b)**
1	0	40	40
2	10	50	45
3	20	52	48
4	40	65	50
5	60	72	55
6	80	80	62
7	100	92	65

Reaction condition: 6.9 mmoles benzaldehyde and 4.6 mmoles phosponium salt crushed with 0.100 g nanocrystalline MgO for 1 hr at RT.

Table 2. Effect of thermal and microwave activation on surface basicity's of nanocrystalline MgO in Wittig reaction.

No	Time of Heating/Min	Surface basicity/ mmoles/g	
		Thermal heating (700 °C) (a)	**Microwave heating (500 W) (b)**
1	0	0.02	0.02
2	10	0.1	0.08
3	20	0.25	0.16
4	40	0.38	0.22
5	60	0.55	0.30
6	80	0.70	0.40
7	100	0.85	0.48

Titration condition: 0.100 g nanocrystalline MgO is suspended in non-polar 10 ml benzene solvent, 1 ml phenolphthalein indicator allowed to adsorb over its surface and titrated with 0.1 N benzoic acid.

Measurement of catalytic activity of thermal and microwave activated nanocrystalline MgO:

With a specific amount of activated nanocrystalline MgO (0.100 g) catalysts 6.9 moles benzaldehyde and 4.76 moles phosponium salt are crushed in mortar and pestle for 1 hr. After workup and purification, Wittig product ethyl cinnamate is characterized by HR-MS and ^{1}H-NMR. Basic catalytic activities are compared for thermal and microwave activation method.

Measurement of surface basicity of nanocrystalline MgO for thermal and microwave heating.

As observed from table 1. Catalytic activity of nanocrystalline MgO differs under thermal and microwave activation methods in Wittig reaction. Under thermal activation methods catalyst is activated within only 20 min so it shows 50 % catalytic activity for 20 min (**Table 1, entry 3a**) and 92 % (**Table 1, entry 7a**) for 100 min also catalytic activity regularly goes on increasing. Under microwave irradiation even after 100 min, catalyst is not activated indicating 45 % to 65 % catalytic activity within 20 to 100 min heating period. Catalytic activity of nanocrystalline MgO shows same trends as observed in FT-IR study. This shows that under microwave heating, MgO shows poor catalytic activity.

Hammett indicator method is used for determination of surface basicity of heterogeneous solid bases [27]. So this method is employed for surface basicity

determination of nanocrystalline MgO for both approaches. The phenolphthalein dye is adsorbed over MgO surface and after adsorption solution shows pink color. It is titrated with benzoic acid. So amount of benzoic acid required in ml is measure of surface basicity of nanocrystalline MgO in mmoles/g.

Table 2. represents the surface basicity's of nanocrystalline MgO activated under thermal and microwave heating. From observation's, it is clear that, surface basicity of nanocrystalline MgO is a function of activation temperature and condition. In thermal heating, change from 0.02 mmoles/g to 0.85 mole observed from 0 min to 100 min so there is approximately 50-fold increase in basicity observed while under microwave activation surface basicity changes from 0.02 to 0.48 mmoles/g from 0 to 100 min suggesting for only 25-fold increase. Poor basicity of nanocrystalline MgO under microwave heating is attributed to coverage of surface active basic sites by CO_2 and water vapors, and very high temperature is required for removal of these. Thermally MgO can be effectively heated for activation while under microwave, due to inability of MgO to absorb heat, it is not activated even up to 100 min.

Thus, FT-IR techniques, catalytic activity in Wittig reaction and Hammett surface basicity measurement was employed in order to compare surface activation of nanocrystalline MgO under thermal and micro-

wave activation. Our techniques for comparisons are easy and useful in order to scaling up the organic reactions assisted by solid bases.

CONCLUSION

Pure nanocrystalline cubic MgO prepared by calcination of hydrothermally prepared nanocrystalline $Mg(OH)_2$. Nanocrystalline MgO is activated under thermal and microwave heating for different scale of time. FT-IR study for different time in min suggests that nanocrystalline MgO can be easily activated under thermal condition rather than microwave. Catalytic activity study in Wittig reaction indicates that thermally activated MgO is more basic over microwave activated and surface basicity measurement by Hammett indicator method shows that under thermal heating nanocrystalline MgO can be quickly activated than microwave heating.

In nutshell, thermal activation of nanocrystalline MgO is superior over microwave activation for catalytic applications.

Analysis of Wittig product: E- Ethyl cinnamate
1)HR-MS: (Mol. Formula $C_{11}H_{12}O_2$)
[M+H] ion for 177.09. (For $C_{11}H_{13}O_2$)
2)^{1}H-NMR (500 MHz, CDCl$_3$) for E-ethyl cinnamate
1.3 ppm (triplet, 3H, J=7.5 Hz), 4.25 ppm (quartet, 2H, J=7.5 Hz), 4.26 ppm (doublet, 1H, J= 16 Hz, 7.34 ppm (triplet, 3H, J=3.5 Hz), 7.48 ppm (quartet, 2H, J=3.5 Hz), 7.67 ppm (doublet, 1H, J=16 Hz).

REFERENCES:

1. Pushpaletha P. Rugmini S. Lalithambika M. *Catalysis Letters,* 2005, 30, 141-153.

2. Hideshi Hattori, *Chem. Rev.* 1995, 95, 537-538.

3. Xinhuan L, Jie H, Run J, Peipei T, Renfeng N, Dan Z. Qinghua X. *Scientific Reports*, 2017,7, 2676

4. Xie Z., Chen Q., Zhang C. Bao J., Cao Y., *J. Phy. Chem. B.* 2000, 104, 2853.

5. Noda C., Alt G. P., Werneck R. M., Henriquesj C. A, Monteiro L. F., *Braz. J. Chem. Eng.* 1998,

6. Biplab R. Anupam S, Asit S., Panda B.,Sk. M. I. Chattopadhyay A. P.*ChemistrySelect*, 2016,1,4778-4784.

7. Moison H., Francoise T-B, Andre F. *Tetrahedron.* 1987, 43(3), 537-542

8. Horst P., *Angewandte Chemie*.1977, 16(7), 423-429.

9. Ferretti C. A., Fuente S., Ferullo, R., Castellani, N., Apesteguiaa, Di Cosimo. C. R. *Appl Catal A: General,* 2012,413-414, 322-331.

10. Corma, A., Iborra, S., *Adv. Catal*. 2006, 49, 239-302.

11. Tanabe, K., Yamaguch, T., *J. of Res. Inst. Catal. Hokkaido University*. 1965,11,179-184.

12. Raghubar S., *Int. J. Ex Know*. 2014, 1(1), 43-36.

13. Walkiewicz J.W., Kazonich G., McGill, S.L, *Mineral and Metallurgical Processing,*1988 5 (1) 39–42.

14. Haque, K.E., *Min. Proc. Extv. Metall. Rev*. 1987, 2,235-253.

15. Standish N., Worner, H. *Iron and Steel Maker*, 1991, 18(5), 59-61.

16. Javad Koleini S. M. and Kianoush B. *Microwave Heating Applications in Mineral Processing*. Chapter 4, 95. Intech Publcation.

17. Kingston H.M., Jassie L.B., 1985, *Introduction to microwave sample preparation, theory and practice,* Chapters 2 and 3, ACS professional reference book, Am. Chem. Soc.

18. Wicks G.D., Clark D.E., Schulz, R.L., Folz D.C, *Microwaves: theory and application in material processing.* 1995,111, pp. 79–89.

19. Kustov L. M., Sinev I. M., *Russian Journal of Physical Chemistry A*, 2010, 84(10), 1676-1684.

20. Ding Y., Zhang G., Wu H., Hai B., Wang L., Qian, Y., *Chem. Mater*. 2001, 13 435-440.

21. Fatima Parveen M.,Umapathy S., Dhanalakshmi V., Unbarasan R., *NANO: Brief Reports and Reviews*.2009 ,4(3), 147-156

22. Hattori H., *J. Jap. Pet. Inst.*, 2004, 47, 67-81.

23. Raman C. V., *Proc. Indian Acad. Sci. A*. 1947, 26, 383-390.

24. Raman C. V., *Proc. Indian Acad. Sci.* 1961,54. 205-222.

25. Philippt R. Fujimoto K. *J. Phys. Chem*. 1992,96, 9035-9038.

26. Aguilar J. Pearce J., *British Ceramics Transactions,* 2003,102(2), 52-56.

27. Jacques C. V., *Res. Chem. Intermed.,*2015, 41:9387–9423

RESEARCH ARTICLE   **OPEN ACCESS**

Dielectric behavior of Al³⁺ substituted Cd ferrites

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ABSTRACT

The spinel ferrite series of $Cd_1Al_xFe_{2-x}O_4$ where x varies from x=0.0 to 0.5 in the steps of 0.1 were prepared by the conventional double sintered ceramic method. The formation of single phase spinel structure was confirmed from XRD. The dielectric measurements were carried out as a function of temperature using two probe method with L-C-R-Q meter. The dielectric constant, dielectric loss and dielectric loss tangent are calculated using the data. The dielectric parameters show normal dielectric behavior. The dielectric properties i.e., dielectric constant (ε'), dielectric loss (tan δ) decrease with Al³⁺ ion doping.

Keywords: Spinal, Ferrite, Composite Material, Dielectric.

INTRODUCTION

The studies in spinel ferrites are of enormous importance because of their attractive combined electric and magnetic properties. The magnetic properties of ferrites make them useful in transformers, ultrasonic generators, modulators, phase shifters, isolators, memory devises, recording devices, sensors etc. In spinels the intra sub-lattice interactions are weaker than the inter sub-lattice interactions. Due to this there are unsatisfied bonds in the ferrimagnetic region which brings various exchange interactions. These exchange interactions in spinels allows variety of magnetic orders. The magnetic order can be controlled by proper cation substitution. By substituting proper magnetic and non-magnetic cations we can get desired properties resulting from cation distribution in crystal lattice.

State level Conference on "Recent Approaches in Environmental Protection & Sustainable Development" 2018

The substitution for Fe^{3+} by another trivalent cation is one of the most effective means to control saturation magnetization. The dielectric study of Al^{3+} substituted Fe_3O_4 ferrite nanoparticles N. Kumari, Vinod Kumar, and S. K. Singh, *Int. J. Mod. Phys. B* **28**, 1450193 (2014) has been studied by K. M. Jadhav et. al. [1]. Electrical Properties of Cadmium Substitution in Nickel Ferrites has been studied by Ande Ashok et. al. [2]. Effects of cadmium on physical and magnetic properties of Co-Cd ferrites has been studied by Saroaut Noor et. al. [3].

In the survey of reported work so far, there is no information available to our knowledge about the systemic study of the dielectric behavior of mixed magnetic oxides $CdFe_{2-x}Al_xO_4$. Therefore, the present work is an attempt to study the effect of substitution of non-magnetic cation Al^{3+} on the dielectric properties Cd ferrites.

METHODOLOGY

Synthesis of $Cd_1Al_xFe_{2-x}O_4$

The spinel ferrite series of $Cd_1Al_xFe_{2-x}O_4$ where x varies from x=0.0 to 0.5 in the steps of 0.1 were prepared by the conventional ceramic method. The chemicals for ferrite systems were of analytical grade high purity oxides CdO, Al_2O_3 & Fe_2O_3 were mixed in proper proportion so as to yield the desired stoichiometry composition. Each of these compositions was ground for half an hour in an agate mortar. This mixture was then presintered at 850°C for 24 hours then slowly cooled to room temperature. The presintered samples were gain milled to fine powder. The powder was then pressed at around 5 tones/square inch of pressure to form pellets of about 1 cm in diameter. Pellets of good quality were obtained by using poly vinyl alcohol as binder & maintaining the pressure for about ten minutes each time. The pellets were finally sintered at 900°C for 24 hours and naturally cooled to room temperature.

The X-Ray diffraction patterns for all the powdered samples were recorded on X-ray diffractometer by using CuKα radiation with wavelength 1.542 Å.

The dielectric measurements were carried out as a function of temperature using two probe method with L-C-R-Q meter.

RESULTS AND DISCUSSION

X-ray diffraction
The X-ray diffraction patterns of all the six samples of the $Cd_1Al_xFe_{2-x}O_4$ system are shown in **figure (1)**. The formation of single phase spinel structure was confirmed from XRD.
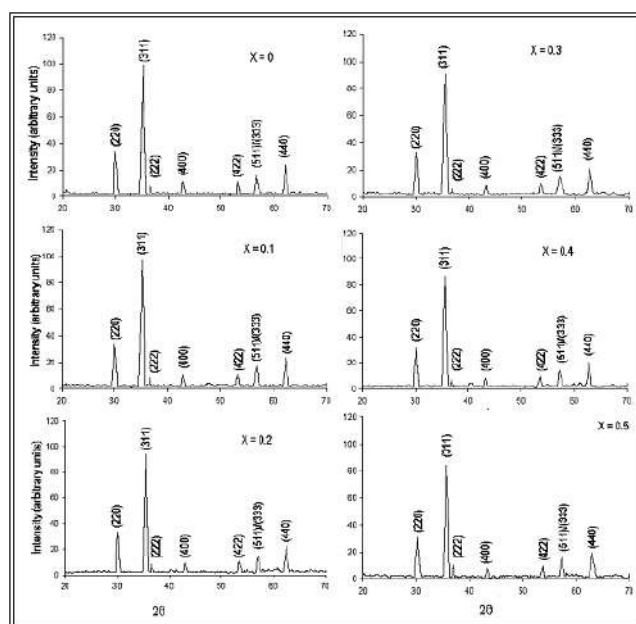
Figure1. Typical X-ray diffractograms of the $Cd_1Al_xFe_{2-x}O_4$ system.

Dielectric Properties
The dielectric measurements were carried out as a function of temperature using two probe method with L-C-R-Q meter at the Dept. of Physics, Dr. B.A.M.U. Aurangabad. The dielectric constant, dielectric loss and dielectric loss tangent are calculated using the data.

The values of dielectric constant (ε′) have been calculated and the variation of dielectric constant as a function of temperature at 1 K.Hz. frequency for all the samples is shown in **figures (2 to 3)**. The dielectric constant is found to increase gradually with increase in temperature up to 600 K and thereafter increases rapidly with temperature.
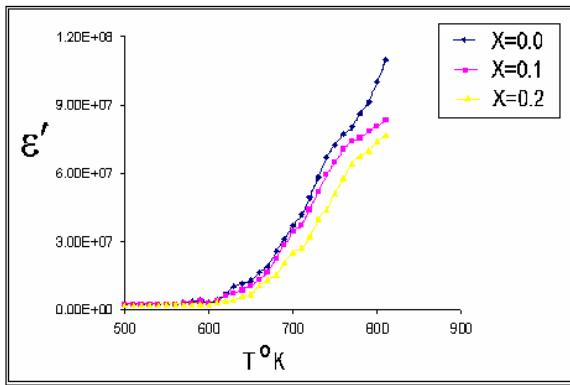
Figure 2. Variation of dielectric constant **(ε′)** as a function of temperature for $Cd_1Al_xFe_{2-x}O_4$
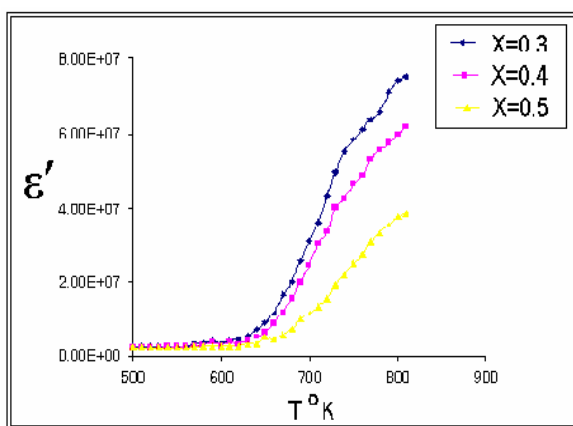
Figure 3. Variation of dielectric constant **(ε′)** as a function of temperature for $Cd_1Al_xFe_{2-x}O_4$

The variation in dielectric constant is due to interfacial polarization. The interfacial polarization arises due to distribution of electrical resistivity in ferrites because of non-uniform distribution of oxygen ions induced during the sintering process. Polarization results in an electronic exchange between Fe^{2+} and Fe^{3+} ions which produces local displacement in the direction of applied external field. Similarly the Al^{3+} and Al^{2+} ions give hole concentration which produces the local displacement opposite to the direction of applied field. These displacements determine the polarization and dielectric properties in the ferrites. The observed decrease in dielectric constant is normal dielectric behavior which may be due to interfacial polarization explained on the basis of Koop's theory [4] in accordance with Maxwell–Wagner two layer modal according to which ferrite materials consist of fairly conducting grains separated by less conducting grain boundaries.

Resistivity and dielectric constant are inversely related to each other. Since resistivity of ferrites decreases with the increase in temperature, the increase in dielectric constant with the increase in temperature is the expected result. Such behaviour has been reported in number of ferrite systems [5-7]. As the temperature rises, the number of charge carriers increases resulting in increase of space charge polarization [8-9] and hence there is increase in dielectric constant. With the increase in Al^{3+} concentration the number of charge carriers decrease resulting in decrease of dielectric constant.
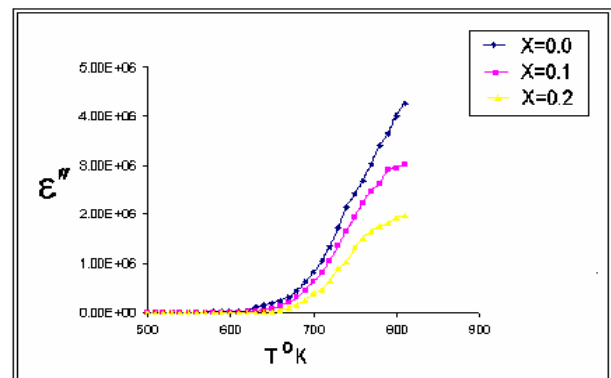
Figure 4. Variation of dielectric loss **(ε″)** as a function of temperature for $Cd_1Al_xFe_{2-x}O_4$
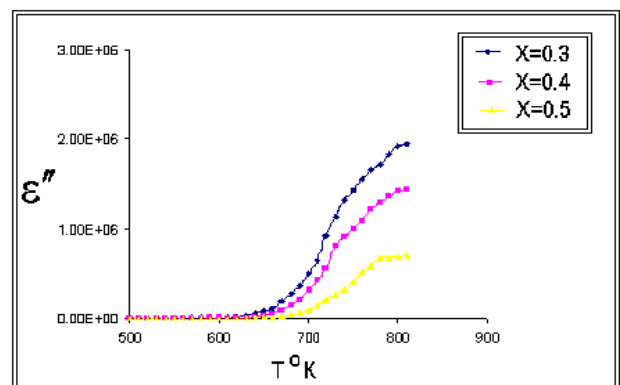
Figure 5. Variation of dielectric loss **(ε″)** as a function of temperature for $Cd_1Al_xFe_{2-x}O_4$

The variation of dielectric loss (ε″) with temperature for all the samples is shown in **figures (4 to 5)**. It is observed that the dielectric loss varies very slowly up to temperature 650 K (approximately) and then increases rapidly with the increase in temperature. This behavior of dielectric loss is in full agreement with that obtained by V. A. Loffe et. al. [10] for spinel

ferrites. The dielectric loss decreases with increase in Al^{3+} concentration of the system [11].
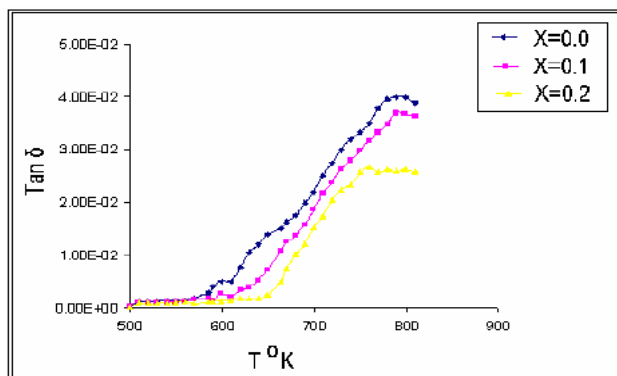
Figure 6. Variation of dielectric loss tangent **(tanδ)** as a function of temperature for $Cd_1Al_xFe_{2-x}O_4$
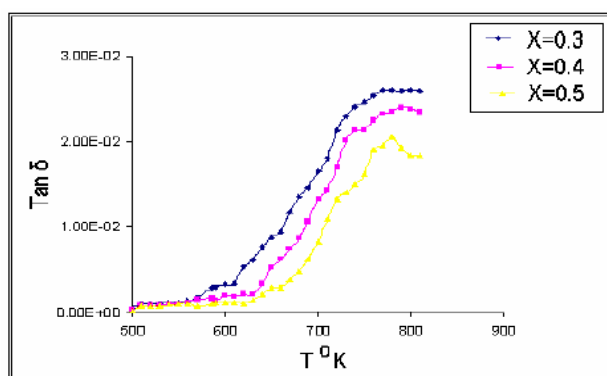
Figure 7. Variation of dielectric loss tangent **(tan δ)** as a function of temperature for $Cd_1Al_xFe_{2-x}O_4$

The thermal variation of dielectric loss tangent (tanδ) is as shown in **figures (6 to 7).** Similar to dielectric loss, dielectric loss tangent (tanδ) also increases slowly in the beginning and then rapidly increases with the increase in temperature. It is in agreement with a general rule that (tanδ) appreciably increases with the rise in temperature [12]. The dielectric loss tangent decreases with increase in Al^{3+} concentration of the system.

CONCLUSION

The system $Cd_1Al_xFe_{2-x}O_4$ with x = 0 to 0.5 (with step of 0.1) has a single phase spinel structure. The dielectric constant decreases with increase in Al^{3+} content. The dielectric loss decreases with the increase

in Al^{3+} content. The dielectric loss tangent decreases with increase in Al^{3+} concentration of the system. The dielectric parameters show normal dielectric behavior in accordance with Maxwell–Wagner two layer models. Low value of dielectric loss (tan δ) obtained in these ferrites is suitable for devices where low core losses are required

REFERENCES

1. Kumari N, Kumar Vinod and Singh SK. *Int. J. Mod. Phys.,*2014, B28:1450193.
2. Ande Ashok et. al. *World J. of Condensed Matter Phys.,*2012, 2:257-266 .
3. Saroaut Noor et. al. *J. of Ban. Acad. of Sci.,*2011, Vol.35, No. 2:229-235,
4. Koops CG. *phys Rev.,*1951, 83:121.
5. Shinde SS & Jadhav KM. *Material Letters,*1998, 37:63-67.
6. Shaikh AM, Bellad SS & Chaugule BK. *J. Magn. Magn. Mater.*, 1999, 195: 384.
7. Bellad SS & Chaugule BK. *Mater. Res. Bull.,* 1998, 33(8): 1165.
8. Iwauchi K. *J. Appl. Phys.,1971,* 10: 520.
9. Rezlescu N & Rezlescu E, *Phys. Status Solidi (a),*1994, 23.
10. Ioffe VA, Khvostenko GI & Zonn ZN. *Sov. Phys. JT*, 1957,27: 1985.
11. Rabin LI & Novikova ZI, Ferrites, *Mansik,* 1960, 146.
12. Tareev B. Physics of dielectric materials, *translated from Russian by Troitky A. English translation, Mir Publishers,*1975, 155-157.

Synthesis of Bioactive Chromone Derivatives

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ABSTRACT

Chromones (1-benzopyran-4-ones) and Chromone
derivatives are naturally occurring compounds
ubiquitously found in the plant kingdom, and therefore
present in representative amounts in a normal human
diet.These phytochemicals possess a wide spectrum of
biological activities – such as anti-inflammatory,
antifungal, antimicrobial, antiviral, antitumour and
anticancer-mainly due to their well-recognized
antioxidant properties, which stem from their ability to
neutralize active forms of oxygen and to cut off free
radical processes. Here we successfully synthesize some
chromone derivatives by using substituted phenols in
laboratory at room temperature. Crystalline products
were characterized using sophisticated techniques such
as FT-IR, NMR and mass spectrophotometric methods.

Keywords: chromones; derivatives; biological activity.

INTRODUCTION

The chromone classes of compounds it was found that
they are associated with various physiological and
biological properties and find importance in medicine.
Taking these facts in to consideration it was thought
worthwhile to synthesize a series of such compounds. A
number of scientists in the past have tried to find out
some relationship between chemical structure and
physiological or biological properties. It is now well
established fact that the activity of the compounds
depends upon three factors. The first and perhaps most
important is the heterocyclic moiety present in particular
compound. The second factor is the nature of the
substituents and the third factor is the position of
substituent's in these compounds.

Chromone derivative have received significant attention owing to their diverse range of biological properties viz. blood platelet disaggregation, antimicrobial, anthelmintic, antibacterial, anti-inflammatory, anti-hypertensive, antifungal.

There is growing interest in the pharmacological potential of natural products is chromones constitute an important group of natural products. Chemically, they consist of open chain flavanoids in which the two aromatic rings are joined by a three carbon α. β unsaturated carbonyl system. The presence of a reactive α, β unsaturated keto function in chromones is found to be responsible for their antimicrobial activity [1]. In recent years a variety of chromones have been reviewed for their cytotoxic, anticancer chemoprevenive and mutagenic as well as antiviral, insecticidal and enzyme inhibitory properties [2,3].A number of chromones having hydroxy, alkoxy groups in different position have been reported to possess anti-bacterial [4], antiulcer [5], antifungal [6], antioxidant [7], vasodilatory [8], antimitotic [9], antimalarial [10], antileshmanial [11] and inhibition of chemical mediators release, inhibition of leukotriene B4 [12], inhibition of tyrosinase [13,14] and inhibition of aldose reductase [15] activities. Appreciation of these findings motivated us to synthesize chromones as a potential template for antimicrobial agents. It must be noted that this scaffold provides substitution pattern on benzylidene acetophenones nucleus.

The main objectives of chromones synthesis are not only for the development of more diverse and complex bioactive compounds for biological activity and structure activity relationship studies but also for the applications in medicinal chemistry, such as preparation of fluorescence probes, due to photochemical properties of chromones. Chromone derivatives have high potential in drug discovery. Synthesis of large compound libraries is a general trend in a modern drug discovery process. In recent yearsa lot of synthetic method to construct the Chromone ring appeared. We want to study the synthesis of chromones by some methods they may include acid as catalyst, base as catalyst, microwave irradiation assisted synthesis, solid-supported synthesis, and other methods.

The present work describes the synthesis of series of some Chromones. With referring literature of the above classes of compounds it was found that they are associated with various physiological and biological properties and find importance in medicine. Taking these facts in to consideration it was thought worthwhile to synthesize a series of such compounds.

METHODOLOGY

The synthesized compound for its own identification like different types of bonds, functional groups, carbon skeleton, polarity, molecular weight, refractive index etc. different chemical and physical methods are available. We know that chemical method becomes time consuming and wastage of much chemicals. Chemical method gives ideas about functional group, aromaticity. Saturation, unsaturation and which elements present in the compounds. However, analysis by chemical method can't reach up to structure of the compound. Overcome this time consuming and wastage of much chemicals, physical method become fast and gives more structural information about chemicals without wastage of chemicals.

The melting points of synthesized compounds were determined in open capillary tubes using melting point apparatus, expressed in °C and are uncorrected. The IR spectra of compounds were recorded on Shimadzu Affinity-1 FTIR in KBr disc and absorption bands are expressed in cm^{-1}. The ^{1}H NMR spectra in DMSO were recorded on Bruker WM 400FT MHz spectrometer and chemical shift were reported as parts per million (ppm) down field using TMS as internal standard. The HRMS spectra on dic-ms600mz were recorded on Bruker Compass Data Analysis. The purity of the compounds was checked by TLC on silica gel Glassplates using ethyl acetate: hexane (1:3) solvent system.

RESULTS AND DISCUSSION

Synthesis of Chromone
Expermental:
Step-I
5gm of phenol, 6 ml acetic anhydride and 5 ml pyridine (dry)take in dry conical flask keep for

overnight,second day pour over crushed ice, Organic layer separated by separating funnel. Organic layer separate by anhydrous sodium sulphate or magnesium sulphate. Distilled organic layer above 200°C.

Substituted Phenol   Ester
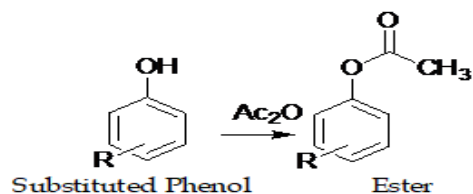
Step-II

Take 6gm anhydrous $AlCl_3$ in 50ml round bottom flask. Attach air condenser and then add above ester to the flask, reaction start within 2 min. After 10 minutes heat the reaction mixture in round bottom flask for two hours between 135-150°C, allow to stands for overnight then keep the flask in ice bath and add crushed ice to the flask. Product will separate within two days. Filter the product and recrystallize with alcohol.

Substituted Acetophenone
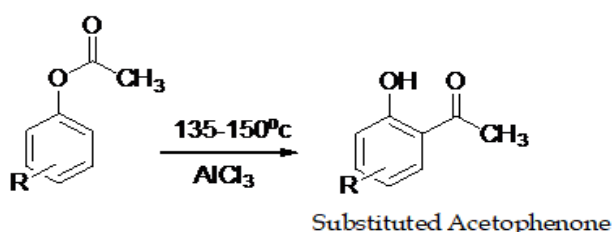
Step-III

5gmacetophenone dissolve in minimum amount of DMF. Take 8-10 ml DMF in round bottom flask and cool to 0°Cand 15ml $POCl_3$ dropwise, this ice cold solution. Add above solution in RBF dropwise exothermic reaction then stir the reaction mixture for 30 min at room temperature. Stir for 15 min. then keep reaction for overnight. Second day pour mixture over crushed ice(25gm). Keep for 2-3 hrs. Solid get separated. Recrystallize with alcohol and if insoluble in alcohol then recrystallize with acetic acid.
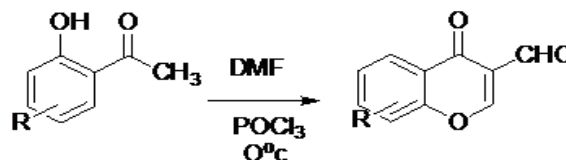
Substituted Chromone
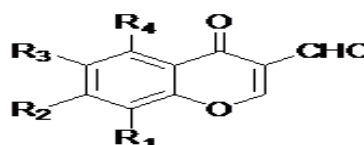
A series of Different Chromones prepared as follows.

Sr.No.	R1	R2	R3	R4
1	Cl	H	Cl	H
2	H	H	Cl	H
3	H	CH_3	H	CH_3
4	H	H	Br	H
5	H	CH_3	H	H
6	CH_3	H	CH_3	H
7	H	H	CH_3	H
8	H	Cl	H	Cl
9	H	CH_3	Cl	H

Sr.No.	Name of the compound	Melting point(°C)	%Yield
1	6,8dichloro-4-oxo-4H-Chromene-3-carbaldehyde	171	79
2	6, chloro-4-oxo-4H-Chromene-3-carbaldehyde	157	83
3	6-bromo -4-oxo-4H-Chromene-3-carbaldehyde	193	85
4	7 methyl -4-oxo-4H-Chromene-3-carbaldehyde	157-165	89
5	6,8 dimethyl -4-oxo-4H-Chromene-3-carbaldehyde	187-190	84
6	6,chloro-7methyl -4-oxo-4H-Chromene-3-carbaldehyde	183-185	90
7	5,7 dimethyl -4-oxo-4H-Chromene-3-carbaldehyde	165	92
8	6, methyl -4-oxo-4H-Chromene-3-carbaldehyde	173	84
9	5,7 dichloro-4-oxo-4H-Chromene-3-carbaldehyde	185	85

Spectral Analysis of some Chromones:

1. **6,8dichloro-4-oxo-4H-Chromene-3-carbaldehyde:**1H NMR: (300 MHz, DMSO)   : 10.079 (s, 1H, -CHO), 9.038 (s, 1H, 7-H), 8.264 (s, 1H,5H), 8.007 (s, 1H, 2-H).HRMS: m/z [M+] Calculated for $C_{10}H_4O_3Cl_2$: 243.4000;Found: 242.9613.

2. **6, chloro-4-oxo-4H-Chromene-3-carbaldehyde:**1H NMR: (300 MHz, DMSO)   : 10.093 (s, 1H, -CHO), 8.952 (s, 1H, 2-H), 8.042 (s, 1H,5-H), 7.933 (s, 1H, 7-H);7.835(s,1H, 8-H);HRMS: m/z [M+]Calculatedfor$C_{10}H_5O_3Cl$:209.0000;Found:209.0004.

3. **6-bromo -4-oxo-4H-Chromene-3-carbaldehyde:**1H NMR: (300 MHz, DMSO)   : 10.098 (s, 1H, -CHO), 8.964 (s, 1H, 2-H), 8.200 (s, 1H,5H), 8.060 (s, 1H, 7-H);7.700(s,1H, 8-H);HRMS: m/z [M+] Calculated for $C_{10}H_5O_3Br$: 253.4000;Found:254.9474.

4. **7 methyl -4-oxo-4H-Chromene-3-carbaldehyde:**1H NMR: (300 MHz, DMSO)   : 10.114 (s, 1H, -CHO), 8.881 (s, 1H, 2-H),8.032(d, 1H,5H), 8.017 (d, 1H, 6-H);7.574(s,1H, 8-H); 3.368(s,3H,CH$_3$)HRMS: m/z [M+] Calculatedfor$C_{11}H_8O_3$:188.8000;Found:189.0549.

5. **6,8 dimethyl -4-oxo-4H-Chromene-3-carbaldehyde:**1H NMR: (300 MHz, DMSO)   : 10.121 (s, 1H, -CHO), 8.921 (s, 1H, 2-H), 7.757(s, 1H,5H), 7.753 (s, 1H, 7-H);3.354(s,3H, 8-CH$_3$); 2.435(m,3H,6-CH$_3$)HRMS: m/z [M+] Calculated for $C_{12}H_{10}O_3$: 203.0000;Found: 203.0708.

6. **6,chloro-7methyl -4-oxo-4H-Chromene-3-carbaldehyde:** 1H NMR: (300 MHz, DMSO)   : 10.088 (s, 1H, -CHO), 8.917 (s, 1H, 2-H), 8.008(s, 1H,5H), 7.828 (s, 1H,8-H);3.353(s,3H, 7-CH$_3$); 2.435(m,3H,6-CH$_3$)HRMS: m/z [M+] Calculated for $C_{11}H_7O_3Cl$: 223.2000; Found:223.0158.

Conflicts of interest: The authors stated that no conflicts of interest.

REFERENCES

1. Rackova L, Firakova S, Kostalova D, Stefek M, Sturdik E and Majekova M. Oxidation of liposomal membrane suppressed by flavonoids: Quantitative structure–activity relationship., *Bioorg Med Chem*, 2005,13:6477-6484.

2. Middleton E, Kandaswami C and Theoharides TC. The Effects of Plant Flavonoids on Mammalian Cells: Implications for Inflammation, Heart Disease and Cancer., *Pharmacol Rev.*,2000, 52:673-751.

3. Nijveldt RJ, van Nood E, van Hoorn DEC, Boelens PG, van Norren K and van Leeuwen PAM. Flavonoids: a review of probable mechanisms of action and potential applications, *Am J ClinNutr.*, 2001,74: 418-425.

4. Harborne JB, Williams CA. Advances in flavonoid research since 1992, *Phytochemistry* 2000; 55: 481-504.

5. Pick A, Muller H, Mayer R, Haenisch B, Pajeva IK, Weigt M, Bonisch H, Muller CE, Wiese M.Structure–activity relationships of flavonoids as inhibitors of breast cancer resistance protein (BCRP), *Bioorg Med Chem* ,2011, 19: 2090-2102.

6. Amaral S, Mira L, NogueiraJMF,Da Silva AP, Florencio MH.Plant extracts with anti-inflammatory properties—A new approach for characterization of their bioactive compounds and establishment of structure–antioxidant activity relationships, *Bioorg Med Chem,* 2009, 17:1876-1883.

7. Mastuda H, Morikawa T, Ueda K, Managi H, Yoshikawa M.Structural requirements of flavonoids for inhibition of antigen-Induced degranulation, TNF-α and IL-4 production from RBL-2H3 cells, *Bioorg Med Chem*,2002, 10:3123-3128.

8. Ferrali M, Donati D, Bambagioni S, Fontani M, Giorgi G, Pietrangelo A. 3-Hydroxy-(4H)-benzopyran-4-ones as potential iron chelating agents in vivo, *Bioorg Med Chem,* 2001, 9:3041-3047.

9. Gong JX, Huang KX, Wang F, Yang LX, Feng YB, Li HB, Li XK, Zeng S, Wu XM, StoeckigtJ, Zhao Y, Qu J. Preparation of two sets of 5,6,7-trioxygenated dihydroflavonol derivatives asfree radical scavengers and neuronal cell protectors to oxidative damage, *Bioorg Med Chem*,2009, 17: 3414-3425.

10. Burda S, OleszekW.Antioxidant and Antiradical Activities of Flavonoids, *J Agric Food Chem*,2001; 49: 2774-2779.

11. Nowakowska Z. A review of anti-infective and anti-inflammatory chromones, *Eur J Med Chem*, 2007, 42: 125-137.

12. Burlando B, VerottaL,Cornara L, Bottini-Massa E.Herbal Principles in Cosmetics: Propertiesand Mechanisms of Action, *CRC Press, Taylor & Francis Group: Boca Raton, FL, USA*,2010.

13. Hadfield JA, Ducki S, HirstN, McGown AT, Progress in Cell Cycle Research, *Editions Life inProgress: New York*, 2003, 5: 309-325.

14. Bandgar B P, Gawande SS, Bodade RG, Totre JV, Khobragade CN. Synthesis andbiological evaluation of simple methoxylatedchromones as anticancer, anti-inflammatory and antioxidant agents, *Bioorg Med Chem* , 2010, 18: 1364-1370.

RESEARCH ARTICLE   **OPEN ACCESS**

Formulation and Evaluation of Diclofenac Sodium Oro Dispersible tablets using superdisintegrant by direct compression technique

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Manuscript Details

ABSTRACT

The present study was aimed to formulate, evaluate and optimized a tablet which disintegrates and dissolves rapidly to show a rapid onset of action. Diclofenac sodium, a non-steroidalanti-inflammatory drug with analgesic and anti-inflammatory properties was selected as a model drug. In the present study, attempt has been made to prepare fast dissolving tablets of Diclofenac sodium using superdisintegrants like sodium starch glycolate and starch by direct compression technique using 3 different concentration of superdisintegrant. The precompression parameters of prepared tablet blend like angle of repose, bulk density, tapped density, Carr's index, Hausner's ratio and the post compression parameters of the tablet like hardness, friability, weight variation, disintegration time, invitro dissolution release rate were evaluated. It was concluded that formulation F9 showed better release characteristics of the drug.

Keywords: Oro dispersible tablets, Diclofenac sodium, Sodium starch glycolate

INTRODUCTION

For decade, oral drug delivery has been known as the most widely used route of drug administration when compared to all the other routes that have been explored for delivery of different dosage forms to systemic circulation.

The reason for such popularity of oral route may be attributed to its ease of administration. Recent advances in novel drug delivery systems (NDDS) aim at formulating a convenient dosage form for administration and to achieve better patient compliance to enhance safety and efficacy of drug molecules. One such approach is oro-dispersible tablet. An oral fast dissolving drug delivery system is a novel tablet dosage form which dissolves or disintegrates in the oral cavity with a good taste and flavor increasing the acceptability of bitter drugs without the need of water or chewing and hence called melt in mouth tablets or oro-dispersible or rapid disintegrating or quick dissolving tablets. The drugs may be absorbed from mouth, pharynx or esophagus while the saliva passes down into stomach. Advantages of the fast dissolving tablets include rapid onset of action, ease of swallowing without the aid of water, enhanced dissolution rate, increased gastric absorption, minimized first pass metabolism, improved oral bioavailability and improved patient compliance. ODT formulation combines the advantages of both conventional tablets and liquid formulations.

In the present study it was proposed to formulate fast dissolving tablets of Diclofenac sodium by using direct compression technique, with the aim of reaching high serum concentration of the drug in a short time period. In this study, effort has been made to formulate the fast dissolving tablets using super dis-integrant like Sodium Starch Glycolate.
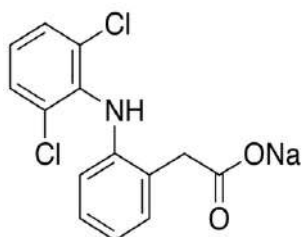
METHODOLOGY

Materials

All the materials used in this present work were commercial samples. Diclofenac sodium was received from Sahyadri scientific research, Islampur, Sodium starch glycolate received from Sahyadri scientific research, Islampur, Lactose purchased from Sahyadri

scientific research, Islampur, Magnesium stearate, Talc. All the reagents used were of analytical grade. Freshly prepared distilled water was used in the work.

Method
Oro dispersible Tablets of Diclofenac Sodium using Direct Compression Technique

Direct compression is used to define the process by which tablets are compressed directly from the powder blends of active ingredients and suitable excipients. No pre-treatment of the powder blends by wet or dry granulation is involved. Direct compression technique does not require the use of water or heat during the formulation procedure and is the ideal method for moisture and heat-labile medications. This is a process of compressing mixed powders into tablets without the need of intermediate granulating step. This technique involves conventional equipment, commonly available excipients and a limited number of processing steps. High doses can be accommodated and the final weight of tablet can easily exceed than that of other production methods.

Fast dissolving tablets of Diclofenac sodium were prepared by direct compression method. All the ingredients were passed through # 60mesh separately. Then the ingredients were weighed and mixed in geometrical order and compressed into tablets of 200mg by using 10-station rotary mini press tablet machine.

EVALUATION
Pre Compression Parameter
1. Angle of Repose
Angle of repose was determined by using funnel method. The blend was poured through funnel that can be raised vertically until a maximum cone height (h) was obtained. Radius of the heap was measured and angle of repose was calculated using the formula,

$$\theta = \tan^{-1} h/r$$

Where, θ is the angle of repose, h is the height of pile
r is the radius of the base of pile

Table 1- Formulation Table

Sr. no.	Ingredient	Formulation Code (mg)								
		F_1	F_2	F_3	F_4	F_5	F_6	F_7	F_8	F_9
1	Diclofenac Sodium	50	50	50	50	50	50	50	50	50
2	Lactose	131	130	133	132	131	134	133	134	135
3	Starch	6	6	6	5	5	5	4	4	4
4	Sodium Starch glycolate	6	5	4	6	5	4	6	5	4
5	Talc	4	4	4	4	4	4	4	4	4
6	Magnesium Stearate	3	3	3	3	3	3	3	3	3
7	Total weight	200	200	200	200	200	200	200	200	200

Table 2: Limits of angle of repose

Angle of repose (θ)	Flow
<25	Excellent
25-30	Good
30-40	Passable
>40	Very poor

2. Bulk density

Bulk density of a powder is defined as the ratio of the mass of the powder and its bulk volume. It is used to describe packing of particles. For bulk determination, a weighed quantity of the powder material was introduced into a graduated measuring cylinder and volume of powder was determined.

Bulk Density= Mass of the powder/ bulk volume

3. Tapped density

For determination of the tapped density, a weighed quantity of the powder was introduced into a graduated measuring cylinder and was tapped mechanically either manually or using a taping device till a constant volume was obtained

Tapped Density= Mass of the powder/ tapped volume

4. Carr's compressibility index

The simplest way of measurement of free flow of powder is compressibility, an indication of the ease with which a material can be induced to flow. The compressibility index is determined by Carr's index, which is calculated by using the following formula,

$C= 100(1-B/T)$

Where, B is bulk density, T is tapped density

5. Hausner's Ratio Hausner's ratio is an index of ease of powder flow. It is calculated by the following formula,

Hausner's Ratio= Tapped density/ Bulk density

Lower Hausner's ratio (< 1.25) indicates better flow properties than higher ones (>1.25)

Table 3: Flow Characteristics

Carr's Index	Flow Character	Hausner's Ratio
<10	Excellent	1.00-1.11
11-15	Good	1.12-1.18
16-20	Fair	1.19-1.25
21-25	Passable	1.26-1.34
26-31	Poor	1.35-1.45
32-37	Very poor	1.46-1.59
>38	Very very poor	>1.60

Post Compression Parameter
1. Hardness
The hardness of the tablet indicates its tensile strength and is measured in terms of load/pressure required to crush it when placed on its edge. Hardness has influence on disintegration and dissolution times and may affect bioavailability. Monsanto hardness tester was used to measure hardness of the formulated tablet. The tester consists of a barrel containing a compressed spring held between two plungers. The lower plunger was then forced against a spring by turning a threaded bolt until the tablet fractures. As the spring was compressed, a pointer rides along a gauge in the barrel to indicate the force. The force of fracture ease record and the zero force reading was deducted from it. It is expressed in kg/cm².

2. Friability

This test evaluates ability of tablet to withstand abrasion and edge damage during packing, handling and shipping. Friability generally reflects poor cohesion of tablet ingredients. Friability was measured by the help of Roche friabilator. 10 tablets were weighed and placed in plastic chamber that revolves at 25 rpm for 4 minutes. Tablets were re-weighed after removal of fines. The friability was calculated by the formula

F= w(initial)- w(final)/w(initial)

3. Weight variation

Tablets are designed to contain a specific amount of drug in a specific amount of tablet formulation. The weight of the tablet is measured to help ensure that a tablet contain the proper amount of drug. 20 tablets were selected randomly from each formulation were individually weighed using an electronic balance. Average weight of the tablets was calculated. The individual weight of the tablet was compared with average weight. The tablets meet the USP specification if not more than 2 tablets are outside the percentage limit and if no tablet differs by more than 2 times the percentage limit.

Weight variation = w_{avg} -$w_{initial}$/ w_{avg} × 100

Where, W_{avg}= Average weight of tablet, $W_{initial}$= Individual weight of tablet

Table 4: Weight Variation

Average weight of tablets(IP)	Average weight of tablets(USP)	Maximum % Difference allowed
Less than 80mg	Less than 130mg	10
80mg-250mg	130mg-324mg	7.5
More than 250mg	More than 324mg	5

4. Disintegration time

The process of breakdown of a tablet into smaller particles is known as disintegration. One tablet was placed in each of 6 tubes of the basket. A disc was added to each tube and the apparatus was run using 6.8pH phosphate buffer maintained at 37^0C as the immersion liquid. The assembly was raised and lowered between 30 cycles per minute in the 6.8pH phosphate buffer. The time in second taken for complete disintegration of the tablet with no mass remaining in the apparatus was measured and recorded. The tablet must be disintegrated **within 3 minutes.**

5. In vitro dissolution studies

In-vitro dissolution studies of the tablets were carried out in USP dissolution apparatus type IIby employing a paddle stirrer at 50 rpm using 900 mL of pH 6.8 phosphate buffer sat 37± 0.5^0C as a dissolution medium. One tablet was used in each test. Aliquots of 5 mL each were withdrawn at specified time intervals (0, 2, 6, 8, 10, and 12) and replaced with equal volume of fresh medium. The withdrawn aliquots were analyzed for drug content spectrophotometrically at $ʎ_{max}$ 275nm. Drug concentration was calculated and expressed as cumulative percent of the drug released.

RESULTS AND DISCUSSION

Diclofenac Sodium Oro dispersible tablet were prepared by direct compression method. Sodium Starch Glycolate and Starch were used as super disintegrant which help in rapid and drug dissolution.

Weight Variation:

All tablets from each formulation passed weight variation test, as the % weight variation was within the pharmacopeia limits.

Friability:

The friability of the formulations was found to be between 0.33-0.75% and was within the official requirement (i.e. less than 1%).

Hardness:

The hardness was maintained to be within 2.8-3.2 kg/cm², no variation in the hardness was found which clearly indicates that the blending was uniform.

Disintegration time

In vitro disintegration time for all the formulations varied from 22.6 to 39.3 seconds. The formulation F_9 shows better disintegration time of 22.6seconds.

Table no 5- Evaluation of pre compression parameter

Formulations	Angle of repose (Mean ±SD)	Bulk density (g/cc)	Tapped density (g/cc)	Carr's compressibility index	Hausner's ratio
F$_1$	30.24±0.52	0.52±0.02	0.63±0.04	16.42	1.21
F$_2$	25.42±1.20	0.38±0.04	0.46±0.34	15.42	1.16
F$_3$	22.74±1.02	0.46±0.12	0.50±0.06	14.78	1.13
F$_4$	26.32±1.22	0.43±0.03	0.62±0.12	11.67	1.15
F$_5$	26.22±0.65	0.45±0.12	0.52±0.01	12.01	1.15
F$_6$	27.42±0.66	0.41±0.02	0.62±0.16	14.87	1.20
F$_7$	28.60±0.74	0.43±0.25	0.56±0.08	14.64	1.22
F$_8$	26.62±0.42	0.46±0.14	0.62±0.05	15.44	1.17
F$_9$	**25.72±0.82**	**o.42±0.03**	**0.62±0.06**	**13.45**	**1.28**

Table 6: Evaluation of Post compression parameter

Formulation Code	Hardness (kg/cm²)	Friability (%)	Weight variation (mg)
F$_1$	2.8±0.22	0.642±0.30	198.6±0.04
F$_2$	2.6±0.12	0.546±0.38	196.1±0.12
F$_3$	3.1±0.14	0.32±0.24	197.5±0.07
F$_4$	2.1±0.03	0.542±0.12	196.3±0.18
F$_5$	2.8±0.16	0.539±0.20	198.1±0.17
F$_6$	2.7±0.02	0.356±0.20	199.4±0.04
F$_7$	2.8±0.02	0.484±0.31	199.5±0.12
F$_8$	2.9±0.04	0.743±0.30	198.5±0.04
F$_9$	**2.8±0.05**	**0.688±0.40**	**198.7±0.07**

Table no 7- Disintegration Time

Formulation code	In vitro Disintegration time (sec)
F$_1$	38.2
F$_2$	36.8
F$_3$	32.4
F$_4$	28.6
F$_5$	29.5
F$_6$	32.6
F$_7$	28.1
F$_8$	29.4
F$_9$	**22.6**

In vitro Dissolution Study

The result of In vitro dissolution study indicates that process used to prepare the orodispersible tablet to enhance the rate and extent of dissolution of Diclofenac. From the In vitro dissolution data it was found that as the concentration of superdisintegrants increased, the drug release also increased. Among the different batches of formulation F$_9$ shows highest dissolution rate were around 84.6

Table 8:

Sr. no	Time in min	Formulation code (% drug release)								
		F$_1$	F$_2$	F$_3$	F$_4$	F$_5$	F$_6$	F$_7$	F$_8$	F$_9$
1	0	0	0	0	0	0	0	0	0	0
2	2	8.77	14.48	15.70	21.79	7.24	12.74	19.39	13.74	**23.84**
3	4	10.24	20.55	22.06	27.64	11.20	17.00	24.50	17.72	**25.90**
4	6	14.53	27.52	26.42	32.36	13.56	23.20	28.34	23.82	**29.84**
5	8	16.11	28.95	32.69	35.54	19.23	27.24	34.50	25.97	**33.40**
6	10	20.54	33.05	35.52	38.50	23.06	32.84	38.93	32.72	**38.62**
7	12	22.83	35.85	37.81	41.60	26.88	41.65	39.56	37.24	**42.07**
8	14	25.65	37.86	42.84	49.74	29.85	43.12	44.17	47.76	**54.87**
9	16	28.63	40.65	45.67	58.67	32.77	48.26	57.62	47.44	**63.08**
10	18	32.0.5	42.82	50.22	67.22	44.65	61.15	86.21	64.52	**84.65**
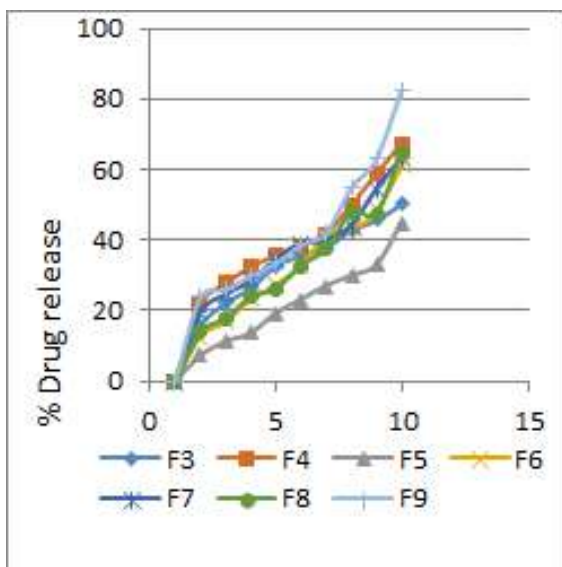
Fig: % drug release

CONCLUSION

Diclofenac Sodium is widely used Non-Steroidal Anti-inflammatory drug for rheumatoid arthritis, inflammation and pain relief. Fast dissolving tablets of Diclofenac Sodium are a useful approach for pain management and a feasible alternative to the available conventional immediate release dosage form. From the results, optimized F_9 formulation showed improved drug release characteristics.

REFERENCES

1. Nitin KK, Vilas PB, Arunadevi S, *Journal of Innovations in Pharmaceuticals and Biological Sciences,* 2015;2, 4: 541-555.

2. Pavan KR, Prakash BM, Shailendra SK, *International Journal of Advanced Research in Pharmaceutical & Bio Sciences,* 2013;3, 2:74-79.

3. Swamy NGN, Sachin, Abbas Z, *Journal of Applied Pharmaceutical Science*, 2012;2, 11:40-49.

4. Ravi S, Teja KM, Sai KV, *Scholars Academic Journal of Pharmacy,* 2013;2, 4:340-353.

5. Patil C and Das S, *African Journal of Pharmacy and Pharmacology,* 2011;5, 1:76-82.

6. Karthikeyan M, Umarul MAK, Megha M, Shadeer HP, *Asian Pacific Journal of Tropical Biomedicine*, 2012; S308:311.

7. Ranjit PS, Nagamani R, Satyajit P, *Journal of Applied Pharmaceutical Science,* 2015;5, 7:94-102.

8. Priyanka K, Vikesh S, *International Journal of Pharma Research & Review*, 2014;3, 7:12-19.

9. Devendra RR, Hemant NG, Vikas VP, Vinod MT, Vijay RP, *International Current Pharmaceutical Journal,* 2012;1, 10:311-316.

10. Nwoko VE, *Innovations in Pharmaceuticals and Pharmacotherapy,* 2014, 2, 2:350-368.

11. Jagadeesh I, Padmaja B, *International Journal of Pharmacy and Pharmaceutical Sciences*, 2012;4, 1:241-248.

12. Raghavendra RNG, Mahesh K, Mettu SR, Kistayya C, Mahipal RB, *International Journal of Pharmaceutical Sciences and Drug Research,* 2012;4, 4:230-235.

13. Santhosh RI, Sivakumar R, Siva P, Sajeeth CI, *International Journal of Pharmaceutical, Chemical and Biological Sciences,* 2013;3, 2:388-397.

14. Jain CP, Naruka PS, *International Journal of Pharmacy and pharmaceutical sciences*, 2009;1, 1:219-226.

15. Amit M, Vandana S, Arun G, Ashish A, *International Journal of Pharmaceutical & Biological Archives*, 2012;3, 4:1003-1007.

16. Md. Nehal Siddiqui, Garima G, Sharma PK, *International Journal of Pharmaceutical Sciences Review and Research*, 2010; 4, 2:87-96.

17. Sudhir B, *Asian Journal of Biochemical and Pharmaceutical Research*, 2011;3, 1:606-616.

18. Priyanka K, Vikesh S, *International Journal of Pharma Research & Review,* 2014;3, 7:12-19.

RESEARCH ARTICLE   **OPEN ACCESS**

Synthesis and characterization of ZnO dopped Fe₃O₄ nanocomposite material and its heterogeneous photocatalytic activity for degradation of phenol

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ABSTRACT

A series of ZnO doped Fe3O4 nano-composite materials were synthesized by using co-precipitation method. The surfactant such as CTAB was used during the synthesis of this nano-composite material. This material then characterized with the help of some sophisticated modern techniques such as XRD, FT-IR, and SEM-EDS. According to these all characterizations results suggested the formation of nano-composite highly crystalline materials. The catalytic activities of synthesized materials were successfully tested on degradation of phenol by using hydrogen peroxide at room temperature. The degradation reaction was monitored by using Ultra-Violet visible spectroscopy (UV-Vis) techniques. It was found that 15 wt% ZnO doped Fe3O4 nano-composite material shows better activity for the degradation of phenol. The degradation reaction is carried out at room temperature with continuous stirring in presence of sun light irradiation. Thus this degradation reaction is different from conventional heterogeneous catalysis or photocatalysis, in which the degradation reaction is carried out in natural light source. Thus this reported phenol degradation reaction is a quite promising green technology, which could be widely applied in practice.

Keywords: Doping, Heterogeneous catalysis, Photodegradation, Nanocomposite Material

INTRODUCTION

Inorganic chemistry is the branch of chemistry, which deals with the study of synthesis and behavior of inorganic and organometallic compound. This field covers all chemical compounds except the organic compounds (carbon based compounds, usually containing C-H bonds). Important inorganic compounds are the oxides, carbonates, nitrate, sulphates and halides. It is deals with the study of coordination compounds, main group compounds, transition metal compounds, organometallic compounds, cluster compounds, bioinorganic compounds and solid state compounds.

During the last decade, nanosize magnetic materials have been largely studied due to their different magnetic properties compared to their bulk counterparts such as super-paramagnetism which can significantly advance the current clinical diagnostic and therapeutic methods. Each superparamagnetic particle is a single magnetic domain in which thermal energy can overcome anisotropy energy barrier and cause free spin reversal. This is to say that no residual magnetism remains after elimination of external magnetic field.

In this regard, spinel ferrite nanocrystals with general chemical composition MFe_2O_4 (where M= Co, Ni, Mn, Fe, Cu, Zn, etc.) form a group of applicable magnetic materials due to their interesting magnetic, magneto-resistive and magneto-optical properties.

On the overlook at the literature survey, it has been observed that metal oxide and mixed metal oxide play an important role in catalytic processes to speed up chemical reactions, in an eco-friendly manner, cost effective, efficient and clean processes. The main objectives of the present work are:

1) To study the effect of various methods on the preparation of metal oxides and mixed metal oxides like co-precipitation, impregnation and sol-gel methods.
2) To study the photocatalytic degradation of industrial wastes like phenol, organic dyes with the help of prepared metal oxides.

3) To replace the toxic, conventional/classical acid catalysts by means of solid heterogeneous catalyst.
4) To develop a clean, efficient and environmentally benign processes for the synthesis of pharmacologically and biologically important heterocyclic compounds.

To take above mentioned aim and objectives here in this research, we successfully synthesized pure ZnO, pure Fe_3O_4 and series of ZnO doped Fe_3O_4 nanocomposite material. We studied the structural and morphological properties of synthesized material by different characterization techniques such as XRD, SEM-EDS and FT-IR. Finally investigates the photocatalytic activity of synthesized material by degradation of phenol.

METHODOLOGY

Chemicals and Reagents:

Ferrous Sulphate, Ferric Sulphate, Zinc Choride, 1:1 Ammonia, Polyethylene Glycol, Ethanol, etc. All chemicals are Analytical grade and used without further purifications.

1. Synthesis of ZnO:

Dissolve 1.6 gm Zinc Chloride in 50 ml deionised water. Add 10 ml alcohol, then add 5 ml polyethylene glycol. Stirrer it continuously till dissolved it completely, then heat the above solution at 80°C for 5-6 hr. Add drop by drop 1:1ammonia to maintain pH 8.5. This solution form white precipitate. Then this precipitate is filtered through Whattman filter paper no 41. Wash it with hot water. Dry this residue overnight at 120°C temperature. After drying obtained material is then kept for calcination in high temperature Muffel furnace at the temperature of 120°C for 3 hrs. White coloured final resulting powder is ZnO nanocomposite material.

2. Synthesis of Pure Fe_3O_4:

Dissolve 3.9 gm ferric sulphate and 2.5 gm ferrous nitrate in 50 ml deionised water in separate clean and dried beaker. Add 10 ml alcohol. Then add 5 ml polyethylene glycol. The solution is heated with continuous stirring at 80°C for 5-6 hr. Add drop by drop 1:1ammonia to maintain pH 8.5. This solution

form dark brown precipitate. These resulting two solutions are then mixed together with stirring which gives black coloured precipitate. Then this precipitate is filtered through Whattman filter paper no 41. Wash it with hot water. Dry this residue overnight at 120°C temperature. After drying obtained material is then kept for calcination in high temperature Muffle furnace at the temperature of 500°C for 3 hrs.

3. Synthesis of series of ZnO doped Fe_3O_4:

Take above prepared solutions (3.9 gm ferric sulphate and 2.5 gm ferrous nitrate) in beaker. Add Zinc Chloride solution (0, 1, 5, 10 and 15 wt%) in the same beaker. Then add 5 ml polyethylene glycol with continuous stirring and heat it at 80°C temperature for 4-5 hrs. Filter the total solution through Whattmann filter paper no. 41. Dry this residue in oven at 120°C for overnight. After drying the product was then crushed in mortar pestle to obtain very fine powder. After drying obtained material is then kept for calcination in high temperature Muffle furnace at the temperature of 500°C for 3 hrs. Finally weight is taken off all these products and fill it in sample bottle and labelled it as 1) pure ZnO, 2) pure Fe_3O_4, 3) 1 wt% ZnO doped Fe_3O_4, 4) 5 wt% ZnO doped Fe_3O_4, 5) 10 wt% ZnO doped Fe_3O_4, and 6) 15 wt% ZnO doped Fe_3O_4.

RESULTS AND DISCUSSION

The X-ray diffraction data was acquired in the 2θ range of 20–80° on a Rigaku Multiflex instrument using Cu Kα (λ = 1.5418 Å) radiation source and a scintillation counter detector (SCD). Crystalline phases present in the samples were identified with the help of JCPDF-ICDD.

The Fourier transformation infra-red spectra (FTIR) were recorded on FTIR spectrometer (SHIMADZU-FTIR/4100), Japan in the range 4000–500 cm^{-1}.
The powder morphology was observed by scanning electron microscope (SEM), analysis was performed on a JEOL JSM6700F electron microscope.

1. XRD Analysis:
The XRD pattern of the powder is studied with the diffraction angle 20-80°. All the peaks are in 100%

phase matching with the ZnO hexagonal phase of JCPDF No. 36-1451. It is shown in **Figure 1**. There are no other characteristic impurities peaks were present which also confirm that the product obtained is in pure phase. Similarly high intensity peaks suggested that the synthesized material is highly crystalline material. The peak at 2 °=36.64 is corresponds to the hkl value 101 suggests that ZnO material with hexagonal crystal structure. The line broadening in the peaks determine the crystallite size of ZnO to be less than 25 nm. The values present in the Table 2 are corresponds to the hkl and FWHM of ZnO material. The average crystalline size of the calcined ZnO powder is estimated by the Scherrer's relation (1).

$$D = 0.9\lambda/\beta COS\theta \quad\quad (1)$$

Where D is the average crystalline size λ is the X-ray wavelength of 1.54 Å, θ is the Bragg diffraction angle and β is the FWHM.

The XRD patterns of the synthesized nanoparticles after calcination for various wt.% of ZnO doped Fe_3O_4 are shown in **Fig. 2**. The peak positions agree with those of the spinel structure. Thus the synthesized nanoparticles are Fe_3O_4 phases. The synthesized nanoparticles seem to be Fe_3O_4 phase. In these diffraction patterns, no trace of the reactants and by-products or ZnO material, so on, can be seen, it indicates total incorporation of ZnO material in to the Fe_3O_4. As revealed, diffraction peaks of all samples completely correspond to standard pattern characteristic peaks of the magnetite cubic inverse spinel structure (JCPDS 19-0629). No secondary phase is detected to prove the formation of other Zn-based structures such as ZnO. So, we might conclude that Fe_3O_4 has been formed with high phase purity and Fe ions have been successfully replaced with Zn^{2+} ions.
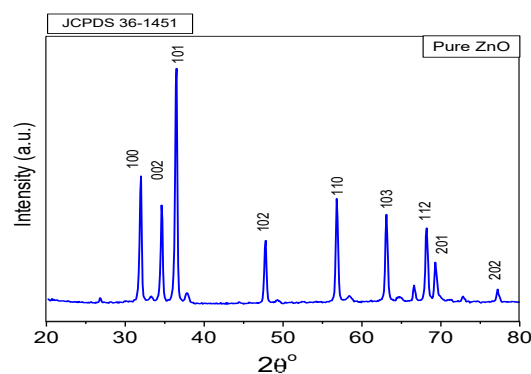
Figure 1: XRD spectrum of Pure ZnO
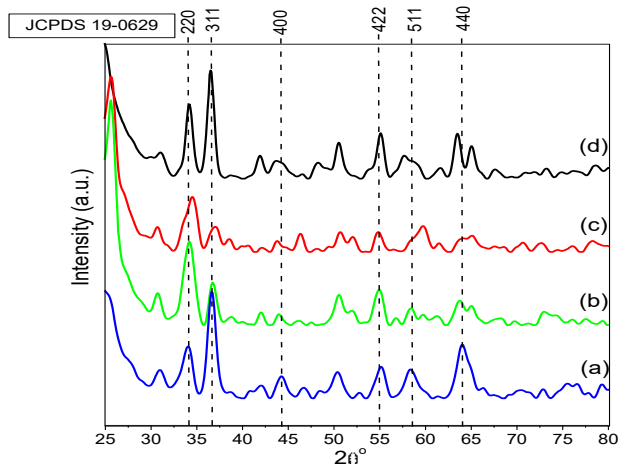
Figure 2: XRD spectrums of (a) Pure Fe_3O_4, (b) 5% ZnO-Fe_3O_4, (c) 10% ZnO-Fe_3O_4, (d) 15% ZnO-Fe_3O_4

2. SEM-EDS Morphology and structure studies:

In order to understand the surface morphology and to know the surface dispersion of the active species on the support, the efficient studies were carried out on the SEM analysis. **Figure 3 (a, b, c and d)** shows SEM morphology photographs for pure ZnO materials with different magnifications which shows highly porous in nature and nanocrystalline structure. In the image of **Figure 3 (c)** clearly shows that ZnO material with 0.5 in size.
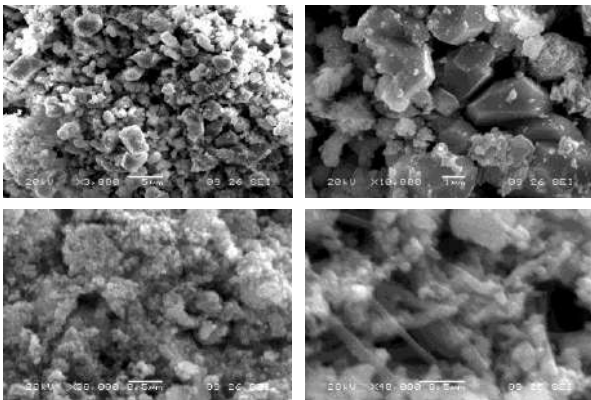
Figure 3 (a, b, c and d): SEM images of Pure ZnO

From the **figure 4 (a, b and c)** it shows good agglomeration of Fe_3O_4 magnetic nanoparticles. It is evident that the sample shows an irregular appearance From the SEM micrograph of the 15 wt.% ZnO doped Fe_3O_4 material is shown in **Figure 5 (a, b and c),** it can be clearly seen that effect of addition of n into Fe_3O_4 material clearly shows alteration in

crystallite size, improvement in morphology and increasing the porosity.
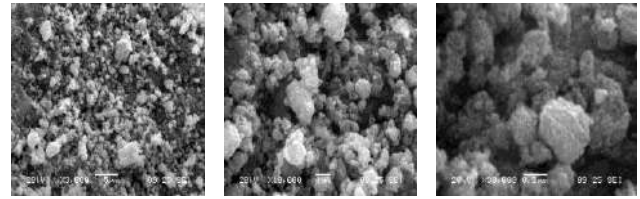
Figure 4 (a,b,c) : *SEM images of Pure Fe_3O_4*
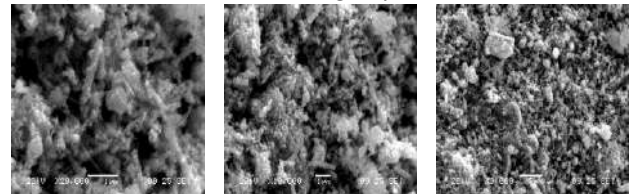
Figure 5: *SEM images of 15wt.% ZnO-Fe_3O_4*
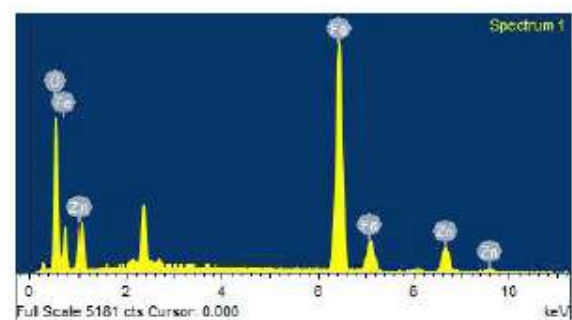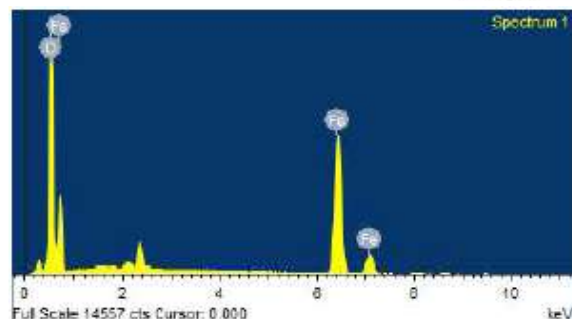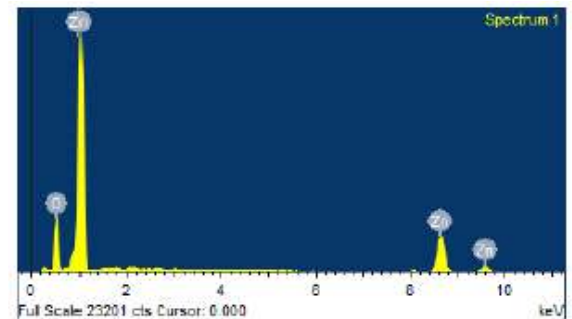
Figure 6: SEM-EDS images of **(a)** Pure ZnO, **(b):** Pure Fe_3O_4, **(c):** 15% ZnO-Fe_3O_4

Table 1: Elemental Data Analysis

Element	Pure Fe₃O₄		Pure ZnO		15 % ZnO-Fe₃O₄	
	Weight %	Atomic %	Weight %	Atomic %	Weight %	Atomic %
O	57.77	82.8	44.08	76.31	36.39	67.32
Zn	0.00	0.00	55.92	23.69	13.37	6.05
Fe	42.23	17.32	0.00	0.00	50.25	26.63
Total	100.00	100.00	100.00	100.00	100.00	100.00
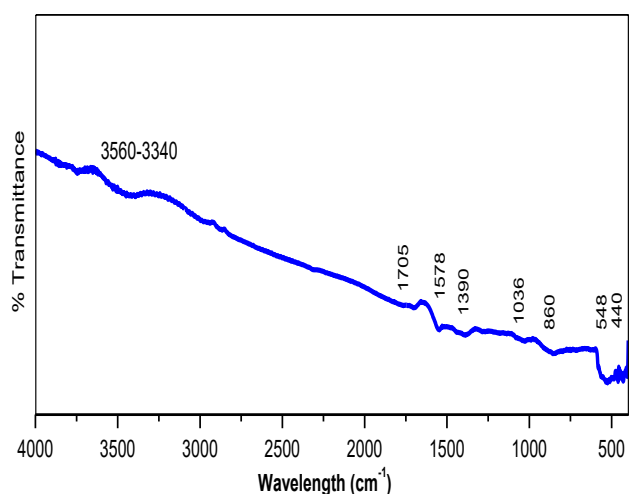
Figure 7: FT-IR spectrum of Pure ZnO
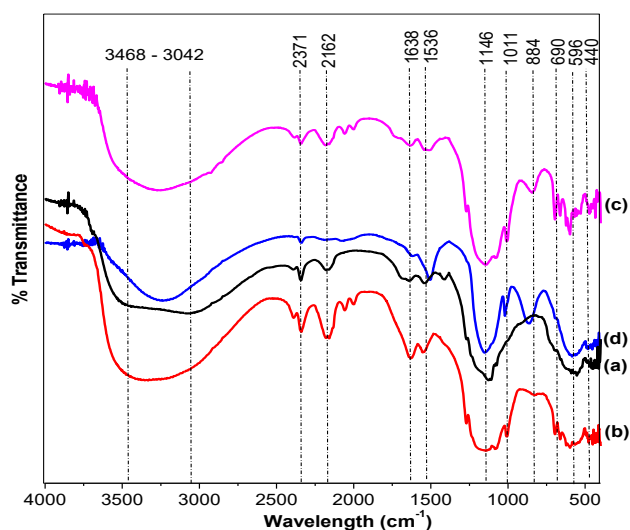
Figure 8: FT-IR spectrums of (a) Pure Fe₃O₄, (b) 5% ZnO-Fe₃O₄, (c) 10% ZnO-Fe₃O₄, (d) 15% ZnO-Fe₃O₄

Elemental compositions of pure ZnO, pure Fe₃O₄ and 15 wt. ZnO doped Fe₃O₄ materials are represented in the EDS spectrums shown in **Figure 6** (a, b and c). The elemental quantitative analysis is also shown in **Table 1**. The observed Zn:O; Fe:O and Zn:Fe:O atomic ratios are fairly close to the expected bulk ratios indicating

good distribution of the metal species inside the sample matrix. This observation supports analysis it was shown that, the minimum stoichiometric ratio for the all sample are maintained.

