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# RECENT ADVANCES IN SCIENCE & TECHNOLOGY

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## *Preface*

This book covers all disciplines in science and technology. The book contains 42 articles from different fields of science and technology. The contributions by the authors include Solar Energy, Synthesis and Pharmaceutical Applications of Heterocyclic Compounds, Synthesis and characterization of Ferrite nano-material, Ionic Liquid, Water Quality and Industrial Waste, Green Chemistry, Trace Element and Human Health, Catalyst, Stem Cell, Nano-Biotechnology, Anti-Corrosion Agent, Doping of Metal Ions, Parasitic infections in Fishes, Coordination Chemistry, Ultrasonic Studies on Molecular Interaction, Technique of fish and plant production, Green Hydrogen Energy, Direct Thermo-elastic problem, GC-HRMS analysis of Bioactive Compound, Water resource Management etc. Data published in this book is very useful for new researchers and academicians working in the field of science and technology.

Now a day, researchers have very much attention in research and academic interest as well as industrial needs especially in the field of Science and technology for different development. Therefore, an attempt has been made to provide a base of essential information of different areas in scientific research, which would help the budding researcher to develop a new model to design and implement the strategies for the development of society and the improvement of the quality of life. The recent advancement in science and technology has brought the revolutionary changes in every aspect of human life.

The book is intended to educate the reader on Current and Futuristic development in terms of Science and technology in India. Pharmacology, Green Energy, Atom Economy, Human Health etc. sustainable options and challenges associated with it to achieve its ambitious goal of Developed India.

I express my sincere thanks to Hon. Shri. Ravindra Patil Shisode, President, and Hon. Shri. Rajendra Patil Shisode, Secretary, Pratishtan Shikshan Prasarak Mandal, Pratishtan Mahavidyalya, Paithan for their kind support, blessing and love.

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I am also Thankful to “Royal Book Publishing” Tamil Nadu for publishing this edited book which is being useful for new researchers in a very short period of time.

I would like to express my thanks to all Co-Editor and Co-authors, for completion of this edited book in a prescribed period of time and to give beautiful shape to it which has only been possible because of the their continuous support.

**Dr. Ajay Manikrao Patil**  
**(Chief Editor)**

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## Chapter -1

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### Solar Energy: Made Simple for a Sustainable Future

Pritam A. Mule

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**Abstract:** Energy sources is available in various forms. Light is a form of energy. Heat and electricity is also form of energy. One form of energy can be turned into another. Electricity is used to light streets and buildings, to run computer and TVs and many other machines to run and appliances at home, at school and at many workplaces. Our planet has limited supply of oil and coal. So, In this chapter details about endless energy. Solar Energy, Energy from sunshine. Solar Energy is produced by the Sunlight is a non-vanishing renewable source of energy which is free from eco-friendly.

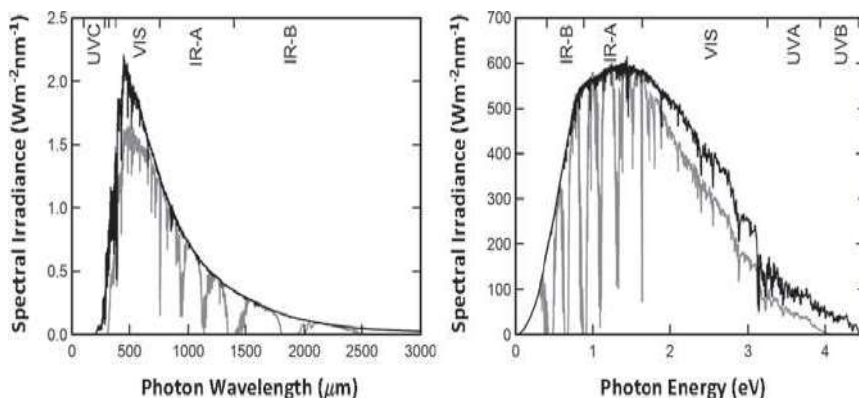
**Keywords:** Renewable Energy, Solar Panel, Photovoltaic Cell, Thermal Electricity

#### 1. Introduction

The sun drives 99.98% of the world's energy supply,<sup>1</sup> including thermal, photovoltaic, photochemical, photo biological and hybrid solar, hydro, wind, wave, and biomass energy conversion. It originally grew the biomass that we now access as fossil fuels. Other sources include tidal, geothermal and nuclear.<sup>1</sup> The sun's energy comes from fusion reactions in its core. These reactions have been "burning" for 4.5 billion years and are expected to continue for another 6.5 billion years. The total power radiated out into space by the sun is about  $3.86 \times 10^{26}$  W. Since the sun is approximately  $1.5 \times 10^{11}$  m from the earth, and because the earth is about  $6.3 \times 10^6$  m in radius, it intercepts only 0.00000045% of this power.<sup>2</sup> This still amounts to a massive  $1.75 \times 10^{17}$  W.

Most of this radiation is in the visible and infrared part of the electro-magnetic spectrum, with less than 1% emitted in the radio, UV

and X-ray spectral bands. The sun's electromagnetic radiation approximates that of a black body with a temperature around 5778K, with its peak in the yellow range of the visible spectrum. This is sometimes "rounded up" to 6000K for simplicity.<sup>3</sup>

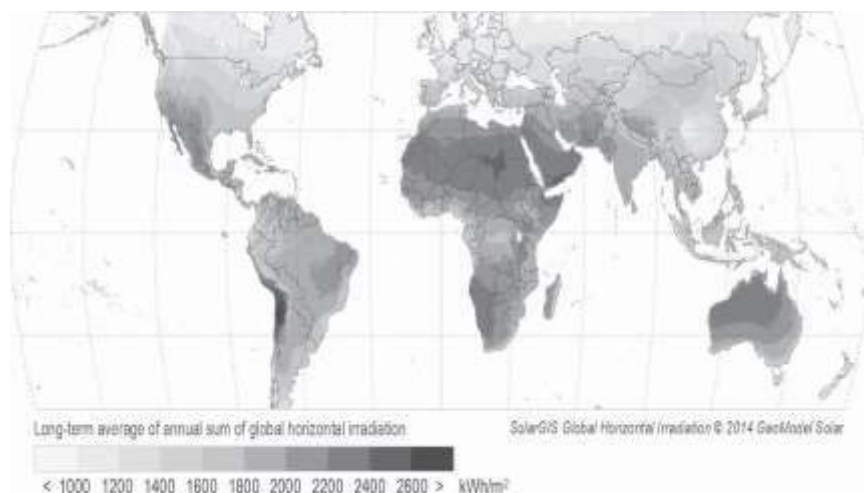


**Fig. 1. The AM0 spectrum of solar radiation, as would be observed at the top of the earth's atmosphere. The spectral irradiance is shown as a function of photon wavelength (left) and photon energy (right). The spectral regions are indicated. Image credit: Prof. Pietro Altermatt.<sup>5</sup>**

Figure 1 shows the spectrum of radiation from ultraviolet to infrared, as seen outside the earth's surface. This standard "AM0" spectrum<sup>13</sup> is that used to model and predict and qualify solar cells for use in space. The "AM" in the name refers to the "air mass", the thickness of terrestrial atmosphere through which the radiation has passed before it is observed, in this case zero.

## 2. Advantages and Disadvantages Associated with Solar Energy Use

Solar energy has several major advantages when compared with other sources. There source is distributed, though unequally, to every location on the globe. There source is abundant, to the extent that many countries have far more than they need to supply their energy needs from solar alone.



**Fig.3. Global solar radiation overt land surfaces of the world. Reprinted with permission from Solar GIS Global Horizontal Irradiation.**

It is effectively renewable on a human timescale, since the sun is expected to maintain similar production of its essential radiation at about the current rate for billions of years before eventually cooling to become a red giant.<sup>28</sup> Arguably, untraded solar energy already dominates the global energy supply as it grows our forests and crops that provide basic energy services to a large fraction of the world's population, warms our passive solar buildings, evaporates seawater to produce our industrial salt supply and even dries our crops, clothes and fuels outdoors.

### **3. History of Solar Energy Use**

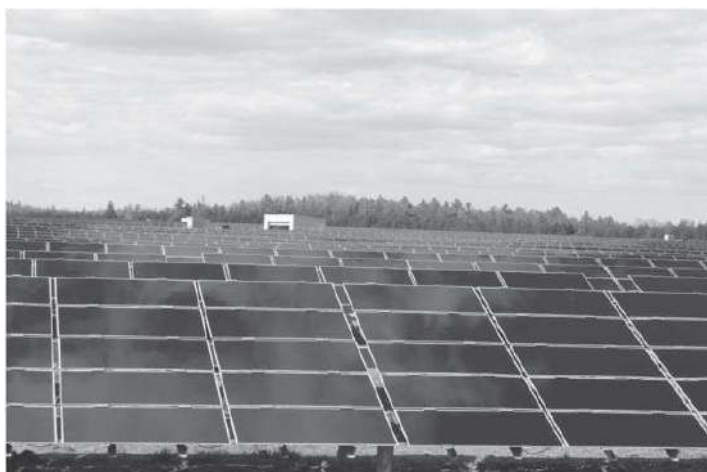
Solar energy has long supported humanity, with at least two forms, passive solar energy and biomass fuel use. Thus solar energy has been our partner throughout the progress of mankind. The growth of agriculture in the sunny "cradle of civilization" played a critical role in the development of civilization.<sup>11</sup> People have used the sun for drying crops, bricks, etc. since prehistoric times. The first known crop drying installation has been found in France and dates from around 8000BC.<sup>10</sup> There is evidence from around the world of dryer development in many civilizations and this relatively simple solar technology continues to change lives and economies for the better, even today, in remote locations all over the planet.<sup>12</sup>

## **4. Modern Applications of Solar Energy**

Methods to collect solar energy and convert it to useful forms range from the simple and traditional to modern and highly sophisticated. Outputs include low grade heat, high temperature industrial process heat, hydrogen, synthesis gas, synthetic hydrocarbons and other chemical energy carriers such as ammonia and metals, and intermittent or dispatchable electricity. These technologies are all at different developmental stages and associated cost of energy. We introduce a range of them in this section before they are treated in detail in the following chapters.

### **4.1 Photovoltaics for Large-Scale Electricity Production**

Photovoltaics have undergone continual development in the decades since 1950 until silicon and cadmium telluride photovoltaic now present credible challenge to fossil fuels and are one of the most promising methods to continue to provide electrical services to society in a carbon-challenged world. Government support programs for photovoltaics integration into electricity grids, most importantly and effectively in Japan and then Germany in the late 20th century, initiated the recent boom in demand and consequent mass production. Research associated with those programs solved the major safety concern about potential creation of live “islands” in otherwise closed grids and power quality and utility scale photovoltaics installations sprang up in many countries to compete with the incumbent whole sale electricity generators (see Fig. 4).



**Fig. 4. Part of a 9 MW utility scale photovoltaics installation at Stone Mills, Ontario Canada.**

**Photocredit: R. Corkish.**

#### **4.2 Photovoltaics for Small-Scale Off-grid Applications**

Prior to the rapid increase in the number of grid-connected systems in the late 20th century, the sustaining markets for the photovoltaic's manufacturing industry wherein those locations where all energy services are expensive. These locations are usually remote from grid electricity and other services, such as islands and rural areas. The main applications for the smaller systems, up to 1 kW, were and are water pumps, lighting and remote homes while those for larger installations were and are for commercial or industrial applications such as telecommunications repeaters, pastoral or mining.

#### **4.3 Concentrating Solar Thermal Electricity**

The concentration of the incident solar flux, for either photovoltaics or concentrating solar thermal (CST) technologies, is limited to regions with consistently high, direct, solar irradiation. This implies a separation from many of the areas where people have chosen to congregate in the world. It also demands mechanical tracking of collectors to keep them pointed at the sun, with consequent reliability concerns. CST technologies have had a much more positive history than has concentrating photovoltaics and several installations have been built in Spain and the USA<sup>7</sup>.



Other countries including China, India, and Australia have also built CST plants mainly on a test basis and the world has approximately 3800 MW capacity in operation with more than double that in the pipeline. The economics are currently not as attractive as those of flat plate photovoltaics but CST has the distinct advantage of easy coupling to relatively inexpensive storage, as sensible or latent heat or in thermochemical conversion (see next section). Integrated storage offers “dispatch able” energy, available on demand.

#### **4.4 Solar Thermochemical Processes**

Options to reduce the impact of intermittency on concentrating solar thermal plants are to store sensible or latent heat or to use it to drive endothermic thermochemical processes.<sup>5</sup> The product chemicals can be transported over long distances or stored and delivered to reformer reactors in which heat is released in exothermic reactions. Alternatively, solar heat may be applied to change the chemical composition of a fossil fuel to increase its calorific content or endothermic industrial chemical conversion processes may be directly utilized. These processes are likely to be applied only at very large scale, and are currently at a pre-commercial stage of development.<sup>6</sup> This storage option has the advantage over sensible and latent heat storage since that storage is at ambient temperature. In the case of solar-produced fuels, significant storage capacity may be afforded by the volume of existing reticulation systems at low cost.

#### **4.5 Solar Water Heating**

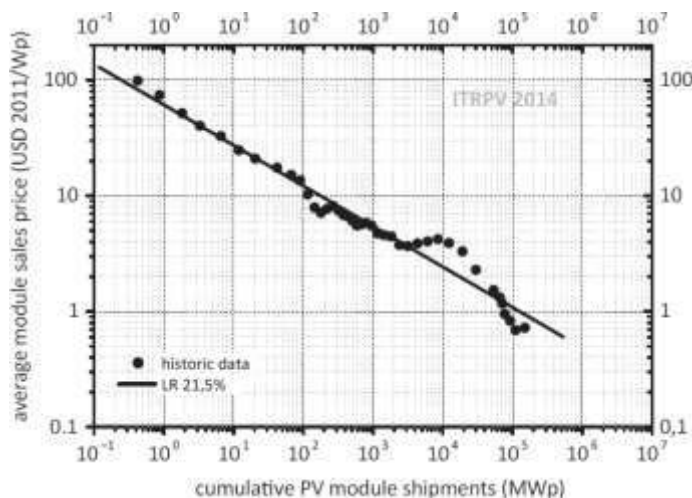
Solar water heating, both domestic and commercial/industrial, is so common and mainstream in many regions of today’s world that it is sometimes overlooked as a significant renewable energy technology.<sup>52</sup> Like non-concentrating photovoltaics, it is a technology that is easily incorporated in to urban infrastructure and displaces imported energy services. Two main collector technologies are already in mass production: flat plate and evacuated tube. In the former, the solar radiation heats a dark metal plate and heat is transferred to either a heat transfer fluid or directly to the water itself.

## 4.6 Evaporative Cooling

Like domestic solar water heating, evaporative cooling is a solar technology that has achieved mass market acceptance and is sometimes overlooked as a contribution of solar energy to human comfort and convenience. Evaporative cooling is a ubiquitous air conditioning choice in many of the less humid climate zones of the world. It works by using solar-heated ambient warmth to extract heat from a fan-forced flow of air by the latent heat of evaporation of water. Even more simply, passive forms rely on prevailing wind to blow air across ponds. It is the technology that supports the cooling towers prevalent in commercial/industrial air conditioning and industrial cooling and in fossil fueled and nuclear generation of electricity so it may be seen that evaporative cooling makes a huge contribution to human energy services but is generally excluded from renewable energy statistics.

## 5. Economics of Solar Energy Use

We write at a time when some solar energy technologies, most spectacularly photovoltaics, are fulfilling their long-term promise to reduce costs and prices to the point of credible direct competition with fossil and nuclear fueled electricity generation.



**Fig.6. Historic price reductions and the so-called “PV experience curve” for PV cells.<sup>61</sup>**

For example, prices in the USA of residential and commercial PV systems fell, on average, 6–7% per annum from 1998– 2013 and then rapidly, by 12–14% per year from 2012–2013. An additional 3–12% decrease is expected in 2014.<sup>7,8</sup> The ranges account for different market dynamics and cost structures for systems of different sizes. Much of the historical cost decrease has resulted from cost reductions in cell and module production, following a beneficial “experience curve” (Fig. 6),<sup>9</sup> which may flatten in the near term as the industry recovers from overcapacity resulting from the global financial crisis. However, drops in balance of system components and installation methods and soft costs<sup>62</sup> (including the non-hardware costs such as sales and marketing, financing, contracting, permitting, grid connection inspection, installation and O&M) are likely to allow the system price to continue falling. USA prices for installed photovoltaic systems have been reported to be more than twice the price in Germany, for instance,<sup>8</sup> suggesting the potential for significant reductions in the USA.

## **6. Summary/Conclusions**

Solar energy is on the verge of a massive boom. Together with wind energy, it directly challenges the incumbent dominant forms of traded energy, fossil and nuclear. This chapter outlines the rapidly improving economics of solar energy, particularly, flat-plate photovoltaics, in an inexorable march towards dominant market share as global concern grows about the impact of atmospheric carbon on climate change. We are already seeing the beginnings of divestment of fossil fueled energy by influential investors, as fears grow about stranded assets. The chapter also addresses the policy environment that has encouraged the development of solar energy in recent decades. However, some of these policies are under threat as energy companies, especially in the areas of electricity transmission and distribution, realize the challenge posed by solar to their traditional business models and learn to adjust those models to include distributed generation.

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## Chapter -2

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### A review on synthesis of imidazole and benzimidazole derivatives

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#### Abstract:

Imidazoles as well as benzimidazoles and their scaffold are extraordinarily essential class of nitrogen bearingazole heterocyclic compounds. They have used in wide area of organic synthesis which have applied in a various application in diverse fields including polymer, medicine, agriculture and chemical industries. Synthesis of imidazoles as well as benzimidazoles has been developed by condensation of simple as well as substituted diamine and substituted aldehydes using various nanocatalyst. Recyclable nanoparticles catalyst most efficient and straightforward protocol for development of imidazoles and benzimidazoles scaffold. Metal nanoparticles (NPs) are very useful as recyclable catalyst to suppress metal leaching into the product. In this review, we summarize the synthesis of reusable NPs and their reactivity in organic synthesis. By using such various types of NPs we have reduced reaction time, increase atom economy, increase product yield, reduced reaction steps and reduce cost of product.

**Keywords:** Imidazoles, benzimidazole, Catalyst, nanocatalysts.

#### 1. Introduction

For the last decades, nanocomposites materials have been widely reported in the scientific literature to afford substantial properties enhancements, even at low nanoparticles content. In nanotechnology, polymer nanocomposites are defined as solids consisting of a mixture of two or more phase separated materials, where one or more dispersed phase is in nanoscale and a polymeric major phase. Materials can be referred to as nanoscaled when their size,

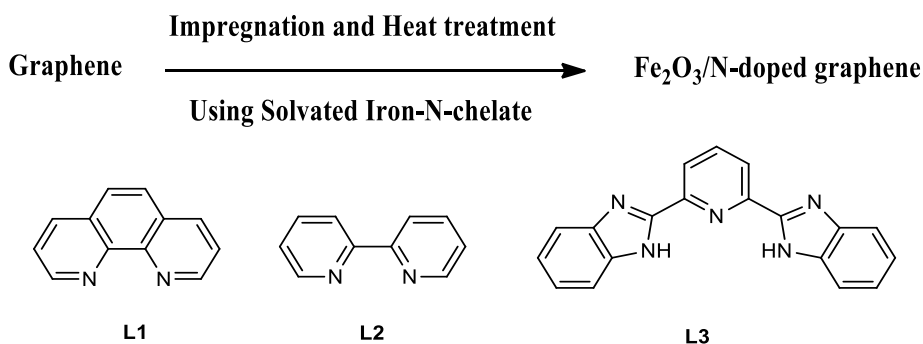
meaning at least one of the three external dimensions range from approximately 1 nm to 100 nm [1]. Nanocomposites can be processed by conventional wet and dry processing techniques, yet in adjusted conditions vs. their neat counterparts. Polymer nanocomposites and nanoparticles can also be applied as nanocoatings meaning a deposited nanoscale layer on selected substrates to reach specific surface behaviour [2,3]. Benzimidazole and its scaffold or 1H-1,3-benzodiazole primarily based heterocycles are structurally much like evidently taking the place of nucleotides, i.e., adenine base of the DNA [4-5]. This is considered in addition to a factor of vitamin B. This feature considerably has been utilized in drug development, synthesis and medicinal chemistry as well as pharmaceutical, showing enormous variety of organic and scientific solicitations. Benzimidazole is also named 3-azaindole, azindiole, benzimiinazole, benzoglyoxine. Benzimidazole is a vital modified structure that presents an extensive number of natural and pharmacologically active molecules. Magnetic nanoparticles (MNPs) have emerged as a new category of catalysts. This is referred to as ultrafine size and high surface area. They exhibit a better catalytic activity than a conventional heterogeneous catalyst. In general, there are artificial approaches for the synthesis of benzimidazole derivatives. The synthesized benzimidazole compounds have been organized from the condensation response between o-phenylenediamine and diverse carbonyl compounds. This process is carried out in the presence of ammonium chloride ( $\text{NH}_4\text{Cl}$ ) as a catalyst. Ammonium chloride is a commercial catalyst. The yield of all benzimidazole scaffold and its derivatives turned to be 75-94 % [6-10].

Herein, we review recent the methodology for the synthesis of recyclable nanomaterial and their reactivity.

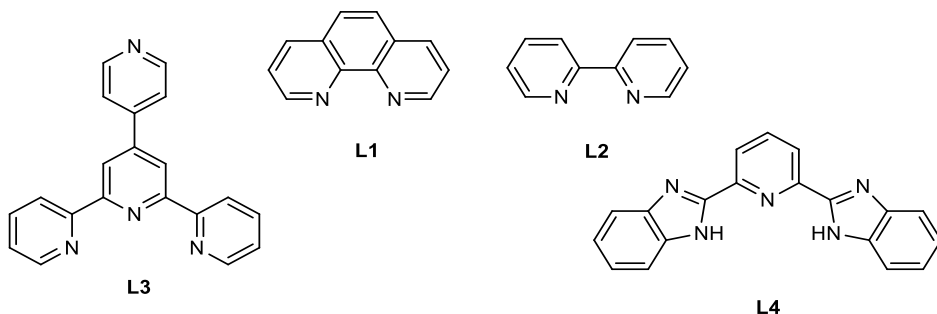
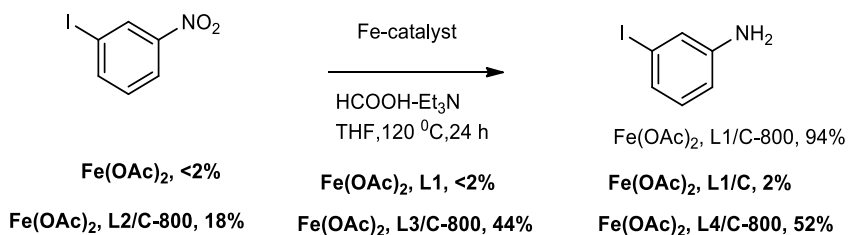
### **Iron nanoparticle catalysts for hydrogenation**

Beller and coworkers, in 2013, applied  $\text{Fe}_2\text{O}_3$ -based nanocatalysts ( $\text{Fe}_2\text{O}_y/\text{NGr}@C$ ) which they had prepared by pyrolysis of in situ-generated nitrogen-ligated iron acetate complexes on a carbon support (Figure 1) [11,12] These nanocatalysts were used for the hydrogenation of nitroarenes to test the scope and selectivity of the

$\text{Fe}_2\text{O}_3/\text{NGr}@C$  (Fe- L1/C-800) catalyst without using hydrogen gas (Scheme 1) [12]. In a model reaction, they found that the ligand on carbon with pyrolyzed iron is important to the reaction. The homogenous iron catalyst Fe-Oy/C and the Fe-phenanthroline catalyst were not active in this benchmark reaction. However, the  $\text{FeO}_3$  prepared from the Fe-1,10-phenanthroline L1 complex on carbon reduced 3-iodonitrobenzene to the corresponding aniline in excellent yield (Scheme 1). Other ligands such as bipyridine, terpyridine, and pyridine bisbenzimidazole had moderate activity.

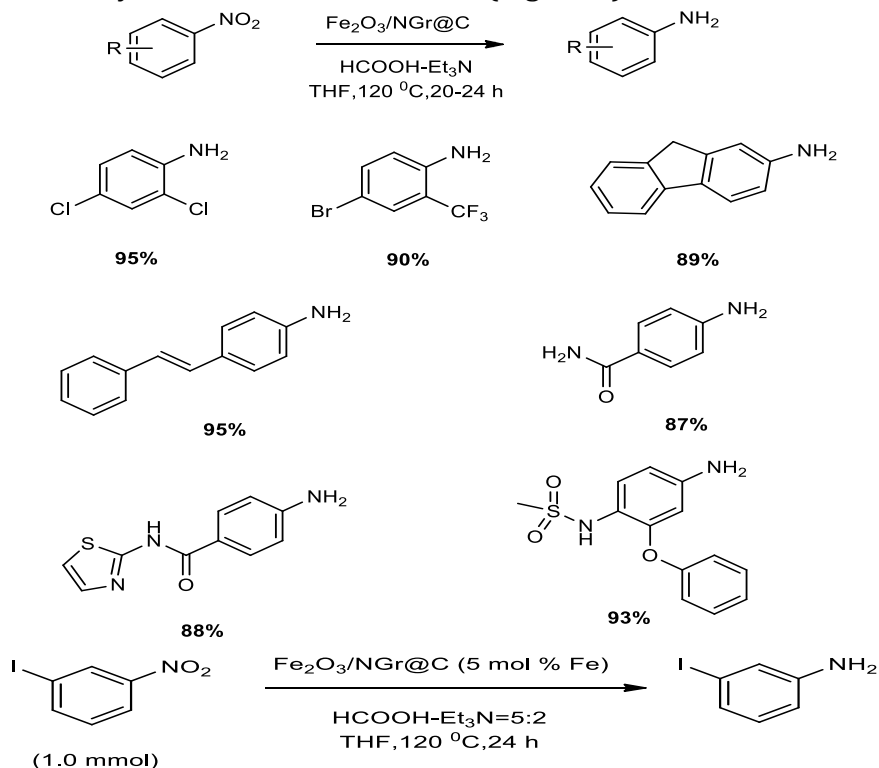
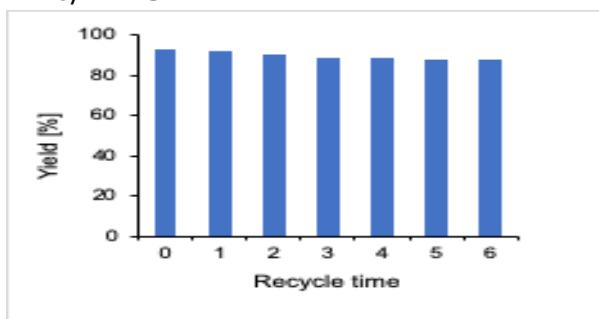


**Figure 1. Preparation of  $\text{Fe}_2\text{O}_3/\text{NGr}@C$ -catalysts using different nitrogen ligands (Fe = 3 wt %, Fe/L = 1:3 mole ratio)**



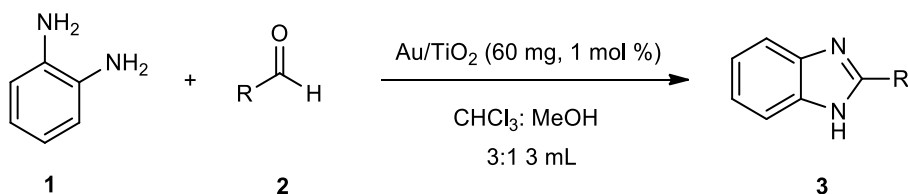
**Scheme 1.** Hydrogenation of 3-iodonitrobenzene with Fe catalysts.

Functionalized nitroarenes with structural diversity were hydrogenated to the corresponding amines such as halogenated anilines, various functional group-substituted anilines, and heterocyclic amines (Scheme 2). Remarkably, the catalyst has high stability and could be recycled more than five times (Figure 2).

**Scheme 2.** Chemoselective transfer hydrogenation of nitroarenes catalyzed by  $\text{Fe}_2\text{O}_3/\text{NGr@C}$ .

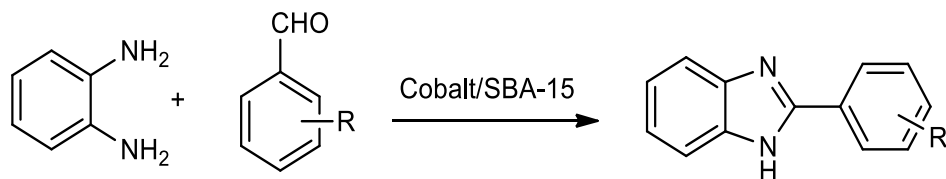
**Figure 2.** Recycling of  $\text{Fe}_2\text{O}_3/\text{NGr@C}$  catalysts in the reduction of 3-iodonitrobenzene.



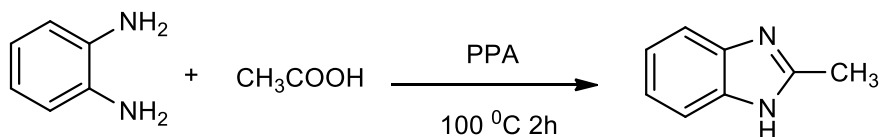


**Scheme 3.** Synthesis of 2-aryl substituted benzimidazoles in the presence of Au/TiO<sub>2</sub>.

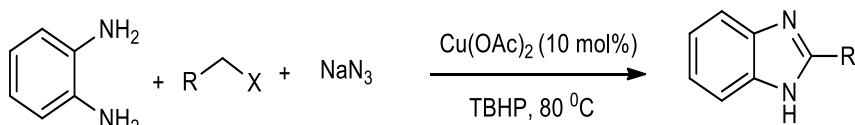
In **Scheme 3**, the commercially available Au/TiO<sub>2</sub> (2-3 nm AuNPs size) was used as an efficient catalyst for the selective synthesis of 2-aryl and 2-alkyl substituted benzimidazoles. The present heterogeneous catalytic protocol includes the one-step reaction between the corresponding aldehyde and the o-phenylenediamine, at ambient conditions, and in CHCl<sub>3</sub>:MeOH (3:1). This reaction has a wide-ranging substrate scope and exemplifies a new heterogeneous methodology for practical C-N bond formation under mild conditions, without additional additives and oxidants. The small-size gold nanoparticles (<5 nm) supported on TiO<sub>2</sub> were found to be the most active species under the present catalytic conditions. The catalyst Au/TiO<sub>2</sub> could be used at least five times without any significant loss of its catalytic efficacy. The present protocol applied to the lab-scale synthesis of 4-tolylbenzimidazole, as well as to the synthesis of the regio-isomer of the antifungal and antiparasitic Thiabendazole. [13] Rajabi et al [14] reported the one-pot synthesis of benzimidazol derivatives via oxidative condensation of aromatic aldehydes with o-phenylenediamine under mild conditions using a cobalt(II) supported on mesoporous silica-type material (Scheme 4).



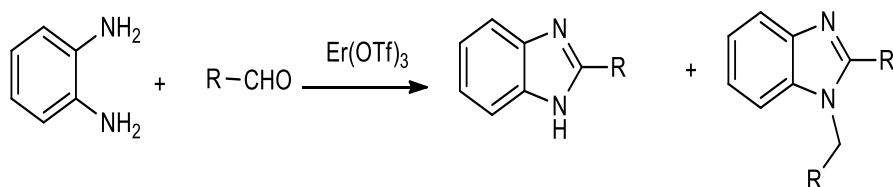
**Scheme 4.** Saini et al [15] reported the polyphosphoric acid is an efficient catalyst for the synthesis of 2-methylbenzimidazole from o-phenylenediamine and acetic acid. 2-methyl benzimidazole is a heterocyclic organic compound having an important pharmacophoric group which is used in medicinal industry (Scheme 5).



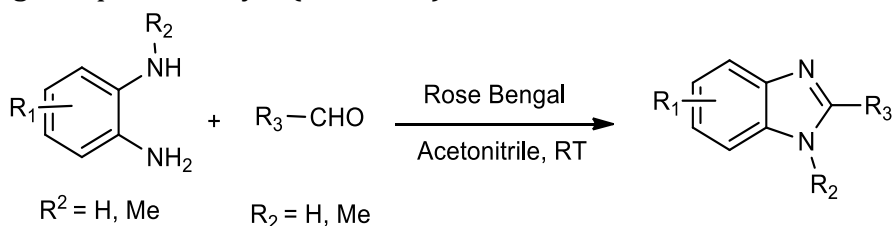
**Scheme 5.** Mahesh and co-workers [16] investigated the synthesis of 2-substituted benzimidazoles in presence of Cu(II) as catalyst. The targeted molecules were prepared by the reaction of aniline, primary alkylhalides and sodium azide (Scheme 6).



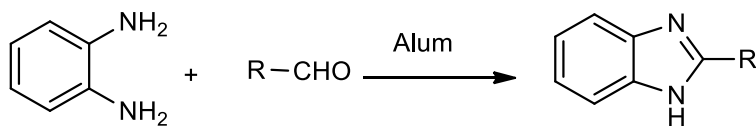
**Scheme 6.** Cano et al [17] developed an efficient protocol for the synthesis of benzimidazole derivatives, starting from *o*-phenylenediamine with diverse aryl aldehydes using  $\text{Er}(\text{OTf})_3$  as the catalyst (Scheme 7).



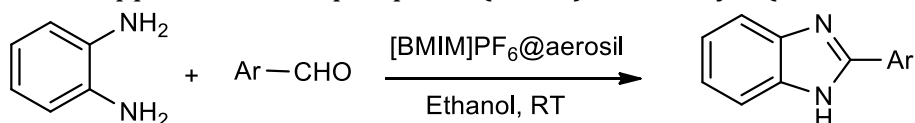
**Scheme 7.** Kovvuri et al [18] developed photocatalytic method for the synthesis of functionalized benzimidazoles via photocatalytic condensation of *o*-phenylenediamine and aldehydes using the Rose Bengal as photocatalyst (Scheme 8).



**Scheme 8.** Gurumeet C. Wadhawa *et al* [19] reported the synthesis of 2-substituted benzimidazoles and 1,5-disubstituted benzodiazepines using aldehydes through the condensation reaction between *o*-phenylenediamine and an aldehyde or a ketone to synthesize 2-substituted benzimidazole and 1,5-disubstituted benzodiazepines respectively (Scheme 9).



**Scheme 9.** Sonawane *et al* [20] reported synthesis of biologically relevant 2-substituted benzimidazoles from *o*-phenylenediamine and aryl aldehydes in high yields under mild reaction conditions by using aerosol supported ionic liquid phase (ASILP) as a catalyst (Scheme 10).



**Scheme 10.**

## Conclusion

Imidazoles, along with benzimidazoles and their framework, constitute a crucial class of nitrogen-containing azole heterocyclic compounds. These compounds play a significant role in a broad range of organic synthesis applications, contributing to diverse fields such as polymer science, medicine, agriculture, and chemical industries. The synthesis of imidazoles and benzimidazoles involves the condensation of both simple and substituted diamines with substituted aldehydes, employing various nanocatalysts and reagents. The utilization of recyclable nanoparticle catalysts represents an efficient and straightforward approach for constructing imidazole and benzimidazole scaffolds. The incorporation of various types of catalyst has demonstrated benefits such as reduced reaction time, increased atom economy, enhanced product yield, fewer reaction steps, and cost reduction in the production process.

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## Chapter -3

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### Study on ferritization temperature of Mn-Zn ferrites synthesized by using Oxalate Precursor method

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#### ABSTRACT:

The oxalate complexes of  $Mn_xZn_{1-x}Fe_2(C_2O_4)_3.nH_2O$  with  $x = 0.30, 0.32, 0.34, 0.36, 0.38$  were synthesized by using oxalate precursors from respective metal acetate solutions in  $CO_2$  atmosphere. STA (Simultaneous thermal Analysis) were carried out on the oxalate complexes in nitrogen atmosphere. The total mass loss % and step wise mass loss % values are in good agreement with theoretical mass loss % values. The studies revealed that the ferritization temperature lies in between the temperature  $367-402.7^\circ C$  and occurrence of simultaneous decomposition and ferritization process.

**KEYWORDS:** Mn-Zn ferrites, oxalate precursors, STA, Ferritization temperature

#### 1. INTRODUCTION:

Ferrite nanoparticles are very important magnetic materials from the view of the application in many fields such as biomedical technology, nuclear magnetic resonance, hydroelectric energy, optoelectronic sensor and catalyst [1-6]. Among other mixed ferrites frequently studied by the researchers, the Mn-Zn ferrites possess a spinel structure in which all the cations are distributed among the tetrahedral and octahedral sites making it a partially inverse spinel and are mostly used in many electronic devices. A special interest has been given to Mn-Zn ferrites because of their high chemical and thermal resistance, low electromagnetic losses, high magnetic permeability, high saturation magnetization and low production costs [7-8]. There

many types of preparatory techniques which are used to synthesize the Mn-Zn ferrites nanoparticles such as Coprecipitation [9-10], sol-gel methods [11], hydrothermal [12-13], combustion method [14], biosynthesis method [15], microwave assisted combustion method [14], auto combustion methods [16], solvothermal method [17-18], citrate precursor methods [19] and micro-emulsion process [20]. Many researchers [21-23] have been synthesized various mixed ferrites using this oxalate precursor method. The oxalate precursors are usually preferred due to their low solubility, low decomposition temperature and very fine particle nature [24]. It is therefore decided that to synthesize the various compositions of Mn-Zn ferrites using oxalate precursor method and investigated for their characterization by STA.

## **2. EXPERIMENTAL**

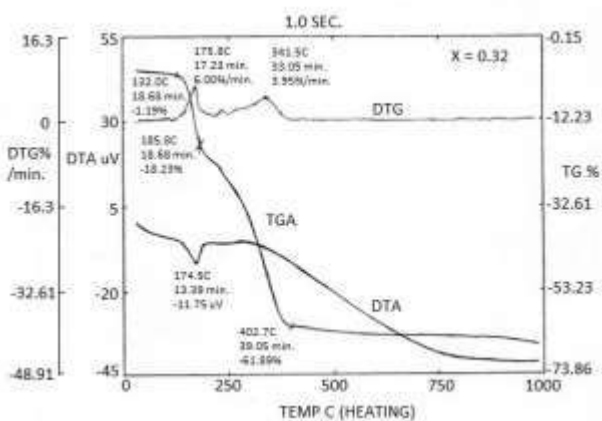
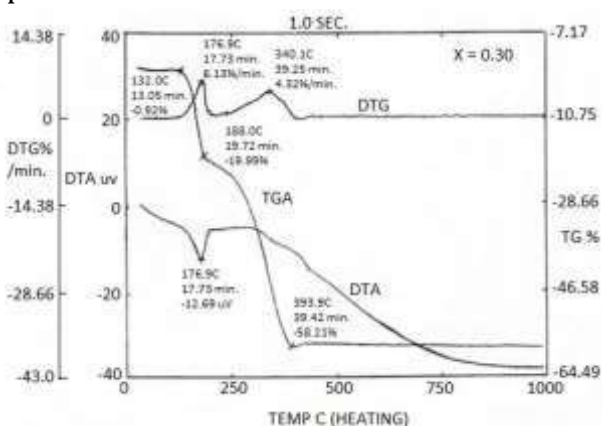
The oxalates were synthesized using the method suggested by Wickham [25] and later-on modified by M.Bremers et al [26] for synthesis of Mn-Zn ferrites. For each composition, ferrous acetate ( $\text{Fe}^{2+}$ ) was prepared by adding the AR grade glacial acetic acid and distilled water to required quantity of iron metal powder. To avoid the oxidation of Fe II to Fe III, the whole reaction was carried out in  $\text{CO}_2$  atmosphere instead of  $\text{N}_2$  [26]. To maintain desired stoichiometry, the required amount of Mn acetate, Zn acetate were dissolved in distilled water and warmed. The synthesized acetates were slowly added to hot ammonium oxalate solution to precipitate the required oxalate complex which then filtered and dried. In this manner, different oxalate complexes such as  $\text{Mn}_x\text{Zn}_{1-x}\text{Fe}_2(\text{C}_2\text{O}_4)_3 \cdot n\text{H}_2\text{O}$  where  $x = 0.30, 0.32, 0.34, 0.36, 0.38$  were synthesized.

## **3. RESULT AND DISCUSSION**

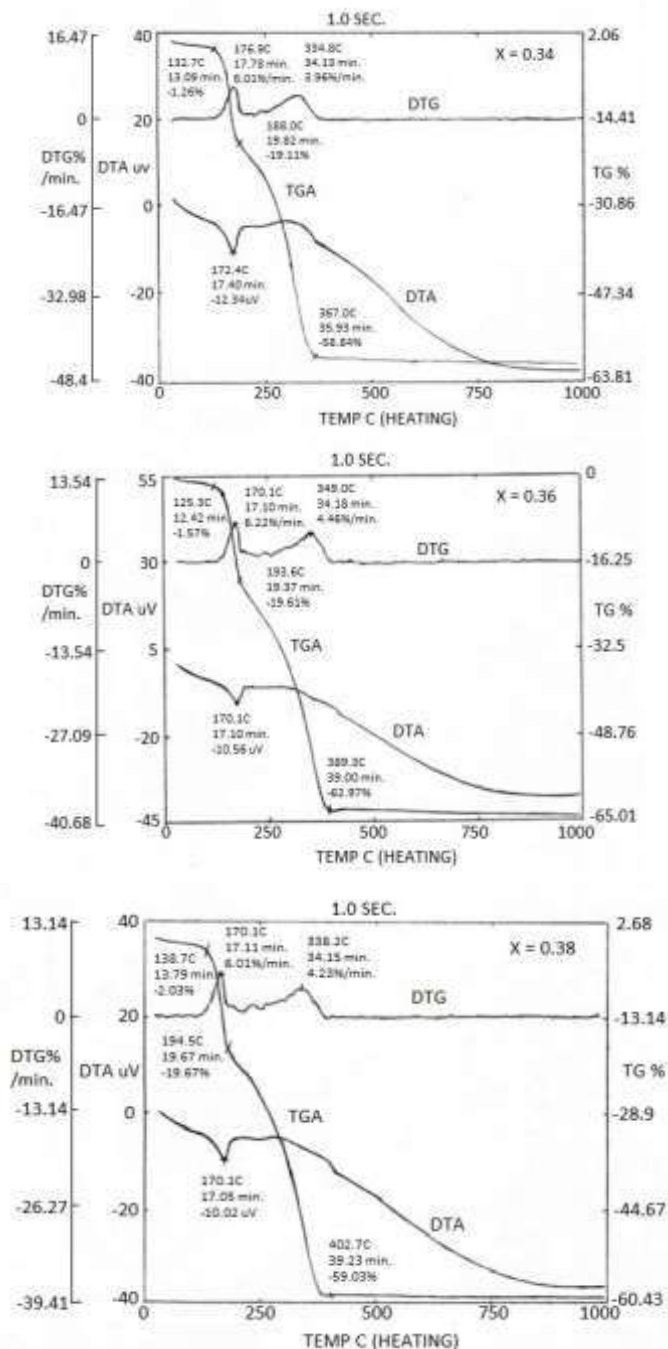
### **3.1 SIMULTANEOUS THERMAL ANALYSIS [STA]:**

The STA curves for present oxalate complexes are presented in Fig. 1. It is observed that the thermal decomposition of present oxalate complexes is take place in two different steps. Step I includes the dehydration step representing the loss of water of crystallization from oxalate complex and Step II includes the decomposition of anhydrous oxalate complex. DTA curves for all oxalate complexes shows that

dehydration is an endothermic reaction represented by a sharp peak whereas decomposition is an exothermic reaction. The observed total loss in % in comparison with total mass loss % based on the theoretical calculations and proposed formulae for respective oxalate complexes are mentioned in Table 1. It is seen that obtained total mass loss % is in good agreement with theoretically calculated values. The total mass loss in % experimentally observed is used to determine the total number of water molecules present in the respective oxalate complex. An initial weight loss about 0.83 to 2.03 % is observed in the temperature range of room Temperature to 138.7°C for all compositions which may be due to the evaporation of physically absorbed water on the surface of the particles. In dehydration, the weight loss of 17.04 to 19.07 % below the temperature about 194.5°C and in decomposition, the weight loss of 38.22 to 44.85 % is observed below the temperature 402.7°C.







**Fig. 1 STA curve for Mn<sub>x</sub>Zn<sub>1-x</sub>Fe<sub>2</sub>(C<sub>2</sub>O<sub>4</sub>)<sub>3</sub>.nH<sub>2</sub>O oxalate complex**

After this temperature, the curves don't show any further weight loss and hence attains the stability confirming the formation of final product and free from any other types of residue. The corresponding

onset, terminate temperature, time required for process such as dehydration and decomposition steps obtained from TG curve are given in Table 2. Table 3 represents the onset, peak and termination temperature for dehydration and decomposition steps and time after which peak occurs in dehydration steps and decomposition steps as derived from DTA curves. There is extremely close agreement between onset and termination temperature during dehydration and decomposition as derive from TG and DTA curves which is expected from theory. Table 4 gives the onset, peak and termination temperature for dehydration and decomposition steps and time after which peak occur in dehydration and decomposition steps as derived from DTG curves. From Table 2, it is seen that oxalate complexes have been dehydrated completely in the temperature range 125.3°C to 194.5°C whereas most of unhydrous oxalate complexes have been decomposed completely at 185°C to 402.7°C.

**Table 1: Stepwise and total weight loss in % obtained from theoretically and from TGA during dehydration and decomposition processes and proposed formulae for oxalate complexes**

Oxalate complex with Theoretical H <sub>2</sub> O molecules	Cal. total Th. wt. loss %	Proposed oxalate complex with actual H <sub>2</sub> O molecules	Th. Cal. total Wt. loss % from proposed oxalate complex	Actual Total ob. wt. loss %	Stepwise and total weight loss % from TGA			
					Initial wt loss	Dehy.	Decomp	Total
Mn <sub>0.30</sub> Zn <sub>0.70</sub> Fe <sub>2</sub> (C <sub>2</sub> O <sub>4</sub> ) <sub>3</sub> .10H <sub>2</sub> O	61.49	Mn <sub>0.30</sub> Zn <sub>0.70</sub> Fe <sub>2</sub> (C <sub>2</sub> O <sub>4</sub> ) <sub>3</sub> .8.5H <sub>2</sub> O	59.73	59.86	0.92	19.07	38.22	58.21
Mn <sub>0.32</sub> Zn <sub>0.68</sub> Fe <sub>2</sub> (C <sub>2</sub> O <sub>4</sub> ) <sub>3</sub> .10H <sub>2</sub> O	61.50	Mn <sub>0.32</sub> Zn <sub>0.68</sub> Fe <sub>2</sub> (C <sub>2</sub> O <sub>4</sub> ) <sub>3</sub> .9.5H <sub>2</sub> O	60.92	61.00	0.09	17.04	44.85	61.89
Mn <sub>0.34</sub> Zn <sub>0.66</sub> Fe <sub>2</sub> (C <sub>2</sub> O <sub>4</sub> ) <sub>3</sub> .10H <sub>2</sub> O	61.54	Mn <sub>0.34</sub> Zn <sub>0.66</sub> Fe <sub>2</sub> (C <sub>2</sub> O <sub>4</sub> ) <sub>3</sub> .8.5H <sub>2</sub> O	59.78	58.98	0.26	17.85	39.73	58.84
Mn <sub>0.36</sub> Zn <sub>0.64</sub> Fe <sub>2</sub> (C <sub>2</sub> O <sub>4</sub> ) <sub>3</sub> .10H <sub>2</sub> O	61.56	Mn <sub>0.36</sub> Zn <sub>0.64</sub> Fe <sub>2</sub> (C <sub>2</sub> O <sub>4</sub> ) <sub>3</sub> .9.5H <sub>2</sub> O	60.99	61.12	0.57	18.04	43.36	62.97
Mn <sub>0.38</sub> Zn <sub>0.62</sub> Fe <sub>2</sub> (C <sub>2</sub> O <sub>4</sub> ) <sub>3</sub> .10H <sub>2</sub> O	61.58	Mn <sub>0.38</sub> Zn <sub>0.62</sub> Fe <sub>2</sub> (C <sub>2</sub> O <sub>4</sub> ) <sub>3</sub> .8.5H <sub>2</sub> O	59.82	60.26	2.03	17.64	39.36	59.03

**Table 2: Data on onset, termination temperature in °C and time in minutes during dehydration and decomposition obtained from TG curve**

X	Dehydration				Decomposition		
	Onset temp.	Time	Termination temp.	Time	Onset temp.	Termination temp.	Time
0.30	132.0	13.05	188.0	19.72	188.0	393.9	39.42
0.32	132.0	18.68	185.8	18.68	185.8	402.7	39.05
0.34	132.7	13.09	188.0	19.82	188.0	367.0	35.93
0.36	125.3	12.42	193.6	19.37	193.6	389.3	39.00
0.38	138.7	13.79	194.5	19.67	194.5	402.7	39.23

**Table 3: Data on onset, peak and termination temperature in °C and occurrence of peak after time during dehydration and decomposition obtained from DTA**

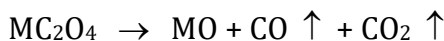
x	Dehydration				Decomposition			
	Onset temp.	Peak temp.	Occurrence of peak after time	Termin. Temp.	Onset temp.	Peak temp.	Occurrence of peak after time	Termin. Temp.
0.30	132.0	176.9	17.73	188.0	188.0	-----	-----	393.9
0.32	132.0	174.5	13.39	185.8	185.8	-----	-----	402.7
0.34	132.7	172.4	17.40	188.0	188.0	-----	-----	367.0
0.36	125.3	170.1	17.10	193.6	193.6	-----	-----	389.3
0.38	138.7	170.1	17.05	194.5	194.5	-----	-----	402.7

**Table 4: Data on onset, peak and termination temperature in °C and occurrence of peak after time during dehydration and decomposition obtained from DTG**

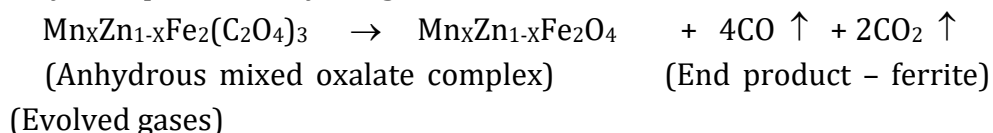
x	Dehydration				Decomposition			
	Onset temp.	Peak temp.	Occurrence of peak after time	Termin. Temp.	Onset temp.	Peak temp.	Occurrence of peak after time	Termin. Temp.
0.30	132.0	176.9	17.73	188.0	188.0	340.1	39.25	393.9
0.32	132.0	174.5	17.23	185.8	185.8	341.5	33.05	402.7
0.34	132.7	172.4	17.78	188.0	188.0	334.8	34.13	367.0
0.36	125.3	170.1	17.10	193.6	193.6	349.0	37.18	389.3
0.38	138.7	170.1	17.11	194.5	194.5	338.2	34.15	402.7

This fact may be attributed to the formation of homogeneous phase during synthesis of present oxalate complexes. It may be assumed that lowering the decomposition temperature of solid solution

oxalate complex is caused by earlier initiation of exothermic decomposition of Fe<sup>2+</sup> oxalate in it, the local heat thus generated being sufficient to effect the decomposition of Mn<sup>2+</sup>, Zn<sup>2+</sup> oxalate as well. From the curves it is clearly seen that dehydration shows a single step in case of oxalate complex of general composition Mn<sub>x</sub>Zn<sub>1-x</sub>Fe<sub>2</sub>(C<sub>2</sub>O<sub>4</sub>)<sub>3</sub>.nH<sub>2</sub>O thereby giving anhydrous oxalate complex. Thus in view of this observation it may be expected that anhydrous oxalate complex having general formula Mn<sub>x</sub>Zn<sub>1-x</sub>Fe<sub>2</sub>(C<sub>2</sub>O<sub>4</sub>)<sub>3</sub> may have been formed. The observation of single step in TG of oxalate complexes during decomposition may be taken as an indication of homogeneous phase during preparation. The decomposition of oxalate of type MC<sub>2</sub>O<sub>4</sub> produces oxides as an end product when they are decomposed in N<sub>2</sub> atmosphere as per reaction

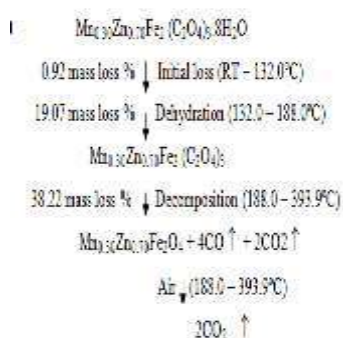


As the mass loss % during decomposition agrees well with theoretically calculated mass loss% values assuming mixed oxide as an end product. The decomposition of solid solution of oxalate complex may be represented by the general reaction

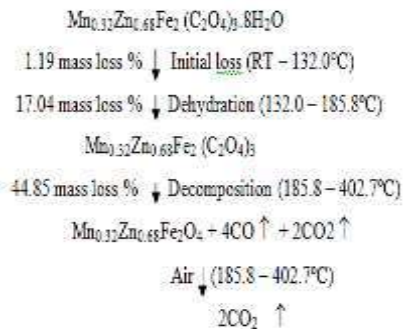


For the present oxalate complexes, dehydration and decomposition reactions can be represented as follows,

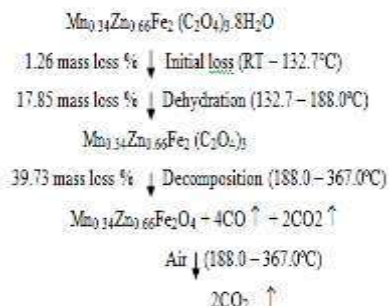
**1] x = 0.30**



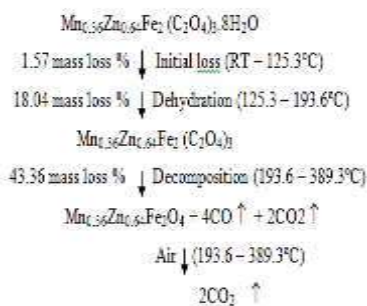
**2] x = 0.32**



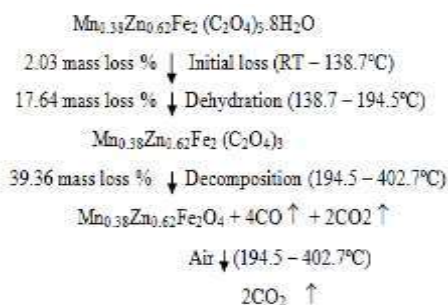
**3] x = 0.34**



**4] x = 0.36**



**5] x = 0.38**



The ferritization and decomposition processes are occurring simultaneously in temperature range as a nascent MO's are highly reactive and therefore, helps ferritization temperature to be lowered. Thermal studies indicates that after the completion of second stage, the TG curve is seen nearly a straight line running parallel to the temperature axis, indicating an absence of any further change i. e. simultaneous occurrence and completion of decomposition and ferrite formation and thereby stability of resultant ferrites.

**4. CONCLUSIONS:**

The following conclusions have been made -

1. Various compositions of Mn-Zn oxalate complexes are synthesized by using oxalate precursors.
2. Depending upon the composition, the ferritization occurs between the temperature ranges 367 to 402.7°C in very short time. The

ferritization temperature is far lower than that required for conventional ceramic techniques.

3. Both the process the decomposition and ferritization take place simultaneously.

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## Chapter -4

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### “Design And Synthesis of Novel 2,3,5-Substituted, 1,3,4-Oxadiazole Derivatives as Biologically Active Scaffolds”

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#### Abstract

New series of novel 1,3,4-Oxadiazole (**4a-c**) derivative bearing Benzofuran and pyrazole moiety was synthesized by acetylation cum cyclisation of (**3a-c**) with acetic anhydride. Reaction of 2-(5-(5-(benzofuran- 2- yl)- 1 -methyl- 1H-pyrazol-3-yl) - 1, 3, 4-oxadiazol- 2-ylthio) acetohydrazide (**1**) with various substituted aromatic aldehyde (**2a-c**) in ethanol produces (**3a-c**). The structures of newly synthesized compounds were corroborated through elemental and spectral studies like IR, <sup>1</sup>H NMR, C<sup>13</sup> NMR and Mass spectra. All the synthesized compounds were *in vitro* screened for their antibacterial activity against panel of pathogenic microorganism including. Gram positive bacterial strains, *S. aureus* and Gram negative strains *E. coli*, *P.vulgaris*, *S. typhi* compare with standard reference Chloramphenicol drug.

**Keywords:** 1, 3, 4-Oxadiazole, benzylidene, Pyrazole, carbohydrazide, Benzofuran.

#### Introduction

Increasing antimicrobial resistance due to misuse and overuse of antibiotic against human pathogenic microorganisms is also a global problem in medicine, placing significant burden on healthcare sectors as well as increasing morbidity and mortality worldwide. To overcome the growth of resistance it is important to yield a new class of antibiotics retaining decent biological Properties. N-containing heterocycles, especially five-membered rings, are of great interest as



they are found in natural products [1]. Heterocyclic compounds have held core stage in the development of molecules to enhance quality of human life. Perhaps, more than seventy per cent of drugs used today are heterocyclic compounds among that 1,3,4-Oxadiazoles containing different functional groups have attracted a great deal of attention from synthetic and medicinal chemists that has led to production of novel compounds with improved pharmacological properties. Oxadiazole a five membered heterocyclic aromatic compound having molecular formula  $C_2H_2N_2O$  belong to the most fertile group and valuable synthetic templates due to their wide-range spectrum of antimicrobial activities. Biological activities of oxadiazole are owing to the presence of  $-N=C-O$  linkage. 1,3,4-oxadiazole motif is of particular Crucial in materials in pharmaceutical chemistry as it can be used as a bioisosteric replacement of acid, ester, and amide functionalities [2-4].

Oxadiazole nucleus is fertile source of bioactivity in the area of drug discovery and its derivatives had been reported to exhibit several biological activities anti-inflammatory and analgesic[5-6], antimicrobial[7,8], antiviral[9], blood pressure lowering properties [10], anticonvulsant[11], anticancer [12],The all-encompassing literature review revealed that the existence of heterocyclic rings at the 2<sup>nd</sup> and 5<sup>th</sup> position of the oxadiazole ring escalations of the biological profile of these compounds to a larger extent. In the view of above observation an attempt has been made in present study to design and synthesized some novel oxadiazole derivatives (**4a-c**).

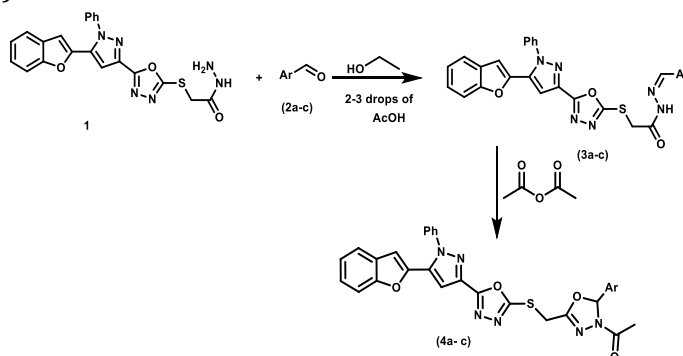
## **Material and Methods**

IR spectra were recorded on a Shimadzu IR Spectrophotometer (KBr,  $\nu_{max}$  in  $cm^{-1}$ ). Positive-ion electrospray ionisation (ESI) mass spectra were obtained with a Waters Micromass Q-TOF Micro, Mass Spectrophotometer. All the obtained products were screened for their antimicrobial activities.  $^1H$  NMR and  $^{13}C$  NMR spectra are recorded on a Bruker AM 400 instrument (400 MHz) using tetramethyl silane (TMS) as an internal reference and DMSO- $d_6$  and  $CDCl_3$  as solvent. Elemental analysis (CHN) was done using Thermo Scientific (Flash-2000).

## Experimental Procedure

**Procedure for the synthesis of N'-(4-methoxybenzylidene)-2-(5-(5-(benzofuran-2-yl)-1-phenyl-1H-pyrazol-3-yl)-1,3,4-oxadiazol-2-ylthio) acetohydrazide (3a):** 2-(5-(5-(benzofuran-2-yl)-1-methyl-1H-pyrazol-3-yl)-1,3,4-oxadiazol-2-ylthio) aceto hydrazide (**1**, 4.34, 10mmol), and 4-methoxyBenzaldehyde (1.21 ml, 10mmol) were taken in absolute ethanol (25mL), 2-3 drops of acetic acid was added as a catalyst, the reaction mixture was refluxed for 2h. Resulting mass was allow to cool, filtered and the product was recrystallized from absolute ethanol to get **3a** (Scheme 1). Similarly, **3b-c** was synthesized from **1** and **2b-i** by adopting the same procedure as described for synthesis of **3a**.

White amorphous solid mp, 198°C; yield, 72% ; ( from absolute ethanol); M.F ; C<sub>29</sub>H<sub>22</sub>N<sub>6</sub>O<sub>4</sub>S. IR(KBr,  $\nu_{\max}$  in cm<sup>-1</sup>): 3187, 3493 (N-H str.), 3070 (C-H str., aromatic), 2996, 2936 (C-H asym. str. aliphatic), 2836 (C-H sym. str., aliphatic), 1501, 1483 (C=C str., aromatic), 1107, 1102 (C-H i.p. def, aromatic), 835 (C-H o.o.p. def, aromatic), 1678 (C=O str., CONH), 1605 (C=N str.), 1254 (C-O-C sym. str.), 1031 (C-O-C asym. str.), 1461 (C-H asym. def, CH<sub>2</sub> and CH<sub>3</sub>), 1372 (C-H sym. def., CH<sub>2</sub> and CH<sub>3</sub>), 744, 776, 697 (C-S-C str.), 640 (C-S str.). <sup>1</sup>H NMR  $\delta$  ppm (DMSO-*d*<sub>6</sub>): 3.77 (s, 3H, -OCH<sub>3</sub> attached to aromatic ring), 4.65 (s, 2H, heteroaryl-S-CH<sub>2</sub>CONHN=CH-Ar), 11.69 (s, 1H, -NHCO-), 8.15 (s, 1H, -CH=N-), 6.67 (s, 1H, at C<sub>4</sub>-carban of pyrazole), 6.69-7.98 (m, 14H, aromatic + heteroaryl).



### Reaction Scheme: 1

**N'-(2-chlorobenzylidene)-2-(5-(5-(benzofuran-2-yl)-1-phenyl-1H-pyrazol-3-yl)-1,3,4-oxadiazol-2-ylthio)acetohydrazide (3b):** White amorphous solid mp, 207°C; yield, 74% ; ( from absolute ethanol); M.F

; C<sub>28</sub>H<sub>19</sub>ClN<sub>6</sub>O<sub>3</sub>S. IR(KBr,  $\nu_{\max}$  in cm<sup>-1</sup>): 3100,3485(N-H str.), 3050 (C-H str. , aromatic), 2975,2930(C-H asym. str., aliphatic), 2830(C-H sym. str., aliphatic), 1521,1453(C=C str., aromatic), 1100,1120(C-H i.p.def, aromatic), 845(-C-H o.o.p.def, aromatic, 1680(C=O str. ,CONH), 1630(C=N str.), 1260(C-O-C sym. str.), 1050(C-O-C asym. str.), 1458(C-H asym.def ,CH<sub>2</sub> and CH<sub>3</sub>), 1362(C-H sym. def. , CH<sub>2</sub> and CH<sub>3</sub>), 750,770,690(C-S-C str.), 640(C-S str.)

**(Z)-N'-(2-fluorobenzylidene)-2-(5-(5-(benzofuran-2-yl)-1-phenyl-1H-pyrazol-3-yl)-1,3,4-oxadiazol-2-**

**ylthio)acetohydrazide(3c):**White amorphous solid mp,205°C; yield, 76% ; ( from absolute ethanol); M.F ; C<sub>28</sub>H<sub>19</sub>FN<sub>6</sub>O<sub>3</sub>S. IR(KBr,  $\nu_{\max}$  in cm<sup>-1</sup>): 3105,3495 (N-H str.), 3050 (C-H str. ,aromatic), 2985,2940 (C-H asym. str. aliphatic), 2830(C-H sym. str. ,aliphatic), 1500,1463 (C=C str., aromatic), 1115,1125(C-H i.p.def, aromatic), 860(C-H o.o.p.def, aromatic), 1688(C=O str. ,CONH), 1632(C=N str.), 1263(C-O-C sym. str.), 1050(C-O-C asym. str. ), 1460(C-H asym.def ,CH<sub>2</sub> and CH<sub>3</sub>),1362 (C-H sym. def. , CH<sub>2</sub> and CH<sub>3</sub>), 751,770,680 (C-S-C str.), 652 (C-S str.).

**1- (5- ( (5- (5- (benzofuran - 2 - yl) - 1- phenyl - 1H - pyrazol - 3 yl)- 1, 3, 4 oxadiazol - 2 - ylthio) methyl) - 2 - (4-methoxyphenyl)- 1,3,4-oxadiazol-3(2H)yl)ethanone(4a):**N'-(4-methoxybenzylidene)-2-(5-(5-(benzofuran-2-yl)-1-phenyl-1H-pyrazol-3-yl)-2,3-dihydro-1,3,4-oxadiazol-2-ylthio)acetohydrazide(3a, 2.76g, 0.005 moles) was taken in acetic anhydride (30mL). The reaction mixture was reflux for 6h, allowed to cool and poured on crushed ice, reaction content kept for overnight, product obtained was filtered, washed, dried and recrystallized from ethanol white crystalline solid (**4a**).Similarly, **4b**-cwas synthesized from **3b-c**by following the same procedure as for the synthesis of **4a**.White amorphous solid m.pt, 170°C; yield, 70 % ; Rf;0.74(from absolute ethanol)M.F;C<sub>31</sub>H<sub>24</sub>N<sub>6</sub>O<sub>5</sub>S.IR(KBr, $\nu_{\max}$ in cm<sup>-1</sup>);3062, 3034(C-H str., aromatic), 2983,2938(C-H asym. str. aliphatic), 2905(C-H sym. str. ,aliphatic), 1373 (C-H sym. def. , CH<sub>2</sub> and CH<sub>3</sub>), 1486,1409(C=C str., aromatic), 1092(C-H i.p.def, aromatic), 896,799(C-H o.o.p.def, aromatic), 1609(C=N str. pyrazole and oxadiazole ring),

1305(C-N str.), 1259(C-O-C sym. str.), 1022(C-O-C asym. str.), 1022,979(N-Nstr.,oxadiazole),1737(C=Ostr.,-COCH<sub>3</sub>), <sup>1</sup>HNM Rδppm (DMSO-d<sub>6</sub>):2.50(s,3H,-COCH<sub>3</sub>), 3.34(s,3H,-OCH<sub>3</sub>), 4.30 (s,2H, hetero aryl-SCH<sub>2</sub>-heteroaryl), 6.68(s,1H,one proton at C<sub>4</sub> of pyrazole ring),7.27-7.65(m, 15H,aromatic,heteroaryl and fused heteroaryl ring).

**1 - (5- ( ( 5- ( 5- (benzofuran-2-yl)-1-phenyl-1H-pyrazol-3-yl)-1, 3, 4-oxadiazol-2-ylthio)methyl)-2-(2-chlorophenyl)-1,3,4-oxadiazol-3(2H)-yl)ethanone (4b):** White amorphous solid m.pt,185°C; yield,72%;Rf;0.63(from absolute ethanol )M.F;C<sub>30</sub>H<sub>21</sub>ClN<sub>6</sub>O<sub>4</sub>S. **IR(KBr,ν<sub>max</sub>incm<sup>-1</sup>);** 3069, 3030(C-H str. , aromatic), 2985,2940(C-H asym. str. aliphatic) ,2900(C-H sym. str. ,aliphatic), 1367 (C-H sym. def. , CH<sub>2</sub> and CH<sub>3</sub>), 1482,1410(C=C str., aromatic), 1089(C-H i.p.def, aromatic), 896,799(C-H o.o.p.def, aromatic), 1609(C=N str., pyrazole and oxadiazole ring), 1305(C-N str.), 1256(C-O-C sym. str.), 1027(C-O-C asym. str.), 1024,975(N-N str., oxadiazole),1732(C=O str. , -COCH<sub>3</sub>).

**1 -( 5- ( ( 5- ( 5- (benzofuran-2-yl)-1-phenyl-1H-pyrazol-3-yl)-1,3,4-oxadiazol-2-ylthio) methyl)-2-(2-fluorophenyl)-1,3,4-oxadiazol-3(2H)-yl)ethanone (4c):** White amorphous solid m.pt, 173°C ; yield, 70% ; Rf;0.70(from absolute ethanol )M.F;C<sub>30</sub>H<sub>21</sub>FN<sub>6</sub>O<sub>4</sub>S. **IR(KBr,ν<sub>max</sub>incm<sup>-1</sup>);**3072, 3035(C-H str., aromatic), 2973,2937(C-H asym. str. aliphatic), 2903(C-H sym. str. ,aliphatic), 1372 (C-H sym. def. , CH<sub>2</sub> and CH<sub>3</sub>), 1478,1409(C=C str., aromatic), 1079(C-H i.p.def, aromatic), 890,789(C-H o.o.p.def, aromatic), 1620(C=N str., pyrazole and oxadiazole ring), 1312(C-N str.), 1256(C-O-C sym.str.),1035(C-O-C asym. str.),1022,983(N-N str., oxadiazole),1725(C=O str. , -COCH<sub>3</sub>).

### Antibacterial activity

The novel synthesized heterocyclic compound(**4a-c**) were screened for their antimicrobial activity (*in vitro*) using cup plate agar disc-diffusion method against panel of pathogenic microorganism including Gram positive bacterial strains, *S. aureus* and Gram negative bacterial strains *E. coli*, *p.vulgaris*, *S.typhi* and there activity was compared with well-known commercial antibiotic Chloramphenicol .

**Table-1-: Antimicrobial Screening Consequence of 2,3,5-Trisubstituted 1,3,4-Oxadiazole derivatives (4a-c) against *S. aureus* and *P. Vulgaris* bacteria.**

Com P- Code	Zone of Inhibition (mm)											
	Gram +ve						Gram -ve					
	<i>S. aureus</i>						<i>P.vulgaris</i>					
Conc. (µg/mL)	Conc. (µg/mL)						Conc. (µg/mL)					
	1000	500	250	125	63.5	31	1000	500	250	125	63.5	31
4a	25	20	21	18	17	20	27	22	21	16	17	14
4b	22	21	19	18	16	17	25	21	19	15	18	11
4c	25	23	18	16	18	19	23	25	22	14	17	15
Std. Drug	24	22	20	19	16	17	28	24	20	17	16	13

**Table-2-: Antimicrobial Screening Consequence of 2,3,5-Trisubstituted 1,3,4-Oxadiazole derivatives (4a-c) against *E. Coli* and *S. Typhi* bacteria.**

Comp. Code	Zone of Inhibition (mm)											
	Gram -ve											
	<i>E. coli</i>						<i>S.typhi</i>					
Conc. (µg/mL)	Conc. (µg/mL)						Conc. (µg/mL)					
	1000	500	250	125	63.5	31	1000	500	250	125	63.5	31
4a	27	21	24	20	18	17	18	12	14	13	10	11
4b	21	20	20	17	16	16	15	11	09	07	08	10
4c	22	19	24	17	18	16	18	11	13	12	10	09
Std. Drug	26	24	23	21	17	14	17	15	12	11	09	08

**RESULT AND DISCUSSION**

The IR spectrum of **3a** shows confirmation of structure by different absorption band primarily 3187 cm<sup>-1</sup>, 3493 cm<sup>-1</sup> -NH- stretch, 1605 cm<sup>-1</sup> (C=N stretch Schiff base, 1678 cm<sup>-1</sup> -C=O stretch in -CONH-), 640 cm<sup>-1</sup> -C-S stretch. <sup>1</sup>H NMR of (**3a**) appearances at  $\delta$  3.77 ppm singlet, 3H, confirms -OCH<sub>3</sub> attached to aromatic ring  $\delta$  11.69 ppm singlet, 1H, -NHCO- group,  $\delta$  8.15 ppm singlet, 1H, -CH=N-,  $\delta$  6.67 ppm singlet, 1H, -CH- at C<sub>4</sub>-carban of pyrazol ring. Synthesis of -1,3,4-oxadiazol derivative (**4a-i**) was carried out by reacting comp. (**4a-i**) with acetic anhydride IR spectrum of (**4a**) shows that 1609 cm<sup>-1</sup> -C=N pyrazol & oxadiazole, 1737 cm<sup>-1</sup> -C=O stretch, 1022 cm<sup>-1</sup>, 979 cm<sup>-1</sup> -N-N stretch in oxadiazole, 1259 cm<sup>-1</sup> (C-O-C sym str), 1022 cm<sup>-1</sup> -C-O-C Assym stretch, <sup>1</sup>H NMR signal at  $\delta$  2.50 ppm singlet, 3H, confirms -COCH<sub>3</sub>,  $\delta$  3.34 ppm singlet, 3H, of -OCH<sub>3</sub>,  $\delta$  4.30 ppm singlet, 2H, of -CH<sub>2</sub>-,  $\delta$  6.68 ppm singlet, 1H, -CH-C<sub>4</sub> of pyrazol ring.

**CONCLUSION**

In summary, We reported new series of novel 1,3,4-Oxadiazole (**4a-i**) derivative bearing Benzofuran and pyrazole moiety all the compound show good to moderate activity against selected strains *S. aureus*, *P. vulgaris* and *E. coli*, *S. typhi* at lower concentration.

**ACKNOWLEDGEMENTS**

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## Chapter -5

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### Bioactive Heterocyclic Ligands are Important Pharmaceutical Agents

**Take S.A.**

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**Abstract:** Schiff bases are potential, intriguing biological substances with a variety of medicinal uses, such as antipyretic, anti-inflammatory, and antibacterial effects. Schiff bases have been synthesised from the reaction of bio-active heterocyclic ligands with different aldehydes. These compounds were evaluated in vitro against a variety of clinically and commercially relevant bacterial strains to assess their antibacterial activity, as well as against many human carcinoma cell lines to assess their anticancer activity. Antimicrobial agent resistance has been caused by the overuse of medication. High morbidity and death have been brought on by the resistant microbes. Research in pharmaceutical and medicinal chemistry has been skewed by schiff bases' simple synthesis, broad spectrum of pharmacological activity, and azomethine functional group (CN) (Malladi et al., 2013).

**Keywords:** Schiff base, Anti-inflammatory, Antibacterial and antifungal, Antipyretic.

#### **Introduction:**

Many bacteria, including those with multi resistance phenotypes, have been shown to develop antibiotic resistance in recent decades [1, 2, 3, 4]. The World Health Organization has stated that this is a concerning scenario, and several researchers have concentrated on creating novel therapy options. Different approaches have been put up by Oldfield and Feng to create a new class of antibiotics (resistance-resistant antibiotics) that will be able to act on several targets at once [5]. As an alternative, some writers have proposed the creation of new antibiotic classes with unique chemical characteristics not found in the classes of antibiotics that are now in use [6]. Schiff bases have received



much attention due to their wide applications in several fields, ranging from industrial uses such as advanced nanomaterial's [7] to chemotherapeutics and new drug development [8,9,10,11]. However, even regarding the chemotherapeutic applications, the reported roles are very divergent, sometimes with proposed roles that could seem contradictory. For example, antioxidant activity has been ascribed to Schiff bases, acting as cell-protective agents [12,13,14,15], but antimicrobial [16,17], antitumor [18,19], analgesic [12], anti-inflammatory [9], and antidiabetic activities [8,18] have also been reported. A detailed review covering the patented therapeutics applications of Schiff bases has been published by Khan and collaborators [18].

### **Anti-inflammatory:**

New Fe(III), Co(II), Ni(II), Cu(II), and Zn(II) complexes incorporating 4 - { [ ( 2- hydroxyphenyl) imino] methyl } phenyl 4-methylbenzenesulfonate Schiff-base ligand (HL) were designed, synthesized, and characterized by Nadia and et al.[19]. Heterocyclic thioamides constitute a diverse class of compounds with important role in drug discovery [20]. Thiophene derivatives and thiadiazoles including derived ortho-condensed heterocycles have been found to have a wide range of biological activities.[21]

### **Antibacterial and antifungal:**

Ternary complexes of Cu(II) with acetylacetone and various salicylic acids (viz., salicylic, 5-chloro-, 3,5-dibromo-, 3,5-dinitro-, thio-, and acetyl-salicylic acids) were synthesised in pure state and these complexes by Y. Anjanuyulu and et al.[22] The antibacterial and antifungal activity studies on these complexes revealed that the ternary complexes are better toxic agents than the binary complexes against certain bacteria and fungi studied.

### **Antipyretic:**

The novel nickel(II) complex was synthesized through the reaction of Ni(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O with 4-aminoantipyrine (AAP) ligand in a 1:3 mole ratio of Ni(II) to AAP by YusicaAmaliaRasyda and et al.[23]. 2-Mercaptoquinoline thiosemicarbazonemetal(II) complexes of the type

[M(L)<sub>2</sub>], (M = Co, Ni, Cu & Zn) were synthesized by GaneshanGokulnath and et al.[24]

**Anticancer:**

Two ruthenium compounds, namely [ImH]trans-[RuCl<sub>4</sub>(Im)(dmso-S)] (NAMI-A, Im = imidazole) and [IndH] trans-[RuCl<sub>4</sub>(Ind)<sub>2</sub>] (KP1019, Ind = indazole) have already completed phase I clinical trials as anticancer agents.[25] The development of new metal anticancer compounds is a challenge for inorganic chemists. We have to face the fact that four decades of research in this field have only produced a small number of clinically used compounds, most often developed through serendipity rather than through rational chemical design.[26]

**Conclusion:**

Metal complexes exhibiting antimicrobial, antibacterial, anticancer, and antipyretic properties hold significant promise for advancing therapeutic interventions. Their diverse activities underscore the potential for targeted applications in combating infectious diseases, cancer, and fever-related conditions. Further research and development in this field are crucial for harnessing the full therapeutic potential of these metal complexes and addressing complex health challenges.

**Antidiabetic:**

Antidiabeticvanadium(IV) and zinc(II) complexes was synthesied and studied by Heromu Sakurai and et al.[27] The action of anti-diabetic vanadium compounds was studied.

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## Chapter -6

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### Choline Based Ionic Liquids and their Applications In Organic Transformation

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#### **Abstract:**

Chemical researchers always want to find new classes of solvents for the chemical reactions and processes. One such class includes the ionic liquids which has become more popular in the recent years. Ionic liquids exhibit some adverse impact on the environment as they are non-biodegradable and toxic to the environment. The choline chloride (ChCl) based ionic liquids has become replacement for imidazole based ionic liquids and are non-toxic, biocompatible, easy accessible and cost-effectiveness. Variety of choline based ionic liquids and their applications in various fields are being reported nowadays.

In this study, we review introduction and background of the ionic liquid choline as cation for ionic liquids and choline based ionic liquids in the organic synthesis. Ionic liquids are the future solvents of the chemical and allied industry of being their greenness property.

**Keywords:** Ionic liquid, Choline, Biodegradable, Green Synthesis.

#### **1.1 INTRODUCTION:**

As an importance of “The Paris UN Climate Conference 2015”, the world needs a new model of growth that is safe, durable and beneficial for all to meet the climate change challenge. Hence, researchers both from the academia and the industry have to pay greater attention on the detrimental effects of chemicals and chemical processes on the environment. In the recent years, struggling for a sustainable developments in the chemical fields has become essential task, which is done by applying green chemistry practices [1]. One of the important criteria for green and sustainable chemical processes is

the use of eco-friendly solvents having minimum or zero impact on aquatic organism as well as on environment by adopting 12 principle of green chemistry [2]. The best alternative solvent for the chemical reactions are the ionic liquids. Ionic liquids are the organic salts with low melting points that are liquids at or near room temperature [3]. They are known as “room temperature molten salt,” “low temperature molten salt,” “ambient temperature molten salt,” “liquid organic salt,” “liquid electrolytes,” “ionic melts,” “ionic fluids,” “fused salts,” “liquid salts,” “ionic glasses,” [4] “solvents of the future” or “designer solvents.” in the literature [5], [6]. The ionic liquids are possible green replacements to organic solvents for chemical reactions, extractions and bio transformations. They are non-volatile, thermally stable and their solvation properties vary by changing the cation and the anion. The high cost, poor biodegradability, biocompatibility and sustainability are the disadvantages of the ionic liquids. Ionic liquids are the liquids that make new technologies possible through environmental feasibility.