3. Fourier Transformation Infra-Red Spectroscopy Study:

The **Figure** 7 shows the FTIR spectrum of pure ZnO material. over the 4000–500 cm⁻¹ range. Two peaks at 1578 and 1390 cm⁻¹ are due to stretching and bending mode of oxygen available in the Zn-O-Zn and the Zn=O indicate that the presence of a ZnO. Two strong vibration bands were detected at 1036, 860 and 548 cm⁻¹, associated to the stretching and bending mode of Zn-O-Zn and the Zn=O indicating the existence of a layered hexagonal ZnO phase. The peak at 1705 cm⁻¹, associated with the vibration mode of the Zn–OH bond. Due to coordinated crystalline water present in material, it gives observed due to OH-stretching at 3300 cm⁻¹. The broad band around 3560-3340 cm⁻¹ is due to O–H stretching vibration modes of the adsorbed water on the surface of the powder.

The **Figure** 8 (a-d) shows the FTIR spectrum of series of catalytic material prepared (a) Pure Fe₃O₄, (b) 5% ZnO-Fe₃O₄, (c) 10% ZnO-Fe₃O₄, (d) 15% ZnO-Fe₃O₄ over the range of 4000–500 cm⁻¹. The peaks at 1146,

1011, 884 cm⁻¹ are due to stretching and bending mode of oxygen available in the Fe-O-Fe and the Fe=O indicate that the presence of a FeO and Fe₂O₃. Three strong vibrations were detected at 690, 596 and 440 cm⁻¹, associated to the stretching and bending mode of Fe-Fe and the Fe-O indicating the existence of a layered Fe₃O₄ phase. The peak at 1638 and 1536 cm⁻¹, associated with the vibration mode of the Fe–OH bond. The broad band between the ranges of 3468-3042 cm⁻¹ is due to O–H stretching vibration modes of the adsorbed water on the surface of the powder.

Table 2: Physico-chemical parameters of phenol before and after degradation

	ZnO	Fe$_3$O$_4$	5 wt.%	10 wt.%	15 wt.%	Blank
pH	1.40	1.37	1.46	1.44	1.34	1.32
COD	42.24	364.8	595.2	566.4	316.8	345.6
O.C.	0.15	0.28	0.22	0.22	0.22	17.5
E.C.	12.60	15.05	17.43	16.28	13.32	16.70
Flame (Na)	42	54	34	53	46	44
Flame (K)	25	28	17	31	27	25
Phenol Degradation in (%)	8.21	9.47	57.98	60.95	91.59	-

4. Catalytic activity measurements:

Batch degradation control experiments of phenol were carried out at pH = 7.0 in a 25 mL of conical flask placed on a magnetic stirrer at room temperature. The reaction suspension was prepared by adding 0.1 g of catalyst amount into 5 mL of pure phenol (45 mM) and 10 ml of 30% H$_2$O$_2$ (100 mM) solution to initiate the reaction. After 1h the reaction mixtures were centrifuged and analyzed in JASCO, V650 UV-Visible spectrophotometer in the range of 200-800 nm. The change in intensity of peak at λ_{max} for phenol at 270 nm was carried out. Each experiment was run in triplicate, and average values and standard deviations are presented.

The catalytic efficiency of the synthesized catalysts was investigated for phenol degradation reaction and the obtained results are shown in **Figure 9**. In each experiment, the reaction suspension was prepared by adding 0.1 g of catalyst amount into 5 mL of pure phenol (45 mM) and 10 ml of 30% H$_2$O$_2$ (100 mM) solution to initiate the reaction. As can be seen from the **Figure 9,** that was remarkable decrease in the concentration of phenol was clearly observed using UV-Visible spectra. Among all the catalytic materials, 15 wt.% catalyst was observed higher phenol degradation activity when compared to that of remaining catalysts. It is mainly attributed to increase an availability of more active sites including more surface area, smaller particle size and ample vacancy defects by addition of the ZnO. The colour intensity of the phenol solution was increases to around 80% for the modified with ZnO doped Fe$_3$O$_4$ catalytic material when compared with the 0, 5 and 10 wt.%,. The chance in colour of phenol in the solution from colourless to dark brown is shown which is attributed to the formation of intermediates like catechol, resorcinol and glycerol etc. during the degradation of phenol in presence of catalyst and H$_2$O$_2$ after 1h.

Figure 9: Photographs of phenol degradation process a)5% b) 10% C) 15% d) pure FeO e) pure ZnO f) blank g) Pure phenol after 120 minutes

The efficiency of phenol degradation was performed overall synthesized catalysts under the reaction conditions same as in **Figure 9** and obtained phenol conversions are presented in the **Table 4.** Although the catalytic activity of phenol degradation is very low in the absence of catalyst, the catalytic activity in the presence of catalysts satisfactorily improved. It was found that 15 wt.% ZnO doped Fe$_3$O$_4$ catalyst show a higher catalytic performance when compared to that of other series of catalysts. It might be attributed to increase an availability of more active sites by addition of the ZnO metal oxide into Fe$_3$O$_4$. We performed the some physico-chemical parameters of phenol before and after degradation reaction such as pH, COD measurement, O.C., Electrical conductance, detection of concentration of Na and K by using Flame photometrically. As well, the measured chemical oxygen demand (COD) for the degradation of phenol before and after the reaction over prepared catalysts

and obtained results are presented in the **Table 2**. The COD values represent the amount of organic compounds in water and a remarkable decrease in the COD value was noticed after the degradation of phenol. The gradual decrease in COD values was observed for all catalysts after 150 minutes of the reaction time. The observed phenol degradation of all catalysts are as ~ 8.21, 9.47, 57.98, 60.95 and 91.59 % for the catalysts such as pure ZnO, pure Fe_3O_4, 5 wt.% ZnO doped Fe_3O_4, 10 wt.% ZnO doped Fe_3O_4 and 15 wt.% ZnO doped Fe_3O_4, respectively. This decrease in COD values shows the degradation of phenol that leads to the conversion of organic compounds into harmless gaseous CO_2 and inorganic ions. This noticeable observation is indeed due to the oxidation and degradation of phenol by radical species into aromatic compounds, low-molecular carboxylic acids, carbon dioxide and water.

CONCLUSIONS

For improving structural and morphological properties of pure Fe_3O_4, ZnO doping effect was studied. XRD pattern of nanoparticles indicated formation of Fe_3O_4 with cubic inverse spinel structure in all samples. Increasing dopant caused to decreasing crystalline size and formation of uniform and spherical shape nanoparticles. The catalytic activities of synthesized materials were successfully tested on degradation of phenol by using hydrogen peroxide at room temperature. The degradation reaction was monitored by using Ultra-Violet visible spectroscopy (UV-Vis) techniques. It was found that 15 wt% ZnO doped Fe_3O_4 nano-composite material shows better activity for the degradation of phenol. The degradation reaction is carried out in natural light source. Thus this reported phenol degradation reaction is a quite promising green technology, which could be widely applied in practice.

REFERENCE

1. Rezai Marand Z, Shahtahmasbia N, Rezaee M, Roknabadia M, Hosseindokht M, Bagheri Mohagheghid M. Helmi Rashid Farimania, R. Etefagh, Proceedings of the 4th International Conference on Nanostructures (ICNS4) 12-14 March, 2012;211-213, Kish Island, I.R. Iran.

2. Zahra Rezay Marand, Mitra Helmi Rashid Farimani, Nasser Shahtahmasebi, *Nanomedicine journal*, 4, 214, 238-247.

3. Yendrapati Taraka Prabhu, Kalagadda Venkateswara Rao, Vemula Sesha Sai Kumar, Bandla Siva Kumari, *Advances in Nanoparticles*, 2013; 2: 45-50.

4. Kotsikau D, Natarov V, Petrova E, Pankov V, *Physics, Chemistry and Application Of Nanostructures*, 2015.

5. Lee JH. *Nature Medicines*, 2007; 13:95.

6. Park J. *Nature Materials*, 2004; 3: 891.

RESEARCH ARTICLE   **OPEN ACCESS**

Synthesis and Characterization of Chalcone and their Fe(III) metal complexes

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ABSTRACT

In present study three chalcones 2-hydroxy-4-methylphenyl-3-phenylprop-2-en-1-one [HMPO], 2-hydroxy-4-methylphenyl-3-(p-tolyl) prop-2-en-1-one [HMTO] and 4-chlorophenyl-2-hydroxy-4-methylphenyl-prop-2-en-1-one [CHMPO] were synthesized by condensing 2-hydroxy-4-methylacetophenone with benzaldehyde, 4-methylbenzaldehyde and 4-chlorobenzaldehyde. The synthesized chalcones were characterized by IR, NMR and Mass spectral studies. With this chalcones Fe(III) metal complexes were prepared and also characterized by different spectroscopic techniques. IR spectra indicates that hydroxyl oxygen and nuetral carbonyl involved in the coordination with Fe(III) ion. All the complexes posses' octahedral geometry.

Keywords: Chalcone, metal complexes, IR, mass, TGA.

INTRODUCTION

Chalcone is an aromatic ketone and an enone that forms the central core for a variety of important biological compounds, which are known collectively as chalcones or chalconoids. The chemistry of chalcones has generated great scientific interest due to their biological and industrial applications. Chalcones are natural biocides and are well known intermediates in the synthesis of heterocyclic compounds exhibiting various biological activities.

Chalcones and their derivatives possess some interesting biological properties such as antibacterial, antifungal, insecticidal, anesthetic, anti-inflammatory, analgesic etc [1-4]. A number of chalcones having hydroxy group at different position having ability to form coordinate bond with different metal ion in order to form transition metal complexes.

METHODOLOGY

All chemicals used were of the analytical reagent (AR) grade and of highest purity available and purchased from SD-Fine Chem Limited. Melting points were determined with an Electro thermal 9100 apparatus and are uncorrected. IR spectra were recorded on a Shimadzu 4300 spectrometer. NMR spectra were recorded with a Brucker 80 instrument using TMS as internal standard. Mass analyses of the products were conducted with a Finnigan-Matt 8430 GC-Mass instrument.

Synthesis of chalcone

A mixture of 0.01 mol 2-hydroxy-4-methylacetophenone and 0.01 mol various aldehyde added into ethanol solvent. To this reaction mixture 20 % NaOH added and heated for several minutes' upto formation of solid residue. By keeping overnight residue nuetralized by ice cold HCl solution, filtered and dried in oven [5-7].

Synthesis of metal complexes

An equimolar mixture of respective chalcone and $FeCl_3.H_2O$ (0.01 mole) were added in RB flask containing ethanol and refluxed for 8-10 hrs to obtained solid residue. Residue filtered, dried and recrystallized with ethanol.
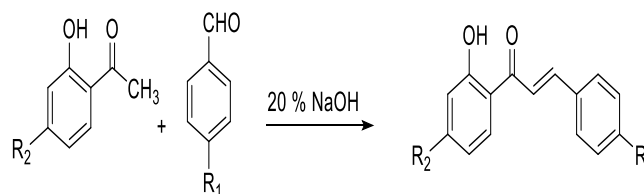
Fig 1: Synthesis of Chalcone

Synthesis of metal complexes

Newly synthesized chalcone (0.01 mol) and $FeCl_3.6H_2O$ (0.01 mol) were taken in R.B. flask already containing ethanol solvent and refluxed for 4-6 hrs to obtained solid residue. Residue filtered, dried and recrystallized by using ethanol.

RESULTS AND DISCUSSION

IR Spectra

IR spectral technique is helpful in order to determine binding mode of ligand towards metal ion. In chalcone hydroxyl stretching band is appeared at 3305-3315 cm^{-1} which is completely disappered in metal complexes confirm the participation of hydroxyl oxygen in coordination with metal ion. This confirms by the upward shift of C-O stretching frequency by 19-32 cm^{-1} is spectra of complexes. A band due to carbonyl group at 1687-1695 cm^{-1} again shifted to lower frequency at extent 20-34 cm^{-1} suggest coordination of carbonyl oxygen with metal ion. At the same time new spectral bands appeared in spectra of complexes in region 530-555 cm^{-1} due to M-O stretching vibration [8-10]

Sr No	Chalcone	R$_1$	R$_2$
1	2-hydroxy-4-methylphenyl-3-phenylprop-2-en-1-one [HMPO]	H	CH$_3$
2	2-hydroxy-4-methylphenyl-3-(p-tolyl)prop-2-en-1-one [HMTO]	CH$_3$	CH$_3$
3	4-chlorophenyl-2-hydroxy-4-methylphenyl-prop-2-en-1-one [CHMPO]	Cl	CH$_3$

Spectroscopic data of 2-hydroxy-4-methylphenyl-3-phenylprop-2-en-1-one
[HMPO]: IR (KBr) v$_{max}$: cm^{-1} : 3305(-OH), 3029(-Ar-CH), 1691(-C=O), 1590(-C=C)
^{1}H-NMR (DMSO-d$_6$) δ: 9.8(1H,s,OH), 6.98-7.60(7H,m,Ar-H), 2.5(3H,s,CH$_3$)
Spectroscopic data of 2-hydroxy-4-methylphenyl-3-(p-tolyl)prop-2-en-1-one
[HMTO]: IR (KBr) v$_{max}$: cm^{-1} : 3312(-OH), 3015(-Ar-CH), 1687(-C=O), 1592(-C=C)
^{1}H-NMR (DMSO-d$_6$) δ: 9.5(1H,s,OH), 6.90-7.74(7H,m,Ar-H), 2.4(6H,s,CH$_3$)

Spectroscopic data of 4-chlorophenyl-2-hydroxy-4-methylphenyl-prop-2-en-1-one

[CHMPO]: IR (KBr) ν_{max}: cm^{-1} : 3315(-OH), 3035(-Ar-CH), 1695(-C=O), 1594(-C=C)

^{1}H-NMR (DMSO-d_6) δ: 9.2(1H,s,OH), 6.98-7.55(7H,m,Ar-H), 2.3(3H,s,CH$_3$)
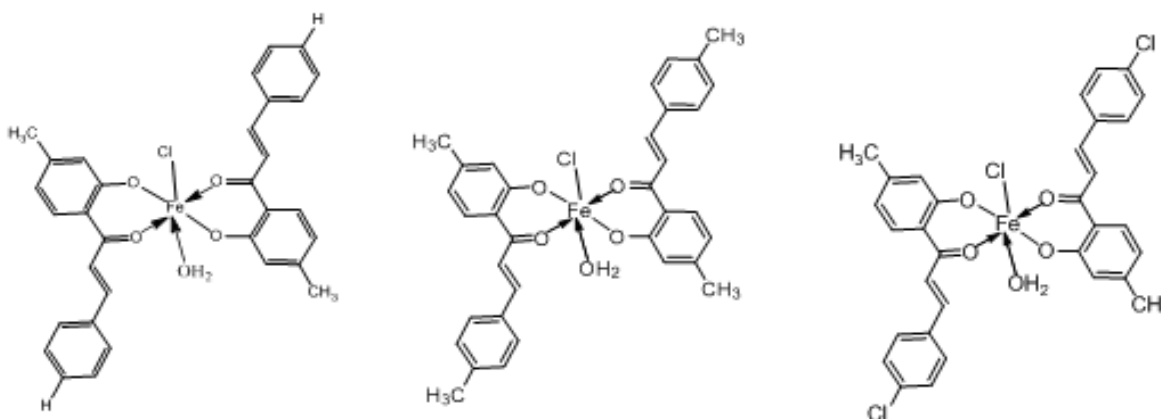
Fig 2: Probable structure for metal complexes

Mass Spectra

Mass spectrometry has been successfully used to determine the molecular ion peak for synthesized compounds. The various fragmentation peaks obtained for chalcone and all Fe(III) complexes are in good agreement with proposed structure. The mass spectral data shows that all Fe(III) complexes are dimeric. Molecular io peak and various fragment ion obtaibed are given below.

Fe(HMPO): m/z- 582, 564, 530, 292, 78.

Fe(HMTO): m/z- 611,592, 558, 502, 306.

Fe(CHMPO): m/z- 651, 633, 597, 326.

Table 1: Analytical data of chalcone and its Complexes

Sr. No.	Compounds	Molecular Formula	Molecular Weight	Colour	M. P. (^{0}C)
1.	HMPO	$C_{16}H_{14}O_2$	238.28	Yellow	120
2.	HMTO	$C_{17}H_{16}O_2$	252.31	Yellow	134
3.	CHMPO	$C_{16}H_{13}ClO_2$	272.73	Brown Yellow	125
4.	Fe(HMPO)	$C_{32}H_{28}ClFeO_5$	583.86	Brown	>300
5.	Fe(HMTO)	$C_{34}H_{32}ClFeO_5$	611.91	Green	>300
6.	Fe(CHMPO)	$C_{32}H_{26}Cl_3FeO_5$	652.75	Green	>300

Table 2: IR spectral bands of Fe(III) complexes of chalcones

Compounds	v(OH)	v(C=O)	v(C-O)	v(M-O)
Fe(HMPO)	--	1671	1309	530
Fe(HMTO)	--	1657	1317	555
Fe(CHMPO)	--	1661	1421	541
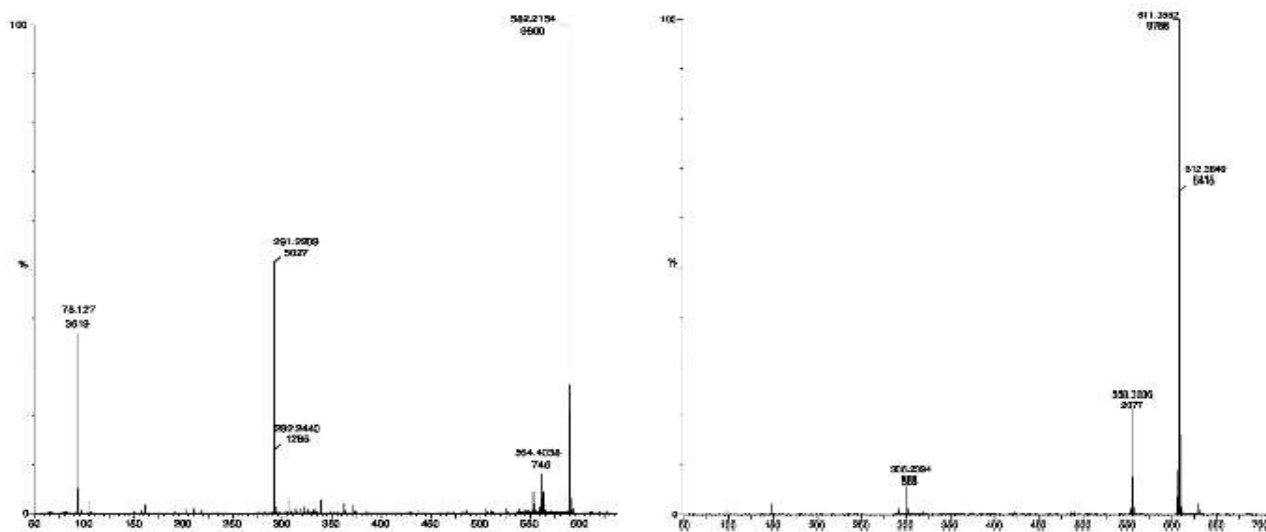
Fig 3: Mass Spectrum of Fe(HMPO) and Fe(HMTO) complex

Thermal Analysis:

Thermogravimetric analysis is the best techniques to find out the thermal stability of complexes. Thermogravimetric analysis was performed in nitrogen atmosphere with heating rate 10^0C per minute and thermograms are recorded in temperature range 40^0C to 800^0C. By using this technique several kinetic parameters like activation energy (Ea), order of reaction (n), entropy change (S) were calculate. The thermal data have been analyzed by using Freemann-Caroll and Sharp-Wentworth methods. All the complexes are stable upto 60^0C and further decomposed in several steps. There occurs no loss upto 120^0 C molecule in all metal complexes indicates the absence of any lattice water molecule. Above 220^0 C the complexes shows the loss of one coordinate water molecule. wt. loss obs/**calcd**: Fe(HMPO)-3.32/**3.34**
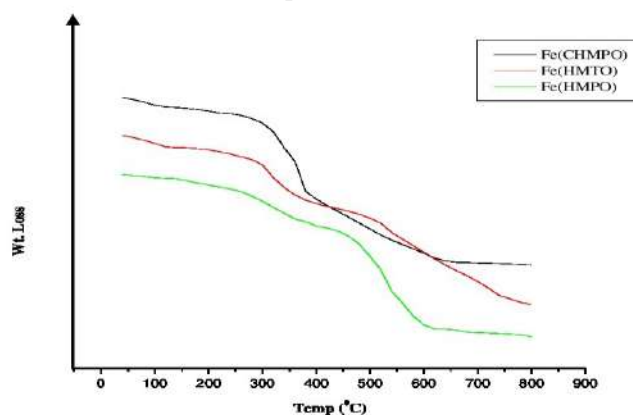
Fig 4: Thermal graph of Fe(III) complexes

Fe(HMTO)- 2.88/**2.94,** Fe(CHMPO)- 2.97/**2.88** Next steps includes the decomposition of coordinate part of ligands and finally all complexes are converted into their respective metal oxides [11].

Table 3: Kinetic Parameter for Fe(III) metal complexes.

Compounds	Half Decomposition Temperature (^{0}C)	Activation Energy Ea (kJ/mole)		Order of Reaction (n)	Entropy Change -ΔS (J/mol/K)	Free Energy Change ΔF (kJ/mol)
		FC	SW			
Fe(HMPO)	440	24.70	23.86	0.98	-145.91	70.65
Fe(HMTO)	490	17.13	17.69	0.99	-148.58	64.63
Fe(CHMPO)	510	21.33	20.89	0.98	-147.63	62.63

CONCLUSION

The present article includes the synthesis and characterization of three chalcones and its Fe(III) complexes. Characterization of compounds includes IR, Mass, ^{1}H NMR and thernogravimetric analysis. Chalcones coordinates to Fe (III) metal ion through hydroxyl oxygen and neutral carbnyl oxygen and act as a dibasic ligand (Metal ligand ratio 1:2) The thermal data shows that complexes were highly stable and its thermal decomposition as well as thermodynamic parameters was studied.

Conflicts of interest: The authors stated that no conflicts of interest.

REFERENCES

1. Bhat BA, Dhar KL, Saxena AK, Shanmugavel M. Synthesis and biological evaluation of Chalcones and their derived Pyrazoles as potential cytotoxic agents, *Bio org. & Med. Chem.*, 2005, 15 (3),:177-3180.

2. Kalirajan R, Palanivelu M, Rajamanickam V, Vinothapooshan G and Anandarajagopal K. Synthesis and biological evaluation of some Chalcone derivatives, *Int. J. of Chem. Sci.,* 2007, 5(1):73-80.

3. Gupta Urmila Sareen, Vineeta, Khatri Vineeta, Chugh Sanjana. Synthesis and antifungal activity of new Fluorine containing 4- (substituted Phenyl azo) Pyrazoles and Isoxazoles, *Indian J. of Het.Chem.*, 2005; 14: 265-266

4. Mishra Rakesh Mani and Wahab Abdul. Synthesis and Fungicidal activity of some new 2, 3-Dihydro-4H-Benzimidazolo [3, 2-b] - [1, 3] - Thiazine-4-ones, *Indian J. of Het.Chem.,* 2003, 13: 29-32.

5. Sreedhar NY, Jaypal MR, Prasad KS and Prasad PR. Synthesis and characterization of 4-hydroxychalcone using PEG-400 as a recyaclable catalyst., *R. J. Pharmaceutical, Biological and Chemical Sciences*, 2010, 1(4): 480.

6. Zangade S, Mokle S, Vibhute A and Vibhute Y. An efficient and operationaly simple synthesis of some new chalcone by using grinding techniques,*Chemical Science Journal,* 2011, 1-6.

7. Arora V, Arora P and Lamba HS. Synthesis and evaluation of chalcone derivatives of 2-acetyl naphthalene for antifungal and antibacterial activity, 2012, 4(2):554-557.

8. Habib SI, Shah NN, Baseer MA, Kulkarni PA. Synthesis and characterization of Cobalt(II), nickel(II), copper(II) complexes of 2-hydroxy chalcones, 2011, 3(1):788-792.

9. Vyas KB, Nimavat KS, Jani GR, Hathi MV. Synthesis and antimicobial activity of coumarin derivatives of metal complexes, 2001, 1(2): 183-192.

10. Bansode T and Habeeb SI. Synthesis and antimicrobial activity of some phenothiazine chalcones and its metal complexes, *I. J. Chem Tech Research*, 2017, 10(9):240-244.

11. Singh K, Kumar Y, Puri P, Kumar M, Sharma C. Cobat, nickel, copper and zinc complexes with 1,3-diphenyl-1H-pyrazole-4-carbox -aldehyde schiff bases: Antimicrobial, spectroscopic, thermal and fluorescence studies. *Europian J. of Medicinal Chemistry*. 2012 ,52:313-321.

RESEARCH ARTICLE

OPEN ACCESS

QSAR and Toxicity profile of Synthesized Derivatives of 5-aryl/di fluro phenyl substituted-1,3,4-Thidiazole-2-amine

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ABSTRACT

The synthesized series of derivatives of 5-substituted-alkyl/aryl-1,3,4-thidiazole-2-amine were confirmed by spectral techniques such as IR, ^{1}HNMR, LCMS etc. Computational study of the synthesized molecule gives us clear cut idea about the toxicity and ADMET parameters. The synthesized derivatives didn't show any toxicity issues which was predicted in result. Toxicity profile of the synthesized derivatives were check out in silicon regarding mutagenic, tumorigenic, effect on reproductive system, eye irritant properties. Antimicrobial studies of synthesized molecules are the parameter of screening of synthesized derivatives which was also produces acceptable result. In that molecular modelling and docking study reveals the antimicrobial properties with selected protein target via literature survey and PDB. Due to increase in AMR problem day by day cause the resistance to antibiotic drugs and it affects the severe problems to community and hospitalized patients as well as healthy persons. Mainly these problems arises due to population, industrialization, pollution and deforestation, new microorganisms are continuously producing and cause diseases such as swine flu, antiviral infection, and asthma in short infectious diseases increased day by day. To fight against these harmful microorganisms there was continuous and consistent effort of scientist to develop new molecules which could be used for the treatment of infections.

Key words: -substituted-alkyl/aryl-1,3,4-thidiazole-2-amine, mutagenic, tumerogenic, effect on reproductive system, eye irritant, IR, ^{1}HNMR, LCMS, AMR etc.

INTRODUCTION

Heterocyclic compounds are wide range of pharmaceutical, agrochemicals, veterinary, antioxidant, corrosion inhibitors, as a copolymer, dye stuff, useful for synthetic purpose [1]. These are very much interest in our daily life, some of them as an antibiotics such as penicillin's, cephalosporin; alkaloids such as vinblastine, morphine, reserpine etc. had heterocyclic moiety. The cyclic organic compounds which contain at least one hetero atom, the most common heteroatom are the nitrogen, oxygen and sulphur but heterocyclic rings containing other hetero atoms are also widely known. [2-3]. To solve the medical life issues synthetic chemistry joined with medicinal chemistry, computational chemistry, drug chemistry which is becomes an important fields of chemistry. Modern drug discovery focuses on the synthesis of specific bimolecular targets, which invariably contain a heterocyclic component or design drug which has been structural and functional analogues to mimic or study the change in pharmacological properties. A key challenge in the synthesis of such targets continues to be the development of new pathways and improvement of existing pathways [4]. Structure based drug discovery (SBDD) is a proven strategy for the rational development of small molecules of therapeutic interest without necessitating its synthesis at the preliminary stages [5-6].

In this research work we especially emphasis on molecular modelling and docking study of synthesized derivatives of 5-substituted -1,3,4-thidiazole-2-amine by computational method chosen for the identification of potential target specific ligands (lead generation), synthesis and computational screening were carried out in pursuit of designing some potential novel antimicrobial compounds carrying 1,3,4-Thiadiazoles rings as core nucleus.

1, 3, 4- thiadiazole core containing drugs are currently in the market: acetazolamide®) and methazolamide® are diuretics, acting through inhibition of carbonic anhydrase; their derivatives display additional activities, including anticonvulsant and selective cerebral vasodilation, as well as the anticipated inhibition of carbonic anhydrase, cefazolin sodium® (CFZL; 3) and cefazedon® (CFZD; 4)-first-generation cephalosporins and megazol® an antiparasitic drug [7-10].
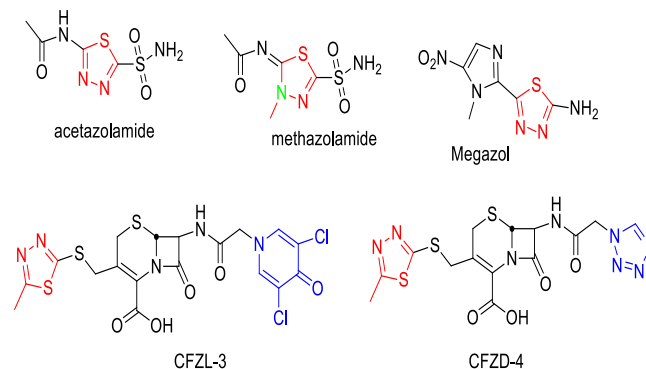
Fig.1: Structures of 1, 3, 4- thiadiazole core containing drugs available in the market

METHODOLOGY

Methods to study the *in silico* activity:

Software and program
Chemsketch was used to draw the ligand compounds. Accelry's Discovery studio v4.0 and Schrodinger's maestro visualization program v9.6 [11] were utilized to visualize the protein-ligand structures, H-bonds, measurement of bond lengths and to render images. Manual Pharmacophore hypothesis generation module of Schrodinger's maestro v9.6 was used for pharmacophore features mapping of the compounds along with location and calculation of distance between the pharmacophore features. MGL Tools version 1.5.6 was used for the preparation of the ligands and protein receptors in pdbqt format and to visualize and estimate the grid box size for docking calculations. Autodock 4.0 [12] is the software used for the docking calculation. Molinspiration and Orisis property explorer and programs were used to predict the ADMET properties of the compounds.

QSAR study of the synthesized derivatives:
Based on the descriptor values predicted by the Molinspiration and Osiris property explorer online servers [13] all the synthesized compounds successfully satisfied all the parameters of Lipinski's rule of five [14] (the mol. wt. must be less than 500 Da, the number of hydrogen donors and log P values should be less than five; the refractivity molar range shall be between 40 to 130 and the number of

hydrogen bond acceptors should not exceed ten.) and all the present investigated synthesized compounds show that all the compounds have a promising oral bioavaibility and ADME. As per the Veber's rule, oral bioavailability of drugs could be measured by the total polar surface area (TPSA) of the compound along with molecular weight, number of H-bonds and the number of rotatable bonds. Good orally bioavailabe small molecules is marked by small molecular weight (less than 500 Da); the number hydrogen donor/ acceptors combined shall be less than 12, TPSA values less than 140 and the number of rotatable bonds must be less than ten [15].

Prediction of toxicity:
The toxicity predictions of the present studied synthesized derivatives using Osiris Property Explorer [16] were based on the functional group similarity for the query molecule with the *in vitro* and *in vivo* validated compounds in the database.

The result of toxicity analysis of all the analyzed compounds is described as follows "HIGH" means high tendency of toxicity; "MEDIUM" means the midcore and "NONE" means low toxic tendency as evident tabulated in table no. 1 and 2.

RESULTS AND DISCUSSION

All these synthesized computational screened *in silco* activity predicted the compounds possessed no toxic tendency, only TDB$_3$ (2-(3-(5-(2,3-difluorophenyl)-1,3,4-thiadiazol-2-yl) ureido) acetic acid) has showed high eye irritant toxicity. Otherwise remaining all synthesized derivatives of 1,3,4-thidiazole having urea moiety (as pharmacophore) as bridge connected with bioactive molecule used as cyclopentyl amine,morpholine and glycine (optically inactive aminoacid) showed acceptable pharmacological properties based on analogues.

Table 1: ADME parameters of the synthesized derivatives:
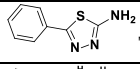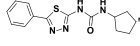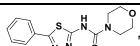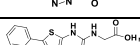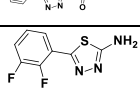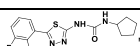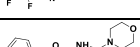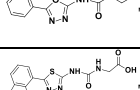
Sr. No	synthesized derivative	Molecular Formula	Mol. wt.	Log P	H-bond donors	H-Bond acceptors	Rotatable bonds	TPSA
1	TDA	$C_8H_7N_3S$	177.2	1.6	1	3	1	80.0
2	TDA$_1$	$C_{14}H_{16}N_4OS$	288.3	2.9	2	5	3	95.1
3	TDA$_2$	$C_{13}H_{14}N_4O_2S$	290.3	1.9	1	6	2	95.5
4	TDA$_3$	$C_{11}H_{10}N_4O_3S$	278.2	0.8	3	7	4	132.4
5	TDB	$C_8H_5N_3F_2S$	213.2	1.8	1	3	1	80.0
6	TDB$_1$	$C_{14}H_{14}N_4OF_2S$	324.3	3.1	2	5	3	95.1
7	TDB$_2$	$C_{13}H_{12}N_4O_2F_2S$	326.3	2.1	1	6	2	95.5
8	TDB$_3$	$C_{12}H_{12}N_4O_3F_2 S$	310.3	1.3	3	7	4	132.4

Table 2: Toxicology profile of the synthesized derivatives

Sr.No	Compound Code	Mutagenic	Tumerogenic	Effect on Reproductive system	Eye Irritant
1	TDA	NONE	NONE	NONE	NONE
2	TDA$_1$	NONE	NONE	NONE	NONE
3	TDA$_2$	NONE	NONE	NONE	NONE
4	TDA$_3$	NONE	LOW	NONE	NONE
5	TDB	NONE	NONE	NONE	NONE
6	TDB$_1$	NONE	NONE	NONE	NONE
7	TDB$_2$	NONE	NONE	NONE	NONE
8	TDB$_3$	NONE	LOW	NONE	HIGH

CONCLUSION

In Silco study of synthesized derivatives e.g (2-(3-(5-(phenyl/2,3-difluorophenyl)-1,3,4-thiadiazol-2-yl)ureido)acetic acid,1-cyclopentyl-3-(5-phenyl-1,3,4-thiadiazol-2-yl)urea has been acceptable pharmacological properties which can be extend our research work. With the help of urea moiety by further modification novel molecule metal complex can be synthesized. Before synthesis designed and virtual screening is essential. Computational drug designing is very good tool in drug chemistry to step in synthesized analogues of standard drug.

REFERENCES

1. Arora Pragi, Arora Varun, Lamba HS, Wadhwa Deepak, Importance of Heterocyclic Chemistry: A Review IJPSR, 2012; Vol. 3(9): 2947-2954.

2. Kunied T, Mutsanga H, The chemistry of heterocyclic compounds,Palmer,B,2002:175

3. Abbas Al-Mulla, A Review: Biological Importance of Heterocyclic Compounds, Der Pharma Chemica, 2017; 9(13):141-147.

4. Katritzky AR & Boulton AJ, "Advances in Heterocyclic Chemistry", Volume 7 Academic Press New York and London, 1966

5. Whittle Peter J., and Tom L. Blundell. *Annual review of biophysics and biomolecular structure* 1994; 23: 349-375.

6. Blundell, Tom L. *Nature* 1996; 23: 384.6604 .

7. Masereel B, Rolin S, Abbate F, Scozzafava A, Supuran CT, J. Med.Chem. 2002; 45: 312– 320.

8. Burger's Medicinal Chemistry and Drug Discovery, 6th ed, (Ed.: D. J. Abraham), Hoboken, Wiley, 2003; 1: 690.

9. Li Y, Geng J, Liu Y, Yu S, Zhao G. Chem Med Chem, 2013; 8: 27-41.

10. Martınez A, Alonso M, Castro A, Pe´rez C, Moreno F. J Med Chem, 2002; 45: 1292-1299.

11. Maestro (Version 9.6). 2013 New York, NY: Schrödinger, LLC

12. Goodsell, David S., Garrett M. Morris, and Arthur J. Olson. *Journal of Molecular Recognition* 1996; 9.1: 1-5.

13. Vanommeslaeghe, Kenno, *et. al,* "CHARMM general force field: A force field for drug-like molecules compatible with the CHARMM all-atom additive biological force fields." Journal of computational chemistry 2010; 31.4: 671-690.

14. http://accelrys.com/products/discoverystudio/visualization-download.php.

15. Morris, Garrett M., *et. al,* Journal of computational chemistry 1998; 19.14: 1639-1662

16. Maestro (Version 9.6). New York, NY: Schrödinger, LLC ,2013.

Formulation and Evaluation of Diclofenac Sodium Gel by Using Carbopol

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ABSTRACT

High Molecular weight water soluble homo polymer of Carbopol are reported to possess very high viscosity in low concentration, transparency, film forming properties and are useful in formation of gel. The objective of this research is to prepare and evaluate 2% polymer containing transdermal gel of Diclofenac sodium. The gel was prepared and evaluated for pH, Spreadability, Consistancy, Homogeneity, Drug Content, Skin Irritation test and In vitro Diffusion Study. The percentage drug release was 97.66%. It can be concluded that preparation of diclofenac sodium by using 2% carbopol 934P Grade were prepared and evaluated.

Keywords – Water soluble polymer, Diclofenac Sodium, Carbopol 934 P, Topical drug delivery.

INTRODUCTION

Topical gel Preparation is intended for skin application and to certain mucosal surfaces for local action of percutaneous penetration of medicament of or for their emollient and protective action [1]. Gel are typically formed from a liquid phase that has been thickened with other components. The continuous liquid phase allows free diffusion of molecules through the polymers and hence release should be equivalent to that from the simple solution [2]. NSAID's are non-steroidal drugs having excellent anti-inflammatory and analgesic activity but NSAID produces GIT ulceration, liver and kidney

trouble especially in case of oral administration. In view, of adverse drug reaction associated with oral formulation, diclofenac sodium is increasingly administered by topical route [3]. Carbapol 934P is used as water soluble or hydrophilic polymers topically in gel drug delivery system[4], due to their non-greasy properties; they can provide easily washable film on the skin and are non-toxic[5]

METHODOLOGY

Diclofenac sodium was received from Sahyadri scientific Islampur, Carbapol 934 P was purchased by research-lab fine chem, Mumbai, and Triethanolamine was purchased by research-lab fine chem, Mumbai. All other Ingredients were of analytical grade.

Procedure of gel preparation:
Diclofenac sodium gel was formulated by using selected concentration of 2% Carbopol 934P polymer for further formulation to getting better result.

About 1g of diclofenac sodium was weighed and dissolved in 30 ml of ethanol (95%), to this solution; specified quantity of propylene glycol was added and dissolved (solution A). Weighed quantity of polymer carbopol 934P was added to sufficient amount of water, mix uniformly by using magnetic stirrer, to that added triethanolamine while continuous stirring (solution B). Solution A and B were mixed thoroughly and the final weight was made up to 100g.

Table 1-

Sr. No.	Name of the Ingredient	Quantity Given	Quantity Taken
1.	Diclofenac Sodium	1g	1g
2.	Carbopol 934P	2g	2g
3.	Triethanolamine	1.5ml	1,5m1
4.	Glycerine	10ml	10ml
5.	Propylene Glycol	10ml	10ml
6.	Ethanol	30ml	30ml
7.	Distilled water	q.s.	Up to 100ml

Evaluation of Carbopol 934 P gel containing diclofenac sodium and marketed gel-

The above formulated Diclofenac Sodium gel containing polymer carbopol 934 P and marketed gel were subjected to evaluation for the following parameter –

A. pH: The of the gel formulations was determined by using digital pH meter(Systronic Instruments, India) by placing the glass electrode completely into the gel system and measure the pH (Table2).

B. Spreadability: It was determined by wooden block and glass slide apparatus. Weights 20g were added to pan and the time was noted for upper slide (movable) to separate completely from the fixed slides. (Table2)[6]. Spreadability was then calculated by using the formula:

$$S = M.L / T$$

Where, S = Spreadability, M = Weight tide to upper slide, L = Length of glass slide, T = Time taken to separate the slide completely from each other.

C. Viscosity: Viscosity measures the flow characteristics of topical gel formulation. Change in viscosity of the product is indicative of change in stability and effectiveness of product. The viscosity of topical gel were determined by using Brook-Field viscometer ML VT115 using Spindle no. 64, at spindle Speed 30rpm at 25°c for 5min.

D. Homogeneity: All developed gels were tested for homogeneity by visual inspection after the gels have been set in the container. They were tested for their appearance and presence of any aggregates.

E. Skin irritation test: Test for irritation was performed on human volunteers. For each gel, 5 volunteer were selected and 1g of formulated gel was applied on the area of 2 square inch to the back of hand. The volunteers were observed for lesions or irritation. (Table 2)

F. Drug content: A specific quantity (100mg) of developed gel and marketed gel were taken and dissolved in 100 ml of phosphate buffer of pH 6.8.

Table 2: Values of evaluation parameters of developed gel and marketed gel

	pH	Spreadability (g.cm/sec)	Viscosity (cp)	Homogeneity	Skin irritation test	Drug content (%)
Formulated Gel	6.8	6.0	100	Good	Nil	99.98
Marketed gel	6.8	5.5	99	Good	Nil	99.90

Table 3: In vitro diffusion studies of Formulated and marketed gel

Sr. No.	Time Interval (min)	Medium pH	% Drug Release	
			Formulated Preparation	Marketed Preparation
1	30	6.8	49.31	50.61
2	60	6.8	64.96	67.12
3	90	6.8	79.24	80.24
4	120	6.8	97.66	98.44
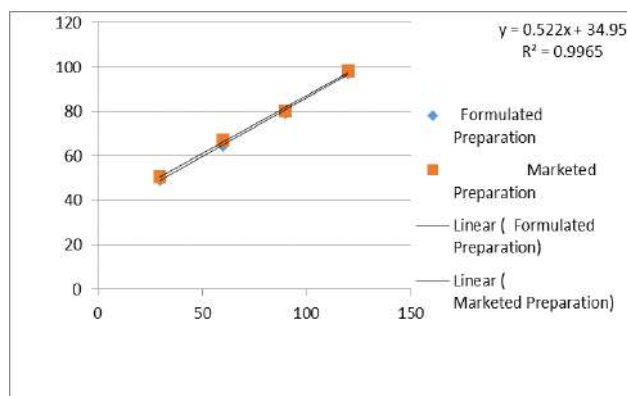
Fig: In vitro Diffusion of Diclofenac Sodium Gel

The volumetric flask containing gel solution was shaken for 2 hrs on mechanical shaker in order to get complete solubility of drug. The solution was filtered and estimated spectrophotometrically at 276.0nm using phosphate buffer pH 6.8 as blank (Table 2) [7].

G. In vitro diffusion Study : Phosphate buffer of pH 6.8 was used for in vitro release as a receptor medium. The cellophane membrane (Prepared from eggs) was used in Franz diffusion cell. The gel sample was applied on the membrane and then fixed in between donor and receptor compartment of diffusion cell. The receptor compartment content phosphate buffer (50ml) of pH 6.8. The temperature of diffusion medium was thermostatically controlled at 37± 1° by surrounding water in jacket and the medium was stirred by magnetic stirrer at 500 rpm. The sample at predetermined intervals were withdrawn and replaced by equal volume of fresh fluid. The samples withdrawn and replaced by equal volume of fresh fluid. The samples withdrawn were spectrophotometrically estimated at 276 nm against their respective blank [8].

RESULTS AND DISCUSSION

The pH values of Formulated and marketed gel was 6.8. The values of spreadability indicated that the gel is easily spreadable by small amount of shear. Spreadability of marketed gel was 5.5g.cm/sec while prepared gel was 6g.cm/sec, indicating spreadability of carbopol 934P containing diclofenac sodium was good as marketed gel.

The consistency (viscosity) reflects the capacity of the gel, to get ejected in uniform and desired quantity when the tube is squeezed. The formulated and marketed gel showed good homogeneity with absence of lumps. The formulated preparations were much clear and transparent as compared to marketed gel.

The skin irritation studies of formulated gel were carried out on human volunteers and that confirmed the absence of any irritation on the applied surface. In vitro permeability study should that permeation studies of formulated and marketed gel were comparable.

It was observed that the Diclofenac Sodium containing polymer carbopol 934P gel produced better spreadability and consistency as compared to marketed diclofenac sodium gel. The formulated gel showed good homogeneity, no skin irritation, good consistency and in vitro permeability was comparable with marketed gel. The carbopol 934P forms water washable gel because of its water solubility and has wider prospects to be used as a topical drug delivery system.

CONCLUSION

The polymer being macromolecules of very high molecular weight remains unabsorbed from the skin and from our studies it can be concluded that carbopol 934P can be used for topical dosage forms for external application.

It has been observed that the formulated gel produces with good consistency, homogeneity, spreadability. Since the polymer is water soluble; consequently, it forms water washable gel and has wider prospect to be used as a topical drug delivery dosage form.

REFERENCES:

1. Narin J, Encyclopedia of Pharmaceutical Technology. Marcel Decker, New Work. 1997.
2. British Pharmacopoeia, International Publication. Vol II. 1993.
3. Williams AC, Topical and Transdermal Formulation. Pharmaceuticals Press Published,New York. 2003.
4. Smart JD, Mortazavi SA, Drug Delivery Research group, *Journals of Pharmacy and pharmacology.*
5. Singh MP, B.P. Nagari Formulation, Development and evaluation, *International Journal of Pharmaceutical Erudition.*
6. Gupta GD, Gound RS, Release rate of nimesulide from different gellants. *Indian J Pharm Sci.1999;* 61: 229-234.
7. Sera UV, Ramana MV, In vitro skin absorption and drug release – a comparison of four commercial hydrophilic gel preparations for topical use. The Indian Pharmacist, **73,** 356-360 (2006).
8. Sahoo SK, Samal AR, Estimation and evaluation of secnidazole The Indian Pharmacist 2006; 5(46): 73.

RESEARCH ARTICLE   **OPEN ACCESS**

Synthesis and Characterization of Transparent Cadmium Sulphide (CdS) Thin Films By Chemical Bath Deposition Technique

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ABSTRACT

In the present manuscript we report the synthesis and characterization of transparent CdS thin films on glass and conducting (FTO) substrate by chemical bath deposition technique.As prepared samples were further characterized by X-ray diffraction (XRD), Scanning electron microscopy (SEM) and UV-VIS spectroscopy for the investigation of structural and optical properties of CdS respectively. XRD pattern recorded for the as prepared sample reveals the nanocrystalline cubic structure of CdS. From the SEM images it observed that the sample exhibits nanocrytalline CdS platelets like morphology. At low magnification structure seems like cabbage. For the study of optical properties of CdS absorption and transmission spectra were recorded. UV-VIS absorption spectra clearly gives the information that the absorption initiates at 524 nm and therefore band gap of CdS sample is ~ 2.36 eV. Similarly, from the UV-VIS transmission spectra, it is observed that transmittance of CdS thin film is approximately 99% above 400 nm.

Keywords: Nanocrystalline, X-ray diffraction, CBD technique, Platelets, Absorption spectra, Transmittance.

INTRODUCTION

Band gap is the fundamental property of semiconductor. Energy separation between the filled valence band and empty conduction band is called as band gap. The band gap of semiconductor increases with decrease in the particle size which is known as the quantum size effect. Currently size quantization effects of nanocrystalline semiconductors have vast attention towards metal-chalcogenide based systems in solar cell. CdX and PbX (X = S, Se, and Te) nanocrystalline thin films having relatively small band gaps and thus capable of harvesting photons in the visible and infrared region, are of great interest. The semiconductors have promising applications in biology, optics, and electronics and transport [1-4]. To synthesize nanocrystalline thin films with various particle size and shape several attempts have beenmade. CdS is a semiconductor with a direct band gap of 2.42 eV. It has interesting optical properties and applications in the field of light emitting diodes, solar cells, optoelectronics devices, photo catalyst, X-ray detectors, solar energy storing and in display devices [5-11]. It is used as a buffer layer in the formation of solar cell devices based on CIS, $CuInSe_2$, $CuInGaSe_2$, CdTe [12-14]. Now adays, CdS nanoparticles are used in Semiconductor Sensitized Solar Cells (SSSCs) to improve the performance of wide band gap semiconductor materials [15-18]. In SSSCs, CdS nanoparticle forms a thin layer on wide band gap semiconductors like ZnO, TiO_2 having nanostructured morphologies like nanorodes, nanotubes, etc. Chemical, physical and electrochemical methods are used for preparation of CdS thin films. They are such as vacuum evaporation [19], sputtering [20], spray pyrolysis [21], chemical bath deposition [22], electro-deposition [23], successive ionic layer adsorption and reaction method (SILAR) [24], screen printing [25]. Chemical bath deposition (CBD) is simple and economic. With the help of CBD method the thickness and transparency of thin film can be controlled easily.

In the present study, we describe the synthesis and characterization of nanocrystalline CdS thin films via CBD technique. The films deposited at various time intervals at low temperature (~60°C) using low precursor concentration. The prepared thin films are further investigated for their structural, surface morphological and optical properties.

METHODOLOGY

All chemical was purchased from s. d. fine-chemicals and used without any further purification. The cadmium sulfate ($CdSO_4 \cdot H_2O$) and thiourea ($H_2N \times CS \times NH_2$) were used as cadmium (Cd) and sulphur (S) precursors. Ammonia (NH_3) was used as complexing agent. For the deposition of nanostructured CdS thin film.

Cadmium sulfide films were prepared from cadmium sulphate and thiourea by CBD in alkaline solution. The typical procedure for the film growth is described as follows, drop by drop 25% NH_4OH solution is added into 500 ml beaker containing 150 ml of 1 mM $CdSO_4$ solution until the initially formed white precipitate is completely dissolved. The clean substrates are mounted vertically in the bath using Teflon holder, in such a way that the substrates are separated by 2 cm distance from each other & the wall of bath. Then 150 ml of 1mM thiourea [CS (NH$_2$)$_2$] is added in the bath solution. The temperature is gradually increased to 60°C under magnetic stirring for all samples. The films removed from the bath after 20 min were highly transparent and uniform with well adhesion to the glass substrate and therefore used for further characterization. The deposited CdS films were rinsed with double distilled water and alcohol and allowed to dry at room temperature, in ambient air. Thicknesses of the thin films of the samples were found to in the range 549 to 1389 Å. Fig 1. show the schematic of CBD method for the preparation of CdS thin films.Thicknesses of the thin films of the samples were found to in the range 549 to 1389 Å.

Figure 1. Schematic of Chemical Bath Deposition

$$CdSO_4 + NH_4OH \leftrightarrow Cd(OH)_2 + (NH_4)_2 SO_4$$
$$Cd(OH)_2 + 4NH_4OH \leftrightarrow Cd(NH_3)_4^{2+} + 2OH^- + 4H_2O$$
$$H_2N - C - H_2N - C = H_2N - C = NH$$
$$H_2N - C - H_2N + OH^- \leftrightarrow H_2N - C = NH + SH^-$$
$$Cd(NH_3)_4^{2+} + SH^- = CdS \downarrow + NH_4^+ + 3NH_3$$
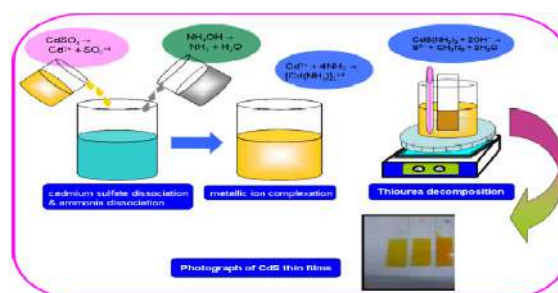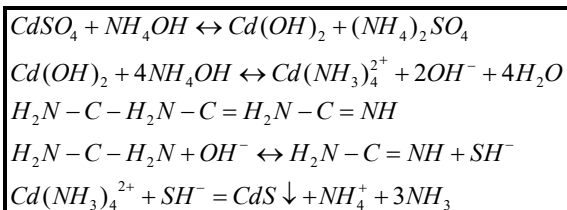
RESULTS AND DISCUSSION

- **X-ray diffraction studies:-**
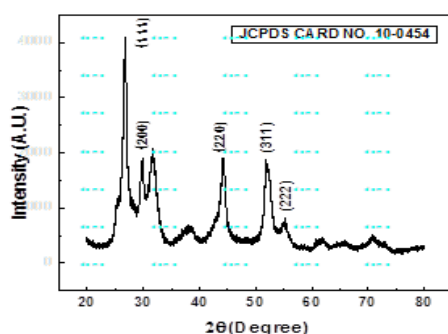
Figure 2. XRD spectra of Cadmium Sulphide (CdS) thin film prepared on glass substrate and

Figure 2 above shows XRD spectra recorded for CdS sample on glass substrate. From this XRD pattern it is observed that the diffraction peaks belonging to planes (111), (200), (220), (311) and (222) were observed at angles 26.76, 29.78, 44.14, 51.88 and 55.10 degrees and are well matching with the cubic phase (with the JCPDS card no.10-0454). From XRD pattern the crystalline nature of the synthesised sample is confirmed. The particle size of the prepared sample was calculated using equation Debay Scherrer's equation given below

$$D = \frac{0.9\lambda}{\beta COS\theta}$$

Where D is the size of nanostructure, β is the broadening of diffraction line measured at half of its maximum intensity (FWHM) and λ is the wavelength of X-ray (1.5406 Å). The calculated particle size of nanostructure is 16 nm.

Scanning electron microscopy (SEM):-

Figure (a) and (b) shows scanning electron micrographs of CdS thin films prepared by CBD method. From figure it is clearly observed that the

sample exhibits nanocrystalline CdS platelets like morphology. At low magnification structure seems like cabbage. The density of these platelets is uniform throughout the sample. The platelets' are having size of 1µm and thickness 10-15 nm. The films are having throughout uniform structure and no cracks are observed in the film.
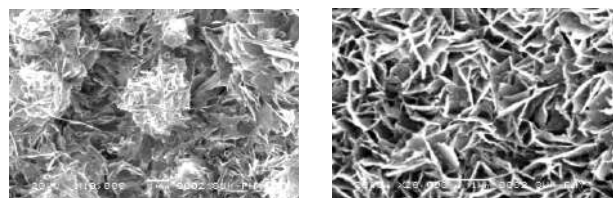
Figure 3. (a) and (b) Scanning Electron Microscopy (SEM) micrograph of Cadmium Sulphide (CdS) thin films prepared on glass by CBD method

UV-VIS Spectroscopy studies:
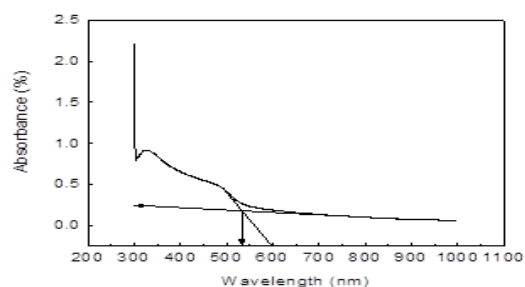
Figure 4. (a) UV-VIS absorption spectra
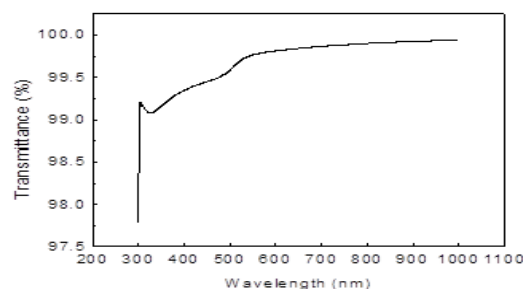
Figure 4.(b) UV-VIS transmission spectra of Cadmium Sulphide (CdS) thin films prepared on glass by CBD method

Above Figure3 (a) and 3 (b) shows absorption and transmission spectra recorded for CdS thin films in the wavelength range 300 nm and 1000 nm. From figure (a) it is clearly observed that the absorption initiates at 524 nm. and therefore, band gap of CdS sample is ~ 2.36 eV. From figure (b) it is clearly observed that the transmittance of CdS film deposited by CBD method is approximately 99% above 400 nm.

CONCLUSIONS

Transparent CdS thin films were successfully deposited on glass substrate by chemical bath deposition technique. XRD pattern recorded for the as prepared sample confirms the nanocrystalline cubic structure of CdS. From the SEM images it observed that the sample exhibits nanocrystalline CdS platelets like morphology. At low magnification structure seems like cabbage. For UV-VIS absorption spectra clearly gives the information that the absorption initiates at 524 nm and therefore band gap of CdS sample is ~ 2.36 eV. Similarly, from the UV-VIS transmission spectra, it is observed that transmittance of CdS thin film is approximately 99% above 400 nm.

REFERENCES

1. Mathew X, Thompson GW, Singh VP, McClure JC, Velumani S, Mathews NR, Sebastian, P. J. Sol. Energy Mater. Sol. Cells 76 2003:293.

2. Kutzmutz S, Lang G, Heusler KE, Electrochimica Acta, 2001;47:955.

3. Shen C, Zhang X, Li H, Mater. Sci. Eng. B, 2001;84:265.

4. Soliman M, Kashyout AB, Shabana M, Elgamal M, Semi. Renew. Energy, 2001;23: 471

5. Sharma RK, Singh G, Rastogi AC, Sol. Energy Mater. Sol. Cells, 2004;82:201

6. Chang YJ, Munsee CL, Herman GS, Wager JF, Mugdur P, Lee DH and Chang CH, Surf. Interface Anal, 2005;37:398.

7. Reynolds JE, *J. Chem. Soc.*,1984; 45: 162.

8. Chopra KL, Kainthla RC, Pandya DK, Thakoor AP, In Physics of Thin Films, Academic Press: New York, 1982;11:167.

9. Lokhande CD, Mater. Chem. Phys., 1991;28:1.

10. Lincot D, Froment M, Cachet H, In Advances in Electrochemical Science Engineering, Alkire RC, Kolb DM (eds), Wiley-VCH: New York, 1998;6:165

11. Nair PK, Nair MTS, Garcia VM, Arenas OL, Pena Y, Castillo A, Ayala IT, Gomez-daza O, Sanchez A, J. Campos, H. Hu, R. Suarez, M. E. Rincon, Sol. Energy Mater. Sol. Cells, 1998;52: 313.

12. Savadogo O, Sol. Energy Mater. Sol. Cells, 1998;52:361.

13. Kaur J, Pandya DK, Chopra KL, J. electrochemistry, 1980;48: 127.

14. Pandya DK, Chopra KL, AcademicPress: NewYork, 1980.

15. Mokrushin S, Tkachev Y, Kolloidn Z. 1961;23: 438

16. Kitaev G, Uritskaya A, Mokrushin S, Russ. J. Phys. Chem**.,** 1965**;**39.

17. Moutinho HR, Albin D, Yan Y, Dhere RG, Li X, Perkins C, Jiang CS, Thin Thin Solid Films, 2003;436:175.

18. Chopra KL, Paulson PD, Dutta V, Prog. Photovolt. Res. Appl. 2004;12: 69.

19. Bruss L, *J. Phys. Chem*., 1986;90:2560.

20. Nozik AJ, Williams F, Nenadovic MT, Rajh T, and MiCiE 0I, *J.Phys. Chem.*, 1985;89:397. Chapter V 163

21. Torimoto, T.Nagakubo, S.Nishizawa, M.Yoneyama, Langmuir, 1998;14:7077.

22. Vossmeyer T, Katsikas L, Gienig M, Popovic IG, Diesner K, Chemseddine A, Eychmiiller A, and Weller H, J. Phys. Chem., 1994;98:7665.

23. Nanda KK, Sarangi SN and Sabu SN, Nanostr. Mat., 1988;10-8:1401.

24. Yao K, Nishimura S, Ma T, Inoue K, Abe E, Tateyama H, Yamagishi A, Eng. Aspects, 2002;201:63.

25. Jinesh KB, Wilson KC, Thampi SV, Kartha CS, Vijaykumar KP, Abe T, Kashiwaba Y, Physica A, 2003;19:303.

Microwave assisted synthesis and biological evaluation of tetrahydropyrimidine derivatives

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ABSTRACT

Twelve [6- (2-methoxy-phenyl)-2-oxo-4-phenyl / substituted phenyl-1, 2,3, 4- tetrahydropyrimidine-5-yl] acetic acid derivatives have been synthesized in a two-step reaction. In the first step benzene or substituted benzene react with succinic anhydride in presence of aluminum trichloride (Friedel Craft reaction) to obtain 4-(substituted phenyl)-4-oxo- butanoic acid. Second step involves synthesis of [6-(2-methoxy-phenyl)-2-oxo-4-phenyl/substituted phenyl-1,2,3,4-tetrahydro pyrimidine -5-yl] acetic acid by reaction between4-(substituted phenyl)-4-oxo- butanoic acid, urea and substituted aldehydes (Biginelli reaction). Their structures are confirmed by IR, ^{1}H NMR. T.L.C. of synthesized compounds performed in chloroform: ethanol (3:1) solvent system. The anti-inflammatory activity of all compounds has been recorded on the basis of reference standard Indomethacin. All the compounds showed tendency to cause a fall in oedema and showed anti-inflammatory activity. The anti-inflammatory data shows that use of anisole in first step plays important role in the activity. Anti-inflammatory activity of all compounds was taken by Carrageenan induced rat paw oedema as described by Winter et al. on albino rats.

Keywords: Pyrimidine, Anti-inflammatory activity, aryl alkanoic acid, NSAID's.

INTRODUCTION

NSAIDs are a mainstay in the treatment of inflammatory disease and are among the most widely used drugs worldwide [1]. The main limitation in using NSAIDs lies in their side effects, which include gastrointestinal ulcerogenic activity and bronchospasm [2]. In recent years, several novel approaches to reducing the GI toxicity of NSAIDs have been taken, with promising results.

In medicinal chemistry pyrimidine derivatives have been very well known for their therapeutic applications [3]. The presence of a pyrimidine base in thymine, cytosine and uracil, which are the essential binding blocks of nucleic acids, DNA and RNA is one possible reason for their activity [4].

The literature indicated that compounds having pyrimidine nucleus possess broad range of biological activities, like 5-fluorouracil as anticancer [5]; idoxuridine and trifluoridine as antiviral [6] zidovudine and stavudine as antiHIV [7]; trimethoprim, sulphamethiazine and sulphadiazine as antibacterial [8]; sulphadoxin as antimalarial and antibacterial [9]; minoxidil and prazosin as antihypertensive [10]; barbiturates e.g. phenobarbitone as sedative [11], hypnotics and anticonvulsant [12]; propylthiouracil as antithyroid [13]; thionzylamine as H_1 –antihistamine [14]; and toxoflavin as antibiotics [15].

The main mechanism of action of NSAIDs is the inhibition of the enzymes possessing cyclooxygenase (COX) activity, which are involved in the formation of prostaglandins and thromboxanes from arachidonic acid contained in cellular membranes [16]. The relationship between the risk of serious GI side effects and the use of nonselective NSAIDs is well established[17]. Side effects to NSAIDs vary from person to person. Common side effects to all NSAIDs are abdominal pain, diarrhoea, nausea, and fluid retention [18].

Synthetic approaches based on chemical modification of some 1,2,3,4 tetrahydro pyrimidine derivatives are undertaken with the aim of improving safety profile. Encouraged by the findings of an exhaustive literature survey, we aimed to develop potent and nontoxic drug.

METHODOLOGY

The first step of reaction is by acylation of substituted benzene and succinic anhydride in the presence of aluminium chloride. The final step in the mechanism is believed to be the condensation between the aldehyde and urea, with some similarities to the Mannich Condensation. The imminium intermediate generated acts as an electrophile for the nucleophilic addition of the ketoesterenol and the ketone carbonyl of the resulting adduct undergoes condensation with the urea NH_2 to give cyclized product [19,20].

Scheme -
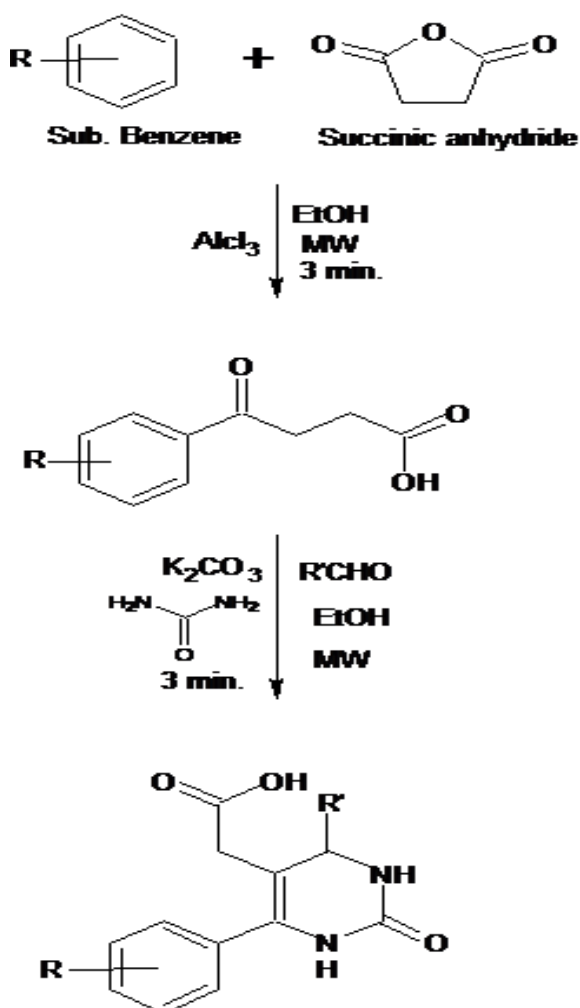
Step – I. Synthesis of the 4-(substituted phenyl) -4-oxo- butanoic acids[21]

Microwave assisted synthesis were carried out using substituted benzene, succinic anhydride and a powdered aluminum chloride. Alcohol used as energy transfer medium. Stirring was provided manually and temperature maintained at constant value for three to five minutes.

Allowed to cool the resulting reaction mixture, added 15 ml of water. The 4-(substituted phenyl) -4-oxo-butanoic acid separated as colorless oil, which soon solidified. Cool in ice, filter off acid at the pump and wash with cold water.

Step – II. Synthesis of [4, 6-(4-substituted aryl)-2-oxo- 1, 2, 3, 4-tetrahydro-pyrimidin-5-yl]-ethanoic acid [22]
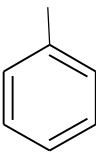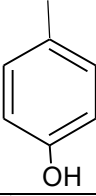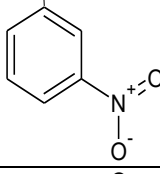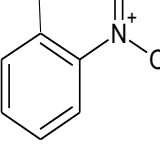
An equimolar reaction mixture of 4-(substituted phenyl) -4-oxo- butanoic acid (0.0022 mol) urea (0.002 mol), substituted aldehyde (0.0022 mol) and K_2CO_3 (0.0022 mol) in 7 ml ethanol were refluxed for three to five minutes. The reaction mixture was cooled and the solid obtained was dissolved in hot water and filtered. The filtrate was neutralized with acetic acid. The product thus obtained was recrystallized from methanol.
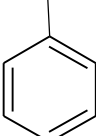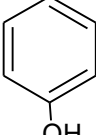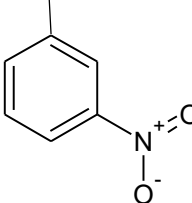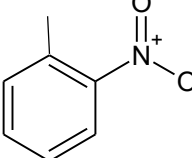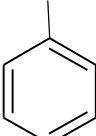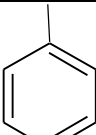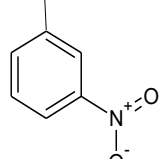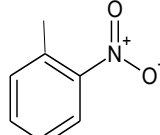
RESULTS AND DISCUSSION

Table 1: Characterization data for 4-(substituted phenyl) -4- oxo- butanoic acids.

Comp.	R	Molecular formula	Molecular weight	% Yield	M.P.
A	CH_3	$C_{11}H_{12}O_3$	192.23	86.12	127^0-128^0
B	H	$C_{10}H_{09}O_3$	177.19	87.48	120^0-121^0
C	OCH_3	$C_{11}H_{12}O_4$	208.23	78.02	136^0-137^0

Table 2. Characterization data for of [6-(2-methyl-phenyl)-2-oxo-4-phenyl/substituted phenyl-1, 2, 3, 4-tetrahydropyrimidine-5-yl] acetic acid.

Comp code	R	R¹	Mol. Formula/Wt	% Yield	M.P.	Rf value Chloroform :Ethanol (3:1)
A1	CH_3		$C_{19}H_{18}N_2O_3$ 322.38	80.78	123	0.661
A2	CH_3		$C_{19}H_{18}N_2O_4$ 338.38	73.43	133	0.592
A3	CH_3		$C_{19}H_{17}N_3O_5$ 367.38	60.45	128	0.622
A4	CH_3		$C_{19}H_{17}N_3O_5$ 367.38	61.09	131	0.643

B1	H		$C_{18}H_{15}N_2O_3$ 307.38		118	0.580
B2	H		$C_{18}H_{15}N_2O_4$ 323.38	69.86	128	0.611
B3	H		$C_{18}H_{15}N_3O_5$ 355.37	68.13	122	0.592
B4	H		$C_{18}H_{15}N_3O_5$ 355.37	57.65	127	0.573
C1	OCH_3		$C_{19}H_{18}N_2O_4$ 339.37	77.57	122	0.540
C2	OCH_3		$C_{19}H_{18}N_2O_5$ 354.38	78.87	131	0.602
C3	OCH_3		$C_{19}H_{18}N_2O_6$ 383.38	65.56	127	0.491
C4	OCH_3		$C_{19}H_{17}N_3O_6$ 383.38	63.89	132	0.511

Spectroscopic data

A1- 3-[6-(4-methylphenyl)-2-oxo-4-phenyl-1, 2.3.4-tetrahydropyrimidine-5-yl] propionic acid-

IR (KBr,cm^{-1}) 3430 (O-H)str.,3068 (Ar-C-H)str,2966 (C-H)str, 1697(C=O)str,1574 (C=C)str,1109(C-N)str.

^{1}H NMR (CDCl$_3$) 7.2-8 (9 H (s) of Ar-H), 6.9 (3 H (s) of pyrimidine), 11.0 (1 H (s) of –COOH), 1.2(3 H (s) of CH$_3$), 1.42 (2H (t) of CH$_2$).

A2-3-[6-(4-methylphenyl)-2-oxo-4-hydroxy-phenyl-1,2.3.4-tetrahydropyrimidine-5-yl]propanoic acid-3337 (O-H) str.,3068 (Ar-C-H) str,1123 (C-N) str, 1698(C=O) str,1574 (C=C) str

A3-3-[6-(4-methylphenyl)-4-(3-Nitrophenyl)-2-oxo-1,2.3.4-tetrahydropyrimidine-5-yl]propanoic acid-3437 (O-H) str.,3068 (Ar-C-H) str, 1699(C=O) str,1534 (C=C) str,1073(C-N) str. 1351(NO$_2$) str.

A4-3-[6-(4-methylphenyl)-4-(2-Nitrophenyl)-2-oxo-1,2.3.4-tetrahydropyrimidine-5-yl]propanoic acid-3437 (O-H) str.,3068 (Ar-C-H) str, 1698(C=O) str,1574 (C=C) str,1073(C-N) str. 1377(NO$_2$) str.

B1-4, 6-diphenyl-2-oxo-1, 2, 3, 4 – tetrahydropyrimidin-5yl-propionic acid –IR (KBr,cm^{-1})3423(O-H)str.,3063(Ar-C-H)str,2935,2850 (C-H)str, 1680(C=O)str,1598 (C=C)str,3172(N-H)str.
^{1}H NMR (CDCl$_3$)7.2-8 (9 H (s) of Ar-H), 6.9 (3 H (s) of pyrimidine), 11.0 (1 H (s) of –COOH), 1.42 (2H (t) of CH$_2$).
B2-4-p-hydroxy-6-phenyl-2-oxo-1, 2, 3, 4 – tetrahydropyrimidin-5yl-propionic acidIR (KBr,cm^{-1})3441(O-H)str.,3030 (Ar-C-H)str,2850 (C-H)str, 1690(C=O)str,1550(C=C)str,3172(N-H)str., 1102(C-N)str., 1650(C=O) amide str.
B3–4-m-nitrobenzen-6-phenyl-2-oxo-1,2,3,4–tetrahydropyrimidin-5yl-propionic acidIR (KBr,cm^1)3338(OH)str.,3068(ArCH)str,2211,2898(CH)str,1697(C=O)str,1574(C=C)str,1344(NO$_2$)str., 1185(C-N)str.
B4-4-o-nitrobenzen-6-phenyl-2-oxo-1,2,3,4–tetrahydropyrimidin-5yl-propionicacidIR (KBr,cm^1)3394(OH)str., 3040(ArCH)str,2966(CH)str,1697(C=O)str,1574(C=C)str,1313(NO$_2$)str. 1193(C-N)str.,3170(N-H)str.
C1 - 3-[6-(4-methoxyphenyl)-4-phenyl-2-oxo-1, 2, 3, 4-tetrahydropyrimidin-5-yl] propionic acid
IR (KBr,cm^{-1})3398(O-H)str.,3032 (Ar-C-H)str,2930 Me- (C-H)str, 1690(C=O)str,1570 (C=C)str,3140(N-H)str,1188(C-N)str,1390 methoxy (C=O)str.
^{1}H NMR (CDCl$_3$) 7.2-8 (9 H (s) of Ar-H), 6.9 (3 H (s) of pyrimidine), 11.0 (1 H (s) of –COOH), 1.42 (2H (t) of CH$_2$),3.9 (3 H (s) of OCH$_3$).
C2--3-[6-(4-methoxyphenyl)-4-(4-Hydroxyphenyl)-2-oxo-1,2,3,4-tetrahydropyrimidin-5-yl] propionic acidIR (KBr,cm^{-1})3390(O-H)str.,3030(Ar-C-H)str,2950Me-(C-H)str, 1650(C=O)str,1580(C=C)str,3125(N-H)str,1292(C-N)str,1399 methoxy (C=O)str.
C3-3-[6-(4-methoxyphenyl)-4-(3-nitrophenyl)-2-oxo-1,2,3,4-tetrahydropyrimidin-5-yl]propionic acidIR (KBr,cm^{-1})3398(O-H)str.,3032 (Ar-C-H)str,2930 Me- (C-H)str, 1690(C=O)str,1570 (C=C)str,3140(N-H)str,1188(C-N)str,1390 methoxy (C=O)str, 1321(NO$_2$)str.
C4-3-[6-(4-methoxyphenyl)-4-(2-nitrophenyl)-2-oxo-1,2,3,4-tetrahydropyrimidin-5-yl]propionic acidIR (KBr,cm^{-1})3390(O-H)str.,3030(Ar-C-H)str,2950 Me- (C-H)str, 1650(C=O)str,1580(C=C)str,3125(N-H)str,1292(C-N)str,1399 methoxy (C=O)str, 1313(NO$_2$)str.
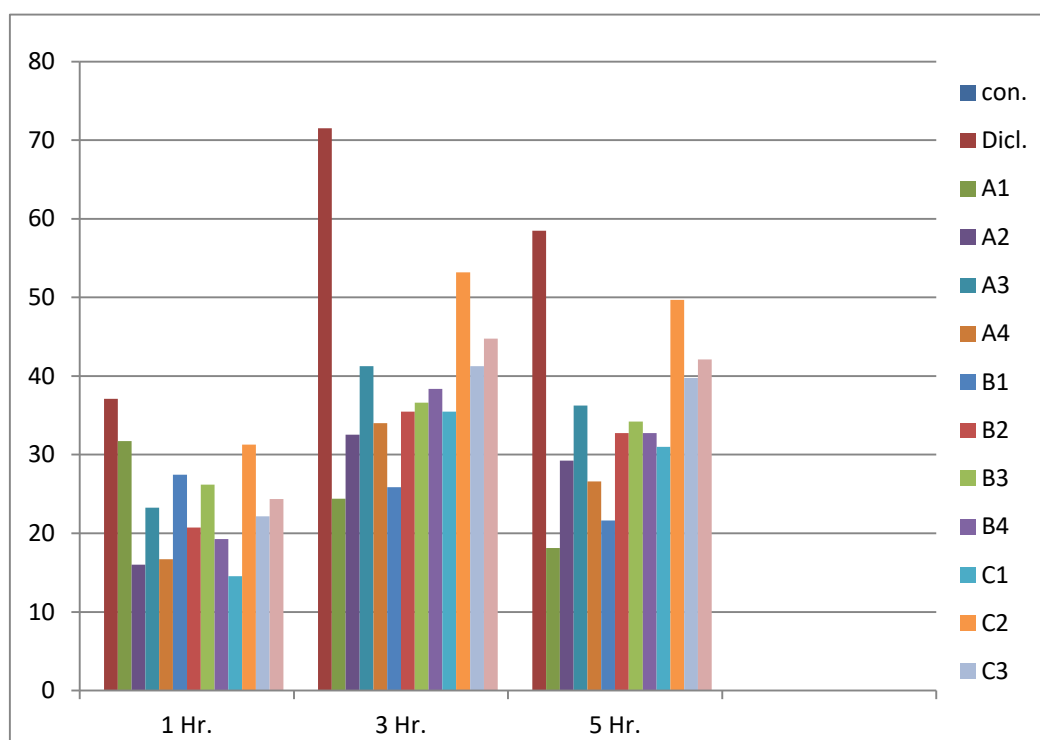
Fig. 1 - statistical graph showing comparisons in anti-inflammatory activity.

Table 3. Anti-inflammatory activity statistical data

Group	Dose	Carrageenan induced paw edema					
		1 Hr.		3 Hr.		5 Hr.	
		EV	EI	EV	EI	EV	EI
control	saline	2.68 ±0.19	-	3.44±0.35		3.42±.32	
Indomethacin	10 mg/kg	1.63±0.25	37.09	0.98±0.02**	71.51	1.40±.25*	56.47
A1		2.36±0.11	31.74	2.58±.06*	24.41	2.80±.05	17.12
A2		2.31±0.03	16.00	2.32±.07*	32.55	2.42±.02	27.23
A3		2.00±0.09	23.27	2.02±.12*	41.27	2.18±.01	36.25
A4		2.29±0.047	16.72	2.27±.03*	33.01	2.51±.017	26.60
B1		2.34±0.016	27.45	2.53±00*	25.87	2.68±.011	21.63
B2		2.18±0.011	20.72	2.22±.05*	35.46	2.30±.052	30.74
B3		2.03±0.034	26.18	2.18±.10**	36.62	2.25±0.60	34.21
B4		2.23±0.060	19.27	2.12±.01*	37.37	2.30±.052	30.74
C1		2.30±0.062	14.54	2.25±.06*	35.46	2.36±.053	30.99
C2		1.89±0.084	31.27	1.60±.06**	53.19	1.72±.027*	49.70
C3		2.14±0.050	22.18	2.02±.10**	41.27	2.06±.024	39.76
C4		2.08±0.103	24.36	1.90±.01**	44.76	1.98±.057	41.10

Values are expressed as mean ± SEM (n=6). EV – Oedema volume, EI – Oedema inhibition.
Significant at $p < 0.05$, ** highly significant at $p<0.01$, *** Very highly significant at $p<0.001$

Evaluation of anti-inflammatory activity using carrageen an   induced rat paw edema mode [23]. Albino rats of either sex (150-200 g) were divided into different groups, containing six animals each. Animals were fasted for 12 h before experiment and only water was allowed. While the first group was a control one and received vehicle (Tween 80 in propylene glycol (10% v/v), 0.5 ml per rat), the second group received Diclofenac sodium (10 mg/kg). The entire remaining group received the test compounds at the same dose orally. All the suspensions for oral dose were prepared in the vehicle mentioned above and administered in a constant volume of 0.5 ml per rat.

CONCLUSION

Twelve   [6-(2-methoxy-phenyl)-2-oxo-4-phenyl/ substituted phenyl-1,2,3,4-   tetrahydropyrimidine-5-yl] acetic acid derivatives have been synthesized in a two-step reaction.

Their structures are confirmed by IR, ^{1}H NMR and TLC.The anti-inflammatory activity of all compounds has been recorded on the basis of reference standard indomethacin.All the compounds showed tendency to cause a fall in oedema and showed anti-inflammatory activity.The anti-inflammatory data shows that use of methoxy at [4]th position of phenyl in product shows increase in activity (c1-c4).

Research, Ale (M.S.) India for providing laboratory facility.

Conflicts of interest: The authors stated that no conflicts of interest.

REFERENCES

1) Holmes JT, Prostaglandins, Leukotrienes and other Eicosanoids in Wilson and Giswold's *Textbook of Organic Medicinal and Pharmaceutical Chemistry, 11th ed.; Block, J.H.; Beale, J.M., Eds; Lippincott-Williams and Wilkins, Philadelphia*, 2004,818-829.

2) Huskisson EC, In Antirheumatic drugs, clinical pharmacology and therapeutic series. *Praeger publishers*, 1983, 3,117-156.

3) Borne RF. Nonsteroidal Anti-inflammatory agents In Foye's Principles of Medicinal Chemistry, *5th ed.; Williams, D. A.; Lemke T.L., Lippincott Williams and Wilkins, New York*, 2002, 751-793.

4) Kadam. SS, Mahadik KR, Bothara KG. Nonsteroidal Anti-inflammatory agents: In Principles of Medicinal Chemistry, *15thedition, NiraliPrakashan, Pune*, 2002, 2, 138-139.

5) Wallace J.L., Bure A .G, GI-Sparing Anti-inflammatory Drugs: A Promising Future. www.gastro.org/user-assets/ documents/ 08-publications/06_GIHep-Annual Review /Articles/Wallace-Buret.pdf

6) Bhosale D, Bharambe S, Gairola N, Dhaneshwar SS, Mutual prodrug concept: Fundamentals and applications, *Ind.J.Pharm.Sci*, 2006,68, 3, 286-294.

7) Bartsch H, Nair J. Chronic inflammation and oxidative Stress I the genesis and perpetuation of cancer: role of lipid peroxidation, *DNA damage, and repair. Langenbecks Arch Surg* ,2006; 391: 499–510.

8) Lu H, Ouyang W, Huang C. Inflammation, a key event in cancer development. *Mol Cancer Res*, 2006; 4: 221–33.

9) Winter et al., Proc. *Soc. Exp. Bio. Med*., 111, 1962, 544.

10) Pirisino R., Bainchini F., Banchelli G. *et al*, 2-phenylpyrazolo-4-ethyl-4, 7-dihydro [1, 5-a] pyrimidine-7-one for its analgesic, *antipyretic and anti-inflammatory activities. Pharmacol. Res. Comm.*, 1996, 18, 241.

11) Modica M, Santagati M, Santagat A e*t al*, Some new Thiadiazolothieno-pyrimidinones for anti-inflammatory and analgesic activities A., *Pharmazie*, 2000, 55, 500.

12) Cenicola ML, Donnoli D, Stella L. *et al*. Someimidazolo [1, 2]- pyrimidines for anti-inflammatory, analgesic and antipyretic activities. *Pharmacol. Res.,* 1990, 22: 80

13) Nargund LV, Badiger VV and Yarnal SM, Substituted 2-mercapto-3-(N-alkyl) pyrimido [5, 4-c] cinnolin-4-(3H)-ones for anti-inflammatory and antimicrobial activities, *J. Pharm. Sci*., 1992, 81: 365.

14) Cottam HB, Wasson DB, Shikh HC. Pyrazolo [3, 4-d] pyrimidine derivatives as potential inhibitor of adenosine kinase et al, *J. Med. Chem.,* 1993, 36:3424.

15) Bruni F, Costanzo A, Selleri S, *et al*. Series of pyrazolo [1, 5-a] pyrimidin-7-one for anti-inflammatory activity, *J. Pharm. Sci.,* 1993, 82:480.

16) Tozkoparan B, Ertan M, Kelicen P. and Demirdamar, New 2-benzylidene-7-methyl-3-oxo-5-(substitutedphenyl)-2,3-dihydro-5H-thiazolo[3,2]pyrimidine-6-carboxylic acid esters for anti-inflammatory activity R., *IL Farmaco*, 1999, 54,:588.

17) Lee CH, Jiang M, Cowart M, *et al*. Some 6-substituted pyridopyrimidine analogues as potential AK inhibitors., *J. Med. Chem.,* 2001, 44:2133

18) Boyle DL, Kowluk EA, Jarvis MF, *et al. J. Pharmacol. Exp. Ther*, 2001, 296,:495

19) Molina P, Aller E, Lorengo A, *et al*. Pyrido [1, 2] pyrimidines on solid phase using the iminophosphorane methodology and tested for effects on leukocyte function in vitro and anti-inflammatory activity. *J. Med. Chem.*, 2001, 44:1011

20) Vidal A,Ferrandiz ML, Ubeda A, *et al*. Hexahydroimidazo [1, 2] pyrimidine derivatives on leukocyte functions *J. Pharm. Pharmacol.* 2001, 53: 1379.

21) Furniss BS, Hannaford AJ, Smith PWG, Tatchel AR. *Vogel's textbook of practical organic chemistry, 5th edition*, 1015-1016.

22) Sodhi Sham M, Dinodia Monica, Rani Resma, Shukla Rakesh. Synthesis, anti-inflammatory and analgesic activity of some pyrimidine derivatives, *Indian J. of Chemistry,*2009, Vol. 49B:273-281.

23) Winter CA, Risley EA, Nuss GW. Carrageenan-induced oedema in the hind paw of rat as an assay for anti-inflammatory activity. *ProcSoc. Exp. Biol. Ther.* ,1962, 111: 544-547.

RESEARCH ARTICLE　　　　　　　　　　　　　　　　　　　　　　**OPEN ACCESS**

Synthesis and characterization of Fe-doped TiO₂ Nanoparticles by modified sol-gel method

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ABSTRACT

Undoped and iron-doped TiO₂ nanoparticles ($Ti_{1-x}Fe_xO_2$ where x = 0.00 – 0.05) were synthesized by acid catalyzed sol–gel method. The synthesized products were characterized by sophisticated instrumental techniques like X-ray diffraction (XRD), transmission electron microscope (TEM) and ultraviolet –visible spectroscopy (UV-Vis-DRS) XRD pattern confirmed the tetragonal structure of synthesized materials. Average grain size was determined from X-ray line broadening using the Debye–Scherer relation. The crystallite size was found to be in the range 5.6 to 17.9 nm when calcined at 500 °C temperature. The doping of 1–5 mole% Fe into TiO₂ proved a great decrease in the size of nanocrystals as compared to undoped TiO₂. The TEM micrographs revealed the spherical-like morphology with average diameter of about 8 to10 nm which is in agreement with XRD results. UV-Vis-DRS clearly showed the shift in the absorption towards visible region of the spectrum.

Keywords: Sol-gel, XRD, TEM, UV-DRS.

INTRODUCTION

TiO₂ is well known for its widespread applications in paints, pigments, cosmetics, food stuffs, environmental treatment and purification purposes [1-5]. There is a particular current interest in the application of titanium dioxide (Titania) in the photocatalytic degradation of organic pollutants and in the photochemical water splitting,

Which are becoming vital importance due to environmental concerns and future energy need. Due to its wide energy band-gap (3.0-3.2 eV) effective utilization of solar energy is limited to about 3-5% of the total solar spectrum [6-10]. Doping of the semiconducting metal oxides [6-8] like CuO, NiO and Fe_2O_3 with low band gap energy is one of the most widely used way to reduce band gap energy (Red Shift) and to lower the recombination of photogenerated e^-/h^+ pairs. Among the various semiconducting metal oxides Fe^{3+} is suitable for doping with Titania because both of them have nearly same ionic radii [8] (Fe^{3+} = 0.690 Å and Ti^{4+} = 0.745 Å). Also iron is among ten most abundantly occurring chief elements from the earth crust. In its (+III) oxidation state it is having half field d^5 orbital acts as a charge carrier trap and inhibits the recombination of photogenerated e^-/h^+ pairs. Owing to this it enhances the photocatalytic activity of doped Titania. Efficient photocatalytic activity of Fe^{3+}-doped Titania is reported by many researchers[9-11] while according to some reports, photocatalytic activity of Titania doped with Fe^{3+}decreases as the dopant acts as a center for electron–hole recombination.[12] It very interesting to study the effect of concentration of Fe as a dopant on photocatalytic activity of Titania.[24] Different methodology are employed for synthesis of nanocrystalline, photocatalytic active Titania including hydrothermal, solvothermal, co-precipitation and sol–gel method.[11] Out of these hydrothermal/solvothermal methods are energy and time consuming, while sol-gel method is very simple, rapid, reproducible, fabricates nanoparticles with precise morphology and particle size[13].

In this article iron doped Titania nanoparticles were synthesized very simple surfactant free acid catalyzed modified sol-gel method and were characterized by various sophisticated instrumental techniques like XRD, TEM, EDX and UV-DRS.

METHODOLOGY

1. Chemicals
For the synthesis of undoped and iron-doped TiO_2 nanoparticles, the materials used were titanium (IV) butoxide [M = 340.76, Ti $(OC_4H_9)_4$], ferric nitrate nonahydrate [Fe $(NO_3)_3$ $9H_2O$)], obtained from Sigma-Aldrich. Isopropyl Alcohol [M =60, $(CH_3)_2CHOH$] and acetic acid were obtain from S.D Fine Chemicals and Ethyl alcohol [M=46, C_2H_5OH] from local distillery. The deionized water was used as a solvent.

2. Synthesis
Synthesis of TiO₂ nanoparticles
The bare Titania powder was synthesized by modified sol-gel method. In a typical experimental procedure 20 gram of titanium (IV) butoxide was slowly added to the solution containing 15 ml isopropanol and 10 ml glacial acetic acid. It was ultrasonically dispersed in ultrasonic bath for 30 minutes and was added to 50 ml solution containing acetic acid–water (1:3) mixture under constant magnetic stirring. The vigorous magnetic stirring was continued for next four hours. It was then dried in hot air oven at 80°C for 12 hours, calcined at 500°C in high temperature muffle furnace for 4 hours in static air atmosphere and it was labeled as PT.

Synthesis of Fe-doped TiO₂ nanoparticles
Fe-doped TiO_2 nanoparticles were synthesized by using sol-gel method in presence acetic acid as solvent. Calculated quantity (1.0, 3.0 and 5.0 mole %) of ferric nitrate hexahydrate were ultrasonically dispersed and dissolved in 50 ml glacial acetic acid - water (1:3) solution (Solution A). The required quantity of titanium (IV) butoxide was slowly added to a mixture of 15 ml isopropyl alcohol and 10 ml glacial acetic acid (Solution B). Then the solution A was slowly added into the solution B with vigorous magnetic stirring and ultrasonically dispersed for 60 min. The resultant solution was continuously stirred for next 4 hours, and then it was dried in oven at 80°C for 12 hours and then it was calcined at 500°C in high temperature muffle furnace for 4 hours. Resultant powders were labeled as x % FeT, where x % is the mole % Fe in TiO_2.

3. Characterization
The synthesized samples were characterized by various sophisticated Techniques. X-ray diffraction (XRD) patterns were carried out by using Philips X-ray diffractometer with diffraction angle 2θ in between 20 to 80° using Cu-Kα radiation of wavelength 1·54058 Å. The microscopic nanostructure and particle size was determined by using a CM-200

PHILIPS transmission electron microscope (TEM) at 200 kV (L= 600, λ = 0.0025 nm). The elemental analysis was carried out using X Flash 6I30 Bruker instrument. The absorption spectra were recorded using a double beam UV-Visible spectrophotometer, Shimadzu -2450, Japan.

RESULTS AND DISCUSSION

After calcination at 500°C, the resultant powders were analyzed to study their surface morphology and composition with the help of sophisticated analytical instrumental techniques.

X-Ray Diffraction

The XRD pattern of undoped and Fe- doped TiO_2 nanoparticles calcined at 500℃ is shown in Fig.1. The XRD peaks of all the synthesized samples were wide confirming nanocrystalline nature of the photocatalyst. The peak values located at 2θ(°) 25.2, 37.6, 48.0, 53.8, 54.9, 62.6, 68.7, 70.2 and 74.9 correspond to the Miller indices (101), (004), (200), (105), (210), (204), (214), (220) and (107) respectively, confirming formation of highly photoactive tetragonal anatase Titania. All the diffraction peaks obtained from XRD agreed with the reported JCPDS card no. 21-1272 for tetragonal anatase Titania. No distinct peaks corresponding to rutile phase or dopant were seen in the X-ray diffractograms, which may be due to the proper incorporation of Fe^{3+} ions into the TiO_2 lattice. The crystallite sizes were estimated with the help of Scherer's equation, by using the most intense reflection ($2\theta^\circ = 25.2$) and were found to be in the range of 5.6 to 17.9 nm (Table 1).

Table 1: Physical parameters of iron doped and undoped Titania nanoparticles obtained from X– Ray Diffraction and Diffuse Reflectance Spectroscopy.

Sr. No.	Catalyst	Crystallite size, (nm)	Band gap energy, (eV)
1.	PT	17.9	3.12
2.	1% FeT	7.0	2.7
3.	3% FeT	5.9	2.5
4.	5% FeT	5.6	2.4
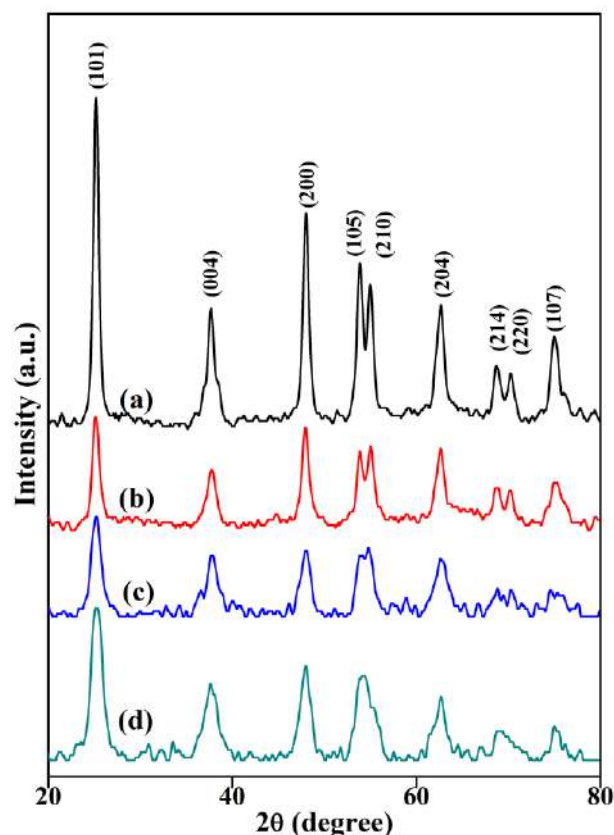
Fig.1. XRD patterns of (a) PT, (b) 1% FeT, (c) 3% FeT, and (d) 5% FeT calcined at 500℃.

Transmission Electron Microscopy (TEM)

The surface morphology of synthesized nanomaterial was studied by TEM analysis, Fig.2 show the TEM image of 1% FeT nanomaterials calcined at 500°C. From TEM analysis, it is clear that the synthesized nanoparticles are having spherical like morphology (Fig. 2 (a-b)) with excellent crystalline nature (Fig.2(c)). The SAED pattern confirms the presence of pure anatase phase (Fig. 2 (d)) which clearly supported XRD analysis. The d-spacing was indexed with the JCPDS card No 21-1272 corresponding to the anatase phase. The grain sizes were calculated from TEM analysis and were found in the range between 8 to10 nm; thus, results obtained from TEM are in good agreements with XRD analysis.

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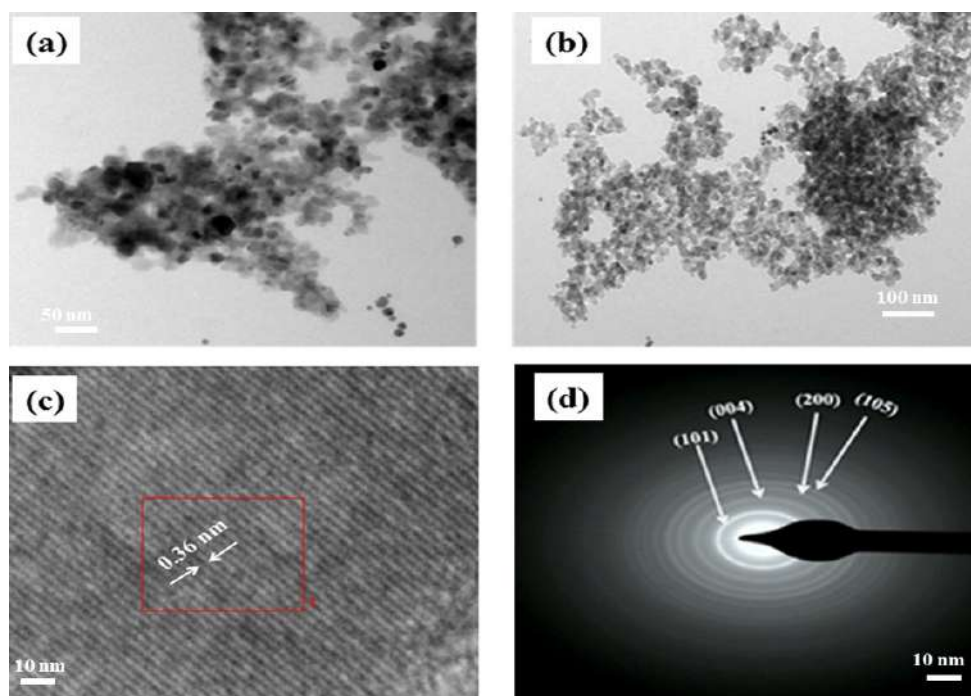
Fig.2. Typical TEM and HRTEM micrographs of synthesized nanoparticles, (a, b) TEM images at different magnifications; (c) HR-TEM image, and (d) SAED pattern of 1 % FeT calcined at 500℃.
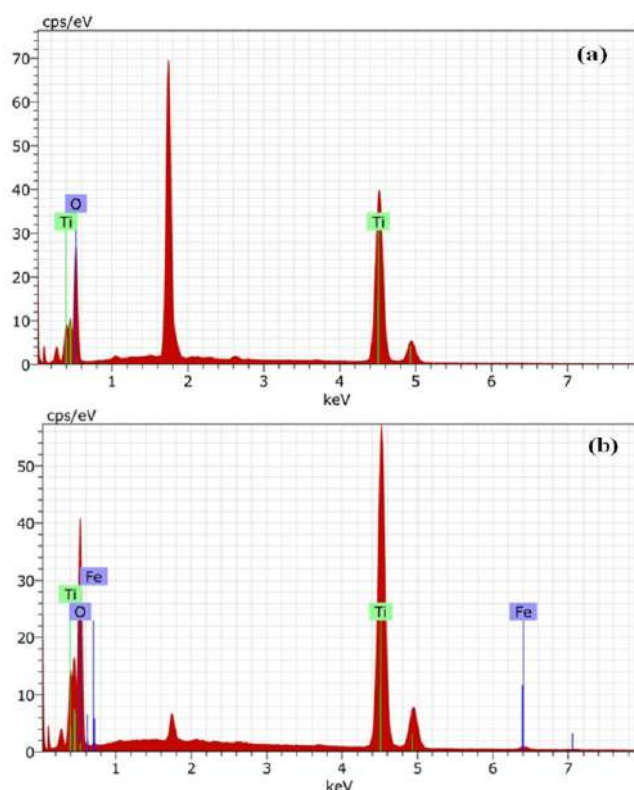
Fig. 3: EDX patterns of (a) PT, and (b) 1% FeT.

Ultra-Violet Visible Diffuse Reflectance Spectroscopy (UV-Vis DRS)

In order to study optical absorption properties, all the synthesized nanomaterials were analyzed by UV-**Energy Dispersive X-Ray Spectroscopy (EDX)**

Elemental analysis of Fe^{3+}-doped and bare Titania was carried out using EDX technique. The EDX patterns for PT and 1% FeT (Fig. 3) confirms the presence of proper proportion of Fe with respective Ti in doped samples and iron doping leads the oxygen deficiencies in the samples

Visible DRS technique in the absorption range of 200 nm to 800 nm and the results are shown in the Fig.4. The bare Titania shows the absorption at 404 nm with band gap energy 3.1 eV, which is near the band gap energy of the anatase Titania (∼3.2 eV). While significant enhancement in the absorption edge was observed for all doped samples indicating red shift in the absorption of wavelength in between 400–520 nm. The colour of the doped samples changed from white to yellowish brown with increase in the dopant concentration. The doping of transition metal ions like Fe^{3+} ions does no modify the position of valance band edge of Titania but it introduces new energy levels into the band gap of Titania. Thus, the dopant energy levels in between valance band and conduction band shifts the absorption edge towards longer wavelength

resulting in the decrease of band gap energy. The band gap energy values were calculated by extrapolation of the absorption band to the x-axis using the following equation,

$$E_{bg} = 1240/\lambda$$

Where, λ is the wavelength in nanometer and E_{bg} is the band gap energy. The calculated band gap energy values were, in the range of 2.4 to 3.1 eV (Table 1).
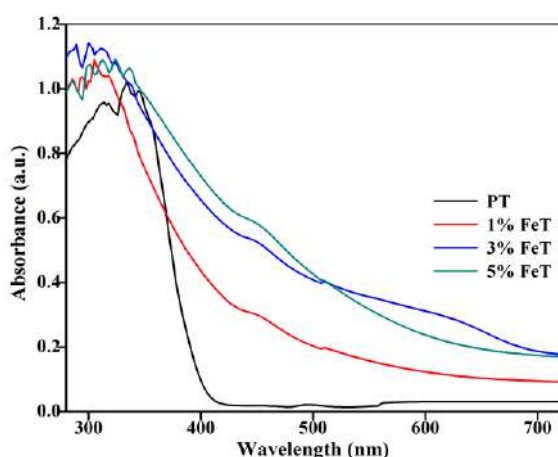
Fig.4. UV–vis DRS spectra of bare TiO_2 and Fe^{3+}-doped TiO_2 nanoparticles.

The enhanced visible light absorptions may be explained by the charge transfer transition between the d electrons of the Fe dopant and the conduction band of TiO_2. These results suggest that iron was incorporated into the crystalline network of TiO_2. The ionic radius of Fe^{3+}(0.69A°) and Ti^{4+}(0.745A°) are similar, and Fe^{3+} ions potentially substitute Ti^{4+} in the structure of TiO_2, which would in turn introduce a new impurity level into the band gap of TiO_2 and reduce the forbidden energy gap of TiO_2.

CONCLUSION

Modified sol-gel synthetic route results the formation nanocrystalline anatase TiO_2 polymorphs with decreased in grain size (17.9 to 5.6 nm) upon iron doping was confirmed by powder XRD.TEM and HRTEM confirm the formation of nanocrystalline Titania with spherical like morphology. The SAED pattern depicts diffraction peaks corresponding to pure anatase polymorphic phase of Titania. The results of TEM and XRD strongly supports to each other. Doping of iron in the Titania lattice was

confirmed from EDX. The red shift in the absorption maxima due the iron doping was observed from UV-Vis DRS results.

Above results clearly confirms that synthesized nanomaterials may further used for environmental remedies.

Conflicts of interest: The authors stated that no conflicts of interest.

REFERENCES

1. Matthews Peter D, King Timothy C and Wright Dominic S.. *Chem. Commun.*, 2014,**50:** 12815.

2. Auvinen J and Wirtanen L. *Atmos.Environ.*,2008, **42:** 4101.

3. Maria Eugenia Carlotti, Elena Ugazio, Lucia Gastaldi, Simona Sapinoa, Davide Vione, Ivana Fenoglio and Bice Fubini. *J. Photochem.Photobiol.*,2009, B **96:**130.

4. Y.Liao and W.Que *J.Alloys Compd.*, 2010,**505:** 243.

5. Mohamed RM and Mkhalid IA. *J. Alloys Compd.*,2010,**501:**143.

6. Molea A, Popescu V, Rowson NA, Cojocaru I, Dinescu A, Dehelean A, and Lazar M. *Ind. Eng. Chem. Res.*,2015, **54:** 7346.

7. Tayade RJ, Kulkarni RG, Jasra RV. *Ind. Eng. Chem. Res.*, 2006,**45:** 5231.

8. Barakat MA, Kumar R. P. A. Enhancement, "Photocatalytic Activity Enhancement of Titanium Dioxide Nanoparticles," *Springer International Publishing,* 2016, pp. 1–29.

9. Sood S, Umar A, Mehta SK, Kansal SK. *J. Colloid Interface Sci.*,2015, **450:** 213

10. Liu W, Zhao X, Borthwick AGL, Wang Y. *J. Ni, ACS Appl. Mater. Interfaces.*, 2015,**7:** 19726.

11. Mohsen M , Yousef M. *J. Mater. Sci. Mater. Electron.*,2015, **26:** 5509.

12. Wang W, Serp P, Kalck P, Faria JL, *Appl. Catal. B Environ.*,2005, **56:** 305.

13. Serpone N, Lawless D, Disdier J, Herrmann JM, *Langmuir.*,1994,**10:**643.

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RESEARCH ARTICLE   **OPEN ACCESS**

Formulation and Evaluation of Luliconazole Emulgel for Topical Drug Delivery

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ABSTRACT

The aim of the present study was to develop an emulgel formulation of Luliconazole using carbopol 934 as a gelling agent. The Luliconazole has anti-Fungal activity. It acts by inhibiting lanosterol demethylase, which is major component of fungus cell wall. Emulgel has emerged as a promising drug delivery system for the delivery of hydrophobic drugs. The prepared emulgel were also evaluated for their physical properties, pH, drug content and rheological properties. Candida albicans was used as a model fungus to evaluate the antifungal activity of the prepared formulations. Stability studies revealed no significant differences in formulation.

It was concluded that Luliconazole emulgel formulation (F3) prepared by using Carbapol 934 as gelling agent, emulsifying agent in its high level and liquid paraffin in its low level was the choice of formula, since it showed the highest drug release and antifungal activity.

Keywords: Emulgel, Luliconazole, Topical drug delivery.

INTRODUCTION

Topical formulations apply a wide spectrum of preparations both cosmetic and dermatological, to healthy or diseased skin [1]. These formulations range in consistency from solid through semisolid to liquids. When gels and emulsions are used in a combined form, the dosage forms are referred to emulgel. [2, 3]As the name suggests they are the combination of emulsion

micro-emulsion and gel. Novel polymers with complex fuctions as emulsifires and thickeners have been widely used due to their gelling capacity which allows the formulation of stable emulsion by decreasing surface and interfacial tension and also by increasing the viscosity of the aqueous phase. Oil/water and water/oil emulsions are used as vehicles to deliver various drugs to the skin [4]. Emulsion gels are has importance due to many reasons; they have better application property in comparison to classical formulation as creams and ointment, they have faster and more complete release of the drug from the vehicle to the skin, also they are convenient to apply on hairy skin due to the absence of greasiness and lack of residue upon application. They permit the incorporation of both aqueous and oleaginous ingredients, so hydrophobic or poorly water soluble drugs as antifungal agents are easily incorporated in such type of vehicles through the proper choice of the oily phase [5].
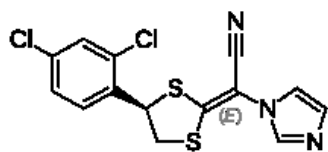
Fig. 1: Structure of Luliconazole

Luliconazole has anti-fungal activity. Luliconazole is inhibiting the enzyme lanosterol demethylase. Lanosterol demethylase is needed for the synthesis of ergo-sterol, which is a major component of the fungus cell membranes. For skin care and the topical treatment of dermatological diseases, a wide choice of vehicles including solid, semisolids and liquid Preparation is available to physician and patients. Within the major groups of semisolid preparations, the use of transparent emulgel has expanded, both in cosmetics and pharmaceuticals. Emulgel or jellified emulsion is stable one and better vehicle for hydrophobic or water insoluble drugs as Luliconazole. Also emulgel has a high patient acceptability since they possess the advantages of both emulsions and gels. Therefore, they have been recently used as vehicles to deliver various drugs to the skin. [6-7]

METHODOLOGY

Materials:
Luliconazole was obtained as a gift sample from A. S. Life Science, Haryana, India. Carbopol 940 was obtained from Loba chemicals Mumbai. Liquid paraffin, propylene glycol, ethanol was procured from Naprod life science, Mumbai. Methyl parabens and propyl parabens procured from Chem. Pure pvt.ltd. Mumbai. All other chemicals used were of analytical grade and were used without any further chemical modification.

Method:
Preparation of Luliconazolee Emulgels:
Emulgel was prepared using carbopol 940, as gelling agents (Table 1). The gels in formulations were prepared by dispersing carbopol in purified water with constant stirring at a moderate speed and then the pH are adjusted to around 6 using tri-ethanol amine. The oil phase of the emulsion was prepared by dissolving span 20 in light liquid paraffin while the aqueous phase was prepared by dissolving tween 20 in purified water. Methyl and propyl parabens were dissolved in propylene glycol whereas drug was dissolved in ethanol and both solutions were mixed with the aqueous phase. Both the oily and aqueous phases were separately heated to 70° to 80°C; then the oily phase were added to the aqueous phase with continuous stirring until cooled to room temperature. Finally the emulgel was prepared by mixing the both gel and emulsion in 1:1 ratio. The composition of different formulations has been discussed in **Table 1**.

Table 1: Composition of different formulation batches (%w/w).

Ingredient	F1	F2	F3	F4
Luliconazole	1	1	1	1
Carbapol 940	1	1	1	1
Liquid paraffin	5	5	7.5	7.5
Tween 20	0.5	1	0.5	1
Span 20	1	1.5	1	1.5
Propylene glycol	5	5	5	5
Ethanol	2.5	2.5	2.5	2.5
Methyl parabene	0.03	0.03	0.03	0.03
Ethyl parabene	0.01	0.01	0.01	0.01
Purified water	q.s	q.s	q.s	q.s

Evaluation of Emulgel:

1. Physical Appearance and pH Determination:
The prepared Luliconazole emulgel were inspected visually for their colour, homogeneity, Consistency and pH. The pH values of 1% aqueous solutions of the prepared emulgels were measured by a pH meter (Orion Research, Inc., USA). [12]

2. Drug Content Determination:
The drug content of Luliconazolee emulgel was measured by dissolving a known weight of the emulgel formulation (one gram) in 100 ml methanol, appropriate dilutions were made and the resulting solution was then filtering using millipore filter (0.45 µm). Absorbance was measured at 296 nm using UV-spectrophotometer (Shimadzu UV 1800). [11] Drug content was calculated using the slope and the intercept obtained by linear regression analysis of standard calibration curve.

3. Rheological Studies:
The viscosity of different Luliconazole emulgel formulations was determined at 25˚C using a Brook-filled Viscometer. Viscosity was measured by using spindle (52).

4. Skin Irritation Test (Patch Test):
A set of 8 rats was used in the study. The emulgel was applied on the properly shaven skin of rat. Undesirable skin changes, i.e., change in colour, change in skin morphology were checked for a period of 24 h.

5. Spreading Coefficient:
Spreading coefficient was determined by apparatus suggested by Mutimer. It consists of a wooden block, which is attached to a pulley at one end. Spreading coefficient was measured on the basis of 'Slip' and 'Drag' characteristics of emulgels. A ground glass slide was fixed on the wooden block. An excess of emulgel (about 2 g) under study was placed on this ground slide. The emulgel preparation was then sandwiched between this slide and second glass slide having same dimension as that of the fixed ground slide. The second glass slide is provided with the hook. Weight of 500 mg was placed on the top of the two slides for 5 min to expel air and to provide a uniform film of the emulgel between the two slides. Measured quantity of weight was placed in the pan attached to the pulley with the help of hook. The time (in s) required by the top slide to cover a distance of 5 cm was noted. A shorter interval indicates better spreading coefficient.

6. *In-Vitro* Release Studies:
The study was carried out using the modified USP apparatus type II (Hanson SR8-plus 80, USA). Two grams of each emulgel was spread on the cellophane membrane previously soaked overnight in the dissolution medium. The loaded membrane was stretched over a glass cup of diameter 3 cm, and then the cup was immersed in 100 ml of the dissolution medium (25%v/v DMF in 0.02N HCl) to maintain sink condition, the temperature was maintained at 37±0.5°C with paddle agitation speed 50 rpm. An aliquot of 5 ml was withdrawn at different intervals of time. The withdrawn samples were replaced by equal volumes of fresh release medium. The samples were assayed using spectrophotometer at λ max 296 nm. The effect of gelling, the liquid paraffin concentration and emulsifying agent concentration was studied. [5].

7. Antifungal Activity Studies: The prepared emulgel formulations were tested against candida albican strain using agar cup method. Cups of 10mm diameter were made aseptically in savoured dextrose agar after being inoculated with the tested fungal suspension strain (106cfu/ml) by spreading on the agar surface..

Table 2: Physical parameter of formulation batches.

Formulation	Colour	Homogeneity	Consistency	Phase separation
F1	White	Excellent	Excellent	None
F2	White	Excellent	Excellent	None
F3	White	Excellent	Excellent	None
F4	White	Excellent	Excellent	None

The cups were filled with each prepared formulation by sterile syringe. The zone of inhibition of each cup was observed and the radius of the zone of inhibition was measured. [17]

8. Stability Studies:

The prepared Luliconazole emulgel were packed in aluminium tubes (5 grams) and subjected to stability studies at 25°C/60% relative humidity (RH) and 40°C/75% RH for period of 3 months. Samples were withdrawn at time intervals of 15 days and evaluated for physical appearance, pH, rheological properties, drug content and drug release. [18]

RESULTS AND DISCUSSION

Physical Appearance:

Emulgel formulations were white viscous creamy preparation with a smooth homogeneous texture and glossy appearance. Results have been discussed in **Table 2**.

1. pH Determination:

pH of Prepared Emulgel were measured by using pH meter (Orion Research, Inc., USA).

The pH of the emulgel formulation was in the range of 5.76-6.236 which considered acceptable to avoid the risk of skin irritation upon application to skin.

Table 3: pH of emulgel formulation

Formulation	F1	F2	F3	F4
pH	5.83	5.76	6.19	6.23

2. Spreading Coefficient:

The spreading coefficient of various emulgel formulations are given below in **Table 4.** It was concluded that all the developed formulation showed acceptable spread ability, **F3** formulation has more spread ability as compare to other formulation i.e**.** 24.2±0.2.

Table 4: Spreading coefficient of the formulation F1–F4(mean ± SD).

Formulation	F1	F2	F3	F4
Spread ability (gm.cm/sec.)	23.7±0.2	21.4±0.1	24.2±0.2	21.5±0.4

3. Drug Content:

The drug content of different emulgel formulations was estimated by using UV Spectrophotometer at 200-400 nm range. The release of drug through prepared formulation was found to be 96.82, 97.65, 98.25and 98.06 respectively.

Formulation	F1	F2	F3	F4
% Drug content	96.82	97.65	98.25	98.06

Table 5 :Drug content of Luliconazole emulgel Formulation.

4. Skin Irritation Test:

Skin irritation study was performed. No allergic symptoms like inflammation, redness, irritation appeared on rats up to 24 h.

5. Rheological Studies:

The tests were performed by using Brook-field Viscometer. Results are given in Table 6, highest viscosity was found in formulation F3. It may be due to high level of the liquid paraffin concentration and low level of emulsifying agent concentration.

Viscosity (mPas)				
RPM	F1	F2	F3	F4
10	3349±0.54	3371±0.98	3671±0.65	3584±0.75

Table 6: Rheological study of emulgel formulation (mean± SD, n =3)

6. Antifungal Activity:

The antifungal activity of Luliconazole emulgel was studied (**Table 7)**. The zone of inhibition was measure for antifungal activity of drug. The greatest activity was observed in **F3** formulation i.e. 49.5mm and the lowest activity were found with F1.

Table 7: antifungal activity of Luliconazole emulgel

Inhibition zone (mm)± SD	FORMULATION			
	F1	F2	F3	F4
	40.1	43.7	49.5	46.8

7. *In-Vitro* Release Study:

The study revel that, the release of the drugs from emulsified gel formulation can be ranked in the following descending order: F3 > F4 > F1> F2 where the amounts of drug release after 240 min were 90.12%, 83.58%, 79.50%, 71.98% respectively **(Fig. 2, Table 8).**

Table 8: Data for in vitro cumulative % drug release data of formulations F1–F4.

Time (min)	F1	F2	F3	F4
0	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00
5	08.90 ± 0.010	11.02 ± 8.65	11.82 ± 6.83	14.55 ± 30.44
10	12.20 ± 0.05	14.92 ± 9.64	26.54 ± 13.30	20.76 ± 10.20
15	20.78 ± 1.30	23.43 ± 6.83	35.44 ± 10.2	28.24 ± 15.70
20	31.70 ± 2.25	32.04 ± 10.2	41.74 ± 0.03	36.75 ± 0.020
30	38.10 ± 31.70	40.95 ± 24.9	52.14 ± 0.38	41.84 ± 0.03
60	48.23 ± 2.85	53.42 ± 2.31	63.25 ± 0.49	56.01 ± 2.30
120	62.59±1.25	65.86±2.28	76.12±0.47	68.25±1.374
240	71.98%±0.26	79.50%±0.39	90.12%±1.41	83.58%±0.65
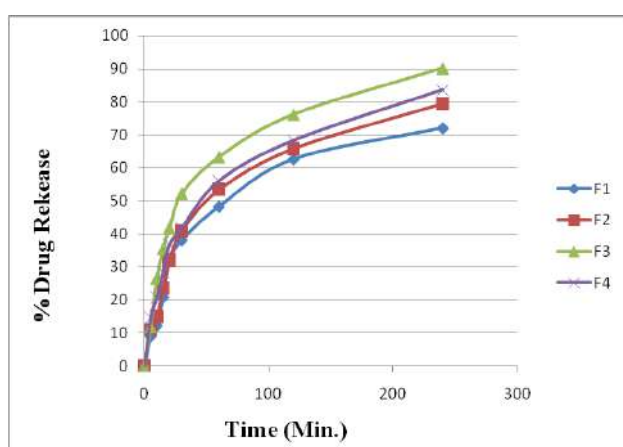
Fig. 2: *In-vitro* **cumulative % drug release of formulation F1–F4**

acceptable physical properties, pH, drug content, viscosity and antifungal activity. Stability studies revealed no significant differences before and after storage for the selected formula. *In vitro* releases of emulgel were also performed to determine drug release from emulgel and duration of drug release. From the in vitro studies, formulation F3 showed maximum release of 90.12% in 240 min. So Luliconazole emulgel can be used as an anti-Fungal agent for topical drug delivery.

Conflicts of interest: The authors stated that no conflicts of interest.

8.   Stability Study:

All the prepared emulgel formulations were found to be stable upon storage for 3 months, no change was observed in their physical appearance, pH, rheological properties and drug content.

CONCLUSION

In the coming years, topical drug delivery will be used extensively to impart better patient compliance. Since emulgel is helpful in enhancing spreadability, adhesion, viscosity and extrusion, this novel drug delivery become popular. Moreover, they will become a solution for loading hydrophobic drugs in water soluble gel bases for the long term stability.

In present investigation topical Luliconazole emulgel was prepared by using carbopol 934 showed

REFERENCES

1. Lawrence HB. Medicated Topicals. Ch.44 in Remington. In: Lippincott Williams and Wilkins. Editors the science and practice of pharmacy 21th ed. Philadelphia; 2006.

2. Mohamed MI. Topical emulsion- gel composition comprising Diclofenac sodium. AAPS Journal, 6(3), 2004.

3. Mohamed MI. Optimization of chlorphenesin emulgel formulation. AAPS Journal, 6(26), 2004.

4. Rieger MM, Lachman L, Lieberman HA. Editors. The theory and the practice of industrial pharmacy 3rd ed. Philadelphia; 1986.

5. Shahin M, Abdel HS, Hammad M, Mortada N. Drug development and industrial pharmacy,37(5):559-568, 2011.

6. British Pharmacopoeia. Appendix ID: A 143(4), 2008.

7. Steven P. Anti- infective. Ch. 90 in Remington. In: Lippincott Williams and Wilkins, editors. Philadelphia: The science and practice of pharmacy 21 th ed.; 2006.

8. Maryadele JO. The Merk Index. Editor. An encyclopedia of Chemicals, USA: Drug sand Biological.14 th ed. NJ, 2006.

9. Howard CA, Loyd V, Allen JR, Nicholns GP. In: Ansel's Pharmaceutical DosageForms and Drug Delivery Systems 8th ed. Lippincott Williams and Wilkins; 2005.

10. Masar BM. Formulation and evaluation of meloxicam as a topical preparation thesis. collage of pharmacy, University of Baghdad; 2004.

11. Monica R, Girish S, Sheetal A, Manmeet K. Optimization of Metronidazole emulgel: a Conceptual framework. Journal of pharmaceutics; 2012.

12. Shalaby S, Abd El-Aal S. Formulation and stability of chloramphenicol gel and emulgel. Bull Fac Pharm,39:89-99, 2001.

13. Costa P, Manuel J. Modeling and comparison of dissolution profiles. Eur. J. Pharm. Sci., 13:123-133, 2001.

14. Kabir A, Biswas B, Rouf A. Design, fabrication and evaluation of drug release kinetics From aceclofenanc matrix tablet using HPMC. Uni. J. Pharm. Sci., 8:23-30, 2009.

16. Gohel M, Panchal M, Jogani V. Novel mathematical method for quantitative expression of deviation from the higuchi model. AAPS Pharm Sci. Tech.,1:1-6, 2000.

17. Helal D, Rhman D, Abdel S, Nabarawi M. Formulation and evaluation of fluconazole topical gel. Int. J. of pharmacy and pharmaceutical Sci., 4(5), 2012. Available: http://www.ijpps. Journal.com/vol. 4 supp5/4593.pdf.

18. ICH Harmonized Tripartite Guidelines, Stability Testing of New Drug Substances and Products. ICH Committee; 2003.

RESEARCH ARTICLE **OPEN ACCESS**

Studies on sensing properties of nanostructured Zn$_2$TiO$_4$

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ABSTRACT

Zn$_2$TiO$_4$ is functional material having wide applications like sensors, solid oxide fuel cell, thermoelectric material and regenerable catalyst. Hence we have synthesized Zn$_2$TiO$_4$ by combustion method at 650 ^{0}C. The as synthesized compounds were characterized by UV-DRS and XRD techniques. The gas sensing properties as synthesized compounds were tested for NH$_3$, H$_2$S, Ethanol, CO$_2$, Cl$_2$, CO, H$_2$, and LPG. Nanostructured Zn$_2$TiO$_4$ showed maximum sensing response for NH$_3$ gas at 300 ^{0}C (23.36 ppm) The sensing performance was tested using static gas system.

Keywords Combustion method, Gas sensors, NH$_3$ and H$_2$S.

INTRODUCTION

Metal oxides gas sensors are widely used because of their high sensitivity, fast response, low cost stability and accuracy but selectivity is very limited. Zinc titanates has been applied in many fields such as paint, pigments, sensors, photocatalysis etc [1,2]. ZnTiO$_3$ has been applied as gas sensor but use of Zn$_2$TiO$_4$ as gas sensors has been less explored. In this work it has been attempted to synthesize Zn$_2$TiO$_4$. As synthesized were characterized by UV-DRS, IR, XRD, FESEM and TEM techniques. The gas sensing properties as synthesized compounds were tested for NH$_3$, H$_2$S, Ethanol, CO$_2$, Cl$_2$ CO H$_2$, and LPG.

METHODOLOGY

Synthesis of Intermediate for synthesis of Zn₂TiO₄:

Aqueous solution of 0.03 mole (6.5847 g) Zinc acetate and 0.015 mole (5.3118 g) Potassium Titanium oxalate were prepared. Then 30 ml 30 % 20 vol H_2O_2 was added into aqueous solution of potassium titanium oxalate. To the orange color solution formed Zinc acetate solution was added drop wise, yellow precipitate formed during addition of zinc acetate solution. Decolorization of solution part observed after stirring mixture for 6 h which ensures the completion of the reaction . Product obtained was filtered and washed with hot water followed by methanol and kept under IR lamp for 3hr. Then yellow product obtained was dried at 120 ^{0}C for 3 hr.

Synthesis of Zn2TiO4 **by combustion method**.

2.45g intermediate, 1.2 g urea, 3.2 g ammonium nitrate , 4.5 g glycene and 3 g starch are mixed uniformly to form paste. Paste formed was added stepwise into combustion tube at 600 ^{0}C. Burning takes place followed by formation of white residue the bottom. Heating further continued for 2 hrs.

Characterization:

As synthesized intermediates and Zinc titanates were characterized by UV-DRS (Perkin Elmer), XRD (Bruker, D-8)

XRD pattern depicted in figure 1 reveals that as synthesized compound possess tetragonal crystal structure (JCPDS 2002, 86-0158, a= 6.000 A° , b= 8.415 A°)
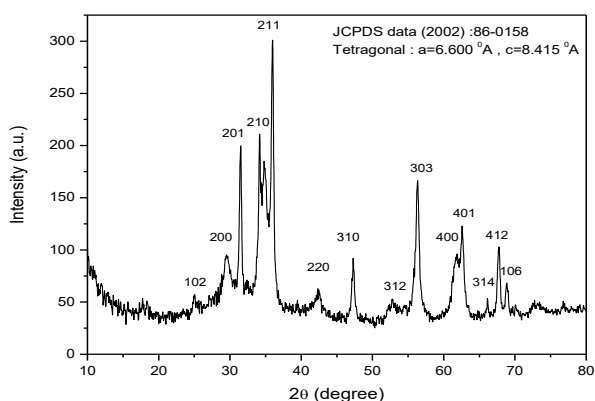
Figure 1: XRD pattern of nanostructured Zn_2TiO_4

UV-DRS absorption spectral study (Figure 2) indicates that band gap of as synthesized nanostructured Zn_2TiO_4 is having band gap 3.19 eV.
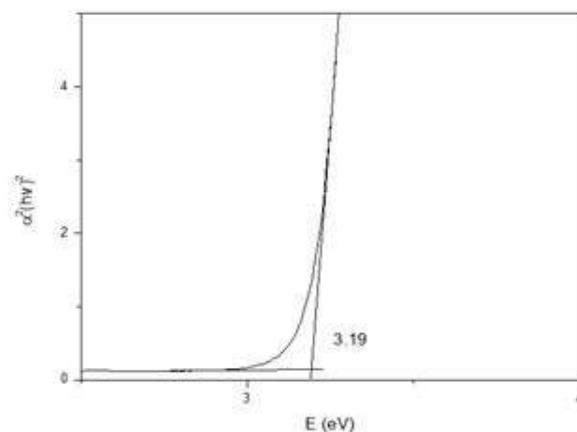
Figure 2: UV-DRS absorption Tauc plot of Zn_2TiO_4

Preparation of Zn₂TiO₄ thick film

The thixotropic paste was formulated by mixing the fine powder of Zn_2TiO_4 with a solution of ethyl cellulose (a temporary binder) in a mixture of organic solvents such as butyl cellulose, butyl carbitol acetate, terpineol, etc. The ratio of the inorganic to organic part was kept at 75:25 in formulating the paste. This paste was screen-printed[1] on glass substrate in the desired pattern. The films were fired at 550 $^{\circ}$C for 30 min.

Gas sensing properties of Zn₂TiO₄ thick film

The sensing performance of the sensors was examined using a 'static gas sensing system' explained elsewhere [3].

RESULTS AND DISCUSSION

Basic gas sensing characteristics

The basic gas sensing characteristics of Zn_2TiO_4 thick film was investigated as a function of operating temperature and test gas concentration. In the present studies the films were characterized by various parameters such as sensitivity, selectivity and response and recovery time.

The sensitivity (*S*) is defined as

$$S = \frac{(Ra - Rg)}{Ra} = \frac{\Delta R}{Ra}$$

where R_g is the resistance in presence of test gas and R_a is the film resistance in dry air, measured at respective temperatures [4,5] . A positive value of S implies film resistance decreases on gas exposure and vice versa.

The selectivity or specificity of a sensor towards an analyzing gas is expressed in terms of dimension that compares the concentration of the corresponding interfering gas that produces the same sensor signal. This factor[5] is obtained by

Selectivity = (Sensitivity of the sensor for interfering gas)/(Sensitivity towards   the desired gas)

The response time is the time interval over which resistance attains a fixed percentage (usually 90%) of final value when the sensor is exposed to full scale concentration of the gas. A small value of response time is indicative a good sensor.

The recovery time is the time interval, over which resistance reduces to 10% of the saturation value when the sensor is exposed to full scale concentration of the gas and then placed in the clean air. A good sensor should have a small recovery time so that sensor can be used again and again.

Sensitivity of Zn_2TiO_4 thick film to various gases with operating   temperature

Figure 3 shows the variation of sensitivity of the Zn_2TiO_4 thick film to various gases (800 ppm) with operating temperature ranging from 50 to 450 °C. For NH_3, the sensitivity goes on increasing with operating temperature, attains its maximum (23.36 ppm) at 300 °C and then decreases with a further increase in operating temperature. It is clear from the figure that, the sensor gives the maximum sensitivity to to H_2S (11.87 ppm) at 350 °C and Cl_2 (9) at 300 °C. The sensor selects a particular gas at a particular temperature. Thus by setting the temperature, one can use the sensor for particular gas detection. The same sensor could be used for the detection of different gases by operating it at particular temperature for a typical gas. This can be attributed to different chemical re-activities of different gases on the sensor surface. Different gases have different energies for adsorption, desorption and reaction on the metal oxide surface, and therefore the response of the sensor at different temperatures would depend on the gas being sensed.

The amount of oxygen adsorbed (O_2^-, O^-, O^{2-}) on the sensor surface goes on increasing with an increase in temperature, reaches to the maximum and then decreases with a further increase in operating temperature. The response to the gas to be detected follows the same behavior. When a reducing gas comes in contact with the sensor surface, it gets oxidized. The rate of oxidation would be the function of the amount of adsorbed oxygen on the surface and the type of gas to be detected. The larger the rate of oxidation, the larger would be the number of electrons released, and in turn the larger would be the gas response. At higher temperatures (beyond about 300 °C), the amount of oxygen adsorbed would be smaller, leading to a slower rate of reduction of a target gas and, therefore, the smaller gas response.
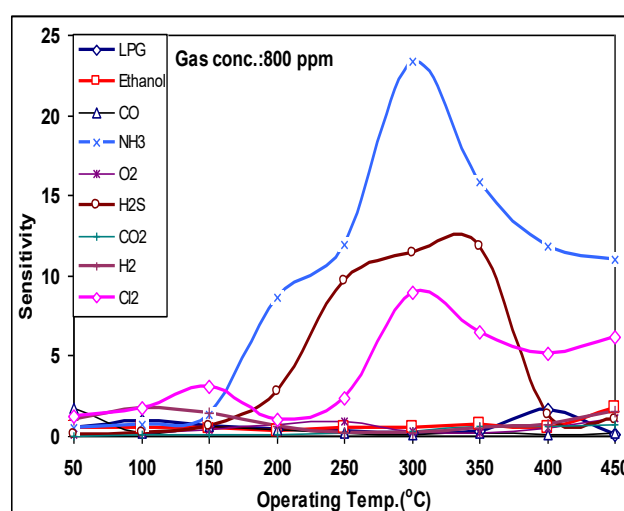
Figure.3 Variation of sensitivity with operating temperature.

Variation in sensitivity with NH_3 gas concentration

The dependence of the sensitivity of the Zn_2TiO_4 thick film on the H_2S concentration at an operating temperature 300⁰C is shown in figure 4. It is observed that the sensitivity increases linearly as the NH_3 concentration increases from 100 to 800 ppm and then decreases with further increase in the H_2S concentration. The linear relationship between the sensitivity and the NH_3 concentration at low concentrations may be attributed to the availability of sufficient number of sensing sites on the film to act upon the NH_3. The low gas concentration implies a lower surface coverage of gas molecules, resulting into lower surface reaction between the surface adsorbed

oxygen species and the gas molecules. The increase in the gas concentration increases the surface reaction due to a large surface coverage. Further increase in the surface reaction will be gradual when saturation of the surface coverage of gas molecules is reached. Thus, the maximum sensitivity was obtained at an operating temperature of 300 °C for the exposure of 800 ppm of NH_3. The linearity of the sensitivity in the low NH_3 concentration range (100-800 ppm) suggests that the Zn_2TiO_4 thick film can be reliably used to monitor the concentration of NH_3 over this range.
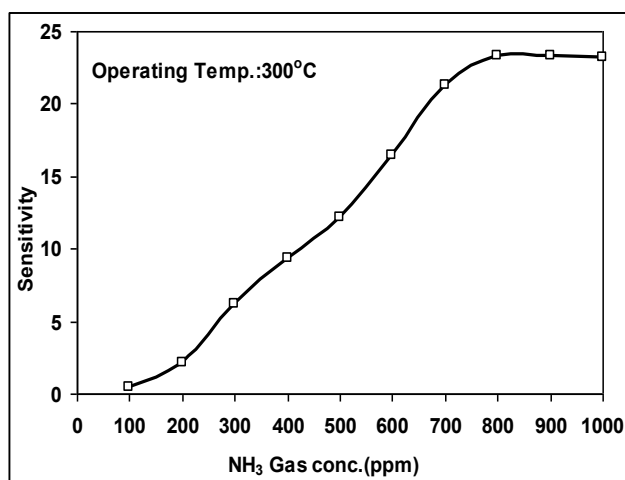
Fig. 4: Dependence of the sensitivity of the Zn_2TiO_4 thick film on the NH_3 concentration at 300 °C

Selectivity of Zn_2TiO_4 thick film for various gases
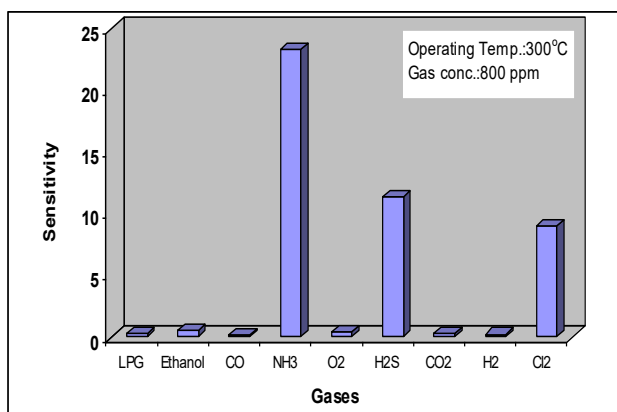
Figure 5. Selectivity of Zn_2TiO_4 thick film for various gases.

It is observed from figure 5 that the Zn_2TiO_4 thick film gives maximum sensitivity to NH_3 (800 ppm) at 300 °C. The films showed highest selectivity for NH_3 against all other tested gases: H_2S, LPG, Cl_2, CO, CO_2, O_2, H_2 and ethanol.

Response time and recovery time
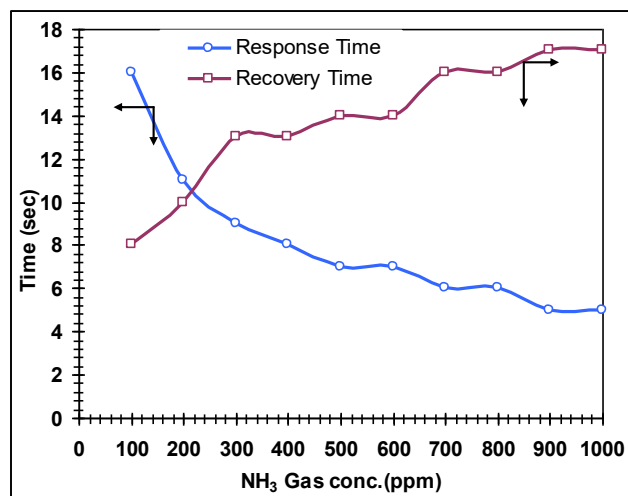
Figure 6. Variation of response and recovery time with concentration of NH_3.