## **1.2 IONIC LIQUIDS:**

The majority of chemical processes till now utilized molecular solvents. Due to their massive use in the chemical industry and laboratories, these molecular solvents may pollute the environment and have a negative impact on organisms and human health. They are also poisonous, combustible, explosive and volatile. In order to reduce the production of chemical waste, dangerous solvents and catalysts use must be avoided<sup>1</sup>. Academic and industry researchers have focused on decreasing the use of hazardous solvents to reduce environmental harm, and the quest for a safer catalyst and solvent continues. The academic and industrial scientific communities are interested in ionic liquids as a potential new green chemical revolution. Due to its advantageous characteristics, this distinctive class of molecular solvents might decrease the use of dangerous and harmful organic solvents.<sup>2</sup> Ionic liquids are characterized as compounds that are entirely made of ions and melt at or below 100°C.<sup>3</sup> Due to their nonvolatile nature, thermal and chemical stability, they have attracted a lot of interest in relation to green chemistry and are green alternatives

to traditional solvents for many chemical processes in the field of sciences.<sup>4-5</sup>

Paul Walden synthesized the first ionic liquid, ethyl ammonium nitrate in 1914<sup>6</sup>. Wilkes and coworkers synthesized first generation room temperature IL using imidazolium and tetrachloroaluminates<sup>7</sup>. This salt is liquid at room temperature and has better physical and electrochemical properties, despite being sensitive to moisture and often containing a small amount of water. The second generation of ILs, which are air and moisture stable, were developed in 1990 when the moisture-sensitive anion was replaced with the tetrafluoroborate ion and other anions.<sup>8</sup> These ILs have been utilized successfully as a reaction medium in a variety of organic processes. By integrating a functional group as a part of the cation or anion, David and colleagues in 1999 developed the third generation IL. These IL can act as a catalyst and a solvent in many chemical processes.<sup>9</sup>

They are useful for a wide range of tasks due to the ions that can be combined with each other and the capability of task-specific molecular design. However, many ILs are unsafe and have an influence on the environment.<sup>10</sup> Therefore, the creation of substitute ILs using relatively affordable, environmentally friendly and biodegradable components is essential if we are to overcome these shortcomings.

### **1.3 CLASSIFICATION OF IONIC LIQUIDS:**

The nature and strength of the cation and anion can be used to categorize the ionic liquid in the following ways.

**Acidic ionic liquids:** Ionic liquids of alkylimidazole or sulfonylalkylimidazole are acidic nature. This class has acidic cations like the ammonium, pyrrolidinium, and imidazolium ions. The  $\text{AlCl}_3$  containing ionic liquid is a well-known example of a Lewis acidic ionic liquid.<sup>11</sup>

**Basic ionic liquids:** Basic ionic liquids made from anions such as lactate, formate, acetate and dicyanamide are examples of basic anions.<sup>12-13</sup> The presence of anions gives them their solubilizing and catalytic properties. The rising number of studies demonstrating the utility of these solvents in base-catalyzed reactions has demonstrated their application.



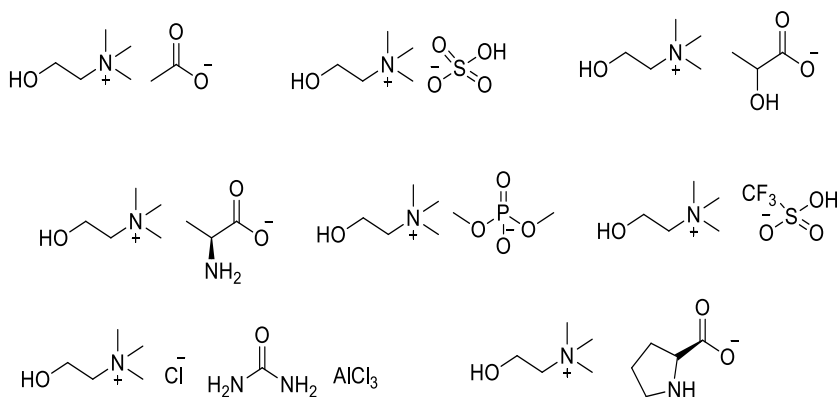
**Neutral ionic liquids:** These ionic liquids display neutrality with regard to acidity and basicity. These ionic liquid has low melting points and viscosity due weak ionic interactions with the cation. They employed as inert solvents in a wide range of applications.<sup>14</sup>

**Amphoteric ionic liquids:** There are very few ionic liquids in this class that can accept or donate protons. Amphoteric ILs include, for instance, the hydrogen sulphate and dihydrogen phosphate anions.<sup>15</sup>

#### 1.4 CHOLINE BASED IONIC LIQUIDS:

Choline is an important biodegradable, low-cost and water-soluble organic salt. In 1862, Adolph Strecker isolated it from pig and ox bile for the first time. In 1865, Oscar Liebreich synthesised choline in the laboratory.<sup>16</sup> ChCl is a B-complex vitamin that is produced in gaseous form from ethylene oxide, trimethylamine and HCl. It can also be derived from natural sources. Choline serves as a precursor molecule for the neurotransmitter acetylcholine. It is also found in cell membrane phospholipids such as phosphatidylcholine and sphingomyelin. Furthermore, choline chloride is a common feed additive supplement.

Choline-based ionic liquids have grown in popularity as a research topic recently because of its unique features and benefits, such as non-toxicity and biodegradability. They were also used to various chemical processes and sustainable, ecofriendly chemistry.<sup>17</sup>



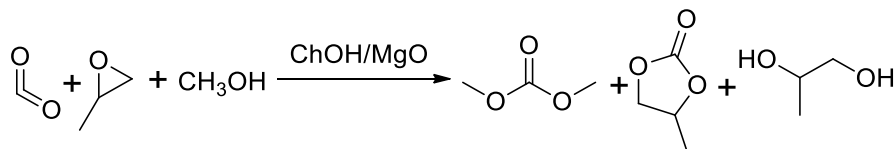
**Figure.1. Examples of choline based ionic liquids**

Ionic liquids based on choline chloride have replaced imidazole-based ionic liquids in many applications and have opened up wide

range of opportunities since they are non-toxic, biocompatible, simple to use and affordable. These are identical to typical imidazolium ionic liquids in terms of their characteristics.

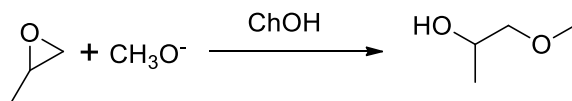
### 1.4.1 Choline based ionic liquid catalyzed organic transformations

A methodology for the synthesis of dimethyl carbonate catalyzed by choline-based ionic liquid was established by Cai et al. This biodegradable ionic liquid was used to synthesize dimethyl carbonate from carbon dioxide, methanol and propylene oxide using a multicomponent reaction.<sup>18</sup>



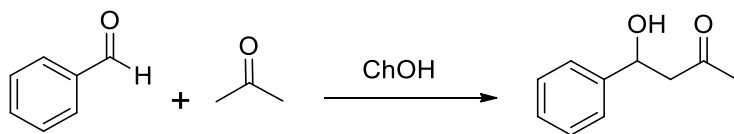
**Scheme1.** Choline hydroxide/MgO catalyzed synthesis of dimethyl carbonate

Using choline hydroxide as a low-cost basic ionic liquid, Lu et al. developed a highly efficient and selective synthesis of propylene glycol ether widely used compound in industry due to its low toxicity from methanol and propylene oxide<sup>19</sup>.



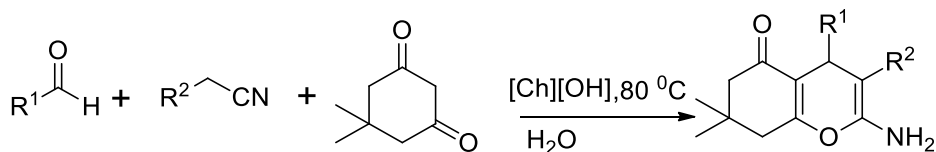
**Scheme2.** Choline hydroxide catalyzed synthesis of propylene glycol ether from methanol and propylene oxide

Hanefeld *et al.* reported an aldol reaction with high yield and selectivity in a shorter reaction time using choline hydroxide as a catalyst<sup>20</sup>.



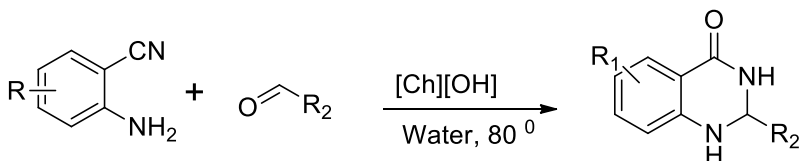
**Scheme.3 Aldol condensation catalyzed by choline hydroxide**

In 2014, Jianguo Yang and colleagues reported a method for preparation of tetrahydrobenzo [b] pyrans in presence of choline hydroxide as a catalyst with good yield and a simple work-up procedure<sup>21</sup>.



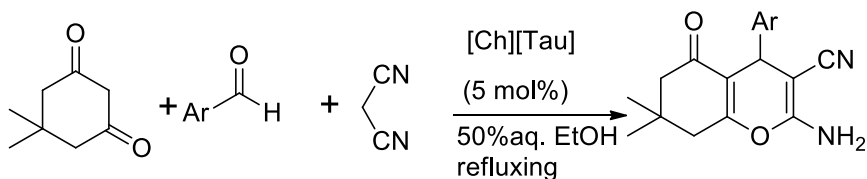
**Scheme4.** Synthesis of tetrahydrobenzo [b] pyrans using recyclable choline hydroxide

In 2016, G. S. Shankarling *et al.* reported protocol for synthesis of 2, 3-dihydroquinazolin-4(1H)-ones in presence of ionic liquid choline hydroxide (ChOH) as an effective catalyst. Desired product obtained in good to high yield with a high atom economy<sup>22</sup>.



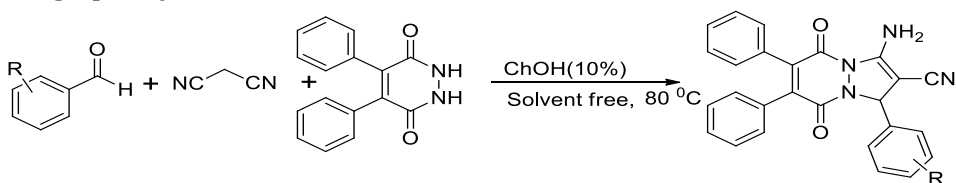
**Scheme5.** Synthesis of 2, 3-dihydroquinazolin-4(1H)-ones using recyclable choline hydroxide

Yanqing Peng *et al.* synthesized a biocompatible amino-functionalized ionic liquid from taurine and choline hydroxide. Employed this ionic liquid 4H-chromene-3-carbonitriles were prepared with good yield<sup>23</sup>.



**Scheme6.** Synthesis of 4H-chromene-3-carbonitriles using choline based ionic liquid

Ganapati S. Shankarling *et al.* demonstrated in 2020 a solvent-free preparation of novel 1H-pyrazolo[1,2-a]pyridazine-2-carbonitriles using choline hydroxide ionic liquid in shorter reaction time and excellent yield. Desired product obtained by using milder condition and in high purity<sup>24</sup>.



**Scheme7. Synthesis of pyrazolopyridazine-2-carbonitriles using recyclable choline hydroxide**

**1.5 CONCLUSION:**

Choline-based ionic liquids have emerged as a new class of biodegradable solvents with environmentally friendly properties. The extensive literature on the use of choline ionic liquids in various fields and methodologies demonstrates their potential for replacing conventional solvents and imidazolium ionic liquids. Choline-based ionic liquids serve as a green solvent and catalyst in organic transformations. Choline ionic liquids have revolutionised industrial applications due to their ease of preparation. There is still plenty of room for improvement by adding more functionality for specific applications. The use of choline-based ionic liquids is significant as a new generation of media with enormous potential.

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## Chapter -7

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### Secondary Metabolites: Biosynthesis, Classification, and Bioactivity Studies

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#### Abstract

This chapter investigates the multifaceted realm of secondary metabolites, exploring their intricate biosynthetic pathways, diverse classifications, and the fascinating spectrum of bioactivities they exhibit. Secondary metabolites, often referred to as natural products, play pivotal roles in the survival and adaptation of organisms across various kingdoms. The classification of secondary metabolites is a complex task due to the vast structural diversity and functional variations observed within this group. Here, we categorize secondary metabolites based on their chemical structures, origins, and biological roles, shedding light on the rich tapestry of compounds that nature has bestowed upon us. From alkaloids and terpenoids to polyketides and flavonoids, each class offers unique insights into the adaptability and evolution of organisms. The therapeutic potential of secondary metabolites is discussed, emphasizing their role as sources of novel drugs and bioactive compounds. Additionally, the ecological significance of these metabolites, including their roles in plant defense mechanisms and inter-species interactions, is examined.

**Keywords:** Secondary metabolites, flavonoids, terpenoids, alkaloids, antioxidants, shikimic pathways.

#### 1. Introduction:

Plants biosynthesize numerous chemicals for their physiological and ecological needs.<sup>1</sup> These compounds can be broadly classified into

primary and secondary metabolites based on their roles and significance within the plant's life processes.<sup>2-3</sup> Primary metabolites are essential for the cell's basic functions such as growth, development, and reproduction. Primary metabolites are carbohydrates, proteins, lipids, and nucleic acids. On the other hand, secondary metabolites are a diverse group of compounds that are not directly involved in the core metabolic pathways but serve various specialized functions.<sup>4</sup> In plants, secondary metabolites play a crucial role in safeguarding and defending plant cells against ecological imbalances or harmful infections.<sup>5</sup> Secondary metabolites often contribute to a plant's adaptation to environmental challenges and interactions with other organisms.<sup>6</sup> They play roles in defense mechanisms, attracting pollinators or seed dispersers, and responding to abiotic stresses. Understanding the dynamic interplay between these metabolites is essential for unraveling the complexities of plant biology and can have implications for agriculture, medicine, and ecology.<sup>7-8</sup> Click or tap here to enter text. Additionally, exploring the biosynthesis and regulatory pathways of these compounds holds promise for the development of novel applications in fields such as pharmaceuticals, nutraceuticals, and bioenergy.

**2. Biosynthesis of Secondary Metabolites:-** The biosynthesis of secondary metabolites in plants is a complex and highly regulated process involving multiple enzymatic pathways.<sup>9</sup> Unlike primary metabolites that are essential for basic cellular functions, secondary metabolites are often produced in response to specific environmental cues, and stressors, or as part of the plant's defense mechanisms. The biosynthesis of secondary metabolites can be broadly categorized into several stages, including precursor formation, the initiation of the biosynthetic pathway, and the modification of the final product. The synthesis of secondary metabolites in plants comprises intricate pathways tailored to the specific type of compound. The glycoside biosynthesis relies on the Pentose pathway, while the Shikimic acid pathway is responsible for the production of phenols. Likewise, the Mevalonic acid and acetate malonate pathways play crucial roles in the biosynthesis of steroids and alkaloids. Figure 1 provides a generalized

overview of some common pathways for the biosynthesis of three major classes of secondary metabolites: alkaloids, flavonoids, and terpenoids.

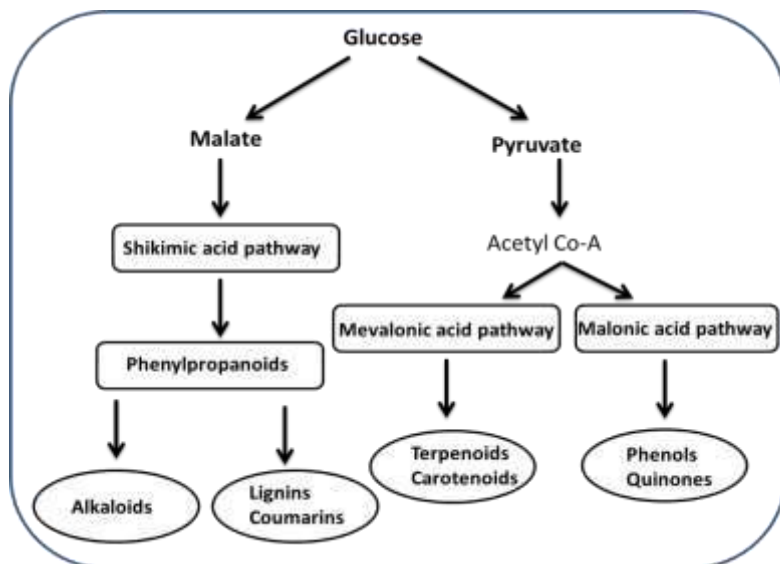


Figure 1: Plant Secondary Metabolites Biosynthesis Pathway

## 2a. Alkaloid Biosynthesis:

**Precursor Formation:** Alkaloids are often derived from amino acid precursors, such as tryptophan, tyrosine, or phenylalanine.

**Initiation:** Enzymes like decarboxylases or transaminases initiate the pathway by converting amino acids into key intermediates, such as tryptamine or tyramine.

**Formation of Alkaloid Backbone:** Additional enzymatic steps modify the backbone structure, leading to the formation of diverse alkaloids. Examples include the conversion of tryptamine to tryptophan-derived alkaloids or the synthesis of nicotine from nicotinic acid.

## 2b. Flavonoid Biosynthesis:

**Precursor Formation:** Flavonoids are derived from phenylpropanoid pathway intermediates, such as phenylalanine.

**Initiation:** Enzymes like chalcone synthase (CHS) catalyze the formation of chalcones from malonyl-CoA and 4-coumaroyl-CoA.

**Formation of Flavonoid Backbone:** Further enzymatic steps, involving enzymes like chalcone isomerase and flavonoid 3'-



hydroxylase, modify the chalcone structure to produce different classes of flavonoids, including flavones, flavonols, and anthocyanins.

## **2c. Terpenoid Biosynthesis:**

**Precursor Formation:** Terpenoids are derived from isoprenoid precursors, namely isopentenyl diphosphate (IPP) and dimethylallyl diphosphate (DMAPP), which are synthesized via the mevalonate or non-mevalonate pathways.

**Initiation:** Geranyl diphosphate (GPP) and farnesyl diphosphate (FPP) are key intermediates, formed by the condensation of IPP and DMAPP, catalyzed by enzymes like geranyl diphosphate synthase.

**Formation of Terpenoid Backbone:** Enzymes such as terpene synthases then catalyze the cyclization of GPP or FPP to form the diverse array of terpenoids, including monoterpenes, sesquiterpenes, and diterpenes.

## **3. Classification of Secondary Metabolites:**

The diversity and complexity of secondary metabolites make their classification a challenging task. In this chapter, we will explore the various ways in which secondary metabolites can be classified based on their chemical structure and biosynthetic origin. **Classification Based on Chemical Structure of Secondary Metabolites:** The following are the main classes of secondary metabolites based on their chemical structure: alkaloids, terpenoids, flavonoids, phenolics, glycosides, and saponins.

**3a. Alkaloids:** Alkaloids, a diverse group of nitrogen-containing organic compounds, are widely distributed in the plant kingdom, where they play pivotal roles in various ecological and physiological processes. The chemical structures of alkaloids are diverse, but many share common features. These compounds often contain a heterocyclic ring, frequently with a basic nitrogen atom, giving them alkaline properties. Plant-derived alkaloids have been a rich source of medicines for centuries, and many modern drugs are either directly derived from or inspired by these compounds. Morphine and codeine from opium poppies are powerful analgesics, quinine from the cinchona tree is a vital

antimalarial agent, and vincristine and vinblastine from periwinkle plants (Apocynaceae) have proven effective against certain cancers.

**3b. Terpenoids:** Terpenoids, also known as isoprenoids, constitute one of the largest and most diverse groups of natural products found in the plant kingdom. These compounds are derived from the basic five-carbon building block, isoprene, and exhibit a wide range of structures and functions. Terpenoids are classified based on the number of isoprene units they contain, leading to the formation of diverse structures. Monoterpenes (C<sub>10</sub>), sesquiterpenes (C<sub>15</sub>), diterpenes (C<sub>20</sub>), triterpenes (C<sub>30</sub>), and tetraterpenes (C<sub>40</sub>) are common classes of plant-derived terpenoids. Some of the important examples include menthol, eugenol, camphor, resins, taxol etc.

**3c. Flavonoids:** Flavonoids represent a diverse group of polyphenolic compounds found abundantly in the plant kingdom. Named after the Latin word "flavus" meaning yellow, flavonoids contribute to the vibrant colors of fruits, flowers, and vegetables. Flavonoids are characterized by a common C<sub>6</sub>-C<sub>3</sub>-C<sub>6</sub> carbon framework, consisting of two phenolic rings (A and B rings) linked by a three-carbon bridge. Major subclasses include flavones, flavonols, flavanones, flavanols (catechins), anthocyanins, and isoflavones. Flavonoids exhibit antioxidant properties, scavenging free radicals and reducing oxidative stress, which is implicated in various chronic diseases, including cardiovascular disorders and certain cancers. Studies suggest that flavonoids may also have anti-inflammatory, antiviral, and neuroprotective effects. Quercetin, quercitrin and kaempferol are the medicinally important flavonoids.

**3d. Saponins:** Saponins are a class of natural compounds widely distributed in the plant kingdom, known for their unique ability to produce foamy lather when mixed with water. Saponins are amphipathic glycosides, featuring a hydrophilic sugar moiety linked to a hydrophobic triterpene or steroid aglycone. This distinctive structure gives saponins their surfactant properties, allowing them to form stable foams. Their bitter taste and detergent-like properties deter herbivores,

while their antimicrobial activity protects the plant from various pathogens. Some saponins exhibit anti-inflammatory, antimicrobial, and immunomodulatory properties. Additionally, they have been investigated for their role in managing cholesterol levels and as potential anticancer agents.

#### **4. Medicinal Importance of Secondary Metabolites:**

##### **4a. Antimicrobial Activity**

The antimicrobial activity of secondary metabolites represents a valuable resource for addressing microbial threats in various fields. Alkaloids, flavonoids, tannins, terpenoids, and phenolic compounds found in various plant species exhibit inhibitory effects against bacteria, fungi, and viruses. For example, alkaloids like berberine and quinine, flavonoids such as quercetin, and essential oils containing terpenoids have been recognized for their antimicrobial properties. The alkaloids interfere with bacterial cell wall synthesis or disrupt membrane integrity, leading to cell death. Flavonoids and phenolic compounds may inhibit bacterial enzymes or disrupt essential cellular processes, while terpenoids can affect membrane permeability.

##### **4b. Anti-inflammatory Activity**

Secondary metabolites with anti-inflammatory properties have been studied for their ability to modulate the inflammatory response and offer potential alternatives for the development of anti-inflammatory drugs. Secondary metabolites often exert their anti-inflammatory effects by modulating key mediators of inflammation. For example, certain flavonoids and polyphenols inhibit the activity of enzymes such as cyclooxygenase (COX) and lipoxygenase (LOX), which are involved in the synthesis of pro-inflammatory eicosanoids. Terpenoids may also interfere with inflammatory signaling pathways, reducing the production of inflammatory mediators. Additionally, Studies have shown that treatment with certain secondary metabolites results in a reduction of inflammatory markers, including cytokines, chemokines, and adhesion molecules. These compounds may help regulate the expression of these molecules, contributing to the suppression of inflammatory processes.

#### **4c. Antioxidant Activity**

In recent years, the exploration of natural compounds with antioxidant properties has gained significant momentum, driven by the increasing recognition of oxidative stress as a contributor to various health conditions. Antioxidants play a crucial role in safeguarding healthy cells from the detrimental effects of oxidative stress. Factors contributing to oxidative stress include free radicals such as superoxide, hydroxyl radicals, and singlet oxygen, which generate reactive oxygen species. Flavonoids, polyphenols and carotenoids play important role as an antioxidants.

#### **4d. Anticancer Activity**

Cancer remains a global health challenge, necessitating the exploration of novel therapeutic strategies. Plant secondary metabolites have garnered significant attention for their potential anticancer properties. Alkaloids, flavonoids, terpenoids, and polyphenols derived from various plant sources, have demonstrated promising inhibitory effects on cancer cell proliferation, migration, and invasion. The mechanisms of action underlying the anticancer effects are thoroughly examined. Plant secondary metabolites exert their influence through modulation of cell cycle progression, induction of apoptosis, inhibition of angiogenesis, and interference with signaling pathways crucial for cancer cell survival. Additionally, their antioxidant properties contribute to the mitigation of oxidative stress, a hallmark of cancer progression.

#### **Conclusion**

In conclusion, this chapter serves as a comprehensive guide to understanding the biosynthesis, classification, and bioactivity of secondary metabolites. By unraveling the intricate processes governing their production, categorizing their diverse structures, and exploring their multifaceted bioactivities, we aim to contribute to the growing body of knowledge surrounding these fascinating compounds and inspire further research in the field.

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## Chapter -8

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### **The Consequences of Waste From Industries on Water Quality in the Mahad Midc Area in Raigad District, Maharashtra**

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#### **ABSTRACT**

Recently, there has been an increase in the number of publications focused on water pollution; in light of this, an assessment of ground water quality in the Mahad M.I.D.C. industrial region has been conducted. Water in this area is used for both residential and drinking purposes, so it is critical to assess its quality. Water pollution is rapidly increasing as a result of increased industrialization and urbanization. As a result, the current study focuses on this topic and seeks to validate heavy metal-induced water contamination. Water samples were taken from rivers around the industrial region on a monthly basis throughout the year. The heavy metals iron, copper, zinc, manganese, nickel, chromium, cobalt, and lead were measured using an Atomic Absorption Spectrophotometer. The findings of this investigation imply that the concentration of a few heavy metals in this area is unexpectedly growing.

**KEYWORDS:-**Water, heavymetal, Pollution.

#### **1. INTRODUCTION**

Metal pollution in water results from both human and natural sources. Certain metals, such as cobalt (Cd), copper (Cu), zinc (Zn), and selenium (Se), are beneficial to humans, but high levels have a negative impact on living creatures. Metals including cadmium (Cd), chromium (Cr), and lead (Pb) are extremely poisonous even at low concentrations [1]. Heavy metals such as Hg, Cd, Pb, and Cr have been discovered to be highly harmful contaminants. Human activities are mostly responsible for the introduction of these metals into water as pollutants. These metals accumulate in the bodies of animals and humans, and they may cause serious disorders such as cancer [2]. Water quality assessment

and monitoring are critical issues for the survival of all living organisms, particularly river water, which is used by humans [3]. Pollution of these sources by diverse anthropogenic activities has a negative impact on both the ecosystem and human health[4-5].The disposal of industrial wastewater is a severe environmental concern. Many chemical processes in industry produce waste including pollutants such as heavy metals. According to the World Health Organization (WHO 1984), heavy metals such as chromium, zinc, iron, mercury, and lead are the most acute concerns. The maximum allowable limit for contaminants in treated waste water is imposed in numerous developed and developing nations. Problems pertaining to soil, water, and natural ecosystem pollution are widely documented [6].Of all the substances thought to harm the aquatic environment, heavy metals are one that are gradually enriched and deposited in biological chains that impact human health[7-9]. Heavy metals originating from these man-made sources pose a significant threat to both human health and the environment because of their toxicity, bioaccumulation, abiotic degradation, and persistence [10-14]. Anthropogenic activities, industrial waste, weathering, municipal trash, household garbage, and atmospheric deposition are some of the possible causes of heavy metal contamination in rivers[15].Heavy metal pollutants are widely dispersed into the water. Wherever streams and rivers lead, industrial regions are the main sources of these. Diminished levels of heavy metals in water have severe effects on the environment, gradually altering the organic chemical balances of plants and animals as they accumulate. Certain organic chemical processes are inhibited or catalytically lead to undesirable directions during thermogenesis. As a result, many organisms will essentially suffer harm and change their natural surroundings. As a result, accurate information regarding the presence of hazardous metals in water is crucial for environmental concerns [16].The study's goal is to evaluate the level of pollution in surface water sources near a significant industrial area in MIDC, Mahad, Raigad, Maharashtra, western Ghats of India. The area is experiencing extensive industrialization and is producing a large number of pollutants that are seriously harming both humans and animals living there.

## 2. MATERIALS AND METHODS

This study set out to determine the level of heavy metal contamination in the groundwater surrounding the study area, Madhava, which is situated on the Arabian Sea in the coastal Kokan region of Maharashtra, south of Mumbai. Latitude 18°6'12"N and longitude 73°28'40"E are the geographic coordinates of the chosen area. The elevation above mean sea level (in meters) is approximately 177.5m. The purpose of the analysis is to determine whether factors like sample location, depth, pH, conductivity, and population burden affect the presence of heavy metal contents and concentration. A total of fifteen (15) water samples were collected from various river places between August 2017 and July 2018. Water samples were collected in empty polythene bottles that were properly labeled to prevent any chance of contamination. Every site was sampled twice, with one sample (50 ml) being combined with 4 ml of HNO<sub>3</sub> (nitric acid) to preserve the sample (APHA, 1992). The sample analyses were completed in accordance with APHA guidelines [17].

## 1. RESULTS AND DISCUSSION

**Table 1.** Month wise mean of metal concentration of heavy metals from different sample stations.

Heavy metal	August	Sept	Oct	Nov	Dec	Jan	Feb	March	April	May	June	July
Fe(ppm)	0.072	0.069	0.072	0.067	0.073	0.077	0.083	0.089	0.072	0.055	0.085	0.07
Zn(ppm)	0.045	0.05	0.05	0.049	0.051	0.049	0.049	0.047	0.05	0.047	0.047	0.042
Cu(ppm)	0.017	0.057	0.074	0.063	0.064	0.076	0.0787	0.079	0.013	0.031	0.024	0.015
Mn(ppm)	0.03	0.022	0.01	0.012	0.02	0.032	0.034	0.037	0.077	0.088	0.04	0.032
Ni(ppm)	0.042	0.035	0.029	0.024	0.031	0.03	0.034	0.034	0.053	0.052	0.055	0.044
Cr(ppm)	0.006	0.012	0.025	0.027	0.024	0.031	0.033	0.037	0.046	0.042	0.026	0.021
Co(ppm)	0.008	0.001	0.003	0.003	0.007	0.004	0.005	0.006	0.002	0.011	0.007	0.001

### Iron

Throughout the year, the mean value of the iron concentration in surface and ground water is 0.073 ppm, with variations ranging from 0.055 to 0.089 ppm. The high iron content found in the research area's red soil may be the cause of the water's iron concentration. It was discovered that the content did not meet WHO (2008) drinking water recommendations. It is in line with the information gathered by Manoj Kumar dev et al. (2017) during their investigation of water analysis close to the Lote industrial sector in Maharashtra [18].



### **Zinc**

Zinc's content varied between 0.042 and 0.051 ppm, with a mean value of 0.047 ppm. The maximum concentration in this analysis for the month of December was 0.051 ppm. Given that the majority of the industries in this area are chemical ones, it's possible that zinc is frequently used as a catalyst for certain chemical processes. The acquired result aligns with the findings of S. Jabeen et al., which indicate fluctuations in zinc concentration throughout water analysis [19].

### **Copper**

A valuable metal found in trace amounts among the heavy metals is copper. Humans and aquatic life are harmed when Cu concentrations rise over allowable levels. The research area's Cu content was determined to be between 0.013 and 0.079 ppm (mean: 0.049 ppm). The month of March had the highest concentration of copper; this could be because chemists use copper more frequently during this time of year. This finding corroborates earlier research by S. Jabeen et al. [19], which connected copper consumption in the chemical industry. Similarly, Rachna Virha et al. discovered that the dumping of trash from surrounding hospitals and companies increased the concentration of copper in the water[20].

### **Manganese**

The highest concentration of Mn (0.088 ppm) was found to exceed the European Union's drinking water quality criteria in the month of May, and the lowest concentration (0.01 ppm) was determined to be below the permitted limit of the water quality regulations set by the EU. This finding adds credence to the theory put up by Manoj Kumar Dev et al. regarding water pollution brought on by anthropogenic activities and industrialization[18].

### **Nickel**

Nickel concentrations vary from 0.024 to 0.055 ppm. The month of June was found to have the highest concentration. The highest figure that was discovered to be over the water quality requirements set by the European Union.

## **Chromium**

The chromium concentration was determined to be between 0.006 to 0.046 ppm. This is below the WHO (2008) and EU (1998) criteria.

## **Cobalt**

The average cobalt concentration in the water ranged between 0.0008 and 0.011 ppm.(Mean = 0.0042 ppm). These findings support the theories of Buyyan, who proposed a link between water pollution and industrialization [19].

## **3. CONCLUSION**

Industrialization and urbanization in the Mahad area during the last decade have been the key causes of the rapid increase in heavy metal concentrations. The study region was divided into sections based on the variation in location surrounding the MIDC area. The results reveal that the fluctuation in metal concentration is not consistent from east to west, as water flows toward the western zone to reach the sea. It should be mentioned that the increasing concentration of metals in the area is the result of industrialization. Furthermore, sample stations influenced by industrial discharge reveal higher metal concentrations. During the study, potential metal contamination such as Cu, Cr, and Ni was found. Metal concentrations above the legal limit may harm animals and plants that come into touch with polluted water. In summary; the current study provides background information for interpreting fluctuations in heavy metal concentrations in water and around the Mahad MIDC area. The data reported here can also be used to monitor water quality in the future surrounding the research region.

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## Chapter -9

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### **A Green Approach for one pot synthesis of polyhydroquinolines using Bael Fruit Extract as a catalyst under solvent free conditions**

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#### **Abstract:**

Using the four-component coupling reaction of aldehydes, dimedone, ethyl acetoacetate, and ammonium acetate in the presence of Bael fruit extract as a green catalyst under solvent-free conditions is a good method for synthesizing polyhydroquinoline derivatives.

**Keywords:** *Dimedone, Bael fruit extract, Hantzsch reaction.*

#### **Introduction:-**

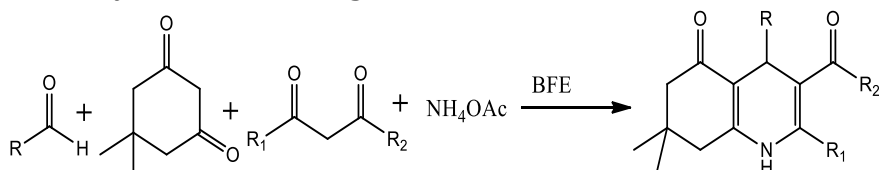
4 Substituted 1,4 dihydropyridine (1,4-DHP) nucleus is a fertile source of biologically important molecules possessing various important pharmacological properties such as vasodilator, antihypertensive, bronchodilator antithrombotic, hepto-protective, antitumor, antimutagenic, geroprotective and antidiabetic agents<sup>1-4</sup>. From recent studies 1-4 DHP shows several medicinal applications which include neuroprotectant and platelet anti-aggregatory activity, in addition cerebral antiischemic activity in the treatment of Alzheimer's disease<sup>5-7</sup>.

A four-component coupling reaction including aldehydes, dimedone, ethyl acetoacetate, and ammonium acetate in the presence of HClO<sub>4</sub>-SiO<sub>2</sub> under solvent-free conditions at 90°C temperature produced an effective Hantzsch condensation polyhydroquinoline derivatives<sup>8</sup>. Through the Hantzsch reaction of aldehydes, dimedone, ethyl acetoacetate, and ammonium acetate at room temperature, Yb(OTf)<sub>3</sub> facilitated the excellent yield of one pot synthesis of polyhydroquinoline derivatives<sup>9</sup>. Numerous studies have been conducted on the photocatalytic oxidation of 1,4 dihydropyridine to pyridine<sup>10</sup>. In this work, a new, effective technique for the solvent-free

synthesis of polyhydroquinoline employing Bael fruit extract catalyst was devised.

### Material and methods:

A mixture of aldehyde (1mmol), dimedone (1mmol), ethyl acetoacetate (1mmol), ammonium acetate (1.5mmol), Bael fruit extract (4 ml) were refluxed under solvent free conditions. The reaction was confirmed by thin layer chromatography, the resulting solid product was treated with EtOAc followed by water and a brine solution and dried with anhydrous Na<sub>2</sub>SO<sub>4</sub>. The solution was concentrated in vacuum to afford the crude product. The pure product was obtained by further recrystallization using absolute alcohol.



**Table: Bael fruit extract catalyzed Hantzsch condensation for synthesis of polyhydroquinolines derivatives under solvent free conditions.**

Entry	R	R1	R2	Time (min.)	Product	Yield	Melting point (°C) Observed	Melting point (°C) Reported
1	C <sub>6</sub> H <sub>5</sub>	CH <sub>3</sub>	OEt	23	2a	82	203-204	202-204 <sup>9</sup>
2	4-F-C <sub>6</sub> H <sub>4</sub>	CH <sub>3</sub>	OEt	25	2b	84	185-186	184-186 <sup>9</sup>
3	4-OCH <sub>3</sub> -C <sub>6</sub> H <sub>4</sub>	CH <sub>3</sub>	OEt	20	2c	81	256-257	257-259 <sup>9</sup>
4	4-CH <sub>3</sub> -C <sub>6</sub> H <sub>4</sub>	CH <sub>3</sub>	OEt	28	2d	85	261-262	260-261 <sup>9</sup>
5	3-NO <sub>2</sub> -C <sub>6</sub> H <sub>4</sub>	CH <sub>3</sub>	OEt	27	2e	80	178-179	177-178 <sup>10</sup>

The structure of the product was determined from their spectroscopic (UV, IR, NMR, Mass) data.

#### 1. Spectroscopic data:

**Ethyl - 1, 4, 7, 8 - tetrahydro - 2, 7, 7 - trimethyl-4-(4-fluorophenyl)-5(6H)-oxoquinolin-3-carboxylate**

**(2b).** Yellow solid, mp 185-186 °C. IR (KBr): 3292, 2959, 1696, 1649, 1608, 1487, 1380, 1219, 1025, 764 cm<sup>-1</sup>. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ = 0.92 (s, 3H, CH<sub>3</sub>), 1.07 (s, 3H, CH<sub>3</sub>), 1.18 (t, *J* = 7.3 Hz, 3H, CH<sub>3</sub>), 2.13-2.25 (m, 4H, 2 ×CH<sub>2</sub>), 2.38 (s, 3H, CH<sub>3</sub>), 4.05 (q, *J* = 7.33 Hz, 2H, CH<sub>2</sub>), 5.02 (s, 1H, CH), 5.8 (s, 1H, NH), 6.85-6.89 (m, 2H, ArH), 7.23-7.27 (m, 2H, ArH). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 14.1, 18.2, 26.4, 29.0, 32.1, 35.2, 50.1, 50.3, 59.0, 103.4, 110.0, 114.2, 114.3, 114.4, 129.0, 129.1, 144.1, 145.1, 149.4, 169.8, 194.2. LCMS: *m/z* = 356 (M-H)<sup>-</sup>. Anal. Calcd for C<sub>21</sub>H<sub>24</sub>NO<sub>3</sub>F: C, 70.58; H, 6.72; N, 3.92; F, 5.32. Found: C, 70.52; H, 6.79; N, 3.87; F, 5.28.

**Ethyl - 1, 4, 7, 8 -tetrahydro-2,7,7-trimethyl-4-(4-methoxyphenyl)-5(6H)-oxoquinolin-3-carboxylate**

**(2c).** Yellow solid, mp 256-257 °C. IR (KBr): 3276, 2956, 1703, 1648, 1606, 1496, 1381, 1215, 1031, 765 cm<sup>-1</sup>. <sup>1</sup>H NMR (200 MHz, CDCl<sub>3</sub> + DMSO-d<sub>6</sub>): δ = 0.95 (s, 3H, CH<sub>3</sub>), 1.09 (s, 3H, CH<sub>3</sub>), 1.21 (t, *J* = 7.2 Hz, 3H, CH<sub>3</sub>), 2.01-2.10 (m, 4H, 2 ×CH<sub>2</sub>), 2.30 (s, 3H, CH<sub>3</sub>), 3.70 (s, 3H OCH<sub>3</sub>), 4.00 (q, *J* = 7.2 Hz, 2H, CH<sub>2</sub>), 4.80 (s, 1H, CH), 6.65 (d, *J* = 7.3 Hz, 2H, ArH), 7.10 (d, *J* = 7.3 Hz, 2H, ArH), 8.65 (s, 1H, NH). <sup>13</sup>C NMR (75 MHz, DMSO-d<sub>6</sub>) δ 14.1, 18.2, 26.4, 29.1, 32.1, 34.7, 50.2, 50.5, 54.8, 58.9, 103.2, 110.1, 113.0, 113.1, 128.2, 128.3, 139.8, 144.6, 149.1, 157.2, 166.9, 194.2. LCMS: *m/z* = 368 (M-H)<sup>-</sup>. Anal. Calcd for C<sub>22</sub>H<sub>27</sub>NO<sub>4</sub>: C, 71.54; H, 7.31; N, 3.79; Found: C, 71.59; H, 7.35; N, 3.84.