Figure 6 shows the response and recovery time vs concentration of NH_3. It is revealed that response time decreased from 16 sec to 5 sec when NH_3 concentration is increased from 100 ppm to 1000 ppm. This may be due to the presence of sufficient gas molecules at the interface for reaction to occur. From the same graph, it is found that for higher concentrations of NH_3, the recovery time was long. This may be due to the reaction products are not leaving from the interface immediately after the reaction.

Gas sensing mechanism

It is known that atmospheric oxygen molecules are adsorbed on the surface of n-type semiconductor oxides in the forms of O^- and O^{2-} thereby decreasing the electronic conduction. Atmospheric oxygen molecules take electrons from the conduction band to be adsorbed as O^-. The reaction is as follows:

$$O_{2\,(g)} \quad +2e \quad \rightarrow \quad 2O^- \quad\quad\quad (1)$$

When reducing gas molecules like NH_3 reacts with negatively charged oxygen adsorbates, the trapped electrons are given back to conduction band. The energy released during decomposition of adsorbed ammonia molecules would be sufficient for electrons to jump up into the conduction, causing on increase in the conductivity of the sensor. The possible reaction is:

$$2NH_3 + 3O^- \rightarrow 3H_2O + N_2 + 3e^- \quad\quad (2)$$
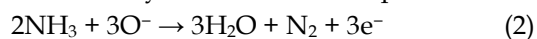
For this reaction to proceed to the right hand side, some amount of activation energy has to be provided thermally. An increase in operating temperature surely increases the thermal energy so as to stimulate the oxidation of NH_3 (Eq. (2)). The reducing gas (NH_3) donates electrons, therefore the resistance decreases, or the conductance increases[6]. This is the reason why the gas response increases with operating temperature. The point at which the gas response reaches maximum is the actual thermal energy needed for the reaction to proceed. However, the response decreases at higher operating temperatures, as the oxygen adsorbates are desorbed from the surface of the sensor[7]. Also, at high temperatures the carrier concentration increases due to intrinsic thermal excitation and the Debye length decreases. This may be one of the reasons for the decreased sensitivity at high temperatures[8]. As the species are desorbed from the surface, oxygen is adsorbed again. As a result, the initial resistance of the film would decrease and the overall change in the resistance on the exposure of gas would be smaller leading to lower sensitivity to the target gas.

CONCLUSION

Sensing performance of Zn_2TiO_4 thick film sensor was observed to be maximum for NH_3 amongst gas tested. The optimum temperature for maximum sensing of NH_3 gas was found to be 300 ^0C. The sensor showed good selectivity to NH_3 gas against LPG, H_2, C_2H_5OH, CO, CO_2 and Cl_2 gases.

Acknowledgement:
Authors are thankful to Dr. B. B. Kale (Director, CMET, Pune) for the support and guidance.

Conflicts of interest: The authors stated that no conflicts of interest.

REFERENCES

1. Zhongchi Liu, Dongxiang Zhou, Shuping Gong, Hui Li , J alloys and compounds,2009;475:840-845
2. Sirajudheen P, '*J Material Sci Eng*' 5,2016; 2
3. Jain GH, Patil LA, Wagh MS, Patil DR, Patil SA, Amalnerkar DP, Sens. Actuators, B, Chem.,2006;117:159-165
4. Jain GH, Gaikwad VB, Kajale DD, Chaudhari RM, Patil RL, Pawar NK, Patil LA, Sens. and Transducers ,2008;90 :160-173
5. Aswal D K and Gupta S K ,*Science and Technology of Chemiresistor Gas Sensors* Nova Sci. Publishers, New York, 2007;37
6. Gaikwad VB, Deore MK, Khanna PK, Kajale DD, Shinde SD, Chavan DN, Jain GH, Recent Advances In Sensing Technology, LNEE 49: 293-308
7. Windichmann H, P. Mark, J. Electrochem. Soc.,1979;126: 627–633
8. Mizsel J, Sens. Actuators, 1995, B 23:173–176.

RESEARCH ARTICLE   **OPEN ACCESS**

Estimation of Frusemide in bulk and tablet formulation by UV spectrophotometric Area under Curve method

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ABSTRACT

The present work was to develop simple UV spectrophotometric method for simultaneous estimation of frusemide in bulk and tablet dosage form and validate as per ICH guidelines. In which two methods, method A is absorption maxima method in that **λmax** was found to be **277 nm.** Method B is Area under curve (AUC). Furosemide is the most commonly used high potency loop diuretics use in clinical practices. A least time consuming efficient and simple UV spectrophotometric method for the assay of frusemide has been developed. Comparison of assay of brands of frusemide (Lasix 20mg) has also been made available in medical store. The assay is based on the ultraviolet UV absorbance maxima at about 277nm wavelength of frusemide using ethanol as solvent. A sample of drug was dissolved in methanol to produce a solution containing frusemide. Similarly, a sample of ground tablet was dissolved in ethanol and various dilutions were made. The absorbance of sample preparation was measured at 277nm against the solvent blank and the assay was determined by comparing with the absorbance of available brand. The developed methods were validated for linearity, precision, accuracy, LOD and LOQ as per ICH guidelines. Both the methods were found to be linear within the conc. Range of 5-25µg/ml for frusemide. The present methods were found to be simple, linear, precise, accurate and sensitive and can be used for routine quality control analysis for the estimation of frusemide in bulk and tablet dosage form**.**

Keywords- Frusemide, Area under Curve, ICH guidelines.

INTRODUCTION

Frusemide (Fu) chemical name is 5-(aminosulfonyl)-4-chloro-2-[(2-furanyl methyl)aminobenzoic acid]. It has the following generic names: Frusemide, Fursemide , Aisemide, Beronald, Desdimin, Lasilix and others. The empirical formula is C12H11ClN2O5S corresponds to molecular weight of 330.77. Frusemide is white to slightly yellow, odourless, almost tasteless crystalline powder, slightly soluble in water, chloroform and ether soluble in acetone, methanol,ethanol, dimethyl formamide [1] and in solutions of alkali hydroxides [2]. It melting point is 206°C; the pH of the aqueous solution is in the range 8.9 to 9.3. The UV spectrum of frusemide (0.01 mg/ml) in 0.1N NaOH was scanned from 190 to 400 nm using DMS 90 Varian spectrophotometer. It exhibited two maxima at 226 and 277 nm. Several methods have been reported for the determination of the components of this important drug (frusemide). Titrimetric methods [3-7], potentiometric methods [8, 9],Ultraviolet methods, Colorimetric methods. Because of cost-effective and minimal maintenance, UV spectrophotometry is always preferred at small scale industries. Literature survey reveals that so far many UV spectrophotometric methods have been reported for the estimation of Furosemide in alone or in combination with other drugs [9]. But out of them only few methods included single estimation of frusemide. Therefore the main objective of the proposed methods were to develop simple, new and economic UV spectrophotometric methods for the estimation of
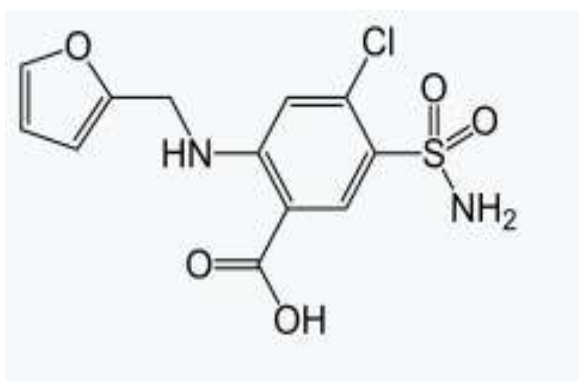
frusemide in bulk and tablet dosage form and validate as per ICH guidelines

Chemical structure of Frusemide

METHODOLOGY

1. Chemicals-

Frusemide was supplied by Sanofi Aventis, Andhari Mumbai, India. Tablet of frusemide 20mg (Lasix) was procured from local pharmacy. Ethanol S.D. Fine Chemicals, Mumbai, India) was used. All chemicals and reagents were of analytical reagent (AR) grade.

2. Instrumentation

A Shimadzu (Kyoto, Japan) model UV-1800 double beam UV-Visible spectrophotometer attached with computer operated software UV probe 2.33 with spectral width of 2 nm, wavelength accuracy of 0.5 nm and pair of 1 cm matched quartz cells was used to measure absorbance of the resulting solutions. Analytical balance, Mettler Toledo (Model JL1503- C).

3. Method
3.1 UV-Spectroscopy Methods
A) Absorbance Maxima Method:

UV-Visible spectroscopy refers to absorption or reflection spectroscopy in the ultra visible spectral region. It means it utilizes the light of visible and adjacent near-UV and near-infrared (NIR) ranges. The absorption or reflectance in the visible range directly affects the perceived colour of the chemical involved. In this range of electromagnetic spectrum, a molecule undergoes electronic transition, absorption measures transition from the ground state to the excited state.[11]

B) Area under curve method

The AUC (area under curve) method is applicable where there is no sharp peak or when broad spectra are obtained. It involves the calculation of integrated value of absorbance with respect to the wavelength between the two selected wavelengths λ1 and λ2. Area calculation processing item calculates the area bound by the curve and the horizontal axis. The horizontal axis is selected by entering the wavelength range over which area has to be calculated. This wavelength range is selected on the basis of repeated observation so as to get the linearity between area under curve and concentration. The above mentioned spectrums were used to calculate AUC. Thus, the calibration curve can be constructed by plotting concentration versus AUC.[15]

4 Experimental Work

a) To check the solubility of Frusemide-

10 mg of frusemide was weighed and solubility of this sample was checked in double distilled water, methanol, ethanol, 1N NaOH, acetonitrile 0.1N HCL. The drug was found to be soluble in ethanol was selected .[16]

b) To identify the λmax of frusemide-

10 mg of the pure drug was accurately weighed and dissolved in 10ml ethanol and the volume was made up to 10 ml with ethanol to give a standard stock solution of 1000μg/ml. Further 2.5ml of 1000ppm solution was withdrawn and was diluted to 25 ml of volumetric flask and 100ppm solution is prepared. Suitable dilutions were made with distilled water to get standard solutions of concentration: 5, 10, 15, 20, 25μg/ml.

C) Sample preparation for analysis of Tablet formulation

Twenty tablets (Frusemide) each tablet containing 20mg of frusemide weighed, average weight calculated and triturated to fine powder and then weighed equivalent to 25mg of frusemide transferred to 25ml of volumetric flask containing proposed diluent, then sonicated for 15 minutes and filtered through whatman filter paper no. 42 to form 1000μg/ml frusemide stock solution of and final volume made up to mark with diluent. From this, 2.5 ml of aliquot transferred in 25 ml of volumetric flask containing diluent to form 100μg/ml of erythromycin stearate stock solution and further dilution of 5, 10, 15, 20, 25ppm and scanned in the range of 200-400nm against ethanol as blank at 215nm and then drug content of solution was calculated by using standard calibration curve.[17]

5. Analytical Method Development

1. Accuracy- It is closeness of the result obtained to the true value. It is often expressed as per cent age recovery by analyzing known added amounts of analyte. Also it can be determined by applying the procedure to quantitatively prepared samples.[18]

2. Precision- The precision of analytical procedure expresses closeness of agreement (degree of scatter) between a series of measurements obtained from multiple sampling of the same homogenous sample under prescribed conditions. It may be considered at three levels: repeatability, intermediate precision and reproducibility. It is expressed as standard deviation or coefficient of variation.[19]

3. Linearity- The linearity of an analytical procedure is the interval between the upper and lower concentration of analyte in the sample for which it has been demonstrated that the analytical procedure is of precision, accuracy and linearity. [20]

RESULTS AND DISCUSSION

Method A

A] Absorbance Maxima Method

Table 1-Calibration Data of frusemide absorbance maxima

CONC	5	10	15	20	25
Abs	0.523	0.886	1.321	1.695	2.046
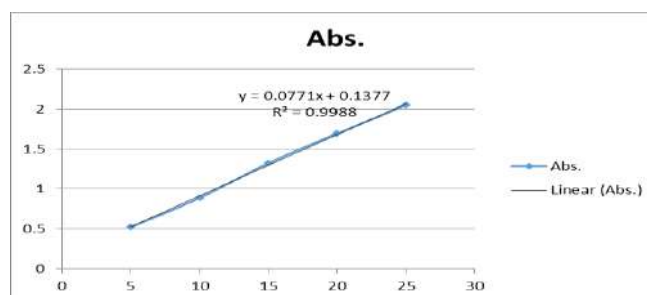
Fig no. 1 Calibration curve of frusemide

Table:

CONC	5	10	15	20	25
AUC	0.905	1.252	1.85	2.288	2.734
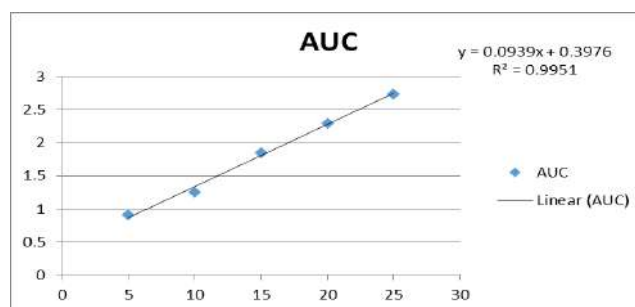
Table 2-Regression Analysis Data for Furosemide by Area under Curve Method

Parameter	AUC
Wavelength Range (nm)	243-277
Concentration Range (µg/ml)	5-25
Slope(m)	0.093
Intercept (c)	+0.397
Correlation Coefficient (r2)	0.995
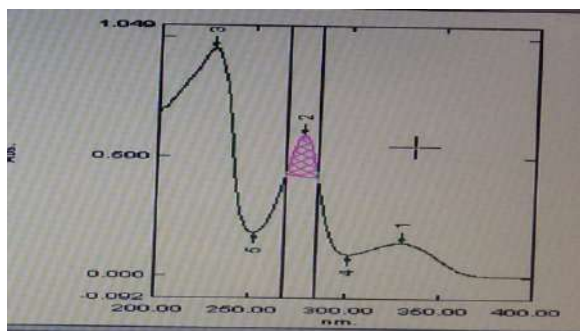
Fig.2 Area under curve of frusemide (10µg/ml)

Table 3: Results of Intra and Inter day precision

Parameter	± S.D.*	% RSD*
Inter day	0.7439	1.48
Inter day	0.8063	1.61

Table 4: Data of Recovery Studies

Level of Mean Recovery (%)	% Mean Recovery	SD*	% RSD*
50%	103.54	0.67	0.65
100%	101.83	0.1616	0.16
150%	102.90	1.1494	1.14

Table 5: Assay Results for the estimation of Furosemide in Pharmaceutical Formulation

Parameter	Label Claim (mg/tab)	Amount Found (mg/tab)	%Labeled Claim
AUC	20	19.84	99.2%

Table no.6. Validation data.

Sr.no	Parameter	AUC Method
1	Linearity	5-25
2	Regression Equation	Y=0.093x+0.397
3	Correlation coefficient	R²=0.995
4	Precision	
4.1	Inter day	0.7439
4.2	Intra day	0.8063

CONCLUSION

Simple UV spectrophotometric methods have been developed and validated for the determination of furosemide in bulk and tablet dosage form. The results of the validation parameters show that the UV spectrophotometric methods were found to be accurate, precise and sensitive. Because of cost-effective and minimal maintenance, the present UV spectrophotometric methods can be preferred at small scale industries and successfully applied and suggested for the quantitative analysis of furosemide in pharmaceutical formulations for QC, where economy and time are essential and to assure therapeutic efficacy.

REFERENCES

1. Merck index, Maryadele J.O. Neil Edu. In: 13 Ed, Merck Research Lab NJ, USA. 2001: 868.

2. Bays HE, Moore PB, Drehobl MA et al. Effectiveness and tolerability of simvastatin in patients with primary hypercholesterolemia pooled analysis of two phase II studies. Cli. Ther 2001; 23 (8): 1209-1230.

3. Arayne MS, Sultana N, Hussain F, & Ali SA. Validated spectrophotometric method for quantitative determination of Simvastatin in pharmaceutical formulations and human serum. *Journal of Analytical Chemistry*, 2007; 62(6): 536-541.

4. Jain N, Jain R, Swami H, Pandey S, & Jain DK. Spectrophotometric Method for simultaneous estimation of Simvastatin and Ezetimibe in bulk drug and its combined dosage form. *Internat. J. Pharmacy Pharm. Sci,* 2009; 1(1): 170-175.

5. Rajput, SJ, & Raj HA. Simultaneous Spectrophotometric estimation of Ezetimibe and Simvastatin in tablet dosage forms. *Indian Journal of Pharmaceutical Sciences,* 2007; 69(6): 759.

6. Mane VB, Babar S, & Kulkarni N. Development of UV Spectrophotometric method for the simultaneous estimation of Simvastatin and Ezetimibe in tablet dosage form by Simultaneous Equation and Absorbance Ratio Method. Development, 2011; 3(3): 1459-1466.

7. *International Journal of Advances in Pharmaceutics 5* (6) 2016 170.

8. Balaji, S, & Sunitha A. Development and validation of Spectrophotometric method for simultaneous determination of Simvastatin and Ezetimibe in tablet formulations. *Pak. J. Pharm. Sci,* 2010; 23(4): 375-378.

9. Bhatia, NM, Deshmukh DD, Kokil SU, & Bhatia, MS. Simultaneous Spectrophotometric estimation of Simvastatin and Ezetimibe in tablet formulation. *J. Chem*, 2009; 6(2): 541-544.

10. Michael E, Swartz Ira S. Krull, Analytical method development and validation, Marcel Dekker, Inc., 1997; 17: 25-2.

11. Christian GD, Analytical chemistry, sixth edition, John Wiley and Sons, 2003; 1-2,604- 620.

12. Skoog, Holler, Nieman, Principles of Instrumental Analysis, fifth edition, Thomson Asia Pvt. Ltd., Singapore, 2004; 300-325.

13. Beckett AH, Stenlake, JB, Practical Pharmaceutical Chemistry, 4th edition, CBS Publishers and Distributors, New Delhi, 2002; 2: 275-295.

14. Samaa, JR, Kalakuntlab RR, Rao VSN, & Reddannaa, P. Simultaneous estimation of Simvastatin and Ezetimibe in pharmaceutical formulations by RP-HPLC method. *J. Pharm. Sci.* Res, 2010; 2(2): 82-89.

15. Hefnawy M, Al-Omar M, & Julkhuf S. Rapid and sensitive simultaneous determination of ezetimibe and simvastatin.

Synthesis, characterization and applications of β-cyclodextrin coated Fe₃O₄-CeO₂

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Manuscript Details

ABSTRACT

Magnetic nanoparticles (MNPs) have emerged as excellent materials in many fields, such as biology, medicine, environment and material science due to their high specific surface area, biocompatibility, low toxicity and strong magnetic responsivity. In this work, we designed and fabricated β-cyclodextrin (β-CD) coated MNPs which could be used in various bio- and environmental applications. Cyclodextrins are natural oligosaccharides which have the molecular Inclusion / complexation capabilities through host-guest interactions with a wide variety of organic and inorganic molecules. Tagging cyclodextrins with magnetic stable nanoparticles makes them magneto-responsive and may lead to a new generation of catalysts which will provide good opportunities for applications in the fields of bio-separation / purification, contaminants removal from waste water in environment pollution cleanup and hydrophobic drug delivery. In this work Fe₃O₄, CeO₂, Fe₃O₄-CeO₂ β-cyclodextrin coated Fe₃O₄, β-cyclodextrin coated CeO₂ and β-cyclodextrin coated Fe₃O₄-CeO₂ were prepared using different co-precipitation method. Functionalized nanoparticles were characterized with XRD and Raman analysis. The catalytic activities were tested for degradation of phenol and oxidation reactions. The degradation of phenol was monitored by chance in colour of the reaction mixture form colorless to dark brown. The chance in the concentration of phenol was monitored by using UV-Vis spectrophotometer.

Keyword:Magnetic Nanoparticles (MNPs), β-cyclodextrin, UV-Vis spectrophotometer.

INTRODUCTION

In Chemical Sciences, nano-structured materials in the form of nanotubes, nanowires, nanorods, nanoribbons and nanosheets [1] have attracted more attention in the last decades due to their unique optical, electrical and magnetic properties and their potential applications in nanodevices. It can be easily understood that metal nanoparticles chemistry differs from that of the bulk materials in terms of size reduction, high surface area to volume ratio which lead to enhanced catalytic activity. Among them magnetite (Fe_3O_4) nanoparticles due to their good biocompatibility, superior superparamagnetic property, nontoxicity and easy preparation process are becoming very popular and promising materials now a days. They have attracted increasing attention and enormous interest in various fields, such as environmental and biomedical applications including enzyme immobilization, protein separation, magnetic resonance imaging, hyperthermia, targeting drug delivery system [2].

Magnetite nanoparticles with superparamagnetism can be easily magnetized with an external magnetic field and demagnetized immediately once the external magnetic field is removed [3]. However, due to high specific surface energy and anisotropic dipolar attraction, magnetite nanoparticles tend to aggregate together into larger clusters which lead to a possible loss of superparamagnetism and limit their applications. Therefore, the surface modification of nanoparticles is necessary and can be modified by inorganic or organic coating to overcome the particles lack. Modification of the surface of MNPs prevents aggregation/agglomeration of the particles, leading to colloidal stability, renders them with water-solubility, biocompatibility, non-toxicity, nonspecific adsorption to cells, and bioconjugation. Moreover, magnetic nanoparticle-based materials can be re-collected by using an external magnet and avoiding conventional filtration or centrifugation processes. Considerable efforts have been made to modify the surface of magnetic nanoparticles and the preparation of organic–inorganic nanocomposites. The combination of inorganic and organic components in a single particle at the nano-sized level has made accessible an immense area of new functional materials [4].

Inorganic materials (silica, gold etc.) natural or synthetic polymers are frequently employed as coating materials to impart surface reactivity. Natural polymers include chitosan, dextran, gelatin, starch, cyclodextrin etc. and synthetic polymers are polyacrylic acid, polyvinyl chloride, poly vinyl alcohol etc.

Cyclodextrins (CDs) are a group of naturally cyclic oligosaccharides, with six, seven, or eight glucose subunits linked by α-(1, 4) glycosidic bonds in a torus shaped structure and are denominated as α-, β-, and γ-CD respectively. The cyclodextrin contains primary and secondary hydroxyl groups at the 2, 3 and the 6 position, respectively [5]. The cyclodextrin assumes a rigid structure because of the formation of a belt of intermolecular hydrogen bonds between hydroxyl groups at the 2 and 3 positions of adjacent glucose units. The rotation of the primary hydroxyl groups is possible, thus reducing the effective diameter of the cavity on the primary side of the molecule. The presence of the hydroxyl groups makes the upper and lower end of the molecule hydrophilic. The cavity of the cyclodextrins is rendered hydrophobic because of the presence of glycosidic oxygens and C–H units. Attention has recently been focused on cyclodextrin based polymeric materials in a wide variety of applications due to their unique sorption properties. Recently, a number of insoluble cyclodextrin polymer or co-polymers have been widely used for various applications such as contaminants removal from wastewater, protein refolding, drug delivery etc. However, the difficulty in separating those powdery cyclodextrin-based adsorbents, except high speed centrifugation, from treated effluent limits their practical applications. Magnetic assisted adsorption separation technology provides an alternative method to separate powdery adsorbents from solution effectively.

Metal-based nanomaterials have also emerged as efficient enzyme mimics. Among them, cerium oxide nanoparticles and magnetic nanoparticles are two most widely used metal oxide catalysts. CeO_2 is one of the most important rare-earth metal oxides and has a wide range of industrial applications, such as in catalysis and solid oxide fuel cells. CeO_2 nanoparticles, which exist in a mixed valence state

(Ce³⁺, Ce⁴⁺), possess many unique properties that have proven to be of high utility in biomedical and catalytic applications. Recently, they have been reported to possess multienzyme, such as SOD, catalase, oxidase, and phosphatase, mimetic properties. For instance, Perez's group reported that nanoceria has an intrinsic oxidase like activity at acidic pH values, as it can quickly oxidize a series of colorimertic dyes without any oxidizing agent. Most recently, it has been found that cerium oxide nanoparticles (nanoceria) possess antioxidant activity at physiological pH values, and the potential use of these materials in biomedical applications, such as protection against radiation damage, oxidative stress, and inflammation, has been reported. The ability of these nanoparticles to act as an antioxidant resides on their ability to reversibly switch from Ce^{3+} to Ce^{4+}. Furthermore, the synthesis of biocompatible dextran-coated nanoceria and its enhanced stability in aqueous solution has been recently reported [6].

The electronic structure and chemical and physical properties of CeO_2 can be easily altered by introducing oxygen vacancies. Such defects can be rapidly formed and eliminated, giving rise to localized or delocalized 4f electron states. Another potentially important application is the diluted magnetic oxides with Curie temperature above room temperature which are a new class for spintronics. Magnetic ordering in a semiconductor depends on a small proportion of transition metal ion dopants that have partially filled d shells, which allow the unpaired electron to mediate ferromagnetism. Many other mechanisms have also been suggested. Introduction of Fe dopant, which normally has a valence of less than +4, may alter the crystal structure and the valence of the Ce ions, changing the chemical or physical properties. The electronic structures around the Ce and Fe sites provide valuable information. Several metals have been successfully used for this purpose, including, ruthenium, cobalt, ruthenium–cobalt, palladium, and nickel. A few examples of mixed oxides (Fe_2O_3–CeO_2) have appeared, namely in dye degradation, oxidations, and synthesis of carbonyl compounds [7]. It is an important and urgent need to design magnetically recyclable nano-catalysts, to reduce their cost of preparation, and to show their efficacy in benign reaction media.

In view of the importance of this research area, we report here the preparation of Fe_3O_4, CeO_2, Fe_3O_4-CeO_2, β-cyclodextrin coated Fe_3O_4, β-cyclodextrin coated CeO_2 and β-cyclodextrin coated Fe_3O_4-CeO_2 and their characterization for catalytic activities for degradation of phenol and oxidation reactions with the following objectives.

METHODOLOGY

Preparation of CeO₂

The investigated CeO_2 were synthesized by coprecipitation method using ultra high dilute aqueous solutions. Coprecipitation was carried out from nitrate precursors $Ce(NO_3)_3 \cdot 6H_2O$ (Aldrich, AR grade). A dilute aqueous ammonia solution was used as the precipitating agent. The requisite quantities of precursors were dissolved in double distilled water under vigorously stirred condition and stirring continued until a clear solution obtained. The mixed solution was precipitated by drop-wise addition of aqueous ammonia solution over a period until the solution pH reached ~8.5. The resulting pale yellow colored slurry was decanted, filtered and washed several times with double distilled water until free from anion impurities. The obtained precipitate was oven dried at 393 K for 12 h and calcined at 773 K for 5 h at a heating rate of 5 K min⁻¹ in air atmosphere.

Preparation of Fe₃O₄

The Fe_3O_4 were synthesized by coprecipitation method using $FeCl_2 \cdot 4H_2O$ (Fluka, AR grade) and $FeCl_3 \cdot 6H_2O$ (Aldrich, AR grade). A dilute aqueous ammonia solution was used as the precipitating agent. The requisite quantities of precursors were dissolved in double distilled water separately, mixed together under vigorously stirred condition and stirring continued until a clear solution obtained. The mixed solution was precipitated by drop-wise addition of aqueous ammonia solution over a period until the solution pH reached ~8.5. The resulting black colored slurry was decanted, filtered and washed several times with double distilled water until free from anion impurities. The obtained precipitate was oven dried at 363 K for 12 h.

Preparation of β-cyclodextrin coated Fe_3O_4-CeO_2 nanoparticles: Prepared Fe_3O_4-CeO_2 nanoparticles were mixed with distilled water to make a colloidal solution. The colloidal solution was mixed with β-cyclodextrin (0.2, 0.5, 0.8 and 1wt% of catalyst) under intense stirring at 40°C. The formed nanoparticles were collected by filtration. The particles were washed with distilled water repeatedly and dried for 24 h in oven at 90°C.

Catalyst characterization

Powder X-ray diffraction analysis: Powder X-ray diffraction data were acquired on a Rigaku Multiflex diffractometer utilizing nickel-filtered Cu Kα (0.15418 nm) radiation source and a scintillation counter detector. The samples were scanned from 2–80° 2θ in 0.02° steps with a counting rate of 1 s/step. The mean crystallite size of the solid solution phases was estimated with the help of Scherrer equation from the line broadening, and the lattice parameter was calculated by a standard cubic indexation method using the intensity of most prominent peak.

The Raman spectra: The Raman spectra of investigated samples were collected on a Horiba Jobin-Yvon HR800 Raman spectrometer equipped with a liquid-nitrogen cooled charge coupled device (CCD) detector and a confocal microscope. A line at 325 nm from a He-Cd laser is used as excitation source of the UV Raman spectroscopy. Visible Raman spectra were recorded with a 632-nm Ar^+ ion (Spectra Physics) laser. The lasers were focused keeping the sample under a microscope with the diameter of the analyzed spot being ~1 μm, under the ambient conditions. The acquisition time was adjusted according to the intensity of Raman scattering. The wave number values reported from the spectra are accurate to within 1 cm^{-1}.

RESULTS AND DISCUSSION

Characterization of catalysts

The XRD patterns of Fe_3O_4 (prepared at 60°C), Fe_3O_4, (prepared at 90°C) and β-cyclodextrin coated Fe_3O_4 samples are illustrated in Fig. 1, 2 and 3 respectively .

In Fig. 1 & 2 all characteristic peaks at 30.20, 35.51, 43.32, 53.71, 57.21 and 62.94 were corresponding to the (220), (311), (400), (422), (511) and (440) crystal planes of a pure Fe_3O_4 with a spinal structure (JCPDS file PDF no.65-3107) [8].
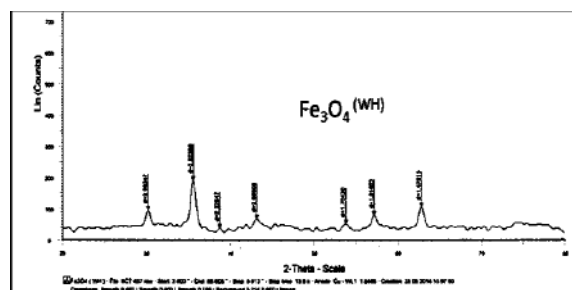
Fig 1: XRD pattern of Fe_3O_4 prepared at 60°C
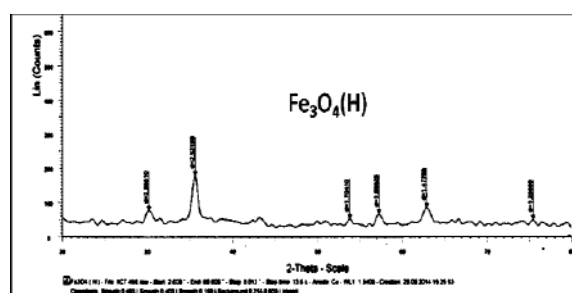
Fig 2: XRD pattern of Fe_3O_4 prepared at 90°C
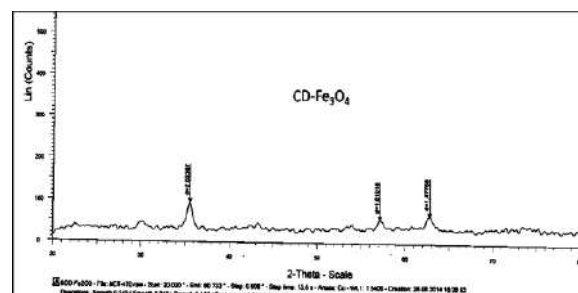
Fig 3: XRD pattern of β-cyclodextrin coated Fe_3O_4

It indicates that the samples are in single phase. The peaks indicate that Fe_3O_4 with a spinal structure and no characteristic peak of impurities are detected in the XRD pattern. This success in fabricating high quality Fe_3O_4 nanoparticles is essential for further research work. All diffraction peaks and positions for Fe_3O_4 match well with those for the β-cyclodextrin coated Fe_3O_4. The peak intensity in the β-cyclodextrin coated Fe_3O_4 was less as compared to the pure Fe_3O_4 due to the coating of β-cyclodextrin on the surface of Fe_3O_4.

Raman Analysis

Raman scattering is an inelastic phenomenon. Although its cross section is very small, recent advances in electronics, lasers, optics, and nanotechnology have made Raman spectroscopy suitable in many areas of application. With the advent of Raman enhancement mechanisms and the progress being made in metal nanomaterials and nanoscale metal surfaces fabrications, surface enhanced Raman scattering spectroscopy has become an extra sensitive method applicable for analysis of foods, drugs and for intracellular and intercellular imaging. Raman spectra are usually used to identify the phase of materials. Figure 4 illustrates the Raman spectra of magnetite nanoparticle Fe_3O_4. As we can see the peaks at 226 cm⁻¹, 496 cm⁻¹, 607 cm⁻¹, and 651 cm⁻¹ were observed. Among them 226 cm⁻¹ and 496 cm⁻¹ peaks attributed to T_{2g} vibration mode of Fe_3O_4. The peaks at 291 and 819 cm⁻¹ were mainly due to the M=O and M-O-M bonding [9]
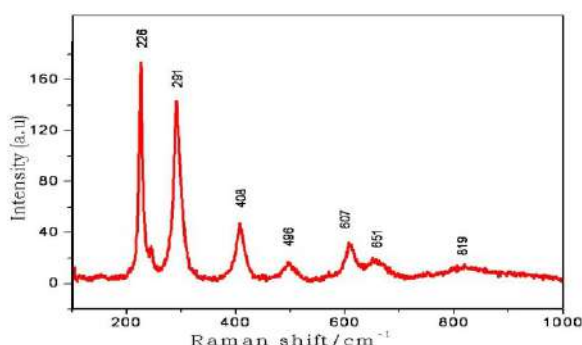
Fig 4: Raman spectra of Fe_3O_4

Phenol degradation studies

Industrial activities generate large amounts of organic hazardous substances in many industries as by-products such as petroleum refining, petrochemical, pharmaceutical, plastic, pesticides, chemical industries, agrochemicals, and pulp and paper industries [10]. The organic pollutants e.g. phenol, are toxic and cause considerable damage and threat to the ecosystem in water bodies and to the human health even at low concentrations. The organics in wastewaters from chemical and related industries cannot be well treated by conventional processes due to degradation of these pollutants being very slow or ineffective and not environmentally compatible.

Heterogeneous catalytic oxidation systems have recently attracted much interest in the waste water treatment processes due to easily recovery and reuse of the catalysts. The Fe_3O_4 MNPs has peroxidase-like activity and could activate H_2O_2, yielding over 85% removal of phenol and about 30% mineralization within 3 h. Also cerium exhibits a redox cycle between the 3+ and 4+ oxidation states, providing a high oxygen storage capacity. This property enhances the performance of transition metal catalysts used in automotive exhaust treatment and waste water treatment. The nanoscaled Fe−Ce oxide hydrate catalyst showed a relatively low rate in heterogeneous Fenton reaction for decolorization of reactive brilliant red X-3B, which was assisted by UV irradiation to achieve almost complete removal of dye within 30 min. We anticipate that nanoscaled cerium oxide (CeO_2) can enhance the catalytic activity of Fe_3O_4 MNPs, and this novel composite is a fascinating and competitive candidate for the catalytic activation of H_2O_2. Furthermore, Heckert et al. suggested that cerium might be capable of redox cycling in the presence of H_2O_2 and behaved similar to iron in a Fenton-like reaction [11]. Based on this we studied the phenol degradation by magnetic nanoscaled Fe_3O_4-CeO_2 and β-cyclodextrin coated Fe_3O_4-CeO_2 composite prepared by the methods described above.

Degradation Experiment

Batch degradation experiments of phenol were carried out in a conical flask (25 mL) placed on a magnetic stirrer with the constant stirring. The reaction suspension was prepared by adding the required amount of catalyst into 5 mL of phenol. A known concentration of H_2O_2 was added to the solution to initiate the reaction. Then, samples were taken at set intervals using a 5 µL syringe and quenched with excess methanol. The reaction mixture was analyzed with the help of a UV-Vis spectrophotomer in the range of 200-800 nm.

Catalytic Activity of Fe_3O_4-CeO_2 Composite

Control experiments were conducted to compare the removal efficiencies of phenol by various processes at pH 7.0. The change in the absorbance of phenol in the reaction mixture was analyzed by JASCO, V650 UV-

Vis spectrophotometer. The change in intensity of peak at λ_{max} for phenol at 270 nm was shown in the Fig. 5.
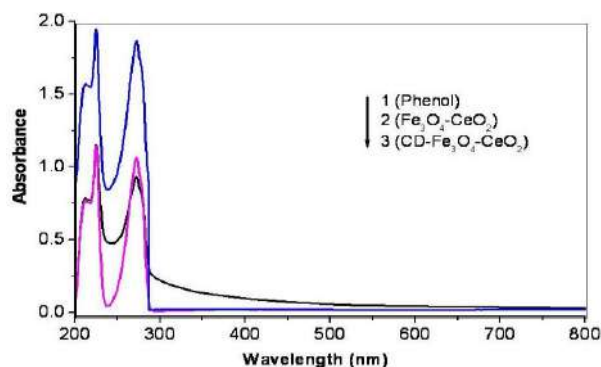
Fig.5: UV-Vis spectra of phenol degradation by using Fe_3O_4-CeO_2 and β-cyclodextrin coated Fe_3O_4/CeO_2

In the presence of H_2O_2, the decrease in absorbance of phenol using β-cyclodextrin coated Fe_3O_4-CeO_2 composite as the heterogeneous Fenton-like catalyst was notably higher than that for pure Fe_3O_4-CeO_2, implying that the catalytic activity was enhanced by the introduction of β-cyclodextrin. The phenol degradation study was also conducted by using CeO_2, β-cyclodextrin coated CeO_2, Fe_3O_4 and β-cyclodextrin coated Fe_3O_4. For CeO_2 and Fe_3O_4 catalyst the observed decrease in absorbance for phenol (λ_{max} 270 nm) was negligible at the specified reaction conditions in presence of H_2O_2 [12, 13].

In Fig 6 the chance in colour of phenol in the solution from colour less to dark brown is shown which is attributed to the formation of intermediates like catechol, resorcinol and glycerol etc. during the degradation of phenol in presence of catalyst and H_2O_2 after 1h. Fig 7 shows the easy separation of catalyst from the reaction mixture by using external magnet. The separation of the catalyst was better in case of CD coated MNP than the Fe_3O_4 nanoparticles [14]. This magnetic separation of catalyst was more convenient than the conventional filtration methods and may be used for different Industrial separation processes.

Figure 6 Photographs of phenol degradation after 1 h,
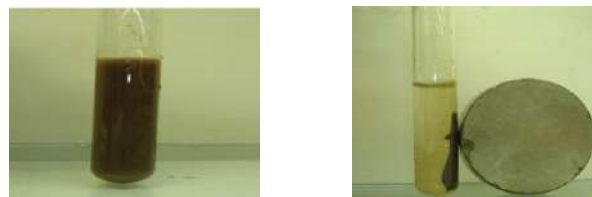
Fig. 7 Separation of catalyst using an external magnet.

CONCLUSION

Fe_3O_4-CeO_2 was successfully prepared by coprecipitation method. The resulting nanoparticles were coated with β-cyclodextrin, the reactivity and the magnetic separation property of β-cyclodextrin coated Fe_3O_4-CeO_2 can be used in catalysis and adsorption with high efficiency. The degradation of phenol and separation of catalyst by using external magnet was more pronounced with CD- Fe_3O_4-CeO_2 than the Fe_3O_4-CeO_2. The interaction between the Fe_3O_4 and β-cyclodextrin results in the formation of a complex with enhanced aqueous solubility. The use of β-cyclodextrin can improve the magnetic properties of the Fe_3O_4. The growth and the particle size distribution of the inorganic materials may be restricted by the use of the β-cyclodextrin. Furthermore due to the high activity of β-cyclodextrins in the resulting product, they have promising applications in much more extensive fields.

REFERENCES

1. Zhang N, J. Li, Z. He, K. Sun, Preparation and characterization of nano-tube and nano-rod structured $La_{0.8}Sr_{0.2}MnO_{3-\delta}/Zr_{0.92}Y_{0.08}O_2$ composite cathodes for solid oxide fuelcells, Electrochem. Comm. 2011;13: 570-573.
2. Gupta AK, Gupta M, Synthesis and surface engineering of iron oxide nanoparticles for biomedical applications, Biomaterials. 2005; 26 :3995-4021.
3. Bruce I.J, Sen T, Surface modification of magnetic nanoparticles with alkoxysilanes and their application in magnetic bioseparations, Langmuir. 2005; 21:7029-7035.
4. Liao MH, Chen DH, Preparation and characterization of a novel magnetic nanoadsorbent, *J. Mater. Chem.* 2002; 12 :3654-3659.

5. Zhao D, Zhao L, Zhu CS, Huang WQ, Hu JL, Water insoluble ß-cyclodextrin polymer crosslinked by citric acid synthesis and adsorption properties toward phenol and methylene blue, *J. Inclusion Phenom. Macrocyclic Chem.* 2009; 63: 195-201.

6. Asati A, Santra S, Kaittanis C, Nath S, Perez J M, Oxidase- Like Activity of Polymer-Coated Cerium Oxide Nanoparticles. Angew. Chem., Int. Ed. 2009; 48: 2308−2312.

7. Lejin Xu, Jianlong Wang, Magnetic Nanoscaled Fe_3O_4/CeO_2 Composite as an Efficient Fenton-Like Heterogeneous Catalyst for Degradation of 4-Chlorophenol, Environ. Sci. Technol. 2012; 46: 10145−10153

8. G. L. Li, Y. R. Jiang, K. L. Huang, P. Ding, and I. I. Yao, Kinetics of adsorption of sacharomyces cereviceae mandelated dehydrogenase on magnetic Fe3O4-chitosan nanoparticles, Journal Colloid Interface Sci. 2008;320: 11-18.

9. D. L. A. De Faria, S. V. Silva, et al., Raman Microspec-troscopy of Some Iron Oxides and Oxyhydroxides, Journal of Raman Spectroscopy 1997; 28: 873-878.

10. Saputraa E, Muhammada S, Suna H, Anga H, Tadéa MO, Wanga S, Manganese oxides at different oxidation states for heterogeneous activation of peroxymonosulfate for phenol degradation in aqueous solutions, App Cat B: Env2013; 142– 143:729– 735.

11. S. Yu, H.J. Yun, Y. H. Kim, J. Yi, Carbon-doped TiO2 nanoparticles wrapped with nanographene as a high performance photocatalyst for phenol degradation under visible light irradiation, App Cat B: Env 2014; 144: 893– 899

12. Ayati A, Ahmadpour A, Bamoharram FF, Tanhaei B, Manttari M, Sillanpaa M, A review on catalytic applications of Au/TiO2 nanoparticles in the removal of water pollutant, Chemosphere 2014; 107:163–174.

13. Y. Zhang, X. M. Dou, J. Liu, M. Yang, L. P. Zhang, Y.Kamagata, Decolorization of reactive Brilliant Red X-3B by heterogeneous photo-Fenton reaction using an Fe−Ce bimetal catalyst, Catal. Today 2007; 126: 387−393.

14. Heckert EG, Seal S, W. T. Self, Fenton-like reaction catalyzed by the rare earth inner transition metal cerium, Environ. Sci. Technol. 2008; 42: 5014−5019.

Synthesis and Characterization of Azopyrazole derivatives

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ABSTRACT

In present investigation 3-(2-(4-substitutedphenyl)-hydrazono)-pentane-2,4-dione synthesized by using substituted aniline and acetyl acetone in presence of sodium nitrite. The synthesized compound further reacted with isoanizide and semicarbazide to obtained 4-(4-substitutedphenyl)diazenyl)-3,5-dimethyl-1H-pyrazol-1-yl)(pyridine-4-yl)methanoneand4-(4substitutedphenyl)diazenyl)-3,5-dimethyl-1H-pyrazol-1-carboximide. All the newly synthesized compounds were characterized on the basis of elemental analysis, respective melting point, IR and ^{1}H-NMR spectroscopic techniques.

Keywords: Aniline, Azopyrazole, IR, ^{1}H-NMR.

INTRODUCTION

Pyrazole is an important heterocyclic compound containing three carbon atom and two adjacent nitrogen atom. Synthesis of azopyrazole derivatives from aniline bears a great attention in recent years. Azo pyrazole derivative exhibeat wide variety of biological activities such as antibacterial, analgesic, antifungicidal anti-inflamatory [1-5]. By considering this point of view the objective of present work is to prepare new derivatives of azopyrazole and characterized by different spectroscopic techniques.

METHODOLOGY

All chemicals used were of the analytical reagent (AR) grade and of highest purity available and purchased from SD-Fine Chem Limited. Melting points were determined with an Electro thermal 9100 apparatus and are uncorrected.

Synthesis of 3-(2-(4-substitutedphenyl)-hydrazono)-pentane-2,4-dione

4-substituted aniline (0.01 mole) was dissolved in a mixture of concentrated HCl (8 ml) and water (6 ml) and cooled to 0°C on ice bath. The cold diazonium salt solution was filtered into a cooled solution of acetyl acetone in presence of sodium nitrite, sodium acetate (0.01 mole) in ethanol (20 ml) and stirred for 2 hrs and resulting solid was filtered, dried and recrystallized by ethanol. (2a-c)

Synthesis of 4-(4-substitutedphenyl)diazenyl)-3,5-dimethyl-1H-pyrazol-1-yl)(pyridine-4-yl)methanone

A mixture of 3-(2-(4-substitutedphenyl)-hydrazono)-pentane-2,4-dione (0.01 mole) and isoaniazide (0.015 mole) in glacial acetic acid (15 ml) is refluxed for 4-5 hrs. The resulting mixture was concentrated and allowed to cool. The resulting solid was filtered, washed, dried and recrystallised from ethanol to obtained pure compound. (3a-c)

Synthesis of 4-(4-substitutedphenyl)diazenyl)-3,5-dimethyl-1H-pyrazol-1-carboximide

A mixture of 3-(2-(4-substitutedphenyl)-hydrazono)-pentane-2,4-dione (0.01 mole) and semicarbazide (0.015 mole) in glacial acetic acid (15 ml) is refluxed for 4-5 hrs. The resulting mixture was concentrated and allowed to cool. The resulting solid was filtered, washed, dried and recrystallised from ethanol to obtained pure compound. (4a-c)
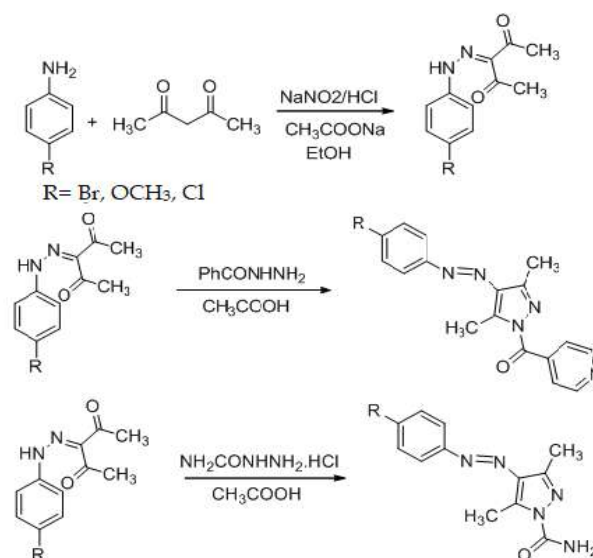
Fig 1: Scheme of Reaction

Table 1: Physiochemical data of all synthesized compounds.

Sr No	Comp.	R	Molecular Formula	Mol. Wt.	% Yield	Elemental Analysis			
						M. P. (°C)	% C	% H	% N
1	2a	Br	$C_{11}H_{11}BrN_2O_2$	283.12	71%	110	46.66	3.92	9.89
2	2b	OCH₃	$C_{12}H_{14}N_2O_3$	234.25	69%	84	61.53	6.02	11.96
3	2c	Cl	$C_{11}H_{11}ClN_2O_2$	238.67	70%	107	55.36	4.65	11.74
4	3a	Br	$C_{17}H_{14}BrN_5O$	384.23	53%	171	53.14	3.67	18.23
5	3b	OCH₃	$C_{18}H_{17}N_5O_2$	335.36	59%	179	64.47	5.11	20.88
6	3c	Cl	$C_{14}H_{14}ClN_5O$	339.78	62%	169	60.09	4.15	20.61
7	4a	Br	$C_{12}H_{12}BrN_5O$	322.16	67%	132	44.74	3.75	21.74
8	4b	OCH₃	$C_{13}H_{15}N_5O_2$	273.29	65%	133	57.13	5.53	25.63
9	4c	Cl	$C_{12}H_{12}ClN_5O$	277.71	70%	129	51.90	4.36	25.22

RESULTS AND DISCUSSION

All the newlly synthesized compound were characterized on the basis of IR and ^{1}H-NMR spectroscopic techniques.

Spectral data of 3-(2-(4-bromophenyl)-hydrazono)-pentane-2,4-dione (2a)

IR (KBr) ν_{max}: cm^{-1} : 3318(-NH), 3072(-Ar-CH), 2927(-Al-CH), 1683(-C=O), 1489(-C=N), 1584(-C=C), 1065(C-O), 741(C-Br): ^{1}H-NMR (DMSO-d$_6$) δ: 14.2(1H,s,NH), 7.6(2H,m,Ar-H), 7.3(2H,d,Ar-H), 2.6(3H,s,CH$_3$), 2.3(3H,s,CH$_3$)

Spectral data of 3-(2-(4-methoxyphenyl)-hydrazono)-pentane-2,4-dione (2b)

IR (KBr) ν_{max}: cm^{-1} : 3315(-NH), 3069(-Ar-CH), 2931(-Al-CH), 1680(-C=O), 1501(-C=N), 1590(-C=C), 170(C-O), 740(C-Br): ^{1}H-NMR (DMSO-d$_6$) δ: 14.3(1H,s,NH), 7.7(2H,m,Ar-H), 7.3(2H,d,Ar-H), 2.5(3H,s,CH$_3$), 2.3(3H,s,CH$_3$)

Spectral data of 3-(2-(4-chlorophenyl)-hydrazono)-pentane-2,4-dione (2c)

IR (KBr) ν_{max}: cm^{-1} : 3320(-NH), 3075(-Ar-CH), 2929(-Al-CH), 1681(-C=O), 1485(-C=N), 1584(-C=C), 1067(C-O), 741(C-Br): ^{1}H-NMR (DMSO-d$_6$) δ: 14.5(1H,s,NH), 7.5(2H,m,Ar-H), 7.4(2H,d,Ar-H), 2.6(3H,s,CH$_3$), 2.3(3H,s,CH$_3$)

Spectral data of 4-(4-bromophenyl)diazenyl)-3,5-dimethyl-1H-pyrazol-1-yl)(pyridine-4-yl)methanone (3a)

IR (KBr) ν_{max}: cm^{-1} : 3060(-Ar-CH), 2921(-Al-CH), 1670(-C=O), 1514(-C=N), 1588(-C=C), 1171(C-O), 741(C-Br): ^{1}H-NMR (DMSO-d$_6$) δ: 7.8(4H,m,Ar-H), 7.5(4H,m,Ar-H), 2.5(3H,s,CH$_3$), 2.2(3H,s,CH$_3$)

Spectral data of 4-(4-methoxyphenyl)diazenyl)-3,5-dimethyl-1H-pyrazol-1-yl)(pyridine-4-yl)methanone (3b)

IR (KBr) ν_{max}: cm^{-1} : 3065(-Ar-CH), 2920(-Al-CH), 1674(-C=O), 1520(-C=N), 1590(-C=C), 1160(C-O): ^{1}H-NMR (DMSO-d$_6$) δ: 7.6(4H,m,Ar-H), 7.2(4H,m,Ar-H), 2.4(3H,s,CH$_3$), 2.2(3H,s,CH$_3$)

Spectral data of 4-(4-Chlorophenyl)diazenyl)-3,5-dimethyl-1H-pyrazol-1-yl)(pyridine-4-yl)methanone (3c)

IR (KBr) ν_{max}: cm^{-1} : 3064(-Ar-CH), 2931(-Al-CH), 1672(-C=O), 1515(-C=N), 1591(-C=C), 1170(C-O): ^{1}H-NMR (DMSO-d$_6$) δ: 7.6(4H,m,Ar-H), 7.4(4H,m,Ar-H), 2.6(3H,s,CH$_3$), 2.4(3H,s,CH$_3$)

Spectral data of 4-(4-bromophenyl)diazenyl)-3,5-dimethyl-1H-pyrazol-1-carboximide (4a)

IR (KBr) ν_{max}: cm^{-1} : 3062(-Ar-CH), 2935(-Al-CH), 1665(-C=O), 1540(-C=N), 1587(-C=C), 1165(C-O), 741(C-Br): ^{1}H-NMR (DMSO-d$_6$) δ: 7.2(2H,d,Ar-H), 7.8(2H,d,Ar-H), 2.2(3H,s,CH$_3$), 2.3(3H,s,CH$_3$)

Spectral data of 4-(4-methoxyphenyl)diazenyl)-3,5-dimethyl-1H-pyrazol-1-carboximide (4b)

IR (KBr) ν_{max}: cm^{-1} : 3069(-Ar-CH), 2940(-Al-CH), 1664(-C=O), 1541(-C=N), 1585(-C=C), 1161(C-O), 735(C-Br): ^{1}H-NMR (DMSO-d$_6$) δ: 7.1(2H,d,Ar-H), 6.9(2H,d,Ar-H), 2.3(3H,s,CH$_3$), 2.5(3H,s,CH$_3$)

Spectral data of 4-(4-chlorophenyl)diazenyl)-3,5-dimethyl-1H-pyrazol-1-carboximide (4c)

IR (KBr) ν_{max}: cm^{-1} : 3070(-Ar-CH), 2933(-Al-CH), 1664(-C=O), 1543(-C=N), 1581(-C=C), 1169(C-O), 743(C-Br): ^{1}H-NMR (DMSO-d$_6$) δ: 7.4(2H,d,Ar-H), 7.6(2H,d,Ar-H), 2.5(3H,s,CH$_3$), 2.8(3H,s,CH$_3$)

CONCLUSION

During this study some azopyrazole have been synthesized by using isoaniazide and semicarbazide. Spectroscopic data obtained matches with the structure of compounds.

Conflicts of interest: The authors stated that no conflicts of interest.

REFERENCES

1. Mohamed A, Hasan SM and Wadood A. *Oriental J Chem*, 18, 2002, 351.
2. Tabarelli Z, Rubin MA, Berlese DB, Sauzem PD, Missio TP, Teixeira MV, Sinhorin AP, Martind MA, Zanatta N, Bonacorso HG and Mello CF. *Brag J Med Biol Res*, 2004,37: 1531.
3. Sahu SK, Banerjee M, Samantray A, Behara C and Azan MA, *Trop J Pharm Res*, 2008,7:961.
4. Maccari R, Ottana R and Vigorita MG. *Bioorg. Med. Chem. Lett.,* 2005,15: 2509.
5. Walcourt A, Loyevsky M, Lovejoy DB, Gordeuk VR and Richardson DR. *Int. J. Biochem. Cell Biol.,* 2004, 36: 401.
6. Patel C, Rami CS, Panigrahi B and Patel CN. *Journal of Chemical and Pharmaceutical Research*, 2010, 2(1):73-78.
7. Amer AM, Mohamed EK, Raslan S and Tahawe H. *Journal of American Science*, 2010, 6 (9):889-892.
8. Zangade SB, Jadhav JD, Lalpod, Vibhute YB, Dawane BS. *J. Chem. Pharm. Res.,*2010, 2(1): 310-314
9. Jayapa MR, Sreenivasa K, Prasad and Sreedhar NY. *J. Chem. Pharm. Res.,* 2010, 2(3):127-132.
10. Plasencia C, Daym R, Wang, Q, Pinski J, Burke TR, Quinn DI, Neamati N. *Mol. Cancer Ther,*2005,4: 1105.
11. Zhao H, Neamati N, Sunder S, Hong H, Wang S, Milne GW, Pommier Y, Burke TR. *J. Med. Chem.,*1997, 40: 937.
12. Terzioglu N, Gursoy A. *Eur. J. Med. Chem.* , 2003, 38:781

RESEARCH ARTICLE   **OPEN ACCESS**

Environmental Sciences Studies of the Physicochemical Parameters of soil samples of Khed Taluka Dist: Pune (Maharashtra)

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ABSTRACT

The term "soil testing" refers to the full range of chemical, physical and biological tests that may be carried out on a submitted sample of soil, though in the present context, only nutritional aspects will be considered. Assessment of land capability for various forms of agriculture, In the present investigation the physicochemical study of soil is based on various parameters like total Organic Carbon, Nitrogen (N), Phosphorus (P_2O_5), Potassium (K_2O), pH and Conductivity. To study the effect of phosphate fertilizer, phosphorus, and application of nitrogen to increase percentage yield of crops . This information will help farmers to decide the problems related to soil nutrients amount of fertilizers to be added to soil to make production economic.

Keywords: Quality of soil, Soil test, EC, PH, Total organic carbon Nitrogen (N), phosphorus (P) and potassium (K).

INTRODUCTION

Soil is a non-renewable resource upon which mankind depends for survival. Historically, the rise of great civilizations has been linked to the quality of soil and the availability of water. Equally, the demise of such civilizations is often attributed to mismanagement of soil and land in its broadest sense. Crop productivity and soil fertility are thus synonymous.

Yields have to be increased from existing land areas; adding fertility to the soil to satisfy the demands of higher-yielding crops is essential. Soils vary greatly throughout the world; they have inherent weakness, primarily deficiencies in nutrients that are essential to growing crops Tests are usually performed to measure fertility and indicate deficiencies that need to be remedied Soil testing can be divided into four steps (1) sampling, (2) analysis, (3) interpretation, and (4) recommendations. Most soils are deficient in nitrogen (N); it is transient in nature, and plants need a lot of it. In many cases, phosphorus (P) is just as critical; soil chemical reactions reduce the effectiveness of P fertilizers. The soils are generally well supplied with potassium (K), and usually don't need fertilization, especially for low-yielding rainfed crops. In recent years, there is a growing realization has grown that other elements, e.g., micronutrients, are deficient in some areas of the region.

METHODOLOGY

Size of Area to Sample:
Traditional Methods:
The size of the area from which a sample is taken may vary from less than one acre (for example, lawns, gardens, etc.) to 15 acres. For most field conditions the size may range from 5 to not more than 15 acres. Variations in soil types, slope, drainage, or past management may require that smaller areas be sampled, resulting in three or more composite samples per field.

Precision Agriculture Methods:
Precision farming uses the modern tools of Global Positioning Systems (GPS), Geographic Information Systems (GIS), and remote sensing to delineate subsections of fields that can be sampled separately. The division of field areas that can be sampled separately is typically based on soil type, field topography, or crop growth or yield maps from the previous season. Although areas of poor crop growth may be due to factors other than plant nutrition, sampling these areas separately helps to determine if lack of fertility may be reducing crop yield

Physico-Chemical Analysis:
The collected samples were analysed for major Physicaland Chemical soil parameter Like pH, Elecrical Conductivity(EC), Organic Carbon (OC), Nitrogen(N), Phosphorus(P), Potassium(K). Organic matter is oxidized with chromic acid(Potassium Dichromate, Sulphuric acid) This method is widely used in Indian laboratories. The K and P analysis by standard methods.

PH was measured using PH meter, EC was measured using a Conductivity meter, OC was measured using colorimeter, Potassium was measured using Flame photometer, Phosphorus was measured using Spectrophotometer. All apparatus are Systronic make. Examination of soil done by Government of Maharashtra Horticulture Training Centre, Talegaon Dabhade, Dist-Pune

RESULTS AND DISCUSSION

Total 10 villages soil samples of Khed Taluka, Dist : Pune were collected in clean polythene bags and brought to the Laboratory it is the permissible standard according to Horticulture Training Centre,. Air dry the soil samples in shade, crush the soil clods lightly and grind with the help of pestle and mortar, pass the entire quantity through 2mm stainless steal sieve, if the gravel content is substantial record as percent of the sample (w/w) as to pass it through 0.2 to 0.5 mm sieves, processing of the samples for analysis.

Determination of Soil
(1) Soil Temperature :-
 Soil temperature is one of the most important soil properties that effect crop growth. The major source of heat is sun and heat generated by the chemical and biological activity of the soil is negligible.

(2) PH :-
The soil reaction or PH is meant to express the acidity or alkalinity of the soil. The PH is very important property of the soil is it determines the capacity. The PH values fluctuated less than 8.5(table-1). The limit of PH value for soil Acidic. < 6.5, Normal 6.5-7.8, Alkaline 7.8- 8.5, Alkali > 8.5.

(3) EC:-

Total soluble salts are estimated from electrical conductivity (EC) of aqueous soil extracts. Standard value of EC in soil- Normal < 0.8 dsm-1, critical for salt sensitive crops, critical for salt tolerant crops 1.6 - 2.5 dsm-1, Injurious to most crops > 2.5 dsm-1. The EC value 04 to 1.8 (table no.1)

(4) OC and Nitrogen (N) :-

Soil organic carbon is the seat of nitrogen in soil and its determination is often carried out as an index of nitrogen availability. In the colorimeter method (Datta et al, 1962), Organic matter is oxidized with chromic acid. OC in Khed taluka 0.23 to 0.85 (table no.1) .Standard value of OC low < 0.50, medium 0.50- 0.75 and high > 0.75.

(5) Phosphorus:-

Phosphorus was found in the range of low, medium, high (table no.1). Inorganic phosphorus as orthophosphate plays a dynamic role in aquatic ecosystem. Phosphorus, the most important micro nutrient, is utilized by plant in the form of $H_2PO_4^-$ & HPO_4^{2-} species.

6)Potassium:

Standard value of K as K2O in soil low < 140 kg K2O ha-1, medium 140-280 kg K2O ha-1high > 280 kg K2O ha-1. Potassium was found in the range of low, medium, high (table no.1). K though present in small amount in soil sample, plays a vital role in the metabolism of fresh water and considered to be an important micronutrient. The K is relatively abundant in the earth's crust, most of it is not accessible to plant.

Experimental value of quality characteristic especially PH, EC, OC, N, P, K, of soil of Khed Taluka are present in the table no. 1. Result are in tune with farming practices followed by farmers of this region. Most of the farmer's are using chemical fertilizer, Urea and Nitrogen fertilizer only since last 25 to 30 years which contains concentrated amount of Nitrogen, OC & Phosphorus. On the basis of these results farmers are advised to use integrated nutrient management practice to maintain optimum concentration of all the essential nutrients for plants. Farmers are also advised to add bio-fertilizers containing organic carbon and nitrogen solubilizing bacteria.

Table 1: Experimental value of quality characteristic especially PH, EC, OC, N, P, K, of soil of Khed Taluka

Sample No.	pH	E.C.	Organic Carbon	N	P	K
01	7.25	0.16	0.84	412.00	20.02	323.40
02	8.10	0.14	1.02	460.14	16.24	342.12
03	7.72	0.12	0.72	354.24	24.12	424.24
04	7.90	0.18	0.64	314.14	20.28	404.12
05	7.48	0.21	0.92	448.46	16.42	332.54
06	8.16	0.24	0.68	454.18	20.26	428.00
07	8.24	0.18	0.82	308.84	24.34	421.32
08	7.80	0.20	1.04	364.14	26.02	522.24
09	7.68	0.22	0.68	452.42	24.84	548.64
10	8.05	0.16	0.82	404.16	18.64	424.32
11	8.26	0.20	0.74	514.34	22.62	436.74
12	7.54	0.24	0.92	426.34	28.34	483.50

EC- mS/cm.   C- org-%,   N,P& K- Kg/hectre,

1) The physico-chemical analysis of the soil samples from western villages have influence of the uncontrolled solid waste disposal practice. The main crops are potato, onion and groundnut.
2) Most of the farmers are using excessive chemical fertilizers and the too much dose of such fertilizers in few soils has rendered high values of P and K. The retention of K could also be due the clay minerals formed by chemical weathering of basalts which is the parent material for the soil.
3) Use of acidic fertilizers and organic manure can be a remedy which can raise the crop yield.
4) Monitoring of micronutrients in the soils should be done periodically as it can be an efficient way to assess the qualitative and quantitative abundances of the metal concentrations.

An examination of soil samples (Table 1) shows that the values for pH range from 7.25 to 8.26 indicating that the soils are alkaline and under such conditions the solubility of minerals decreases creating nutrient deficiencies in the soils. Plant growth is therefore limited by deficiencies in iron, manganese, zinc, copper and boron. Electrical Conductivity value ranges from 0.12 mS/cm to 0.24mS/cm. However some samples shows excess content of soluble salts which may due to excess use of fertilizer like P and K. Electrical conductivity is used to estimate the soluble salt concentrations in soil and is commonly used as a measure of salinity. Soil with EC below 0.4mS/cm are considered marginally or non-saline, while soils above 0.8 mS/cm are considered severely saline. The organic carbon (%) ranges from 0.64 to 1.04 % .The organic soil matter includes all the dead plant materials and live or dead animals. Most living things in soils, including plants, insects, bacteria and fungi, are dependent on organic matter for nutrients and energy. Soils have varying organic compounds in varying degrees of decomposition. Organic matter holds soils open, allowing the infiltration of air and water, and may hold as much as twice its weight in water. Phosphorus is one of the key macronutrient required for plant growth and metabolism. Inorganic phosphate supplied to the soil as a fertilizer is rapidly converted into unavailable form. Soluble P converted into insoluble phosphate involves microorganisms. Phosphorous in the present soils vary from 16.24 Kg/hectare to 28.34 Kg/hectare.The highest value in sample may be due to use of excessive phosphorous fertilizers. Application of phosphorus (P) is necessary for maintaining a balance between the other plant nutrients and ensuring the normal growth of the crop.Potassium fixation occurs when soils dry and the potassium is bonded between layers of clay. Under certain conditions, dependent on the soil texture, intensity of drying, and initial amount of exchangeable potassium. From the analyzed samples potassium ranges from 323.40 Kg/hectare to 548.64 Kg/hectare, indicating sufficient K in most of the sample.

Its activities has many enzyme reaction involved in the metabolism of organic acids P and N it is also involved in the photosynthesis and protein synthesis and also, manganese function along with Fe (Lindsay and Norvell, 1978) in formation of chlorophyll. Showing the variation in different parameter of soil samples from Eastern and Western region of Khed taluka

CONCLUSION

This can be concluded from this study that the available EC, PH, OC N, P, K, deficient soil is recommended rich fertilizer. To predict the probable crop response to applied nutrients. To identify the type and degree of soil related problems like salinity, alkalinity and acidity etc. and to suggest appropriate reclamation / amelioration measure. To find out suitability for growing crops.To find out suitability for irrigation.To study the soil genesis. The soil sample studied area of Khed Taluka. Dist : Pune has been found to be fit for crop productivity.

Conflicts of interest: The authors stated that no conflicts of interest.

REFERENCES

1. Swanti. A, Jain MS. Jagtap K. P. Patel Physico-Chemical Characterization of farmyard Soil used in some villages of Lunawada Taluka. Dist :Mahisagar (Gujarat) International Journal of Scientific and Research Publications. 2014**;**4:3,

2. Berger KC and Truog E. Boron determination in soils and plants. Ind. Eng. Chem. II., 1939;11:540-545

3. Chavhan DM and Wagh GS. Physicochemical and microbiological analysis of soil around Lonand City and adjoining area of MIDC. *Int. J. of Life Sci.* 2013;.1(1):67-70

4. Doran JW and Zeiss MR. Appl. Soil Ecology, 2000;15: 3-11.

5. Emnova, EE. Stiinte biol., chim. Siagr, nr, 2004;4(294): 63-70.

6. Ganguly P. Science Article, Buzzle.com 2007.

7. Graham RD and Webb, M. Micronutrients and resistance and tolerance to disease. Micronutrients in Agriculture, 2nd Edn. Soil Science Society of America Madison, Wisonsin, 1991;329-37

8. Gupta PK. Soil, Plant, Water and Fertilizer analysis. 2nd Ed. Agrobios, (India), Jodhpur. 2007:1-5.

9. Gupta UC. A simplified method for determining the hot water soluble boron in podzol soils. Soil Sci. 1967;103: 424-428.

10. Jabsing KT. Ph.D Thesis, Annamalai University, 2002.

11. Jackson ML Soil Chemical Analysis Prentice Hall of India Pvt. Ltd., New Delhi 1967; 205.

12. Joffe JS. Pedology: Pedology Publ. New Brunswick, N. J. 1949

13. Lindsay WL, Norvell WA. Development of DTPA soil test for zinc, iron, manganese and copper. Soil Sci. Soc. Am .J. 1978;42:421-428.

14. Nannipieri P, Ceccanti B and Grego S. Ecological significance of biological activity in Soil. Soil Biochemistry, Vol.6 Marcel Dekker, New York, 1990;6:293-355.

15. Olsen SR, Cole CV Watambe FS and Dean LA. Estimation of available phosphorus in soil by extraction with NaHCO3, U.S.D.A. Ciraza (Quoted from, Method of soil Analysis, C.A. Black 2nd ed.) 1165 Am. Soc. Agron., Inc. Medision Wisconsin, USA. 1954

16. Sayyed MRG, Wagh GS and Supekar A. Assessment of impact on the groundwater quality due to urbanization by hydrogeochemicalfacies analysis in SE part of Pune city, India, Proceedings of the International Academy of Ecology and Environmental Sciences, 2013;3(2): 148-159

Biofuels: Today's Need of Conventional Fuel Replacement

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ABSTRACT

A biofuel is a fuel that is produced through modern biological processes, such as agriculture and anaerobic digestion rather than a fuel produced by geological processes such as those involved in the formation of fossil fuel, such coal and petroleum from prehistoric biological matter. The biomass can be converted to convenient energy containing substances in three different ways: thermal conversion, chemical conversion, and biochemical conversion. This biomass conversion can result in fuel in solid, liquid or gases form. This new biomass can also be used directly for Biofuels.

Bioethanol is an alcohol made by fermentation mostly from carbohydrates produced in sugar or starch crops such as corn, sugarcane, or sweet sorghum. Cellulosic biomass derived from non-food sources. Ethanol production from sugarcane is currently the most attractive and alternative to fossil fuel a it achieves significant GHG emission reduction

The high cost of vegetable oils likely would push the biodiesel cost to over $1.00 per litre. Costs in a small plant would be about 10 % cents per litre higher than the tallow cost.

The current global scenario shows that our country deems very low percentage of utility and production, so it is challenge to our science and technology to build up utility and production of biofuel.

Keywords Biofuel, Bioethanol, Biomass, Biodiesel, Resources.

INTRODUCTION TO BIODIESEL

A biofuel is a fuel that is produced through modern biological processes, such as agriculture and anaerobic digestion rather than a fuel produced by geological processes such as those involved in the formation of fossil fuel, such coal and petroleum from prehistoric biological matter. biofuel can be derived directly from plant or indirectly from agricultural commercial domestic or industrial wastes [1]. Generally biofuel involve existing carbon fixation such as those that occur in microalgae through the process of photosynthesis. Biofuels are also made through the use or conversion of biomass (referring to recently living organism most often referring to plant or plant derived material). This biomass can be converted to convenient energy containing substances in three different ways: thermal conversion, chemical conversion, and biochemical conversion. This biomass conversion can result in fuel in solid, liquid or gases form. This new biomass can also be used directly for Biofuels.

Oil +alcohol $\rightarrow$ biodiesel + glycerine

Fig. 1. Biofuel separation.

Bioethanol is an alcohol made by fermentation mostly from carbohydrates produced in sugar or starch crops such as corn, sugarcane, or sweet sorghum. Cellulosic biomass derived from non-food sources, such as trees and grasses is also being developed as a feedstock for ethanol production. Ethanol can be use as a fuel for vehicles in its pure form, but it is usually widely use in USA and Brazil. Current plant designed does not provide for converting the lignin portion of plant raw material to fuel component by fermentation.

Biodiesel can be use for vehicle in its pure form, but it is usually used as a diesel additive to reduced level of particulate carbon monoxide and hydrocarbon from diesel powered vehicles. Biodiesel is produced from transesterification and is the most common biofuel in Europe.

NEED AND OBJECTIVES

As compared to India abroad foreign countries are being high biodiesel, biofuel as its major consumption of fuel for motorcorp, car's for transportation purpose. Majority in US rate of biofuel is 46% as compare to India very low percent.

The major drawback of conventional fuel like diesel, petrol is increases of carbon and it's unsaturated derivative results in air pollution which finally affect the mankind can also give birth to disease like lung cancer, Upper respiratory tract infection, Lower respiratory tract infection, Nasopharyngeal pneumonia. To overcome this type of disease and air pollution and finally to same biodiversity of nation, the very basic need is to use biofuel as a source of fuel.
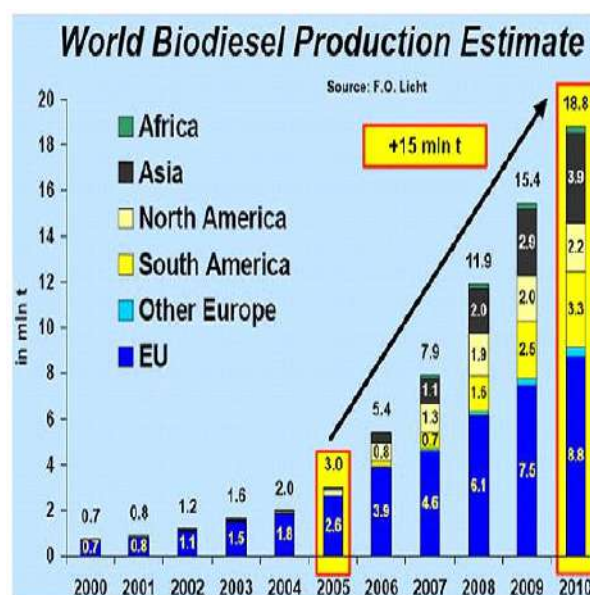
Fig. no. 2- The graph of world wise Biodiesel Production Estimation.[2]

NATURAL RESOURCES OF BIOMASS [3]

1. Sugarcane:

Ethanol production from sugarcane is currently the most attractive and alternative to fossil fuel a it achieves significant GHG emission reduction. It is obtained from renewable biomass sugarcane and biogases. Brazil and USA are the largest producers of ethanol from sugarcane with both countries according for about 86% of total Bioethanol production in 2010 in Brazil introduction of ethanol in automobiles reduced carbon monoxide emission from 50g/km in 1980 to 5.8g/k min 1995. The Brazilian economy has grown is sustainable biofuel production and consumption of ethanol.

2. Corn

The biggest biofuel in the United States right now, corn sometimes gets a bad wrap. Corn ethanol is more sustainable than petroleum, but it has been a centrepiece for debates on using agricultural crops for fuel. It's true that corn used for fuel is corn that could have been someone's dinner, but Runge explained that even once the corn oil has been extracted for ethanol, there is still a by-product of distiller corn that can be fed to animals. "It is taking food and putting it into fuel, but there is a by-product of doing that that still can go to animals. It's not one to one, but it's not all bad," Runge said. Even given the possible by-products it seems that corn is, at best, a short-term solution. Much like sugar cane, corn is one of the best options we have available now, but because the process is expensive and has high energy consumption rates, Runge felt it should be high on the list, but may fade from use over time.

3. Soy

Soy has been a popular biofuel for several years now. In a process called trans-esterification, producers squeeze the oil from seeds and use it in products such as biodiesel and jet fuel. It is a relatively easy and inexpensive rendering process, according to Runge. "You could make it in a bathtub if you really wanted to," Runge said. "I don't encourage it because there's methanol involved and methanol can make you blind. But it's very easy to do." As is the case with many agricultural crops, there is debate over the extent to which soy could be utilized. Crops like soybeans are

dietary staples to many people, and researchers are reluctant to rely too heavily on traditional food crops as fuel sources. The seed oil that goes in a gas tank could have gone to someone's stomach, and it may prove difficult to say one destination is more valuable than the other. While soy is used widely, it is not as popular as corn or sugar cane, nor does it have the amount of resources that cellulose or algae provide. This makes it a short-term solution that deserves attention, but not a higher spot on the list.

4. Camelina and Jatropha

Camelina and Jatropha are both plant-based fuel sources that are found all over the world and are the up and comers for the biofuel revolution, according to Runge. These flowering plants have an advantage over other seed-based fuels like soy because they can be grown in very dry areas. Thus, they aren't diverting land that could be used for agriculture in the way soybeans are, and can be grown in a wider variety of places. While Jatropha has the added benefit of making even poor soil more fertile over time, Peter Taglia, a scientist with the renewable energy advocacy group Clean Wisconsin, still had some doubts about it and other feedstock fuels. If a fuel is successful, Taglia thinks it could expand beyond marginal land use, and this could create new problems. "If used on more fertile lands, and either displacing food crops or native ecosystems we are, perhaps, creating additional problems in our search to replace petroleum," Taglia said. Camelina and Jatropha both offer a lot of options that other feedstocks don't provide, but much of their potential has gone unrealized, leaving them in the middle of this biofuel countdown.

5. Animal Fat

Pigs, chickens, and cows -- all are fairly common in everyday life, but they could have uncommon potential for biofuel. Leftover fat from animal food products can be rendered into oil and then used as a fuel for cars and trucks. It may seem like an odd source, but Taglia believes the benefits of animal fat are large. "Any time you can use something that was either going to a landfill or was going to be, and that's not necessarily the best use of it, then that's something we really want to be used efficiently to produce fuel," Taglia said. But gathering fat isn't as easy as picking it up from the landfill or your local fast food restaurant.

Animal fat is used in many industrial products, so there would be competition for resources. This combined with the natural limit on the resource means animal fat could never displace petroleum by itself, Taglia said, and this placesk animal fat low of the list of biofuels.

Table no.: The given table shows that the detailed agricultured biofuel potential in India[5]

Sr no.	Crop type	Oil yield potential '000l/ha
1	Micro algae	47.5 – 142.5
2	Oil palm	6.o
3	Corn	0.2
4	Rapeseed	1.2
5	Sunflower	1.0
6	Soya bean	0.5

METHOD OF PREPARATION OF BIOFUEL
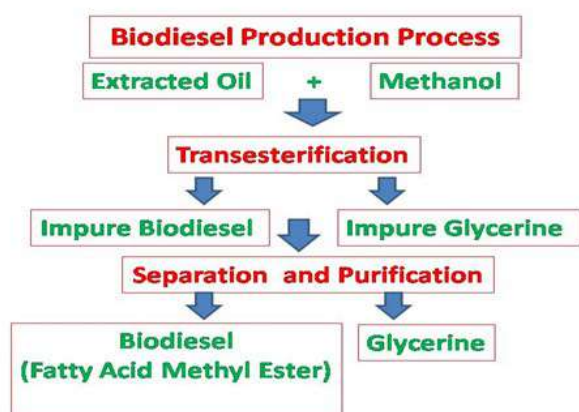
How much does 1 Litre of biodiesel cost?

The high cost of vegetable oils likely would push the biodiesel cost to over $1.00 per litre. Costs in a small plant would be about 10 cents per litre higher than the tallow cost, although, when using waste cooking oil, this additional cost would be largely offset by the cheaper feedstock. •

What are some of the benefits of biodiesel?

It's safer to handle and has virtually the same energy efficiency as petroleum diesel. In addition it has lubricity benefits that fossil fuels do not. Biodiesel blends as low as B2 have been found to significantly reduce the amount of toxic carbon-based emissions.

ADVANTAGES OF BIOFUELS [4]

1. Cost Benefit: As of now, Biofuels cost the same in the market as gasoline does. However, the overall cost benefit of using them is much higher. They are cleaner fuels, which mean they produce fewer emissions on burning. Biofuels are adaptable to current engine designs and perform very well in most conditions. This keeps the engine running for longer, requires less maintenance and brings down overall pollution check costs. With the increased demand of Biofuels, they have a potential of becoming cheaper in future as well. So, the use of Biofuels will be less of a drain on the wallet.

2. Easy to Source: Gasoline is refined from crude oil, which happens to be a non-renewable resource. Although current reservoirs of gas will sustain for many years, they will end sometime in near future. Biofuels are made from many different sources such as manure, waste from crops and plants grown specifically for the fuel.

3. Renewable: Most of the fossil fuels will expire and end up in smoke one day. Since most of the sources like manure, corn, switchgrass, soya beans, waste from crops and plants are renewable and are not likely to run out any time soon, making the use of Biofuels efficient in nature. These crops can be replanted again and again.

4. Reduce Greenhouse Gases: Fossil fuels, when burnt, produce large amount of greenhouse gases i.e. carbon dioxide in the atmosphere. These greenhouse gases trap sunlight and cause planet to warm. The burning of coal and oil increases the temperature and causes global warming. To reduce the impact of greenhouse gases, people around the world are using biofuels. Studies suggest that Biofuels reduces greenhouse gases up to 65 percent.

5. Economic Security: Not every country has large reserves of crude oil. For them, having to import the oil puts a huge dent in the economy. If more people start shifting towards biofuels, a country can reduce its dependence on fossil fuels. More jobs will be created with a growing biofuel industry, which will keep our economy secure.

6. Reduce Dependence on Foreign Oil: While locally grown crops has reduce the nation's dependence on fossil fuels, many experts believe that it will take a long time to solve our energy needs. As prices of crude oil is touching sky high, we need some more alternative energy solutions to reduce our dependence on fossil fuels.

7. Lower Levels of Pollution: Since Biofuels can be made from renewable resources, they cause less pollution to the planet. However, that is not the only reason why the use of biofuels is being encouraged. They release lower levels of carbon dioxide and other emissions when burnt. Although the production of Biofuels creates carbon dioxide as a byproduct, it is frequently used to grow the plants that will be converted into the fuel. This allows it to become something close to a self sustaining system.

DISADVANTAGES OF BIOFUELS [4]

1. High Cost of Production: Even with all the benefits associated with Biofuels, they are quite expensive to produce in the current market. As of now, the interest and capital investment being put into biofuel production is fairly low but it can match demand. If the demand increases, then increasing the supply will be a long term operation, which will be quite expensive. Such a disadvantage is still preventing the use of Biofuels from becoming more popular.

2. Monoculture: Monoculture refers to practice of producing same crops year after year, rather than producing various crops through a farmer's fields over time. While, this might be economically attractive for farmers but growing same crop every year may deprive the soil of nutrients that are put back into the soil through crop rotation.

3. Use of Fertilizers: Biofuels are produced from crops and these crops need fertilizers to grow better. The downside of using fertilizers is that they can have harmful effects on surrounding environment and may cause water pollution. Fertilizers contain nitrogen and phosphorus. They can be washed away from soil to nearby lake, river or pond.

4. Shortage of Food: Biofuel are extracted from plants and crops that have high levels of sugar in them. However, most of these crops are also used as food crops. Even though waste material from plants can be used as raw material, the requirement for such food crops will still exist. It will take up agricultural space from other crops, which can create a number of problems. Even if it does not cause an acute shortage of food, it will definitely put pressure on the current growth of crops. One major worry being faced by people is that the growing use of Biofuels may just mean a rise in food prices as well.

CONCLUSION

On the basis of above study we have to conclude that the utility and production of biofuel in our country is very poor. All the required natural sources or biodiesel production are available in all over India e.g. Sugarcane, Soy, Corn, Animal fat but current global scenario shows that our production and utility is poor so it is challenge to our technology to build up production of biofuel and hence also utility. The major drawback of conventional fuel like petrol, diesel, gasoline is increase of various harmful disease and also air pollution, hence it required to control it to save biodiversity of nation, the very basic need is to use biofuel as a source of fuel.

Conflicts of interest: The authors stated that no conflicts of interest.

REFERENCES

1. Mukta M. Abhyankar , Supriya G. Jagtap, Textbook of Natural Drug Technology, Semester VII. Page no- 10.32.
2. https://images.search.yahoo.com/yhs/search;_ylt=A8 6J77iHldaoQ4AngoPxQt.
3. https://w.w.w.seeker.com/top10_sources_for_biofule 1769457447.html.
4. Juliet Ben lwo vasilde manovic Philip conghurst by renewable and sustainable energy Reviews, volume 63, sep.2016, page no- 172-192.
5. Aculom G, Purohit P, Fisher G, (2013), Second generation biofuel potential in India, sustainability concideration 8th edition, *conference on sustainable development of energy, water and environment system, Dubrovnik Croatia, Dubrovnik Neretva.*

RESEARCH ARTICLE   **OPEN ACCESS**

The Impact of Global Warming on the Biodiversity

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ABSTRACT

Environment means to the sum total of condition, which surrounds point in space and time. But the term 'Environment' is changing from place to place and time to time. Therefore, in the ancient period the term environment was used in limited sense. It was referred to only physical aspects of the earth's land, air and water as biological communities. With the passage of the time, the term environment was extended the environment through their social, economic and political activities. Therefore, today the term environment is used for climate change, global warming, natural disasters, and soil and land degradation, loss of biodiversity, air and water pollution etc. Moreover, it is, nevertheless, clear that humans are responsible to change the earth's climate in different ways. The production of carbon dioxide, cutting of forests and agriculture which produces methane, another greenhouse gas (GHG) etc. are responsible for global warming. It is scientifically supported global climate change (GCC) is an emerging issue which is proved as a peril to ecosystems, nonhuman species populations, and human populations etc. Moreover, It is responsible for reduced regional food yields, freshwater shortages, increased frequency of extreme weather events, coastal population displacement, changes in the ecology and geography of infectious agents, declines in farming community incomes, and biodiversity losses with accompanying disruption of ecosystem functions. It is nevertheless, true that these consequences are raised due to the global warming. Therefore, this research paper is an endeavor to illustrate the adverse impact of global warming on the biodiversity.

Keywords: Climate, Global warming, biodiversity, ecosystem, surplus, disease, disaster, penalty, freedom, control, impact, remedy, judiciary, fine, constraint, policy, public, constitution, right and legislation.

INTRODUCTION

Global warming is the increasing of temperature of the earth. It is due to accumulation of greenhouse gases in the atmosphere. The climate change is the general term for any persistent change in climate, occurring over decades. The scientists are of the view that this global warming is resulted due to the enhanced greenhouse effect [1]. The projected increase in atmospheric concentrations of anthropogenic GHGs could have an adverse impact on the earth's temperatures [2]. It is proved scientifically beyond the reasonable doubt that global warming is one of the significant perils to the, biodiversity. The projected temperature increase of 1 to 3.6°C over the next 100 years could exceed rates of change for the last 10,000 years [3].

Objectives
Following are the objectives of the research paper.

1. To study the impact of Global warming.
2. To understand the impact of Global warming on biodiversity.
3. To give suggestions to face the impact of Global warming on biodiversity.

Nature and Scope of the study
The nature and scope of the study covers the comprehensive analysis of the current state of the impacts impact of Global warming in India and the way how its measures became more effective. The main points to be focused through the current research paper are the definition of Global warming, its background, review of literature, international aspect, judicial contribution, constitutional provisions, impact on biodiversity and suggestions to tackle it successfully. The Biological Diversity Act, 2002, Article 21, 48A, 51A[4] and the impact of Global warming on biodiversity is the scope of this research paper.

Hypothesis
Following is the hypothesis to guide the study.
1. There is an adverse impact of Global warming on the biodiversity.

METHODOLOGY

This is the conceptual research paper. Therefore, the secondary data like the books, newspapers, and websites, views of the authors expressed through the research papers, journals, case laws and commentary on the concept of the impact of the biodiversity are studied and necessarily used to complete the research paper.

1. Meaning and definition of Global Warming
Global Warming means a gradual increase in the overall temperature of the earth's atmosphere generally attributed to the greenhouse effect caused by increased levels of carbon dioxide, CFCs, and other pollutants. The Global warming is defined as "an increase in the earth's average atmospheric temperature that causes corresponding changes in climate and that may result from the greenhouse effect."[5]

2. Meaning and definition of Biodiversity
Biodiversity means the variety of plant and animal life in the world or in a particular habitat, a high level of which is usually considered to be important and desirable for the survival of lives on the earth. There are three types of Biodiversity i.e. genetic diversity, species diversity, and ecosystem diversity. International Council for Bird preservation (1992) defines that Biodiversity is the total variety of life on earth. According to the Biological Diversity Act, 2002, Biological Diversity means the variability among living organisms from all sources and the ecological complexes of which they are part and includes diversity within species, between species, and of ecosystems [6].

RIGHT TO LIFE

1. Article 21 reads as:
"*No person shall be deprived of his life or personal liberty except according to a procedure established by law.*"
According to Bhagwati, J., Article 21 "embodies a constitutional value of supreme importance in a democratic society." Iyer, J., has characterized Article 21 as "the procedural *magna carta* protective of life and liberty.

　　　　　　http://www.irjse.in

2. Judicial interpretation for biodiversity protection

It is however, indisputable that the right to life as enshrined in the article 21, includes within its ambit something more than survival and not mere an animal existence. It therefore, nevertheless, includes all the aspects of life necessary to make a man's life meaningful, complete and worth living.The Supreme Court in Maneka Gandhi Vs. Union of India[7], the Board of Trustees Vs. Dilip [8] and in Ramasharan Vs. Union of India [9], held that the right to includes all that gives meaning to a man's life, for example, his tradition, culture, heritage and protection of that heritage in its full measure[10].

The Supreme Court in Sachidananda Pandey Vs. State of West Bengal & Ors[11] held that "Whenever a problem of ecology is brought before the Court, the Court is bound to bear in mind Article 48 A of the Constitution of India, which enjoins that the state shall endeavor to protect and improve the environment and to safeguard the forests and wild life of the country, and Article 51A (g), proclaims it to be the fundamental duty of every citizen of India to protect and improve the natural environment including forest, lakes, rivers and wild life, and to have compassion for living creatures"[12].

THE IMPACT OF GLOBAL WARMING ON THE BIODIVERSITY

Biodiversity is defined as "the variety and variability among living organisms and the ecological complexes in which they occur[13]." The air we breathe, the soil we farm, the plants we harvest, and the water we drink all depend on ecosystem services. A significant portion of this value is generated by biological sources. The global warming is a significant determinant of vegetation patterns. It has a significant influence on forest distribution, species dominance, and plant productivity and, in general, ecology of forests. The biodiversity is closely associated with favorable climate on the earth. But change in climate will alter the biodiversity. Even if animals and species are valued solely by reference to human preferences, consideration of animal welfare dramatically increases

the argument for aggressive responses to climate change[14].

It is found that, plants and animals are very sensitive to global warming. Their range move when the climate patterns change-species die out in areas where they were once found and colonize new areas where the climate becomes newly suitable. Moreover, it is traced from the fossil records that some species have become completely extinct because they were unable to find suitable habitat when climate change made their old homes unlivable. For forestry, the climate change-induced modifications of frequency and intensity of forest wildfires, outbreaks of insects and pathogens, and extreme events such as high winds, may be more important than the direct impact of higher temperatures and elevated CO2[15].

According to IUCN Red List of Threatened Species, there are at least 1,141 of the 5,487 mammals on Earth are known to be threatened with extinction, and 836 are listed as Data Deficient. The main factor that causes endangered species is loss of habitat. Whenever we cut down a tree or destroy a forest to build a shopping mall we are destroying the homes of several species. It also means that a food source for another animal may be destroyed as well. In some areas certain species are hunted for sport faster than they can reproduce[16].

Ecosystems, with their specific flora and fauna, are especially vulnerable to negative impact from global warming. In India the major threat to such Biodiversity rich regions is due to disproportionate destruction of forest and even climate change negatively affects forests including biodiversity wiping out the species[17].

CRITICISM

The Global warming is always criticized on certain grounds. The depletion of biodiversity also leads to extinguishment of certain species, so it has become an urgent issue necessary to be tackled. The most serious defect in the Global warming is the failure of the state to mitigate its hazardous impact on the biodiversity. The state is vulnerable so as to achieve the

technological mastery to face the unfavorable impacts of the global warming. The global warming impact is totally contrary to the upliftment of human status. Thus, the rich biodiversity of India is under severe threat owing to habitat destruction, degradation, fragmentation over-exploitation of resources and global warming. Increasing the speed of economic growth without exhausting the resources and at the same time fulfilling the basic needs of a large growing population is one of the great challenges facing India[18].

Hypothesis

There is an adverse impact of Global warming on the biodiversity.

It is verified and found true on the basis of the secondary data.

FINDINGS AND CONCLUSIONS

The Global warming has different hazards on the biodiversity. It is found that 50% of crop yield from the genetic resources of the plant. Moreover, the medicinal bio-compounds from the coral reefs are the sources of medicine for cancer and other diseases. It may be concluded that the biodiversity has the significant impact of global warming. It is found through this research paper that if state failed to take appropriate measures to deal with the effects of global warming on biodiversity, half of the common plants on earth and one third of animals will be extinct up to 2080. Therefore, this research paper has revealed the fact that the biodiversity is now at the mercy of the global warming. Since, the human survival in future merely depends upon the technological progress of the state so as to face the hazardous impacts of the global warming. Thus, however, it is undisputable that the poor in this regard may be more vulnerable and easy prey to global warming. Since, the conservation of biodiversity has gained worldwide momentum.

Conflicts of interest: The authors stated that no conflicts of interest.

REFERENCES

1. Dr. Sukanta K. Nanda, *Environmental Law*, 1st edition, Allahabad: Central Law Publications, 2007, p. 324.
2. Ramachandran R. 'The Play of Environmental Politics', *the ICFAI Journal of Environment Law*, , Jan. 2003,Vol. 11.
3. Stuart E Eizenstat, "The Kyoto Protocol: A Framework for Action", Focus, *Global Issues Electronic Journal,* May, 1998, pp. 6-7.
4. Article 48 A and 51 A (g) have been inserted in the Constitution of India by 42nd amendment in 1976.
5. Global warming. (n.d.). Dictionary.com Unabridged. Retrieved January 5, 2018 from Dictionary.com website http://www.dictionary.com/browse/global-warming.
6. Clause (b) of section 2 of Biological Diversity Act, 2002
7. AIR 1978 SC 597
8. (1993) 1 SCC 124
9. (1989) Supp. (1) SCC 251
10. Ibid
11. AIR 1987 SC 1109; 1987 (2) SCR 223; 1987 (2) SCC 295; 1987 (1) JT 425.
12. Ibid
13. Dr. Sukanta K Nanda, "*Environmental Law*", Central Law Publications, I Edition, Allahabad , 2007, pp. 338-339
14. Hsiung Wayne and Sunstein Cass R. Climate Change and Animals, University of Pennsylvania Law Review, Vol. 155, No. 6, Symposium: Responses to Global Warming: The Law, Economics, and Science of Climate Change (Jun., 2007), pp. 1695-1740
15. Andrei P. Kirilenko and Roger A. Sedjo, Climate Change Impacts on Forestry, Proceedings of the National Academy of Sciences of the United States of America, Vol. 104, No. 50 (Dec. 11, 2007), pp. 19697-19702.
16. http://www.ask.com/question/how-many-endangered-species-are-there accessed on 10/01/2018
17. Mehdi, "Climate Change and Biodiversity: India's Perspective and Legal Framework", *JILI,* Vol. 52:3 & 4, p. 352
18. Rathore MS, *Environment and development: Areas of Conflict and Convergence*, Environment and development, Jaipur and New Delhi: M S Rathore, Rawat Publications, 1996, p. 20

An Analysis and Perspective related with Global Warming

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ABSTRACT

Actuaries are becoming more aware of the combined impact of climate change and limitations of resources — two separate and very significant issues — putting at risk the sustainability of the current socio-economic systems that support our way of life. Climate change is more than global warming. The rise in average temperature is only one indicator of broader changes also translating into extreme temperatures, drought, flooding, storms, rising sea levels, impacts on food production, and infectious diseases. Although the scientific community has been aware of the link between greenhouse gases (GHGs) and climate change for many years, world leaders have been slow to react and implement measures to mitigate the risks. he potential impact on actuarial methods and assumptions, especially future growth expectations, is pervasive in the work of actuaries and affects traditional life and non-life, health and pensionsareas, investment practices, and newer areas like enterprise risk management.

Keywords: Stratospheric-ozone depletion, photochemical oxidants, aerosols, acidification, toxic metals, ecologically active substances, hot-spot regions, chemical time bombs, long-range transports of air pollutants, organic substances.

INTRODUCTION

Extensive discussions are taking place throughout the world in all forms of media on the subjects of global warming and climate change. These discussions point to the global dangers posed by the earth's warming. Global warming will not only be felt many decades from now — it is already happening and its impacts are clearly visible.

Globalizing the environmental policy agenda must not divert attention from concerns about unresolved, regional environmental problems. Global warming, albeit with unclear manifestations in regional and local climates, is likely to aggravate impacts from already existing environmental stress (including acidification, rising tropospheric oxidant levels, stratospheric ozone depletion, the flux of heavy metals in soils and waters). International law not only recognizes that there is evidence that climate change is caused by human beings, but generally that human beings or countries have been the greatest contributors to climate change

METHODOLOGY

What is Climate Change?

Climate change is the subject of how weather patterns change over decades or longer. Climate change takes place due to natural and human influences. Increases in global temperatures may have different impacts, such as an increase in storms, floods, droughts, and sea levels, and the decline of ice sheets, sea ice, and glacie

Extreme events

It is difficult to blame a particular extreme weather event (such as a heat wave, flood-producing storm, or drought) on global warming, since weather fluctuates naturally. However, climate theory and models predict that global warming will increase the frequency and/or intensity of some types of extreme events.

Indirect and Direct Effects

Among the most obvious areas of concern to societies worldwide, with respect to facing the effects of climate change, is agriculture. Perhaps the challenges involved are often seen as one of agricultural sensitivity to "global warming as such", i.e., to increasing temperatures in terms of global averages. alues of meteorological parameters (temperature, precipitation, etc), the real implications of climate change may very likely pertain to shifts in the frequency distribution of these parameters.

Recognition of Risks

Due to the nature of the subject, the international discussions on climate change are driven by worldwide climate scientists. There would be a few members of the CIA who would have a good understanding of the issues involved. But the majority of actuaries may not have such understanding. Global warming is no longer just a pre-diction—it is actually happening. It is undisputed that the average temperature at the surface of the Earth has increased over the past century by about 1°F(0.6°C), with both the air and the oceans warming. Precisely determined how much of the recent warming was caused by human activities, the consensus among climate scientists is that most of the warming over the past 50 years was probably caused by human-produced greenhouse gases.

Torrential rains and flooding

According to the available data, a significant increase in the intensity of precipitation events occurred over the second half of the 20th century. Since higher temperatures speed up evaporation and increase the amount of water vapor in the air, leading to heavier downpours.

Process of Global Warming

Earth receives energy through radiation from the sun. Play an important role of trapping heat, maintaining the earth's temperature at a level that can sustain life. This phenomenon is called the greenhouse effect and is natural and necessary to support life on earth without the greenhouse effect, the earth would be approximately 33°C cooler than it is today. In recent centuries, humans have contributed to an increase in atmospheric GHGs as a result of increased fossil fuel burning and deforestation. The rise in GHGsis the primary cause of global warming over the last century.

Global action to address climate change

1. UN Framework Convention on Climate Change-
There is currently only one single universally legally-binding treaty governing international action to address climate change. This is the UN Framework Convention on Climate Change. They discussed the

need for a binding commitment to and the setting of measurable objectives and timelines for greenhouse gas reductions by developed countries, establishing a financial mechanism for climate action, ensuring technology transfer from developed to developing countries.

2. International Human Rights Law

Human Rights and Climate Change – Recent Initiatives United Nations Human Rights Council addressed the link between climate change and human rights in a specific resolution. In the resolution, the Council also recognized that "human beings are at the centre of concerns for sustainable development and that the right to development must be fulfilled so as to meet equitably the development and environmental needs of present and future generations." Several other international and regional human rights bodies have also addressed climate change. For example, the Committee on the Elimination of Discrimination against Women adopted a Statement on Gender and Climate Change and the plenary parent body of the Inter-America Commission for Human Rights, the General Assembly of the Organization of American States also adopted a resolution on Human Rights and Climate Change.

3. Mitigation and Adaptation for Climate Change

Keeping Global Warming under2°C . An international agreement had been reached at Copenhagen that global warming should be limited to 2°C.

Mitigation Measures for Reducing Carbon Emissions

The need for reducing the CO2 equivalent emissions will affect many sectors of the economy: energy creation, transport, buildings, industry, agriculture, human settlements

Energy Creation

Availability of adequate energy supply is fundamental to modern living. Currently, a major portion of the energy is generated using fossil fuels—coal, oil, and natural gas (in decreasing order of CO emissions). These will need to be replaced by low or zero carbon fuels, such as wind, solar, and nuclear

Transportation

Advancements will need to take place in areas of energy efficiency, improved vehicle performance, use of electrical vehicles,

Carbon Pricing

Cap and Trade is a carbon pricing system where the government sets a cap on the amount of emissions, and companies can meet the cap by reducing their emissions or paying another entity to do so.
Trading of emission allowances enables emissions to be reduced at a lower cost than requiring each company to lower their own emissions.

Carbon Tax

This is a tax on carbon emissions, based on the carbon content of the fuel. This would, for example, add more to the price of coal than to the price of gasoline, due to the higher carbon content of coal

CONCLUSION

Global warming has wide-ranging effects on many aspects of human life. It threatens economies, lives and traditional ways of life. Although individual events or phenomena may not always be easy to link to global warming, the increase in frequency and intensity of such phenomena, and their simultaneous occurrence around the world, provides stronger evidence for such a linkage.

Conflicts of interest: The authors stated that no conflicts of interest.

REFERENCES

1. IPCC. 2001.Climate Change 2001: TheScientific Basis.Contribution of Working Group I to the Third Assessment Report ofthe Intergovernmental Panel on ClimateChange. Edited by J.T. Houghton, Y. Ding,D.J. Griggs, M. Noguer, P.J. van der Linden,X. Dai, K. Maskell, and C.A. Johnson.Cambridge and New York: Cambridge University Press. (http://www.grida.no/climate/ipcc_tar/wg1/index.htm)

2. National Climatic Data Center .2005 .ftp://ftp.ncdc.noaa.gov/pub/ data/ anomalies/ annual_land.and.ocean.ts

3. Lindberg S.E., Zhang H., Gustin M., et al. (1999) Increases in mercury emissions from desert soils in response to rainfall and irrigation. Journal of Geophysical

4. Lindgren E. (2000) The New Environmental Contextfor Disease Transmission.PhD Thesis, Department of Systems Ecology, Stockholm University, ISBN 91-7265-117-2. [A document that addresses the effects of climate change on observed and potential spread of, inter alia, insect-borne diseases (such as tick-borne encephalitis)].

Environmental pollution and Global Warming

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ABSTRACT

Everything that surrounds us is directly or indirectly connected to the environment. Not only the man, but also other living beings as well as the nature (volcanic eruptions, earthquakes) have effects on environmental pollution. Environmental pollution is present from the very beginning of life, but today it is a serious problem that threatens the survival of mankind. During the preparation of scientific research, we noticed a problem: "What is the influence of the waste management on the environment?" Today, every person living on planet Earth is worried about environmental pollution because the consequences faced every day, through the air we breathe, the food and water we consume, through pollution and radiation we are exposed to. Also, the consequences of environmental problems are manifested through the lack of natural resources, extinction of plant and animal species, as well as the problems in the global ecosystems and bio- chemical processes. Based on the research problem we can hypothesize: Yes, waste management has a great impact on the environment.

Keywords: environment, waste management, enviro-nmental pollution, global warming.

INTRODUCTION

The man, along with all other living beings from the beginning of its existence is closely linked with the entire inanimate and living nature that surrounds it. This interaction is the basis of the whole modern right of environmental protection. Through his own development, the man developed his interest in the way and manner that would harmonize with the nature that surrounds it, to ensure the conditions necessary for their survival.

With each new discovery (ranging from tools for tillage and wheel all the way to modern computer technology) man makes bigger part of the eternal desire to reconcile nature and its needs. Contemporary urban, industrial, economic and technological development has provided great benefits to man, but the industrial air and water pollution, uncontrolled deforestation and their conversion into agricultural land, destruction of the ozone layer and global warming of the planet followed by climatic changes, the accumulation of various wastes, including radioactive as well as the eradication of certain plant and animal species, are just some of the negative consequences of human activities, which, however, seriously endangering his own survival. At present time, the protection of the environment is of great importance in the prevention and elimination of these contradictions. The right to protect the environment today should be seen as a unique supranational (international), national and local unit. Therefore, in order for the normative framework to succeed, actions must be taken at the universal, national, regional and local levels.

Today we can say that we live in a world of waste; because of population growth and production increasing amounts of waste that makes landfill are becoming more numerous and increasingly de- grade the environment. Every day a huge amount of waste, equally as in the villages and in agricultural areas is produced. Every year, about 10 million tons of oil products reaches rivers and oceans and has more than 500 billion tons of industrial waste. Industrial facilities and transport throw into the atmosphere about a billion tons of aerosols and ash. At the landfill waste is collected for years. In the wild landfill reaches up to 70% of total waste. The biochemical processes of decomposition of waste adversely affect the environment. As for municipal waste that contaminates the soil and plants, air, groundwater and surface water on them in huge quantities reproduce rats, mice and insects, which contributes to the spread of infection. This new situation poses a threat to human health, for both present and future generations. This imposes the problem of protecting the environment through waste management. The composition of waste is very different and includes municipal solid waste, industrial, and agricultural, medical, electronic and other wastes of mixed composition.

A particular problem is hazardous waste (chemical, biological and nuclear) with a strong polluting effect and the many negative consequences for human health and the environment. In the early 90s of the 20th century in developing countries, there are 100 - 330kg of solid waste per capita in the European Union, the number was 414, and in North America 720kg. The global problem of all countries of the world is that there is an increase of waste, both in the amount of waste produced, as well as the amount of waste per person. The big problem is that this waste is not processed, instead, it is disposed of in landfills that are huge and located in the vicinity of cities and represent a dangerous pollutants to air, water and land.

ENVIRONMENTAL POLLUTION

The result of deteriorating environmental situation in various countries and regions where environmental pollution is the most intense climate is warming, ozone layer is depleting, desertification. According to the definition adopted by the UN organization, "pollution are exogenous chemical substances encountered on a suitable place, at the appropriate time and in inadequate quantities.[1]. According to the analysis (taken in early 20th century), it is concluded that the most polluted spheres are atmosphere and hydrosphere. Even the state of cosmic space around our planet raises serious concerns. In order to define the concept of the environment, we must consider the basic ecological unit that has its own laws, which is characterized by complex factors of animate and inanimate nature. This unit is called an ecosystem. The man as a conscious being has a great influence on the environment. According to the methodology of the World Health Organization, there are 26 risk factors to health, some dating from the environment that are considered to cause many diseases in the population of children aged 0 to 19 years.

The impact of economic and other activities on the environment may be different in character: direct (immediate) and indirect synergy. From the perspective of environmental effects of pollution are usually described in terms of which we have already pointed out: degradation, devastation, endangering the environment, in different time periods. It is possible to perceive the effects of pollution in air, water, soil,

wildlife, human health, and so on. We can speak of long-termed and short- termed effects of pollution. It can be very important to understand the concept of the working environment, if it is connected with the concept of environmental protection in the context of discussions on environmental management. This can be done for many reasons. First, because of the connection of the working environment as part of the environment, there are connections and processes related to the operation and are of importance to the issue of the environment. There are several provisions in the law on safety and health at work and other regulations governing this area, which are directly relevant to the understanding of the relationship between the working environment and the environment. It is necessary to know not only the notion of working environment but also to bear in mind the definition of a number of other terms such as "hazardous materials", "danger", "dangerous phenomenon", "risk", "risk assessment", and so on. According to this law work environment defines the working environment in which the work is performed under specific operating conditions in the workplace, working procedures and relationships in the work process. Unfavorable changes in the environment caused by human activities, causing a change in the inflow of energy, radiation levels physicochemical and microbiological composition of environmental pollution can be defined. The harmful effect of pollution, their effect is manifested in the anatomical and morphological structure, metabolism, growth process, at all levels of cellular organization, from the molecular to the cellular level, through individual and population to biogenesis and ecosystems.

WASTE MANAGEMENT

In the Middle Ages, food waste was dumped on the streets, so the rodents and insects transmitted many infectious diseases and dangerous epidemics. Today, because of inadequate treatment of waste could be a higher number of infectious diseases. The general interest of society in our country, governed by the Law on Waste Management is the management of waste. The objective of this law is to provide and ensure the conditions for waste management in a way that does not endanger human health and the environment.
Law relating to waste management, is based on the following principles:

1. The principle of optimal choice of options for the environment
2. The principle of proximity and regional approach to waste management
3. The principle of hierarchical waste management
4. The principle of accountability
5. The 'Polluter Pays' Principle.

Prevention of waste, reuse of waste and recycling, separation of recyclable materials from the waste and the use waste as an energy resource development processes and methods of waste disposal, remediation of unregulated dumps, and developing awareness of waste management involves waste management. The concept of waste management refers to the activities of collecting, transporting, sorting, recycling, disposal, tracking and monitoring of waste. The biggest problem is the collection of waste for recycling, ie, waste sor- ting, because some parts of the process must be performed manually, which increases the cost of recycling. But there are problems with the categorization of garbage, so let's say PET bottles are not the same category of waste as PVC containers and cannot be recycled together. As the best solution for the protection of the environment from waste, would be not to use products or packaging that is biodegradable and does not release toxins into the atmosphere. As a consumer, you can choose the product you are buying. Products with excess packaging not only pollute the environment, packaging is included in the price you pay, and the placing of 30 grams in a package which can hold 60 grams of leads to visual misleading. Japan introduced legislation on recycling in 1997 and currently there are 44 categories of waste. Every resident of Japan has received a 27-page instruction on the procedure for sorting their waste. There are categories describe to you in detail, so if you have a lipstick that you spent, he throws himself into the category of combustion, but after you remove it from the packaging and plastics dumped into the category of 'small plastic and metal. Japan is a country-specific, because with 127 million people, on average, 336 people live in an area of 1 km2. Logically Japan has to take care of as little use of land for the purpose of disposal.

In the world's major cities, there are 3.5 kg of waste per capita a day. With increased population and living standards, the waste also increases. According to the Basel

Convention, the world annually produces about 4oo.ooo.ooo tons of hazardous waste. Military waste and radiochemical industry, which uses various raw materials and products that have hazardous properties is especially present. The production of hazardous waste can occur in combat, destruction of chemical plants in NHB accidents (transport, storage, accidents, natural disasters, natural disasters) and so on. The harmful and dangerous contaminants working environment includes the physical (solid waste, dust, noise, vibrations), chemical (aerosols, gases, vapors, fumes, dust, waste), radiation (ionizing, UV, oils, infrared, radar, laser , ultrasound, x-ray) and biological (viruses, bacteria, mold, fungi, parasites, insects, rodents). Anthropogenic substances go into the environment in different ways. Wastewater is discharged into surface and underground waterways and basins. Solid waste is stored in special landfills, buried and deposited in abandoned mines. Agriculture uses fertilizers and pesticides (about 70,000 different harmful and hazardous substances, and the list is supplemented annually with new 900-1000 terms). Synthesized new compounds that are not found in the working environment and living organisms are able to decompose (PVC materials). It is estimated that close to 40 million plastic bottles and bags, a large number of lost and discarded fishing nets, nylon and others float on seas and oceans.

WASTE MANAGEMENT STRATEGY

The collection, transport, storage and treatment of waste carry a number of risks to safety and health of employees in the waste industry".[14] The system activities and activities which include the prevention of waste by reducing packaging materials, waste reduction, developing a habit of sorting waste in the population represents waste management.

Waste Management Strategy provides us with the following activities:

1. The introduction of formal legal mechanisms, such as the acquisition of knowledge in the field of waste management.

2. Acquisition and improvement of education and training of persons who manage waste

3. Establishment of a national body responsible for the development of educational programs and training in the field of waste management. There are ways

to intensify the use of waste, to improve the quality of the processed raw materials and the number of participants in the recycling process which leads to greater environmental protection. Most countries accept the strategy of the European Union relating to:

4. Research and development of new technologies for recycling,

5. optimization of the system of collecting and sorting waste,

6. reduction of external costs of re-use of waste,

7. computerized systems of waste management,

8. exchange and sale of scrap material.

CONCLUSION

The environment is changing through development of the industrial revolution and the beginning of the use of fossil fuels. people are thin- king about this problem more and more, becau- se it should not allow the industry to continue to develop and harm the environment. This way of thinking has led to the emergence of the concept of sustainable development. This concept impli- es the continued development of the industry in a way that has minimal environmental impact. To change the quality of the ecosystem brings harmful effects of pollutants present in the environment and thus to increase the potential negative impacts on human health in several ways. The survival of man and nature are brought into question trough this industrial mode of production that was supposed to make the man the master of nature. Pollution of the basic elements of the environment (air, water and land) reached alarming results. Landscapes and spaces that are beyond the reach of human activities still exist. People recognized and legally protected such natural goods. The life and working life of the man carried out in the urban areas and industrial zones, and only rare moments of rest in an oasis of pure nature. Some types of waste represent a major potential threat to the environment and human health. The company did not immediately and fully understand this danger. In many countries there are still no regulations on waste management. The amount of hazardous waste has increased dramatically in the last period due to different types of pesticides that are applied in agriculture and

industrial waste containing toxic and cancerogenicc substances. Worsening situation of global environmental global warming, ozone layer depletion, acidification of the environment with the occurrence of acid rain and its consequences of global environmental problems: deforestation, soil degradation, loss of biodiversity and stocks of clean drinking water is the result of deteriorating environmental situation in different countries and regions where environmental pollution is most intense. Reducing environmental pollution is an important goal of sustainable waste management. Recycling is one of the useful methods aimed at maximum utilization of energy and raw materials from waste. Most countries are op- ting for recycling because in addition to eliminating waste they see economic solutions. Recycling keeps raw materials and energy. From discarded and useless products obtained raw materials that would be in a different situation should draw from natural resources. The growth of population and production of larger quantities of waste to landfill seems to become increasingly numerous and increasingly degrade the environment. Landfills cover large areas of arable land, grow in uncontrolled dumps with high risk of miraculous situation and require huge costs of eliminating them. In order to reduce the amount of generated waste it is necessary to improve waste management strategies starting from minimizing waste at source, via rebooting the use of secondary raw materials, recycling and disposal too.

Conflicts of interest: The authors stated that no conflicts of interest.

REFERENCES

1. Bjelajac Z. Ecological policy of EU and its criminal-legal draft, MP4, 2011.
2. Heleta M. System management projecting of envi- ronmental, University Singidunum, Beograd 2010.
3. Jovanovic L. Ecomanagement in function of envi- ronment protection in towns. Ecological movement of Novi Sad, Novi Sad 1999.
4. Jovanovic L. Ecomanagement in function of envi- ronment protection in towns. Ecological movement of Novi Sad,
5. Jovanovic L. Recycling as an important part of su- stainable developmentISO14000, Aranđelovac 2000.
6. Jovanovic L. Recycling as an important part of su- stainable development ISO14000, Aranđelovac 2000.
7. Kolomeiceva-Jovanovic L. Chemistry and enviromen- tal protection, Belgrade 2010,
8. Rajovic G. "Environmental awareness as a basis for sustainable development of rural areas of Mon- tenegro", Ecologica, scientific and professional soci- ety for Environmental Protection of Serbia, No. 49, Belgrade, 2007.
9. Todić D. Environmental management conditions of globalization, Megatrend University, Belgrade, 2008.
10. http://www.forum.ftn.uns.ac.rs/index.php?act= attach&type=post&id=5558
11. http://hr.wikipedia.org/wiki/odlagaliste_otpada
12. http://staklenozvono.rs/?p=1254
13. www.dominomagazin.com
14. http://krize.medijskestudije.org
15. www.ecologija.org.rs/casopis.htm
16. http://www.reciklaza.in.rs

Environmental pollution due to mobile phone radiation, high voltage powerline radiation and investigating it's effects on heart rate and blood pressure

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ABSTRACT

Pollution due to Electromagnetic Radiation (EMR) is inevitable in today's world and it had affected our lives. Currently we are totally dependant upon the technologies which are the major sources of EMR. In many situations these radiation are more powerful than natural sources of radiation. A study was conducted in Pune city (India) to find out the effect of EMR emitted by Global System for Mobile (GSM) and high voltage powerline (HVPL) on health parameters like blood pressure and heart rate, for 20 healthy volunteers (10 male and 10 female), within age group 18-25 years. In the study, heart rate (HR) and blood pressure along with other health parameters are measured using multiparamonitor (Caddo 19B, Multipara set-up). The transmitter and receiver handset (Moto G 3rd Gen-XT 1550), GSM SIM (AIRCEL), distance between caller and receiver, time of observations and other external parameters are kept fixed. These observations are analyzed using software "students online t test". Significant effect is observed on heart rate and blood pressure for level $p<0.05$. In high voltage powerline study, the changes in diastolic or systolic blood pressure and heart rate when volunteers were subjected to electromagnetic radiation due to power line for 30 minutes of time are statistically not significant for groups at level $p < 0.05$.

Keywords: Electromagnetic radiation (EMR), Mobile tower radiation, radiation pollution, HVPL study

INTRODUCTION

Speedy developments in various fields of science and technology in recent years have exaggerated the human interference into the natural environment. Such interference is linked with physical, biological and ecological systems resulting in various accidental and unwanted negative impacts on environment. With economic, social and scientific development, there is awareness about environmental pollution recent times. Everyday our life is encompassed by man-made EMR: food is heated in microwave ovens, airplanes are guided by radar waves, television sets receive electromagnetic waves transmitted by broadcasting stations, and infrared waves from heaters provide warmth. Infrared waves also are given off and received by automatic self-focusing cameras that electronically measure and set the correct distance to the object to be photographed. During night, incandescent or fluorescent lights are turned on to provide artificial illumination, and cities glow brightly with the colorful fluorescent and neon lamps of advertisement signs. Ultraviolet radiation cannot be seen by eye but its effect is felt like pain from sunburn. Mobile tower, cell phone and HVPL are also pouring radiation in these.

The intensity of man made EMR has become so ubiquitous and it is now increasingly being recognized as a form of unseen and dangerous pollution that might affect life forms in multiple ways [1]. Most of the short-term studies related to the thermal impacts of EMR exposure on biological systems have neither succeeded to discover any statistically significant changes in the biological processes at the background levels of exposures [2]. On the other hand, long-term studies have reported frightening observations, detecting negative consequences on immunity, health, reproductive system, behavior, communication and co-ordinations [3]. The electromagnetic radiations are classified in to radio frequency field (RFF), Intermediate frequency fields (IFF), extremely low frequency field (EFF) and static fields [4].

The consequence of mobile phone radiation and HVPL on human health is the subject of recent interest due to enormous increase in mobile phone usage throughout the world. Mobile phones use EMR in the microwave range. Some national radiation advisory authorities have suggested actions to diminish exposure to their citizens as a preventive approach. The speedily growing mobile phone technology increased public anxiety about the possibility of associated undesirable health effects. There is some evidence for biological consequences also, which, may be necessarily hazardous for humans [5]. Radiation from mobile base stations (towers) may also have an effect on the local abundance of insects or other invertebrates and thereby indirectly influencing the number of house sparrows. The adult house sparrows are seed-eaters, and need insects and other invertebrates to feed their young, such that it is likely that they will prefer areas with high abundance of invertebrates at the beginning of the breeding period. Several investigators have postulated that the lack of invertebrates might be an important factor in the reported decline of house sparrow populations in urban areas. Short-term exposure of pulsed mobile phone radiation with carrier frequency 900 MHz resulted in a 50-60 % decrease of the reproductive capacity of insects. Similar results were also found with microwave radiation at other frequencies.

Some study showed that long-term exposure to low-intensity (pulsed) electromagnetic radiation from base stations may have significant effects on populations of wild birds. When birds are exposed to weak electromagnetic field, they disorient & begin to fly in all directions. This explains migratory birds undermining navigational abilities. Honey bees appear to be very sensitive to EMR [6]. Plant showed responses to high frequency electromagnetic fields [7]. Other wildlife such as amphibians and reptiles also appear to be at high risk with possible interference of EMR. Biological effects due to HVPL on plants, insects and animals, as well as the human body are less harmful when exposed to levels that are below the standardized threshold values. Probable risks from high voltage powerline like leukemia, breast cancer, neuropsychological disorders, decrease in blood sugar and reproductive outcomes have been reported [8,9].

METHODOLOGY

Caddo 19B Multipara Monitor with silver/silver chloride ECG disposable electrodes (single use) and ECG paper rolls are used for experiments. Multipara monitor machine is a light weight, compact and easily portable (Fig. 1). Using the machine one can view important signals such as electrocardiogram (ECG), respiratory rate, percentage of saturated oxygen (Sp02), blood pressure and body temperature. These parameters can be easily displayed on monitor. The power switch is on the front side of panel. The charge marker and the power switches are illuminated when the machine is powered on. The alarm display is on the front panel. The alarm indicator shows lights when alarm takes place. The sensors have sockets at the left side and the recorder socket is at the right. Other sockets and power connectors are on the rear panel. The monitor is easy tool to use with functions carried out by a small number of buttons and a rotating switch on the front panel. The multipara monitor has a printer with network arrangement and provision to display patient's very important health parameters on the screen. The data so obtained is used for measurement of various parameters and keeping record for diagnosis and treatment. This data can be stored, printed and transferred. There are four sensors or electrodes, used in multiparameter which is capable of giving information of electrocardiogram and health parameters (Fig. 2).

Fig1:Multiparamonitor   Fig 2: Multiparameter electrodes

SAMPLE COLLECTION AND STUDY PLAN

For mobile radiation study, 20 students including males and females within age in between of 18-25 years were selected from urban backgrounds. These volunteers were using cell phones and enrolled in the Colleges of Pune city. Any individual, employees, guest and servants were excluded from study. College students selected were using cell phone for more than 3 years. The purpose of study was explained to all volunteers and their consents were obtained.

All subjects were informed to avoid drinks like caffeinated drinks. They are also asked not to have unnecessary activities together with physical exercises and aerobatics within 12 hours prior the collecting data. They were also informed not to have food or tea prior to experiments. Volunteers were given information regarding the equipments being used for examining the health parameters, procedure of study and experimentation. Mobile study was completed in a silent, airy and spacious room in seating position. The recording of respiratory rate (RR), saturated oxygen (SpO2) along with systolic blood pressure (SBP), diastolic blood pressure (DBP), pulse rate (PR) and body temperature (Temp) of a volunteer under observation was completed with the help of simple six channel multipara monitor of Scientech Company of Model Caddo 19B.

ECG electrodes are positioned on arms, legs and chest as per standard medical practice or guidelines given in the user's manual provided by supplier of machine. Recording of ECG generally takes five to ten minutes for a single patient. After recording ECG, the electrodes positioned to body are removed from their locations. An ECG recording is absolutely trouble-free. A volunteer at fixed distance (100 m) was talking continuously on one mobile (Moto G 3rd Gen-XT 1550) with volunteer under experiment using another mobile GSM SIM (AIRCEL). Recording of various health parameters were also noted.

Same methodology was used for HVPL study except that volunteer was asked to sit below high voltage powerline.

RESULTS AND DISCUSSION

All the parameters indicated distinct trend in variations in the data except temperature parameter for mobile radiation emitted due to one hour communication. This was because the change in temperature was very small in magnitude as

compared with other parameters. It was observed that there were significant changes in HR and BP as compared with other health parameters. It had been noticed that after 20 minutes of exposure of cell phone radiation, health parameters under investigation showed a trend in the value of parameter. Hence time of exposure was kept time of 20 minutes for further observations. Figure 3 indicates comparative changes in health parameters after mobile phone radiation exposure of 20 minutes.
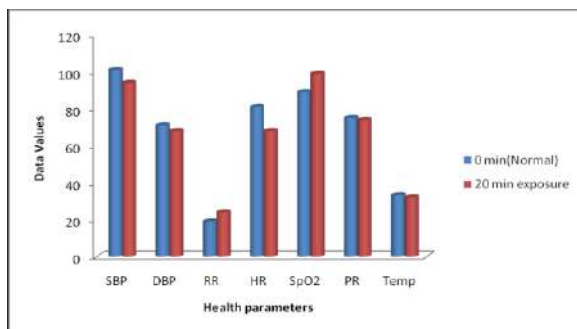
Fig 3: Typical variation in health parameter after 20 min of mobile communication of a volunteer

Heart rate is associated with BP. Observations from 20 volunteers were compared with un radiated normal values of health parameters. These values were compared using online "t test calculator". Hence p values for blood pressure and heart rate are shown in Table 1.

Gender wise comparison of various parameters is shown in Table 1, showed that for male the variation in heart rate was significant at the level of $p < 0.05$, while in case of female volunteers the change in heart rate was not statistically significant. Similarly, observed variation in diastolic blood pressure is significant for female group.

Same observations were noted by asking volunteer to seat below HVPL. The statistical p values of these observations are indicated in Table 2.

Gender ↓	Age years	p value for heart rate for exposure of		p value for DBP for exposure of	
		10 min	20 min	10 min	20 min
Male	22± 1.05	0.0273	0.0023	0.504	0.662
Female	21.67 ± 2.53	0.6303	0.2321	0.286	0.022

Table 1. Statistical p values for HR and DBP

The analysis carried from Table 2, indicated that the variations in diastolic BP, Systolic BP, HR, pulse rate, respiration rate and SpO2, when a volunteers were subjected to electromagnetic radiation due to powerline for 30 minutes was statistically not significant for groups at level $p < 0.05$ This results confirmed the study of other investigators [10].

Table 2. Min, Max, and S.D values of studied variables for male students before and after exposure to EMR from HVPL

Variables	For male				For female			
	Min.	Max.	S.D.	p value	Min.	Max.	S.D.	p value
SBP (a)	105	144	12.50	0.207	95	117	7.86	0.652
SBP (b)	101	121	10.28		110	100	9.38	
DBP (a)	61	89	13.41	0.952	60	91	9.62	0.592
DBP(b)	65	96	7.35		59	63	8.37	
RR (a)	14	21	2.97	0.931	14	21	1.93	0.267
RR (b)	18	12	2.02		21	11	3.03	
HR (a)	65	97	12.06	0.805	70	96	7.67	0.447
HR (b)	73	92	11.35		75	74	7.18	
PR (a)	73	104	12.0	0.196	73	94	8.17	0.760
PR (b)	87	86	6.50		78	73	6.13	
SpO2(a)	84	99	4.41	0.469	73	98	6.63	0.0923
SpO2(b)	90	98	2.63		95	97	1.25	

CONCLUSION

In the study of continuous communication on the mobile phone reported, it had been observed that the call with a mobile phone changed heart rate of healthy volunteer. Mobile phone radiation affected heart rate variability but the effect of speaking on mobile phone cannot be ignored. The result indicated increase HRV parameters even near to head while taking for 20 minutes. Analysis of the data showed statistical significant effect due to mobile phone exposure of 20 minutes on health parameters. The HVPL study indicates that there is no significant effect of powerline voltage on health parameters measured in heart rate variability. The results due to fact that frequency of powerline is of 50 Hz only, hence it possess low energy. Human body absorbs energy of high frequency field more easily. Cells within body possess bioelectric field due to bioelectric potentials.

REFERENCES

1. Mahajan Ankur *et al*. Human Health and Electromagnetic Radiations. *International Journal of Engineering and Innovative Technology,* 2012; 1(6): 95-97.

2. Kapdi M, Hoskote S & Joshi SR. Health hazards of mobile phones: an Indian perspective. *JAPI,* 2008; 56: 893–97.

3. Levitt BB, Lai, H. Biological effects from exposure to electromagnetic radiation emitted by cell tower base stations and other antenna arrays. Environmental Reviews, 2010; 18: 369–395.

4. Grzegorz Redlarski *et al*. The Influence of Electromagnetic Pollution on Living Organism: The Historical Trends and Forecasting Changes. *Bio Med Research International*, 2015:18.

5. Bhargavi K, *et al*. Mobile Phone Radiation Effects on Human Health. *International Journal of Computational Engineering Research,* 2013; 3(4):196-203.

6. Balmori, A. Electromagnetic pollution from phone masts. Effects on wildlife. Pathophysiology, 2009; 16:191–199.

7. Alain Vian *et. al*. Plant responses to high frequency electromagnetic fields. *Bio Med Research International,* 2016.

8. Ahmadi H *et al*. Electromagnetic fields near transmission lines – problems and solutions. Iran. *J. Environ. Health sci.Eng*., 2010;7(2):181-188.

9. DIB Djalel and Mordjaoui Mourad. Study of Influence High-Voltage Power Lines on Environment and Human Health (Case Study: The Electromagnetic Pollution in Tebessa City, Algeria). *Journal of Electrical and Electronic Engineering,* 2014; 2(1): 1-8.

10. Anderson LE, Kaune WT. Electric and magnetic fields at extremely low frequencies. WHO. Reg Publ Eur Ser, 1988 ;25: 175-243.

RESEARCH ARTICLE

OPEN ACCESS

The Human Population is a Main Cause of Global Warming

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ABSTRACT

The Global warming, an important aspect of climate change is closed associated with the accumulation of greenhouse gases in the atmosphere. The Climate change is a general ter. It means change in climate. But the Global warming is a kind of climate change. Global warming means the gradual increase in the temperature of the earth. Scientists are supporting to the fact that this global warming is resulted from the enhanced greenhouse gases in the atmosphere. Therefore, it is closed associated with the greenhouse gases. It mainly includes carbon dioxide, methane and nitrous oxide etc. It is proved that in these gases allows the incoming short-wave radiation from the sun, but they are responsible to block outgoing long-wave radiation from the earth. It results into increase of the earth temperature. Thus, the climate change is occurred. Since, the greenhouse gases are mainly responsible to the global warming. They are produced due to human activities i.e. the burning of fossil fuels, deforestation, the application of fertilizers and the growth of particular types of agricultural production etc. Thus, the population is a significant cause of global warming. The luxuries of population like automobiles and refrigeration are mainly responsible to global warming. There is a close relationship between the population and the release of the green house effect gases in the environment. Therefore, the present research paper is mainly concerned with the research problem that the growth of population on the earth stimulated the global warming as a global concern. The increased proportion of CO2 and other GHGs are the main causes of the global warming. Nevertheless, global warming is the human population induced danger to the planet. Thus, the growth of human population is the main cause of global warming on the green planet.

Key Words: global, warming, surplus, climate, change, penalty, freedom, control, effect, greenhouse effect, policy, public, constitution, right and legislation.

INTRODUCTION

The term greenhouse effect was firstly used by the Swedish chemist Svante Arrhenius in 1897.It is closely associated with the global warming. Global warming means increase in the temperature of the earth. It is proved that the earth temperature has increased by about 0.5°C in last century. The scientists have supported to the view that the increased proportion of CO2 and other GHGs are the main causes of the global warming. Since, it is the human induced mechanism to trap the survival of the earth.

There are different factors are responsible for the destruction of the green house effect like the burning of fossil fuels, land clearing and agriculture, etc. It affects the temperature on the earth and leading to the global warming. Moreover, the human activities are mainly responsible for the global warming. It is only recently, however, that some scientists specializing in the great variety of aspects involved in climatic studies began to evince serious concern that the warming process was so far underway, following the dramatic growth in world population, industrialization and other factors, that dire consequences for humankind might already be irreversible. Scientific rather than legal studies have thus dominated research in this field to date.(1)

"Climate change" means a change of climate which is attributed directly or indirectly to human activity that alters the composition of the global atmosphere and which is in addition to natural climate variability observed over comparable time periods.(2) Scientists believe that this global warming trend is resulted from the enhanced greenhouse effect.(3) The earth's atmosphere alone is being barraged by the effects of almost every human activity imaginable; from our diets and farming practices to the way we heat and energize our homes, travel, work and recreate. Projected increase in atmospheric concentrations of anthropogenic GHGs could have an adverse impact on the earth's temperatures.(4)

Objectives
Following are the objectives of the research paper.
1. To study the global warming.
2. To understand the causesof global warming.

3. To give suggestions to face the effects of global warming.

Nature and Scope of the study
The nature and scope of the study covers the comprehensive analysis of the current state of the causes of global warming in India and the way how its measures became more effective. The main points to be focused through the current research paper are the definition of global warming, background, and review of literature, international aspect, judicial contribution, constitutional provisions and suggestions to cure the lacuna. The Environmental Protection Act, 1986, Article 21 of the Indian Constitution is the scope of this research paper.
Hypothesis
The human population is a main cause of global warming.

METHODOLOGY

This is the conceptual research paper. Therefore, the secondary data like the books, newspapers, and websites, views of the authors expressed through the research papers, journals, case laws and commentary on the concept of the causes of the global warming are studied and necessarily used to complete the research paper.

1. Meaning and definition of global warming.
A gradual increase in the overall temperature of the earth's atmosphere generally attributed to the greenhouse effect caused by increased levels of carbon dioxide, CFCs, and other pollutants. It is also defined as changes in the world's weather, in particular the fact that it is believed to be getting warmer as result of human activity increasing the level of carbon dioxide in atmosphere."(5)

2. The Right to life also includes right to purity of environment

1). Article 21 reads as:
"No person shall be deprived of his life or personal liberty except according to a procedure established by law."(6)

According to Bhagwati, J., Article 21 "embodies a constitutional value of supreme importance in a democratic society." Iyer, J., has characterized Article 21 as "the procedural magna carta protective of life and liberty.

2). Judicial interpretation of right to purity of environment

i).Right to get Pollution Free Water and Air: In **Subhas Kumar v. State of Bihar(7),** it has held that a Public Interest Litigation is maintainable for insuring enjoyment of pollution free water and air which is included in 'right to live' under Art.21 of the constitution. The court observed: "Right to live is a fundamental right under Art 21 of the Constitution and it includes the right of enjoyment of pollution free water and air for full enjoyment of life. If anything endangers or impairs that quality of life in derogation of laws, a citizen has right to have recourse to Art.32 of the Constitution for removing the pollution of water or air which may be detrimental to the quality of life."(8)

ii)Right to Clean Environment: Indian judiciary is an activist judiciary. Since, it is held by the judiciary that the Right to Life under Article 21 of the Constitution includes the right to dignity i.e. to live in a proper environment free from the dangers of diseases and infection. Following are the cases supporting this judicial philosophy. In M.C. Mehta v. Union of India(1988)(9), the Supreme Court ordered closure of tanneries that were polluting water. In M.C. Mehta v. Union of India(1997)(10), the Supreme Court issued several guideline and directions for the protection of the Taj Mahal, an ancient monument, from environmental degradation. In Vellore Citizens Welfare Forum v. Union of India(11), the Supreme Court took cognizance of the environmental problems being caused by tanneries that were polluting the water resources, rivers, canals, underground water and agricultural land. The Court issued several directions to deal with the problem.(12) In Milk Men Colony Vikas Samiti v. State Of Rajasthan(13), the Supreme Court held that the „right to life" means clean surrounding which lead to healthy body and mind. It includes right to freedom from stray cattle and animals in urban areas. In M.C. Mehta v. Union of India **(2006)[14],** the Court held that the blatant and large-scale misuse of residential premises for commercial use in Delhi, violated the right to salubrious sand decent environment. Taking note of the problem the Court issued directives to the Government on the same. In **Murli S. Deora v. Union of India[15],** the persons not indulging in smoking cannot be compelled to or subjected to passive smoking on account of act of smokers. Right to Life under Article 21 is affected as a non-smoker may become a victim of someone smoking in a public place.[16]

3. The Green House Effect
The earth's climate is driven by solar radiation. In the long term the energy absorbed from the sun must be balanced by outgoing radiation from the earth and the atmosphere. Part of this outgoing energy is absorbed and re-emitted by radioactive atmospheric gases ('greenhouse gases'), thereby reducing net emission of energy to space. To maintain the global energy balances, both the atmosphere and the surface will warm until the outgoing energy equals the incoming energy. This is the green house effect.[17]

The greenhouse effect is perfectly natural and has always existed since theearth took its present form: indeed without it, the earth would be very much colder and subject to violent fluctuations in temperature so that it would be uninhabitable. More recently, the term greenhouse effect has taken on a new shade of meaning to refer to an increase in greenhouse gases in the atmosphere from man-made missions, which is leading to more of the sun's heat being trapped and the earth becoming warmer.[18]

3.The Population is a significant cause of global warming:
It is found that there are different causes are responsible for the global warming. But the most important cause is the human population. Human population is mainly responsible to develop the Halocarbon. Halocarbon is a collective term for the group of partially halogenated organic species, including the chlorofluorocarbons (CFCs), hydro chlorofluorocarbons (HCFCs), hydro fluorocarbons (HFCs), halons, methyl chloride, methyl bromide, etc. These halocarbons have large Global Warming Potentials. Moverover, The World Wildlife Fund (WWF) International has observed that the population

explosion, sustained by human science and technology, is causing almost insoluble problems for future generations. It is responsible for the degradation of the environment through the pollution of the air and water; it is consuming essential as well as non-essential resources at a rate that cannot be sustained. Above all, it is condemning thousands of our fellow living organisms to extinction.[19]

Degradation of the world's environment, income and equality, and potential for conflict exist today because of over-consumption and over-population. If the unprecedented population growth continues, future generations of children will not have adequate food, housing, medical care, education, earth resources and employment opportunities. Humankind cannot continue to grow indefinitely. We are now adding more people to the world's population every decade than we used to add in a century.[20]

Criticism

It is found through the research paper that the human population is the main cause of production of halocarbons. It leads to destruction of the green house effect. Nevertheless, it is the sole criteria of the global warming. It is the unfavorable climate change. Therefore, it is the danger to the life of the green planet. Human luxurious activities are mainly responsible greenhouse gases. It is the root cause of global warming.

Different of the GHGs like carbon dioxide, methane, nitrous oxide etc. are human originated. It is beyond the Earth's natural capacity to remove these gases from the atmosphere. Since, the human population is mainly responsible for the global warming.

Hypothesis

1. The human population is a main cause of global warming.

It is verified and found true on the basis of the secondary data.

CONCLUSION

It may be concluded that as the population grows, it influences directly and indirectly on the environment.

Therefore, it is the pressure on the planet. If the planet like earth failed to deal with the impact of the factors produced by the human population then it is a threat rather than a challenge to the survival of the planet in future. Since, the need of the time is to control the rate of growth of the human population and to adopt other technological measures to face the peril of the global warming. It may be also concluded that the destruction of forests and the use fertilizers and pesticides should be minimized so as to avoid danger of global warming. Moreover, deterrent penal provisions should be followed for causing environmental pollution. Thus, the population should act as a catalyst in prevention of global warm in grather than an obstacle in it.