**2. Result and Discussion:**

Polyhydroquinoline derivatives are traditionally prepared by reacting aldehyde with ethyl acetoacetate and ammonia, either in acetic acid or by refluxing in alcohol. Unfortunately, there are a number of issues with this technique, including a lengthy reaction time, an excess of organic solvent, and a reduced product yield<sup>11</sup>. Aldehyde, dimedone, ethyl acetoacetate, and ammonium acetate in a 1:1:1:1 mixture are all starting ingredients. The reaction is green and solvent-free when it is catalyzed by Bael fruit extract catalyst.

**3. Conclusion:**

Finally, we reported on the recyclable and reusable nature of Bael fruit extract as a green catalyst for the Hantzsch reaction. Several

effective aspects of this approach include the experiment's simplicity, mild reaction conditions, good yield, quick reaction time, and ease of setup. Therefore, this method's simplicity and clarity make it appealing for the synthesis of derivatives of polyhydroquinolines.

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## Chapter -10

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### Importance of Trace Elements and Some Bulk Elements for Betterment of Human Health

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#### INTRODUCTION:-

Trace elements refer to any chemical element or minerals that are present in the body in very small amounts usually less than 0.1% by volume. There are different types of trace elements in the human body. The nutritionally essential elements are required for proper physiological and metabolic functions, At least 21 trace elements have been described in the human body and each of the trace elements has different roles and functions within the body, and a deficiency or excess may lead to various clinical manifestations and affect one's growth and development. In addition to the nutritionally essential trace elements, there are a variety of probably essential elements and potentially toxic elements. Most trace elements can be classified as nutritionally essential, probably essential, or potentially toxic. The probably, nutritionally essential trace elements include iron, copper, cobalt, zinc, selenium, chromium, iodine, and molybdenum.

Meanwhile, the potentially toxic elements include fluoride, lead, cadmium, mercury, arsenic, aluminium, lithium, and tin.

#### What are trace elements in nutrition?

Nutritionally essential trace elements are required parts of an individual's nutrition. These elements contribute to vital bodily functions, including metabolic function, tissue repair, growth, and development. Because the human body cannot naturally synthesize these elements, it is essential that people consume them through their diet or by using supplements.



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Excess consumption of these elements can have potentially toxic effects. Nutritionally essential trace elements include iron, copper, cobalt, zinc, selenium, chromium, iodine, and molybdenum.

**BACKGROUND**

- ANY CHEMICAL ELEMENT PRESENT in the HUMAN BODY in VERY SMALL AMOUNTS  
- USUALLY < 0.1% by VOLUME
- CLASSIFICATIONS: NUTRITIONALLY ESSENTIAL, PROBABLY ESSENTIAL, or POTENTIALLY TOXIC

**EXAMPLES**

<b>CHROMIUM (Cr)</b> METABOLIC FUNCTION	<b>IRON (Fe)</b> OXYGEN TRANSPORT	<b>COPPER (Cu)</b> RED BLOOD CELLS, ENZYMATIC FUNCTIONS	<b>SELENIUM (Se)</b> METABOLIC FUNCTION (ANTIOXIDANT)
<b>MOLYBDENUM (Mo)</b> DIGESTION & EXCRETION	<b>COBALT (Co)</b> ORGANIC; AMINO ACIDS & NEUROTRANSMITTERS	<b>ZINC (Zn)</b> CELLULAR & IMMUNE SUPPORT, METABOLIC FUNCTION	<b>IODINE (I)</b> THYROID HORMONES

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### TRACE METAL AND SOURCES

Sr. No	Metal	Sources of metals
1.	Na (1.8 g)	❖ Salty foods, milk, baking soda, baking powder, some vegetables
2.	K (2.6 g)	❖ Vegetables, fruits, milk, meat, whole grains, legumes
3.	Mg (0.5 g)	❖ Chlorophyll, nuts, legumes, whole grains
4.	Ca (22 g)	❖ Milk, milk products, fish bones
5.	Fe (75 mg)	❖ Liver, meat, egg yolk, green leafy vegetables, whole grains, bread, cereals
6.	Cu (2 mg)	❖ Liver, kidney, egg yolk, whole grains
7.	Se ( 2mg)	❖ Seafood, organ meats, and Brazil nuts
8.	Zn (28 mg)	❖ Liver, pancreas, meat, shell fish, milk, nuts, legumes.
9.	Co (3 mg )	❖ Liver, pancreas mushrooms.
10.	Mo (5 mg )	❖ Liver, kidney, whole grains, legumes, leafy vegetables
11.	Cr (5 mg)	❖ Grape juice, Broccoli, Turkey breast, English muffin, mashed, Green beans

### IRON (Fe)

Iron plays an important role in transporting oxygen throughout the body through the blood. Iron deficiency can lead to anemia (i.e., deficiency of healthy red blood cells) and has also been linked to upper alimentary tract cancers.

- It helps the oxygenated throughout blood and making haemoglobin, a protein in red blood cells
- It also plays an important role in metabolism as a component of some proteins and enzymes in converting blood sugar to energy
- It boosts the immune system that is necessary for immune cells proliferation and maturation, particularly lymphocytes, associated with the generation of a specific response to infection
- It aids cognitive function; supplementation benefitted the intelligence and memory of anemic school-age children, who are at higher risk of impaired cognitive development
- It supports healthy skin, hair and nails. Iron supports enzyme systems that are involved in the synthesis of collagen and elastin

### **COPPER (Cu)**

The third most abundant trace element in the human body works with iron to form healthy red blood cells and is an essential component of many enzymes involved in chemical reactions throughout the body. It also plays an important role in maintaining the strength and health of blood vessels, nerves and bones. Copper contributes to many central processes in the human body, thereby augmenting both physical fitness and mental wellness. Some of the vital functions of copper include: Adjoining with proteins to form metalloenzymes known as cuproenzymes, to facilitate energy production, neuron activation and synthesis of bones, connective tissues. Incorporating with bioactive constituents in the bloodstream to result in ceruloplasmin (CP), a carrier and transport protein, which is crucial for iron metabolism, distribution and absorption in bodily cells, tissues Integrating as a key component of superoxide dismutase enzymes, which portray powerful antioxidant traits, for safeguarding cells from oxidative damage by toxins, harmful free radicals. Carrying out the vital role of maintaining the body's structural proteins – collagen and elastin, which enrich skin texture and elevate heart wellness. Helping strengthen bones, joints and increasing bone mineral density, to lower the risk of debilitating illnesses like osteoporosis and arthritis. Regulating blood pressure, blood sugar and cholesterol levels, by ensuring proper glucose, lipid metabolism and decreasing the chances of acquiring chronic conditions of hypertension, diabetes. The preserving smooth relay of signals between nerves to ensure optimal brain functions, and responses across all organs in the body. Controlling fundamental biological processes of new blood vessel formation or angiogenesis, gene expression in cells, tissues, neurological development, neurohormone production and pigment compounds i.e. melanin secretion. Contributing to adequate white blood cells within the system, to support immunity, keep seasonal illnesses and infectious diseases due to bacteria and viruses.

### **COBALT (Co)**

It can be found in organic and inorganic forms. In the organic form it forms a vital part of vitamin B12 (i.e., cobalamin) and

contributes to the formation of amino acids and neurotransmitters. Conversely, inorganic forms of cobalt can be toxic to the human body. Cobalt is an integral part of vitamin B12 and therefore essential for the function of cells. It is also involved in the production of red blood cells and the production of antibacterial and antiviral compound that prevent infections. Cobalt also plays a key role in the metabolism of fats and carbohydrates as well as the synthesis of protein and conversion of folate in their active form. In the nervous system, cobalt is responsible for preventing demyelination leading to multiple sclerosis, which is condition that results in damage to the membrane that covers the nerve fibers in the brain and spinal cord such prevention ensures the efficient transmission of nerve impulses.

### **ZINC (Zn)**

Contributes to many functions in the body but is most importantly associated with cell division, cell growth, tissue repair, and metabolic function. It also aids the immune system in fighting off viruses and bacteria. Zinc is necessary for carrying out many primary functions in the body such as:

- Bolstering immunity.
- Enhancing nervous system function, brain activity, memory and concentration.
- Lowering inflammation in certain conditions in the body like acne
- Accelerating wound healing process, in instances of tissue injury
- Preventing chronic diseases in old age, like diabetes, heart disease and age-related macular degeneration, by fostering healthy ageing process
- Preserving a normal sense of taste and smell
- Promoting optimal cell growth, division as well as DNA and protein synthesis

### **SELENIUM (Se)**

Selenium isplays an important metabolic role as an antioxidant (known to prevent or reduce damage caused by oxidation in the body). Chromium also contributes to metabolic function, as it plays a key role in regulating sugar, fat and protein levels in the blood. Being a crucial

constituent of many biochemical complexes in cells and tissues, selenium carries out various bodily tasks by means of more than two dozen selenoproteins that control DNA synthesis. DNA i.e. Deoxyribonucleic Acid being the fundamental genetic material that influences the formation and function of cells, tissues and organs, selenium is highly essential for conserving overall health of the body. Furthermore, selenium is involved in many significant processes, such as: Promoting the cognitive abilities, memory, concentration, thinking, by positively influencing brain and nerve cell operations. Preserving reproductive wellness, fertility in both men and women. Safeguarding thyroid gland from undergoing oxidative stress and increasing the synthesis of thyroid hormones, for optimal growth, development, metabolism and immunity. Averting the onset of chronic ailments like cancer, diabetes and Alzheimer's disease, by scavenging harmful free radicals circulating in the bloodstream. Contributing noteworthy antioxidant properties to flush out toxins in the system and lower the risk of heart disease, stroke. Decreasing volumes of harmful inflammatory compounds and raising levels of antioxidant glutathione peroxidase in the body, thereby augmenting cardiac functions and heart wellness. Enhancing lung power and lessening breathing problems, respiratory distress experienced in asthma.

### **CHROMIUM (Cr)**

Chromium is an essential trace mineral with various benefits, including improving insulin sensitivity and enhancing protein, carbohydrate, and lipid metabolism. It is a metallic element that people need very small quantities. There is limited information about the exact amount of chromium required, and what it does, as studies have so far produced conflicting results.

Recent results suggest Trusted Source that chromium picolinate supplements may have benefits for some people, but experts recommend diet, rather than supplements, as the best source of chromium. Chromium is a mineral that humans need in very small quantities. Good sources include broccoli, liver, and brewers' yeast. Chromium supplements may enhance muscle mass, weight loss, and glucose control, but researchers are still working to confirm this.

Nutritional supplements are like medications and those considering taking supplements should use them with caution. Healthful food is the best and safest source of nutrients.

### **IODINE (I)**

Iodine is a very important element within the thyroid hormones thyroxine (T<sub>4</sub>) and triiodothyronine (T<sub>3</sub>). These hormones are essential in metabolism, growth, and development of the human body. Iodine is involved in several critical functions in the human body, such as: Maintaining normal synthesis and operation of thyroid hormone, by catalyzing the conversion of thyroid stimulating hormone (TSH) into triiodothyronine (T<sub>3</sub>) and thyroxine (T<sub>4</sub>), thus uplifting immunity, heart wellness and metabolism. Preventing the incidence of hypothyroidism, i.e. an underactive thyroid gland. Ensuring optimal neural development in the growing fetus in pregnancy. Lowering the risk of goitre, which results in enlargement of the thyroid gland? Promoting memory, concentration, intelligence, rational thinking and myriad other brain operations. Averting occurrence of thyroid cancers and other autoimmune conditions like Graves' disease.

### **MOLYBDENUM (Mo)**

Molybdenum is required for a few enzymatic functions involved in digestion and excretion. Excited Mo character in scrubs, Mo is the only element of second and third transition series known to be essential to life. It is present in all plant and animal tissues. Largest amounts are found in liver, kidney, bone and skin.

- Mo is absorbed as molybdate  $\text{MoO}_4^{2-}$  ion, Mo enzymes:
- Nitrogenase enzyme : biological  $\text{N}_2$  fixation
- Nitrate reductase: catalyses reduction of  $\text{NO}_3^-$  to  $\text{NO}_2^-$  the first step for assimilation of inorganic  $\text{N}_2$ .
- Xanthine oxidase, (catalyses oxidation of Xanthine to uric acid, key step in purine metabolism).
- Xanthine dehydrogenase
- Aldehyde oxidase: conversion of aldehydes to carboxylic acids.
- Sulphite oxidase: oxidation of  $\text{SO}_3^{2-}$  to  $\text{SO}_4^{2-}$

## **SODIUM (Na)**

Sodium is an alkali metal that plays many important roles in the human body. It helps cells transmit nerve signals, regulates water levels in tissues, and maintains the proper balance of water and minerals. Sodium is also used to regulate blood pressure and blood volume. Sodium plays a key role in normal nerve and muscle function. The human body requires a small amount of sodium to conduct nerve impulses, contract and relax muscles, and maintain the proper balance of water and minerals. Sodium is an essential nutrient involved in the maintenance of normal cellular homeostasis and in the regulation of fluid and electrolyte balance and blood pressure (BP). When the concentration of sodium in the blood is abnormally low, it's called hyponatremia.

The recommended daily intake of sodium for healthy adults is 2,300 mg per day. Adults with high blood pressure should further limit salt quantities to less than 1,500 mg per day.

- Na<sup>+</sup> ions are highly mobile and are present in largest concentration in body fluids.
- Concentration is highest in the extra-cellular fluids – plasma & intestinal fluids.
- Na<sup>+</sup> ions maintain normal hydration & osmotic pressure.
- Concentration of Na<sup>+</sup> in extra-cellular fluids regulates the flow of water across the cell membrane.
- Na<sup>+</sup> ions are involved in the processes by which foods & cellular building blocks – glucose & amino acids are transported into the cells.
- This transport takes place by a pumping action – Na<sup>+</sup> out and K<sup>+</sup>, glucose, amino acids in.

## **POTASSIUM (K)**

Potassium is an important mineral nutrient in the human diet. We need potassium because its roles in the human body include fluid regulation, muscle contraction and regulation of nerve signals. What are some effects of potassium on health? Scientists are studying potassium to understand how it affects health. Here are some examples of what this research has shown. High blood pressure and stroke, Bone health Kidney stones, Blood sugar control and type 2 diabetes.

- K<sup>+</sup> ions are the most abundant cations in the intracellular fluids. High concentration is needed for the synthesis of enzymes and proteins which control the chemical reactions of the cell.
- Active transport mechanism, Oxidation of glucose.
- Transmission of nerve signals.
- Heart is particularly sensitive to K<sup>+</sup> ions exert relaxing effect on heart muscle.
- Hypopotassemia (low K) –Hyperpotassemia- less common.
- There are alterations in electrocardiogram (ECG) and distinct histological changes in myocardium.

### **MAGNESIUM (Mg)**

Magnesium is the energy mineral; Magnesium (chemical symbol Mg) is an alkaline earth metal and one of the ten most common elements on the planet. It occurs naturally as a bivalent cation, usually in the form of inorganic salts, including carbonate such as magnesite(MgCO<sub>3</sub>) and dolomite (CaMg[CO<sub>3</sub>]<sub>2</sub>) as well as magnesium sulphate (MgSO<sub>4</sub>) and magnesium chloride (MgCl<sub>2</sub>).

In foods, magnesium is more commonly included in compound form, associated with proteins or in the form of salts. Magnesium belongs to the group of essential bulk elements. The human body contains 11.5-16.5 mmol of magnesium/kg of body weight. For a 70 kg adult, this equates to a quantity of around 24 g. Roughly 60-65 % of the body's total stores are found in the bones, 27 % in the musculature. The remaining 6-7 % are distributed amongst other soft tissue (liver, kidneys and heart). Only around 1 % are allotted to plasma (normal range in serum: 0.8-1.2 mmol/l), of this 53 % are present in ionised free form; the rest are complexed or protein-bound. In the human organism, magnesium is present exclusively as a bivalent cation (Mg<sup>2+</sup>) and fulfils a variety of functions as a nutrient and active ingredient.

Magnesium play an important role for, Bones and masticatory system, Enzyme catalysis, Ion transport, Magnesium is a physiological opponent (antagonist) of calcium. A latent magnesium deficit is comparably common; in around 50 % of cases, a cellular magnesium deficiency is present in often normal serum or plasma values.



## **Calcium (Ca)**

Calcium is the most abundant mineral in the body. Calcium is a nutrient that all living organisms' need, why we need calcium, Calcium plays various roles in the body. These include the following: Around 99% of the calcium in the human body is in the bones and teeth. Calcium is essential for the development, growth, and maintenance of bone. Calcium helps regulate muscle contraction, cardiovascular system and other roles.

Calcium is a co-factor for many enzymes. Without calcium, some key enzymes cannot work efficiently. Studies have also suggested that consuming enough calcium can result in:

- A lower risk of developing conditions involving high blood pressure during pregnancy
- Lower blood pressure in young people
- Lower blood pressure in those whose mothers who consumed enough calcium during pregnancy
- Improved cholesterol values
- A lower risk of colorectal adenomas, a type of non-cancerous tumor

## **CONCLUSION**

Trace elements function primarily as catalysts in enzyme systems; some metallic ions, such as iron and copper, participate in oxidation-reduction reactions in energy metabolism. Iron, as a constituent of haemoglobin and myoglobin, also plays a vital role in the transport of oxygen. The function of each elements or acts as a vital role, Calcium – the bone mineral, Magnesium – the energy mineral, Iron – the elixir of life for an “steely” health, Zinc – spark plug of the metabolism, Selenium – the trace element for cell, Iodine – the thyroid element and so on *etc.*

Trace elements are vital for human body to maintain normal yet complex physiological functions including metabolic function, tissue repair, related to body's growth & development. The trace minerals are just as vital to our health as the major minerals, but we don't need large amounts. The trace elements are participating for metabolism of proteins, carbohydrates, lipids, and energy and synthesis of some hormones and connective tissue.

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## Chapter -11

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### A New Versatile Catalytic Material Coal Fly Ash

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#### Introduction:

#### Coal fly ash

Coal fly ash is waste product of coal combustion in a coal fired power stations. Large quantities of coal fly ash are produced in electric power plants throughout the world every year. Efficient disposal of coal fly ash is a worldwide issue because of the huge amount produced and its harmful effects on the environment. It is estimated that the world production of fly ash generated is around 500 metric tonnes and will continue to grow in the future. Current practice of fly ash disposal is to dump it into the sea or into landfills. The major environmental concern of the dumped fly ash is the possibility of the leaching of metals and organic compounds and their migration into groundwater or nearby surface water. In addition, the airborne particulates from fly ash could also affect human health through direct inhalation. More importantly, the use of land and its maintenance results in a long-term financial burden. Thus in recent years, there is an increasing interest in the bulk utilization of fly ash. In the past two decades, a great deal of effort has been conducted for the application of fly ash as value-added products. These major applications include building and construction materials such as cement, geopolymers, ceramic materials, soil amendment, zeolite synthesis, low cost adsorbents for gas and water cleaning, nuclear waste stabilization and material recovery[1-6]. Except the utilization in construction materials, no other applications have been put into practice. Even though the civil engineering application could not catch up with the generation rate of fly ash, leaving large quantities of the waste dumped in landfills. It is estimated that worldwide recycling of fly ash is only about 25% of fly ash generated each year.

It is known that coal fly ash is a silica - aluminates material, the major chemical constituents of coal fly ash are  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$  and  $\text{Fe}_2\text{O}_3$  (

60-70 Wt %, 16-20 Wt% and 6-7 Wt % respectively). After high temperature combustion, these oxides are formed with high thermal stability, which results in fly ash having good catalyst support. In addition, minor components of other metal oxides such as TiO<sub>2</sub>, CaO, MgO, K<sub>2</sub>O, and Na<sub>2</sub>O could also be used as effective catalyst components. It is noted that fly ash also contains some trace elements such as Hg, As, Ge, Ga and traces of heavy metals (Cr, Co, Cu, Pb, Mn, Ni and Zn) and rare earths and minerals like mullite, hematite, magnetite, ferrite and rutile. For many catalyst systems, transition metal oxides are the active parts, and alkali or alkaline earth metal oxides are catalyst promoters. It is deduced that fly ash could be employed as catalyst and catalyst support for many catalytic reactions.

Utilization of fly ash for other industrial applications provides a cost effective and environmentally benign way of recycling this solid waste, to reduce detrimental effects on environment.

### **Characteristics of fly ash:**

#### **a) Classification of fly ash**

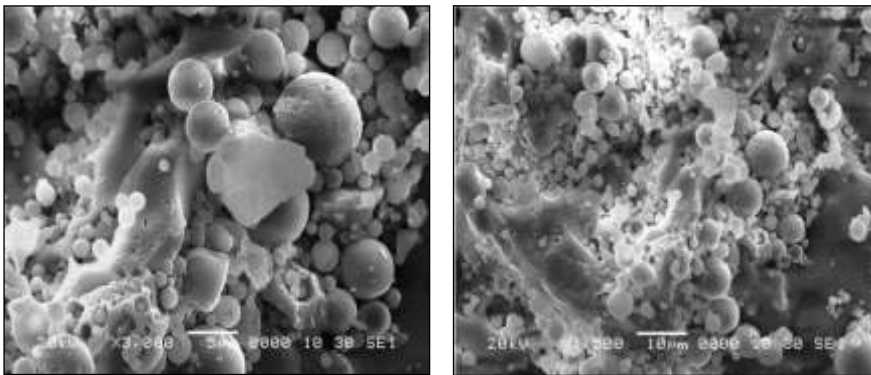
Fly ash is a heterogeneous material with various physical, chemical, and mineralogical properties, depending on the mineralogical composition of burned coal and on the combustion technology. Currently, a specification that is used for ash classification is based on CaO content. Two classes of fly ash are defined by ASTM C618, class F and class C [7]. Typical chemical composition of class F and Class C fly ashes are given in (**Table 1**). The class C fly ash refer to those having more than 8% CaO and is usually from lignite and subbituminous coal combustion. The class F fly ash is from bituminous and anthracite coals and has less than 8% CaO. During coal combustion and collection of fly ash, the particles are suspended in the exhaust gases, thus fly ash particles are generally spherical in shape and range in size from 0.5 to 100  $\mu\text{m}$ . The particle size distribution is an important property of fly ash, with the smaller particles having greater surface areas. The chemical composition depends on coal sources and combustion conditions. For heterogeneous catalytic application, surface area and surface properties are important factors influencing the adsorption and dispersion of the active component. In general, the surface area of fly ash is around 0.52-7.92 m<sup>2</sup>/g [8-10].

**Table 1 Chemical compositions of class F and class C fly ashes (in wt %)**

Component	Class F		Class C	
	Low-Fe	High-Fe	High-Ca	Low-Ca
SiO <sub>2</sub>	46-57	42-54	25-42	46-59
Al <sub>2</sub> O <sub>3</sub>	18-29	16.5-24	15-21	14-22
Fe <sub>2</sub> O <sub>3</sub>	6-16	16-24	5-10	5-13
CaO	1.8-5.5	1.3-3.8	17-32	8-16
MgO	0.7-2.1	0.3-1.2	4-12.5	3.2-4.9
K <sub>2</sub> O	1.9-2.8	2.1-2.7	0.3-1.6	0.6-1.1
Na <sub>2</sub> O	0.2-1.1	0.2-0.9	0.8-6.0	1.3-4.2
SO <sub>3</sub>	0.4-2.9	0.5-1.8	0.4-5.0	0.4-2.5
Loss on Ignition	0.6-4.8	1.2-5.0	0.1-1.0	0.1-2.3
TiO <sub>2</sub>	1-2	1-1.5	<1	<1

**b) Size and Shape**

Fly ash consists of silt-sized particles which are generally spherical, typically ranging in size between 5 and 100 micron (Figure 1). These small glass spheres improve the fluidity and workability of fresh concrete. Fineness is one of the important properties contributing to the pozzolanic reactivity of fly ash. These small size also important for catalyst synthesis.



**Figure 1. SEM micrograph of fly ash at different magnification**

**c) Color**

Fly ash can be tan to dark gray, depending on its chemical and mineral constituents. Tan and light colors are typically associated with high lime content. A brownish color is typically associated with the iron

content. A dark gray to black color is typically attributed to an elevated unburned carbon content. Fly ash color is usually very consistent for each power plant and coal source.



**Figure 2. Appearance of fly ash**

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## Chapter -12

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### Host pathogen interaction in plants

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#### **INTRODUCTION :-**

For a pathogen to infect a plant, it must be able to make its way in to and through the plant, obtain nutrients from the plant, and neutralize the defense reactions of the plant. Pathogens accomplish these activities mostly through secretions of chemical substances that affect certain components or metabolic mechanisms of their hosts. Penetration and invasion, however, seem to be aided by, or in some cases entirely by, the result of the mechanical force exerted by certain pathogens on the cell walls of the plant.

#### **Infection Process:-**

Plants exist in a world which is full with microorganism. The microorganisms continue to grow in the same environment as the plants and trees throughout the growing season or for many years. The surfaces of these plants are constantly exposed to bacteria, fungi, nematodes, and possibly parasitic plants. Plant pathogens have accumulated many adaptations to enable themselves to adhere to plants, overcome the plant defense mechanisms, and colonize plant tissues for growth, survival, and reproduction. Once established inside the plant, they have at least temporarily escaped the intense competition from saprophytic organisms on plant surfaces and in the soil.

The "infection process" can be broadly divided into following three phases:

- a. Adhesion
- b. Penetration
- c. Colonisation

It encompasses the germination or multiplication of an infective propagule in or on a potential host till the establishment of a successful parasitic relationship between the pathogen and the host. The process of infection is influenced by properties of the pathogen, the host and the external environment. If any of the stages of the infection process is inhibited by any of these factors, the pathogen will not be able to cause disease in the host. While some parasites colonise the outside of the plant (ectoparasites), pathogens may also enter the host plant by penetration, through a natural opening (like a stomatal pore) or via a wound. The symptoms of the diseases, produced by these pathogens, result from the disruption of respiration, photosynthesis, translocation of nutrients, transpiration, and other aspects of growth and development.

### **Adhesion:-**

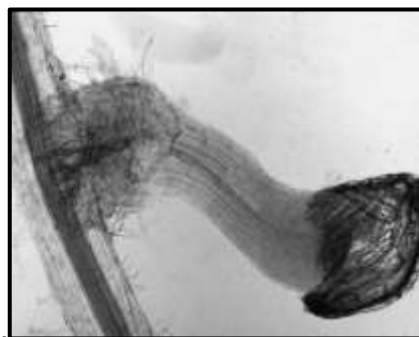
Before a pathogen can penetrate a host tissue, a spore must germinate and grow on the surface of the plant. Many fungi, on encountering their host or some other solid substrate, germinate or start producing germ tubes which may differentiate into infection structures. Adhesion is also crucial to the successful parasitism of plants by pathogens. In fungal-substratum adhesion that occurs on the plant host surface before penetration, adhesion serves multiple functions.

The various functions of a plant-pathogen adhesion can be as follows:

- Adhesion keeps propagules of pathogens from being displaced by not being blown or rinsed by water and/or wind from a potentially suitable environment.
- It is required for host penetration via mechanical pressure,
- It is required for thigmomodification.
- It is required for thigmotropism.
- It facilitates interaction between pathogen and host.
- It increases the surface area of contact with its host.
- It also limits germination to potential host tissue (which is required for contact stimulated germination).



Many bacteria produce fimbriae and they play a role. In the case of motile pathogens, they must find the host and negotiate its surface before entering the host. Some pathogens develop specialised penetration structures, such as appressoria, while others utilise pre-existing openings in the plant's surface, such as wounds or stomatal pores. Plant viruses are often transported and introduced into the plant via vectors such as fungi or insects. The initial contact between infective propagules of a parasite and a potential host plant is called inoculation. Pathogens use a variety of stimuli to identify a suitable entry point. Several fungi use topographical cues on the plant surface to guide them towards a likely stomatal site. Once the hypha reaches a stoma, volatile compounds escaping from the pore appear to provide a signal for the formation of a specialised penetration structure, the appressorium (Fig 1). Sugars, amino acids and minerals secreted by plants at the leaf surface can non-specifically trigger spore germination or provide nutrition for the pathogen. Some pathogenic spores will not germinate in the absence of these substances.



**Fig. 1 : Germinating seed of *Striga hermonthica* giving rise to a appressorium which has attached to a host root**

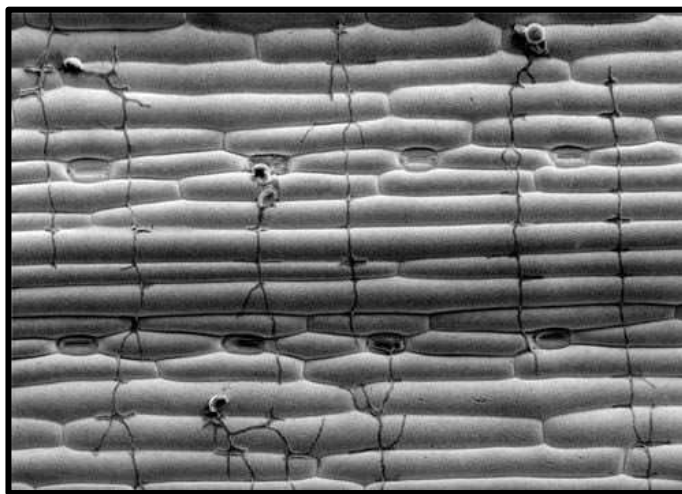
Pathogen development is influenced by temperature, moisture, light, aeration, nutrient availability and pH, whether contact is required can depend upon the environmental conditions. The conditions necessary for survival and successful infection differ between pathogens.

### **Penetration:**

Pathogens normally exploit every possible pathway to enter their host, although individual species of pathogen tend to have a

preferred method. The host plays an equally important role in penetration of the pathogen by providing certain stimuli. The stimuli provided by the host for germination, growth and the differentiation of infection structures can be hydrophobicity, hardness, chemical components and topographical features of the host plant. Several chemical components of host plants have been implicated in the germination of propagules of plant pathogens and the differentiation of infection structures.

In particular, the wax on the surface of aerial parts of the plant is a rich source of diverse compounds, which may play these roles. The topologies of plant surfaces provide signals to many fungal pathogens. For example, rust fungi usually enter their hosts through stomata, their topology triggering the development of infection structures. For rust fungi which enter via stomata, locating a stoma may be facilitated by responding to other topological signals. For example, germ tubes of *P. graminis f. sp. tritici* (Fig 2) orient themselves at right angles to leaf veins which, owing to the manner of their distribution, maximize the chance of the tube encountering a stoma.



**Fig. 2 : Germ tubes of *P. graminis f. sp. tritici* orienting themselves at 90° to the grooves on the host surface corresponding to the epidermal cell junctions**

### **Colonisation:**

Once a pathogen has arrived in the vicinity of a potential host plant or, as may happen in the case of soil-borne pathogens, a plant root

has arrived in the vicinity of a pathogen, subsequent events depend on the production and perception of signals by both partners. In soil, pathogens may be influenced by compounds exuded from the host root. Motile stages may be attracted or repelled and the germination of sessile propagules stimulated or inhibited. Air-borne pathogens generally rely upon large populations of propagules to ensure that at least some of them alight on a suitable host. At this point, adhesion is a necessity to prevent the propagule being washed off the plant and, for at least one fungal pathogen; adhesion has been established as a prerequisite for germination. Following adhesion, germination, which may be under the control of topological or chemical signals from the host, occurs and in some instances such signals lead to the differentiation of infection structures. These, too, require firm anchoring to the surface of the plant if any mechanical force is to be exerted. A successful infection requires the establishment of a parasitic relationship between the pathogen and the host, once the host has gained entry to the plant. There are two broad categories of pathogen-biotrophs (those that establish an infection in living tissue) and necrotrophs (those that kill cells before colonising them, by secreting toxins that diffuse ahead of the advancing pathogen). These two kinds of pathogens are also sometimes known as 'sneaks' and 'thugs', because of the tactics they use to acquire nutrients from their hosts. . Biotrophs often feed through haustoria, which penetrate the host cell wall, almost certainly through the agency of degradative enzymes, and invaginate but do not penetrate the host plasma membrane.

Necrotrophs do not produce specialised penetration structures. Instead, they kill host cells by secreting toxins, then degrade the cell wall and middle lamella, allowing their hyphae to penetrate the plant cell walls and the cells themselves. The toxins produced by necrotrophs can be specific to the host or non-specific. Non-specific toxins are involved in a broad range of plant-fungus or plant-bacterial interactions, and will therefore not usually determine the host range of the pathogen producing them. Necrotrophs often enter the plant through wounds and cause immediate and severe symptoms. For necrotrophs the role of degradative enzymes seems clear. They are required not only for penetration and colonization of plant tissue but also to reduce the high molecular weight components of these tissues to

products which they can metabolize. In the case of soft rotting organisms, this often results in the 'mushy' symptoms that give these diseases their name.

An intermediate category of parasite is the hemibiotrophs, which start off as biotrophs and eventually become necrotrophic, employing tactics from both classes of pathogen. In hemibiotrophic infections, intercellular hyphae can form haustoria in living mesophyll cells, but as the lesion expands under favourable conditions, those heavily parasitised cells at the inner, older part of the colony collapse and die. Pathogens that colonise the surface of plants, extracting nutrients through haustoria in epidermal or mesophyll cells are termed ectoparasites. The haustoria are the only structures that penetrate the host cells. Some parasites colonise the area between the cuticle and the outer wall of the epidermal cells, penetrating host epidermal and mesophyll cells with haustoria. These are called sub-cuticular infections. Pathogens can also form colonies deeper in the plant tissues. These are mesophyll and parenchyma infections, and can be necrotrophic, hemibiotrophic or biotrophic relationships. Colonization of the host by viruses is a special case. Viruses move from cell to cell through plasmodesmata but they may be replicated at sites that are some distance away. In the case of systemic infections long-distance movement of viruses occurs through the phloem or xylem and normally requires an intact capsid protein. Once in the conducting tissues of the plant, movement of the virus and unloading follows as that of solutes but the mechanisms remain unknown.

Viruses, mildews and rusts develop specialised biotrophic relationships with their hosts. Intercellular hyphae of downy mildew colonise host mesophyll cells and form haustoria. The mildew sporulates and the infected cells eventually die, although necrosis is delayed and contained, compared to that caused by necrotrophic pathogens. Rust fungi can also delay senescence in infected cells while they sporulate. Vascular infections usually cause wilting and discoloration as a result of the physical blockage of infected xylem vessels. True vascular wilt pathogens colonize the vascular tissue exclusively, although other pathogens can cause the same symptoms if they infect the vascular system as well as other tissues. There are a few pathogens that manage to achieve systemic infection of their host. For example, many viruses can spread to most parts of the plant, although

not necessarily all tissues. Some downy mildews can also systemically infect their host by invading the vascular tissue and growing throughout the host, causing deformation, rather than necrosis. Finally, there are some pathogens that complete their entire life cycle within the cells of their host, and may spread from cell to cell during cytokinesis. These are endobiotic infections.

### **Summary**

Since many pathogens have to breach the barriers of plant waxes, cutin and suberin that cover plants as well as plant cell walls before establishing a parasitic relation with their hosts. Some soil-borne pathogens locate their hosts through chemical signals and these are also important in subsequent events such as the germination of propagules, chemotropism of germ tubes and the differentiation of infection structures, the last of these also being influenced by physical features of the host. Adhesion is often required for successful penetration, particularly where this is achieved by the exertion of mechanical force. However, enzymes that degrade the surface layers of plants, such as waxes, cutin and suberin are also critical for entry by many pathogens. Once past these surface layers the pathogen usually has to breach the cell wall and for this a range of pectolytic enzymes, cellulases and xylanases as well as enzymes involved in the degradation of lignin are required. In some instances, other enzymes are inferred to have important roles to play in pathogenicity or virulence such as proteases and membranolytic enzymes. The products of degradative enzymes acting on host tissues are sources of nutrition for necrotrophic pathogens but the subtler biotrophic pathogens feed through specialized structures called haustoria. Viruses and viroids lack motility and therefore face a particular problem in colonizing their hosts after entry. Long distance spread is by passive movement in the xylem or phloem.

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## **Chapter -13**

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### **Stem Cells: An Overview**

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#### **Abstract**

Stem cells are the cells with the ability to develop into various types of cells in the body. They act as repair mechanism of the body. Stem cells are distinguished by two main properties: a) the capability to self-renew and b) the potential to differentiate into different types of cells. They are divided into two types: embryonic stem cells and adult stem cells. Due to their ability to repair, regenerate, and develop into certain specialized cell types, stem cells offer great promise as therapy for a number of diseases.

Stem cells are found in all the developmental stages in embryos, fetus and adults. In general terms, stem cells can be called as raw materials of human body. These are the special cells that require proper conditions to produce other cells with unique function. They new daughter cells become either new stem cells or differentiate with more specific function like heart cells, brain cells or bold cells to name a few. No other cells in the living body have the ability to create new cells types (Chagastelles and Nardi, 2011).

Although the concept of stem cells is very old and in past decades its biology and therapeutic potential are studied intensively, still a lot it to be explored (Ramalho-Santos and Willenbring, 2007). They are the machinery of prolonged self-renewal that leads to rise of mature and differentiated cells (Till and McCulloch, 1980; Weissman, 2000). The main difference between a stem cell and the differentiated cell is observed in the cells DNA. In former cell, DNA is loosely arranged with working genes. During the differentiation process, genes which are no longer required are shut down but those necessary for specialized function will remain active. This process can be reversed, and such pluripotency can be achieved by interaction in gene sequences (Zakrzewski et al., 2019).

### **Embryonic stem cells**

Embryonic stem cells (ESCs) are outcome of isolation and cultivation of cells from the blastocyst that forms at approximately 5 days after fertilization (Edwards, 2001). ESCs are pluripotent and possess ability to differentiate into cells that are acquired from germ layers; these cells are immortal in culture and may be maintained for several hundred passages in the undifferentiated state; importantly they control a normal chromosomal composition. First ESCs lines were first obtained in 1981 from mouse blastocysts whereas first human ESCs line was developed in 1998 (Choumerianou et al., 2008). ESCs lines can be preserved in permanent culture and frozen or thawed. More than 250 human ESCs lines are developed worldwide. Crucial advantage of ESCs over adult stem cells is associated to their pluripotency and limitless expansion in culture due to their ability to give rise to all cell types composing the adult organism. Human ESC-derived oligodendrocyte progenitor cells were first to be used in clinical trials in 2010. Due to safety concerns, the clinical use of human ESCs is much more controlled than that of adult stem cells (Chagastelles and Nardi, 2011).

### **Adult stem cells**

Adult stem cells (ASCs) are quiescent cells with very limited self-renewal and differentiation capacity. All the tissues have their own compartment of stem cells because different types of precursor cells have been isolated in adult tissues (Chagastelles and Nardi, 2011). ASCs are accountable for renewing cells that die within a given organ, either due to physiological or pathological processes. The first ASCs were obtained and utilized for blood production in 1948, which was later expanded in 1968 when the first adult bone marrow cells were used in clinical trials for blood disease.

Different types of ASCs are Hematopoietic stem cells, Mesenchymal stem cells, Neural stem cells, Epithelial stem cells and Skin stem cells. Hematopoietic stem cells have been in clinical use for more than 50 years in bone marrow and cord blood transplantation. Mesenchymal stem cells (MSCs) are originated from stroma and can be isolated from any tissue in the organism (da Silva Meirelles et al., 2006;

Meirelles and Nardi, 2009). MSCs are widely used for clinical therapy due to their easy in vitro expansion and ability to differentiate and modulation of immune response (Nardi and da Silva Meirelles, 2006). ASCs possess ability to self-renew for long period and give rise to mature cell types with specialized functions, which highlights their potential of asymmetric division. The cells produced are called progenitor cells that after several rounds of mitosis, give rise to differentiated cells. The mechanism behind the fate of stem cells is not clear but they depend on the interaction between these cells and their microenvironment/niche. They are composed of different cells, extracellular matrix, signaling factors that in combination with intrinsic characteristics of stem cells, define their potential and properties (Fuchs et al., 2004). The role of niche is important, as stemness is regulated only when the cells are attached to it. ASCs proliferate and differentiate once they leave the niche. The ASCs plasticity is still not very clear. The tissue specific stem cells are capable to transdifferentiate across lineage boundaries were subsequently shown to be largely due to technical artifacts (Raff, 2003). ASCs such as MSCs possess more plasticity and therefore can important candidate for cell therapeutic applications.