REFERENCE

1. Patricia Birnie, *International Environmental Law and Global Climate Change*, 1st edn, London: Graham & Trotman Publishers Group,1991, p. 1.
2. UNFCC Article 1(2).
3. Dr. Sukanta K. Nanda, *Environmental Law*, 1st edition, Allahabad: Central Law Publications, 2007, p. 324.
4. R. Ramachandran, 'The Play of Environmental Politics', *the ICFAI Journal of Environment Law*, Vol. 11, Jan. 2003.
5. **Definition** of "**global warming**" from the **Cambridge** AcademicContent **Dictionary**© **Cam bridge** University Press).
6. Article 21 of the Indian Constitution.
7. 1991 AIR 420, 1991 SCR (1) 5
8. Ibid
9. AIR 1988 SC 1037 : (1987) 4 SCC 463
10. .AIR 1997 SC 734 : (1997) 2 SCC 353
11. AIR 1996 SC 2721 : (1996) 5 SCC 647
12. Ibid.
13. (2007) 2 SCC 413
14. (2006) 3 SCC 399
15. AIR 2002 SC 40 : (2001) 8 SCC 765
16. Ibid.
17. P C. Sinha, *Global Warming*, International Encyclopedia of Sustainable Development Series, IEdition, New Delhi: Anmol Publications Pvt. Ltd., 1998, p.1.
18. 52 Robin Churchill, *Controlling Emissions of Greenhouse Gases*, 1st edition, Boston: Graham &Trortman, 1991, pp. 147-148.
19. 70 Richard D. Lamm, The Future of the Environment, *Annals of the American Academy of Political andSocial Science*, Vol. 522, The Future: Trends into the Twenty-First Century (Jul., 1992), pp. 59-66.

E-Waste-An Environmental Impact

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ABSTRACT

It is here that the relics of Information Age, with their miraculous microscopic circuits transistors Capacitors and semi conductors are bludgeoned and torched with stone age technology. For the residence in this squalor and filth make they living, first by hauling and then smashing, gutting and burning the television and Computers in a most ungreen form of "recycling" to recover metals- cropper, steel and aluminum. The Information and communication Technology has become the power of the global economy. Software and hardware parts of the Information Technology has entered most of the parts of the social, technical and natural environment. Obviously the increase in production of electric and electronic components and especially computer hardware manufacturing companies. Currently usage, dumping and recycling of all these electric and electronic components are directly affecting to human life and environment. The men, women and even the children using the ways which are not only environmentally harmful but also life frightening. The electronic market particularly computers hazardous impact of different chemicals disposed in environment in the process of computer usage, disposal and inefficient recycling.

Keywords: : IT, economy, electric and electronic, e-waste,   hardware   and   software, manufacturing ,environment, hazardous

INTRODUCTION

E-waste comprises of waste generated from used the electronic devices and house hold appliances which are not fit for their original use and output to the user having not in well condition for recovery, recycling or disposal. Such waste covers wide range of electrical and electronic devices such as record players, radios, transistors, VCRs and black-and-white TVs. later on CD and DVD players, computers, mobile phones, including household appliances like refrigerators, air conditioners, water coolers, water filters, heater etc. E-Wastes contain many hazardous substances like heavy metals, PVC plastics and brominates flame retardants more than thousands of different components which are toxic and potentially hazardous to environment and also human health. E-Waste : 4,500 Eiffel Towers in 2016, 44.7 million metric tons of e-waste was generated. This is equivalent to almost 4,500 Eiffel Towers!

Global e-waste generated		
Year	Total E-waste	Per Person E-waste
2016	44.7 mt	6.1kg
2021	52.2mt	6.8 kg
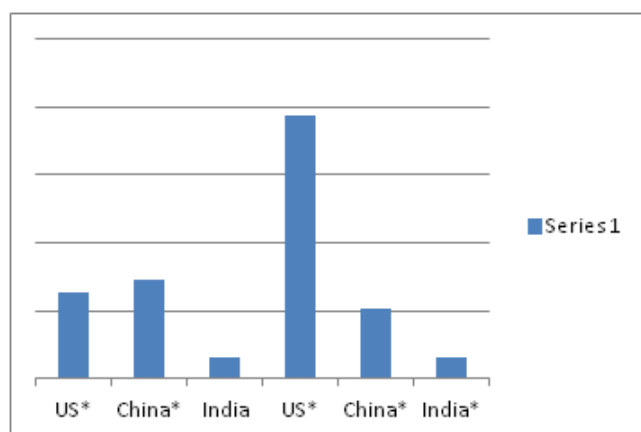
Recycled or dumped	E-waste max in Asia
20% (8.9) of e-waste is documented to be collected and properly recycled	Asia ,which has 45 contries with 4.4 billion population, accounts for
4% (1.7mt) of e- waste in the higher income countries is thrown into the residual waste.	40.7% (18.2mt) of global e-waste
76% (34.1mt) of e-waste is likely dumped. Traded, or recycled under inferior conditions	4.2kg or e-waste per capita

Alarming data on the role of electronic waste and its effects on environment.(1.47 lakh tones or 0.573 MT per day in India, in which Maharashtra state generates approximately 20270.6 tones, Mumbai contributes 11017.06 tons, Maharashtra state ranks first in generating E-waste in India)

Manufacturing process of some components:
Microchip fabrication has over 400 distinct steps involve 4 general phases like layering having thin layer of desired material, usually silicon or aluminum, which then requires oxidation can change the semiconducting silicon layer into an insulating silicon oxide layer which then patterning process, can carving of a dense, maze-like set of furrows into layer and last step is etching can be made by using the solvents or particles bombardment to alter the layer pattern. The process requires great deal of ultra-pure water and the chips are bathed in chemical solvents the resources used are fossil fuels, chemicals, water for bathing the printer, circuit board ,CRT monitors, LCD/LED monitors.

Chemical elements used for lead in shouldering of printed circuit boards and other components also in glass for CRTs.It is estimated that 1.2 billion tons of lead was used in computer components between1997 to 2014.

Mercury is used in batteries, switches, housing and printed circuit boards. Mercury is also found in medical equipment, data transmission equipment, telecommunication equipment, and cell phones as well as estimated that 22% of the yearly use of Mercury is in electrical and electronic equipment although a small amount of Mercury is used in nearly all computer construction. Cadmium is used in resistors for chips, infrared detectors and in semiconductors and also in older CRTs.Plastics is found throughout the computer, largely from casings but also internally to hold components together.
Specific form of plastics used is polyvinyl chloride (PVC) which is used in cabling and housing also.PVC is difficult to recycle and production and burning of PVC generates dioxins and furans.The plastics in computers are often treated with flame retardant chemicals,particularly brominates flame retardant.

List of Examples of devices containing these elements

✓ Tin:Shoulder
✓ Aluminum:Nearly all electronic goods using more than a few watts of power
✓ Iron:Steel chassis, cases and fixings
✓ Silicon:Glass,Transistors,ICs,Printed circuit boards
✓ Nickel and Cadmium: Nickel-Cadmium rechargeable batteries
✓ Lithium:Lithium –Ion battery
✓ Zinc:Plating and Steel parts.
✓ Gold:Connector platting,Primarily in Computer equipment
✓ Sulpher:Lead-acid Battery.
✓ Carbon:Steel,Plastics,Resistors

ENVIRONMENTAL IMPACT

1. The process of dismantling and disposing of electronic waste lead to a number of environmental impacts. Liquid and atmospheric release end up in bodies of water, ground water, soil and air and therefore in aquatic and terrestrial animals, in crops eaten by both animals and human and in potable water.
2. Cathode ray tubes used in TVs, computer monitors, ATM, video cameras are more Lead, Barium and other heavy metals leaching into ground water and release of toxic phosphor.
3. Printed circuit board image behind table-a thin plate on which chips and other electronic components are placed de-shouldering and removal of computer chips,open burning and acid baths to remove final metals after chips are removed air emissions as well as discharge into rivers of glass dust, tin, lead, brominates, dioxin, beryllium, cadmium and mercury.
4. Chips and other gold plated components chemical stripping using nitric and hydrochloric acid and burning of chips, hydrocarbons, heavy metals, brominate substances discharged directly into rivers acidifying aquatic organisms and flora. Tin and lead contamination of surface and ground water.
5. Air emissions of brominates, dioxins,heavy metals and plastics in and low temperature melting to be reused emissions of brominated dioxins, and hydrocarbons.
6. Computer accessories like cable burning and stripping to remove copper hydrocarbon ashes released into air water and soil.

IMPACT ON HUMAN BODY

1. The problem of lead can cause damage to the central and peripheral nervous systems, blood systems, kidneys, endocrine system and cause negative effects on child brain development, lead accumulates in the environment and has a toxic effects on flora ,fauna and microorganisms. Electronics contribute 40% of the total amount of lead found in landfills and can make its way from landfills into the water supplies.
2. The problem of mercury spreads out in water transforming into methylated mercury which easily accumulates in living organisms such as it enters the food chain through fish which can swim in polluted waters. Methylated mercury can cause chronic brain damage. Mercury will leach when certain electronic devises such as circuit breakers are destroyed .
3. Cadmium compounds accumulate in the human body, particularly the kidneys. Cadmium is absorbed through respiration and also food intake. When ruminated flame retardant plastic are cadmium containing plastics are land filled PBDE and cadmium have the potential to leach in to the soil and ground Water.
4. Cadmium has a half-life of 30 years so that cadmium can poison a human body slowly through human life.
5. Hexavalent Chromium (Chromium VI) is used to treat steel plates(an anti-corrosive) and is estimated that between 1997-2014,1.2 million pounds were used in computer components.

CONCLUSION AND SUGGESTIONS

We are willingly allowing hazardous substances to percolate into our water and air so that we may enjoy a new technology and this problem is not going away because the global population is growing and the demand of newer and better technology is creating huge amounts of old and outdated electronics. Most of

the countries discarded computers and monitors and TVs, eventually end up in landfills. There are better ways to dispose of this E-waste which include buy second hand electronic devices, try to fix it before throwing it away, and try to sell so that someone does not buy a new one. Currently there are very few rules and regulations on the disposal of E-waste and cannot be strictly implemented. Implement stricter laws and regulations to help and facilitate a moment towards recycling and reusing. Educate the population better so that they fully understand the potential consequences to improper disposal. Many large corporate sectors such as buy back versions will take back old and unwanted electronic devices. Always contact the large retailers before throwing away the electronic devices.

Conflicts of interest: The authors stated that no conflicts of interest.

REFERENCES

1. IRG Systems South Asia Pvt.Ltd.New Delhi-110017(March 2007)"Report on Assessment of Electronic Waste in Mumbai-Pune Area" fromhttp://mpcb.gov.in/images/pdf/ewaste report 1.pdf

2. Vijay N.B.Trupti S (February-2014), **E-**waste: A new environmental Challenge(Volume $,Issue 2,ISSN no:2277-128-X)

3. ErachB(2010) "Environmental Studies" for Undergraduate Courses,Universities Press Hyderabad (India) Private Limited (ISBN 978- 81-7351-540-2)

4. www.google.com

5. http://en.wikipedia.org/wiki/Electronic waste

6. http://   www.trade   2 save.com/blog/2008/07/06/   wall-e-highlights the   problem- of-electronics-e-waste

RESEARCH ARTICLE   **OPEN ACCESS**

The Impact of Global Warming on the Health of Human Being

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ABSTRACT

Global warming has endangered the existing life-support systems and conditions of earth. There are different adverse effects of global warming. It is responsible for reduced regional food yields, freshwater shortages, increased frequency of extreme weather events, coastal population displacement, changes in the ecology and geography of infectious agents, declines in farming community incomes, and biodiversity losses with accompanying disruption of ecosystem functions. It will converge adversely on human biology and health. Global warming, ultimately, is a threat to our biological health and survival. The Global warming is the evolution of global change and climate change impact. Recently, government and researcher had tried to create awareness about the Global warming and measures to control it. It results in the knowledge about the nature of, consequences of, and responses to, Global warming. Generally, these processes produce a report or other document. It is wonderful to note that the scientific community believes we are already facing the effects of Global warming. Therefore, the developing countries like India is required to concentrate more on the measures to deal with Global warming successfully. That's why this research paper has dealt with the different adverse impacts of the Global warming on the health of the human being.

Key Words Climate, Global warming, health, money, surplus, disease, disaster, penalty, freedom, control, impact, remedy, judiciary, fine, constraint, policy, public, constitution, right and legislation.

INTRODUCTION

Global warming is one of the most important challenges facing the international community today. Scientists have presented overwhelming evidence that climate change is indeed occurring, that human activity has contributed to the problem, and that it will have far-reaching implications for ecosystems, including human settlements.[1] It is scientifically proved that the human beings are more affected due to the Global warming. It harmfully affects human health. It is responsible to make the favorable atmospheric conditions to increase the geographic range of malaria and dengue. It is predicted that due to increasing the atmospheric temperature the earth's surface may not be habitable by 2300.

Objectives
Following are the objectives of the research paper.
1. To study the impact of Global warming.
3. To understand the impact of Global warming on health.
3. To give suggestions to face the impact of Global warming on health.

Nature and Scope of the study
The nature and scope of the study covers the comprehensive analysis of the current state of the impacts impact of Global warming in India and the way how its measures became more effective. The main points to be focused through the current research paper are the definition of Global warming, background, and review of literature, international aspect, judicial contribution, constitutional provisions and suggestions to cure the lacuna. The Environment Protection Act, 1986, Article 21 and the impact of Global warming on health is the scope of this research paper.

Hypothesis
Following is the hypothesis to guide the study.
There is an adverse impact of Global warming on the health of the human being.

METHODOLOGY

This is the conceptual research paper. Therefore, the secondary data like the books, newspapers, and websites, views of the authors expressed through the research papers, journals, case laws and commentary on the concept of the impact of the health are studied and necessarily used to complete the research paper.

1. Meaning and definition of Global Warming
Global Warming means a gradual increase in the overall temperature of the earth's atmosphere generally attributed to the greenhouse effect caused by increased levels of carbon dioxide, CFCs, and other pollutants. The Global warming is defined as "an increase in the earth's average atmospheric temperature that causes corresponding changes in climate and that may result from the greenhouse effect."[2]

2. The impact of Global warming on developing countries.
The reports of IPCC indicate that developing countries would be affected by Global warming. It is significant to note that the poor are vulnerable to its hazards more. Since, the developing countries like India may be suffered more due to the scanty technical measures to tackle with the unfavorable atmospheric condition created due to the Global warming. Thus, the Global warming has different hazards. Following is the detailed account regarding the adverse impact of the Global warming on the health of the Human being.

3. **The impact of Global Warming on the health of human being.**
1) Right to Health Article 21 reads as:
"No person shall be deprived of his life or personal liberty except according to a procedure established by law."
According to Bhagwati, J., Article 21 "embodies a constitutional value of supreme importance in a democratic society." Iyer, J., has characterized Article 21 as "the procedural magna carta protective of life and liberty.

2) Judicial interpretation of right to health
In **State of Punjab v. M.S. Chawla [3]** it has been held that- the right to life guaranteed under Article 21 includes within its ambit the right to health and medical care. In **Consumer Education and Research Centre v. Union of India [4]**The Supreme Court laid down that:"Social justice which is device to ensure life

to be meaningful and livable with human dignity requires the State to provide to workmen facilities and opportunities to reach at least minimum standard of health, economic security and civilized living. The health and strength of worker, the court said, was an important facet of right to life. Denial thereof denudes the workmen the finer facets of life violating Art. 21."[5]

3). Global warming impact on health

The health means, "The state of being free from illness or injury". Global warming will affect the human health in many ways. Global warming increases the risk of mortality from heat stress. It is scientifically proved that the diseases that may survive and spread more in the warmer climates are malaria, dengue, yellow fever, encephalitis and cholera etc. as the mosquitoes are increased in the atmosphere. Global warming causes the more carbon dioxide in atmosphere. It affects adversely human health by reducing blood PH. Since, it may cause problems like breathing, rapid pulse rate, headache, hearing loss, sweating and fatigue etc. Further, it may cause foetal abnormalities also. There are direct and indirect impacts of global warming. Although the indirect effects will ultimately be more serious, the so-called direct effects are surprisingly marked.[6]

Many important diseases that affect developing countries are sensitive to climate variations, according to the report, and even a proportionally small change in the global incidence of some diseases could result in significant public health impacts far into the future.[7] Warmer temperatures will increase the chances of heat waves and can exacerbate air quality problems such as smog, and lead to an increase in allergic disorders. Diseases that thrive in warmer climates, such as dengue fever, malaria, yellow fever, encephalitis, and cholera are likely to spread due to the expansion of the range of disease-carrying organisms. By 2100, there could be an additional 50-80 million cases of malaria each year.[8]

Global climate change magnifies the effects of pollution and will worsen the incidence of asthma, allergies, infections and cardiovascular diseases worldwide. Smoke and particulate matter from more wildfires, which are known to increase in frequency as the climate warms, and the presence of air-borne particles from dust storms caused by desertification.[9]

Researchers have found that poison ivy has grown more potent and lush because of increased carbon dioxide in the atmosphere. As climate changes, it's projected to play an even greater role." High levels of airborne particulates cause respiratory diseases, as well as inhibiting plant growth and requiring costly filtration equipment in certain high-tech manufacturing industries. Long-term exposure to particulates appears to be associated with a reduction of life expectancy of up to 2 years and higher prevalence of bronchitis and reduced lung function in children.[10]

4). Hazardous elements of global warming and their impact on the health.

Following are the hazardous elements resulted of the global warming affecting the health of human being very worst.

i. Nitrogen oxides (NOX): It is responsible to irritate mucous membranes; aggravate existing respiratory illnesses, cause coughs, headaches and shortness of breath.

ii. Carbon monoxide (CO): Carbon monoxide is responsible to reacts with hemoglobin in the blood and reduces the blood's ability to carry oxygen. It is responsible to provide reduced oxygen supply to heart.

iii. Lead: lead is an important element of petrol. It is environmental pollutant causing health problems among infants, children, men and women. It affects mental development, blood chemistry, kidneys, and nervous, reproductive and cardiovascular systems etc.

iv. Ozone: Ozone is an element of photochemical smog. It impairs lung functions and causes cough and chest discomfort, chronic lung injury, leading in some cases to fibrosis, chronic bronchitis and heightened susceptibility to respiratory infections.[11]

v. Ebola: Global warming is also responsible for the evolution of new diseases as in warmer temperatures

is favorable to bacteria as compared to cold temperatures. Hence the mosquitoes are increased in warmer atmosphere. New diseases due to ebola, hanta and machupo virus are found due to warmer climates.[12]

4. Impact of Global Warming in India

India is also a sufferer country of Global warming. It affected reproduction rate very badly the disease malaria affected badly to the most of the central and eastern states of India. According to Stern Report (UK) if global warming is not checked by 2050, 200 million lives will be at risk, it will amount to 20 percent loss in the GDP, one in twenty individuals will lose their home and it will also lead to more than three trillion pounds worth of losses and has suggested the idea of imposing carbon tax on states based on the amount of carbon dioxide emitted.[13]

5. Criticism

The Global warming is always criticized on certain grounds. The most serious defect in the Global warming is the failure of the State to mitigate its hazardous impact on the health. The state is vulnerable so as to achieve the technological mastery to face the unfavorable impacts of the global warming. The global warming impact is totally contrary to the upliftment of human status. Since, it is very rightly said by the swami Vivekananda that "The sign of vigour, the sign of life, the sign of hope, the sign of health, the sign of everything that is good, is strength. As long as the body lives, there must be strength in the body, strength in the mind, [and strength] in the hand".[14]

Hypothesis

1.There is an adverse impact of Global warming on the health of the human being.

It is verified and found true on the basis of the secondary data.

CONCLUSIONS

It may be concluded that in developing country like India that the impact of Global warming is on the health of human being in particular and on the creatures is in general. Researchers found that if

nothing is done to counter the effects of global warming being felt at present, by the year 2080, half of the common plants on earth and one third of animals will lose their climate range. This means the ecosystem or the habitat of the animal or plant will be lost and this will automatically lead to species becoming endangered and the biggest threat will be to plant life, reptiles and amphibians. Thus, the environment should act as a protector rather than a trap to threaten the survival. Therefore, this research paper has revealed the fact that the human health is now at the mercy of the global warming. Since, the human survival in future merely depends upon the technological progress of the state so as to face the hazardous impacts of the global warming. Thus, poor in this regard may be more vulnerable and easy prey to global warming.

REFERENCE

1. Idean Salehyan, "Climate Change to Conflict? No Consensus Yet", *Journal of Peace Research*, 2008;45(3): 315-326.
2. Global warming. (n.d.). Dictionary.com Unabridged. Retrieved January 5, 2018 from Dictionary.comwebsite http://www.dictionary.com / browse/global-warming.
3. AIR SC 1997;1225
4. AIR 1995;922, SCC (3) 42
5. Ibid.
6. Paul Brown, *Global Warming – Can Civilization Survive*, 1st edition, Universal Press, Hyderabad, 1999:125.
7. Jan Gilbreath, "Climate Change: Global Warming Kills", Environmental Health Perspectives, 2004;112(3):160
8. Focus, "The Kyoto Protocol; A Framework for Action", *Global Issues Election Journal*, May 1998: 7.
9. Air quality worsens asthma, *Deccan Herald*, March 16, 2012:16
10. Climate Change Trade in sustainable development, United Nations, New York, 2006: 14
11. Vehicular pollution: A Silent Killer, *Indian Auto*, June 1999:28
12. Ibid
13. Dr. Neeraj Patil, "Putting the heat on Global Warming Threat", *Deccan Herald*, Feb 17, 2007
14. The Complete Works of Swami Vivekananda/Volume 6/Lectures and Discourses/Formal Worship

Factors Affecting "Air Pollution"

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ABSTRACT

The study was carried out over a period of 12 months to get representative data incorporating seasonal variations that have bearing on air quality of Pune city. A total of seven air monitoring stations were installed at various locations. As per the finalized protocol, air quality monitoring for the city of Pune was carried in three sites each with background site common in each set in three seasons. The statistical analysis of the observation of PM_{10} is presented. The suggested model can be used for a prediction of the daily mean value of PM_{10} using selected factors and their previous values.

Keywords Regression,PM_{10} (Particulate matter (size < 10 microns)),Pb- Lead content ,Cd- Cadmium content ,Cu- Copper content, Cr- Chromium content, NOx- Nitrogen oxide content,SO_2- Sulphur dioxide content in PM_{10}.

INTRODUCTION

Air pollution levels are very high in large cities in India. This poses a threat to human health. Causes of air pollution are many, but mostly anthropogenic (i.e. due to human activity). The main anthropogenic factors responsible are, high population density, increasing number of vehicles on road, road dust, factories etc. A study was conducted (in year 2000) to assess relative pollution levels at three sites in Pune. Bhosari is an industrial area, Mandai represents densely populated area and SNDT is a site with crowded road traffic causing emission of toxic gases and large size particulate matter. The observation was recorded in a week. On each

selected day air samples were also taken from university campus. It also has low population density (residential), less vehicle traffic etc. The interest is in checking differences across sites, differences within a day, fluctuations in different seasons etc. It is of interest to study variation not only in total particulate matter but also its break up into different components (metals, gases etc).

Objectives:

Three areas of Pune district were studied

For this research work. The study covers the following

Objectives: 1. To analyze and examine season wise effect of components on air pollution. 2. To evaluate and forecast the future PM_{10} (Particulate matter (size < 10 microns)) of some important polluted element.

METHODOLOGY

The data for the study has been collected from all possible sources as Websites, Online journals, government and cooperative reports etc.

Table: Analysis and Interpretation of data: Descriptive Statistics of Pb, Cd, Cu, Cr, NOx, SO2

Variable	N	Mean	Median	StDev	Minimum	Maximum	Q1	Q3
Pb	53	0.912	0.61	0.758	0.07	3.49	0.41	1.09
Cd	53	0.0008	0	0.003	0	0.02	0	0
Cu	53	0.4289	0.21	0.412	0.01	1.44	0.08	0.7
Cr	53	0.46	0.37	0.357	0	1.54	0.2	0.71
NOx	53	47.79	40.07	25.22	17.07	121.46	29.3	59.5
SO2	53	25.11	22.44	10.52	10.15	51.08	17.5	30.8

Forecasting of actual value and predicted value
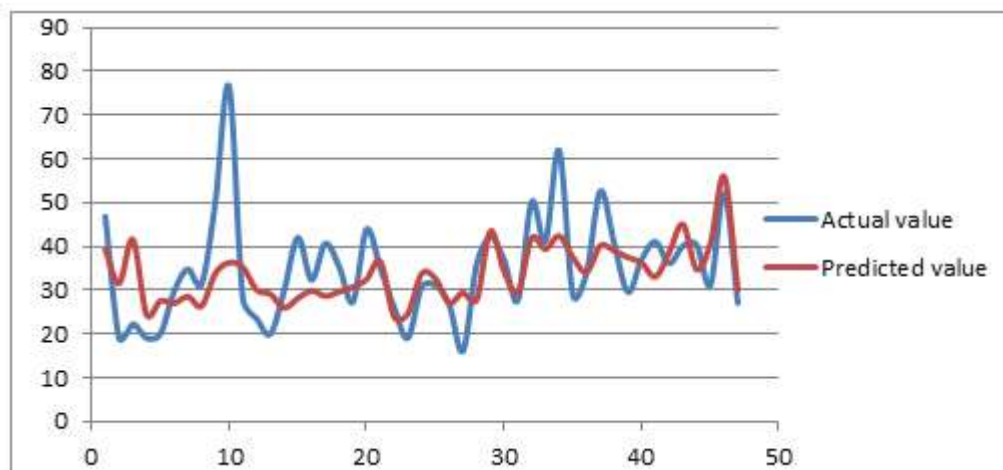
Fig. 1: shows that Actual value and predicted value are approximately equal.

Winter Season

H₀: Data is normally distributed   V/S   **H₁:** Data is not normally distributed

Decision Criteria: If p-value is less than 0.05 then we reject H₀.
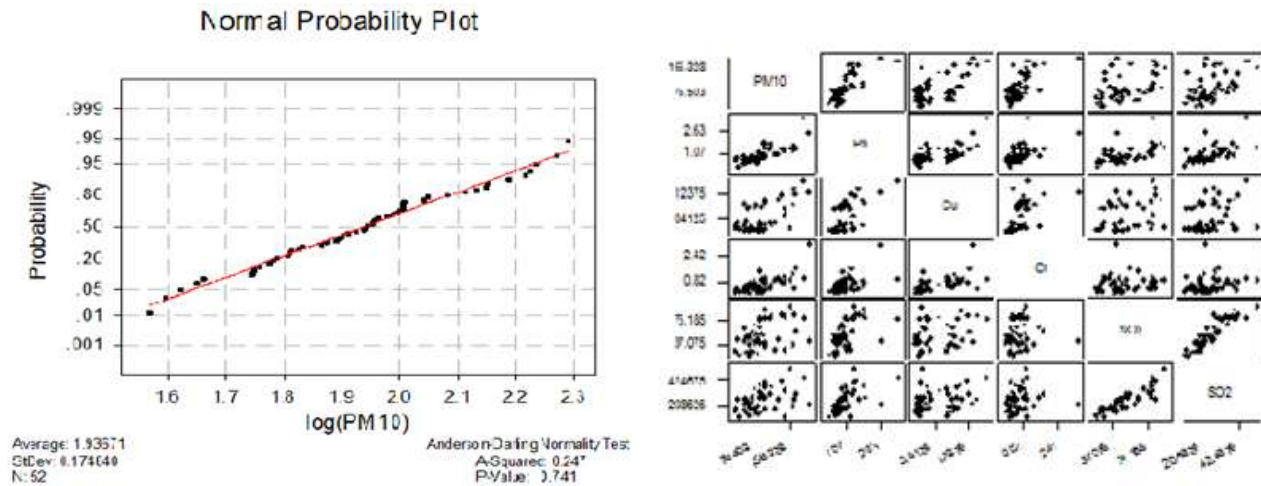
Fig.2a) Matrix Plot of winter season
(fig.2b) shows that PM₁₀ and Pb, PM₁₀ and Cr, NOx and SO2 are positively correlated.

Summer Season
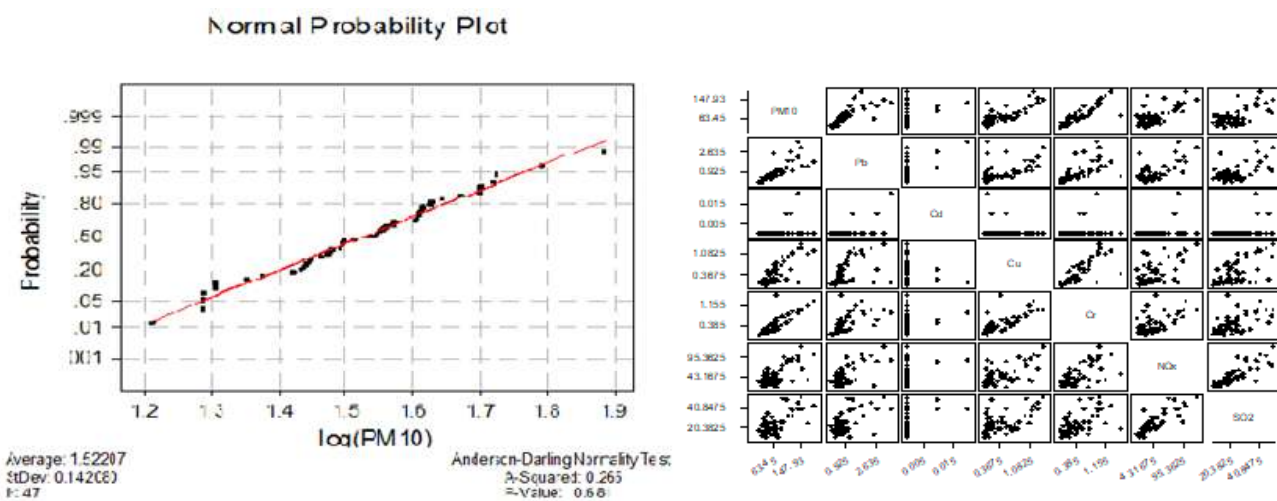
Fig.3   Matrix Plot of Summer season
Fig.3) shows that PM10 and Pb, Cu, Cr, NOx and SO2 are positively correlated.

Monsoon Season

H_0: Data is normally distributed   **V/S** H_1: Data is not normally distributed

Decision Criteria: If p-value is less than 0.05 then we may reject H_0.
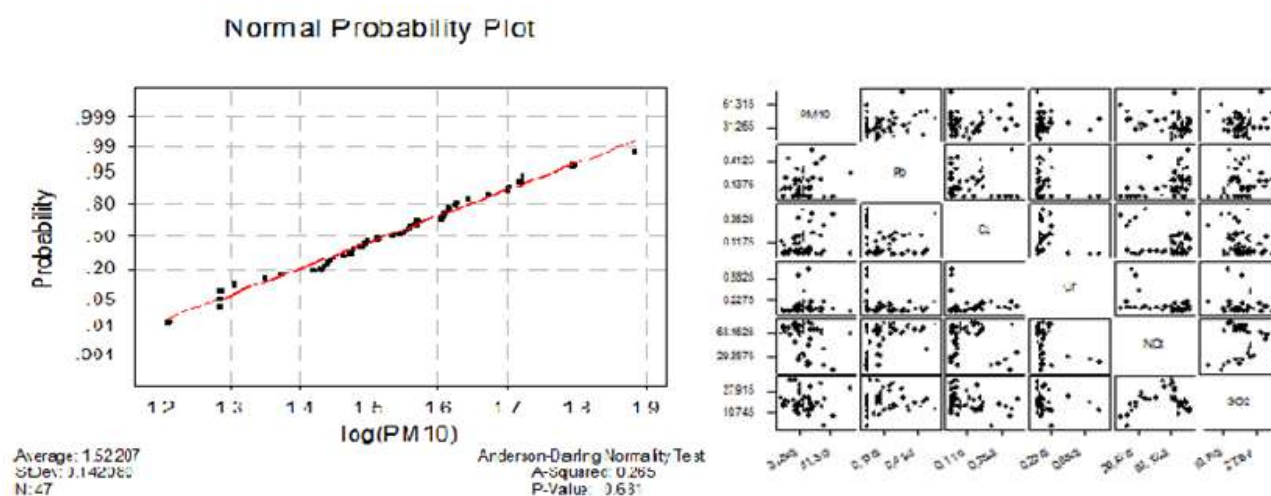
(Fig. 4) Matrix Plot of Monsoon season
(Fig. 4) shows that there is no correlation.

Stepwise Regression: log (PM$_{10}$) versus Pb, Cu, Cr, NOx, SO2

Backward elimination Alpha-to-Remove: 0.1
Response is log (PM10 on 5 predictors, with N = 52

Step	1	2	3
Constant	1.656	1.655	**1.644**
Pb	0.115	0.123	**0.118**
T-Value	2.19	2.55	**2.51**
P-Value	0.033	0.014	**0.015**
Cu	0.018		
T-Value	0.35		
P-Value	0.725		
Cr	0.122	0.128	**0.127**
T-Value	2.72	3.08	**3.08**
NOx	0.0024	0.00255	**0.00181**
T-Value	1.4	1.54	**1.9**
P-Value	0.168	0.13	**0.064**
SO$_2$	-0.0017	-0.0021	
T-Value	-0.43	-0.55	
P-Value	0.669	0.587	
S	0.115	0.114	**0.114**
R-Sq	60.64	60.54	**60.28**
R-Sq(adj)	56.36	57.18	**57.8**
C-p	6	4.1	**2.4**

Season wise content of regressors viz Pb, Cu and Cr is tabulated below

Season	Elements	Average content in atmosphere(μg/m^3)
Summer	Pb	0.912
	Cu	0.4289
	Cr	0.46
Winter	Pb	1.0248
	Cu	0.4687
Monsoon	Pb	0.1496
	NOx	58.72

Statement of Hypothesis

H_0: The data follow a specified distribution.

H_1: The data do not follow a specified distribution.

Use the corresponding p-value (when available) to test if the data come from the chosen distribution. If the p-value is less than a chosen alpha (usually 0.05 or 0.10), then reject the null hypothesis that the data come from that distribution.

Analysis of Variance

Source	DF	SS	MS	F	P
Regression	3	2.17665	0.72555	82.05	0.000
Residual Error	48	0.42443	0.00884		
Total	51	2.60107			

F_0 = 82.05 and $F_{\alpha,k,(n-k-1)}$ = 2.798061

The F Ratio table show for 3 degrees of freedom for smaller sum of square, critical value of F=2.17665 at 0.05 level of significance.The computed value ,i.e.82.05 is much greater than table value.Hence it is not significant .Consequently we reject Ho and accept H1.The air quality of pune district is not satisfactory.

abstracts,The seventeenth International conference On Qualitative methods for the Environmental Sciences,TIES 2006;Kalmar Sweden,18-22.6.2006.

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CONCLUSION

From the analysis of summer season, we conclude that Pb, Cu and Cr highly affect the response variable PM_{10}.For winter season analysis, it is observed that the response variable PM_{10} is highly influenced by the regressors Pb and Cu.For monsoon season the regressors Pb and NOx significantly affect the response variable PM_{10}.From the overall analysis, it is seen that the Pb is responsible for air pollution in all season, so we should try to minimize its amount in atmosphere.

REFERENCES:

1. Applied Linear Statistical Models,(Fifth Edition)-Michael H. Kutner, Christopher J. Nachtaseim, John Neter, William Li
2. Introduction to Linear Regression Analysis, (Third Edition)-Douglas C. Montgomery, Elizabeth A. Peck, G. Geoffrey Vining.
3. Colls, J. *Air Pollution*. New York: Spon Press 2002.
4. European Public Health Alliance, *Air, Water Pollution and Health Effects*. Retrieved from http://www.epha.org/r/54 2009.
5. Stadlober E, Hormann S, Pfeiler B. Quality and performance of a PM10 daily forecasting model. Sparse estimates in over complete time series models. 2009:307-326.
6. Hrdlickova Z, Kolar M. Michalek J. Vesely V, The Statistical analysis of air pollution by suspended particulate matter in Brno.program and

RESEARCH ARTICLE **OPEN ACCESS**

Ceramide from *Celocia argentea* Leaves by LC-MS

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ABSTRACT

Celocia argentea, a traditional medicinal plant belongs to family Amaranthaceae is well known for its antioxidant, hepatoprotective, cytotoxic, immunomodulating, antidiabetic activities. The dried powder of C. argentea leaves was extracted in $CH_3OH:H_2O$ (1:4) v/v, concentrated to its $1/10^{th}$ volume. The exhaustive analysis of C. argentea leaves extract was separated and characterized using HPLC-MS-Q-TOF. The structures were studied and proposed by ESI-MS/MS with MassHunter software. The CerP(d18:1/22:0), is ceramide with phosphate identified at m/z 722.5232 and at retention time 12.03 min is due to adduction of potassium ion followed by loss of water molecule having combination of elements is $C_{40}H_{80}NO_6P$ shows 1.94 ppm error. Present study will support in future to propose the plant as a good source of lipid, which will support as a dietary supplement and can be used as a component of cosmetic materials. Apart from this there are compounds of biological origin are the medicines used for chronic as well as severe diseases like cancer, tuberculosis etc.
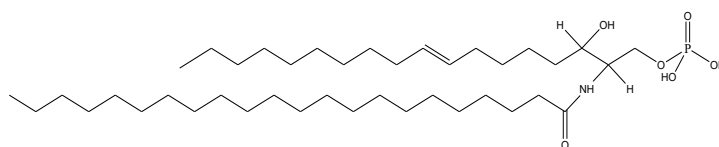
Keywords: Celocia argentea, HPLC-MS-Q-TOF, Ceramide, Herbal medicine

INTRODUCTION

Celocia argentea L. commonly known as Cockscomb, Lagos spinach, is weed found worldwide belonging to Amaranthaceae family. It is traditional medicinal plant in Asian country commonly used by rural population. Indian origin *C. argentea* have more than sixty varieties over the world. The entire plant is utilized, and some of its benefits were reported by tribal people in folk medicine. Historically, this plant has been widely used, the composition of its seeds being more studied than that of leaves. Leaves are applied in traditional medicine for treatment of eczema, diarrhea, and throat. The secondary metabolites, glycosides isolated from seed are used as skin whitening agent and for strengthening of eyes. The plant is used to cook in the time scarcity as a leafy vegetable. Recently, the secondary metabolites isolated from the seeds have been reported for various biological activities. Seed from *C. argentea* are rich in betalains which confer antioxidant activity. Isolated flavonoids from seed are responsible for good antioxidant, hepatoprotective, cytotoxic, immunomodulating, antidiabetic activities [1,2]. *C. argentea* leaves has relevance for developing dietary role due to valuable phytochemical carbohydrates, vitamins and essential macro and micronutrients.

There is need to investigate bioactive phytoconstituents for developing nutraceuticals and dietary supplements. A prerequisite for investigating bioavailability and biochemical effect of any dietary phytochemicals is to know the occurrence of metabolites group in a given plant species with their qualitative and quantitative composition. In agricultural and food science the metabolism is very important growing field of analytical chemistry for identification of small metabolites present in small plant tissues [3]. Metabolite profiling is the analytical method for relative quantification of number of metabolites from biological samples, restricting itself to a certain range of compounds or event to screening a pre-defined number of members of compounds of class [4]. Thus, mass spectrometric methods are most widely used in the field of metabolomics, LC-DAD-MS with electron spray ionization (ESI) being used increasingly for screening botanical metabolites [5,6].Identification of phenolic acids, lipids and other polar phytoconstituents from *C. argentea* has been usefully performed by RP-HPLC coupled with mass spectrometry. Highly efficient resolution and characterization of wide range of polar compounds is achieved for separation of compounds by polarity differences in LC-MS hyphenated technique [7]. The mass accuracy and true isotopic pattern provided by Quaternary Time of Flight-Mass Spectrometry (Q-TOF-MS) for both precursor and fragment ions enabled the determination of many well-known compounds present in *C. argentea* by facilitating additional information key for determining the elemental composition.

The aim of this study is to present a complete exhaustive analysis of *C. argentea* leaves extract. HPLC-MS-Q-TOF was used for characterization and identification of untargeted phytochemicals covers wide mass range and target many compound classes, representing overall richness of plant. Extraction program was performed as per the guidelines for analysis of phytochemicals by J. Horborne. The structures were studied and proposed by ESI-MS/MS with MassHunter software [8]. In future these bioactive can be isolated and analyze quantitatively will be useful for various biological activities and formulations of new herbal medicine. LC-MS detects the large group of plant secondary metabolites such as alkaloids, saponins, phenolic acids, phenyl propanoid, flavonoids, glucosinolates, polyamines; depending upon type of stationary phase used [9].

Actually, from early human history, natural products from plants have always been investigated and utilized to treat numerous diseases. Medicinal plants are productive sources to provide large number of bioactive molecule, which could be screened to find potential lead compound for drug discovery [10]. The efficacy, safety and quality of phytomedicine are the important need for therapeutic purposes. Among all the identified hundreds of phytoconstituents, only a few constituent are bioactive, means having potential for biological activity. Hence it is necessary to identify and measure all the bioactive phytoconstituents of medicinal plant to ensure the reliability and repeatability of clinical research and enhance the quality from the pharmacological beneficial and or hazardous perspectives. Because of complexity and

variability of botanical extracts, it has presented significant challenges for separation and detection methods enabling rapid analysis of the chemical composition of medicinal plant. Identification and quantification of trace metabolites of natural products from complex biological matrices requires sophisticated analytical methods with high sensitivity and selectivity.

METHODOLOGY

The analysis of extracted sample was carried out on LC-MS-Q-TOF spectrometer for metabolite profiling of *C. argentea* leaves powder using the solvent gradient elution method. Liquid chromatography is coupled with high resolution tandem mass spectrometer, has greatly facilitated explicit identification and highly sensitive quantification of trace component present in complex matrices, which can accomplish qualitative analysis in short time. The present study summarizes recent developments of identification and measuring phytochemical constituents and their metabolites, as well fingerprinting analysis for *C. argentea* are highlighted. Metabolite profiling of phytochemical constituents from matrices helps to confirm known compounds and structure of unknown compounds. After profiling phytoconstituents need to separate chemically and followed by bioassay for new active chemical entity from natural sources. Significance of the study of metabolite profiling is still growing due to their numerous functions in plants as well as their beneficial effects on human health. Identification of phenolic metabolites presents in complex plant extracts is an important task during studies in many areas of biomedical research. Secondary metabolites present in complex plant extracts are often isomeric or isobaric compounds, with different substituents to parent compound shows very similar chromatographic properties. These phytochemical properties results in co-elution of numerous metabolites due to lack of resolving power of HPLC column. The substituents such as sugar moieties, consisting of simple hexoses and pentoses (glucose or xylose), deoxysugars (rhamnose) or sugar acids (glucuronic acid), acyls (aliphatic and aromatic acids) and alkyls, demands for a usage is special analytical

techniques for identification of secondary metabolites. There is need to more developments on isolation and purification of phytochemical from natural product extracts, which is laborious, tedious and time consuming to identify the compound by earlier IR, MS and NMR techniques are known (dereplication) or uninteresting. Now LC-MS can rapidly identify known or unknown compounds using fragmentation pattern of molecular species.

In leaves of *C. argentea*, all the compounds are verified in terms of retention time, accurate mass measurement data, elemental composition and error in ppm by comparing the results with those in literatures and a number of established database of LC-ESI-Q-TOF-MS from exact mass measurement. The data were processed using MassHunter® software in Agilent Q-TOF-MS.

Extraction *C. argentea* leaves for LC-MS

Prior to LC-MS analysis of secondary metabolites, sample preparation is regarded as an obligatory procedure because extracting the desired chemical component from the herbal materials is the important first step in analytical process. The popular soxhlet extraction technique is used for the extraction, clean-up, and concentration of analytes from different matrices can effectively reduces matrix effect which has significant impact on the accuracy, precision and robustness of bio-analytical methods.

The extract procedure was carried out as per the standard methods of phytochemical analysis. The dried powder of *C. argentea* leaves was extracted in $CH_3OH:H_2O$ (1:4) v/v, the extract was concentrated to its $1/10^{th}$ volume. The resultant extract was acidify using 2M HCl and extracted with chloroform. The chloroform layer was concentrated in vacuum. The aqueous layer was excluded from further study. The LC-MS-Q-TOF analysis performed and tentatively identified compounds is highlighting retention time, elemental composition, precursor ion, m/z, exact mass, error and identification of metabolite. Ceramides- drug of biological origin were unambiguously identified, and tentative structures were proposed for compounds.

RESULTS AND DISCUSSION

Ceramides contain fatty acids linked by an amide bond to the amine group of long chain base. They play an important role for biosynthesis of the complex spingolipids and play role for cellular signaling, regulation of apoptosis, cell differentiation, transformation and proliferation. They are esterified to terminal hydroxyl group, prevents the loss of moisture through the skin. The glycoceramides found in skin of plant and animal tissues contain basic ceramide unit linked by a glycosidic bond by a carbon 1 of the long-chain base to glucose or galactose, which were reported in brain lipids called as cerebroside. They are act as biosynthetic precursor of lactoceramide and are part of water permeability barrier.

The CerP(d18:1/22:0), **cpd**-406 is ceramide with phosphate identified at m/z 722.5232 and at retention time 12.03 min is due to adduction of potassium ion followed by loss of water molecule having combination of elements is $C_{40}H_{80}NO_6P$ shows 1.94 ppm error. The dihydroceramide C2, **cpd**-169 appeared on chromatogram at m/z and retention time 326.3053 and 8.93 min respectively due to protonation followed by loss of water molecule possesses $C_{20}H_{41}NO_3$ elemental combination with 0.28 ppm error. The glucosylceramide, cpd-**366** and cpd-**536** are the ceramide unit is linked to glucose appeared on chromatogram at 11.49 and 15.40 min for m/z to molecular ion is 72.536 and 661.536 are caused due to adduct of sodium and ammonium ion have elemental combination is $C_{40}H_{77}NO_8$ and $C_{36}H_{69}NO_8$ with 2.6 and 0.4 ppm error. The cerebroside C **cpd**-515 is identified at m/z of molecular ion is 771.6082 and at retention time 14.64 min caused due to adduction of ammonium ion form molecule, having elemental combination is $C_{43}H_{79}NO_9$ with only 0.4 ppm error.
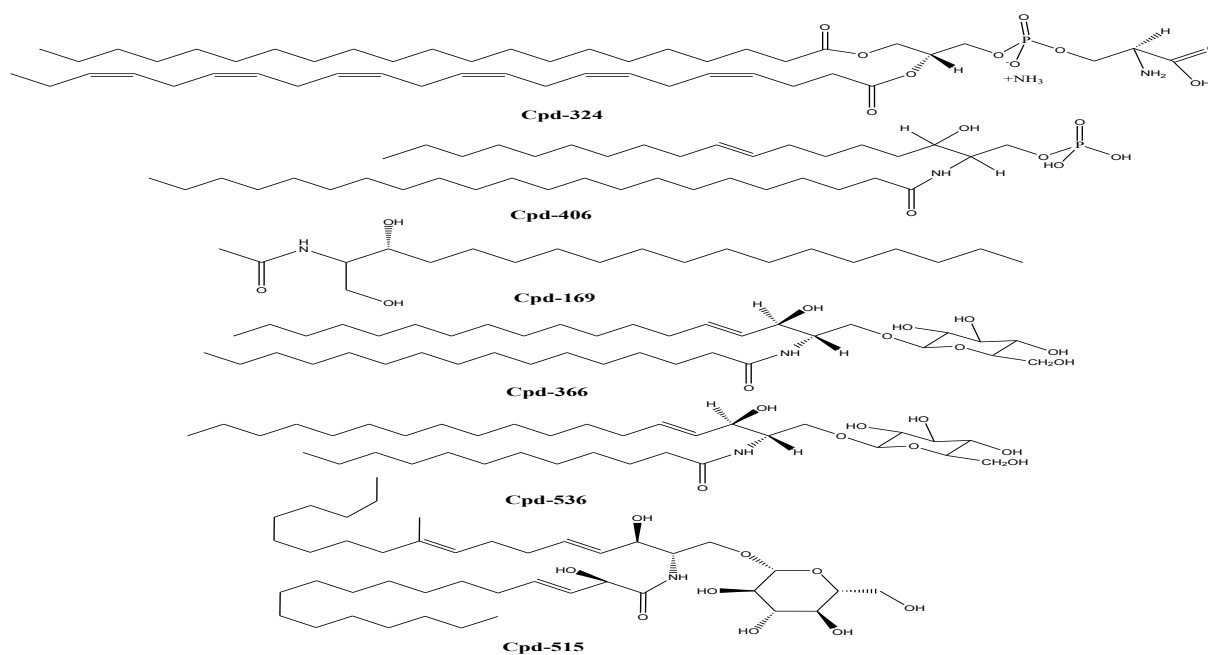
Figure: Structures of phytochemical metabolites- Ceramide

CONCLUSION

The qualitative analysis of *C. argentea* leaves extract performed with HPLC-MS-Q-TOF identify the presence Ceramides were identified, and tentative structures were proposed for compounds. Globally, phytomedicine manufacture needs to maintain the purity of extract from active phytoconstituents quote active marker compound for quality of active extracts.

Present study will support in future to propose the plant as a good source of Ceramides as a lipid, which will support as a dietary supplement and can be used as a component of cosmetic materials. Apart from this there are compounds of biological origin are the

medicines used for chronic as well as severe diseases like cancer, tuberculosis etc.

In this work the extensive qualitative identification of phytochemical metabolites using HPLC-Q-TOF-MS method was employed for quality determination of herbal medicinal products and formulations containing *C. argentea* leaves have been established. The extract may play significant role in prevention of degenerative diseases**.** Ceramides of biological origin have been identified using literature survey, providing the first comprehensive characterization available on the phytochemical composition of the leaves of *C. argentea,* highlighting it is an abundant source of antioxidant phenolics and phytochemicals. The result obtained may develop the current knowledge on *C. argentea*, boost further research toward exploring of bioactive compounds and may encourage more consumption of this important functional food.

Conflicts of interest: The authors stated that no conflicts of interest.

REFERENCES

1. Jong T T, Hwang CC. *Planta Med.,* **1995**, *61*: 584.
2. Perveen, S, Yousaf M, Zahoor AF, Rasool N, Jabber A. *Toxicol. Environ. Chem.,* **2015**, 1.
3. Cevallos-Cevallos JM, Futch DB; Shilts T, Folimonova, SY, Corcuera RD. *Plant Physiol. Biochem.* **2012**, *53*:69.
4. Fiehn O., *Plant Mol. Biol.* **2002**, *48*:155.
5. Harnly JM, Bhagwat, S, Lin LZ. *Anal. Bioanal. Chem.,* **2007**, *389*: 47.
6. Mari A; Lyon D, Fragner L, Montoro P, Piacente S; Wienkoop S, Egelhofer V, Weckwerth W. *Metabolomics.,* **2013**, *9*: 599.
7. Wu, H, Guo J, Chen S, Liu X, Zhou Y, Zhang X, Xu X. *J. Pharmaceut. Biomed.,* **2013**, *72*: 267.
8. Abu-Reidah IM, Ali-Shtayeh MS, Jamous RM, Segura-Carretero A. *Food Res. Int.,* **2015**, *70*:74.
9. De Vos R, Moco S; Lommen A, Keurentjes J, Bino, RJ, Hall RD. *Nat. Protoc.,* **2007**, *2*, 778.
10. McChesney, JD; Venkataraman SK, Henri JT. *Phytochemistry.,* **2007**, *68*:2015.

RESEARCH ARTICLE   **OPEN ACCESS**

Phytochemical and Physicochemical screening of different extracts of *Butea monosperma* flowers

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ABSTRACT

India has well recorded and practiced knowledge of traditional herbal medicines. Plants are used as medicinal and cosmetics purposes since in ancient times. Butea monosperma commonly known as Flame of forest has been widely used in traditional Indian medical system of 'Ayurveda' for the treatment of variety of diseases. The aim of this study is extraction and isolation of components present in flowers of Butea monosperma

Keywords: *Butea monosperma,* medicines, flowers etc.

INTRODUCTION

It is evident that without nature human being life is impossible. There are three basic necessity of humans is food, clothes and shelter and now the fourth one is good health, which provided by plant kingdom. Nature stands a golden mark and provided the storehouse of remedies to cure all ailments of mankind. Plant kingdom represents a rich house of organic compounds, many of which have been used for medicinal purposes and could serve as lead for the development of novel agents having good efficacy in various pathological disorders in the coming years. Herbs have always been the principal form of medicine in India and presently they are becoming popular throughout the world, as people strive to stay healthy in the face of chronic stress and pollution, and to treat illness with medicines that work in count with the body's own defense. There is a widespread belief that green medicines are healthier and more harmless or safer than synthetic ones [1]

Plant products are part of phytomedicines. This can be derived from barks, leaves, flowers, roots, fruits, seeds. Knowledge of the chemical constituents of plants is desirable because such information will be value for synthesis of complex chemical substances [2, 3, 4].These compounds are synthesized by primary or rather secondary metabolism of living organisms. Secondary metabolites are chemically and taxonomically extremely diverse compounds with obscure function. They are widely used in the human therapy, agriculture, scientific research, veterinary and many other areas [5].

Phytochemical constituents are the basic source for the establishment of several pharmaceutical industries. The constituents present in the plants play a significant role in the identification of crude drugs. Phytochemical screening is very important in identifying new sources of therapeutically and industrially important compounds like alkaloids, flavonoids, phenolic compounds, saponins, steroids, tannins, terpenoids etc.Medicinal plants are rich sources of antimicrobial agents. Plants are used medicinally in different countries and are the source of potential and powerful drugs [6].

According to World Health Organization (WHO), medicinal plants would be the best source to obtain variety of drugs. About 80% of individuals from developed countries use traditional medicines, which has compounds derived from medicinal plants. However, such plants should be investigated to better understand their properties, safety, and efficiency [7].Now day's phytochemical studies have attracted the attention of plant scientists due to the development of new and sophisticated techniques. Plant synthesizes different types of chemical compounds, which can be differentiated on the basis of their chemical class, functional groups and bio synthetic origin into primary and secondary metabolites. Knowledge of the chemical constituents of plants is desirable, not only for the discovery of therapeutic agents, but also because such information be of value in disclosing new resources of such chemical substances.

METHODOLOGY

Butea monosperma

Butea monosperma (Lam.) Taub (Syn. Butea frondosa Willd. Family Faboideae), a deciduous tree, is found chiefly in the mixed or dry deciduous forests of Central and Western India. This plant is popularly known as dhak or palas, palash, mutthuga, bijasneha, khakara, chichara and commonly known as "Flame of the forest" This tree grows to 50 ft high, with stunning flower clusters. Tree is almost leafless during spring season forming an orange- red hue of flowers on the upper portion, giving the appearance of flame from a distance.[8,9] Flowering & Fruiting: March to May.

i. Collection of plant material:

Plants *Butea monosperma* will be collected from remote area of Soegaon region near Ajantha caves. Subsequent visit will be planned to photograph the plant in proper blooming period and confirm the folk of medicinal uses. The plant parts will bring to the laboratory. After completion of description, identification and noting medicinal uses the plant will be subjected for phytochemical constituents through literature. The plant parts will be dried, crushed to make powder and the powder material used for the study of phytochemical test.

The collected flowers were cleaned and shade dried. Fresh samples were used for anatomical studies and dried parts were powdered, sieved and stored in an airtight container for further use.

ii. Extraction of Plant Drug:

The fine powdered plant material will be subjected to extraction in soxhlet apparatus. The powdered plant material will be subjected to successive solvent extraction using different organic solvents such as methanol, Ethanol, Acetone, chloroform, and Ethyl acetate. Twenty grams of powdered plant material will be used to soxhlet extraction with 250 ml of the various organic solvents for 8 hrs. Each time before extracting with next solvent, the powder residue will dried. Extract obtained in each solvent will be concentrated, solidified and used for preliminary phytochemical analysis.

iii. Phytochemical Screening:

For preliminary phytochemical screening powder tuber of *Butea monosperma* will be subjected to various qualitative chemical tests to determine the presence of various phyto-constituents like glycosides, tannins, phytosterols, proteins, amino acids, flavonoids, saponins etc

iv. Physicochemical evaluations:

Physico-chemical parameters of the powdered drug such as total ash, water-soluble ash, acid insoluble ash and sulphated ash were determined. Extractive value, solubility tests, moisture test, mineral content and nutritive value (ash, fat, fiber, protein and carbohydrate) of flowers were determined as per standard procedures

RESULTS AND DISCUSSION

Phytochemical analysis was performed on the hot water, methanol acetone chloroform and ethyl acetate extract of *Butea monopserma*. Hot water extract was found to contain proteins, amino acids, alkaloids, phenolic compounds, glycoside, carbohydrate protein. Methanolic extract contains carbohydrates, glycosides, alkaloids, flavonoids, saponins Acetone extract conain

steroid carbohydrate protein glucoside .chloroform extract contain carbohydrate glucoside and ethyl acetate extract contain alkaloids phenolic compounds carbohydrate protein. (Table 1.)

This work is beneficial for analyzing the quality and purity of the crude drug. In this study different parameters used for the evaluation of *Butea monosperma* like moisture content, extractive values ash values. Physicochemical evaluation of *Butea monosperma* was shown that the results of physicochemical constants found within limit. (Table no. 2) This indicates that the quality and purity of raw material was good enough.

Table no.2: Physico-chemical analysis of *Withania somnifera*

Sr. No.	Physicochemical parameters	Values (in %w/w)
1	Total ash value	7.3%
2	Water soluble ash	3.02%
3	Acid insoluble ash	1.04%
4	Moisture content	4.80%
5	Alcohol soluble extractive value	9.25%
6	Water soluble extractive value	7.16%

Table 1: Preliminary Phytochemicals Screening of *Butea monosperma* flower

Sr.N.	Test	Hot water	Methanol	Acetone	Chloroform	Ethyl Acetate
I	**Alkaloids**					
	Mayer's Te	+	+	-	-	+
II	**Flavonoids**					
1	Shinod test	-	+		-	-
2	Lead acetate test	-	+			
III	**Phenolic compound and Tannins**					
	FeCl3 test	+	+	-	-	+
IV	**Terpenoids**					
	Liebermann Burchards Test	-	-	-	-	-
V	**Steroids**					
	salkowski test	+	+	+	-	-
VI	**Carbohydrates**					
	Fehling's Test	+	+	+	+	+
VII	**Protein**					
	Millon's Test	+	+	+	-	+
VIII	**Saponins**					
	Foam Test	+	+	-	-	-
IX	**Glucosides**					
1	Keller-Killian test	+	+	+	+	-
2	Legal's test	+	+	+	+	-

The result of moisture content 3.64%w/w implies that the drug is properly dried and stored. The value of moisture content 4.80%w/w implies that the drug is properly dried and stored. The physicochemical parameter like total ash is an important as it shows the purity of drug, which implies presence or absence of foreign material like metallic salts etc. The physicochemical analysis result for total ash was found to be 7.3. W/w. The ash value lies within limit implies purity and quality of crude drug. The water soluble extractive value found to be 7.16%. While ethanol soluble extractive value found to be 9.25% w/w.

CONCLUSION

The phytochemical screening confirmed the presence of various phytochemical constituents such as alkaloids, flavonoids, amino acids, carbohydrate, proteins, saponin and tannins. Phytochemical constituents confirmed utilization of flower for therapeutic medical treatment. The people of India are turning to usage of medicinal plants and phyto-chemicals in health care. Different Physicochemical parameters such as, total ash, acid insoluble ash, water soluble ash, water soluble extractive and alcohol soluble extractive value were observed.All studied standardization parameters like phytochemical screening and physicochemical parameters provide the knowledge in the identification authentication of *Butea monosperma.*

Conflicts of interest: The authors stated that no conflicts of interest.

REFERENCES

1. Sindhia VR, Bairwa R. Plant review: Butea monosperma. International Journal of Pharmaceutical and Clinical Research 2010; 2(2): 90-94.
2. Ayitey Smith E., Addae-Mensah I. W., *Afr. J Pharmacol Drug Res, 1977; 4, 7-8.*
3. Cooke T, *Flora of presidency of Bombay*, 1967; 3B, 236-37.
4. Cragg GM., David JN, *J Pharm Biol,*2001;39, 8-17.
5. Gaikwadi SS, Vadlamudi VP, Waghmaee, Maral SP, Ranteke VJ, Dhok AP., *Phytochemical analysis of aqueous extract of few medicinal plants*,*PKV. Res. J.,* 2003; 27(1): 91-92.
6. Gill LS, *Ethnobotanical uses of plants in Nigeria*, ,*University of Benin Press,* 1992:350.
7. Hey, SA and N Sheng. Utilization and Conservation of Medicinal Plants in China with Special Reference to Atractylodes Lancea, in, Medicinal Plants for Forest Conservation and Health Care, G.C. Bodeker (Ed.) FAO, Rome. 1997.
8. Geeta R, Prakash R, et al "Butea monosperma (LAM.) KUNTZE: A Review" *Int Res J of Pharma,* 2011; 2 (7):98-108.
9. Varsha S, "Therapeutic Significance of Butea monosperma: A Review" *J of Drug Delivery & Therapeutics,* 2011; 1(2): 63-67.

RESEARCH ARTICLE   **OPEN ACCESS**

Recent developments in *Actiniopteris radiata*

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Manuscript Details

Cite this article as:

ABSTRACT

Actiniopterisradiata (family: Actiniopteridaceae) is commonly used as Indian traditional medicine. *Actiniopteris radiata* has the common names of Morpankhi, Mayurishika and Peacock's tail. Traditionally *Actiniopteris radiata* is used alone or with other medicinal plants to treat a variety of ailments. The plant contains rich in tannins, flavanoids, steroids, glycosides, hydrocarbons, Coumarins, triterpenoids and phenolic compounds. The plant used injaundice, typhoid fever, epilepsy, diarrhea, tuberculosis, etc. The plant extract also possesses different pharmacological activities such as, anti-microbial activity, analgesic activity, anti-oxidants, anthelmintic, anti-stress and anti-allergic activities. The present review could form a sound basis for further investigation in the potential discovery of new natural bioactive compounds and provide preliminary information for future research.

Keywords: Actiniopterisradiata, bioactive compounds, medicine.

INTRODUCTION

The history of herbal medicine beginning from the ancient human civilization [1], man has familiarized himself with plants and used them in a variety of ways throughout the ages. In search of food, man began to differentiate those plants suitable for alimentary purpose from others with definitive pharmacological action [2]. The use of plants as herbal medicines is as old as human civilization and number of the ancient medicinal systems

such as Ayurveda, homeopathy, naturopathy, siddha, unani and other alternative medicinal systems has been utilizing plants as effective source of medicines to cure many diseases [3]. Herbal medicine, as an alternative to synthetic drugs, have played an important role at the level of basic, public health care in various countries, especially in Asia. During the previous several years, number of various natural products from plants has been characterized as useful as pharmaceuticals or nutraceuticals, due to this number of the herbal medicine have drastically increased in market availability and public usage worldwide [4]. In a report of World Health Organization (WHO), medicinal plants would be the best source to obtain a variety of drugs. It is reported that more than rocky crevices 400, 000 plant species of tropical origin have medicinal properties. About 80 % of individuals in developed countries are using traditional medicine, of plant origin and the natural product for their health due to minimal side effect and cost effective Therefore, in Current research by major pharmaceutical companies such plants should be investigated to understand their properties, safety and efficacy and for a search of new potent activity [3,5,6] .*Actiniopteris radiata*is a tiny terrestrial fern found throughout India. It is also called as Peacock`s tail. It is of limited distribution, and in areas where it occurs is restricted to depleted walls and of steep slopes of exposed hilly areas, up to the altitude of 1200 m above msl. *Actiniopteris radiata*(Sw.) Link. Belonging to *Actiniopteridaceae* family is an herb with great medicinal value. According to Ayurvedic texts

Mayurashikhaa (*A.radiata*) is used as astringent, anti-inflammatory, tonic to genitourinary tract, alleviates vitilated blood, indicated in cough, bronchitis, asthma, diarrhea, dysentery, dysuria, Antihistaminic and Anticholinergic Activity, used internally as well as externally for infected wounds, ulcers, erysipelas, *A. radiata*has been reported to possess anti-helminthic and styptic property [3,4,7,8].

Geographical distribution
Actiniopteris radiata belonging to family (*Actiniopteridaceae*) is a tiny terrestrial fern, commonly known as Peacock`s tail. It is an important medicinal plant widely distributed throughout India and also in Burma, Sri Lanka, Afghanistan, Iran, Arabia, Yemen, South Eastern Egypt, Tropical Africa, Australia and Madagascar. It is of limited distribution, and in areas where it occurs, is restricted to depleted walls and rocky crevices of steep slops of exposed hilly areas, roadside, up to the altitude of 1200 m [9]. Black or reddish gravelly soil is best suited for its growth.

Morphology of Actiniopterisradiata
The Small xerophytic plantis 8-25 cm high rooting in the crevices of rocks or in between in the joints of bricks walls in most and rocky soil long roadside usually in lime rich/alluvia soil. The rhizomes is oblique to horizontal, 1.5 to 2.0 cm in length, densely covered with wiry roots, palaea and leaf bases The young leaves show cricinate venation but the lamina becomes flat at an early stage of development.

Table: Scientific Classification and Local names of *Actiniopterisradiata*

Scientific Classification: [1,10]	Local names:[1,7]

• Kingdom: Plantae – Plants.
• Phylum: Magnoliophyta– Flowering.
• Class: Magnoliopsida – Dicotyledons.
• Order: Malpighiales.
• Family: Actiniopteridaceae.
• Subfamily:Euphorbioideae.
• Synonym : A. australis (L. f.) Link.
    A. radiata (Sw.) Link.
    A. dichotoma Kuhn.
• Tribe :Euphorbieae.
• Genus :*Actiniopteris*- Linnaeus, 1753.
• Specific epithet: *radiate.*
• Botanical name: *Actiniopterisradiata*
	• Hindi– Morpankhi.
	
	• Sanskrit – Mayurishika ,
	  Madhuchhadaa,Sahastrahi, Vahrishikhaa.
	
	• Telugu– Nemaliadugu.
	
	• Tamil–Mayilatumshikhai.
	
	• English– Peacock's tail.

Fig.No.1 Whole plant of *Actiniopteris radiata*

The laminae are fan-like, stiff and rough to touch. The sporangia are sub-marginal on an inter-marginal vein covering almost the entire biaxial surface of dichotomously segment [11, 12].

Ethno-medicaluses

Whole plant paste is directly applied on cuts surface and wounds: past with sugar is given to kill intestinal worms twice for 3 days. The plant paste with sugar candy is given as a cooling agent in case of syphilis .The paste of two fronds is given daily two times a day to children to cure rickets. The whole plant paste mixed with cow's milk is given for the treatment of piles and leucorrhoea. In milk is given twice a day for 2-3 days. In case of epilepsy, plant paste with sugar candy is given[1]. 5-6 leaves paste with sugar is also given two times a day as an aphrodisiac, also used as tonic to increase the potency[11].The whole plant is also use for the treatment Typhoid[13]. The Fresh paste/dried powder of leaves (5-6) with 1 tsp. honey is given twice a day for the treatment of leucorrhoea and also increase fertility. The paste of 5-6 leaves mixed with fresh cow milk (nearly 200ml) is taken for a week or so, to overcome irregularly in menstrual period. The leaves or leaves ash (approx. 2-3 g) of the plant mixed with fresh cow milk (200 ml) is given to a lady for fortnight after menses for conception. On the other hand the paste of 8-10 leaves mixed with thin curd (nearly 250 ml) is given for birth control. Paste with sugar is used orally for twice a day, as strength tonic to increasing the potency in women [14,15,16]. Plants are dried and one tea spoonful powder is taken orally, once a day for four days in the case of cough. Plants are soaked overnight in a glass of water and

taken orally in morning for control of blood pressure and decoction of leaves is also used in tuberculosis .The whole plant is also use for the treatment asthma, fever, leprosy and hair growth. The juice extracted from the stem is taken orally twice a day to treat diarrhea[17,18].

PHYTOCHEMICAL:

Several chemicals have been identified from *Actiniopteris radiate* plant are known to be alkane hydrocarbon chains (hentriacontane, hentriacontanol) and flavonoid glycoside (quercetin-3-rutinoside) (Rutin) ,steroidal compounds (β-sitosterol, β-sitosterol palmitate, β-sitosterol-D-glucoside)[19] .glycosides are (2-(3, 4-O – Diglucos cinnamoyl) – 4 – hydroxyl furan and 1-Heptaloyl, 8-hexyl, 3-(O – diglucos), 10 – methyl, 9. 10 – dihydro naphthalene.)[20].The other Phytochemical are Dec-3-enyl, 2-(heptyloxy)-1-mercaptpoethyl terephthalate and 1-(Phenylethy-lbutanoate ether), 2-(3-ene heptanoate) ethane [21]. Glucose, fructose, alkaloids, Flavonoids, tannins, Saponins, Quinones, Cardio glycosides, Terpenoids, Coumarins, betacyanin and phenols [22]
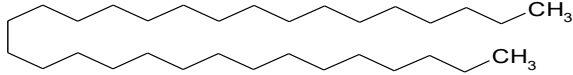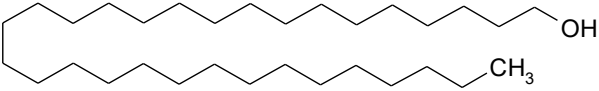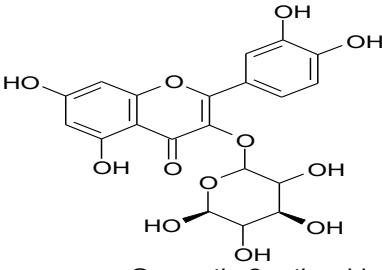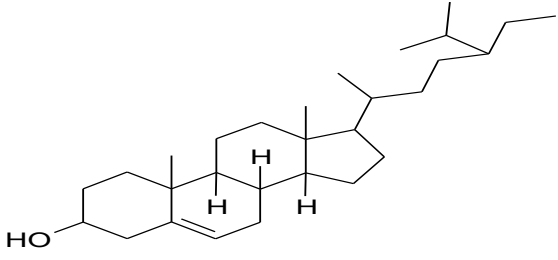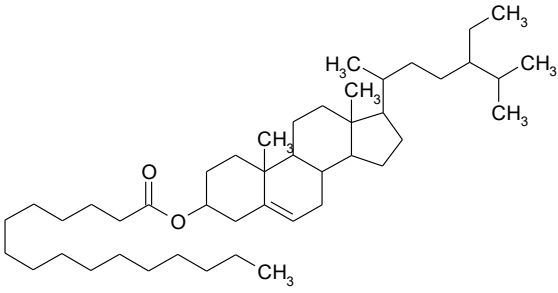
PHARMACOLOGICAL PROPERTIES
Analgesic activity:

The effect of the aqueous and ethanolic extract of plant Actinopteris radiata has been investigated in mice to evaluate the analgesic activity by using Acetic acid induced writhing and Tail flick method. Administration of A. radiata extracts shown effective analgesic activity in the Acetic acid induced Writhing

method, whereas both extracts shown non-significant analgesic activity in Tail flick method. Therefore it is indicated that both plant extracts shows only potent peripheral mediated analgesic activity and inhibits predominantly peripheral pain mechanism. The extracts have found to produce marked analgesic effect due to the presence of alkaloids, tannins, flavonoids and phenolic acid. [1]

Invitro Antihistaminic and Anti cholinergic activity:
The crude ethanolic extract *Actiniopteris radiata* (Sw.) Link has been investigated to assess the spasmolytic effect on isolated rat ileum. The crude extract at a dose of 2, 4, 10 mg/ml dose dependently relaxed the rat ileum which was contracted due to the presence of agonist like histamine and acetylcholine [1,8].

Name of compound	*Structure*
Alkane Hydrocarbon Chains • *hentriacontane*	 Hentriacontane
• *hentriacontanol*	 Hentriacontanol
Flavonoid Glycoside • *quercetin-3-rutinoside*	 Quercetin-3-rutinoside
Steroidal Compounds a) *β-sitosterol*	 ß- Sitosterol
b) *β-sitosterol palmitate*	 ß-sitosterol palmitate

c) *ß-sitosterol-D-glucoside*

ß-sitosterol-D-glucoside

Glycosides
a) *2-(3, 4-O – Diglucoscinnamoyl) – 4 – hydroxyl furan.*

2-(3, 4-O–Diglucos cinnamoyl) – 4 – hydroxyl furan

b) *1-Heptaloyl, 8-hexyl, 3-(O – diglucos), 10 –methyl, 9. 10 – dihydro naphthalene.)*

c) *Dec-3-enyl, 2-(heptyloxy)-1-mercaptpoethyl terephthalate*

d) *1-(Phenylethylbutanoate ether), 2-(3-ene heptanoate) ethane*
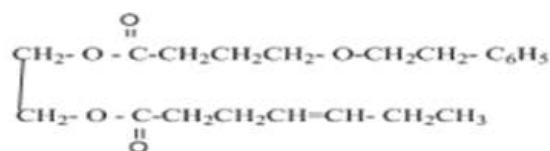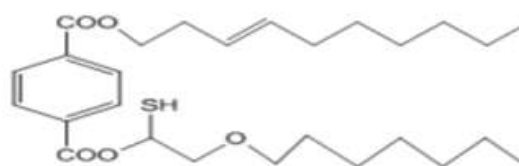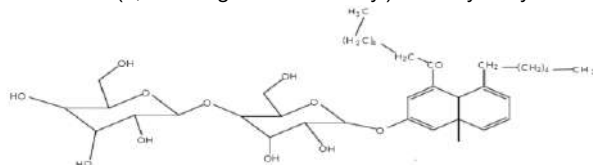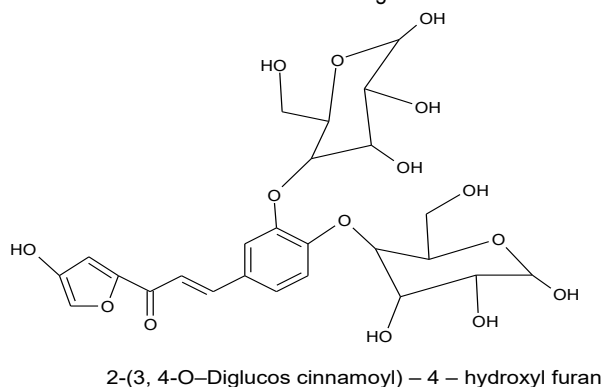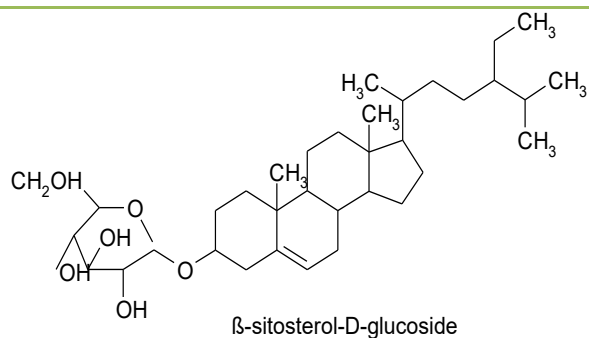
Antimicrobial activity
Anti-bacterial activity
Thestudy was undertaken to investigate the antibacterial activity of the fern, Actenopteris radiata by well diffusion method. The whole plant was extracted successively with certain alcoholic and aqueous solvents (petroleum ether, chloroform, acetone, methanol and water). Different concentrations of crude extracts such as 100, 200 and 300 µg/mL were prepared to found antibacterial activity. Chloramphenicol was used as standard drug.

The results of the shows that water and methanolic extracts of the plant had strong antibacterial activity in all the concentrations than the other solvents used. whole plant methanol and water extracts inhibits effectively the growth of the Gram-positive bacteria viz.,*Bacillus subtilis* and *Staphylococcus aureus* and Gram-negative bacteria viz., *Escherichia coli* and *Salmonella paratyphii* at all concentrations (100 to 300µg/mL) than that of other solvent extracts used Therefore, the plant extract may be used to control the diseases caused by pathogenic bacteria[3].

Antifungal Activity

Plant extract of *Actiniopteris radiata* and *Caralluma adscendens* was evaluated against *Candida Albicans, Aspergillus niger* and *Mucor* by Cup-plate method at the concentration of 150 mg/mland 300mg/ml using Griseofulvin used as standard drug. Diameters of the zones of inhibition were determined as an indication of activity after incubating the plates at 250C for 72 hrs. An average of three independent determinations was recorded. The anti-microbial activity was shown significant results due to the presence of flavonoids, tannins and sterols [9].

Wound healing activity

The ethanolic extract of the *Actinopteris radiata has* been investigated toevaluated for its wound healing activity in rats. Wound healing activity was studied using Excision, Incision and Dead space wound models in rats following topical application and compared with a standard 5% w/w Povidone-iodine ointment. 10% w/w *Actinopteris radiata* ointment was prepared for topical application and 5% w/w Povidone-iodine ointment was used as a standard. On excision and incision wound models the alcoholic extract of *Actinopteris radiata* produces good wound healing activity and comparable with that of 5% w/w Povidoneiodine ointment. The alcoholic extract of *Actiniopteris radiata* shown dose dependent effect on granulation tissue and hydroxyproline content. The results clearly indicated good wound healing activity [1]

Antitubercular activity

The Antitubercular activity of n-Hexane, Chloroform, Ethanol extracts was prepared from whole plant of *Actiniopteris radiata Linn* was evaluated against *Mycobacterium tuberculae*using Microplate Alamar Blue assay. Minimum inhibitiory concentration (MIC) was taken to assess antitubercular activity. The minimum inhibitory concentration of n-Hexane, Chloroform and Ethanolic extracts was (12.5, 3.125, 25µg/ml) respectively and compared with Pyrazinamide (3.125µg/ml) and Streptomycin (6.25µg/ml) was taken as standard drug. The results shown that Chloroform extract has more significant antitubercular activity as compared to n-Hexane, Ethanolic extracts [23].

Antioxidant and Radical Scavenging Activity

Medicinal plants are recognized as sources of natural antioxidants that can protect biological system from oxidative stress. In this study, the antioxidative potential of different solvent extracts (*n*-hexane, chloroform and ethyl acetate, ethanol and aqueous) of plant *Actiniopteris radiata* (Sw.) link were evaluated using different *in vitro* methods. From all the solvent extracts, ethanol extract of *A. radiata*, showed potent activity and the inhibitory concentrations of extract at 50% (IC50) were 742.11, 670.80, 505.902, 372.43 and 425.23 µg/mL for DPPH, superoxide and hydroxyl radical scavenging activities, metal chelating and anti-lipid peroxidation activities, respectively and has nearly similar to that of the standard antioxidant tested. Moreover, ethanol extract of *A. radiata* showed strong reducing power, which denotes the antioxidant capacity of the extract. The results shown that the ethanolic extract of *A. radiata* is a good source of natural antioxidants [4].

The antioxidant activities of methanolic extracts of *Actiniopteris radiata* and *Equisetum ramosissimum.* The analyses carried out were DPPH radical scavenging, ABTS +, reducing power. From the analyses, *Actiniopteris radiata* and *Equisetum ramosissimum*, were found to have potent antioxidant activity against DPPH with the IC50 value of 93.48 and 78.58 respectively. *Actiniopteris radiata* had the highest values for ABTS + radical scavenging activity (2523.11µ TE/g) and reducing power assay (0.853 absorbance at 700µg/ml)[24].

Anthelmentic activity

The Ethanol extract of whole plant of *Actiniopteris radiata Linn*. has been evaluated for *invitro* anthelminthic effect. In present study different concentration of Extract 50 mg/ml, 100 mg/ml, 200 mg/ml were investigated for its anthelminticactivity. The paralysis time [P] and death time [D] was comparable with standard Albendazole 20 mg/ml.The result indicates that the 200 mg/ml Extract posses significant anthelmentic activity. Dose dependent effects were observed with 50 mg/ml, 100 mg/ml, and 200 mg/ml [25]

Anti-stress and anti-allergic effect

The extracts of whole plant of *Actiniopteris radiata was*screened for its therapeutic potential as an antiallergic and antistress agent in asthma using specific *in vivo* animal models. Only ethanol extract at a higher dose of 100 mg/kg i.p significantly (p<0.05) decreased milk induced eosinophilia by 16.20 ± 2.235 when compared with control group while even lower doses of 50 mg/kg, i.p exhibited significant inhibition (P<0.05) of leukocytosis induced by milk in mice. Other extracts like petroleum ether, ethyl acetate and methanol unable to exhibit that significant potential. Results obtained thus validate the traditional claim of the *Actiniopteris radiata* utilization in different aspect of asthma due to presence of various polar secondary metabolites in ethanol extract [26]

Hepatoprotective Activity

The study has been investigated phytoconstituents, acute oral toxicity and Hepatoprotective Activity of ethanolic extract of *Actiniopteris radiata* by using CCL4 induced Hepatotoxicity in wistar albino rat. The ethanolic extract at the dose of 100,200,400 mg/kg p.o and standered drug used as silymarin 100mg/kg was administered in rat and Hepatoprotective Activity was assessed by estimating various bicohemical parameters like SGPT, SGOT, ALP, serum bilirubin, followed by histo pathological studies. Ethanolic extract of *Actiniopteris radiata* at the doses of 200 and 400 mg/kg (p.o.) offered significant (p<0.01) hepatoprotective activity by reducing the serum marker enzymes like SGPT, SGOT, ALP, bilirubin. Whereas at the dose of 100 mg/kg (p.o) EEAR exhibited moderate hepatoprotection (p<0.05) compared with the of the standard silymarin (p<0.001). Histo pathological studies of ethanolic extract of *Actiniopteris radiata* revealed that, the normal cellular architecture was retained as compared to silymarin, there by further confirming the potent hepatoprotective effect. Hepatoprotection exhibited by ethanolic extract of *Actiniopteris radiata* against CCI4 induced hepato toxicity was found to be dose dependent and significant as evidenced by the biochemical and histo pathological parameters. The protective effect of ethanolic extract of *Actiniopteris radiata* against CCL4 may be due to the presence of flavonoids, sterols, triterpenoids and phenolic compounds [27].

CONCLUSION

From the detailed literature review, it is concluded that the plant is a potential source of drugs that can be used in the treatment of different dermal ailments and disease areas. This plant is important traditional plant containing different steroidal and glucosidal compounds which can be used in skin disease treatment and owning different anti-microbial activities. A complete exploration and development work should be commenced for the preservation of *Actiniopteris radiata* and development of products for their restored fiscal and therapeutic application. Furthermore, there is a space for exploration to establish lead compounds for drug development. This evidence will be supportive for pharmacognosists, ethnobotanists, botanists and pharmacologists.

Conflicts of interest: The authors stated that no conflicts of interest.

REFERENCES

1. Ramalingeswara Reddy P. and Sreenivasulu M, Chand Basha. S,*Actiniopteris radiata* (linn.): a comprehensive review, Journal of Global Trends in Pharmaceutical Sciences, April–June 2012, Vol.3, pp. 612-618.
2. R. Ansari and k. Ekhlasi-kazaj, a review on adiantum capillus-veneris. L, *j adv sci res*, 2012, 3(4): 15-20.
3. Moorthy D, S. Paulsamy, K. Nandha Kumar1 and M. Saradha, In vitro antibacterial activity of the fern, *Actiniopteris radiata* (Sw.) Link.inhabiting the Shervaroyan Hills, the Eastern Ghats. J. of Chem. and Pharmaceutical Research, 2013, 5(7):211-214.
4. M. Manjunath, G. Lavanya, R. Sivajyothi and O.V.S. Reddy, Antioxidant and Radical Scavenging Activity of *Actiniopteris radiata* (Sw.) Link., Asian J. Exp. Sci., Vol. 25, No. 1, 2011; 73-80.
5. Santhosh Kumar s, samydurai p, ramakrishnan r and n nagarajan, gas chromatography and mass spectrometry analysis of bioactiveconstituents of adiantum capillus-veneris l.,international journal

of pharmacy and pharmaceutical sciences,l 6, issue 4, 60-63.

6. Hubert d. Nyarko, victor y. A. Barkuand joseph batama,antimicrobial examinations of cymbopogon citratus and adiatum capillus-veneris used in Ghanaian folkloric medicine, international j. Of life science and research, vol 2/issue 2/apr-jun 2012.

7. R.Manonmani and sara preliminary phytochemical analysis of A.radiata international Research journal of pharmacy,2013.

8. Vivek R. Alai, Dr. Gautam P. Vadnere, Invitro Antihistaminic and Anticholinergic activity of *Actiniopteris radiata* (Sw.) Link. On isolated rat ileum, International Journal of pharmtech Research, Vol. 3, No.1, pp 05-07, Jan-Mar 2011.

9. J.B Naik, Jadege D.R., Antibacterial and Antifungal activity of *Actiniopteris radiata* (Sw.) Link, 2010, 2, 1751-1753.

10. C.P. Khare (Ed.), Indian medicinal and aromatic plants, Springer Science, 2007.

11. Shweta Singh and Rita Singh,ethno medicinal use of pteridophytes in reproductive health of tribal women of pachmarhi biosphere reserve, Madhya Pradesh, India, ijpsr, 2012; vol. 3(12): 4780-4790.

12. Pradeep parihar and leena, some pteridophytes of medicinal importance from Rajasthan, natural product radiance, vol.5(4),2006.

13. Sharad maroti Porte,overview of folk medicine used for Typhoid in India. Int.j.res in Ayurvedapharm 5(2), Mar-Apr 2014.

14. Shweta Singh and RitaSingh, ethno gynecological use of pteridophytes in pachmarhi biosphere reserve, MadhyaPradesh, India, life sciences leaflet 9, 1st sept 2012 *life sciences leaflets* 9: 63-71(iii),2012.

15. R.S.Gupta and Rakhi Sharma review on medicinal plant exhibiting antifertility activity in male, *Natural product radiance*, .5 (5), 2006 pp.839-410.

16. Shweta Singh and RitaSingh, utilization of pteridophyte of Achanakmar-Amarckantak biosphere reserve, central India in women's health and beauty care practices , International research journal of pharmacy, 2013, Vol. 4, pp235-240.

17. R. Revathi, R. Muthuraja, Binu Thomas and K. Raju, ethno medicinal fern and fern-allies used by tribe malayalis of kolli hills,Eastern Ghats, Tamil nadu, pteridological research, 15 June, 2013; vol.2(1):1-10.

18. Santhosh Kumar S, Samydurai P and N Nagarajan, Indigenous Knowledge on Some Medicinal Pteridophytic Plant Species among the Malasar Tribe's in Valparai Hills, Western Ghats of Tamilnadu, *American Journal of Ethnomedicine*, 2014, Vol. 1, No. 3, 164-173.

19. K. Hemalatha, n. Sunandana rao and n. Shivaji, phytochemical constituents of *Actiniopteris radiata* Linn. Whole plant, annals of phytomedicine an international journal, vol. 1(1): pp. 34-38, 2012.

20. Lakshmi narasimhaiah, m. J. N. Chandrasekhar and s. Alexander,isolation of new glycosides from pteridophyte plant, international journal of pharmacy and pharmaceutical sciences, vol 4, suppl 4, 2012.

21. Lakshminarasimhaiah* and y. Vamshi Vishnu,isolation of new phytoconstituents from actiniopteris radiate, international journal for pharmaceutical research and review.

22. Kalpana DeviRajesh, Subramanian vasantha, nakulan valsala Rajesh, annamalai panneerselvam, qualitative and quantitative phytochemical analysis in four pteridophytes, int. J. Pharm. Sci. Rev. Res., 27(2), July – august 2014; article no. 72, pages: 408-412.

23. Sreenivasulu munna, S. Chand Basha, P. Ramalingewara Reddy,N. Pramod,y. Pradeep kumar,g. Mahaboob basha, antitubercular activity of *actiniopteris radiata* Linn, journal of global trends in pharmaceutical sciences volume- 5, issue -1-pp-1443-1445, January – march 2014.

24. S. Paulsamy, d. Moorthy, k. Nandakumar and m.saradha, evaluation of in vitro antioxidant potential of methanolic extracts of the ferns, *actiniopteris radiata* (sw) link. And equisetum ramosissimum desf, international journal of research and development in pharmacy and life sciences,April - May, 2013, vol. 2, no.3, pp 451-455.

25. V. Sreenivasul1, s. Chand basha, y. Adinarayan1, v. Lingaswam1, U. N. Praveen Kumar, anthelmentic activity of *actiniopteris radiata* (linn.), journal of global trends in pharmaceutical sciences vol5(2)-(2014) pp. 1716 –1718.

26. Gautama prakash vadnere, aslam ramjan Pathan, abhay KumarSonghai, bhakti uday kulkarni1 and jogendra chandrakant hundiwale, anti-stress and anti-allergic effect of *actiniopteris radiata* in some Aspects of asthma,pak. J. Pharm. Sci., vol.26, no.1, January 2013, pp.195-198.

27. K. Naga sravanthi, P. Rajeshwara rao, J. Niranjan, Hepatoprotective activity of ethanolic extract of Actiniopetris radiate Linn against ccl_4 induced hepatotoxicity in Wistar albino rats, International , 2012, Vol. 1, pp201-206.

RESEARCH ARTICLE　　　　　　　　　　　　　　　　　　　　**OPEN ACCESS**

Role of public sector banks in Environmental protection

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ABSTRACT

The present era of Economic Development and Globalization has added a lot of comfort and luxury to human life. But on the other hand it has resulted in the exploitation of the natural environment which in turn has disturbed the ecological balance. The disturbance in ecological balance has adversely impacted the human and its surrounding environment Many research studies have pointed out that increase in carbon emissions and reckless development without paying attention to environment can be a reason of occurrence of natural disasters. All over the world institutions are concerned about the overall impact of depletion of environment Understanding the importance of the existence of human health & well being governments as well as the organizations started taking various initiatives for the concern of environmental protection and sustainability Banks and financial institutions are also playing a major role in optimizing or reducing carbon footprints. Green Banking is also a part of India's mission of "Go Green" and plays an active role to take environmental and ecological aspects as part of their lending principle. Green banking is comparatively a new development in the financial world. Green banking means promoting environmental friendly practices and reducing carbon footprint from banking activities. . It is a form of banking taking into account the social and environmental impacts and its main motive is to protect and preserve environment. This paper is focus on how Public Sector banks in India are initiating themselves in Environmental protection

Keywords Environmental Sustainability; Green Banking; exploitation; Environmental protection.

INTRODUCTION

Banks are the most important institutions that help business activities. And it play major role in the development of country's economy. According to section 5(1) (b) of Banking Regulation Act, 1949; banking as; "Accepting, for the purpose of lending or investment, of deposits of money from the public, repayable on demand or otherwise, and withdraw able by cheque, draft, order or otherwise." As of 31 March 2017 the Indian banking system consists of 27 public sector banks,26 Private sector banks,46 Foreign Banks,56 Regional Rural Banks, 1,574 urban Co operative banks and 93,913 rural Co operative banks. The public sector banks contribute maximum to 70 % of total credit and deposits businesses in India. The banking sector has contributed 7.7 percent of total GDP of the country in the financial year 2016- 2017. The banking sector in India is growing very rapidly, it assets had reached up to Rs US $ 1,518 billion in year 2017 as compared to Rs 7.5 trillion in the year 1998. As the banks are among one of the major sources of financing instrument for commercial projects so they can play a major role in promoting environmental sustainability by funding the socially and environmentally responsible investment projects.

Objective of the Study:
1. To understand the role of Banking sector in sustainable development of country
2. To discuss the various strategies used by different banks in Environmental protection
3. To highlight the initiatives taken by Indian banks to make them environment friendly.

METHODOLOGY

The study mainly includes literature review from secondary data. The Secondary data sources include articles, research papers, annual reports of the respective banks and other relative information published on the banks and company's official websites. The study also includes the primary data collection through personal visit to bank and in- depth interview of the branch managers and staff.

ENVIRONMENTAL SUSTAINABILITY

Environmental Sustainability is 'the ability to maintain the things that are valued in the natural and biological environments. Environmental sustainability could be defined as 'a condition of balance, resilience, and interconnectedness that allows human society to satisfy its needs while neither exceeding the capacity of its supporting ecosystems to continue to regenerate the services necessary to meet those needs nor by our actions diminishing biological diversity'. Environmental system seeks to sustain the global life support system indefinitely. In then developing countries the problems related to the environment have become very critical and their dependence on natural resources for the growth and development underline the need of implement policy and plans for sustainable resource use. Due to increasing effect of industrialization, urbanization, increasing population density and poor environment management system in India the environmental problems have become alarming issues. So the time demands the need of using the natural resources in a sustainable way; i.e. meeting the needs in such way that it not only meet the needs of present but also for the future generation

GREEN BANKING

The concept of Green Banking is attached to Triodos bank (established in 1980) from Dutch origin which started the environmental sustainability in the banking sector from the very first day. In the year 1990 the bank launches 'Green fund' for funding environment friendly projects and all other projects follow later. Taking example from this bank the banks all over the world start taking green initiatives in the banking sector. Institute for Development and Research in Banking Technology defines Green Banking as '*Green Banking* is an umbrella term referring to practices and guidelines that make banks sustainable in economic, environmental, and social dimensions. It aims to make banking processes and the use of IT and physical infrastructure as efficient and effective as possible, with zero or minimal impact on the environment'. The India Banks Association defines it as "Green bank function like a normal bank along with considering the social and environmental

factors for the protection of natural resources". According to RBI (IDRBI, 2013) "Green banking is to make internal bank processes, physical infrastructure and Information Technology effective towards environment by reducing its negative impact on the environment to the minimum level" . Green Banking concept can be defined in a number of ways in a broader perspective; it is the environment friendly banking practices that promote their customers to reduce the carbon footprint through their banking activities. In December 2007, the Reserve Bank of India (RBI) issued a circular (RBI 2007-2008/216) highlighting the importance for banks to act responsibly and contribute to sustainable development and emphasizing the need for Indian banks to establish institutional mechanisms to enshrine sustainability.

State Bank of India (SBI)

SBI is the India's oldest and largest banking institution in almost every parameter such as net profit, revenue, assets and market capitalization. The bank was established in 1806 as Bank of Calcutta and became as the State Bank of India in the year 1955 during the nationalization of Imperial Banks in India. The bank offers various ranges of activities such as investment banking, consumer banking, commercial banking, assets management, pension, insurance, mortgages and credit card, through it   of branches in country and overseas. As of 31 March 2017, it had assets of 27, 05,966 corer and 24,017 branches 25.85 lakh customers and 2, 09,567 employees are working in its branches. It is involved in community services activity since1973 and throughout the country the bank participates and sponsors various social cause and welfare activities

a. SBI launched the concept of SBI Digital Village to convert certain identified village into a cashless eco system 21 villages were launched on 1st July 2016.
b. SBI launched SBI Mingle the social media banking platform for facebook and twitter users ,the bank customers can do a host of banking services like checking account balance & requesting mini statements on their face book and twitter accounts.

c. On the occasion of State Bank Day, the bank had launched 'Green Channel Counter' (GCC) facility on 1st July 2010 at 57 select branches spread across the country and the same was expanded to more than 14,981 branches in 2017. 8.00 lakh transaction are being routed through GCC per day.This is a pioneering concept which is eco-friendly and convenient. This approach helps to make paper less banking up to some extent
d. SBI became the first bank in the country to venture into generation of green power by installing windmills for captive use in 2010.) Collaboration with Suzlon Energy Ltd. to use wind power at the place of thermal power in its business operations and currently using wind power in its most of offices located in Gujarat, Tamil Nadu and Maharashtra .
e. SBI offers an interest discount of 10 basis points on all environment friendly projects.
f. More over SBI is the largest deployer of solar ATMs in this country.
g. SBI initiated the carbon disclosure projected in the financial sector in India, for the sake of environmental concern and safety by becoming a signatory to the Carbon Disclosure Project of World Wide Fund (WWF).
h. The Bank has put in place SMART i.e. Specific, Measurable, Achievable, Realistic and Time bound Green Banking Goals.
i. Initiated the carbon disclosure projected in the financial sector in India, for the sake of environmental concern and safety
j. SBI and Export- Import Bank of India (EXIM Bank) both jointly provide a long term loan to a Spain based companies Grupo T- Solar Global SA and Astonfield Renewable Resources for building solar plant in India. Most of the financial institutions avoid giving long term loans to such projects because of their uncertainty and chnological changes.
k. Green home loan scheme which supports environment friendly housing projects and offer subsidy and interest rates reduction.

Punjab National Bank(PNB)

PNB is among one of the India's oldest bank and was established on 19 May, 1894. Presently it's the second

largest public sector bank by assets in India. As of 31 March 2017, it had assets of 667390 corer and 6937 branches 80 million customers and 70,801 employees are working in its branches. It offers a wide variety of financial services there for customers like consumer banking, corporate banking, equity, mortgage, credit card, finance & insurance banking, wealth management etc. Aside from financial activities the bank is also involved in various social initiatives such as farmer empowerment, environment friendly initiatives, education & health initiatives for the financially disadvantaged category.

a. A separate green audit sheet is being employed by the bank to access the impact of various green banking initiatives implemented in the bank.
b. The bank has sanctioned nine commercial projects of wind energy comes with total sum of Rs. 1850.81 million to push and develop the renewable supply of energy.
c. The bank introduced Green Banking by completing transformation into Core Banking Solutions (CBS) Banking.
d. Bank has started using energy efficient appliances & conducting the electricity auditing of their offices.
e. The bank has conjointly placed guideline for supply of term loan to business units and commercial projects that are producing renewable energy.
f. The bank introduced e-solutions by paperless dealings in complaint & vigilance procedures by a speedier communication system through e-network which drastically cuts down cost & time.
g. The bank has introduced "Green practices" to conserve resources "Green audit" of all functional units to sensitize the staff towards sustainable practices.
h. Bank has gone for installation of Solar UPS at selected ATM sites like in states of U.P and Bihar.

Bank of Baroda (BOB)

BOB is an Indian state owned (public sector) bank established in the year 1908 in the princely state of Baroda. It provides various ranges of banking products and financial services through its branches such as corporate banking, investment banking, retail banking, asset management etc. to the consumers. In year 2017 the bank was ranked 1145 on Forbes Global 2000 list. As of 31 March 2017, it had assets of 694,875.42 corer and 5538 branches and 52420 employees are working in its branches The bank also has its international presence with 100 branches in 24 countries outside India with its presence in major financial center such as New York, Dubai, and Hong Kong, Singapore etc. The bank has taken various corporate social responsibility initiatives upheld inclusive growth high on its agenda.

a. Internet banking, mobile banking was added as alternate delivery channel to reduce the use of paper in banking procedure.
b. As a part of green banking initiatives various changes were made such as backup consolidation, server and desktop virtualization.
c. While financing the commercial projects the banks give a due weight age to green projects such as windmills and solar power projects which helps in earning the carbon credit.
d. The bank insisted to implement water treatment plant and obtain NOC from central/ state government pollution control board while lending the loan to manufacturing units which emit toxic polluting substance.
e. Promotion of measure of pollution control and efforts for environmental protection & conservation and cleaning of environment.

Canara Bank

Canara Bank is an Indian public sector bank and was established as Canara Hindu Permanent Fund in 1906 and further renamed as Canara Bank in 1910 As of 31 March 2017, it had assets of 552,960.78 corer and 6639 branches and 59413 employees are working in its branches The bank has a widespread network of 3564 branches and 4000 ATMs spread across the country. The bank also has abroad offices in Dubai, Hong Kong, London, Moscow and Doha. The bank was ranked 816 by Forbes Global 2000 list. Various financial services such as asset management, commercial banking, investment banking, consumer banking, credit card and mortgages are being provided by the bank. It also sponsors two regional rural banks in Kerala and Karnataka. In the year 2003

the bank partnered with UNEP for solar project under development project initiatives. Along with this bank is also involved in activities such as rural development and environment friendly initiatives.

a. The bank has implemented various green banking initiatives such as internet banking, tele-banking & mobile banking. Solar power biometric ATMs has been implemented in a few rural areas.
b. Now the bank is not extending the finance to the new units which are involved in producing and consuming Ozone depleting substances. The bank has also stopped extending the finance small/medium scale unit engaged in the manufacturing of Aerosols by using CFC.
c. The bank insisted to manufacturing units which emit toxic polluting substance to implement water treatment plant and obtain NOC from central/ state government pollution control board while lending the loan.
d. The banking is providing loans for implementing solar lighting system, till the date the bank has financed 50,000 such unit lending 5-8 lac Rs to each unit

Bank of India (BOI)

BOI is commercial bank, founded in 1906, it has been government owned since nationalization in 1969 BOI As of 31 March 2017, it had assets of 609,913.93corer and 5,100 branches and 45,613 employees are working in its branches

a. Provision of Internet banking, mobile banking, phone banking and electronic cards was added as alternate delivery channel to reduce the use of paper in banking procedure.
b. Reduction in paper usage by issuing e-transaction advices to corporate customers and encouraging electronic statements among retail customers.
c. Various green initiatives such as distribution of tree saplings and creating awareness among society have been undertaken by the bank.
d. The bank has undertaken initiatives to popularize use of solar street lights and rain water harvesting in rural areas.

e. Bank has establishment of a 2 KV Solar Plant under Lok Birdhari Prakalp –project of The Maharogi Seva Samiti, a non-profit organization to take care of healthcare needs of tribal people.

CONCLUSION

Indian economy is an emerging economy and there is a huge potential of growth of Indian banks by adoption of innovative approach in their strategy making process. The banking sector is getting modernized and new facilities such as net banking, mobile banking are being prioritized at the place of traditional approaches. These new approaches adopted by the banks are beneficial to customers as well as banks themselves. Now the banks understood the importance and necessity of environment along with the economic progress of a nation. Learning from their western counterparts the banks in India are also adopting various environmental practices and initiatives in their day to day business operations for the environmental concern and playing an important role in maintaining the ecological balance. There is a need of an approach towards paradigm shift by setting up of the business model which would consider all the three aspect of triple bottom line approach i.e. the people, the planet and the profit. Indian banking sector is still at the initial stage to adoption of innovative approach in their strategy making process .As most of the banks are adopting and focusing only on those green initiatives which provides win-win situation for the bank, that help to show the concern for the environment along with helping the bank in cost savings and improved operational efficiency. So the time demands a little focus on the initiatives such as creating awareness among society, and helping smaller firms to change their process so they can be more environmentally friendly in nature and that will also widespread the concept of environmental sustainability.

Conflicts of interest: The authors stated that no conflicts of interest.

REFERENCES

1. Mishra DK, Green Strategies: Response of Indian Banks to Climate Change.

2. Todaro, Michael P and Stephen C Smith. Economic Development, 8e. Delhi: *Pearson Education, 2003.*

3. Misra SK and Puri. Growth and Development. Mumbai:,*Himalaya Publishers*, 2005.

4. Bank of Baroda Annual report 2016-17,

5. SBI Annual report 2016-17,

6. BOI Annual report 2016-17,

7. Canara Bank Annual report 2016-17,

8. PNB Annual report 2016-17,

9. "The Indian Banker", monthly journal published by Indian Banks Association edition February 2016.

Effects of Global warming and Climatic Change

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ABSTRACT

A comprehensive assessment of the impacts of climate change is a grave threat faced by humankind. Changes in the environment-reportedly largely due to greenhouse gases released into the atmosphere by human activity-threaten to make earth uninhabitable for humans. The unnatural climate change we are expecting is the manifestation of a change in the climate of human kind over the past several centuries. To this end an integrated ecological-economic modeling framework is employed, encompassing climate scenarios, agro-ecological zoning formation, socio-economic drivers, as well as world food trade dynamics. Specially, first, impacts of different scenarios of climate change on bio-physical soil and crop growth determines of yield are evaluated on a 5'X5' latitude global grid; second, the extent of potential agricultural land and related potential crop production is computed. The detailed bio-physical results are then fed into an economic analysis, to assess how climate impacts may interact with alternative development pathways, and key trends expected over this century for food demand and production, and trade, as well as key composite indices such as risk of hunger and malnutrition, are computed. This modeling approach connects the relevant bio-physical and socio-economic variables within a unified and coherent framework to produce a global assessment of food production and security under climate change. The results from the study suggest that critical impact asymmetries due to both climate and socio-economic structures may deepen current production and consumption gaps between developed and developing world; it is suggested that adaptation of agricultural techniques will be central to limit potential damages under climate change.

Keywords: climate, greenhouse gases, bio-physical and socio-economic

WHAT IS GLOBAL WARMING?

Global warming is the increase of Earth's average surface temperature due to effect of greenhouse gases, such as carbon dioxide emissions from burning fossil fuels or from deforestation, which trap heat that would otherwise escape from the earth. This is a type of greenhouse effect. Global warming can be defined as an increase in earth's atmospheric, oceanic temperatures and on overall change in earth's atmosphere including a rise in sea levels and variability of snowfalls. Climatic change and associated impacts vary from region to region around the globe .Due to increase in greenhouse effect resulting especially from pollution and other activities such as, greenhouse gas emissions produced by human activities mainly industrial processes and transportation.

Causes of Global Warming:

Global warming is primarily a problem of too much carbon dioxide in the atmosphere which acts as a blanket, trapping heat and warming the planet. As we burn fossil fuels like coal, oil and natural gas for energy or cut down and burn forests to create pastures and plantations, carbon accumulates and overloads our atmosphere. Certain waste management and agricultural practices aggravate the problem by releasing other potent global warming gases, such as methane and nitrous oxide.

GLOBAL WARMING IMPACTS

Rising Seas:

Inundation of Freshwater marshlands, low lying cities and islands with seawater.Global sea levels rose about 15mm between Nov2014 and Feb2016 as a result of El Nino,well above the post-1993 trend of 3 to 3.5 mm per year.

Change in Rainfall pattern:

Droughts and fires in some areas, flooding in other areas.

Increased likelihood of extreme events:

2016 is slated to become the hottest year on record with parts of the Arctic now 20 degrees celcius above normal-that's akin to 50 centigrade .

Melting Glaciers:

Significant melting of old glaciers is already observed.

Widespread vanishing of animal Populations:Wide spread habitat loss

Spread of disease:Migration of diseasesand spread of epidemics

Bleaching of Coral reefs:

Due to warming of seas and acidification due to carbonic acid formation-one third of coral reefs now appear to have been severely damaged by warming seas.

Loss of Plankton due to warming seas:

The enormous 900 mile long Aleutian island ecosystems of orcas, sea lions, sea otters, sea urchins, kelp beds and marine economic populations, appears to have collapsed due to loss of plankton. As our climate changes, the risk of injury, illness and death from the resulting heat waves, wildfires, intense storms and floods increases.

THE CAUSES OF GLOBAL WARMING

It took more than 25 years to broadly classify to broadly accept that mankind is causing global warming with the emission of greenhouse gases. the drastic increase in the emission of Carbondioxide with in the last 30 years caused by burning fossilfuels has been identified as the major reason for the change of temperature in the atmosphere. More than 80% of the world –wide energy demand is currently supplied by the fossil fuels coal, oil or gas. It will be impossible to find alternative sources, which could replace fossil fuels in the short or medium term. The energy demand is simply too high.

EFFECT OF GLOBAL WARMING

Where do you take evidence that "the temperature rising and falling does drive Carbon dioxide levels "in the atmosphere? Neither vegetation nor oceans "produce carbon dioxide .The ocean water can store some carbon dioxide the warmer the water the lower the amount of carbon dioxide which can be absorbed earlier. This is called a feedback-effect of the warming

of the oceans. It should not be mixed up with the cause of warming. Almost 100% of the observed temperature increase over last 50 years has been due to the increase in the atmosphere of greenhouse gas concentrations like water vapor, carbon dioxide, methane and ozone. Greenhouse gases are those gases that contribute to the greenhouse effect.The largest contributing source of greenhouse gas is the burning of fossil fuels leading to the emission of carbon dioxide.

The Greenhouse effect:

When sunlight reaches the earth's surface some is absorbed and warms the earth and most of the rest is radiated back to the atmosphere at a longer wavelength than the sunlight. Some of these longer wave lengths are absorbed by greenhouse gases in the atmosphere before they are lost to space. The absorption of this long wave radiant energy warms the atmosphere. These greenhouse gases act like a mirror and reflect back to the earth some of the heat energy which would otherwise be lost to space. The reflecting back of heat energy by the atmosphere is called the "greenhouse effect".

Greenhouse gases in the atmosphere act like a mirror and reflect back to the earth a part of the radiation, which would otherwise be lost to space. The higher the concentration of greenhouse gases like carbon dioxide in the atmosphere, the more heat energy is being reflected back to the earth. The emission of carbon dioxide in to the environment mainly from burning fuels like oil, gas; kerosene etc. has been increased dramatically over past 50 years.

SOLUTIONS TO GLOBAL WARMING

There is no single solution to global warming, which is primarily a problem of too much heat-trapping carbon dioxide, methane and nitrous oxide in the atmosphere. The technologies and approaches outlined below are all needed to bring down the emissions of these gases by at least 80 percent by mid-century. To see how they are best deployed in each region of the world.

Greening transportation:

The transportation sector's emissions have increased at a faster rate than any other energy using sector over past decade.A variety of solutions are at hand,including improving efficiency in all modes of transport, switching low-carbon fuels,and reducing vehicle miles traveled through smart growth and more efficient mass transport system.

Revving up renewable:

Renewable energy sources such as solar, wind, geothermal and bioenergy are available around the world. Multiple studies have shown that renewable energy has the technical potential to meet the vast majority of our energy needs. Renewable technologies can be deployed quickly, are increasingly cost-effective, and create jobs while reducing pollution.

Managing forests and Agriculture:

Taken togeather, tropical deforestation and emissions from agriculture represent nearly 30 percent of the world's heat trapping emissions. We can fight global warming by reducing emissions from deforstations and forest degradiation and by making our food production practices more sustainable.

Exploring nuclear:

Nuclear power results in few global warming emissions, an increased share of nuclear power in the energy mix could help reduce global warming-but nuclear technology poses serious threats to our security and as the accident at the Fukushima plant in Japan illustrates to our health and the environment as well. The question remains: can the safety, proliferation, waste disposal, and cost barriers of nuclear power be overcome.

Developing and deploying new low –carbon& Zero-carbon technologies:

Research into the development of the next generation of low-carbon technologies will be critical to deep mid-century reductions in global emissions. Current research on battery technology, new materials for solar cells, harnessing energy from novel sources like bacteria and algae, and other innovative areas could provide important breakthroughs.

Ensuring sustainable development:
Various countries of the world most of the least developed vary dramatically in their contributions to the problem of climate change and in their responsibilities and capacities to confront it. A successful global compact on climate change must include financial assistance from richer countries to poorer countries to help make the transition to low-carbon development pathways and to help adapt to the impacts of climate change.

CONCLUSION

Global warming is a big issue for concern which has found its place in big discussion rooms and is an alarm with no snooze button for the human beings which wakes them up from deep slumber where they dreamt for acquiring profits for their means and in a long run forget to look after their guardians our nature, which is the source of everything we live on,from food, water and even the air we take into respire. But we are polluting the same water we will drink, polluting the same air we take into respire and extensive use of strong chemical fertilizer which is running our crops instead of making them healthy. We as human expect a lot from others, but are we giving our nature back the same thing? The answer is yes, but we are giving our nature the return gift for the favors of the nature the return gift for the favor of the nature, protecting us as pollution of water and air, destruction of forests and exploitation in the form of a mining of our mother earth.

In order to make our own world we are destroying worlds of millions who have their home on earth, which includes the animals, birds and insects as well, who have supported and protected every time we were in trouble. Not only this contribution and sacrifice of these animals, trees and the whole ecosystem around us are the elements which have helped us in evolution from a nescient to an effective thinker. Is this that they deserve? Our heart pumps and our mind throttles back saying, of course not at all. We have to nurture the nature, love it and to do that first we will have to understand what constant troubles we the social animals are creating for mother earth, and the trouble which forces us each of us to ponder and analyze before it is too late.

Conflicts of interest: The authors stated that no conflicts of interest.

REFERENCES

1. Arunkumar tawar,Global Climate Change, (2012) *Common wealth Publishers,New Delhi-110059*

2. Manoj Singh, Climatology (2012) *Sharada Pustak Bhavan,Allahabad*

3. Rajendra Singh. Pollution& world water Problem (2014) *S. Chand Publication, New Delhi*

4. Rudra Dutta, KP Sundaram, Indian Economy (2015) *Himalaya Publications*,New Delhi

Proximate analysis of *Phyllanthus amarus* leaves

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ABSTRACT

Natural products have traditionally provided many of the drugs in use. Despite the achievement of synthetic chemistry and advances towards rational drug design, natural products continue to be essential in providing medicinal compounds and as starting points for development of synthetic analogous. With the increasing power of screening programme and increasing interests in the reservoir of untested natural products, many future drug developments will be based on natural products.

Phyllanthus amarus is an annual weed commonly known as bhumi amla in India and is traditionally used to treat flu, dropsy, diabetes, and jaundice. It is also used to treat hepatic and urolitic diseases and have diuretic, antiviral, anticancer, hepatoprotective, antioxidant anti-inflammatory activity. It mainly contains phyllnathin and hypophyllanthin as active ingredients. The aqueous extract of this plant had been employed for treatment of nervous debility, epilepsy, as medhya (intellect promoting) and in vata disorders. In the present study, we reports the proximate survey and phytochemical screening of *Phyllanthus amarus.* The whole plant material was tested for proximate survey shows **1.96**% foreign organic matter, **7.32**% ethanol soluble extractives, **17.21**% water soluble extractives, **7.22**% total ash, **4.68**% Acid insoluble ash, 11.38% loss on drying and **2.00**% moisture content.

Keywords: phytochemicals, proximate analysis, bhumiamla, Phyllanthus amarus

INTRODUCTION

Most of the crude drugs (Plant materials) are usually put in quarantine store and they remain there for long time. During storage proper ventilation, humidity controls, suitable temperature and light conditions should be ensured to maintain their original pharmacological action. However, it is observed that, crude plant materials, before being taken for processing, are not analyzed which can lead to changes in original characteristics. To avoid this, the crude drugs should be tested for the following tests as per the USP and Indian Herbal Pharmacopoeia (IHP). The Study includes Foreign organic matter, Ethanol soluble extractives, Water soluble extractives, Total ash contents, Acid insoluble ash, Water soluble ash, Loss on drying and Percentage moisture content.

Foreign Organic Matter

Medicinal plant materials should be entirely free from visible signs of contamination, i.e. moulds, insects and other animal contamination, including animal excreta, fungus and dust. It is seldom possible to obtain marketed plant materials that are entirely free from some form of innocuous foreign matter. However, no poisonous, dangerous or otherwise harmful foreign matter or residue should be allowed. Any soil, stone, sand, dust and other foreign organic matter must be removed before medicinal plant materials are cut or ground for testing. Macroscopic examination can conveniently be employed for determination of foreign matter in whole or specific plant material.

METHODOLOGY

SAMPLING

Phyllanthus amarus plant material of selected plants were collected from various places in Junnar Taluka in bulk, washed thoroughly with water to remove the dust particles on the surface of the plant and the soil particles adhering to the roots. Excess water was allowed to drain off by spreading the plant material on filter papers. Then 500gm of the washed and drained plant material of each plant was taken and spread as a thin layer on a white, clean muslin cloth. Foreign matter was sorted by visual inspection and by using magnifying lens (6x). The portions of the sorted

foreign matter were weighed and the contents of foreign matter in grams per 100 grams of the sample were calculated. The procedure was carried out for a total of five sets.

Extractable Matter

This method determines the amount of phytoconstituents extracted with solvents from a given amount of medicinal plant material in the form of powder. Here according to Indian Herbal Pharmacopoeia ethanol and water were used as common solvents to determine the extractable matter.

Procedure

Accurately weighed five grams plant material was placed in glass-stoppered conical flask. To it 100 cm^3 of water was added. The flask was shaken frequently for six hours, and then allowed to stand for eighteen hours. The contents were filtered rapidly to avoid loss of solvent. The filtrate was transferred to a previously weighed clean beaker and evaporated to dryness on a water-bath. After evaporation the extract was dried at 105°C for six hours and kept in a desiccator for cooling. The beaker was weighed and percent extractable matter in water was calculated. The above procedure was repeated thrice for determination of water-soluble extractable matter.

Ethanol soluble extractable matter was determined by following the above procedure except ethanol was used instead of water, as extracting solvent. The experiment was repeated for three times.

Ash Content

The ash remaining following ignition of medicinal plant materials is determined by three different methods, which measures

The Total Ash method is designed to measure the total amount of material remaining after ignition. This includes both 'physiological ash', which is derived from the plant tissue itself, and 'non-physiological ash', which is the residue of the extraneous matter (e g sand and soil) adhering to the plant surface.

Acid-Insoluble Ash is the residue obtained after boiling the total ash with dilute hydrochloric acid and igniting the remaining insoluble matter. This

measures the amount of silica present as sand and siliceous earth.

Water-Soluble Ash is the difference in weight between the total ash and the residue after treatment of the total ash with water.

Total Ash

The total ash was obtained by taking Accurately weighed 2 g of the dried plant material was taken in a tarred Silica dish and was ignited with a flame of Bunsen burner for about one hour. The ignition was completed by keeping it in a muffle furnace at 550°C ± 20°C till grey ash was formed. It was then cooled in desiccators and weighed. The process was repeated (ignition, cooling and weighing) till the difference in the weight between two successive weighing was less than 1 mg.

Acid Insoluble Ash

Acid Insoluble Ash was obtained by following method.

Procedure

Accurately weighed 2gm of the dried plant material was taken in a porcelain/silica dish and was ignited with a bunsen burner for about one hour. The porcelain dish was kept in a muffle furnace at 550°C ± 20°C till grey ash was obtained. The ash was moistened with concentrated HCl and evaporated to dryness after which it was kept in an electric air oven maintained at 135°C ± 2°C for 3 hr. After cooling, 25 cc. of dilute HCl was added, and was kept covered with watch glass and heated on a water bath for 10 minutes. It was then allowed to cool, and was filtered through Whatmann filter paper No. 41. The residue was then washed with hot water till washings were free from chloride (as tested with $AgNO_3$ solution). The filter paper and the residue were put in a dish and ignited in a muffle furnace at 550°C ± 20°C for one hour. The process of cooling in a desiccators and weighing was repeated till the difference between two successive weights was found to be less than one mg.

Water-Soluble Ash

Water soluble ash was obtained by following method.

Procedure

Twenty-five cm3 of distilled water was added in a silica dish containing the total ash and boiled for ten minutes. The insoluble matter was collected on an ash-less filter paper. The residue was washed with hot water and ignited in a crucible for fifteen minutes at a temperature not exceeding 450°C. The weight of this residue was subtracted from the weight of the total ash and the water-soluble ash was calculated.

Loss on Drying

The percentage of loss on drying was obtained by following method.

Procedure

Five grams of plant powdered sample was weighed in wide mouthed stoppered weighing bottle. The bottle was then placed with lid open in an air oven maintained at 100°C ± 2°C. The sample was kept in an oven for 2 hours. The bottle was then removed, covered and placed in a desiccator. The bottle was weighed after cooling to room temperature and weighed.

The bottle was again kept in the oven for 2 hrs. and the above procedure was repeated (heating. cooling and weighing) till the difference in the weight between two successive weighing was less than 1 mg. Three readings for each sample were recorded.

Moisture Content

The moisture of plant powders were obtained by Karl-Fischer Titrimetric Method

Procedure

Reaction vessel was rinsed thoroughly with methanol magnetic stirring rotor was inserted in the vessel and placed in proper position. The large rubber cork was removed and some K/F grade methanol was added using funnel, to the reaction vessel just enough to submerge the metal wires of sensors in the reaction vessel. The cork was replaced immediately. The K/F reagent and methanol bottles were placed in position. Then the instrument was turned on and the speed of magnetic stirrer was adjusted. Methanol was neutralized and the titer factor was determined by calibrating the K/F reagent. This was done by adding 10 µl of distilled water with the help of a µL syringe in the reaction vessel and completing the titration. The calibration of the reagent was done in triplicate. The readings were noted and the titer factor was calculated. The data for determination of titer factor is

given in following table QC 8 and it was calculated using the following formula.

RESULTS AND DISCUSSION

The results of proximate analysis were obtained and are found to be **1.96%** foreign organic matter, **7.32%** ethanol soluble extractives, **17.21%** water soluble extractives, **7.22%** total ash, **4.68%** Acid insoluble ash, 11.38% loss on drying and **2.00%** moisture content

Conflicts of interest: The authors stated that no conflicts of interest.

REFERENCES

1. Lee CD, Ott M, Thyagarajan SP, Shfritz DA, Burk RD, Gupta S. Phyllanthus amarus down regulates hepatitis B virus m RNA transcription and translation, *Euro J Clin Invest*,1996; 26: 1069-1076.

2. Joy KL, Kuttan R. Antioxidant activity of selected plant extracts. *Amala ResBull* ,1995,15: 68-71.

3. Prakas A, Satyan KS, Washi SP, Singh RP. P. urinaria, P. niruri and P. simplex, on carbon tetrachloride induced liver injury in the rat. Phytother Res,1995; 9: 594-596.

4. Rajesh Kumar NV, Kuttan R. Phyllanthus amarus extract administration increases the life span of rats with hepatoprotective carcinoma,*J Ethnopharmacol* ,2000; 73: 215-219.

5. Shirish S. Pingale, "Phytochemical Analysis of Root Bark of *Argemone Mexicana* L", *Annals of Plant Sciences, ISSN: 2287-688X* (2012-2013),2013, 02 (04):117-118,

6. Sharma AC, Kulkarni SK. Evidence for GABA-BZ receptor modulation of short- term memory Passive avoidance task paradigm in mice. *Methods Find Exp Clin Pharmacol* ,1990,12: 175-180.

7. Shirish S. Pingale, "Proximate Analysis of Leaves of *Phyllanthus Amarus*",*International Journal of Multidisciplinary Research, ISSN 2277 9302*, October-2013, Vol II. Issue 7(V): 41-43,

8. Somanabandhu A, Nityangkuru S, Mahidol C. 1H and 13C NMR assignments of phyllanthin and hypophyllanthin lignans that enhance cytotoxic responses with cultured multidrug-resistant cells. *J Nat Prod* ,1993; 56: 233-239.

9. Uttam B. Shelar, Shirish S. Pingale, Manohar G. Chaskar, 'Recent Developments of Caesalpinia Decapetala', *International Journal of Scientific Research in Science and Technology(IJSRST), Print ISSN: 2395-6011, Online ISSN: 2395-602X*, November-December2017,3(9):100-105,.
URL: http://ijsrst.com/IJSRST173920.php

10. Shirish S. Pingale, Rajeshwari S. Oza, 'Proximate Analysis of Terminalia Chebula Leaves ', *International Journal of Scientific Research in Science and Technology(IJSRST), Print ISSN: 2395-6011, OnlineISSN:2395-602X*,2017,3(10):43-45,
URL: http://ijsrst.com/IJSRST1731011.php

11. Shirish S Pingale and Bharat P More, "Phytochemical Screening of Terminalia chebula fruits", *CTBC's International Research Journal, Special Issue on Advanced Analytical Techniques, ISSN 2350-0905. December 2014, Volume 1, Issue 3 (Special Issue) : 24-27.*

12. *Shirish S. Pingale, Sunita S. Salunke-Gawali and Bharat P. More*, "Proximate Analysis of Terminalia Chebula Fruits", *IJPRD, February-2015 (69-72), International Journal of Pharmacutical Research and Development, ISSN 0794-9446,www.ijprd.com, 2014; Vol 6(12): 69-72.*

Review of Air Quality in Pimpri - Chinchwad, MS, India.

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ABSTRACT

Air pollution is the introduction into the atmosphere of chemicals, particulates, or biological materials that cause discomfort, disease, or death to humans, damage other living organisms such as food crops, or damage the natural environment or built environment. Pollutants with the strongest evidence for public health concern include particulate matter (PM), ozone (O_3), nitrogen dioxide (NO_2) and sulphur dioxide (SO_2). Pimpri-Chinchwad is fastly growing city and Asia's one of the biggest automobile hub. Due to increase in industries and urbanization, the air and water pollution has become a big problem. Due to huge number of vehicles, the percentage of Sox, NOx and RSPM is very high and air in many areas has become unhealthy. Long-term exposure to polluted air can have permanent health effects such as accelerated aging of the lungs, loss of lung capacity and decreased lung function, development of diseases such as asthma, bronchitis, emphysema, and possibly cancer.

Keywords biological materials, chemicals, particulates, diseases such as asthma, bronchitis, emphysema, and possibly cancer.

INTRODUCTION

World Health Organization (WHO) has defined that air pollution is contamination of the indoor or outdoor environment by any chemical, physical or biological agent that modifies the natural characteristics of the atmosphere. Air pollution can be outdoor or indoor. Outdoor air pollution has its origin from natural and anthropogenic sources.

Natural sources contribute substantially to local air pollution whereas anthropogenic sources lead to air pollution at global level. The contribution from human activities far exceeds natural sources. Human activities that are major sources of outdoor air pollution include –

1. Fuel combustion from motor vehicles
2. Heat and power generation plants and boilers
3. Industrial facilities such as manufacturing factories, mines, and oil refineries
4. Municipal and agricultural waste sites and waste incineration/burning and
5. Residential cooking, heating, and lighting with polluting fuels

According to the World Health Organization (WHO) India is home to 13 of the 20 most polluted cities in the world. Half of Delhi's 4.4 million children have permanent lung damage that they will never fully recover from. Pune is ranked 52 in the world's most polluted cities and 20 in India. [1] The 2017 State of Global Air report, published by the Health Effects Institute, shows that air pollution-related deaths in Indian between 1990 and 2015 rose by almost 150%. 3 million deaths every year as a result of exposure to ambient (outdoor) air pollution. There are 4.3 million deaths every year as a result of household exposure to smoke from dirty cook stoves and fuels. [2] An estimated 3 million premature deaths globally are linked to ambient air pollution, mainly from heart disease, stroke, chronic obstructive pulmonary disease, lung cancer, and acute respiratory infections in children.[3]

Classification of Air Pollution

Air pollution is classified into two types-

1. Primary Pollutants- Primary pollutants are substances that are directly emitted into the atmosphere from sources. The main primary pollutants known to cause harm in high enough concentration are CO, CO_2, CH_4, NO, N_2O, NH_3, Sulfur, H_2S.

2. Secondary Pollutant- Secondary Pollutants are not directly emitted from sources but instead from in the atmosphere from primary pollutants (also called precursors). The main Secondary Pollutants known to cause harm in high enough concentration are NO_2, HNO_3, NO, O_3, VOC, H_2SO_4, nitric acid. Organic aerosols formed from VOC in gas to particle reactions. The following table shows sources and effects of the main pollutants.

Pollutant	Sources	Effects
Sulfur Dioxide (SO_2)	Thermal power plants and industries, industrial boilers and processes, coal-burning stoves, heaters.	eye irritation, dead aquatic life, lung damage, acidic precipitation, damage to property and forests
Nitrogen Oxides (NOx)	Vehicles, industrial boilers, power plants, commercial and residential heaters. coal burning stoves	Lung damage, forms acid rain and destructs roads, buildings, statues and forests
Hydrocarbons	Dry cleaning operations. auto paint shop, service stations, chemical plants, auto emission.	Carcinogenic, damage Lungs.
Particulates	Diesel engines, Power plants, steel industry, flour mills, windblown dust, wood stoves.	Eye irritation, damage to lungs, damage to crops, reduce visibility,
Ozone	Vehicle exhaust. photochemical smog	Eye irritation, Lung damage, respiratory tracts problems.
Lead	Vehicles burning, leaded gasoline, power plants, metal refineries.	Brain, kidney damage, Smog formation.
Carbon monoxide	vehicles burning, gasoline, kerosene- or wood-burning stoves, dry cleaners	Headache reduced mental alertness, damage to heart. death, smog
PM10 and PM2.5	Transportation, Brick kilns, Stone crushing units, automobile industries.	Respiratory Diseases, Skin and eye related problems, Carcinogenic.

Measurement of Air Pollution

Air pollution is measured in terms of air quality index (AQI) . An air quality index (AQI) is a number used by government agencies to communicate to the public how polluted the air currently is or how polluted it is forecast to become. As the AQI increases, an increasingly large percentage of the population is likely to experience increasingly severe adverse health effects. An increase in air quality index signifies increased air pollution and severe threats to human health. In most cases, AQI indicates how clear or polluted the air in our surrounding is, and the associated health risks it might present. The AQI centers on the health effects that may be experienced within a few days or hours after breathing polluted air.

AQI calculations focus on major air pollutants including: particulate matter, ground-level ozone, sulfur dioxide (SO_2), nitrogen dioxide (NO_2), and carbon monoxide (CO). Particulate matter and ozone pollutants pose the highest risks to human health and the environment. For each of these air pollutant categories, different countries have their own established air quality indices in relation to other nationally set air quality standards for public health protection.

Air Quality Index Categories
The AQI is divided in six categories and each category is meant to correspond to different health concern levels. Below is an explanation of the categories and their meanings.

Study Area
Pimpari Chinchwad is situated at a height of 530 m above the sea level. It has pleasant climate all the year round. There are three rivers Pavana, Mula and Indrayani flow through this area. Pimpri Chinchwad is a part of Pune Metropolitan City in the state of Maharashtra, India. It is the urban agglomeration of Pune. It consists of the towns of Pimpri, Chinchwad, Nigdi, Akurdi, Ravet, Bhosari, Pimple Gurav, Moshi, Punawale, and Sangavi, which are governed by a common municipal body - Pimpri Chinchwad Municipal Corporation or PCMC. It is located to the north-west and is connected to the centre of Pune city via the Old Pune-Mumbai Highway.

The Pimpri Chinchwad is adjoining city of Pune. The Pimpri-Chinchwad Municipal Corporation has developed very fast in terms of industrial, commercial and residential growth. The MIDC has also set up industrial centers in the area known as Chinchwad, Pimpri and Bhosari. Geographically, the area falling in Pimpri-Chinchwad Corporation is situated in between River Mula & River Indrayani. Air, water and sound pollution in Pimpri Chinchwad is on the rise. This is attributed to increasing urbanization, growth of industries, and changed lifestyle.

Meanings of AQI categories

Numerical Value	Air Quality Index Levels of Health Concern	Meaning
0 to 50	Good	Air quality is considered satisfactory, and air pollution poses little or no risk
51 to 100	Moderate	Air quality is acceptable; however, for some pollutants there may be a moderate health concern for a very small number of people who are unusually sensitive to air pollution.
101 to 150	Unhealthy for Sensitive Groups	Members of sensitive groups may experience health effects. The general public is not likely to be affected.
151 to 200	Unhealthy	Everyone may begin to experience health effects; members of sensitive groups may experience more serious health effects.
201 to 300	Very Unhealthy	Health warnings of emergency conditions. The entire population is more likely to be affected.
301 to 500	Hazardous	Health alert: everyone may experience more serious health effects

Source: http://airnow.gov/index.cfm?action=aqibasics.aqi

Sources of air pollution in Pimpri Chinchwad

Pune is one of the major industrial hubs in Asia and many of these industries are situated in Pimpri-Chinchwad area. Industrialization started in 1954 with the arrival of Hindustan Antibiotics Limited in this area. PCMC is now home to the Indian operations of major automobile companies like Premier Limited, Mahindra & Mahindra Ltd - Truck & Bus Division, Mahindra Engineering services, Bajaj Auto, BEL Optronic Devices Limited, TATA Motors (formerly TELCO), Kinetic Engineering, Force Motors (formerly Bajaj Tempo) Daimler Chrysler, Thermax and Autoline Industries. In addition to this, several heavy industries such as Forbes-Marshall, Thyssen Krupp and GEA Ecoflex, Alfa Laval & Sandvik Asia have their manufacturing units in the town and also the German company KSB Pumps, Swedish bearing company SKF [4].

Database and methodology

The Central Pollution Control Board (CPCB), back in 1984, initiated National Ambient Air Quality Monitoring (NAAQM) at the national level to regularly monitor ambient air quality of selected major urban cities and industrial towns of the country. This was later renamed as the National Air Monitoring Programme (NAMP). The ambient air quality monitoring network involves measurement of a number of air pollutants at different strategic locations in the country. The task of any monitoring network thus involves the selection of pollutants, the selection of locations, frequency, duration of sampling, sampling techniques, infrastructural facilities, manpower, operation, and maintenance. [5]

In Pimpri-Chinchwad city instruments have been installed to measure air pollution, at two places - Sector23, Water Purification Center, Nigadi and Growth Lab, Nasik Road, Bhosari, with the help of IITM - (Indian Institute of Tropical Meteorology). Also LED DISPLAY (Light Emitting Diode), showing the level of air quality, have been installed at Pimpri Chowk circle and Chaphekar Chowk. National Ambient Air Quality Standards

Sr. No	Pollutants	Time Weighted	Industrial, Residential, Rural and Other Area	Ecological Sensitive area (Notified by Central Government)
1	SO$_2$	24 hours	80	80
2	NO$_X$	24 hours	80	80
3	RSPM(PM10)	24 hours	100	100

Courtesy- http://www.pcmchelpline.in/helpline/english/callcenter-faq.php?type=21

Real-time Air Quality Index (AQI) from January to November 2017 is given in the table.

| Month	SOx			NOx			RSPM		
	Min	Max	Avg	Min	Max	Avg	Min	Max	Avg
January	13	59	25.4	55	158	110.12	85	266	146.6
February	18	68	30.12	9	166	85.41	61	271	136.76
March	13	50	28.92	29	177	67.85	31	186	89.96
April	15	40	24.68	13	80	37.68	39	211	81.96
May	13	31	25.71	21	142	54.67	26	191	74.25
June	9	39	19.12	12	67	29.75	15	378	56.58
July	8	39	16.96	18	59	32	15	115	34.96
August	9	27	16.92	20	79	36.96	11	69	29.46
September	13	31	19.7	19	62	40.7	6	81	29.13
October	6	27	16.15	20	96	54.04	27	378	104.46
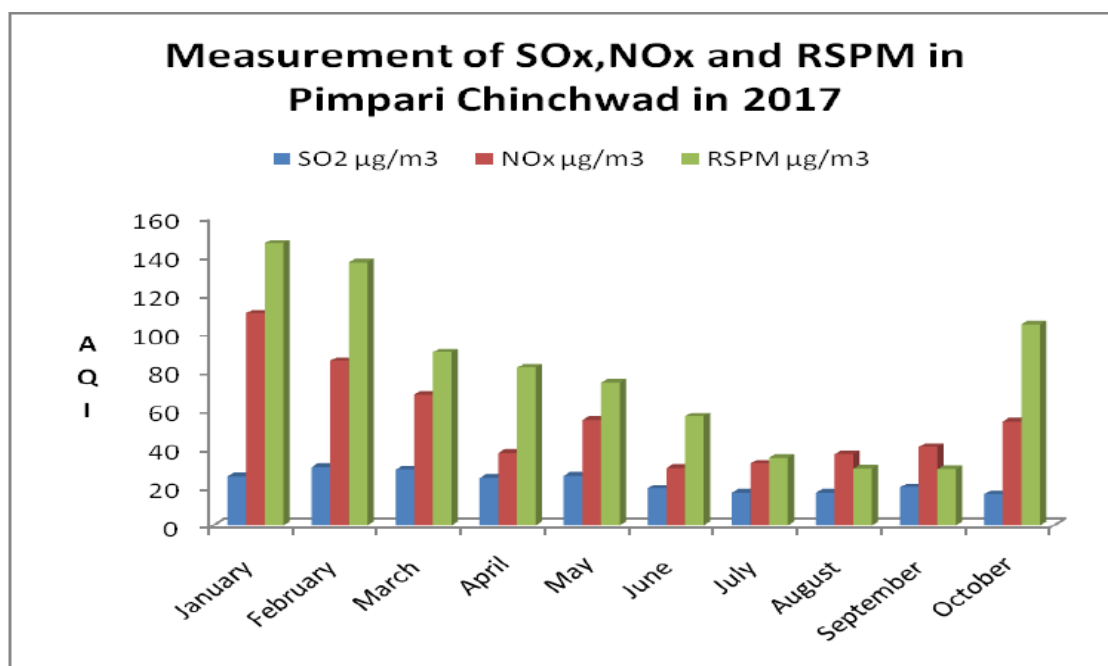
Fig. : Average Pollutionin Pimpari Chinchwad in 2017.

DISCUSSION

The air quality in the PCMC area is mainly declining due to vehicular pollution and industrial pollution.

Vehicular Pollution
According to the Pune RTO's statistics, till February end, the vehicle population of Pune including Pimpri Chinchwad was 34,90,569. Out of the total vehicle population, Pune city has a total of 24,53,717 vehicles while Pimpri Chinchwad has a population of 10,36,852 vehicles.

Out of the total 24,53,717 vehicles in Pune, nearly 75% of them that is 18,40,834 are two-wheelers. Apart from two-wheelers, the city has 4,30,440 four-wheelers which includes 3,74,445 small cars. In the year 2003, Pune had a total of 10,57,379 vehicles registered with Pune RTO.

The twin city Pimpri Chinchwad is also facing a problem of poor public transport system and it has a total of 10,36,852 vehicles of which 7,72,655 are two-wheelers and 1,55,943 are four-wheelers. [6]

Due to the development of the urban areas as centres of trade, commerce and industries, the growth in vehicular traffic has been alarming in Pune city and Pimpri- Chinchwad. Air pollution from vehicles is of serious concern. The health effects caused by vehicular pollution in the cities are indicated through increasing incidences of cough, headache, nausea, irritation of eyes, and various bronchial and respiratory diseases besides visibility problems.

The main pollutants emitted from automobiles are hydrocarbons, lead/ benzene, carbon monoxide, sulphur dioxide, oxides of nitrogen and suspended particulate matter. Though the number of vehicles is one of the major factors on which vehicular pollution depends, there are other reasons for the increased vehicular pollution in urban areas. These include: fuel quality, vehicle maintains, traffic congestion, poor road conditions and old automotive technologies.

Industrial Pollution.
Pimpri- Chinchwad area has a cluster of small scale industries in Bhosari MIDC. These companies are mainly related to automobile sector and are main cause of cause air pollution. The brick kilns and stone crushing units are also significantly declining the air quality in the city. These companies should adopt the rules and regulations of Maharashtra Pollution control Board.

In order to tackle the issue, it wants to take a multi-pronged approach, including awareness on air pollution , tree plantation drives, increase CNG buses, ban on vehicles aged over 15 years and to promote use of public transport.

Nearly 70% of air pollution is caused by the increasing number of vehicles. For this, PCMC wants to reduce the number of private vehicles and improve public transport. The Pune Mahanagar Parivahan Mahamandal Ltd will add 200 mini buses, 800 BRT buses, 50 Tejaswini buses (for women), and 550 buses by private contractors to its fleet. The civic body has proposed a clean development mechanism to reduce air pollution.

CONCLUSION

The main source of pollution in Pimpari Chinchwad area is vehicular emission. The air quality is fast deteriorating because of bad public transport. The only solution to the issue is that we need more efficient public transport, so that it discourages citizens to use private vehicles. Increasing the green cover in the city will not reduce particle pollution. We have to reduce vehicular emission, which is the biggest source of pollution in the cities. The industries have been reducing pollution because it is related to their profit margin. The more energy efficient they are all the more they will save. It is mainly the sheer number of vehicles on the city roads that is adding to the pollution. Maharashtra Pollution Control Board has to take strict actions against the companies who are polluting the atmosphere of city. Metro rail is a good option but it may take at least 20 years before we can actually use it. The PMPML has to be improved and side-by-side there has to be constraints over private vehicles.

Conflicts of interest: The authors stated that no conflicts of interest.

REFERENCES

1. https://www.numbeo.com/pollution/rankings_current.jsp
2. http://www.who.int/gho/phe/outdoor_air_pollution/burden/en
3. http://www.who.int/airpollution/ambient/health-impacts/en
4. "History And culture" PCMC Web Portal Retrieved 5 January 2017.
5. Central Pollution Control Board. Guidelines for Ambient Air Quality Monitoring. National Ambient Air Quality Monitoring Series; CPCB, Ministry of Environment and Forest, Government of India: Delhi, India, 2003.
6. http://www.dnaindia.com/pune/report-soon-city-vehicle-population-to-cross-35-l-mark-1816794

Estimation of Pazufloxacin Mesylate in bulk and formulation by UV-Spectrophotometric area under Curve Method

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ABSTRACT

The current work is carried out to for estimation of Pazufloxacin mesylate in bulk and pharmaceutical dosage form by utilizing area under curve (AUC) method. For this purpose the wavelength range 229-260nm was selected. Distilled water was used as a solvent throughout the work. Linearity was observed in concentration range 2-12µg/ml (r^2 =0.999) for the method. Recovery studies for area under curve were found to be 99.32%. The method developed was validated for linearity, precision, accuracy, LOD, and LOQ as per ICH guidelines.[1] The present method was found to be simple, linear, precise, accurate and sensitive which can be used for routine quality control analysis for spectrophotometric estimation of Pazufloxacin mesylate in bulk and dosage form.