The important clinical use of stem cells is to renew or replace tissue that are damaged due to injury or disease. These clinical applications can be utilized in cell therapy and tissue engineering approaches (Pessina and Gribaldo, 2006). The use of ASCs nulls the ethical hurdles related to the use of ESCs. Other problems such as spontaneous differentiation with the risk of teratoma formation can be avoided. As seen from various preclinical and clinical studies, ASCs play important role in regenerative medicine. Even hematopoietic stem cells have been used for more many years for hematologic diseases in bone marrow and cord blood transplantation, the therapeutic use of stem cells for non-hematologic disorders has been recently explored. Different numbers of clinical and preclinical studies have been performed. The group of study subjects in most trials is too small at clinical level and controls are not adequately tested to allow accurate testing of the capability of such treatments. Meta-analysis of stem cell therapy clinical trials has exhibited good results but, also indicate the



necessity for controlled trials (Martin-Rendon et al., 2008; Chagastelles and Nardi, 2011).

ASCs are most commonly isolated from the bone marrow. The therapeutic outcome exhibited for hematologic diseases are may be due MSCs that are present in this tissue (Meirelles and Nardi, 2009). Adipose derived MSCs are also successfully used in clinical trials (Nardi and da Silva Meirelles, 2006), but major drawback with ESCs are ethical issues, immunological rejection, and the potential of developing teratomas. The above issues are avoided with the use of ASCs. But still true therapeutic potential of the stem cells for non-hematologic diseases is not fully explored.

### **Medical applications of Stem cells**

They possess great potential and play important role in medicine. The study of stem cells reveals complex events occurring during human development. Incorrect differentiation/cell division leads to birth defects or cancer. Various stem cell therapy treatments are available for retinal and macular degeneration, heart failure, spinal cord injury, and diabetes type 1. In-depth research in stem cells could find the treatments for currently incurable diseases. Stem cells are also important target during pharmacological testing. Stem cells are involved in rejuvenation by cell programming. They also have great importance in Cell-based therapies, incurable neurodegenerative diseases and fertility treatments (Zakrzewski et al., 2019).

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## Chapter -14

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### Applications of Metal Supported MCM-41

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#### **Abstract:**

Mesoporous silica sieves have attracted a lot of interest in recent times, both in terms of both research and application. Particularly, MCM-41 has become popular among researchers due to its versatility in terms of chemical flexibility and application in various fields. Metal incorporation on MCM-41 is found to be effective to enhance its activity. Recently, MCM41 (Mobil Composition Matter), molecular sieves with transition metal incorporation have been utilized as a growth catalyst in various transformations.

#### **Introduction:**

The unique properties of MCM-41 based silica materials make them attractive candidates for applications in catalysis, production of novel materials by encapsulating metals, semiconductors and biofluids. The application of these composites in biotechnology, such as medication delivery, biosensors, and biocatalysis, is given special consideration[1].

In order to utilize MCM 41 as efficient catalyst, it is necessary to generate the appropriate catalytically active sites to the silicate framework of MCM41. The main objective of the support material is to give the active component a large surface area and porosity. The dispersion and morphology of catalyst are directly affected by the interaction between the catalyst and the support material [2].

#### **Applications:**

##### **Copper deposited on MCM-41 in the synthesis of 5-substituted 1H-tetrazoles**

Two different post-functionalization modifications of MCM-41 were used to create Cu-functionalized mesoporous MCM-41 materials:

MCM-41@Cu (without ligand) and MCM-41@Histidine@Cu (with ligand). Cu-functionalized mesoporous MCM-41 sample was also prepared directly by condensation of tetraethyl orthosilicate (TEOS) with CuCl<sub>2</sub> and decyltrimethylammonium bromide (DTAB) (Cu-MCM-41) [3].

### **Ni/Al-MCM-41 catalysts for selective hydrodeoxygenation of anisole to cyclohexane**

The hydrodeoxygenation activities of the Ni/Al-MCM-41(x) catalysts were much higher than those of the reference Ni/Al<sub>2</sub>O<sub>3</sub> and the Ni/MCM-41 catalyst without Al. Nickel loading in the catalysts was maintained constant (5 wt%), while Al content in the supports was varied (Si/Al molar ratios  $x = 120, 90, 60$  and  $30$ ). In addition, they showed high selectivity in the formation of the deoxygenated products, with more than 96% selectivity to cyclohexane.. With Ni/Al-MCM-41(90) catalyst, the best activity and selectivity values were obtained [4].

### **Effective mineralization of Diclofenac by catalytic ozonation using Fe-MCM-41 catalyst**

This study analyzed the efficiency of Diclofenac (DCF) detoxification and degradation in the Fe-MCM-41/O<sub>3</sub> process. The removal of DCF was effective with both the O<sub>3</sub> and Fe-MCM-41/O<sub>3</sub> systems, and the presence of Fe-MCM-41 greatly improved its mineralization. The Fe-MCM-41/O<sub>3</sub> method produced high mineralization efficiency (76.3%), which was 2.8 times greater than that of ozonation[5].

### **Biodiesel production and engine performance study using ZnO/MCM-41**

This work demonstrates the role that powdered lemon peel plays a role in the synthesis of ZnO nanoparticles. ZnO/MCM41 with a core-shell morphology was easily synthesized using a one-pot method. The effectiveness of various ZnO coating percentages (10%, 20%, and 30%) over MCM-41 in the conversion of biodiesel was investigated. With the catalyst ZnO/MCM-41 (20%), a maximum conversion of 99.4% of Jatropha oil was noted. [6].

### Ti-Modified MCM-41 for Oxidation of Secondary Alcohol with H<sub>2</sub>O<sub>2</sub>

In this work, wet impregnation and direct hydrothermal synthesis were utilized to create titanium-containing MCM-41, which was then utilized to oxidize secondary alcohol to a carbonyl compound in the presence of hydrogen peroxide. The synthesized mesostructured catalysts were characterized by FT-IR, BET, XRD, SEM-EDX, and Raman spectroscopy; the results showed that the Ti was successfully incorporated into the MCM-41 framework. The mesoporous material of all samples retained its hexagonal structure, according to XRD data, while the Ti-MCM-41(20) sample had a slightly deformed porous structure. To test the catalytic performance, 1-phényl-1-propanol was oxidized in dichloromethane using aqueous 30% H<sub>2</sub>O<sub>2</sub> as an oxidant.

Ti placed straight into the mesoporous silica gel exhibited superior catalytic activity (95-98%) [7].

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## Chapter -15

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### Biological Significance of Five and Six membered Heterocyclic Compounds

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#### **Abstract:**

Heterocyclic compounds are of huge importance biologically and industrially and the largest of classical division of organic chemistry is formed by heterocycles. Today there are a number of heterocyclic compounds are known, day by day the number is increasing fast due to the enormous synthetic research and also their synthetic utility. Most fields of sciences such as medicinal chemistry, biochemistry also another area of sciences important role of Heterocyclic compounds. In this chapter, recently synthesized heterocyclic compounds shows enormous biological activity such as antifungal, anti-inflammatory, antibacterial, antioxidants, anticonvulsant, antiallergic, herbicidal and anticancer.

**Keywords:** Heterocyclic compound, Biological active, Five & Six Membered Heterocycles etc.

#### **Introduction**

Heterocyclic compounds are of huge importance biologically and industrially and the largest of classical division of organic chemistry is formed by heterocycles. More than a century, the largest area is being occupied by heterocyclic compounds in the area of research. Organic chemists weekly synthesize hundreds of new heterocyclic compounds. In most cases specific reason for the synthesizing of particular heterocyclic compounds generally, based on the theoretical consideration, pharmaceutical chemistry and biological mechanism or all of three combinations.

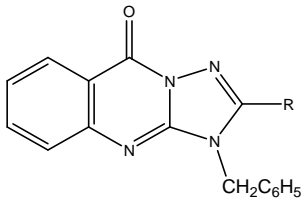
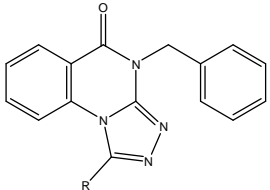
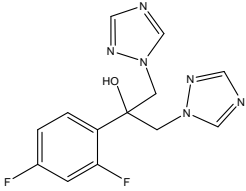
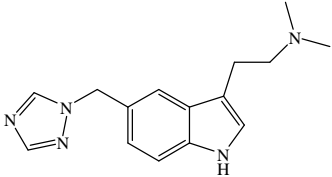
Cyclic compounds containing one or additional hetero atoms in the ring are called heterocyclic compounds. Most of the heterocycles are individually having five or six membered rings and containing nitrogen (N), Sulphur (S) and oxygen (O) are common heteroatoms but heterocyclic rings containing other heteroatoms are also generally known. Heterocyclic chemistry is the branch of pure organic chemistry that involves synthesis and evaluation of properties and applications of these heterocycles. It has become an important branch of organic chemistry and contributes medicinal chemistry to a greater extent. Nitrogen heterocycles are naturally abundant than those of Sulphur or oxygen heterocycles [1] and which are widely distributed in nucleic acids, involvement in approximately every physiological process of plants and animals.

Organic researchers more attentions to the synthesis of heterocyclic compounds over the years due to their therapeutic values.[2] Nitrogen, oxygen and sulfur heteroatom containing poly-functionalized aliphatic or aromatic heterocyclic compounds proven as significant in the process of drug discovery.[3] Drugs analyses in the market shows that more than sixty percent are heterocycles.[4] Hence, it is clear that the past decades due to chemotherapeutic value of heterocyclic nuclei containing compounds gain much importance in the development of novel biologically active scaffolds, such as anti-malarial, analgesic, local anesthetic, antioxidant, antimicrobial, anti-diabetic, anti-tubercular, anti-helminthic, anti-inflammatory, anti-epileptic, anti-anxiety, antiviral, antihistaminic, anti-neoplastic, antidepressant, antihypertensive, and anti-Parkinson's agents, etc.[5-6] The heterocyclic compounds are generally distributed in environment and are extremely essential for the living organisms; they play a fundamental role in metabolism of all living cells, such as the pyrimidine and purine bases of the genetic material DNA, the essential amino acids, proline, tryptophan and histidine, the coenzymes, vitamins, the photosynthesizing pigment chlorophyll, and the oxygen transporting pigment haemoglobin etc. A vast number of synthetic and naturally occurring heterocyclic compounds found their use in medicines, agrochemical, and polymers etc.

Cimetidine, chlorpromazine, metronidazole, diazepam, famotidine, azidothymidine, barbiturates, methotrexate, antipyrine, and captopril are synthetic drugs which are heterocyclics. H<sub>1</sub> receptor blockers such as Cetirizine [7] and Chlorpheniramine, antiretroviral drugs such as Zidovudine/azidothymidine (AZT) reverse transcriptase inhibitor [8-10] (also called ZDV), Acyclovir (ACV), anticancer drugs Cladribine (Leustatin) [11] and Pentostatin [12] are heterocyclics. Many antibiotics including penicillin, cephalosporin, norfloxacin, and beta-lactamase inhibitors Clavulanic acid and Sulbactam contain heterocyclic rings.

### Biological Significance

**Table 1. Biologically Active 1,2,4-Triazole Derivatives**

Compound	Structure	Biological Activity [Ref]
3-benzyl-2-substituted-3H-[1,2,4] triazolo[5,1-b]quinazolin-9-ones		Antihypertensive activity [13]
4-benzyl-1-substituted-4H-[1,2,4] triazolo[4,3-a]quinazolin-5-ones		Antihistaminic agents [14]
Fluconazole		Antifungal Agent [15]
Rizatriptan		Treatment of migraine headaches [16-17]



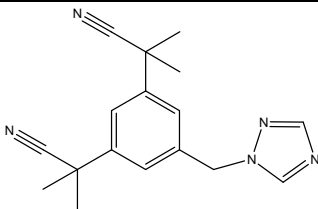
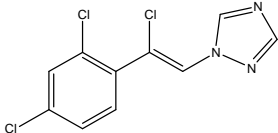
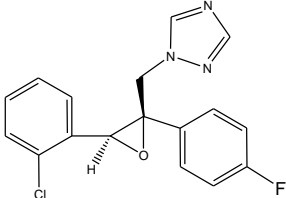
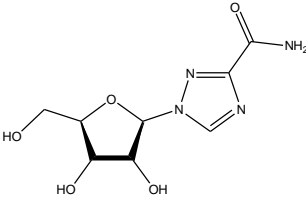
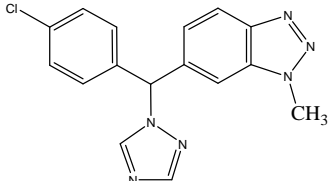
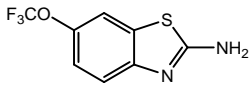
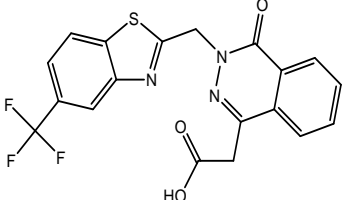
Anastrozole		Treatments for breast cancer [18]
Loreclezole		Anticonvulsant activity [19]
Epoxiconazole		Fungicide active ingredient [20]
Ribavirin (1-β-D-Ribofuranosyl-1,2,4-triazole-3-carboxamide)		Antiviral activity [21-22]
Vorozole		Antineoplastic agent [23]

Table 2. Biologically active benzothiazoles derivatives

Compound	Structure	Biological activity [Ref]
Riluzole		Anticonvulsant [24]
Zopolrestat		Aldose Reductase Inhibitor [25]

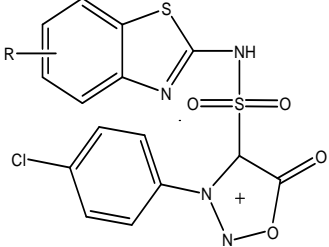
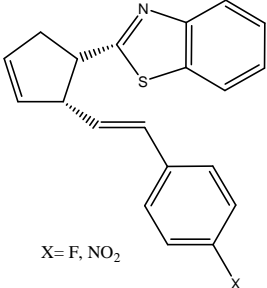
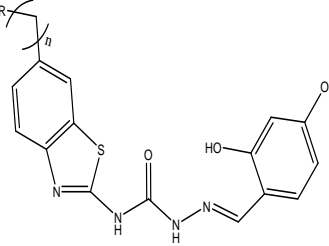
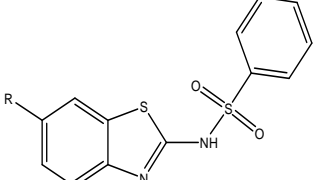
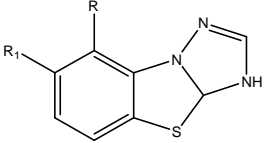
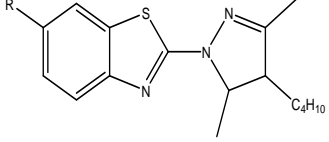
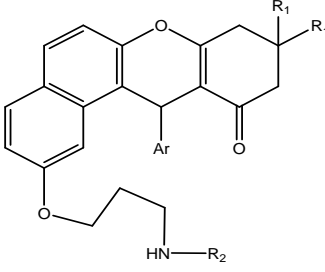
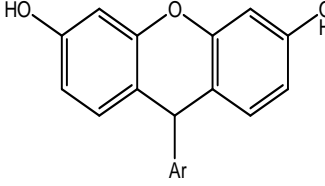
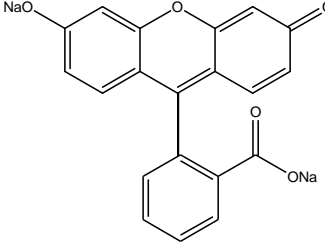
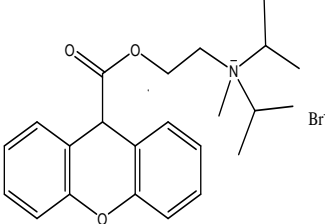
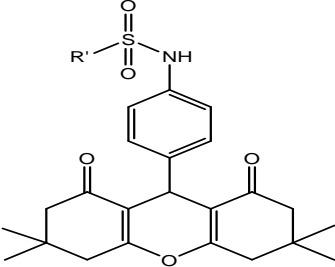
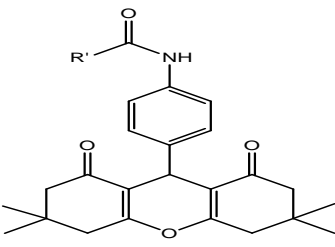
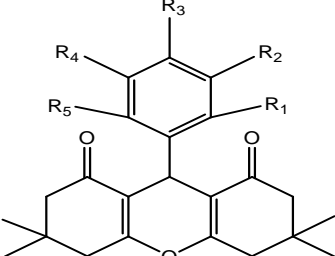
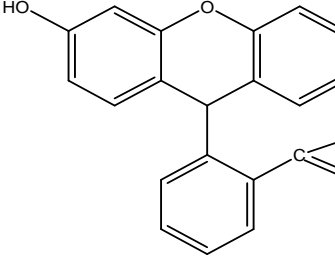
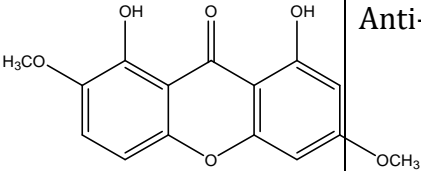
<p>4-[(Substituted-1,3-benzothiazol-2-yl)sulfamoyl]-3-(4-chlorophenyl)sydnones</p>		<p>Antimicrobial [26]</p>
<p>2-Substituted benzothiazole derivatives</p>	 <p>X = F, NO<sub>2</sub></p>	<p>Anticancer and Antioxidant [27]</p>
<p>Benzothiazole and <i>ortho</i>-hydroxycarbamoylhydrazone moieties</p>		<p>Antitumor Agents [28]</p>
<p>2-(substitutedarylsulfonamido)-6-substituted</p>		<p>Anti-bacterial activity [29]</p>
<p>8-fluoro-9-substituted benzothiazolo 1,3,4-triazoles</p>	 <p>R = Aniline, Nitro aniline R<sub>1</sub> = F, Br</p>	<p>Anthelmintic activity [30]</p>
<p>2-(4'-butyl-3',5'-dimethylpyrazol-1'-yl)-6-substituted benzothiazoles</p>		<p>Anti-inflammatory activity [31]</p>

Table 3 Biologically active xanthenes derivatives

Compound	Structure	Biological activity [Ref]
2-(3-aryl/alkylaminopropoxy)-12-aryl-xanthene derivatives		Antibacterial and Antifungal activity [32]
9-aryl-9H-xanthene-3,6-diol derivatives		Anti-inflammatory and analgesic agents [33]
Fluorescein		Fluorescein angiography or angioscopy of the retina and iris vasculature
Propantheline bromide		Anti-muscarinic agent [34]

<p>Xanthene sulfonamide and carboxamide derivatives</p>	 <p>Xanthene sulfonamide</p>  <p>Xanthene carboxamide</p>	<p>Antimicrobial activity [35]</p>
<p>2,2,5,5-Tetramethyl-9-aryl-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione Derivatives</p>		<p>Antioxidant, Antimicrobial and Anti-proliferative Activity [36]</p>
<p>2-(3,6-dihydroxy-9H-xanthen-9-yl) benzamide</p>		<p>Anti-cancer activity [37]</p>

1,8-dihydroxy 3,7-dimethoxy-xanthene-9-one		Anti-malarial Activity
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## Conclusion

Heterocyclic compounds one of the important types of organic compounds, which is taking a wide range in the medicinal chemistry this due to the enormous number of heterocyclic compounds that used in medicine as drugs for varied diseases. The drugs which contain the core of heterocyclic its skeletons such as Antifungal activity, anti-inflammation, anti-bacterial, antioxidants, anticonvulsant, antiallergic, herbicidal activity and anticancer, etc.

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## Chapter -16

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### Nano-Biotechnology: Present Uses and Prospects

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#### ABSTRACT:-

The field of nano-biotechnology focuses on creating products at the nanoscale through the use of biologically inspired frameworks and evolution. In this article, we looked at nano-biotechnology, a novel field of study that combines the expertise of biologists, chemists, physicists, engineers, and medical professionals. This field has undergone a revolution since the discovery of nanoparticles. There are many different kinds of nanoparticles, including metallic, polymeric, carbon-based, and ceramic nanoparticles. They are created using specialized methods, including top-down and bottom-up approaches. The production of fuel and energy, bio-formulations, nanofabrication, and biomimetic are just a few of the many applications for nano-biotechnology. Another amazing accomplishment of Nano biotechnology is the creation of Nano robots. Scientists are working to introduce innovations and improvements to this rapidly developing field, but there are some unavoidable limitations and challenges in this area.

**Keywords:** Principles, Nano-particles, Applications, Therapeutics, Advancements, Challenges

#### INTRODUCTION:

While biotechnology has only been around for a few generations and its boundaries are still being defined, nanotechnology and bio-nanotechnology are relatively new concepts from the late 20<sup>th</sup> century [1]. Biotechnology is the application of scientific knowledge and instruments to the manipulation of genetic, molecular, and cellular



phenomena in order to create products or commodities that are used in a wide range of industries, from agriculture to medicine [2]. The branch of nanotechnology known as "bio-nanotechnology" combines atom-level engineering and manufacturing with biologically inspired elements. It is also closely related to biotechnology, but it also involves the possibility of creating and modifying the atomic-level details of the things that are produced. Bio Nanotechnology equipment is designed at the atomic level and clearly performs 3D molecular tasks, including processes for individual control integrated into their framework [1].

By limiting the applications of digital technology, nanotechnology can be defined as an innovation that allows for the controlled initiation of nanomaterials and their implementation, that is, impacting or simply viewing them for their intended use. As a result, it has the potential to taper many biological sciences pathways, and nanotechnology is already deeply ingrained in common biological matters [3, 4]. It can also change our preconceived notions and cognitive processes and clearly distinguish between biology, physics, and chemistry [5]. The field of nanotechnology is incredibly diverse, ranging from the addition of new techniques based on molecular self-build to the development of novel products using nanoscale real estate and the direct manipulation of atomic barriers. This idea involves applying a wide range of scientific disciplines, including surface science, organic chemistry, semiconductor physics, cell genetics, and micro fabrication [6].

### **History:**

Michael Faraday studied the properties or formation of colloidal suspensions of "Ruby" gold particles in 1857. They are among the most fascinating nanoparticles because of their unique electrical and visual properties. Faraday demonstrated the formation of multicolored fluids by gold nanoparticles under suitable lighting conditions [7]. In 1959, Caltech physicist Richard Feynman introduced the concept of nanotechnology in a talk titled "There's Plenty of Room at the Bottom." Feynman hinted that although he never used the word "nanotechnology," it will eventually be able to successfully control atoms and molecules [8].

Nanotechnology has advanced since Feynman's initial theories and concepts in 1981, when physicists Gerd Binnig and Heinrich Rohrer created the Scanning Tunneling Microscope at IBM Zurich Research Laboratory (STM) [9]. It was demonstrated in 1985 by Robert Curl, Harold Kroto, and Richard Smalley that carbon can survive to very stable spheres known as fullerenes or buckyballs [10]. Nano informatics exclusively works with the obtaining, sending, scheduling, mapping, and interpreting data and information at the critical nanoscale level. By improving the nano-modeling of tumor cells and making it simpler to identify drug-resistant malignancies, nano informatics also supports chemotherapy. Localized gene therapy and targeted medication delivery based on hyperthermia are the most effective Nano informatics approaches for cancer prevention with the least amount of negative direction [11].

### **Nanoparticles:-**

A wide class of materials known as nanoparticles (NP) is made up of particulate matter with a minimum size of 100 nanometers. Depending on their shape, these materials can have a 1D or 3D shape. A range of metal ions, surfactants, and polymers can be used to structure the surface layer of NPs, which are three-layered molecules. Similarly, a variety of metal ions, surfactants, and polymers can be used to structure the inner layer of NPs.c) The shell layer, which differs from the core material in terms of structure and chemistry (Figure 1) [12].

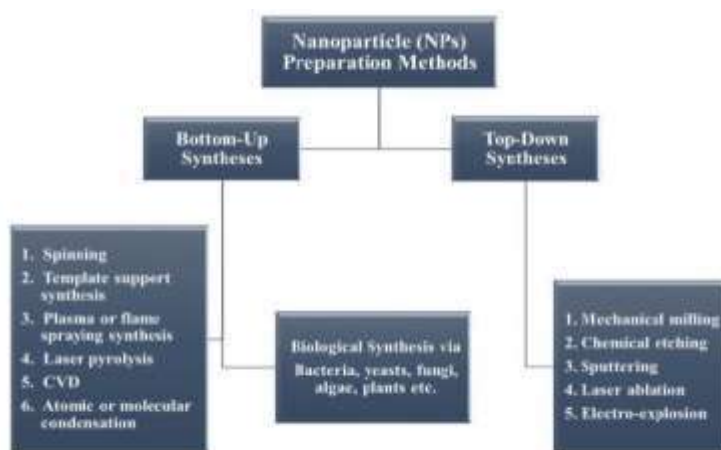


Figure 2 Typical synthesis methods for Nanoscale Particles (NPs)

**NP classification:-**

NPs fall into a number of categories based on their size, shape, and chemical makeup. Based on their physical and chemical characteristics, the following is a list of some of the more well-known

**NP classes:**

NPs based on carbon: Two types of carbon-based NPs are distinguished: Carbon nanotubes and fullerenes (CNTs). Fullerenes are carbon-based chemical elements whose molecules consist of individual carbon atoms joined by single or double bonds to form a dense or partially blocked network of compact rings. A molecule may resemble a tube, a spheroid, or exist in a wide range of other forms and dimensions. Carbon tubes, or carbon nanotubes (CNTs), usually have a diameter of a few nanometers. Single-Walled Carbon Nanotubes (SWCNT) with diameters in the nanometer range are commonly discussed in relation to carbon nanotubes. The allotrope of carbon that exists between planar graphene and carbon cages is comprised of single-walled carbon nanotubes [13]. Polymeric nanoparticles: Also known as polymeric nanoparticles (NPs), these particles range in size from one to a thousand nm. They can have charged compounds adsorbed on their surface or enclosed in the polymer core. Targeted drug delivery using polymeric nanoparticles (NPs) has considerable promise for treating a range of illnesses. Polymer Nanoparticles (NPs) have garnered significant attention recently due to their small size [14]. Metal precursors are used to create metal particles. These nanoparticles are created by chemical, electrochemical, and photochemical processes. These can adsorb small molecules and have a high surface energy. These nanoparticles are used in environmental and bioanalytical applications, biomolecule detection and imaging, and biomedical research. The sample is coated with gold nanoparticles prior to being examined in the SEM. Increasing electronic flow is often achieved by doing this, which helps to produce high-quality SEM images [15]. Ceramic nanoparticles: Another name for ceramic nanoparticles is metalloid solid. Heating and cooling processes are used to create ceramic nanoparticles. Amorphous, polycrystalline, solid, hollowed-out, and nonporous ceramic nanoparticles are all possible. Because of these

nanoparticles' numerous uses in chemical reactions, dye photodegradation, photocatalysis, and imaging applications, researchers focus on them [16].

### **Manufacturing of Nanoparticles:-**

To supply the various nanoparticles, coatings, dispersions, or composites, specific synthesis techniques are employed. Acquiring such size-structured particle functions requires defined manufacturing and response scenarios. pH, concentration, temperature, soil adjustments, chemical composition, and engineering control can all be used to control the chemical composition, particle size, crystallinity, and shape. Top-down and bottom-up approaches are the two main methods utilized to supply nanoparticles. The mechanical grinding of the storage tissue using grinding techniques is generally referred to as the "top-down" period in this context. Building the system through chemical and self-conference tactics is the bottom-up approach. The preferred functions exactly for the nanoparticles and their chemical makeup determine which technique is best [17].

### **Application of Nanoparticles:-**

Nanoparticles have a variety of potential uses. A few of these crucial applications are listed below: Regarding environmental remediation and renewable energy: Natural nanoparticles have been shown to have the ability to restore the environment in certain circumstances. The effective treatment of air, water, and soil through the use of nanoparticles or nano-treatment has been a part of environmental restoration for over ten years. Nanoparticles are used in the disinfection, filtration, and evaporation of surface water. The main purpose of nanoparticles is to stop household and commercial waste from becoming sludge.

### **Nano biotechnology Applications:-**

Nanobiotechnology in bio-formulation: This area of study addresses the limitations related to large-scale development and the commercialization of bio-inoculant formulations. The creation of biopesticides and biofertilizers in conjunction with nanobiotechnology

is known as bio-formulation [18]. Biomimetic: Applying the principles of underlying mechanisms to potential medical, scientific, or engineering applications [19]. Biomimicry used some biological solutions to discover new technologies. A few biomimetic techniques have been in use for a long time. For instance, synthetic production of specific vitamins and antibodies. Biomimetics has been proposed recently for use in data converters, machine hearing systems, and signal amplifiers. Additional potential uses for biomimetics include nano-robot antibodies, various electronic devices, and artificial organs. Solving human problems involves analyzing nature, its workings, and the role models it provides. It is the study of nature and natural phenomena with the goal of drawing inspiration from it [19]. The process of designing objects and materials with nanoscale dimensions is known as nanofabrication. Because a large-scale economy is developed with the same machinery and minimal material, it is an economical method. It makes use of cutting-edge technology to produce different types of silicon chips, microcontrollers, and microchips [20].

### **Nanorobots:**

New avenues in robotics have been opened up by nanobiotechnology. These robots will be utilized to introduce novel concepts to the human body. Nanobots are capable of intracellular surgery and other procedures. An onboard computer can coordinate various tasks, such as pathology, diagnosis, and lesion correction or removal [21]. Though they are still in the early stages of development and research, they are capable of carrying out their particular function at the molecular and cellular levels. Other names for nano-robots include nano-mites, nano-machines, and nano-ids [22]. Because of their advancements in cancer diagnosis and drug delivery, nano-robots are currently causing havoc in the biomedicine industry [23].

### **Applications of Nanoparticles for Tracking Stem Cells:-**

Superparamagnetic Iron Oxide (SPIO), a nanoscale particle, is emerging as the perfect test for non-invasive cell tracking. However, its low intracellular labeling proficiency has limited its use and stimulated attentiveness in the development of recently discovered labeling

policies. The unusual frequency of the fluorine amalgamation can be adjusted in nanoparticle Neuro-micro scans. With this method, backcloth gestures that interfere with medical imaging are eliminated. Incompatible compounds could be used to stamp cells with contradictory designations, which could subsequently be independently detected by adjusting the MRI scanner. Prina-Mello and colleagues have demonstrated that nickel nanowires and growth in alumina membranes can be introduced into adherent and suspended cells and used for cell manipulation, including proof and partitioning. The author has further stated that internalized nanoscale wires within adherent cells can be manipulated (rearranged) without causing any anisotropy in the occupants of the cells [24].

#### **Applications for Health:-**

Nanobiotechnology can create novel antidote formulations with less side effects and more efficient drug delivery systems.

#### **Gene Transfer:-**

Current gene therapy systems are beset by the inherent limitations of successful pharmaceutical processing and manufacturing, in addition to the engineered mutant risk of reversion to the natural category. In this context, viral vectors of immunogenicity that are used for gene delivery are also a concern. [3].

**The Use of Nanoparticles in the Development of Biomarkers:-**Recent advances in molecular diagnostics have been applied to identify biomarkers of various infections. The identification of biomarkers has been corrected by nanobiotechnology. Ingenious molecular diagnostic tests assume that certain biomarkers are also present. Nanoparticles' high surface areas and physicochemical properties make them ideal for developing biomarker-harvesting platforms. It is possible to customize the surface of nanoparticles, which is defined as the range of available nanoscale particle advancements, to specifically bind a subset of biomarkers and isolate them for further investigation using highly sensitive proteomic tests [25].

**Particles at the nanoscale for molecular diagnostics:-**

A small number of nanoparticles have been applied to diagnosis. This indicates that QDs, magnetic nanoparticles, and gold nanoparticles are the most commonly used [25].

**Now Nanotechnology in Bio-Research:-**

Nowadays, bio-nanotechnology is a significant topic; it is undoubtedly a developing field. The field of bio-nanotechnology is experiencing exciting times right now. The development of nanomedicines has made it possible for scientists to alter the human body's mechanisms for the better, fixing faulty genes and curing illness. The familiar natural materials we work with, like wood, bone, and shells, provide the building blocks needed to create materials tailored to our needs on a nanoscale. The biological process of retrieving and storing nanoscale information is being productively applied to the interpretation of computational complexity and cybercriminals [1].

**Prospects for Nano-Biotechnology in the Future:-**

There are a lot of conversations concerning the implications of nanobiotechnology in the future. It might create and suggest the development of a variety of novel materials and devices that could be useful in the fields of medicine, biomaterials, hardware, and energy production. However, this methodology presents significant challenges as does any new technology, such as questions about the ecological impact and toxicity of nanoscale materials, their potential effects on global economics, and speculation about countless end-of-the-world scenarios. It makes sense to have specific legal regulations for nanobiotechnology. These examinations have served as a forum for debate between legislators and advocacy organizations. [26].

**CONCLUSION:**

An overview of Nano biotechnology and its applications across a range of industries is given in this review article. It focuses on the traits and varieties of various nanoparticles, such as metal, ceramic, polymeric, and carbon-based nanoparticles. These nanoparticles play a key role in important processes such as biomarker discovery and stem

cell tracking. Furthermore, the importance of Nano biotechnology in bio formulations, biomimetic, and nanofabrication is also emphasized in this article. The field of medical science could greatly benefit from the advancement of Nano biotechnology, resulting in better health care practices. Medical science is on the verge of new advancements thanks to the concept of applying Nano biotechnology knowledge to drug delivery, gene delivery, stem cell-based therapy, and cancer diagnostics. It is undeniable that some scientists are concerned about the biosafety, environmental risks, and contamination that nanomaterials may cause. These issues need to be addressed.

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## Chapter -17

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### Ternary complex formation equilibria of ligand with Cu(II) and dipeptides

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#### **Abstract-**

Ternary complex formation equilibria of Cu(II), metal involving tetra dentate Schiff base ligand (L) 4-hydroxy-3(1-{2-[(4-chlorobenzylideneamino) phenylimino] ethyl} - 6 methyl-2H- Pyran2-one as primary ligand and dl- alanyl glycyl ( $r_1$ ), glycyl- dl-valine( $r_2$ ) dipeptides as secondary ligands have been carried out in 60% (v/v) THF-Water medium and ionic strength of 0.1 M ( $\text{NaClO}_4$ ) investigated potentiometric ally. Ternary complexes are formed by simultaneous reactions. The stability constant of binary complexes  $\text{ML}$ ,  $\text{ML}_2$  are determined under similar experimental conditions. The species formed during ternary complexation such as concentration of total metal, total ligand, free metal, free ligand are discussed by Irving Rossottii technique calculated by using "SCOG" computer programme. The comparative study of Cu(II) ternary complexes is discussed.

**Keywords-** Ternary complexes, tetra dentate Schiff base ligand, dipeptides.

#### **I. INTRODUCTION:-**

The ligand involved in ternary complexation have the functional groups such as  $-\text{COOH}$ ,  $\text{NH}_2$  and  $\text{CONH}_2$ . Which are identical with those present in enzyme. Schiff base transition Metal Complexes have important and popular area for research due to their simple synthesis [1]. The importance of ternary metal complexes is from application of such complexes in many analytical and biological reactions [2,3]. Metal ternary complexes with Oxygen and nitrogen donor ligands show remarkable high stability [3-6] ONH, Schiff Life and its appearance are connected with specific and distinct macroscopic structures. Complexes of amino acids and peptides are involved in the exchange and transport

mechanism of trace metal ions in the human body [6, 7]. Hence it is worthwhile to study ternary complexes of Cu(II) dipeptides MLR complexes Where M = (Cu) and L = Schiff bases & R = dipeptides. Copper is ubiquitous in plants and animals, and its redox chemistry is involved in a variety of biological oxidation processes. Copper usually binds to proteins (copper proteins) in living organisms. As ternary complexes containing Cu(II) as a metal are relatively well investigated. The stability of ternary complexes will help towards understanding of the driving forces that lead to the formation of such complexes in biological systems. In the present study Cu(II) metals is used.

In continuation of our earlier work [8-10] we have prepared tetra dentate Schiff base with known method.

## **II. EXPERIMENTAL:-**

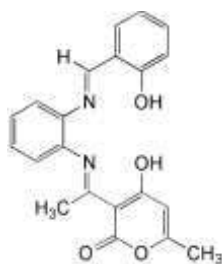
THF, NaOH used in present investigation Titrations were obtained from E. Merk. THF was further purified by known literature method [11]. All metal chlorides used for preparation of metal ion solutions obtained from their AR grade metal chlorides. These metal solutions were further standardize by known literature method [11]. A standard 0.2N NaOH solution was used for titrations. Standard solutions of AR grade HClO<sub>4</sub>, NaClO<sub>4</sub> (1.0M) were prepared and standardized by known methods [11]. Experimental procedure by potentiometric titration technique performed in inert atmosphere (nitrogen) at ionic strength of 0.1 M (NaClO<sub>4</sub>). The ligand solution (0.1M) was prepared in distilled THF. Schiff base used in present investigation found to be insoluble in water. THF: water (60:40) was used as a solvent for potentiometric studies. The potentiometric technique for study of the mixed ligand complexes include following titrations.

- 1). Free HClO<sub>4</sub> → A
- 2). Free HClO<sub>4</sub> + Ligand (L) → A + L
- 3). Free HClO<sub>4</sub> + Primary Ligand (L) + Metal ion (M) → A + L + M
- 4). Free HClO<sub>4</sub> + Secondary Ligand (R) → A + R
- 5). Free HClO<sub>4</sub> + Secondary Ligand (R) + Metal ion → A + R + M
- 6). Free HClO<sub>4</sub> + Primary Ligand (L) + Secondary Ligand (R) + Metal ion → A + L + R + M

The solutions were titrated pH metrically against (0.2N) NaOH. The commercial alcohol was distilled over freshly ignited dry calcium oxide to obtain absolute alcohol. The fraction distilled at 78°C was collected after discarding 20 ml of distillate. Anhydrous methanol and super dry ethanol were obtained from commercial methanol and ethanol (AR) respectively by distillation after treatment with magnesium metal turnings and iodine [11]. All other solvents used during the entire research work were of AR grade.

## II.Apparatus:-

The titrations were carried out using Elico digital pH meter (model LI-127) equipped with a CL-51B combined electrode for pH measurements. Before titrations pH meter was calibrated against standard buffers (pH 4.02 and 9.18) readings were corrected for THF: water media. The pH and volume of NaOH piloted to determine protonation constants and stability constants of the Schiff bases and their complexes were determined. Throughout the experimental work glass distilled water was used. This was obtained by double distillation of deionised water in presence of crystals of potassium permagnate and potassium hydroxide pallets. For synthesis of Schiff bases, ethanol and methanol were used as commercial solvents. Structure of Ligand is as follow

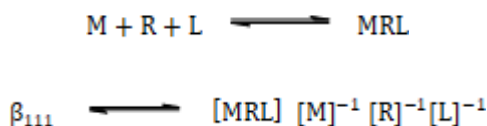


**Figure1. 4-hydroxy-3(1-{2-[(4-chlorobenzylideneamino) phenylimino] ethyl} - 6 methyl-2H- Pyran2-**

## II.Potentiometric measurements:-

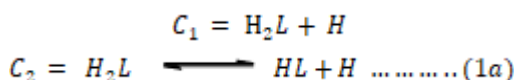
The synthesis and characterisation of ligand is reported earlier [12]. Stock solutions of ligand (0.1M) was prepared in THF. The protonation constants of primary ligand (L) and secondary ligands DL-Alanyl Glycyl (R<sub>1</sub>), Glycyl- DL-Valine (R<sub>2</sub>) with Cu ions determined in

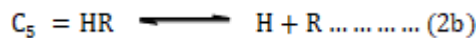
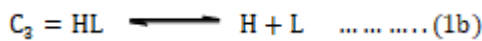
60% (v/v) THF:Water medium. The Irving-Rossetti technique was used after appropriate pH corrections determined by using method suggested by Van Uitert [13]. The protonation constant and metal-ligand stability constant of ligands is given in the (Table 1). The pH values of titrations were found in between pH 3 to 11 from pH metric data the stoichiometry, deprotonation and stability constants were calculated. Primary and secondary ligands form both 1:1 and 1:2 complexes with Cu(II) ions. Here two systems are represented in the above work. The Concentration of various species HL, L, R, CuL, CuR, CuLR, at different pH were obtained by using SCOGS computer programme [14, 15]. The concentration of these species were plotted against pH values to study complexing equilibria and predominance of mixed ligand complexes over binary complexes. Equilibrium constant  $\beta_{111}$  for the reaction



The plots of systems i.e. Cu(II)-DL-Alanyl Glycyl (L), Cu(II)-Glycyl-DL-Valine (L), systems were presented (Fig.1-8). The possible equilibria in the ternary chelation was done by analyzing the graphs. The pH titration curves for systems Cu(II)-L-R<sub>1</sub>/R<sub>2</sub> are represented in (Fig. 1-2).The precipitation pH of mixed ligand complex was about 7.2. The mixed ligand curves coincide with A+L curve .at pH 1.5 and then deviates. The mixed ligand ligand curve from the theoretical composite curve towards left indicate the formation of ternary complex. The mixed ligand curve did not coincide with A+L+M and A+R+M metal complex titration curves, indicates the formation of 1:1:1 complex by a simultaneous equilibria.