Keywords: Pazufloxacin mesylate, Area under curve (AUC), and ICH guidelines.

INTRODUCTION

Pazufloxacin mesylate is a newer broad-spectrum fluoroquinolone antibiotic. Chemically is (3R)-10-(1-aminocyclopropyl)-9-fluoro-2,3-dihydro-3-methyl-7-oxo7H-pyrido[1,2,3-de]1,4-benzoxazine-6-carboxylic acid methanesulfonate (Fig. 1) [2]. The potent broad-spectrum activity of the molecule is due to the presence of the aminoacyl group at C10, imparting against gram negative and gram positive bacteria including a variety of resistant strains and anaerobic bacteria.

Mechanism of action shown by Pazufloxacin mesylate is multimodal and it inhibits both DNA gyrase and topoisomerase IV enzyme, leading to increase its antibacterial spectrum via DNA gyrase-dependent process such as polymerization of DNA, (ATP-dependent) DNA supercoiling and chromosome fragmentation. It has also shown DNA antagonistic action. The multimodal mechanism is linked to the low potential for the development of resistance in Pazufloxacin.[3] Many analytical methods have been overviewed in the survey. In the present work, the development and validation of Pazufloxacin mesylate using area under curve (AUC) method has been estimated for pure drug and marketed formulation.
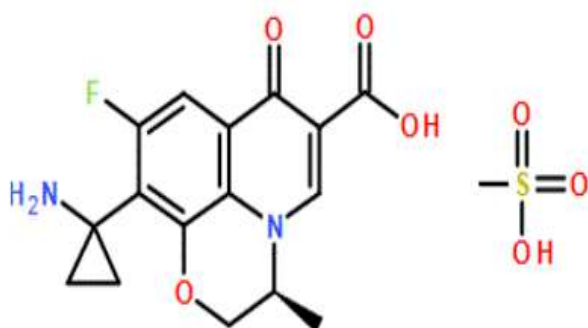

Fig 1. Chemical structure of Pazufloxacin mesylate

METHODOLOGY

1 Chemicals:

Pazufloxacin mesylate was supplied by Macleod's Pharmaceuticals, Sarigam, Gujarat. Pazumac infusion (500mg) was procured from local pharmacy. Methanol (S.D. Fine Chemicals, Mumbai, India) was used. All chemicals and reagents were of analytical reagent (AR) grade.

2 Instrumentation:

A Shimadzu (Kyoto, Japan) model UV-1800 double beam UV-Visible spectrophotometer attached with computer operated software UV probe 2.33 with spectral width of 2 nm, wavelength accuracy of 0.5 nm and pair of 1 cm matched quartz cells was used to measure absorbance of the resulting solutions. Analytical balance and Mettler Toledo (Model JL1503-C).

3 UV-Spectroscopy Methods

A) Area under curve method:

The AUC (area under curve) method is applicable where there is no sharp peak or when broad spectra are obtained. It involves the calculation of integrated value of absorbance with respect to the wavelength between the two selected wavelengths λ1 and λ2. Area calculation processing item calculates the area bound by the curve and the horizontal axis. The horizontal axis is selected by entering the wavelength range over which area has to be calculated. This wavelength range is selected on the basis of repeated observation so as to get the linearity between area under curve and concentration. The above-mentioned spectrums were used to calculate AUC. Thus, the calibration curve can be constructed by plotting concentration versus AUC.[5,6]

4 Experimental Work

a) To check the solubility of Pazufloxacin mesylate:

10 mg of Pazufloxacin mesylate was weighed and solubility of this sample was checked in double distilled water, methanol, ethanol, 1N NaOH, 0.1N HCl.

b) To identify the λmax of Pazufloxacin mesylate:

10 mg of the pure drug was accurately weighed and dissolved small portion of methanol and volume was made up to 10ml using distilled water to give a standard stock solution of 1000μg/ml. Further 2.5ml of 1000ppm solution was withdrawn and was diluted to 25 ml of volumetric flask and 100ppm solution is prepared. Suitable dilutions were made with distilled water to get standard solutions of concentration: 2, 4, 6, 8, 10, 12μg/ml.

C) Sample preparation for analysis of infusion formulation

Sample solution was prepared from infusion PAZUMAC (500 mg/100 ml). A sample volume equivalent to 10 mg was pipetted out from the infusion container and was placed in 10ml volumetric flask; volume was made up with solvent to get the concentration of 1000μg/ml. From this, 2.5 ml of aliquot transferred in 25 ml of volumetric flask containing diluent to form 100μg/ml of Pazufloxacin mesylate stock solution and further dilution of 2, 4, 6, 8, 10, 12ppm and scanned in the range of 200-400nm against water as blank at 249nm and then drug

content of solution was calculated by using standard calibration curve.

5 Analytical Method Development and Validation:
The developed method was validated as per ICH guidelines.

1. Linearity: The linearity of an analytical procedure is the interval between the upper and lower concentration of an analyte in the sample.[1]For which it has been demonstrated that the analytical procedure is of linearity, accuracy and precision.

Standard solution of pazufloxacin mesylate (2, 4, 6, 8, 10 and 12μg/ml) was pipette out in to a separated series of 10ml volumetric flask. The volume was adjusted to the mark with distilled water and mixed well. The absorbance maxima and area under curve for the solutions was measured at 249nm and range of 229-260nm for two methods respectively against distilled water as blank.

2. Precision: The precision of analytical procedure expresses closeness of agreement (degree of scattering) between a series of measurements obtained from multiple sampling of the same homogeneous sample under prescribed conditions. It may be considered at three levels: repeatability, intermediate precision, and reproducibility. It is expressed as standard deviation or coefficient of variation.

The repeatability studies were performed by analysis of same solution (2-12μg/ml) on the same day. Intermediate precision of the method was checked by repeating studies on the two different days. The %RSD of both determinations was calculated.

3. Accuracy: It is closeness of the result obtained to the true value. It is often expressed as percentage recovery by analyzing known added amounts of analyte. Also, it can be determined by applying the procedure to quantitatively prepared samples.

4. Sensitivity: The sensitivity of the method was determined in terms of limit of detection (LOD) and limit of quantitation (LOQ).

The LOD and LOQ were calculated by using formula,

LOD= 3.3×σ/S and **LOQ= 10×σ/ S.**
Where, σ is the standard deviation of regression line and S is the slope of line.

RESULTS AND DISCUSSION

1) Linearity & Range:

A] Calibration curve for pure drug:

Absorbance maxima method:
Under the experimental conditions described, the graph obtained for the absorbance maxima for pure drug showed linear relationship (Fig.1).Regression analysis was made for the slope, intercept and correlation-coefficient values. The regression equations of calibration curve were $Y = 0.071x+0.170 (r2 = 0.998)$ at 249.20nmfor absorption maxima the range was found to be 2-12μg/ml for the UV spectrometry.

Table 1: Calibration curve of Pazufloxacin mesylate

CONC.	ABS
2	0.325
4	0.452
6	0.589
8	0.729
10	0.901
12	1.027
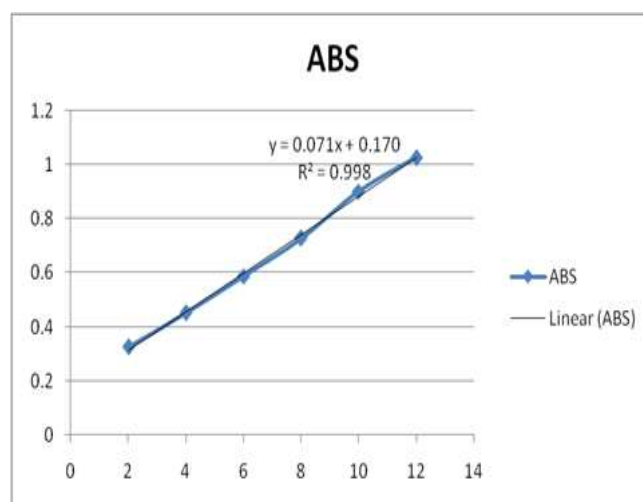
Fig 2: Calibration of pazufloxacin mesylate (Pure Drug)
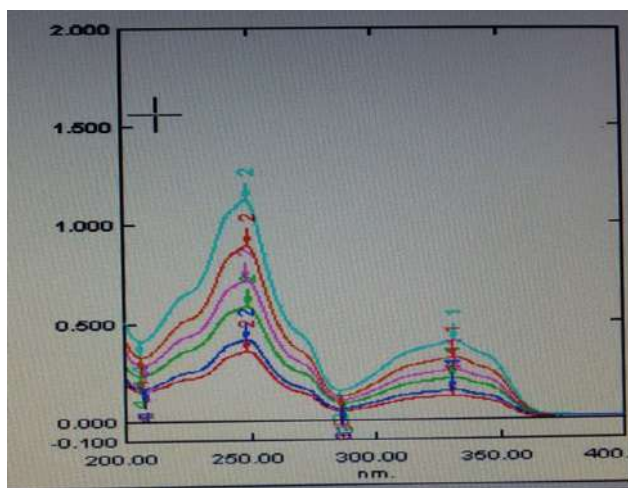
Fig.3: Overlay of spectra of Pazufloxacin Mesylate

Table.2: Calibration curve of Pazumac

Conc.	ABS.
2	0.308
4	0.469
6	0.615
8	0.748
10	0.895
12	1.064
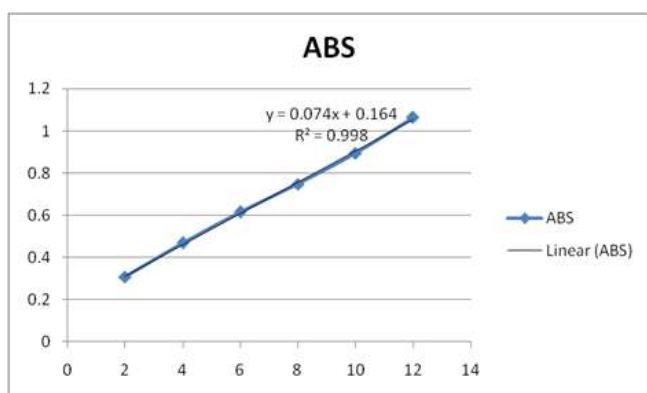
Fig 4: Calibration of formulation of pazufloxacin mesylate (Pazumac)

b) Area under curve method:

Under the experimental conditions described, the graph obtained for area under curve spectra showed linear relationship (Fig.4& Fig.5). Regression analysis was made for the slope, intercept and correlation coefficient values. The equation is y = 0.565x+1.652 (r2=0.999) at 229.20-260.20 nm for area under curve spectrophotometry. The range was found to be 2-12μg/ml for area under curve spectrophotometric analysis.

Table 3: Calibration curve of pazufloxacin mesylate by AUC

Conc.	AUC
2	2.715
4	3.985
6	5.057
8	6.224
10	7.227
12	8.452
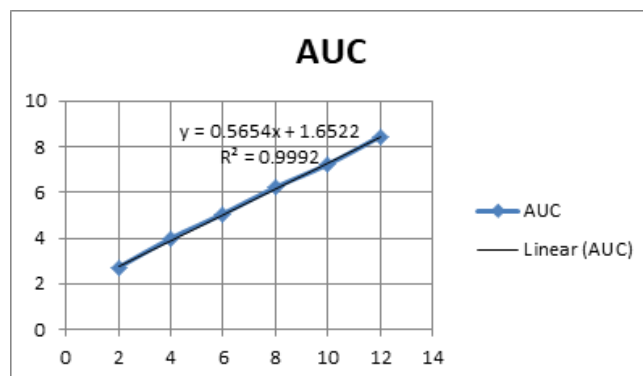
Fig 5: Calibration curve by AUC for pure drug Pazufloxacin mesylate
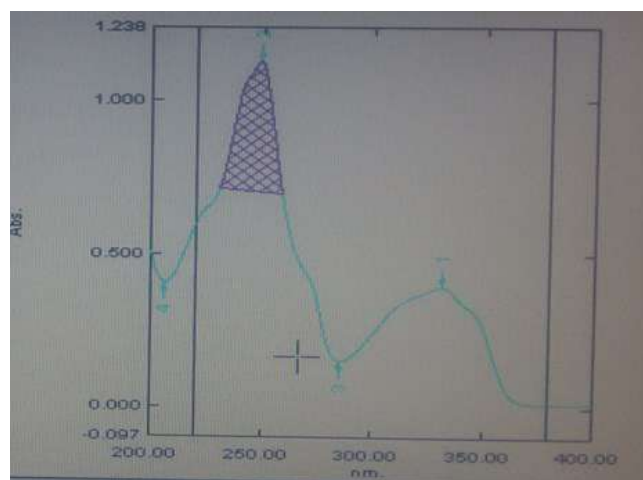
Fig 6: .Area between 229-260nm selected for pazufloxacin mesylate (10μg/ml)

Table 4: Calibration curve by AUC for pazufloxacin mesylate formulation

CONC.	AUC
2	1.363
4	2.824
6	4.239
8	5.678
10	7.264
12	8.671
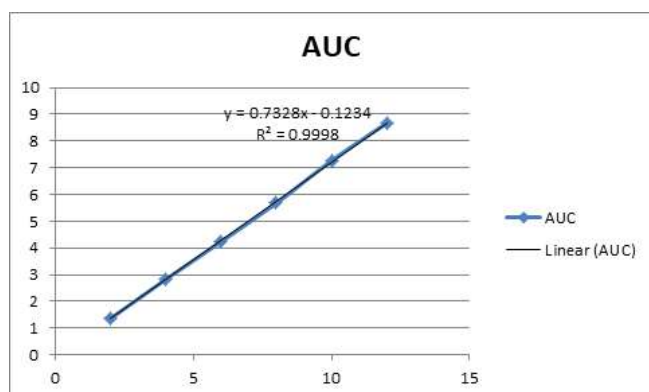
Fig.7. Calibration curve by AUC for pazufloxacin mesylate formulation
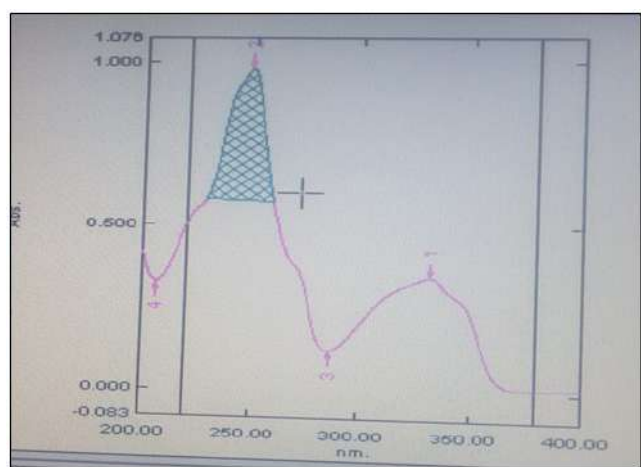
Fig 8: Area between 229.20-260nm selected for Pazufloxacin mesylate infusion formulation (10µg/ml)

Table 4: Regression Analysis Data for Pazufloxacin mesylate by the AUC method

Parameter	AUC
Wavelength range (nm)	229-260
Concentration range (µg/ml)	2-12
Slope(m)	0.565
Intercept(c)	1.652
Correlation coefficient(r2)	0.999

Precision:

Precision of the method was verified by repeatability and intermediate precision studies. Repeatability studies were performed by analyses solution (2-12µg/ml) on the same day. The %RSD of six determinations was calculated. Intermediate precision of the method was checked by repeating studies on

two different days. The %RSD of the determinations was calculated.

Table no.5.Result of interday and intraday precision

Parameters	±S.D.*	RSD	%RSD*
Interday	0.0585	0.009	0.975
Intraday	0.0656	0.010	1.00

*n=6

**Table 4: Data of Recovery studies**

Level of mean recovery (%)	% mean recovery	SD*	%RSD
80%	101.12%	0.0721	1.20
100%	99.32%	0.0595	0.991
120%	100.23%	0.0651	1.08

Table 5: Assay Results for estimation of Pazufloxacin in Pharmaceutical Formulation

Parameter	Label claimed	Amount found	%Label clamed
AUC	10µg/ml	9.93	99.32

Sensitivity:

The limit of detection (LOD) and limit of quantitation (LOQ) were calculated by using the equations LOD= $3.3 \times \sigma/S$ and LOQ= $10 \times \sigma/S$, where σ is the standard deviation of the intercept, S is the slope. The LOD and LOQ were found to be 0.0093 and 0.0283 respectively for the area under curve method.

Recovery studies:

To stock solution in 3 different volumetric flask, aliquots of 8ml, 10ml and 12ml of the standard stock solution were added, volume was made upto 10ml with water to give concentration of 8µg/ml (80%), 10(100%) and 12(120%). Absorbance was determined at 249nm. Procedure was repeated 3 times for 80%, 100% and 120% for recovery studies

Table no.6 Summary Data of Validation Parameters

Sr.no	Parameter	AUC Method
1	Linearity	2-12
2	Regression equation	y =0.565x+1.652
3	Correlation Co-efficient	r2=0.999
4	LOD(µg/ml)	0.0093
5	LOQ(µg/ml)	0.0283

6	Precision	
6.1	Interday	0.975%RSD
6.2	Intraday	1.00%RSD

CONCLUSION

Simple UV spectrophotometric methods have been developed and validated for the determination of Pazufloxacin mesylate and infusion. The results of the validation parameters show that the UV spectrophotometric methods were found to be accurate, precise and sensitive. Because of cost-effective and minimal maintenance, the present UV spectrophotometric methods can be preferred at small scale industries and successfully applied and suggested for the quantitative analysis of pazufloxacin mesylate in pharmaceutical formulations for QC, where economy and time are essential and to assure therapeutic efficacy.

Conflicts of interest: The authors stated that no conflicts of interest.

REFERENCES

1. ICH Harmonised Tripartite Guideline: Validation of analytical procedures: text and methodology Q2 (R1).
2. Merck index, Maryadele J.O. Neil Edu. In: 13 Ed, Merck Research Lab NJ, USA. 2001; 868.
3. Chemical structure available online at URL - http://www.chemicalbook.com/ChemicalProductProperty_USCB3188646.aspx; (Accessed January 2015).
4. http://www.aksci.com/item_detail.php?cat=M798; (Accessed January 2015).
5. Michael E. Swartz, Ira S. Krull, Analytical method development and validation, Marcel Dekker, Inc., 1997; 17: 25-2.
6. Christian G.D., Analytical chemistry, sixth edition, John Wiley and Sons, 2003; 1-2,604- 620.
7. www.ukessays.com/essays/biology/estimation-of-pazufloxacin-mesylate-from-pharmaceutical-dosage-form-biology-essay.php; (Accessed October 2014).
8. Sudesh D. Shambharkar , P. D. Hamrapurkar, AbhijeetParate, et.al. Optimization and Validation of RP-HPLC Stability Indicating Method for Determination of Pazufloxacin Mesylate and Its Degraded Product.International Journal of Applied Science and Engineering 2013; 11, 4: 423-432; (Accessed November 2014). http://www.cyut.edu.tw/~ijase/2013/11%284%29/6_024016.pdf(Accessed November 2014).
9. www.researchgate.net/publication/260419864_HPLC_and_Densitometric_TLC_Methods_for_Simultaneous_Determination_of_Pazufloxacin_with_Some_Coadministered_Drugs_in_Human_Plasma;(Accessed November 2014).
10. Nehad A. Abdallah; Validated Stability-indicating HPLC and Thin Layer Densitometric Methods for the Determination of Pazufloxacin; J Chromat Separation Techniq ISSN: 2157-7064 JCGST; Volume5 Issue2 1000218.
11. http://omicsonline.org/open-access/validated-stabilityindicating-hplc-and-thin-layer-densitometric-methods-for-the-determination-of-pazufloxacin-application-to-pharmaceutical-formulation-2157-7064.1000218.pdf; (November 2014). www.wjpr
12. Skoog, Holler, Nieman, Principles of Instrumental Analysis, fifth edition, Thomson Asia Pvt. Ltd., Singapore, 2004; 300-325.
13. Beckett, A. H., Stenlake, J. B., Practical Pharmaceutical Chemistry, 4th edition, CBS Publishers and Distributors, New Delhi, 2002; 2: 275-295.
14. Quin Li, Rui wang, fei pei, Determination of the Pazufloxacin mesilate in human plasma and urine by HPLC, Asian Journel of Drug Metabolism and Pharmaceuticals, 289.
15. Zhefeng Zhang, Gengling Yang, Dexian Wang, Guijian lian, YI chen, Determination of pazufloxacin mesilate by high performance liquid chromatography, Micro chimica acta, 159, 299-301.
16. Chunyan Yang, Zhujun Zhang, Suming Chen, Feng yang, Development of moleculaely imprinted on-solid extraction combined with chemiluminesence for the determination of pazufloxacin mesilate. Spinger, 2007, 159, 299-301.

Assessment of groundwater pollution in the vicinity of Kurkumbh MIDC Kurkumbh, tal -Daund, Pune-district, Maharashtra, India

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ABSTRACT

The paper aim at determining the suitability of water near, Kurkumbh MIDC. The present work deals with assessment of variation of seasonal water Quality Index of selected location of village near Kurkumbh MIDC. In the present study, Ground water sample of in selected location of village were taken for investigation and analyzed for various parameters with regard to drinking water standards and assessed for their suitability for drinking water .After analysis it has been observed that ground water quality of location are not suitable for drinking propose .We can highlighting the major issues of drinking water availability and measure to be adopted due to growing industrialization and unhealthy human activity. Water samples were collected from twelve different sites covering borderline area of MIDC and neighboring village downstream to it. These samples were analyzed for various parameters such as pH, EC, TDS, total hardness, Ca/Mg hardness, total alkalinity, fluoride, nitrate, sulphate, D.O., BOD, COD using standard method. It was found that values of TDS, total hardness, total alkalinity and sulphate of the samples are out of the highest desirable limit or exceeded the permissible limit. Some samples are extremely hard and highly saline and absolutely unfit for consumption, domestic use and irrigation. Half of these twelve sites are moderately hard and saline for the study reveals no major changes in water quality during study period i.e. pre and post monsoon. This is because of industrial effluent, which is percolated through ground as well as mixed with water stream causing deterioration of water resources.

Keywords: Ground water, quality, Physico-chemical parameters, MIDC, irrigation, effluent.

INTRODUCTION

The water is precious gift of nature to human being is going to be polluted day by day with increasing urbanization, although three fourth part of earth is being surrounded by water but a little portion of it can be used for drinking purposes. Study physico-chemical parameters of drinking water near Kurkumbh MIDC, Daund- taluka. Today human activities are constantly adding industrial, domestic and agricultural waste to ground water. Ground water contamination is reversible i.e. once it is contaminated. It is difficult to restore the original water quality. The problem has been further aggravated by the rapid increases in population there by increasing the demand of water supply for irrigation, human and industrial consumption. Surface water and ground water are the major source of drinking water in India. Water is prime natural resource and physiological necessity to mankind. Therefore drinking water must not carry harmful chemicals as well as biological contaminants for wellbeing some of chemicals like fluoride is arsenic calcium, lead, chromium and nitrate in drinking water over definite limit cause serious health problem.

Kurkumbh MIDC is Chemical Industrial Zone. There are no of pharmaceutical chemical, dye and oil industry. Waste water generated foam various industries is discharged directly on waste land; Nalas, Canals Roti ghat etc. Therefore surface water and ground water is contaminated by industrial and domestically nest. The ground water near Kurkumbh MIDC contaminated with heavy metals, persistent organic pollutants and nutrients. The chemicals like fluoride, arsenic, lead, petrochemicals, other heavy metal cause serious health effect. The impact of heavy metal in drinking water affects the public health to a great extent. They cause biochemical effect such as inhibition of enzymes, genetic damage, and hypertension. Proper management of water resources has become the need of the hour as this would ultimately lead to a cleaner and healthier environment.

METHODOLOGY

Sample Collection:
Water samples were collected in pre-cleaned sterilized polypropylene bottles with necessary precaution from different sites. Samples were collected in Pre and Post monsoon. Various physico-chemical parameters are analyzed as given in standard manual of water and waste water analysis. The main aim of the study is to investigate the physico-chemical characteristics of water samples near Kurkumbh MIDC, because most of these samples are located in the vicinity of the MIDC.

Laboratory analysis:
The samples collected were characterized by different parameters such as P^H, conductivity, TDS, total alkalinity, total hardness, Calcium, Magnesium, fluoride, Nitrate, Sulphate, DO, BOD,COD etc. The temperature, P^H is recorded by P^H meter(Model No. EQ-610 Equiptronics), conductance is measured by conductivity bridge (Model No. EQ-660, Equiptronics). The other parameters are determined by using standard method available in literature [5].

RESULTS AND DISCUSSION

The results of analysis were reported in table. The P^H value of the samples in the study area varied 7.34 – 8.30 during Study area. The observed variation may be due to leaching of effluent and excessive use of fertilizers in local agricultural operation. The electrical conductivity of ground water from study area was ranging in between 850 to1960 micromohs/cm. Whereas permissible limit ranging between 250 to 750 micromohs/cm for domestic use. The total dissolved solids (TDS) ranges from 544 – 10228 mg/l. Dissolved inorganic salts, small amounts of organic matter and gases contribute to TDS. The total hardness varies from 212 to 668 mg/l at study area. This may be due to the presence of calcium and magnesium. The calcium and magnesium was found under permissible limit the high value of magnesium is at sites 10 and 11 indicates that the ground water was polluted because of industrial effluent.

Table 1: Assessment of Ground Water June 2017

parameters	WS 1	WS 2	WS3	WS4	WS5	WS6	WS7	WS8	WS9	WS10	WS11	WS12
PH	7.70	7.60	7.85	8.30	7.90	7.85	7.87	7.90	7.27	7.34	8.22	7.50
Conductance	1030	1214	1014	1790	1380	1200	850	1025	1400	1960	1920	1850
TDS	1659	1777	1649	1145	1878	1768	544	1656	896	1254	10228	1184
Total Hardness	380	324	274	454	288	236	212	278	384	668	712	408
Calcium	88.9	84.36	64.13	120.2	66.5	65.7	58.1	63.3	100.2	135.4	157.9	118.6
Magnesium	38.9	28.26	27.77	37.5	29.7	17.5	16.0	29.2	32.6	80.4	77.4	27.2
Total alkalinity	200	296	256	364	240	264	344	316	224	340	352	288
Chloride	402.2	245.8	344	456.2	234	307.8	425	362	576	298	502.1	374.4
D.O.	2.82	2.54	4.20	2.54	3.25	2.90	5.30	4.85	3.50	3.98	2.95	2.58
BOD	5.75	6.70	8.95	5.50	6.50	6.23	9.55	7.84	7.35	10.45	13.45	12.55
COD	28.50	24.00	28.00	46.00	22.00	15.20	30.00	42.00	56.00	54.23	34.80	24.00
Sulphate	169.4	173.9	248	100	46	290	82	102	86	423	530	319
Fluoride	0.12	0.40	0.20	0.60	0.50	0.20	0.70	1.00	1.30	0.80	1.57	0.90
Nitrate	24.0	22.0	2.0	98.0	36.0	53.0	28.0	16.0	114.0	142.0	175.0	103.0

Excess alkalinity gives bitter taste to water and reacts with cations to forming precipitates, which can damage the pipes, valves, etc. Total alkalinity ranges from 200 mg/l to 364 mg/l during study period the maximum value (364 mg/l) was recorded in study area. The chloride content in study area has shown variation from 234 to 576. The higher value of chloride suggests leaching of effluent from industrial effluent into the ground water [14]. The site numbered 2,3,4,5,6,8,9 & 11 are shows higher value of chloride which are higher than permissible limit of 200 mg/l according to WHO. The extremely high concentration of chlorides as these sites are directly affected due to industrial discharge .Dissolved oxygen ranged from 2.54 to 5.50 mg/l. BOD and COD value indicated present organic matter due to industrial pollution. The sulphate varies ranges from 46 to 530 mg/l. The fluoride value varies from 0.12 to 1.57 mg/l. WHO standard suggested limit for fluoride as 1mg/l. But Ws 9 & Ws 11 show amount of fluoride crossing prescribed limit. The nitrate concentration in groundwater collected from the study area ranged between 2.0 to 175 mg/l .The concentration of nitrate above 45 mg/l in drinking water may cause child disease named as methamoglobinemia.

CONCLUSION

On the basis of above discussion it may be conclude that Ground water is hard and highly contaminated with reference to all physico-chemical parameters studies almost all site near kurkumbh MIDC at daund Taluka. Analysis of sample it is observed that WS 10 and WS 11 are highly polluted. Other sites WS 1, WS 3 and WS 7 are can be used for domestic and irrigation purpose after proper treatments. Remaining are moderately polluted. All the parameters from study area show slight variations, no major changes are observed in water quality during study period. This is because of industrial effluents.

Some of parameters tested under investigation are out of limit according to WHO. Sampling stations show pollution of ground water and not suitable for irrigation as well as domestic use. It is recommended that effluents from industries will be properly treated before discharge.

Conflicts of interest: The authors stated that no conflicts of interest.

REFERENCES

1. Wagh CV, Kokate SJ, Aher HR and Kuchekar SR. *RASAYAN J. Chem*, **2,** 234 (2009).
2. Kamble PN, Kokate SJ, Aher HR and Kuchekar SR. *RASAYAN J. Chemistry.,* **1**, 63 (2008).
3. Karle KK, Bhusal SS, Gunjal PS and Kuchekar SR. *Poll. Res.*, **11**, 65 (1992).
4. Gunjal PS, Zinjad DG, Kuchekar SR and Aher HR. *Poll. Res.*, **12**, 219 (1993).
5. Gunjal PS, Kuchekar SR, Thorat DG and Aher HR. *J. Aqua. Biol.*, **10**, 26 (1995).
6. Trivedy RK and Goel PK. Chemical and biological methods for water pollution studies, 1986.
7. APHA, Standard Methods for Examination of Water and Waste Water.21st ed.American Public Health Association, Washington, DC (2005).
8. Trivedi RK and Goel PK. Chemical and Biological methods for Water Pollution Studies.

Assessment of water quality by physico-chemical parameters

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ABSTRACT

An effective water purification method for the community is the key to human survival and development, as water management is a current global concern. Water is the basic resource necessary for sustaining all human activities, so its provision in desired quantity and quality is of utmost important. Contaminated water is the main source of infectious disease viz. Amoebiasis and Malaria, Cholera, Dysentery, Paratyphoid, Fever, Typhoid, Jaundice. The WHO (World health organization) report that one sixth of the world's population (1.1billion people) does not have access to safe water. Due to use of contaminated water, human population suffers from water borne diseases. Therefore it is necessary to check the water quality at regular interval of time. An assessment on the basis of temperature, pH, Turbidity can also provide an indication about water quality. The present cross-sectional study is focused on measuring the quality of drinking water, soil water, and laundry wastes water in rural areas of Pune district of Maharashtra state and its effect on human health in these areas. Various analyses including physical and chemical assessment were carried out on the water sample collected from the village. The different samples were showing variations in physicochemical properties. At that time water quality was also questionable in most of the cases as majority of people living in these areas. The people were not safe from various water borne diseases.

The principal sources of water are lakes, river and relatively shallow ground water of dam and the study argues about the need and importance of water purification and water management systems in current times. In this review, we are assessing the water pollution to sustainable use of water and try to ensure the highest protection of water from all pollutants.

Keywords: Water Pollutant, Water quality, Infectious diseases, Water treatment, Filtration, Activated charcoal.

INTRODUCTION

Water is sustenance of the life cycle. It must be protected and preserved from all type of pollutant. It is the fundamental right of every individual to get pollution free water, but man disturbing water bodies viz. rivers, wells, stream, and seas. Water pollution occurs when undesirable effluents disperse in water system it results into change in water quality. The natural water system is being polluted by addition of industrial wastes, urban wastes, pesticides related pollutant as well as domestic sources that are primarily sewage, laundry wastes generated in house, apartments, and other dwellings. Pervious as well as impervious surfaces collect hundreds of pollutants such as animal waste, bacteria, oil, grease, sediment, pesticides and deposits from airborne pollutants. These hazardous materials can easily enter in our commercial water sources making water unsafe for human use. Urban sewage is handled by established government agencies as they can usually be effectively controlled [1].

Most of people do not seem to realize how important water is to our progress. Everyone in country use it for their benefits that affect all aspects of water leading to decrease in water quality. There is no other source to target but water to tackle this issue. This cannot be possible until local, state, and national storm water regulations are allow to altered changes in our lifestyle [2].

Water pollution can be prevented by stopping pollutants from contaminating nearby water sources. There are a number of water treatments to prevent pollution and water quality such as biological filters, chemical additives and sand filters. These simple technique cost money to maintain, but prevention is much cheaper than cleaning up water pollution that has already occurred [3].

Keeping the above fact in mind, this study was carried out to assess the physical and chemical parameters of water.

Objectives
The study covers the following objectives for research work
1. To check physicochemical characteristics of four different type of water.
2. Assessment of physicochemical characteristics after filtration (with the help of activated charcoal).
3. To evaluate and forecast the water quality after water treatment.

METHODOLOGY

The different water samples for the study have been collected from nearby villages of Hon. B.J. College Ale village of Junnar Tahsil. (Pune, Maharashtra).

Table 1: Main Source of Drinking Water in Junnar Tahsil of Pune.

Main Source of drinking water	In Numbers			In Percentage		
	Total	Rural	Urban	Total	Rural	Urban
Tap water from treated source	31,745	26299	5446	38.4	34.2	97.3
Tap water from un-treated source	7244	7165	79	8.8	9.3	1.4
Covered well	2282	2274	8	2.8	3.0	0.1
Un-covered well	24372	24338	34	29.5	31.6	0.6
Hand pump	6814	6813	1	8.2	8.8	0.0
Tube well/Borehole	8343	8326	17	10.1	10.8	0.3
Spring	398	394	4	0.5	0.5	0.1
River/Canal	485	482	3	0.6	0.6	0.1
Tank/Pond/Lake	351	350	1	0.4	0.5	0.0
Other sources	572	566	6	0.7	0.7	0.1

Source: Junnar Collector Office Pune 2011.

Water Quality and Pollution

http://www.irjse.in

Water covers over 71% of the earth's surface and is a very important natural resource for people [4]. But only 2 to 5 % of the earth's water is fresh. India has 16 % of the world's population, has only 2.5 % of the worlds land area and 4 % of the world's water resources at its disposal. Precipitation in the form of rain and snowfall provide over 4,000 trillion liters of fresh water to India. It is prove that 80% of India's surface water is polluted. It is fundamental right of every individual to get pollution free water. Water pollution come from industry, agriculture or household, chemical wastes in the water have negative effect on living organism in water and subsequently on our health. Water pollution effects drinking water, rivers, lakes and oceans all over the world which consequently harms human health and natural environment.

Water quality parameters
PH:
PH is the measure of acidity of a solution of water. The pH scale commonly ranges from 0 to 14. The scale is not linear but rather it is logarithmic form e.g. a solution with a pH of 6 is ten times more acidic than a pure solution with a pH of 7. Pure water is said to be neutral with a pH of 7. Water with pH bellow 7 is considered to be acidic while water with pH greater

than 7.0 is considered basic or alkaline since the pH of pure water is 7.
Conductivity:
Conductivity is a numerical expression of an aqueous solution's indicating a capacity to carry an electric current. This ability depends on the presence of ions, their total concentrations, mobility, valence and relative concentrations, and on the temperature of the liquid. Solutions of most inorganic acids, bases, and salts are relatively good conductors. In contrast, the conductivity of distilled water is less than 1 μmhos/cm because conductivity is the inverse of resistance, the unit of conductance.

Alkalinity:
Alkalinity is the sum total of components in the water that tend to elevate the pH to alkaline side of neutrality. It is measured by titration with standardized acid to a pH value of 4.5 and is expressed commonly as milligrams per liter as calcium carbonate (mg/L as $CaCO_3$). Alkalinity is a measure of the buffering capacity (ability to resist changes in pH) of the water, and since pH has a direct effect on organism as well as indirect effect on the toxicity of certain other pollutant in the water, the buffering capacity is important to water quality. Commonly occurring material in water that increase alkalinity are carbonates, bicarbonates, phosphates

Table 2:

Compounds	General Formulae	Odor Produced
Ammonia	NH3	Ammonic, pungent
Amine	CH3NH2, (CH3)3N	Fishy
Chloro phenol	ClPhOH	Medicinal, phenolic
Chlorine	Cl2	Chlorine
Diamines	H2N(CH2)4NH2,H2N (CH2)5NH2,	Rotten fish
Organic	(CH3)3S, CH3SSCH3	Strong delayed cabbage
Skatole	C8H5NHCH3	Fecal, repulsive
Sulphur dioxide	SO2	Pungent, acidic

Table 3: Physical Parameter

Characteristic	Distilled water	Tap water	Soil water	Laundry waste water
Taste	Agreeable	Agreeable	Not Agreeable	Not Agreeable
Odour	Unobjectionable	Unobjectionable	Unobjectionable	Unobjectionable
Turbidity	No turbidity	No turbidity	Turbid	Turbid
Appearance	Clear	Clear	No Clear	No Clear
Colour	colorless	colorless	Brownish	Turbid White
Chemical assessment pH	6.70	7.84	8.15	9.85

Table 4 : Physical Parameter

Characteristic	Distilled water	Tap water	Soil water	Laundry waste water
Taste	Agreeable	Agreeable	Not Agreeable	Not Agreeable
Odour	Unobjectionable	Unobjectionable	Unobjectionable	Unobjectionable
Turbidity	No turbidity	No turbidity	No turbidity	No turbidity
Appearance	Clear	Clear	Clear	Clear
Colour	colorless	colorless	Yellow	colorless
Chemical assessment pH	7.10	7.80	7.60	7.66

and hydroxides. Limestone bedrock and thick deposits of glacial till are good sources of carbonate buffering. Lakes with in such areas are usually well-buffered.

Water quality characters including: Taste, Odour, Temperature, Turbidity, Appearance (clarity), and Colour.

Physical Properties

The vast majority of the large solids such as faces and paper have broken up into very small particles and made turbidity with visible particles of organic material. The water color becomes gray and change to yellow- brown, according to the time day.

Odour:

Wastewater (becomes anaerobic) has a musty smell that is not at all offensive. Microorganisms that produce such odour are Cyanobacteria, Oscillatoria, Moreover, certain industrial waste have distinctive odour that caused by gasses involved from decomposition of various fractions of the organic matter. The rotten eggs is the commonest odor that caused by hydrogen sulfide produced by anaerobic bacteria (reduction of sulfate to sulfide). On the other hand, volatile fatty acids odor produced during food processing treatment and storage (Table No. I) [5, 6]

WATER TREATMENT METHOD

We will present only a brief, general account of typical step.

Filtration

Filtration is any of various mechanical, physical or biological operations that separate solids from fluids by adding a medium through which only the fluid can

pass. The water pumped from the settling areas into tanks that equipped with sand filters. The water is cleared from the most remaining impurities, including numerous bacteria and other microorganisms. [7]

Sedimentation

Sedimentation is used as simple pretreatment of water before starting purification treatment such as filtration and disinfection method. It removes undesirable small particulates suspended matters (sand, silt and clay) and some biological contaminants from water under the influence of gravity. The longer the water is kept for sedimentation, more the suspended solids and pathogens will settle to bottom of the container. Special chemicals or some natural coagulants can accelerate sedimentation. Commonly used chemicals are aluminum sulphate, poly aluminum chloride (PAC or liquid alum) and ferric sulphate. [8]

Analysis

In analysis first the 7pH tablet is mix with water and prepared the water solution having pH-7 in beaker. pH meter is calibrated for pH-7 by using this solution i.e. pH of that water solution is exactly 7. Samples of four different waters are places as follows respectively in first beaker filled with distilled water , second one with tap water, third with soil water and in last beaker laundry waste water is present. The physicochemical principal and chemical assessment (pH) of all types of water is done Observations are recorded in Table No-II to find out which one is pure and impure water samples.

CONCLUSION

Activated charcoal (pure carbon) chemical name is mineral carbon. It is used for emergency toxic removal purpose. The activated charcoal powder mix with

polluted water, i.e. in third and forth water sample. After filtration chemical assessment (pH value) of this water shows that it results in appearance change. (Table No.III) It is good sign for the treatment of water where as 7in absence of filtration this water is not good for human life it is turbid and pH is also high. The filtration results in decrease of turbidity, conductance and pH. The water becomes clean and clear compared to earlier one. So, this filter water can be used for various purposes but not for drinking water purposes. But still after filtration there is no growth of bacteria or pollutant is developed.

Water pollution is a serious problem in India as almost 80 percent surface water is polluted. It is contaminated by biological, toxic, inorganic and organic pollutants. This water is considered unsafe for human consumption as well as various activities. Many of researchers have tried to find out economically feasible method to treat polluted water. These methods include chemical precipitation, electrolysis, coagulation, ultra filtration, adsorption, osmosis to treat waste water. The suggested measures improve the water quality by conducting total ban on the activities that causes pollution. Result of water quality assessment clearly showed that most of the water quality parameters show positive changes with filtration process. In future we need to develop these methods to improve water quality.

Conflicts of interest: The authors stated that no conflicts of interest.

REFERENCES

1. Boyd CE and Tucker CS. Water quality management", Springer science & Business media, 2012.

2. Nesaratnam ST. Water Pollution Control," John Wiley & Sons., 2014.

3. WHO (World Health Organization). water treatment. WHO Geneva. Retrieved, 2012.

4. National Environmental Research Council (NERC). The Oceans: Scientific certainties and uncertainties. Swindon, England, 2017, 172-185.

5. Dague RR. Fundamentals of odor control," *Journal Water Pollution Control Federation*, 1972, 583-594.

6. Loan, NT, Phuong NM and Anh NTN. The Role of Aquatic Plants And Microorganisms In Domestic Wastewater Treatment," *Environmental Engineering and Management Journal*, 2014, 13, 2031-2038.

7. Cui X and Choo KH. National organic matter removal and fouling control in low-pressure membrane filtration for water treatment, *Environmental Engineering Research*, 2014, 19.1-8.

8. García LSM, Martínez CAT & DÍAZ AE. Analisis of waste water treatment plant processes (WWTP) "sedimentation"," *Visión Electrónica: algo más queun estado sólido*, 2014, 8

9. Almeida M, Butler D and Friedler E. At-source domestic wastewater quality, *Urban water*, 1999, 1, 49-55.

10. Barker DJ and Stuckey DC.A review of soluble microbial products (SMP) in wastewater treatment systems, *Water Research*, 1999, 33, 3063-3082.

11. Huang, M.-H., Li, Y.-M. & Gu, G.-W. (2010)."Chemical composition of organic matters in domestic wastewater," Desalination, 262, 36-42.

12. Shon H, Vigneswaran S and Snyder S. Effluent organic matter (EfOM) in wastewater: constituents, effects, and treatment," *Critical Reviews in Environmental Science and Technology*, 2006, 36, 327-374.

13. Vikranthpridhvi Y and Musalaiah M.A review on water and sewage water treatment process, 2015.

14. White EB and Sharma MN. Methods for use in water purification particularly sewage treatments," Google Patents, 1978.

15. Williams R, Neal C, Jarvie H, Johnson A, Whitehead P, Bowes M and Jenkins A. Water Quality, 2015.

RESEARCH ARTICLE   **OPEN ACCESS**

Isolation and partial purification of lipase from *Erythrina indica*

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ABSTRACT

Lipases (triacylglycerol acyl hydrolase) are enzymes that catalyze the hydrolysis of triacylglycerol's at the oil-water interface to release glycerol and free fatty acids as well as their ability to catalyzevarious types ofsynthetic reactions in non-aqueous environment. These include acidolysis, alcoholysis, aminolysis, esterification, inter-esterification. The Lipase used in each application is selected based on its substrate specificity and stereo specificity as well as temperature and p^H stability. Lipasehas attracted more attention in recent years because of its diverse biotechnological applications. Lipase occurs widely in nature, but plant lipase is significant because of its substrate selectivity/specificity. Lipase is one of the most important enzymesused in many food processes, pharmaceuticals, detergents and biofuel industries. Italso Known as Indian coral tree or Tropical coral tree or Tiger's clow or Moochy wood tree or Variegated coral tree, sunshine tree, coral bean, Pangara (Marathi). It grows wild throughout the costal forest of India. Bark, root, leaves and fruits are used in fever, liver ailment. Rheumatism, relieve joint pain, and to kill worms.

Approaches: The Lipase produced by Erythrina Indica seeds was partially purified and characterized in terms of optimal p^H and temperature for activity as well as substrate specificity.

Keywords: Erythrina Indica seeds, protein estimation by assay, Dialysis Spectrophotometry, Gel Filtration, PAGE electrophoresis, optimum pH, optimum temperature, effect of metal ions, effect of surfactant and detergents, wash performance analysis

INTRODUCTION

Enzymes are biocatalyst that increases the rate of reaction. The first enzyme to be determined was urease in 1926 by James B. Sumner. Enzymes are proteins have an active site consisting of catalytic and binding sites. The binding site formschemical bond with specific ligands. Enzymes can increase the rate of reaction 10^{17} fold and showing specificity towards their substrate. The catalytic activity of an enzyme is regulated by other molecules or ions and by feedback inhibition as seen many biosynthetic pathways. In absence of enzymatic catalysis biochemical reactions are very slow that they would not occur mild conditions of temperature and pressure that are compatible with life. Lipases are among the most important classes of industrial enzymes. In recent years the demand of lipolytic enzymes has been increased due to its potential application in various manufacturing processes industrial goods such as detergent industry, food industry and cosmetics flavor enhancers and in pharmaceutical industry. Lipase may be used to produce fatty acids, aroma and flavor compounds lubricants and solvent esters, polyesters amides, thiol esters and bio modified fats.

Lipases are widespread in nature and present in animals, plants, and microorganisms. In plants lipase activity has been identified various tissues but relatively high concentration is found in seeds. Seeds are generally rich in triacylglycerol, which serve as compact source of energy for new plant.

Aims and objectives:

The identification of novel sources of lipases with unique patterns of reaction selectivity remains a strategic objective of lipase studies. Lipases are produced by plants, animals, bacteria and moulds. Manufacture of lipases by microbial fermentation constitutes a process that is currently popular. A less explored avenue is that of lipase extraction from natural sources. So we focus on the isolation of the lipase from easily available plant source; i.e., Erythrina Indica seeds.

METHODOLOGY

Materials:

Seed collection: seeds of Erythrina Indica were purchased from local markets.

Chemicals and biochemical's: acetic acid, acetone, acrylamide, ammonium sulphate, blue dextran bovine serum albumin, cadmium chloride, calcium chloride, cobalt chloride, comassie brilliant blue, copper chloride, copper sulphate, Disodium hydrogen phosphate, ethanol, ferrous sulphate, folin phenol reagent, glacial acetic acid, glycine, hydrochloric acid, lead oxide, magnesium chloride, mercuric chloride, papain, pepsin, p-nitro phenyl palmitate, potassium chloride, sephadex G100 , sodium acetate, sodium carbonate, sodium chloride, sodium dodecyl sulphate, sodium hydroxide, sulphuric acid, tetrametyl ethylenediamines, tris buffer, zinc chloride.

Isolation and purification of lipase from Erythrina Indica:

The 50 g seeds of Erythrina Indica were soaked in 200 ml water and kept for overnight at room temperature. The seed coats were peeled off and the soft cotyledons were homogenized in a mixer with saline (0.85% Nacl). The extract was filtered with cheese cloth kept for overnight. All further purification steps were carried out at $4\text{-}7^{0}$c. The homogenate was mixed with 1-butanol (20ml/100ml homogenate) and stirred for 4 hours at 4^{0}c on magnetic stirrer. The butanol layer was removed by centrifuged at 8000 rpm at 4^{0}c for 20 minutes. The supernatant thus obtained were collected and subjected to fractional salt precipitation with solid $(NH_4)_2SO_4$.

The highest lipase activity was found in the 30-80% ammonium sulphate salt precipitation. This fraction was dialyzed overnight against distilled water, saline, phosphate buffer p^H=8.0.50mM. Mobile phase for purification is phosphate buffer P^H=8.0; 50mM in dialysis bags. Dialysis was carried out in dialysis bags made up of cellophane placed in a plastic beaker containing desired solution against which contents of the bag was dialyzed. 3-4changes gives to remove the ammonium sulphate. The dialysis bags were washed in hot water prior to each use.

The dialyzed enzymes were used as partially purified enzyme and used for enzyme characterization. The dialyzed sample was clarified by centrifugation and loaded onto cell hydrophobic interaction column using phenyl sepharose matrix for purification. Fraction were collected by decreasing ionic strength showing lipase activity then eluted in gel filtration chromatography using G-100 matrix.

Lipase assay:

The spectrophotometric method was used for determination of lipase activity. The activities were determined by para-nitro –phenyl palmitate as substrate and by measuring the release of p-nitrophenol at 410 nm.one unit of Lipase activity is defined as the amount of enzyme that hydrolyzes 1μmol of p-nitrophenol per minute

Lipase assay:

	Blank	EB(ml)	SB(ml)	Test(ml)
Buffer	2.0	1.3	1.7	1.0
Enzyme	-	-	0.3	0.3
Incubate 5 min at 50^0 c				
Substrate	-	0.7	-	0.7
Incubate for 20 minutes at 50^0c and then cool at 8^0c for 10 minutes				

Protein Estimation:

Protein concentration of soluble enzyme preparation was quantified by method of Lowry et al using Bovine Serum Albumin (BSA) as standard. Plot the graph of O.D. verses μg of BSA. Using the standard calibration the protein concentration of the sample is calculated.

Lipase characterization:

Polyacrylamide Gel electrophoresis:

Enzyme purification or purity was checked on non-denaturing native PAGE using 10% gel concentration with slight modification on method described by Holt and Hartman. Page was performed at room temperature in Tris Glycine (p^H8.0). Gel was stained using the comassie Brilliant Blue R-250 staining solution. Total enzyme protein used for page was of 10 μg for crude as well as partially purified lipase.

Gel filtration chromatography:

Molecular weight of lipase from seeds of Erythrina Indica was estimated by gel filtration chromatography using sephadex G-100. The void volume of the column was determined by Blue dextran. The BSA (M.W.66kD), Pepsin (M.W.35kD), Trypsin (M.W.23kD), Lysozyme (M.W.14kD) proteins was used as molecular markers. The molecular weight was calculated by plotting a graph of Log (molecular weight) Vs elution volume/void volume.

Effect of p^H and Temperature:

For optimum p^H the activity of lipase was investigated within the p^H range of 4-10 using the acetate buffer (p^H=4-5, 0.05M), Phosphate buffer (p^H=6-8, 0.05M), Tris-Hcl (p^H=8-9), Glycine-NaOH buffer (p^H=9-10, 0.05M). 300μl of enzyme and 1 ml buffer of different p^H were incubated at 50^0c for 5 minutes 700μl of substrate was added and again incubated 50^0c for 20 minutes to start reaction. To stop the reaction cool at 8^0c. The absorbance was taken at 410nm. For optimum temperature, the enzyme assay was performed as discussed above expect that incubation period was done at temperatures from 20-90^0c.

Effect of metal ions:

The purified enzyme was incubated with 10mM EDTA (1:1v/v) at p^H=7 for 3 hours at 37^0c.The enzyme then dialyzed against mille Q water. The enzyme activity was checked by pre-incubating the demetalized enzyme. For the study of activator and inhibitor action on lipase, metal ion compounds of Al^{3+}, Hg^{2+}, Cu^{2+}, K^+, Zn^{2+}, Co^{2+}, Cd^{2+}, Ca^{2+}, Mg^{2+}, Fe^{3+} were used with 5 mM concentration incubated for 60minutes with demetalized enzyme(1:1)and assay was then carried out. Absorbance was taken at 410 nm.

Effect of surfactant and commercial detergents:

The detergent and surfactant solution were prepared as 1%w/v or v/v and then that solution were incubated in boiling water bath for 60 minutes to degrade the proteins present in detergent. The enzyme sample was incubated in presence of deproteinized surfactants viz. Triton X-100, tween-80, sodium taurocholate commercial detergents like tide, wheel, rin, surf excel, Ariel, ujala at 1:1 proportion for

1 hourat room temperature and then assay was carried out. Absorbance was taken J at 410 nm.

Wash performance analysis:

The wash performance of lipase was examined for removal of oily stains from cloths cotton swatches (2*2 cm) were stained with coconut oil. The cloth piece was dried and soaked separately in tap water, tap water with lipase, detergent with lipase.

Statistical analysis:

All experiments were conducted in triplicate and results were represented with standard deviation calculated by Microsoft excel program.

RESULTS AND DISCUSSION

Lipasecharacterization:

In the present investigation, an attempt has been made to isolate and purify the Lipase from Erythrina Indica seeds which is an important constituent in detergents. Erythrina Indica seeds were partially purified by acetone ammonium sulphate fractionation, followed by dialysis, hydrophobic interaction chromatography and gel filtration chromatography. Partial purification showed cut off of unnecessary proteins and was evidenced on native PAGE.
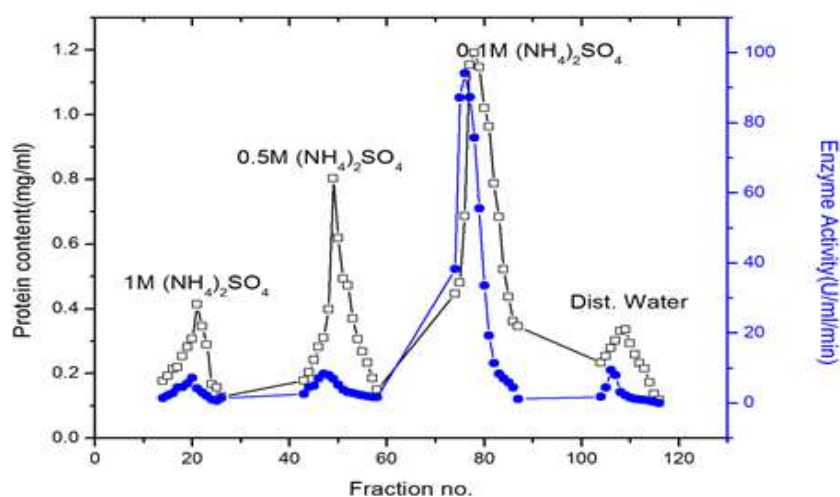
Hydrophobic interaction chromatography:

Fig 1: Elution Profile of Lipase by Hydrophobic interaction chromatography shows highest activity at 0.1 M $(NH_4)_2SO_4$.
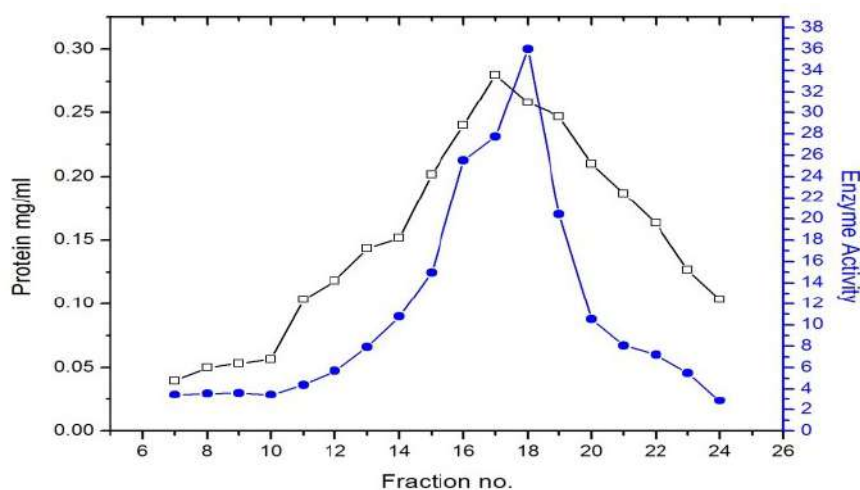
Gel filtration chromatography:

Fig 2: Elution Profile for gel filtration chromatography

Purification summary of lipase:

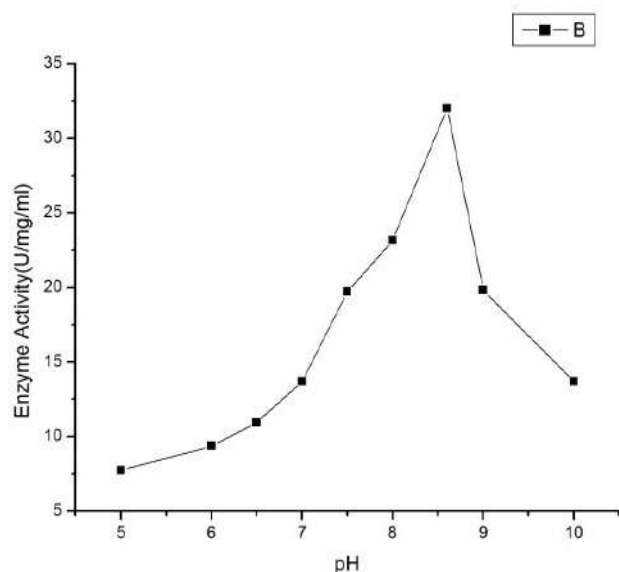| Stages of purification	Activity (units/ml/min)	Protein (mg/ml)	Specific activity	Fold Purification
Saline Extraction	120.7	60.00	2.011	1
Fraction A	111.1	39.40	2.8197	1.4021
Hydrophobic interaction chromatography	94.11	1.190	79.08	39.32
Gel filtration chromatography	36.00	0.280	128.57	63.94

Fig 3:

Fig 4:

Fig 3: Effect of p^H on Lipase activity from Erythrina Indica geminating seeds. Lipase assay performed at 50^0c and at various p^H values
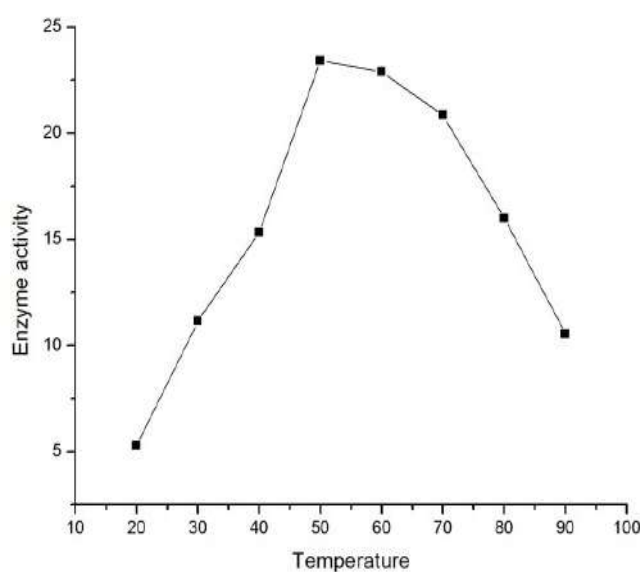
Fig 4: Effect of temperature on activity of the Lipase of the Erythrina Indica Seeds .Lipase assay was performed at $p^H 8$ and at various temperatures.

From above table it concludes that after gel filtration chromatography it gives highest purification value 63.94 folds.

Effect of pH on lipase activity:
The enzyme activity increased with an initial increase in p^H and optimum activity noted at $p^H 8.6$ suggesting alkaline nature of the enzyme. Further increased in p^H beyond optimum caused rapid decreased in enzyme activity (Fig. 3)

Effect of temperature on lipase activity:
The enzyme activity increased with an initial increase in temperature and optimum activity noted at 50^0c.

Further increased in temperature beyond optimum caused rapid decreased in enzyme activity (Fig. 4)

Effect of Metal ions and inhibitors:
Metal ions as Ca^{2+}, Co^{2+}, Cu^{2+}, Mg^{2+}, Zn^{2+} and k^+ at lower concentration shows highest activity on Lipase whereas at higher concentration the lipase activities were found to be inhibited. EDTA, Fe^{3+}, Cd^{2+}, $Hg2^+$ Co^{2+} inhibited the enzyme activity.

Effect of surfactant and commercial detergents:
Lipase exhibits highest activity in the presence of commercial detergentslike tide, wheel, rin, surf excel, Ariel, ujala
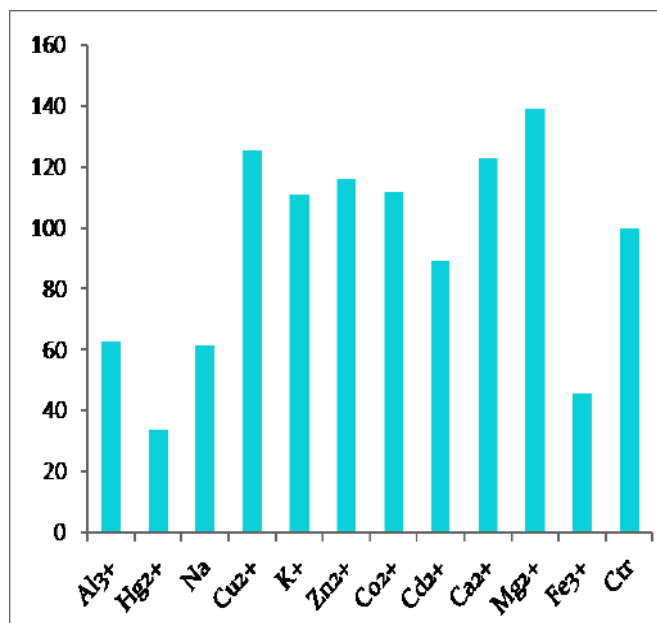
Fig 5:

Fig 6:

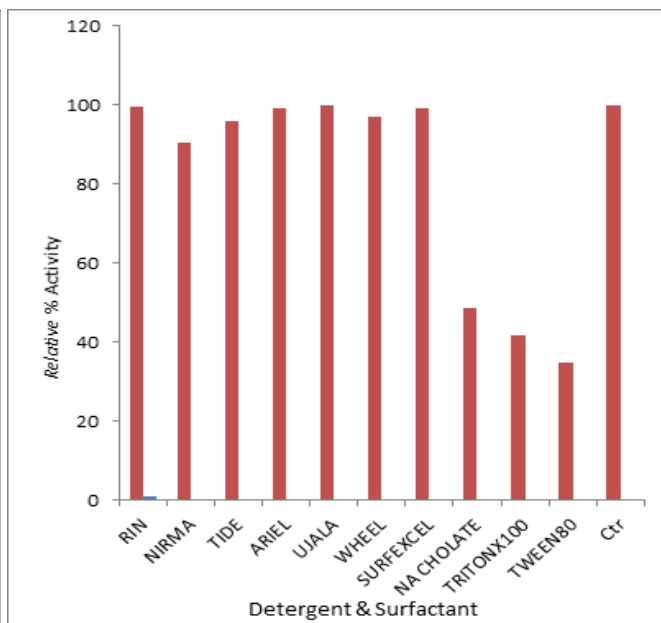**Fig 5:** Graph shows enzyme activity vs metal ions on Lipase.
Fig 6: Effect of surfactant and detergents on Lipase activity.

DISCUSSION

Lipase was isolated from Erythrina Indica seeds and purified. The results clearly indicate that Lipase of Erythrina Indica seeds are well suited for use as an additive in detergents formulation. Maximum activity at alkaline condition is compatible with harsh washing conditions. Maximum activity of lipase showed at temperature 50^0 c. the current thrusts for novel enzymes that tolerate oxidative stress makes the present lipase of high commercial value.

CONCLUSION

Lipases of Erythrina Indica seeds has so far shown the properties best suited for use in detergents and has superior properties to all existing Lipases. Lipase was isolated from Erythrina Indica seeds with saline and purified by dialysis, hydrophobic interaction chromatography and gel filtration. 63.94 folds purification was obtained. The enzyme shows maximum activity at p^H=8.6 and temperature at50^0c.Lipase of Erythrina Indica seeds has so far shown the properties best suited for use in detergents.Metal ions as Ca2+, Co2+, Cu2+, Mg2+,

Zn2+ and k+ at lower concentration shows highest activity on Lipase whereas at higher concentration the lipase activities were found to be inhibited. EDTA, Fe3+, Cd2+, Hg2+ Co2+ inhibited the enzyme activity. Thus, the lipase was successfully isolated and its characterization was carried out.

Conflicts of interest: The authors stated that no conflicts of interest.

REFERENCES

1. Abigor RD, Opute FI, Opoku AR and Osagie AU. Partial purification and some properties of the lipase present in oil palm (Elaeis guineensis). *J. Sci. Food Agric*., 1985; 36:399-406. DOI: 10.1002/jsfa.2740360711

2. Abigor RD, Uadia PO, Foglia TA, Haas MJ and Scott K et al. Partial purification and properties of lipase from germinating seeds of Jatropha curcas. L. *J. Am. Oil Chem. Soc*., 2002; 79: 1123-1126. DOI: 10.1007/s11746

3. Athawale V, Manjrekar N and Athawale M. Effect of Reaction Parameters on Synthesis of Citronellyl Methacrylate by Lipase-Catalyzed Transesterification. *Biotechnol. Prog*. 2003; 19: 298-302. DOI: 10.1021/ bp 0202867

4. Bahri S. Lipase activity in germinating sunflower seedlings. *Biochem. Soc. Trans*., 2000, 28: 771-773. PMID: 11171202

5. Barros M, Fleuri LF and Macedo GA. Seed lipases: sources, applications and properties. A review. *Braz. J. Chem. Eng.*, 2010, 27: 15-29.

6. Brockerhoff H and Jensen RG. Lipolytic Enzymes, 1st Edn., Academic Press, New York, 1974; ISBN-10: 0121345505, pp: 330.

7. Cancino M, Bauchart P, Sandoval G, Nicaud JM and Andre I et al. A variant of Yarrowia lipolytica lipase with improved activity and enantioselectivity for resolution of 2-bromo arylacetic acid esters. *Tetrahed, Asymmet.*, 2008., 19: 1608-1612.

8. Corzo G and Revah S. Production and characteristics of the lipase from Yarrowia lipolytica. *Bioresour. Technol.*, 1999, 70: 173-180.

9. Ejedegba BO, Onyeneke EC and Oviasogie PO. Characteristics of lipase isolated from coconut (Cocos nucifera linn) seed under different nutrient treatments. *Afr. J. Biotechnol.*, 2007; 6: 723-727. ISSN 1684–5315

10. Enujiugha VN, Thani FA, Sanniand TM, Abigor RD. Lipase activity in dormant seeds of the African oil bean (Pentaclethra macrophylla Benth). *Food Chem*. 2004, 88: 405-410.

11. Eze SOO and Chilaka FC. Lipolytic activities in some species of germinating cucubitaceae: Cucumeropsis manii naud, Colocynthis vulgaris L. and Cucubita moschata schrad. *World J. Agric. Sci.*, 2010; 6: 700-706. ISSN 1817-3047

12. Gandhi NN. Application of lipase. *J. Am. Oil Chem. Soc.*, 1997, 74: 621-634.

13. Hills G. Industrial use of lipases to produce fatty acid esters. *Eur. J. Lipid Sci. Technol.*, 2003, 105: 601-607. DOI: 10.1002/ejlt.200300853

14. Holt SM and Hartman PA. A zymogram method to detect endoglucanases from Bacillus subtilis, Myrothecium verrucaria and Trichoderma reesei. *J. Ind. Microbiol. Biotechnol*. 1994, 13: 2-4. DOI: 10.1007/BF01569654

15. Huang AHC. Involvement of glysosomal ingredients lipase in the hydrolysis of storage triacylglycerols in the cotyledons of soybean seedlings. *Plant Physiol.*, 1982, 70: 108-112.

16. Kumar A and Gross RA. Candida antarctica lipase b-catalyzed transesterification:New synthetic routes to copolyesters. *J. Am. Chem. Soc.*, 2002, 122: 11776-11770. DOI: 10.1021/ja002915j

17. Linder M, Matooba E, Fanni J and Parmentier M. Enrichment of salmon oil with n-3 PUFA by lipolysis, filtration and enzymatic re-esterification. Eur. J. Lipid Sci. Technol., 104: 455-462. DOI: 10.1002/1438-9312(200208)

18. Lowry OH, Rosebrough NJ, Farr AL and Randall RJ. Protein measurement with the Folin phenol reagent. *J. Biol. Chem.*, 1951, 193: 265-275.

19. Maliks SV, Kalia V and Pundir CS. Immobilization of porcine pancreas lipase on zirconia coated alkylamine glass using glutaraldehyde. *Indian J. Chem. Technol.*, 2000, 7: 64-67. ISSN 0971-457X

20. Michael JH, Cichowicz DJ and Dierov JK. Lipolytic activity of California-Laurel (Umbellulariacalifornica) seeds. *J. Am. Oil Chem. Soc.*, 2001, 78: 1067-1071. DOI: 10.1007/s11746-001-0390-0

21. Miled BDD, Zarrouk M and Chérif A. Sodium chloride effects on lipase activity in germinating rape seeds. *Biochem. Soc. Trans.*, 2000, 28: 899-902. PMID: 11171250

22. Neklyudov AD, Berdutina AV, Ivankin AN and Karpo BS. Kinetic characterization of enzymatic hydrolysis of complex protein substrates for producing nutrient media. *Appl. Biochem. Microbiol.*, 2002, 38: 328-334.

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1.Hofmann T. The Cluster-Abstraction Model: unsupervised learning of topic hierarchies from text data. Proceedings of the International Joint Conference on Artificial Intelligence. 1999.

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