In Cu(II)-L-R<sub>2</sub> system from (Fig.2 )it was observed that the mixed ligand titration curve coincides with A+L curve at pH 3.8 and later on deviates. Non super impossible nature of the composite curve over mixed ligand curve confirms the formation of ternary complex.





The relative stability of ternary complexes as compared to binary complexes indicated by  $\Delta \log K$ ,  $K_r$ ,  $K_R$  and  $K_L$  parameters. These parameters are defined by the equations.

$$V. \Delta \log K = \log \beta_{111} - \log K_{10} - \log K_{01}$$

$$VI. K_r = \beta_{111}^2 / K_{20} K_{02}$$

$$VII. K_R = \beta_{111} / K_{10}$$

$$VIII. K_L = \beta_{111} / K_{10}$$

## V. RESULT AND DISCUSSION

### i). Cu(II)LR<sub>1</sub> SYSTEM

The tetradentate Schiff base (L) as primary ligand forms both 1:1 and 1:2 complexes with Cu(II), while the secondary ligand, dipeptides, DL-Alanyl Glycyl (R<sub>1</sub>) forms only 1:1 metal-ligand complex with copper. The curves of (F<sub>M</sub>), (F<sub>L</sub>) and (F<sub>R</sub>) are shown in (Fig.5-7).

The initial percentage of free metal at pH = 3.2 is very less i.e. 3.6 which indicates the maximum consumption of metal ions in the complex formation at this pH. It sharply decreases up to pH 5.2 after which the concentration remains constant indicating the maximum complexation in the pH- range 3.2 to 5.2. The percentage of free ligands L and R<sub>1</sub>, though extremely low (~0.03%) found to increase with increasing pH. The percentage of F<sub>L</sub> and F<sub>R</sub> reaches to 6 and 0.64 percent respectively at pH 5.5 and then remains constant. Due to the dissociation of excess ligands present in the system increase in free ligand concentrations.

The values of  $\beta_{111}$ ,  $\beta_{20}$ ,  $\beta_{02}$ ,  $K_L$ ,  $K_R$ ,  $K_r$  and  $\Delta \log K$  are presented in (Table. 2). These values express relative stabilities of binary and ternary complexes.

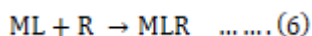
The comparative study of  $\beta_{111}$  with the product of  $\beta_{20}$  and  $\beta_{02}$  i.e.  $K_R$  of the system confirm formation of ternary complexes over binary complexes of primary as well as secondary ligands. Since they show positive value. Relatively higher positive value of  $K_R$  than  $K_L$  reveals greater stability of ternary complexes with respect to the binary complexes of secondary ligand than that of primary ligand.

The binary 1:1, Cu(II)-L and Cu(II)- $R_1$  complexes are stable than ternary complex confirm by the negative  $\Delta \log k$  value. Further negative  $\Delta \log k$  value shows higher stability of binary complexes than the ternary one and may be due to the reduced number of co-ordination sides.

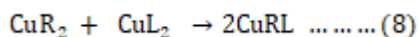
To understand the mechanism and extent of formation of ternary complex at different pH, the species distribution curves are most useful. The speciation diagram obtained for the system Cu (II)-L- $R_1$  is presented in (Fig. 3)

From the curve  $C_9$ , it can be observed that the initial concentration of ternary species is very less i.e. 5.54 percent. Above this pH, it increases very sharply to attain maximum value at pH 5.2 and then remains constant at about 93 percent for the whole pH range up to pH 7.2. Thus more than 93 percent metal ion is present in the form of ternary complex at pH 7.2.

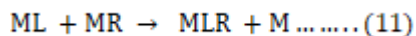
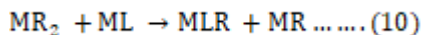
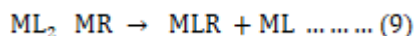
The reactions 1b and 2b represent the formation of HL( $C_2$ ) and R( $C_5$ ). There is continuous decrease in HL( $C_2$ ) and R ( $C_5$ ) with increasing pH indicate the formation of Cu(II)- L-R by reaction (5). It can be seen from the (Fig.3) that the concentration of HL is 93% and that of R is about 4% at the initial pH 3.2 which sharply decrease up to pH 5.2. The ternary complex takes place to the maximum extent i.e. up to 92% in the same pH range which supports the possibility of reaction (5). The formation of CuLR in the present investigation is also possible by equilibria



The another way of characterizing the ternary complexes is by following disproportion reactions



If primary ligand (L) and secondary ligand (R) form 1:1 and 1:2 complexes individually with the metal then disproportionation reaction is possible. Other disproportionation reactions are



The reaction 9 correspond to the system consisting of one ligand which forms only 1:1 complex and reaction 10 correspond to ligand which forms both 1:1 and 1:2 complexes. The reaction 11 represent the system containing primary and secondary ligands which form only 1:1 binary complexes of metal .This reaction is possible in sufficient concentration of ML and MR.

In addition to this, ternary complex may be formed by reaction 7. Since concentration of MR ( $C_8$ ) decreases from 90 to 6.2%. However disproportionation reaction (11) cannot be ruled out. Since in the present system, primary ligand (L) forms both 1:1 as well as 1:2 complexes, while the secondary ligand(R)) forms only 1:1 complex, the formation of MLR is also possible by the disproportionation reaction (9).

**ii). Cu (II)-L (R<sub>2</sub>) SYSTEM**

In this system the primary ligand (L) and secondary ligand (R<sub>2</sub>) form 1:1 and 1:2 metal ligand binary complexes. The species distribution diagram of this system (Fig.4). Show considerably high initial percentage i.e.77% of CuLR<sub>2</sub> species at pH 3.2. The initial percentage of the ternary complex of Cu(II)-L-R<sub>1</sub> system was comparatively very less. This observation indicates that the formation of ternary complex with Glycyl-Valine as a secondary ligand is more favorable at lower pH region and also it is stable. The curve C<sub>10</sub> representing the formation of ternary complex reaches to maximum at pH 4.2 i.e. 97 percent and then attains the value of more than 98% Such a high stability of the 1:1:1 complex may be attributed to the more ligational property of the secondary ligand which forms both 1:1 and 1:2 metal complexes. Initial percentage values of HL and R (C<sub>2</sub> and C<sub>5</sub>)



are 4.6 and 16.63 respectively, decrease to nearly zero. The reaction (5) indicates ternary complex formation.

In addition with reaction (5) there is a possibility of formation of 1:1:1 complex by reaction (6). This can be justified from the observation that the concentration of R and ML decrease to minimum in the same pH region where ternary complex formation possible. The ternary complex formation by the disproportionation reaction (11). Since the concentration of ML and MR decreases to negligible value in this pH range. In this system the percentage distribution curves of free metal ( $F_M$ ) and free ligands  $F_L$ ,  $F_R$  (Fig8-9) show exactly similar trend as in case of Cu(II)-L-R<sub>1</sub> system.

From (Table.2) it can be found that the  $\beta_{111}$  value of Cu(II)-L-R<sub>2</sub> system is considerably higher than that of Cu-L-R<sub>1</sub> system. Comparison of these values suggests that the ternary complex of the system containing Glycyl-Valine as a secondary ligand are highly stable than the complex of the system, containing Alanyl-Glycyl.

The  $\beta_{111}$  value of Cu(II)-L-R<sub>2</sub> system is comparable with  $\beta_{20}$  indicate the equal possibility of formation of 1:1:1 and binary complexes of primary ligand. Whereas, the lower value of  $\beta_{02}$  than  $\beta_{111}$  indicate formation of ternary complex over the binary complex of secondary ligand which may be considered as additional support to the observed more stability of the ternary complex. In case of Cu(II)-L-R<sub>1</sub> system the  $\beta_{111}$  value was notably less than  $\beta_{20}$ . which also supports the observation that the ternary complex formed in this system at initial pH is very less more over similar to the previous system the higher values of  $\beta_{111}$  compared to  $\beta_{20}$  in this system indicate the preferential formation of ternary complex over binary complex of secondary ligand.

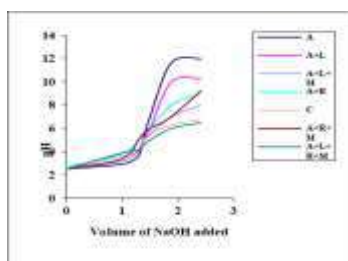
It is seen from the (Table.2) that the  $k_R$  and  $k_L$  values in Cu(II)-L-R<sub>2</sub> system are considerably higher than those in the system Cu(II)-L-R<sub>1</sub>. Comparison of these values show additional stability of ternary complex in Cu(II)-L-R<sub>2</sub> system. The  $\Delta \log k$  values in both the systems are negative. The lower values of  $\Delta \log k$  towards negative side in Cu(II)-L-R<sub>2</sub> system indicate the extra stabilization of ternary complex of this system than the previous system

**Table1. pK and logk values of Cu(II) chelates of ligand and dipeptides**

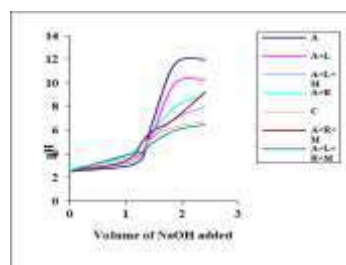
Ligands(L) / peptide(R <sub>1</sub> , R <sub>2</sub> )	pK <sub>1</sub>	pK <sub>2</sub>	pK <sub>3</sub>	logk <sub>1</sub>	Logk <sub>2</sub>	logβ
Ligand (L)	2.93	9.97	10.59	10.15	7.97	18.12
DL-Alanyl Glycyl(R <sub>1</sub> )	4.13	8.45	-	6.05	-	6.05
Glycyl-DL-Valine(R <sub>2</sub> )	4.33	8.59	-	7.62	4.87	12.49

**Table. 2 Parameters of formations of mixed ligand complexes of in 60 %( v/v) THF-water medium at Temp: 30 °C and ionic strength μ= 0.1 M NaClO<sub>4</sub> Cu(II) with primary ligand (L) and dipeptides (R<sub>1</sub>,R<sub>2</sub>)**

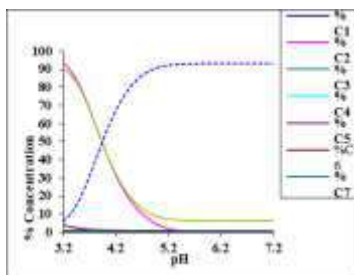
Dipeptides	logβ <sub>20</sub>	β <sub>02</sub>	β <sub>11</sub>	k <sub>L</sub>	k <sub>R</sub>	k <sub>r</sub>	Δlogk
DL-Alanyl glycine(R <sub>1</sub> )	18.13	6.05	12.65	2.49	6.6	-	-3.55
Glycyl valine(R <sub>2</sub> )	18.13	12.49	17.02	6.87	9.40	0.15	-0.75



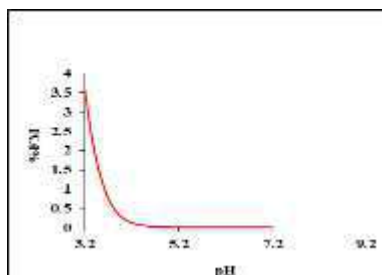
**Figure. 1 pH titration curve of Ternary Complex formation of L-R<sub>1</sub>-Cu (II) system in 60%THF-water at 30°C**



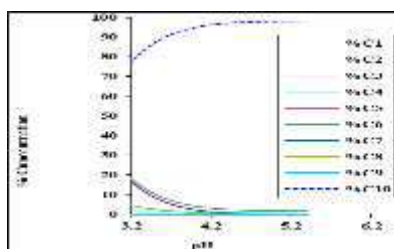
**Figure 2 pH titration curve of Ternary Complex formation of L-R<sub>2</sub>-Cu(II) system in 60%THF-water at 30°C**



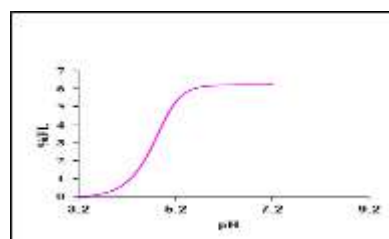
**Figure 3 Percentage distribution curves of L-R<sub>2</sub>-Cu(II) system**



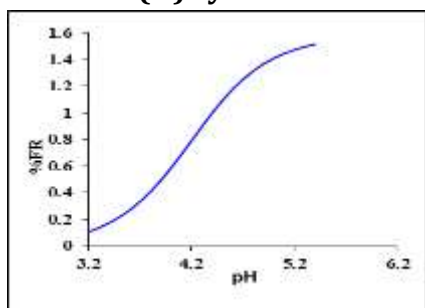
**Figure 5 Plot of % F<sub>M</sub> vs pH in LR<sub>1</sub>Cu System.**



**Figure 4 Percentage distribution curves of L-R<sub>2</sub>Cu(II) system**



**Figure 6. Plot of % F<sub>L</sub> vs pH in LR<sub>1</sub>Cu System.**



**Figure 7 Plot of % F<sub>R</sub> vs pH in LR<sub>2</sub>Cu System.**

## VI. CONCLUSION

Present investigation include the study of - Ternary complex formation equilibria of Cu(II) metal involving tetra dentate Schiff base ligand (L) 4 - hydroxy - 3 ( 1 - { 2 - [ ( 4 - chlorobenzylideneamino) phenylimino) ethyl) - 6 methyl-2H- Pyran2-one as primary ligand and DL- Alanyl Glycyl (R<sub>1</sub>), Glycyl- DL-Valine(R<sub>2</sub>).

Ternary complex formation takes place in a stepwise method. It is seen from the (Table.2) that the  $K_R$  and  $K_L$  values in Cu(II)-L-R<sub>2</sub> system are considerably higher than those in the system Cu(II)-L-R<sub>1</sub>. Comparison of these values show additional stability of ternary complex in Cu(II)-L-R<sub>2</sub> system. The  $\Delta \log k$  values in both the systems are negative. The lower values of  $\Delta \log k$  towards negative side in Cu(II)-L-R<sub>2</sub> system indicate the extra stabilization of ternary complex of this system than the previous system.

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## Chapter -18

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### Investigation of organic compounds with -NH<sub>2</sub> and -COOH groups as anti-corrosion agents

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#### Abstract –

Metal oxidation is a gradual process that is called corrosion. Metal corrosion occurs in a variety of media. Some organic substances function as corrosion inhibitors. The effectiveness of several substances, including p-nitroaniline, m-nitroaniline, p-toluidine, benzoic acid, salicylic acid, phthalic acid, and cinnamic acid, was investigated through a number of trials. The values indicate that organic molecules hinder the action of corrosion.

**Keywords:** Inhibition efficiency, organic compounds, -NH<sub>2</sub>, -COOH functional group.

#### INTRODUCTION:

According to the references, organic molecules with -NH<sub>2</sub> or -COOH groups slow down metal corrosion. This could be caused by the oxidation process being slowed down or by the development of a surface layer. The different organic compounds with -NH<sub>2</sub> and -COOH groups are utilized to investigate the effectiveness of organic compounds' inhibition. The outcomes are really intriguing.

#### EXPERIMENTAL:

To research the effectiveness of organic chemicals' inhibition. The easy experiments were completed. The beakers in this experiment were designated 1 through 12, and the following were added: 1, 2, 3, 4, 25 ml 0.5NHNO<sub>3</sub>, 0.5NHCl in beakers 5, 6, 7, and 8, and 0.5N H<sub>2</sub>SO<sub>4</sub> in beaker number 9, 10, 11, 11, and 12. Different organic compounds, such as p-nitroaniline, m-nitroaniline, and p-toluidien, as well as acids, such as benzoic acid, salicylic acid, cinnamic acid, and pthalic acid, were introduced to each beaker. The temperature was noted.

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$$I. E. = \frac{W_u - W_i}{W_u} \times 100$$

Where

IE=Inhibition efficiency, Wi=Weight loss of metal in inhibitor solution, Wu=Weight loss of metal in inhabited solution (control) relation. Inhibition efficiency was determined. The inhibition efficiency of organic compound in different oxidizing medium were used for comparisons.

## RESULTS AND DISCUSSION

### Regarding organic bases-refer table No.1

All organic acids are shown to be working as inhibitors by inhibition efficiency data; however, while the inhibition action is still present in the same proportion, not all organic bases exhibit the same inhibition efficiency.

The inhibition efficiency table shows that p-Toludien is a potent inhibitor of corrosion. In the medium of nitric acid, its inhibitory efficiency value is 84.38. The value of inhibitory efficiency is 34.18 in HCl medium and 28.41 in H<sub>2</sub>SO<sub>4</sub> media. Compared to m-nitro aniline and p-toludiene, the inhibition efficiency value of p-nitro aniline is weaker. When compared to organic bases having methyl radical, the inhibitory efficacy of organic bases including nitro group is lower. The nitro group-containing organic bases have an electron density directed towards them.

**Regarding organic acids refer Table No.2**

In order to investigate inhibitory efficacy, several acids are employed. Salicylic acid is an organic acid with an OH group, benzoic acid is a simple acid, cinnamic acid is an unsaturated acid, and phthalic acid is a dicarboxylic acid. The efficiency of inhibition varied there. The results show that phthalic acid has a low level of inhibitory efficacy in HNO<sub>3</sub>, HCl, and H<sub>2</sub>SO<sub>4</sub>. Cinnamic acid's inhibitory efficiency is higher, and the data shows that this efficiency is also dependent on the type of oxidizing media. In the case of hydrochloric acid medium table 2–6, the anti-oxidizing activity of various organic acids is more pronounced.

If the inhibitory efficacy of various bases and acids in an oxidizing agent is compared. It has been discovered that bases reduce corrosion more than organic chemicals do.

**Table1: Effect of organic bases on corrosion of steel in 0.5 N Nitric acid media**

Beaker No	Organic compound	Initial weight	Final weight	Loss in Weight	%loss in weight	Inhibition efficiency
1	Control	1.358	1.134	0.224	16.5	0
2	P-Nitroaniline	1.37	1.181	0.189	13.8	15.63
3	m-Nitroaniline	1.371	1.201	0.17	23.6	24.11
4	p-toluidine	1.098	1.063	0.035	3.5	84.38

**Table-2 : Effect of organic bases on corrosion of steel in 0.5N sulphuric acid media**

Beaker No	Organic compound	Initial weight	Final weight	Loss in Weight	%loss in weight	Inhibition efficiency
5	Control	1.115	0.939	0.176	15.7	-
6	P-Nitroaniline	1.120	0.952	0.168	15.0	4.50
7	m-Nitroaniline	1.084	0.924	0.160	14.7	9.09
8	p-toluidine	1.144	1.018	0.126	10.8	28.41

**Table-3: Effect of organic bases on corrosion of steel in 0.5N Hydrochloric acid media**

Beaker No	Organic compound	Initial weight	Final weight	Loss in Weight	% loss in weight	Inhibition efficiency
9	Control	1.171	1.013	0.158	13.5	-
10	m-Nitro aniline	1.047	0.917	0.130	12.4	17.72
11	p-Nitro aniline	1.099	0.979	0.120	10.9	24.05
12	p-toluidine	1.166	1.069	0.104	8.9	34.18

**Table-4 :Effect of organic acid on corrosion of steel in 0.5 N Nitric acid media**

Organic compound	Initial weight	Final weight	Loss in weight	%loss in weight	Inhibition efficiency
Benzoic acid	1.172	0.971	0.201	17.1	10.5
Salicylic acid	1.118	0.921	0.197	17.6	12.05
Cinnamic acid	1.202	1.01	0.192	15.9	14.20
Phthalic acid.	1.263	1.055	0.208	16.5	7.14

**Table5: Effect of organic acid on corrosion of steel in 0.5 N sulphuric acid media**

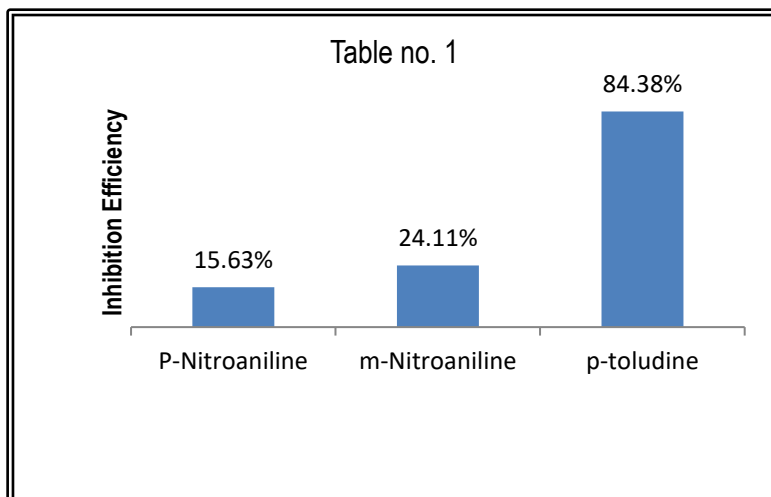
Organic compound	Initial weight	Final weight	Loss in weight	%loss in weight	Inhibition efficiency
Benzoic acid	1.274	1.104	0.170	13.3	3.40
Salicylic acid	1.252	1.080	0.172	13.7	2.27
Cinnamic acid	1.239	1.404	0.165	13.3	6.25
Phthalic acid.	1.216	1.045	0.171	14.0	2.84

**Table 6: Effect of organic acid on corrosion of steel in 0.5 N Hydrochloric acid media**

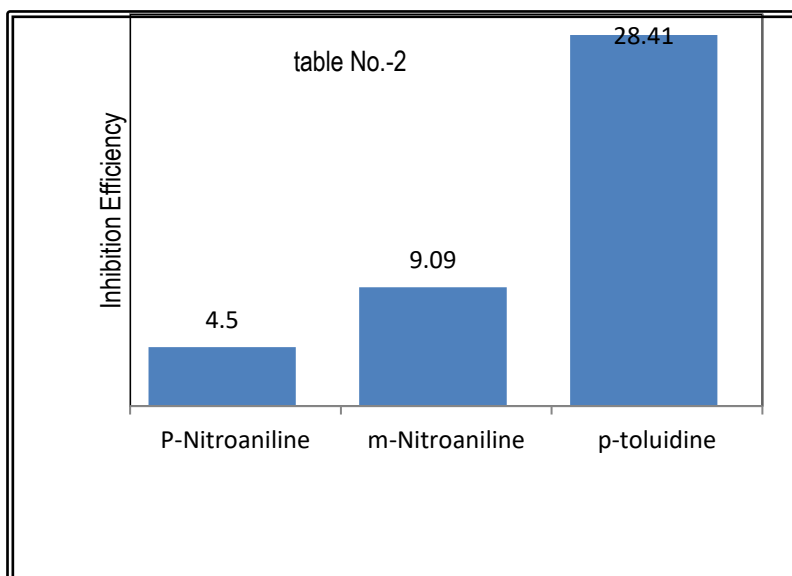
Organic compound	Initial weight	Final weight	Loss in Weight	%loss in weight	Inhibition efficiency
Benzoic acid	1.279	1.134	0.145	11.5	8.22
Salicylic acid	1.259	1.11	0.149	11.8	5.69
Cinnamic acid	1.252	1.117	0.135	10.7	14.55
Phthalic acid.	1.192	1.042	0.150	12.6	5.06



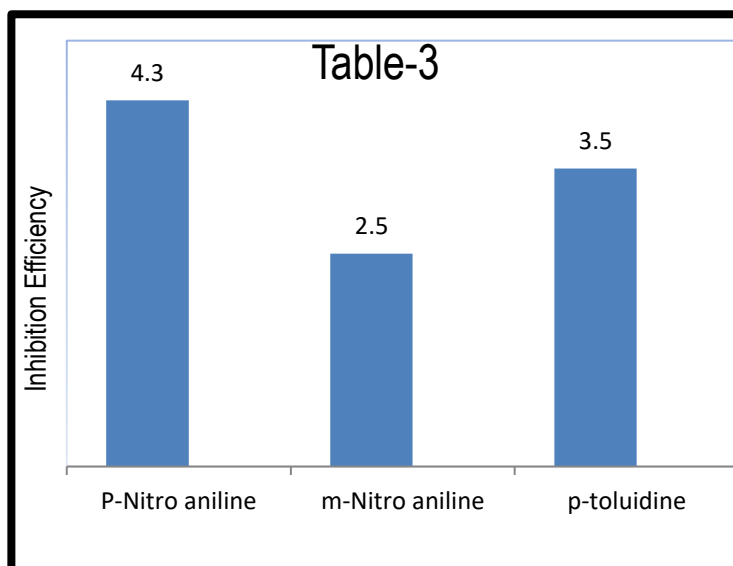
**Table1: Effect of organic bases on corrosionsteel in 0.5 N Nitric acid media**



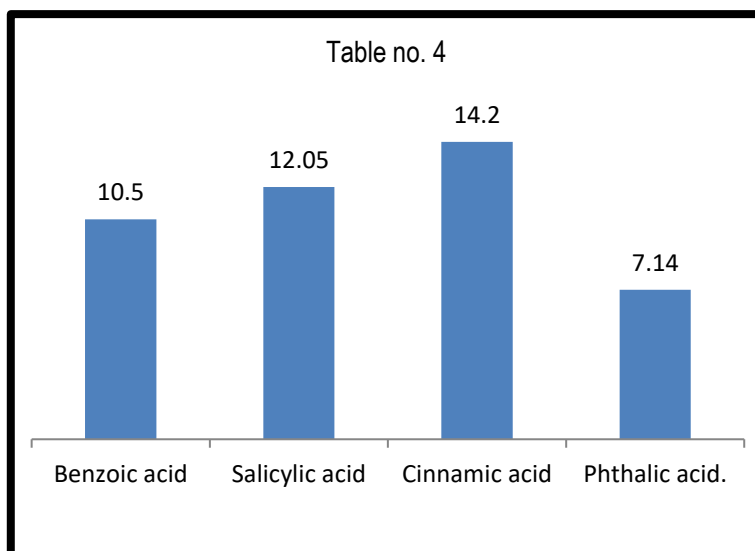
**Table-2 : Effect of of steel in 0.5N sulphuric acid media**



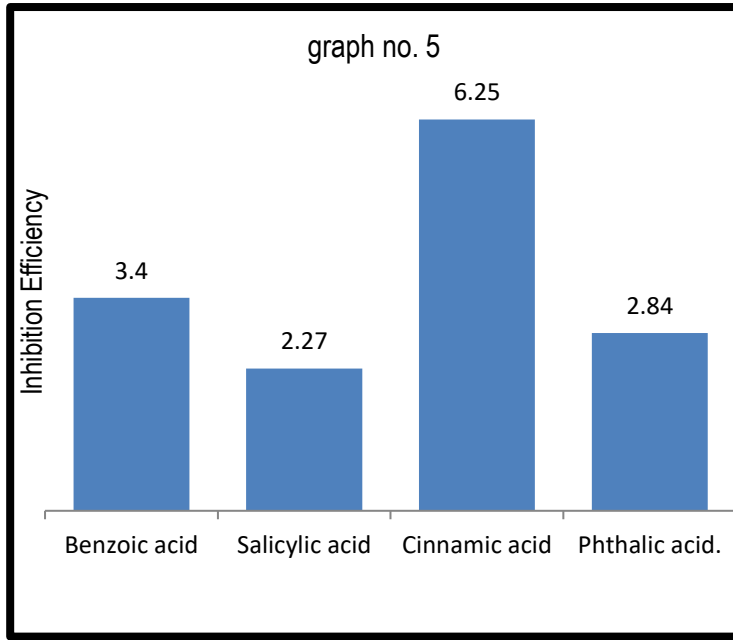
**Table-3: Effect of organic bases on corrosion of steel in 0.5N Hydrochloric acid media**



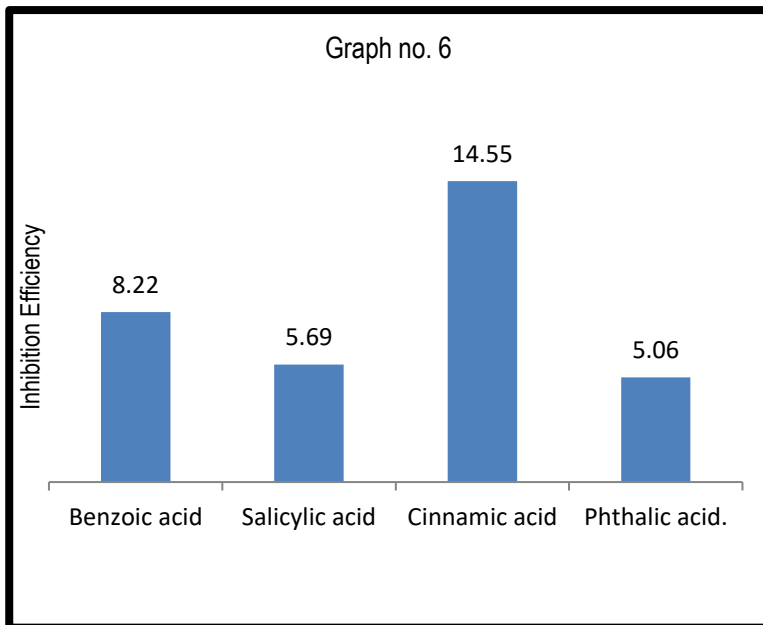
**Table-4 :Effect of organic acid on corrosion of steel in 0.5 N Nitric acid media**



**Table5: Effect of organic acid on corrosion of steel in 0.5 N sulphuric acid media**



**Table 6: Effect of organic acid on corrosion of steel in 0.5 N Hydrochloric acid media**



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## Chapter -19

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### On Bi-Interior Ideals of $\Gamma$ -semihyperring

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#### Abstract:

The concept of  $\Gamma$ -semihyperring is a generalization of semiring, semihyperring and a  $\Gamma$ -semiring. In this paper, bi-interior ideals are characterized with respect to different ideals of  $\Gamma$ -semihyperring. Some important results are proved in this respect.

**Keywords:** Bi-ideals, Interior ideals and regular  $\Gamma$ -semihyperring.

#### 1. Introduction:

In 1964, Nobusawa [1] introduced  $\Gamma$ -ring as a generalization of ternary rings. Barends [2] weakened the conditions in the definitions of  $\Gamma$ -ring in the sense of Nobusawa. The notion of  $\Gamma$ -semiring was introduced by Rao [3, 4] as generalization of  $\Gamma$ -ring as well as of semiring. Also, S. Krishnamoorthy and Doss [5] introduced the notion of commuting regular  $\Gamma$ -semiring.

The French mathematician Marty [6] was introduced algebraic hyper structure represent the natural extension of classical algebraic structure. Davvazet. Al. [7, 8] introduced the notion of a  $\Gamma$ -semihypergroup as generalization of a semi hyper group. In [9-11], S. O. Dehkordi and B. Davvaz studied the notion of a  $\Gamma$ -semi hyperring as a generalization of semiring, a generalization of a semi hyperring, and a generalization of a  $\Gamma$ -semiring.

In this paper many classical notions of  $\Gamma$ -semiring [12] and  $\Gamma$ -hyperring [10] have been extended to  $\Gamma$ -semihyperring. In section-2 some preliminaries are given which are useful for us to take intensive idea about paper while reading. In section-3 we introduced the concept of regular (strongly regular)  $\Gamma$ -semi hyperring with some examples and results. In section-4 we introduced the notion invertible sets (elements) in  $\Gamma$ -semihyperring; idempotent  $\Gamma$ -semihyperring and we come across some important results. In section-5 conclusion is made of present paper.

## 2. Preliminaries:

Here we present some useful definitions further reader is requested to refer [10].

**Definition 2.1.:** Let  $H$  be a non empty set and  $o : H \times H \rightarrow \wp^*(H)$  be a hyperoperation, where  $\wp^*(H)$  is the family of all non-empty subsets of  $H$ . The couple  $(H, o)$  is called a hypergroupoid.

For any two non-empty subsets  $A$  and  $B$  of  $H$  and  $x \in H$ ,  $AoB = \cup_{a \in A, b \in B} aob$ ,  $Ao\{x\} = Aox$  and  $\{x\}oA = xoA$ .

**Definition 2.2.:** A hyper groupoid  $(H, o)$  is called a semihypergroup if for all  $a, b, c$  in  $H$  we have  $(aob)oc = ao(boc)$ .

In addition, if for every  $a \in H$ ,  $aoH = H = Hoa$ , then  $(H, o)$  is called a hyper group (for more details of hyper group, semihypergroup see [13-15]).

In [16], Vougiouklis studied the notion of semihyperring in form in which sum and product are hyperoperations; also see [16].

**Definition 2.3.:** A semihyperring is an algebraic structure  $(R, +, \cdot)$  which satisfies the following properties: (1)  $(R, +)$  is a commutative semihypergroup; that is,  $(x + y) + z = x + (y + z)$  and  $x + y = y + x$  for all  $x, y, z \in R$ . (2)  $(R, \cdot)$  is semihypergroup. (3) the hyperoperation  $\cdot$  is distributive with respect to hyperoperation  $+$ ; that is  $x \cdot (y + z) = x \cdot y + x \cdot z$ ,  $(x + y) \cdot z = x \cdot z + y \cdot z$  for all  $x, y, z \in R$ . (4) the element  $0 \in R$  is an absorbing element; that is  $x \cdot 0 = 0 \cdot x = 0$  for all  $x \in R$ .

**Definition 2.4.:** A semihyperring  $(R, +, \cdot)$  is called commutative if and only if  $a \cdot b = b \cdot a$  for all  $a, b \in R$ .

The concept of  $\Gamma$ -semihyperring is studied and introduced by Dehkordi and Davvaz[9-11].

**Definition 2.5.:** Let  $R$  be a commutative semihypergroup and  $\Gamma$  be a commutative group. Then,  $R$  is called a  $\Gamma$ -semihyperring if there is a map  $R \times \Gamma \times R \rightarrow \wp^*(R)$  (images to be denoted by  $a\alpha b$  for all  $a, b \in R$  &  $\alpha \in \Gamma$ ) and  $\wp^*(R)$  is the set of all non-empty subsets of  $R$  satisfying the following conditions:

1.  $a\alpha(b + c) = a\alpha b + a\alpha c$ .
2.  $(a + b)\alpha c = a\alpha c + b\alpha c$
3.  $a(\alpha + \beta)c = a\alpha c + a\beta c$
4.  $a\alpha(b\beta c) = (a\alpha b)\beta c$ ,  
for all  $a, b, c \in R$  and for all  $\alpha, \beta \in \Gamma$ .

In above definition, if  $R$  is a semigroup, then  $R$  is called a multiplicative  $\Gamma$ -semihyperring.

**Definition 2.6.:**-A  $\Gamma$ -semihyperring  $R$  is called commutative if  $a\alpha b = b\alpha a$ , for all  $a, b \in R$  and  $\alpha \in \Gamma$ .

**Definition 2.7.:**-A  $\Gamma$ -semihyperring  $R$  with zero, if there exists  $0 \in R$  such that  $a \in a + 0$  and  $0 \in 0\alpha a, 0 \in a\alpha 0$  for all  $a \in R$  and  $\alpha \in \Gamma$ .

Let  $A$  and  $B$  be two non-empty subsets of a  $\Gamma$ -semihyperring  $R$  and  $x \in R$  then

$$A + B = \{x \mid x \in a + b, a \in A, b \in B\},$$

$$A\Gamma B = \{x \mid x \in a\alpha b, a \in A, b \in B, \alpha \in \Gamma\}.$$

**Definition 2.8.:**-A non empty subset  $R_1$  of  $\Gamma$ -semihyperring  $R$  is called a  $\Gamma$ -sub semihyperring if it is closed with respect to the multiplication and addition, that is,  $R_1 + R_1 \subseteq R_1$  and  $R_1\Gamma R_1 \subseteq R_1$ .

**Definition 2.9.:**-A right (left) ideal  $I$  of a  $\Gamma$ -semihyperring  $R$  is an additive sub semihypergroup of  $(R, +)$  such that  $I\Gamma R \subseteq I$  ( $R\Gamma I \subseteq I$ ). If  $I$  is both right and left ideal of  $R$ , then we say that  $I$  is a two sided ideal or simply an ideal of  $R$ .

Also to study examples on  $\Gamma$ -semihyperring and to study the notions of Noetherian, Artinian, simple  $\Gamma$ -semihyperring, regular relation on  $\Gamma$ -semihyperring, see[10].

### 3. Bi-Interior Ideals of $\Gamma$ -semihyperring:

In this section, we are going to introduce the notion of bi-interior ideal as a generalization of bi-ideal and interior ideal of  $\Gamma$ -semihyperring and studied some important properties in this respect.

**Definition 3.1.:**-A non-empty subset  $R$  of a  $\Gamma$ -semihyperring  $R$  is said to be bi-interior ideal of  $R$  if  $R\Gamma B\Gamma R \cap B\Gamma R\Gamma B \subseteq B$ .

**Definition 3.2.:**-A  $\Gamma$ -semihyperring  $R$  is said to be bi-interior simple if  $R$  has no bi-interior ideals other than  $R$  itself.

**Theorem 3.3.:**-Let  $R$  be a  $\Gamma$ -semihyperring. Then arbitrary intersection of bi-interiors ideals of  $\Gamma$ -semihyperring is a bi-interior ideal of  $\Gamma$ -semihyperring, provided that the intersection is non-empty.

**Proof:** -Let  $R$  be a  $\Gamma$ -semihyperring and  $B_i$  be a bi-interior ideals of  $\Gamma$ -semihyperring, where  $i \in \Delta$ . Then to show  $\bigcap_{i \in \Delta} B_i = B$  be a bi-interior ideal of  $\Gamma$ -semihyperring.

Now,  $R\Gamma(\bigcap_{i \in \Delta} B_i)\Gamma R \cap (\bigcap_{i \in \Delta} B_i)\Gamma R\Gamma(\bigcap_{i \in \Delta} B_i) \subseteq R\Gamma B_i\Gamma R \cap B_i\Gamma R\Gamma B_i \subseteq B_i$ , for all  $i \in \Delta$ .

Therefore we get,  $R\Gamma(\bigcap_{i \in \Delta} B_i)\Gamma R \cap (\bigcap_{i \in \Delta} B_i)\Gamma R\Gamma(\bigcap_{i \in \Delta} B_i) \subseteq \bigcap_{i \in \Delta} B_i = B$ . Hence the proof.

**Theorem 3.4.**Let  $R$  be a  $\Gamma$ -semihyperring. Then every left ideal (right ideal, ideal) is a bi-interior of  $R$ .

**Proof:** -Let  $R$  be a  $\Gamma$ -semihyperring and  $L$  be a left ideal of  $\Gamma$ -semihyperring. Then  $R\Gamma L\Gamma R \cap L\Gamma R\Gamma L \subseteq R\Gamma L\Gamma R \cap L \subseteq L$ . Hence the proof.

**Theorem 3.5.**Let  $R$  be a  $\Gamma$ -semihyperring. Then intersection of a right ideal and a left ideal of  $R$  is a bi-interior ideal of  $R$ .

**Proof:**-Proof easily hold by Theorems 3.3 and 3.4.

**Theorem 3.6.**Let  $R$  be a  $\Gamma$ -semihyperring and  $B$  be any subset of  $R$ . Then  $B\Gamma R$  and  $R\Gamma B$  are bi-interiors ideals of  $R$ .

**Proof:** - Let  $R$  be a  $\Gamma$ -semihyperring and  $B$  be any subset of  $R$ . Then it clear that  $B\Gamma R$  is a right ideal of  $R$  and  $R\Gamma B$  is a left ideal of  $R$ . Therefore by Theorem 3.4, we get  $B\Gamma R$  and  $R\Gamma B$  are bi-interiors ideals of  $R$ .

**Corollary 3.7.**Let  $R$  be a  $\Gamma$ -semihyperring and  $B$  be a bi-interior ideal of  $R$ . Then  $B\Gamma R$  and  $R\Gamma B$  are bi-interiors ideals of  $R$ .

**Theorem 3.8.**The intersection of a bi-interior ideal  $B$  of  $\Gamma$ -semihyperring  $R$  and a  $\Gamma$ -subsemihyperring  $A$  of  $R$  is a bi-interior ideal of  $R$ .



**Proof:** Let  $B$  be a bi-interior ideal of  $\Gamma$ -semihyperring  $R$  and  $A$  be a  $\Gamma$ -subsemihyperring of  $R$ . Suppose  $C = B \cap A$ . Then  $C$  is a  $\Gamma$ -subsemihyperring of  $R$ ,  $C \subseteq A$  and  $C\Gamma A\Gamma C \subseteq A\Gamma A\Gamma A \subseteq A \dots (1)$ .  $C\Gamma A\Gamma C \subseteq B\Gamma A\Gamma B \subseteq B\Gamma R\Gamma B$ ,  $R\Gamma C\Gamma R \subseteq R\Gamma B\Gamma R$ . It gives that  $C\Gamma A\Gamma C \cap R\Gamma C\Gamma R \subseteq B\Gamma R\Gamma B \cap R\Gamma B\Gamma R \subseteq B$ ,  $C\Gamma A\Gamma C \cap R\Gamma C\Gamma R \subseteq A$  from (1). Therefore  $C\Gamma A\Gamma C \cap R\Gamma C\Gamma R \subseteq B \cap A = C$ . Hence the theorem.

**Theorem 3.9.** Let  $A$  and  $C$  be a  $\Gamma$ -subsemihyperring of  $\Gamma$ -semihyperring  $R$  and  $B = A\Gamma C$ . If  $A$  is the left ideal then  $B$  is a bi-interior ideal of  $R$ .

**Proof:** Let  $A$  and  $C$  be a  $\Gamma$ -subsemihyperring of  $\Gamma$ -semihyperring  $R$  and  $B = A\Gamma C$ . Suppose  $A$  is the left ideal of  $R$ . Then  $B\Gamma R\Gamma B = A\Gamma C\Gamma R\Gamma A\Gamma C \subseteq A\Gamma C = B$ . Therefore we get,  $B\Gamma R\Gamma B \cap R\Gamma B\Gamma R \subseteq B\Gamma R\Gamma B \subseteq B$ . Hence the theorem.

**Corollary 3.10.** Let  $A$  and  $C$  be a  $\Gamma$ -subsemihyperring of  $\Gamma$ -semihyperring  $R$  and  $B = C\Gamma A$ . If  $C$  is the right ideal then  $B$  is a bi-interior ideal of  $R$ .

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## Chapter -20

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### Effect of various doping of metal ions and rare earth ions on the properties of Ni-Zn ferrite materials: A review

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#### **Abstract:**

Substitution, addition or incorporation of different metal ions/rare earth ions shows their effect on the structural, magnetic, electric, optical and thermal properties of the ferrite materials. Every ions having tendency to occupy their own preferred sites, hence due to this site distribution, basic ferrite composition shows totally different properties. The significant change in the structural and magnetic properties makes these ferrite materials technologically, commercially and bio-medically applicable in different fields.

**Keywords:** Ferrites, Doping, Metal ions, Rare earth ions

#### **Introduction**

Ferrites are magnetic materials in which oxygen anions and metal cations arranged themselves to create crystal lattice. Based on their structure, ferrites are classified into four categories such as - Garnet, Ortho-ferrites, Hexagonal ferrites and Spinel ferrites. In spinel ferrite unit cell, there are 8 divalent metal ions, 16 ferric ions and 32 oxygen ions are present. Here oxygen anion forms closed packed face centred cubic (FCC) structure which contains two types interstitial sites namely tetrahedral A-site and octahedral B- sites. One eight of the tetrahedral sites and one half of the octahedral sites are occupied by the cations. [1,2] Based on divalent and trivalent cation distribution the spinel ferrite is classified into three categories such as, normal spinels - in which tetrahedral sites are occupied by the divalent cations and octahedral sites are occupied by trivalent cations, this type of

distribution is known as normal spinel. Inverse spinels - half of the octahedral sites and tetrahedral sites occupied by trivalent cations and remaining half octahedral sites occupied by divalent cations, this type of cation distribution is known as inverse spinel and third one is spinel – in which the divalent cations occupy both sides, this type spinel is known as mixed spinel. It contains general formula  $AB_2O_4$  where A represents divalent cations and B represents trivalent cations [3, 4]. Fig. 1 and 2 represents the classification of ferrites and the crystal structure of spinel ferrite. Cation and anion distribution in spinel ferrite shows interesting structural, magnetic, electrical, optical and thermal properties. These properties are changed according to the distribution of ions, site occupancy, their charge and nature. Site distribution of divalent, trivalent, tetravalent cations revealed the tremendous change in their structural, magnetic, electric, optical and thermal properties. The rare earth ions are also affecting to these properties of ferrite materials due to their large ionic radius. Rare earth ions have a tendency to occupy octahedral site and limited solubility in ferrites. In the ferrite composition the proper selection of rare earth ions are important to get desired properties which are very useful in different field [5, 6].

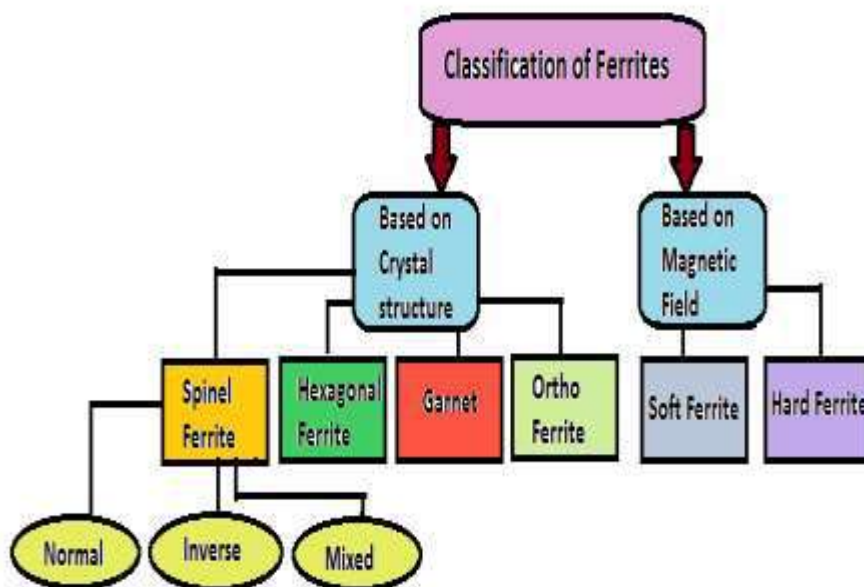
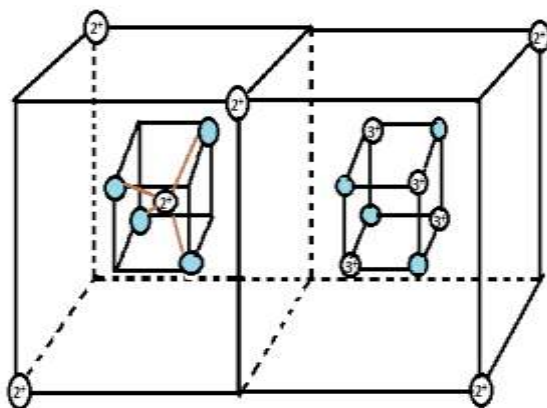


Fig.1 Types of Ferrites



**Fig.2 Spinel Structure**

### **Effect of various doping on the properties of Ni-Zn ferrites:**

The present work deals with the change and improvement in the properties due to various doping and incorporation of different ferrites with each other. Many researchers studied the mixed spinel ferrite with different properties such as nickel ferrite, zinc ferrite, copper ferrite, cobalt ferrite, manganese ferrites, magnesium ferrite, Indium ferrite, Lithium ferrites, Chromium ferrite, Cadmium ferrite also with rare earth ions substitution such as, Gadolinium (Gd), yttrium (Y), ytterbium (Yb) terbium (Tb), dysprosium (Dy), cerium (Ce), samarium (Sm), erbium (Er), neodymium (Nd), holmium (Ho), lanthanum (La), praseodymium (Pr), europium (Eu), thulium (Tm) and scandium (Sc) in these mixed spinel ferrites. It is known that with addition or substitution of very few amounts of these rare earth ions can alter the properties of parent ferrite composition. Many researchers now still are scrutinizing their work on ferrites, due to their applications in many technological and biological fields.

In 1953, Hastings and Corliss [7] first time incorporate the zinc with nickel to analyse the chemical and magnetic properties of Ni-Zn ferrite and found inverted magnetic structure with interesting properties. After the successful experimental result, the researchers are moved towards the mixed ferrite formation to get desire properties. The combination of more than two metallic and rare earth ions depicted tremendously different characteristics. Ravinder Kumar and

et.al [8] studied the cobalt doped Ni-Zn ferrite and found that the structural properties are increases with increase the amount of cobalt. S. J. Azhagushanmugam and et.al [9] observed that substitution of zinc in Ni-Co ferrites showed that the particle size, saturation magnetization and coercivity decreases where as lattice parameter increases with the increase in zinc concentration. A gradual increase in lattice constant has been observed with increasing cobalt content, in this system indicated agglomerated structures and increase in particle size and increase in particle size with increasing  $\text{Co}^{2+}$  content. A decrease in Curie temperature was observed with the increase in  $\text{Co}^{2+}$  due to the weakening of A-B super-exchange interactions with the incorporation of  $\text{Co}^{2+}$  content in the Ni-Zn nanoferrite matrix. R. S. Devan [10] studied the effect of Cobalt substitution on Ni-Cu ferrites, synthesized by the standard double sintering ceramic technique. Variation in grain size, grain size boundaries and porosity causes the resistivity, dielectric constant is inversely proportional to the resistivity, and it shows a decreasing trend with increasing cobalt content. The magneto-crystalline anisotropy increases with increased in cobalt content, which contributes the increase in saturation magnetization.

P. Venkata Srinivasa Rao and et.al [11] reported the Magnetic and Electrical Properties of Gd Doped Ni-Cu-Zn- $\text{Fe}_2\text{O}_4$  synthesized by using oxalic-based precursor technique. It is observed that at the certain temperature susceptibility falls to zero indicating the Curie temperature ( $T_c$ ) and ferrimagnetic samples are converted into paramagnetic sample at that temperature. The Curie temperature ( $T_c$ ) was observed to decrease with increasing Gd concentration and the dielectric constant is decrease at initial position when increasing frequency but then after it is constant at higher frequency. The M-H plots reveal the ferrimagnetic behaviour of nanocrystalline  $\text{Cu}^{2+}$  ions incorporated Ni-Zn spinel nano-ferrite samples. The structural, magnetic, electrical properties shows effect of Jahn Teller ion ( $\text{Cu}^{2+}$ ) in present Ni-Zn ferrite system, the overall,  $\text{Cu}^{2+}$  substitution weakens the A-B super-exchange interactions [12].

Namahari Kumar and et.al [13] reported on structural, optical and High temperature thermo electrical Power studies on Cu substituted Ni-Zn ferrite, substitution of copper in Ni-Zn ferrite system

decreases lattice parameter and increases the x-ray density, Optical studies UV-DRS is observe to decrease in wavelength with increase in Cu content and according to positive value Seedback coefficient, prepared sample indicated p-type semiconducting nature in high temperature region. The electrical conductivity shows decreasing with increasing in temperature showing a nature of the prepared ferrite samples from para and ferrimagnetic nature. M. Kalyan Raju [14] studied the FTIR spectra of copper substituted Nickel-Zinc ferrite within the range 300 to 1000  $\text{cm}^{-1}$ . Two significant absorption bands are observed in system and the change in the band position is due to the change in the  $\text{Fe}^{3+} - \text{O}^{2-}$  inter nuclear distances for the tetrahedral and octahedral sites, If the radius of the impurity ion is larger than the displaced ion then the bond length increases, decreases force constant. The frequency shift is due to replacement of  $\text{Ni}^{2+}$  with  $\text{Cu}^{2+}$  ions having larger ionic radius which effects  $\text{Fe}^{3+} - \text{O}^{2-}$  , larger ionic radius of  $\text{Cu}^{2+}$ (0.72 Å) than the displaced  $\text{Ni}^{2+}$  ion (0.69 Å) indicate the variation in elastic properties.

Q.J. Han [15] have synthesized  $\text{Cu}^{2+}$  substituted Ni-Zn Ferrite, and the cation distributions in the samples were estimated. The saturation magnetization of the samples increased with increasing Zn content, and decreased with temperature. There are four important factors which affect the cation distribution in spinel ferrites, first, there is a square potential barrier between a cation-anion pair, second the height and third width of the potential barrier is related to the ionization energy of the last ionized electron and fourth is the distance between neighbouring cations and anions. The fitted magnetic moments values are very close to the experimental results. The various error indicator parameters all lie within acceptable ranges and indicate that quantum mechanical method is effective for estimating the cation distributions in  $\text{Cu}^{2+}$ -doped Ni-Zn ferrites. N.D.Chaudhari et.al [16] studied  $\text{Li}^{1+}$ -substituted Ni-Zn ferrites and observed that initial permeability decreases with increasing  $\text{Li}^{1+}$  content. The magnitude of wall permeability is found higher than rotational permeability. From the thermal variation of initial permeability, the peak height decreases with increase in  $\text{Li}^{1+}$  content. Loss factor shows increasing trend with

addition of  $\text{Li}^{1+}$  and may be due to thermal randomization of domains. Chaudhari et.al [17] studied the variation of magnetic parameter in case of  $\text{Li}^{1+}$  substituted Ni-Zn ferrites. The saturation magnetization increases with  $\text{Li}^{1+}$  up to  $x = 0.175$  thereafter it decreases. Decreasing trend in YK angles indicated that the  $\text{Li}^{1+}$  does not cause additional canting. Coercive force and remanence ratio ( $M_r/M_s$ ) is found to increase with  $\text{Li}^{1+}$  content which may be due to increase of anisotropy constant.

Ebtesam E. Ateia and et.al [18] reported the effect of rare earth ions and  $\text{La}^{3+}$  ions on the Ni-Zn ferrites, they prepared sample by ceramic technique and some rare ions i.e. Er, Tb, Gd, Dy, and Sm are substitutes in Ni-Zn ferrites, these ions and  $\text{La}^{3+}$  ions highly affected the electrical and magnetic properties of composition. Er - substituted samples show minimum Curie temperature ( $T_c$ ), Er and Gd are ferromagnetic but other possess antiferromagnetic behaviour, hence highest magnetization is found in Er and Gd substituted samples. These rare ions will lead to the formation of the secondary phase on the grain boundaries and do not inter in spinel lattice of Ni-Zn ferrites.

Effect of  $\text{Al}^{3+}$  ions on Ni-Cu-Zn ferrites studied by M.M.Eltabey and et.al [19] and observed that with addition of  $\text{Al}^{3+}$  ions the value of lattice constant is decreased and porosity and grain size, saturation magnetization and initial permeability is increased but there is no particular effect on dc resistivity due to  $\text{Al}^{3+}$ . Improvement in magnetic properties of  $\text{Nd}^{3+}$  ions substituted Mn-Ni-Zn ferrites reported by M.M. Eltabey [20]. The study reveals that the  $\text{Nd}^{3+}$  ions shows effect on grain boundaries more than the grain, the initial permeability and curie temperature increases as the  $\text{Nd}^{3+}$  increases but saturation magnetization shows increasing trends up to 26% more than un-substituted sample. S.A. Mazen and et.al [21] reported their work on the comparative studies of the divalent  $\text{Zn}^{2+}$  and tetravalent  $\text{Ge}^{4+}$  ions on nickel ferrites, the lattice constant and magnetization is increased with  $\text{Zn}^{2+}$  and decreased with addition of  $\text{Ge}^{4+}$  ions. Due to intra and inter granular pores the porosity is increased with  $\text{Zn}^{2+}$  and slightly changed with of  $\text{Ge}^{4+}$ .

Rainer Hochschild and Hartmut Fuess [22] investigated effect of rare earth cations i.e. Pr, Nd, Eu, Ho, Tm, Lu on Ni-Zn ferrites with very



small substitution of iron ions indicated that the all rare earth ions composition shows secondary phase during sintering and finally they are disappeared in prepared ferrite system. Crystallite size of rare earth ion doped samples is slightly lower than the undoped samples; no single phase found in all samples, means the rare earth ions forms the ortho-ferrites and hexagonal ferrites. Secondary phased samples reveal the antiferromagnetic or paramagnetic in nature. R. Suresh [23] presented review on recent advancements of spinel ferrite based binary nano-composite photo catalysts in wastewater treatment. The spinel ferrites ( $MFe_2O_4$ , where, M is divalent metal ion) and their binary nanocomposites are used as Photo catalysts. This review Enlighten the optical, magnetic and electric properties of  $MFe_2O_4$  based binary nanocomposites. The binary nanocomposites such as  $MFe_2O_4$ /metal oxide,  $MFe_2O_4$ /carbon based materials,  $MFe_2O_4$ /polymer and  $MFe_2O_4$ /metals and  $MFe_2O_4$ /other metal compounds, show superior photo catalytic degradation efficiencies.

Dong-Yun Li et.al [24] studied the structural and magnetic properties of  $Dy^{3+}$  substituted Ni-Zn ferrites synthesized by sol gel auto combustion technique and observed that a reduction in crystal size whereas specific area enhanced. The magnetic parameters such as  $M_s$ ,  $H_c$  and  $M_r$  are found to increase initially with  $Dy^{3+}$  content thereafter they decrease. Haikui Zhu et.al [25] investigated the sintering, microstructure and magnetic properties of low temperature co-fired Ni-Cu-Zn ferrites with  $B_2O_3$  and  $WO_3$  additives which enhance the densification. They concluded that the appropriate addition of these additives can efficiently increase an initial permeability and saturation flux density whereas remanent flux density and coercive field strength reduces. B.Yan et al [26] studied the effect of  $Pr^{3+}$  substitution on microstructure, specific surface area and magnetic properties of  $Ni_{0.5}Zn_{0.5}Pr_xFe_2O_4$  nanoparticles synthesized using sol gel method and observed that as the  $Pr^{3+}$  content increases, the saturation magnetization, remanance magnetization and coercivity increases up to 0.05 and then decreases. Y. Koseoglu [27] investigated the structural and magnetic properties of  $Cr^{3+}$  doped Ni-Zn ferrite nano-particles prepared by surfactant assisted hydrothermal method and

characterized by XRD, FE-SEM, EDX, FTIR. Hysteresis curve exhibits the reduction in saturation magnetization and coercivity by  $\text{Cr}^{3+}$  ion substitutions.

Korkmaz [28] investigated the effect of rare earth (RE) metal ions  $\text{Eu}^{3+}$ ,  $\text{Tb}^{3+}$  and  $\text{Dy}^{3+}$  substitution of  $\text{Ni}_{0.4}\text{Cu}_{0.2}\text{Zn}_{0.4}\text{RE}_{0.02}\text{Fe}_{1.98}\text{O}_4$  ferrite nanoparticles by a sono-chemical method. He investigated the crystal structure, chemical bonding, morphology and magnetic characteristics of the Ni-Cu-Zn nano size ferrite by X-ray powder diffraction (XRD), Fourier – transform infrared spectroscopy (FT-IR), scanning electron microscopy (SEM) and vibrating sample magnetometry (VSM) and revealed that the rare earth substitution has a significant effect on magnetic properties of Ni-Cu-Zn nano-size ferrites.

Shinde et.al [29] studied the effect of  $\text{La}^{3+}$  substitution on structural and magnetic parameters of  $\text{Ni}_{0.7}\text{Cu}_{0.1}\text{Zn}_{0.2}\text{La}_x\text{Fe}_{2-x}\text{O}_4$  nano – ferrites synthesized by oxalate co-precipitation method and characterized by thermo- gravimetric and differential temperature analysis (TG-DTA), energy-dispersive X-ray analysis (EDAX)), X-ray diffraction (XRD) , Fourier transform infrared spectroscopy (FTIR), field – emission scanning electron microscopy (FE-SEM) and vibrating sample magnetometer (VSM) techniques. They observed that there is a decrease in saturation magnetization and magnetic moment of Ni-Cu-Zn ferrite as  $\text{La}^{3+}$  content increases. Jacobo and Bercoff [30] studied the structure and electromagnetic properties of  $\text{Y}^{3+}$ -substituted Ni-Zn spinels prepared by a sol-gel method.  $\text{Y}^{3+}$  substitution slightly reduces the average crystallite size and unit cell parameters, and it modifies the saturation magnetization, permeability, and permittivity. The dielectric constant decreases with the Y content, showing a constant behavior in the explored frequency range.

Liu et al. [31] explored the doping effect of  $\text{Sm}^{3+}$  on the magnetic and dielectric properties of Ni-Zn spinels.  $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Sm}_x\text{Fe}_{2-x}\text{O}_4$  samples were synthesized by a conventional two-step solid sintering method. It was found that the substitution by the  $\text{Sm}^{3+}$  cations decreased the dielectric loss in the frequency range of 1–100 MHz, increased in the frequency range of 100–1000 MHz.

## **Conclusion**

Spinel ferrites having significant characteristics, its feasibility makes its more versatile to form number of solid solutions when addition, substitution and incorporation with other metal and rare earth ions. Structural, magnetic, electric, optical and thermal properties of spinel ferrites are strongly dependent on chemical composition and Preparation techniques. Doping of different metal ions shows difference in properties, same as the rare earth ions shows variation and their effect on parent content. Metal ions show the single phase crystal same as rare ions shows Secondary phase in lattice during sintering, after particular temperature the existence of these ions are disappeared. Present work reveals the effect on properties of ferrites materials due to different dopant.

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## Chapter -21

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### Importance of medicinal chemistry in pharmacy

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#### **Abstract:-**

The origins and developments of pharmacy, medicinal chemistry, and drug discovery are interwoven in nature. Medicinal chemistry provides a comprehensive understanding of the chemical basis of drug action equips pharmacy students with the ability to answer rationally the “why” and “how” questions related to drug action and it sets the pharmacist apart as the chemical expert among health care professionals. By imparting an exclusive knowledge base, medicinal chemistry plays a vital role in providing critical thinking and evidence-based problem-solving skills to pharmacy students, enabling them to make optimal patient-specific therapeutic decisions. This review highlights the parallel nature of the history of pharmacy and medicinal chemistry, as well as the key elements of medicinal chemistry and drug discovery that make it an indispensable component of the pharmacy curriculum.

**Keywords:** curriculum, medicinal chemistry, history of pharmacy, drug discovery

#### **Introduction:**

It was not until the mid-19th century that pharmacy emerged as a professional entity in the United States. In 1869, William Proctor Jr defined pharmacy as the “art of preparing and dispensing medicine” which “embodies the knowledge and skill requisite to carry them out to practice.”<sup>1</sup> Thus, preparation and dispensing have been at the heart of pharmacy practice since the beginning of the profession, with sound knowledge of chemical compatibility, solubility, and stability of the drugs deemed essential to effectively accomplish the “preparation” component of the prescription.<sup>1</sup> Over the last 4 decades, the role of a

pharmacist progressively shifted from being a compounder and supplier of pharmaceutical products to a service and information provider, and eventually to a comprehensive patient care provider.<sup>2</sup> In 1990, Hepler and Strand called for a paradigm shift to “pharmaceutical care,” a concept that is defined as “the responsible provision of drug therapy for the purpose of achieving definite outcomes that improve a patient's quality of life.”<sup>2</sup> The Omnibus Budget Reconciliation Act of 1990 (OBRA 1990) increased the clinical responsibility of pharmacists by requiring them to counsel Medicaid patients and participate in prospective and retrospective drug utilization review programs. Responding to this challenge, US colleges and schools of pharmacy moved toward offering the PharmD program.

The advent of this new direction for the pharmacy profession prompted an increase in the clinical coursework in the pharmacy curriculum. Unfortunately, this paradigm shifts also initiated a debate over the relevance of medicinal chemistry, a basic pharmaceutical science, in pharmacy education. The Accreditation Council for Pharmacy Education (ACPE) Standard No. 13 clearly states the need to provide a thorough scientific foundation for the achievement of desired professional outcomes.<sup>3</sup> To achieve this goal, ACPE requires the curriculum to contain biomedical sciences, pharmaceutical sciences, social/behavioural/administrative sciences, and clinical sciences.<sup>3</sup> Additional guidelines specify the medicinal chemistry requirements under the umbrella of pharmaceutical sciences

The Brazilian National Curriculum Guidelines <sup>4</sup> for the pharmacy courses defines the current generalist profile of the pharmacist professional as one who must be capable to “act in all healthcare levels, based on scientific and intellectual rigor”. These guidelines also define the key program contents to pharmacy graduation courses must include the “theoretical and practical knowledge related to research and development, production and quality assurance of pharmaceutical raw materials, ingredients and products”, where medicinal chemistry is inserted. Moreover, among the 31 specific skills and competencies necessary to the formation of the pharmacist, are included “to act in research, development, selection, manipulation, production, storage and quality control of ingredients, natural, synthetic

and recombinant drugs, medicines, cosmetics, sanitizings andcorrelates” and “perform individual and collective pharmaceutical assistance”. Regarding this, medicinal chemistry should contribute not only with drug design and development skills, but also with valuable knowledge of structure-activity relationships (SAR) which gives advantage to perform the practice in pharmacovigilance and pharmaceutical care. Recently, the Brazilian Federal Council of Pharmacy published resolution n° 586,5 which regulates the independent pharmacist’s drug prescription in Brazil, assigning new responsibilities to the pharmacists.

Independent pharmacist’s drug prescription can be defined as the exercise of drug prescribing by a pharmacist autonomously within his/her clinical competence, and this is already implemented in several countries worldwide.<sup>6-9</sup> The presence of medicinal chemistry discipline in pharmacy curriculum should play an important role to the construction of a differential knowledge to a pharmacist from other prescribers regarding pharmacotherapy. In addition, medicinal chemistry concepts must be in mind among the determinants of pharmacotherapy decisions, especially the SAR background of the involved drugs to a high-level practice on clinical pharmacy besides the classroom strategies. Of course, this kind of contribution is not only necessary in Brazil, but in several other countries. Several papers in this Journal have been published dealing with this topic, such as the articles published by Faruk Khan et al.<sup>10</sup> Alsharif et al.<sup>11</sup> and Beleh et al.,<sup>12</sup> emphasizing the importance of this knowledge for pharmacy graduates worldwide. Moreover, this can be noted by the shift in classical textbooks of medicinal chemistry such as those edited by Lemke et al.<sup>2</sup> and Currie and Roche<sup>13</sup> to emphasize the clinical relevance of the discipline and which are adopted by many medicinal chemistry courses around the world

### **Conclusion:**

This article emphasizes the relevance of medicinal chemistry in the pharmacy curriculum, its role in the evolution of pharmacy, and its paradigm shift to pharmaceutical care, as well as its history and intellectual domains. Medicinal chemistry provides a comprehensive



understanding of the underlying principles of drug action and behaviour within the body, which is fundamental to today's pharmaceutical care and patient counselling. Because apprehensions regarding the relevance of medicinal chemistry continue to exist, a change in the approach to how medicinal chemistry content is presented is necessary to better fit this basic science into the pharmacy curriculum under the newly set goals. Some educators are already engaged in this endeavour. Future research/reviews should address the scope of medicinal chemistry in the pharmacy curriculum with appropriate drug class examples. Historically, medicinal chemistry has developed hand-in-hand with the pharmacy profession and has always been at the forefront of drug design and discovery. The components of drug design and discovery contribute to the pharmacy student's foundational knowledge base and will have a tremendous impact in advancing the professional leadership of a pharmacist in the pharmaceutical and health care sectors.

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## Chapter -22

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### Synthesis, Characterization and Antimicrobial Screening of Some New Pyrazolines Bearing Pyrazole Moiety

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#### Abstract

To synthesize pyrazoline derivatives bearing pyrazole moiety, a simple, efficient and green procedure was used. When acrolein, and  $\alpha$ ,  $\beta$ enone reacted with phenylhydrazine provided the simple pyrazoline formation through Cyclization. This method has the advantages of operational simplicity, and high yield of products via a simple experimental and work-up procedure. Further the structures of synthesized product were confirmed on the basis of various spectral data. The compounds were screened for their in vitro antibacterial activity using Gram-positive and Gram-negative bacteria.

**Keywords:** Chalcone, Hydrazine Hydrate, Pyrazoline, Gram-positive and Gram-negative.

#### Introduction

Pyrazoline derivatives refer to a class of organic compounds that contain a pyrazoline ring, which is a five-membered ring consisting of three carbon atoms and two nitrogen atoms.

These derivatives have been of interest in medicinal chemistry and organic synthesis due to their diverse biological activities and potential pharmaceutical applications. Researchers have explored the synthesis of pyrazoline derivatives and studied their pharmacological properties, such as anti-inflammatory<sup>1</sup>, antimicrobial<sup>2-5</sup>, antitumor<sup>6</sup>, antiamoebic<sup>7-8</sup>, antiviral<sup>9</sup>, neuroprotective<sup>10</sup>, antiviral<sup>11</sup>, amine oxidase inhibitory<sup>12</sup> and antidepressant<sup>13</sup> activities.

It's important to note that the biological activities of pyrazoline derivatives can vary significantly based on the specific substituents and structural features. Researchers continue to investigate new methods for synthesizing pyrazoline derivatives and exploring their potential applications in drug discovery and development.

## Experimental

All the chemicals required for this synthesis were purchased from Sigma Aldrich and SD Fine Chemicals. Melting points were recorded in open capillaries and were uncorrected.  $^1\text{H}$  NMR spectra were recorded on Bruker Avance II 400 MHz NMR Spectrophotometer in  $\text{DMSO-d}_6$  and TMS as an internal standard. The infra-red spectra were recorded as potassium bromide disk using FT-IR Spectrophotometer Model RZX (Perkin Elmer). Mass spectra were recorded on Macromass mass spectrophotometer (Waters) by electro-spray method (ES). The purity of the synthesized compounds was checked by TLC silica gel coated plates obtained from Merck as stationary phase and solvent mixture of hexane/ethyl acetate (80:20) as mobile phase.

## General procedure

Compound **1c** Chalcone (0.01mol) was dissolved in 15ml of ethanol. To this reaction mixture, 0.02 mol of hydrazine hydrate was added. Contents were heated under mild reflux for 4 hours and then to the reaction mixture 4-5 drops of glacial acetic acid was added and heating was continued further for 3hours and then cooled to room temperature. Cold water (50ml) was slowly added to the flask and the separated product was filtered, washed with cold water for several times and crystallized from ethanol. The compounds **2(a-g)** were prepared by following the general procedure. Physical data are recorded in **Table 1**. Their structures have been confirmed by IR,  $^1\text{H}$ NMR and Mass spectra.

**IR (2c)** ( $\text{cm}^{-1}$ ):965(C-Cl), 1058(Ar-Br), 1559(C=C), 1598(C=N), 3126(O-H), 3328(N-H).

**$^1\text{H}$  NMR (2c)** ( $\text{DMSO-d}_6$ ) $\delta$ ppm: 3.3241-3.3247(dd, 1H,  $-\text{CH}_a-$ ), 3.5987-3.6024(dd,1H, $-\text{CH}_b-$ ), 3.8380-3.8961(ddd,1H,  $-\text{CH}_c-$ ), 6.8752-

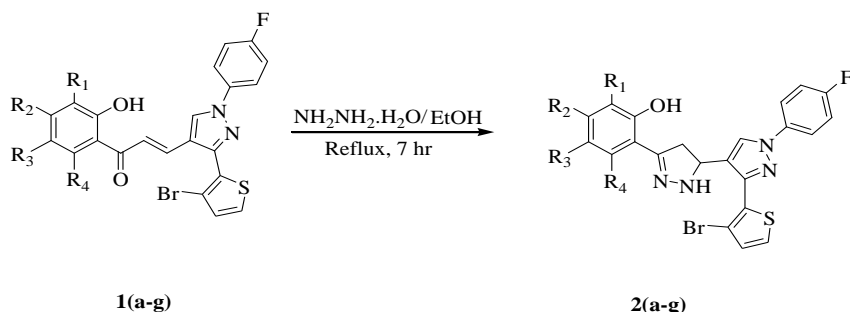
6.9365(d, 1H, -NH-), 7.1864-7.2498(m, 2H, Ar-H), 7.3471-7.3659(m, 2H, Ar-H), 7.4295-7.4305(dd, 2H), 7.8234-7.8269(d, 1H, Ar-H), 7.8687(s, 1H, Ar-H), 7.8901(s, 1H, Ar-H), 8.4251(s, 1H, pyrazole-H), 11.0984 (s, 1H, Ar-OH).

**ES-MS (2c)** (m/z): 518(M+1), 520(M+3).

**IR (2g)** (cm<sup>-1</sup>): 951(C-Cl), 1063(Ar-Br), 1571(C=C), 1591(C=N), 3133(O-H), 3356(N-H).

**<sup>1</sup>H NMR (2g)** (DMSO-d<sub>6</sub>)δ ppm: 3.2689-3.2701(dd, 1H, -CH<sub>a</sub>-), 3.4178-3.4205(dd, 1H, -CH<sub>b</sub>-), 3.7984-3.8002(ddd, 1H, -CH<sub>c</sub>-), 6.8684-6.8691(d, 1H, -NH-), 7.0214-7.1098(m, 2H, Ar-H), 7.2391-7.3871(m, 2H, Ar-H), 7.4354-7.4361(dd, 2H), 7.7954-7.8003(d, 1H, Ar-H), 7.8475(s, 1H, Ar-H), 7.8687(s, 1H, Ar-H), 8.3987(s, 1H, pyrazole-H), 11.1026 (s, 1H, Ar-OH).

**ES-MS (2g)** (m/z): 561(M+1), 563(M+3).



➤ Scheme 1: Synthesis of various 2-(5-(3-(3-bromothiophen-2-yl)-1-(4-fluorophenyl)-1H-pyrazol-4-yl)-4,5-dihydro-1H-pyrazol-3-yl)phenol

**Table 1: Physical data of compounds (2a-g)**

Comp.	R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	M.P. (°C)	Yield (%)
<b>2a</b>	H	H	H	188-190	74
<b>2b</b>	H	H	CH <sub>3</sub>	176-178	79
<b>2c</b>	H	H	Cl	192-194	71
<b>2d</b>	Cl	H	Cl	198-200	83
<b>2e</b>	H	H	F	208-210	80
<b>2f</b>	H	CH <sub>3</sub>	Cl	188-190	69
<b>2g</b>	H	H	Br	174-176	86

## Result and Discussion

The derivatives of Pyrazoline were synthesized successfully in moderate to good yields. The newly synthesized compounds were identified based on melting point range, IR, <sup>1</sup>H NMR and Mass spectral analysis. All the newly synthesized derivatives were screened for antimicrobial activity using the disc diffusion method.

### Antimicrobial activity:

Compounds **2(a-g)** were screened for their in vitro antimicrobial activity against *Pseudomonas aeruginosa* (ATCC 27853), *Escherichia coli* (ATCC 25922), *Staphylococcus aureus* (ATCC 25923) using Gentamycin as a reference standard drug by paper disc diffusion method. Using Nystatin as a standard drug the antifungal activity was evaluated against *Candida sp.* All the tests were evaluated at 100 µg/ml concentration. The culture media was Muller Hinton agar. The zone of inhibition was measured in mm after 24 hours of incubation at 37°C. Microbial data for corresponding compounds is summarized in Table 2.

**Table 2: Antimicrobial Analysis Data**

Sr. No.	Comp.No.	<i>Escherichia coli</i> (ATCC 25922)	<i>Pseudomonas aeruginosa</i> (ATCC 27853)	<i>Staphylococcus aureus</i> (ATCC 25923)	<i>Candida sp.</i>
1	2a	2.3	--	3.9	4
2	2b	--	1.9	--	8.1
3	2c	6	7.8	7.2	--
4	2d	7.5	--	1.8	5.6
5	2e	--	7.4	9.1	--
6	2f	5.3	2.1	--	3.7
7	2g	2.1	--	3.2	9.2
8	Gentamycin	28 mm	23 mm	32 mm	--
9	Nystatin	--	--	--	23 mm

## Conclusion

Newly Seven novel pyrazolines derivatives containing pyrazole have been synthesized successfully and characterized by IR, <sup>1</sup>H NMR and Mass spectral data, Compounds 2a-g are screened for their anti-

inflammatory using the disc diffusion method. From the results obtained, it is concluded that compound 2a-2g shown moderate antimicrobial activity.

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## Chapter -23

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### **An overview of biological active Thiazole containing thiosemicarbazone derivatives**

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#### **Abstract:**

A Sulfur atom at position 1 and a nitrogen atom at position 3, thiazole is one of the most prevalent five-membered heterocyclic compounds. Both naturally occurring and artificially synthesized versions of thiazole show distinct biological behaviors. In the process of finding novel drugs, thiazole ring substituents are changed to produce compounds with strong biological activity. This article discusses thiazole chemistry, thiazole synthesis methods, and their biological functions in order to produce new molecules of thiazole derivatives with more potent biological activity. This article tries to clarify the structural and biological significance of thiazoles in drug discovery and development by examining recent studies on a range of compounds with thiazole rings and their varied biological properties.

**Keywords:** Thiazole, Thiosemicarbazone, Drug Discovery, Anticancer

#### **Introduction:**

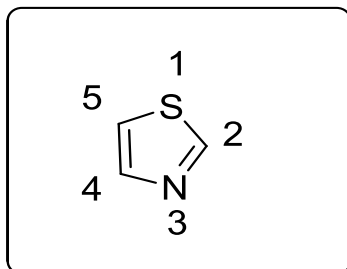
One or more nitrogen-based heterocycles are included in approximately 75% of FDA-approved small-molecule medications. Creating heterocyclic molecules containing nitrogen is crucial to the drug-discovery process. Thiazole, sometimes referred to as 1,3-thiazole, is a member of the azole group of compounds, having atoms of nitrogen and sulfur at positions one and three, respectively. The thiazole nucleus is one of the heterocycles that has been investigated in great detail and is essential to many physiologically active substances. 1,3-Thiazole is a crucial part of several pharmacological formulations. Thiazole-



containing products include anti-neoplastic agents such as tiazofurin and dasatinib, anti-HIV drug ritanavir, antifungal agent ravuconazole, antiparasitic agent nitazoxanide, anti-inflammatory agents meloxicam and fentiazac, antiulcer agent nizatidine, and thiameth.1. concentrating on current research concerning different chemicals with thiazole rings and their derivatives. Thiazole containing compounds have various properties, such as antipsychotic, antibacterial, anticancer, antiallergic, antihypertensive, anti-inflammatory, antimalarial, and antifungal.

### Features of Structure:

Thiazole, also called 1,3-thiazole, is a transparent to pale yellow flammable liquid with the chemical formula  $C_3H_3NS$ . has an odor similar to pyridine. It is a five-membered ring with three members that contain carbon and two vertices that contain sulfur and nitrogen. Below is a list of the thiazole derivative naming conventions.



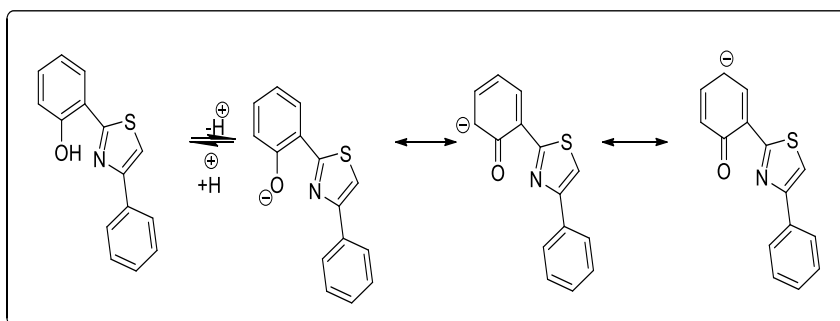
Structure of thiazole

### Thiazole chemistry:

Thiazole creates a stable heterocyclic molecule by utilizing both an electron-donating group (-S-) and an group that accepts electrons (C=N). It is believed that thiazoles, an important class of heterocycles, and its analogues, such as oxazole, exhibit a range of biological features. The thiazole compound and the azole compound isothiazole are isomeric; they both have the same atoms (nitrogen and sulfur) but in opposite positions. Only somewhat soluble in water, thiazole is soluble in ether and alcohol. It is a transparent, pale yellow liquid with a boiling point of 116 to 118°C. Huckel's rule states that a thiazole is a heterocyclic ring with six delocalized electrons from the single pair of electrons in the sulfur atom.

Thiazole derivatives are preferred model compounds for chemistry because of their planar and aromatic structure, which exhibits more electron delocalization than oxazole. Investigation.

The thiazole ring's aromatic behavior was verified by identifying the chemical shift of the protons in  $^1\text{H}$  NMR spectroscopy, which occurred between 7.27 and 8.77 ppm. The thiazole derivatives ring's reactivity was strained as a result of the insertion of different substituents at the C-2, C-4, and C-5 locations; this may call for additional structural thought. For example, when the methyl group (electron donating group) substituent was placed at any location on the thiazole ring, it clearly affected the ring's basicity and nucleophilicity. However, the reductions in basicity and nucleophilicity occur when a strong electron-withdrawing group, such as a nitro group, is added to the molecule.

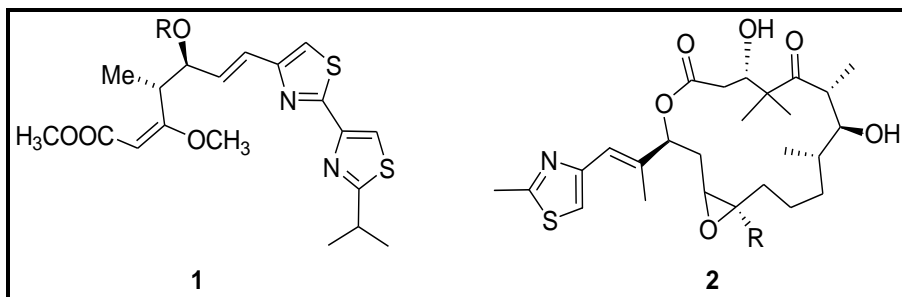


**Figure.1: Resonating Structures of Thiazole**

The diverse building blocks of thiazoles as bioactive chemicals result in a unique molecular structure. Numerous investigations have revealed that the majority of synthetic and natural chemicals with a diverse range of biological features contain the thiazole ring. One such is vitamin B1, sometimes referred to as thiamine, which naturally aids the neurological system by aiding in the manufacture of acetylcholine. Thiazole derivatives also possessed an amphiphilic characteristic since they included both hydrophilic (lipophilic) and hydrophobic (lipophilic) components. Its capacity to readily permeate into the bacterial cell membrane and inhibit activity is enhanced by this characteristic Mixtures

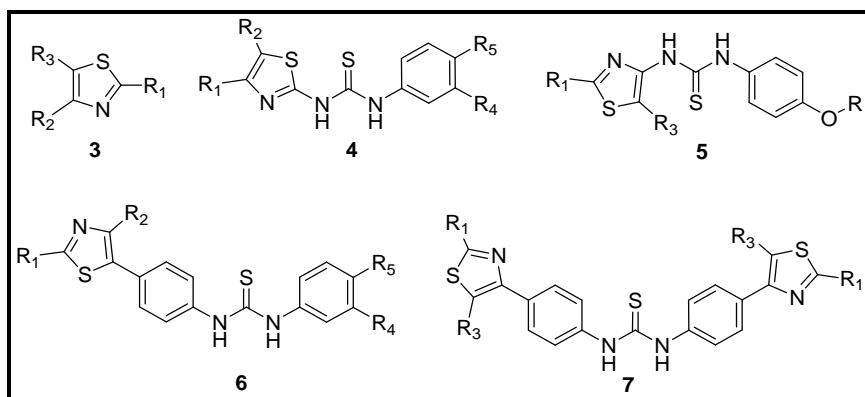
### Biological Activities of Thiazole containing thiosemicarbazone derivatives:

Thioamides are the building blocks in the Hantzsch thiazole<sup>1</sup> and amidines<sup>2</sup> synthesis. The chemistry of thiazole containing heterocyclic compounds continues to draw the attention of synthetic organic chemists due to their varied biological activities,<sup>3</sup> such as antibacterial, antitubercular, anti-cancer, antifungal and anti-inflammatory activities. Sakagami and co-workers reported the isolation of six secondary metabolites, designed cystothiazoles A-F, **1** (Figure 1) as a series of new antibiotics from the myxobacterium culture broth of *Cystobacter fuscus*.<sup>4</sup> These bis-thiazoles has demonstrated potent antifungal activity against the phytopathogenic fungus *Phytopathora capsici* and has shown activity against a broad range of additional fungi with no effect on bacterial growth. Nicolaou K. C. and co-workers<sup>5</sup> have reported chemical biology of epothilones<sup>6</sup> **2** (Figure 1), thiazole containing an antitumor agent. Thiazole nucleus is also important in organic synthesis as it acts as a formyl group equivalent.<sup>7</sup>



**Figure.1 Antibiotic Compound**

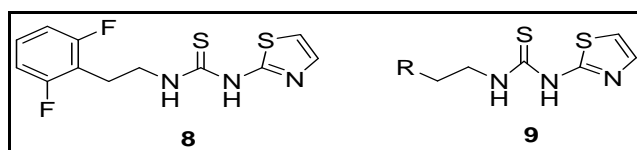
A wide range of substituted thiazole derivatives,<sup>8-12</sup> can be obtained in the reactions of thioamides derived from aliphatic, aromatic, and heterocyclic acids with appropriately functionalized carbonyl compounds. Kulkarni K. S. and co-workers<sup>13</sup> have synthesized substituted thiazolyl thiocarbanilides (**3-7**), (Figure 2) and reported for antitubercular activity.



**Figure.2 S.thiazolyl thiocarbanilides**

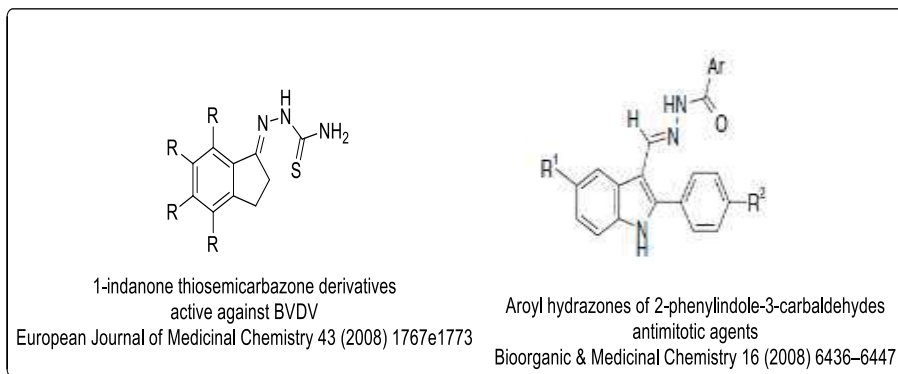
2 - substituted - anilino / phenyl / benzyl - 5 - substituted - 4 - phenylamide - (3 - (2 - chlorophenyl) - 5 - methylisoxazolyl) thiazoles and reported them as potential antitubercular and antimicrobial agents<sup>14</sup>. Ohkubo M. and co-workers<sup>15</sup> have reported synthesis and anti-anoxic activity of ethyl 4-(3/4-nitrophenyl)-2-phenyl-5-thiazolecarboxylate derivatives.

Bell, F.W. and co-workers<sup>16</sup> have reported synthesis and basic structure activity relationship studies of phenethylthiazolethiourea compounds **8**. (**Figure 3**) The synthesized compounds were screened for HIV-1 reverse transcriptase inhibitors activity.



### Thiosemicarbazone

Biological properties of thiosemicarbazone derivatives have been studied since 1946<sup>17</sup>, when their activity against *Mycobacterium tuberculosis* was reported. Since then, this and other biological properties of thiosemicarbazone derivatives such as antibacterial<sup>18</sup>, antitumoral<sup>19</sup>, antiprotozoal<sup>20</sup> and cytotoxic activity<sup>21</sup> have been described. In 1950, Hamre et al.<sup>22</sup> found that thiosemicarbazone derivatives from several benzaldehydes were active against neurovaccinial infection in mice when given orally.



### Conclusion:

Numerous pharmacological effects of thiazole compounds have been demonstrated, including antidiabetic, anticonvulsant, antioxidant, antifungal, antimalarial, and antitumoral properties. Generally, Thiazole containing thiosemicarbazone derivatives are known to possess intriguing biological properties like antibacterial and anticancer properties. It has been observed that changes to the thiazole moiety shown beneficial biological functions.

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*RECENT ADVANCES IN SCIENCE & TECHNOLOGY*

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## Chapter -24

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### Aquatic Weeds control from fishery pond

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#### **Abstract:-**

The unwanted plants that grow in the wetland are termed as aquatic weeds. They generally grow in the fields and compete with crops for water, soil nutrients, light and space, and crop yields are reduced. They also harbour insect pests and microorganisms. Weeds belong to the class Angiospermae (flowering plants) which have two subclasses: Monocotyledoneae (monocots) and Dicotyledoneae (dicots). On the basis of the habitats, they are divided into terrestrial and aquatic categories, and on the basis of the duration of life, they are divided into annuals, biennials and perennials. Water hyacinth is highly invasive aquatic vegetation that poses a serious threat to the environment, society, and economy due to its rapid growth, ecological resilience, and global distribution. Aquatic plants have adapted to living in or on aquatic environments and constitute a problem in culture fisheries. Aquatic weeds can be controlled by several methods like biological, chemical and physical. Each method has its benefits and drawbacks. There are several popular control mechanisms for preventing the spread or eradication of aquatic weeds. Physical methods are suitable only for small scale infestation but when applied in large water bodies become ineffective due to high cost and regrowth. Chemical control has been practiced against aquatic weeds since long time in India but it is not prevalent. This comprehensive Chapter provides valuable insights into an integrated approach for successful Indian major carp aquaculture, promoting sustainable practices for improved productivity and long-term viability of aquatic life.

**Keywords:** - Aquatic weeds control, Biological control, Chemical control

**Introduction: -**

Ponds are ideal habitats for aquatic plants, and some will always be present. Plants are a necessary component of pond ecosystems because they perform valuable functions. Photosynthesizing plants produce oxygen that is needed to sustain fish life. Also, plants assimilate ammonia that is excreted by fish thereby helping to prevent accumulation of potentially toxic concentrations of ammonia. Nevertheless, plants can cause problems in ponds and control measures often must be used to eliminate or reduce their abundance.[01]

**Types of Aquatic Plants:-**

**Higher aquatic plants:-**

**Submersed plants: -**

Submersed plants spend their entire lifetime beneath the surface of the water, although the flower may extend above the surface. Usually the plants are rooted in the mud, but masses of plants may tear loose and float free in the water. These plants are objectionable because they interfere with fishing and fish harvest. The most common submersed higher aquatic plants in ponds are *Najas guadalupensis*, *Potamogeton pectinatus*, *Ceratophyllum demersum*

**Emergent plants:-**

Emergent aquatic plants are rooted in the bottom mud and grow above the water. Many can also grow under strictly terrestrial conditions. The plants are rigid and not dependent on the water for support. Emergent plants usually infest only the pond margins and other shallow areas resulting from inadequate pond construction, water-shortage conditions, or excessive bank erosion. If stands of emergent plants become too dense or widespread, they may interfere with fishing, seining or feeding of fish. They can also create a habitat that harbors snakes. Fast-growing emergent plants such as smartweed (*Polygonum pennsylvanicum*) inhabit shallow areas quickly and can often outpace the rising water level when ponds fill slowly (e.g. during dry seasons or if only a slow flowing well is available to fill the pond). The most common emergent weeds in ponds are: *Polygonum* spp, *Typha* spp., *Salix* spp.



**Floating plants:-**

This category includes free-floating plants such as duckweeds and water meal , and floating-leaf plants, such as water lilies. Many floating plants are present only when pond waters are relatively stagnant and sheltered from winds. Small recreational ponds often have problems with duckweed (*Lemna* spp.) and watermeal (*Wolffia* spp.). In aquaculture ponds, on the other hand, periodic draining and refilling, and frequent fish harvest activity make conditions unfavorable for these plants; these larger ponds are often unsheltered from the wind, and duckweeds are continually washed ashore where they dry up and die. Aquatic plants play an important role in the functioning of aquatic ecosystems [02] However, introduced invasive alien aquatic plants (IAAPs) may threaten ecosystems due to their excessive growth and have both ecological and economic impacts[03]

**Biological control of weeds using pathogens: -**

Weeds can be controlled by pathogens like fungi, bacteria, viruses and virus like agents. Among the classes of plant pathogens, fungi have been used to a larger extent than bacteria, virus or nematode pathogens. In some cases, it has been possible to isolate, culture, formulate and disseminate fungal propagules as mycoherbicides. [04].

**Chemical control of weed:**

Chemical herbicides enable control of aquatic weeds quickly and efficiently, albeit temporarily. Nonetheless, chemical control is the predominant and dependable means of aquatic weed management. The present generation of aquatic herbicides is generally safe when used according to the labelled directions. Misuse of herbicides, deliberate or due to a lack of understanding of proper use, as well as worker protection, are frequent concerns. Misuse can damage the surrounding habitats. Even proper use of herbicides can cause nutrients to be released from decaying vegetation into the water and trigger temporary algal bloom, depress oxygen level, and cause fish kill, especially during hot months. The amount and persistence of chemical residues in treated waters and the increase in the amounts of organic matter that sediments are two other problems. Many herbicides and algaecides require either waiting periods of several hours or days before the water can be used; more stringent restrictions may apply if

the water is used for drinking, irrigation, recreation, or fishing. This will inevitably disrupt the use of the treated water. Presently, chemical pesticides are facing unprecedented scrutiny and restrictions due to regulations such as the Food Quality Protection Act and the Clean Water Act in the USA. In many countries, herbicide use in multi-use waters is banned or severely restricted. Presently, a critical issue facing submerged aquatic weed control is the extremely limited choice of herbicides. Coupled with this, the prospects of resistance buildup to a widely used herbicide, fluridone [5] raises a new level for concern. Hitherto, herbicide resistance has not been a problem in aquatic weed control. Aquatic plant treatments can be complex. Professional aquatic herbicide applicators can be employed to treat pond weed problems. Commercial applicators may be useful in selecting the right chemical, calculating the dosage rate, and safely applying the herbicide. Herbicide treatment should not be considered as a total cure for a pond weed problem. Rather, a combination of methods should be used and supplemented with chemical control agents.[6]

**Conclusion: -**

The development of new techniques for aquatic weed control has been glacial. The reasons for this are principally because of the relatively small “market” in aquatic weed control, and the social unacceptability of many of the available methods. Despite these problems, new techniques in herbicide delivery have been developed, and are now widely used, often with superior results. Prevention is the best way to reduce aquatic plant problems. It is cheaper and easier to prevent weed growth than to control weeds in your pond. Cost of weed control should, therefore, be a major consideration in selecting control methods, especially when monetary resources are limited.

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## Chapter -25

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### **An efficient and green approach for the Synthesis of 2-mercaptobenzimidazole and its derivatives**

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#### **Abstract**

2-mercaptobenzimidazole derivatives were synthesized by reaction of o-phenylenediamines with N-aminorhodanine. This reaction represent a new synthesis of 2-mercaptobenzazole. The structure of the obtained products was established by spectroscopic data.

**Keywords:** 2-mercaptobenzimidazole, o-phenylenediamines, N-aminorhodanine.

#### **Introduction**

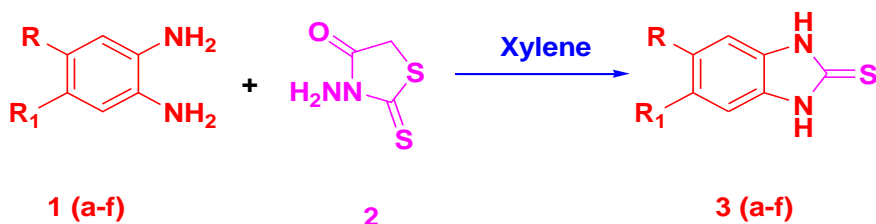
Organic compounds are carbon based compounds. Organic compounds are subdivided into aliphatic, aromatic and heterocyclic compounds. These may be natural or prepared in laboratory. An organic heterocyclic is one which possesses at least one atom other than carbon such as nitrogen, sulfur and oxygen. Such atoms are said to be the heteroatoms. They play a key role in medicines, biochemistry, agriculture and industries Organic heterocycles are classified as 3, 4, 5, 6, and 7 member rings and fused rings.<sup>1,2</sup> A crystalline base C<sub>7</sub>H<sub>6</sub>N<sub>2</sub> that is composed of benzene ring fused with an imidazole ring and is structurally similar to purine ; also: any of various derivatives (such as thiabendazole omeprazole, or benomyl) of this base typically possessing therapeutic properties including broad-spectrum of biological activities such as anthelmintic, fungicidal, or antimicrobial action. Literature survey reveals that when one heterocyclic compound is coupled with another, a new compound results and its biological activity is enhanced. Therefore heterocyclic compounds have got much

attention in the modern era.<sup>3</sup> Benzimidazole, which is a heterocyclic aromatic organic compound, is a privileged structure in medicinal chemistry.<sup>4</sup> It contains a phenyl ring fused to imidazole ring. It was synthesized by Hoebrecker in 1872 who obtained 2,5(or 2,6)-dimethylbenzimidazole by the reduction of 2-nitro-4methylacetanilide.<sup>5</sup> This nucleus plays a role in synthesis of different bioactive derivatives which are used as medicinal compounds. These compounds act as anti-ulcer, anti-viral, anti-tumor, anti-microbial anti-hypertensive and anti-oxidants etc.<sup>6</sup>

Thiols are very useful building blocks for the synthesis of various organosulfur compounds: they have several applications in organic synthesis, in bioorganic, medicinal and heterocyclic chemistry.<sup>7</sup> Also, in addition to that, thiols can act as safety-catch linker in peptide chemistry.<sup>8</sup> Moreover, thiols have been employed as sulfur-based ligands in transition metal complexes.<sup>9</sup> In this respect, a number of synthetic methods for the preparation of thiol derivatives by the reaction of o-phenylenediamine with carbon disulphide,<sup>10</sup> thiourea,<sup>11</sup> O-isopropylxanthic acid potassium salt,<sup>12</sup> 5-phenyl-1,3,4-oxadiazole-2(3H) thione derivatives and N-phenylisothiocyanate,<sup>13</sup> thiocyanic acid ammoniac salt.<sup>14</sup> In continuation of our research concerning benzimidazoles.<sup>15</sup> The condensation of o-phenylenediamine with N-aminorhodanine was carried out.

### Experimental procedure for the synthesis of mercaptobenzimidazole

O-phenylenediamines (0.065 mol) was heated with N-aminorhodanine (0.065 mol) in xylene (50 ml) for 5 hours. The obtained residue was filtered and was crystallized from aqueous alcohol (charcoal). The obtained solid was recrystallized in ethanol.



**Scheme 1:** The synthesis of 2-mercaptobenzimidazole derivatives.

## Results and discussion

In this work, we report a novel method for synthesis of 2-mercaptobenzimidazole derivatives. 2-Mercaptobenzimidazoles are interesting starting compounds because of their chemical reactivity and biological activities. A mixture of o-phenylenediamines 1(a-g) and N-aminorhodanine in xylene was heated during 8 hours. (Scheme 1). The structure of the products 3(a-g) has been determined by NMR and mass data. <sup>1</sup>HNMR spectra showed the presence of the signal of NH and the signal of the C=S was observed in <sup>13</sup>CNMR spectra which confirms the structure of the products 3(a-g). The key step is at the intermediate A, in which the cyclization goes to the C=S group not to C=O group in order to lead to the benzotriazepine product. In the reaction, the only 2-mercaptobenzimidazole product was obtained.

**Table 3. Preparation of 2-mercaptobenzimidazole and its derivatives.**

Entry	Substituted o-phenylenediamines (1a-f)		Product (3a-f)	Yield (%)
	R	R <sub>1</sub>		
1	H	H	2-mercaptobenzimidazole (3a)	90
2	NO <sub>2</sub>	H	2-mercapto-5-nitrobenzimidazole (3b)	92
3	CH <sub>3</sub>	H	2-mercapto-5-methylbenzimidazole (3c)	90
4	Cl	H	5-chloro-2-mercaptobenzimidazole (3d)	96
5	Cl	Cl	5,6-dichloro-2-mercaptobenzimidazole (3e)	95
6	CH <sub>3</sub>	CH <sub>3</sub>	5,6-dimethyl-2-mercaptobenzimidazole (3f)	91

### Spectral Data:

2-mercaptobenzimidazole 3a Yield = 87 %; mp>250°C. <sup>1</sup>HNMR (DMSO-d<sub>6</sub>): 7.10 (m, 2Har); 7.27 (m, 2Har); 12.42 (s,NH). <sup>13</sup>CNMR (DMSO-d<sub>6</sub>): 119.43 (CH); 126.36 (CH); 138.82 (C); 167.12 (C=S). HRMS, m/z: 150(M), calcd for C<sub>7</sub>H<sub>6</sub>N<sub>2</sub>S: 150.02517, found: 150.0251.

## Conclusion

In this work, we developed a novel and improved method for 2-mercaptobenzimidazole derivatives by the condensation of o-phenylenediamines with N-aminorhodanine. The current research will open a new era in the chemistry of 2-mercaptobenzimidazole. The work may be utilized in future for deriving synthetic analogues of 2-mercaptobenzimidazole.

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## Chapter -26

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### ***Ixora arborea* (L): Green, efficient and cost effective alternative for sulfur in the preparation of incense stick**

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#### **Abstract:**

*Ixora arborea* (L) commonly known as torch plant. It is rich in volatile oil so it burns in wet condition. In this paper, *Ixora arborea* leaves powder has taken as alternative oxidizer instead of potassium nitrate, sodium nitrate or sulfur/sulfur compounds. All these are inorganic compound containing Nitrogen and sulfur. When it burns it emits several air polluting gases such as CO, NO, NO<sub>2</sub>, SO<sub>2</sub> gases. These gases are very harmful to respiratory track. It produces many of respiratory diseases. To avoid respiratory complications we have replaced nitrogen and sulfur based inorganic compounds by natural and green source *Ixora arborea*.

**Keywords:** *Ixora arborea*, Oxidizer, nitrogen and sulfur based compounds, respiratory complications

#### **Introduction**

Generally in the form of sticks, coils, and cones, incense is used across the world. Amongst the globe, India has a rich tradition of using incense in many social and religious occasions since time immemorial. Irrespective of cultural beliefs, incense is used in Taoist/Buddhist, Hindu, and Shinto temples and Christian churches [1]. In every Indian household, agarbattis, Dhup are an essential part of prayer. Without agarbattis, Dhup the puja (ritual) is incomplete. Although it is available in every home, there are no known health benefits of incense sticks, apart from filling the atmosphere with scented aromas. However, what



you are unaware of the fact is that this everyday ritual can cause more harm than good to your health.

According to a recent study, incense sticks, dhup, coils, come with a health threat [2]. The results proved that burning agarbattis inside the house generate air pollutants, namely carbon monoxide, Sulfur dioxide, nitric oxide, etc. [3] It causes indoor air pollution that may lead to inflammation of the lung cells, putting you at an increased risk of respiratory complications, increases your risk of COPD and asthma, skin allergies, triggers neurological symptoms, heightens risk of respiratory cancer, increases the toxic load on the body, etc. Following table gives the details about health impact on incense smoke constituents.

**Table 1: Health impact of incense smoke constituents**

<b>Gas evolved</b>	<b>Source/features</b>	<b>Impact on human health</b>	<b>Reference</b>
Carbon monoxide (CO)	Incomplete combustion of hydrocarbons, wood, incense, cigarette, and fossil fuels	Reduces oxygen-transport ability of blood by forming carboxyhemoglobin. Even low-level inhalation causes dizziness, headaches, weakness, and nausea. High concentrations can cause severe illness.	[4]
Sulphur dioxide (SO <sub>2</sub> ) and nitrogen dioxide (NO <sub>2</sub> )	May form complex compounds with other pollutants	Reduced work capacity, elevated cardiovascular complications, pulmonary impairment, respiratory illness, lung irritation, and perturbation in self-defense	[5]
Volatile organic compounds (VOCs)	Chemicals with low boiling points, evaporate easily at room temperature, and include benzene, xylene, toluene, and isoprene	Ophthalmic inflammation, nose and throat irritation, nausea, vomiting, headaches, asthma exacerbation, dizziness. Chronic exposure leads to cancer, liver damage, and central nervous system damage	[5,6]

A variety of materials have been used in making incense. Historically there has been a preference for using locally available ingredients. Along with this, different types of combustible bases (like  $\text{NaNO}_3$ ,  $\text{KNO}_3$ , Sulfur etc.) are used for direct and continuous burning of the sticks. Oxidizer on burning evolves gases like  $\text{SO}_2$ ,  $\text{NO}$ ,  $\text{NO}_2$ , etc. So we have strong need to replace such kind of hazardous oxidizer by green, efficient and cost effective material.



**Fig. 1: a) *Ixora arborea***

**b) *Eucalyptus microtheca***

Considering aforementioned discussion, and importance of *Ixora arborea* plant leaves, in present work, we describe a complete replacement of inorganic carcinogenic air pollution causing oxidizer by green, efficient, renewable, cost effective and plant based material for manufacturing of incense sticks, dhoops, coils, etc.

### **Materials and Methods:**

The plant materials collected from different resources were well recognized and authenticated by taxonomist. Double distilled water was taken for preparation of incense sticks and dhoops. All the chemicals and essential oils were of A grade.

### **Experimental: I**

*Ixora arborea* is also called ‘Torch Plant’ was collected from Giroli ghat (Kolhapur, India). Taxonomic identification was made from, department of botany, Y. C. Warana Mahavidyalaya, Warananagar. Collected material was put for shed drying.

Dried powder of *Ixora arborea* (70g) as oxidizer, wood powder (500g) as base and *Eucalyptus microtheca* dried leaves powder (60g) for essence and anti mosquito agent taken into pot. It was well mixed and wet by water. Wet pulp then rolled on the bamboo sticks and kept for drying.

### **Result and Discussion:**

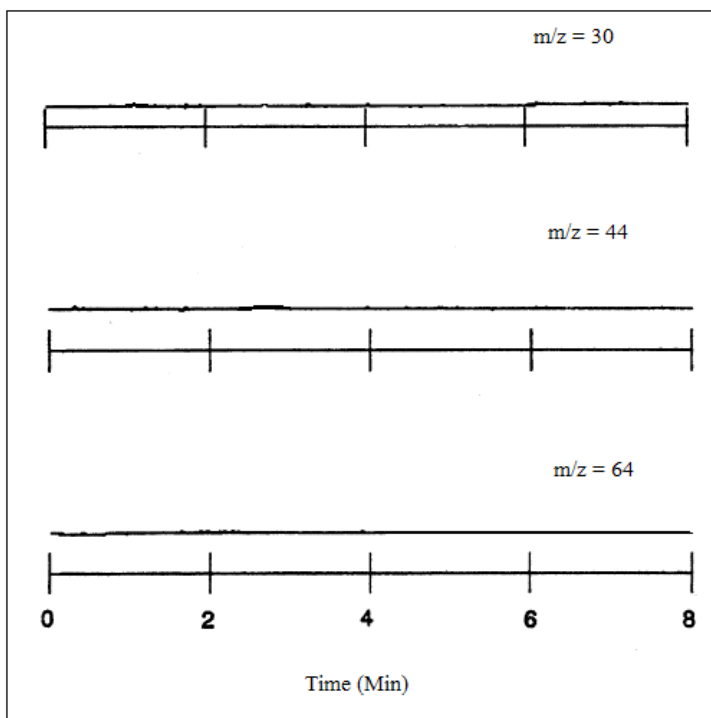
*Eucalyptus microtheca* is well mosquito repellent with good essence. Prepared incense sticks and dhups burnt complete with evolving little smoke. Primary qualitative analysis of smoke was taken in laboratory by taking samples.

### **Tests for NO, NO<sub>2</sub> and SO<sub>2</sub> gases:**

**Test for NO:** Saliva strip was taken for testing NO gas. There was no any color change in strip when it is in contact with the gaseous sample. It is confirmed that, there is absence of NO gas in collected gaseous sample.

1. **Test for NO<sub>2</sub>:** Collected smoke sample is purged with freshly prepared FeSO<sub>4</sub> solution. In this mixture then concentrated H<sub>2</sub>SO<sub>4</sub> is added along the sides of the test tube. A brown ring was not formed at the junction of the two liquids. It confirmed that there is absence of NO<sub>2</sub> in the smoke sample.
2. **Test for SO<sub>2</sub>:** Colorless gas with a pungent smell, like burning sulphur which turns acidified potassium dichromate solution green. While testing smoke collected from incense stick and dhoop, there were no pungent smell and acidified of potassium iodide solution containing starch not turned to blue. This confirmed that, there is absence of SO<sub>2</sub> gas in smoke sample analyzed.

It was also confirmed by analyzing gaseous sample in gas chromatography. From the following gas chromatogram, it was confirmed that, there were no any specific peak observed. Hence it clear that, there is absence of NO, NO<sub>2</sub> and SO<sub>2</sub> in the gas sample collected by burning incense stick.



**Fig. 2: SIM chromatograms of ca. 1 mg/m<sup>3</sup> of NO ( $m/z= 30$ ), N<sub>2</sub>O ( $m/z= 44$ ), and SO<sub>2</sub> ( $m/z= 64$ ) in nitrogen by direct chromatography. Chromatograms were run isothermally at 30°C.**

### Conclusion:

We have successfully replaced hazardous oxidizer by natural source *Ixora arborea* (Torch Plant). Also, prepared product is best mosquito repellent with good essence and burnt very smoothly without evolving carcinogenic gases. The results summarized here demonstrate that GC-MS offers a viable alternative technique to those presently used to determine various nitrogen and sulfur oxides. GC-MS offers the possibility of determining individual species, often at sensitivities not available with other techniques. GC-MS also offers the possibility of time-resolved analysis not available with techniques such as ion chromatography. While cost and size may prevent replacing many analyzers presently used with this technique, GC-MS offers a new alternative where the characteristics mentioned above are important.

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## **Chapter -27**

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### **Status of initiative to improve water resource management**

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#### **Introduction**

Water scarcity affects more than 40% of the global population. The global population is growing fast, and estimates show that with current practices, the world will face a 40% shortfall between forecast demand and available supply of water by 2030. Climate change will worsen the situation by altering hydrological cycles, making water more unpredictable and increasing the frequency and intensity of floods and droughts. The roughly 1 billion people living in monsoonal basins and the 500 million people living in deltas are especially vulnerable. Flood damages are estimated around \$120 billion per year (only from property damage), and droughts pose, among others, constraints to the rural poor, highly dependent on rainfall variability for subsistence. The fragmentation of this resource also constrains water security. There are 276 trans boundary basins, shared by 148 countries, which account for 60% of the global freshwater flow. Six hundred sixty three million people in the world do not have access to clean water, and over 2.7 billion people face water shortages for at least one month out of the year. Water usage has increased at twice the rate as that of the world's population growth. The agriculture industry is attributed to 70 percent of the world's water withdrawal. Women around the world spend a total of 200 million hours a day traveling to collect water, taking time away from their economic activities and families. Fifty percent of those facing water scarcity live in India and China, and 500 million people live in regions where the water supply cannot keep up with the rate of water consumption.

Though there is sufficient water on the planet to meet the needs of the world population, water distribution, usage, climate and

pollution limit the amount of consumable water available from region to region, causing water stress or scarcity. To strengthen water security against this backdrop of increasing demand, water scarcity, growing uncertainty, greater extremes, and fragmentation challenges, people will need to invest in institutional strengthening, information management, and (natural and man-made) infrastructure development.

### **How India is addressing its water needs?**

The country has 18 percent of the world's population, but only 4 percent of its water resources, making it among the most water-stressed in the world. A large number of Indians face high to extreme water stress, according to a recent report by the government's policy think tank, the NITI Aayog (2018). India's dependence on an increasingly erratic monsoon for its water requirements increases this challenge. Climate change is likely to exacerbate this pressure on water resources, even as the frequency and intensity of floods and droughts in the country increases. Groundwater is one of the most important sources for irrigation as well as for rural and urban domestic water supply. However, overexploitation of this valuable resource has led to its depletion. The World Bank is helping the government's national groundwater program, the Atal Bhujal Yojana, to help improve groundwater management. Implemented in 9000 gram panchayats across seven Indian states, this is the world's largest community-led groundwater management program. Since groundwater conservation lies in the hands of hundreds of millions of individuals and communities, the program is helping villagers understand their water availability and usage patterns so they can budget their water use accordingly.

The "Paani Bachao, Paisa Kamao" (Save Water, Earn Money) scheme in Punjab incentivizes farmers to reduce groundwater usage. Around 300 enrolled farmers were given cash incentives to save electricity used for irrigation, resulting in water savings of between 6 and 25 percent without any adverse effect on the yield. Villages in the mountain state of Uttarakhand, suffered from a lack of water supply as the steep Himalayan terrain made it difficult to build and maintain the required infrastructure. For many villagers, particularly women,

obtaining fresh water for domestic use meant traveling distances of over 1.6 kilometers. Between 2006-15 the World Bank-financed Uttarakhand Rural Water Supply and Sanitation Project helped over 1.57 million people in the state by improving sustainable rural water supply and sanitation services across underserved areas. The project focussed on building infrastructure and institutional capacity, including that of the village communities, that would be resilient from natural disasters in the mountain state that often experiences flash-floods, earthquakes and landslides.

The southern state of Kerala receives one of the highest levels of rainfall in the country, however, its undulating terrain drains most of the rainwater into the sea. Rapid growth of built-up areas across the state has led to depleting water sources. Since the early 2000s, the World Bank has been supporting the state government in ensuring that rural families receive a dependable supply of piped water in their homes, at a price that even low-income households can afford. *Jalanidhi I* (2000-2008) and *Jalanidhi II* (2012-2017) have helped bring water into village homes by putting local communities in charge of managing their own water supply schemes for the first time in their lives.

Continuous piped water supply has been a pipe dream for fast-urbanizing Indian cities. Most urban households receive water for a couple of hours a day at most, and often only on a few days a week. This particularly affects the poor, women and children, who spend time and money securing water for their daily needs. The southern state of Karnataka has now proved that 24/7 water supply is indeed possible, affordable, and sustainable in urban areas. The World Bank-supported Karnataka Water Supply Improvement Project helped pilot this approach in the three-water stressed cities of Hubballi-Dharwad, Belagavi and Kalaburgi; a follow-on project, the Karnataka Urban Water Supply Modernization Project, is now scaling up to cover the entire population of the three cities.

In 2019 the southern metropolis of Chennai reeled under a severe water crisis and water was brought in by train from some 200 kilometers away to save the city. Today, Chennai has become the first Indian city to recycle its wastewater at scale to meet the non-drinking water needs of its industries. One completed, two Tertiary Treatment



Reverse Osmosis (TRO) plants will be able to recycle about 20 percent of Chennai's sewage, enabling the city to reduce its consumption of fresh water.

### **Managing India's most iconic river**

The Ganga is India's most important and iconic river and is worshipped by millions as a living goddess. However, the Ganga today is facing formidable pressures of rapid urbanization along its banks as over 100 towns and cities pour their domestic sewage into the river. The World Bank has been supporting the Government of India's efforts to rejuvenate the Ganga River since 2011. Two World Bank projects, worth \$1 billion, are helping set up the institutions needed to manage the river and build the infrastructure to keep it clean. Sewage from cities is the biggest source of organic pollution in the Ganga. By building and maintaining sewage treatment plants and a network of drains, sewage water from houses in several of these cities now gets treated before reaching the river.

### **Tracking floods and droughts**

India is prone to droughts as well as floods even as climate change is increasing unpredictability in weather patterns and leading to more extreme weather events. Reservoirs can help mitigate these extreme events by storing water and releasing it when needed. However, reservoir operators often do not have the technological tools to help them take crucial decisions that can avoid floods.

### **SOME GOVERNMENT INITIATIVES**

1. Water Deficit Banda Shows the Way Through KuanTalab Jiao Abhiyan
2. MazhaPolima (Participatory Well Recharge Programme), Thrissur District, Kerala
3. Practice Manual for Use of Technical Textiles in Water Resources Works
4. Saguna Rice Technique - SRT
5. Best Practices of Ground Water Harvesting in Different Parts of India (Central Ground Water Board Initiatives)

6. Best Practices of Ground Water Harvesting in Different Parts of India (Government Research Institutions Initiatives)
7. Best Practices of Ground Water Harvesting in Different Parts of India (State Departments & Local Bodies Initiatives)
8. Best Practices of Ground Water Harvesting in Different Parts of India (State Governments Initiatives)
9. Rajasthan makes rapid strides in Water Conservation
10. Pani Panchayats for Water Management
11. Tackling drought through farm ponds
12. Groundwater levels rise in Chittor, locals and farmers benefit
13. Two blocks of Odisha get water round-the-year, people rejoice
14. MeraPani Meri Virasat scheme for crop diversification in Haryana
15. A Reverberating Revival Story of Kosi River
16. Rejuvenation of Traditional Water Bodies Makes Puducherry District Water-Rich
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## Chapter -28

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### Billionth of Meter Size Particle

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#### **Timeline for billionth of meter size particle -**

2000 years ago Sulphide Nano particles are used by Greeks and Roman to dye hairs. 1000 years ago gold Nano particles of different sizes used to provide different colors in stained glass windows. This means the Nano particles are used in ancient period but the peoples were unknown about their shape size and the proper applications. Twentieth century the American physicist and Nobel Prize laureate Richard Feynman introduce the concept of nanotechnology in 1959. During the annual meeting of the American Physical Society, Feynman presented a lecture entitled "There's Plenty of Room at the Bottom" at the California Institute of Technology (Caltech).



1974 Taniguchi first time uses the term Nanotechnology. In 1981 IBM develop scanning tunnelling microscope. 1985 "Bucky ball" scientist at Rice University and university of Sussex discover C<sub>60</sub>. 1986 "Engines of creations" published first book on Nanotechnology by K. Eric Drexler. Atomic force microscope invented by Binnig Quate and grebe. 1989 IBM logo made with individual atoms. 1991 carbon Nano tubes discovered by S. Lijima. 1999 "Nanomediclne" first Nano medicine book by Freitas.

**Introduction: -**

Nano – Greek word means –“Dwarf”

1 nm = one billion<sup>th</sup> ( $10^{-9}$ ) of meter.

Size range between 0.1 & 100 nm.

Nanotechnology is also known as molecular manufacturing. According to the dictionary, the definition of Nano is small or minute. So the definition of nanotechnology is the study, design, and application (or manufacturing) of microscopically small things. Nanotechnology is the development and use of techniques to study physical phenomena and develop new devices and material structures to the Nano size. Nanotechnology impacts all areas of our lives. Nano science and nanotechnology are the study and application of extremely small things and can be used across all the other science fields, such as chemistry, biology, physics, materials science, and engineering. Prof. C.N.R. Rao is considered as the "Father of Indian Nanotechnology". The behaviour of the elements at their normal atomic size is normally known to the scientist but below about 100 nm the rules that governs the behaviour of the elements of our known world starts to give way to the rules of quantum mechanics and everything changes. (Physical, mechanical, electrical, medicinal etc.). E.g. carbon in the form of graphite (i.e. lead pencil) is soft with the help of which we can write / draw easily but when the same graphite is dropdown to its Nano size particles its characters changes. It becomes stronger than steel and is six time lighter than it's original.

Nano particles of copper is highly elastic metal at room temperature stretching to 50 times its original length without breaking. Means it become more elastic. We observed the gold which shiny but when we dropdown the gold elements to its Nano size at that time gold changes its colour to brownish black and as the size changes its colours changes depending upon the size of the particle. The changing characteristics of the elements at Nano scale are useful for the living things if we apply these particles in proper manner. So it is important to study billionth of metre scale particle. But these particles are not user friendly. Their size is very small it may badly affect the human body. Elements of the different materials can be converted into Nano size basically by two methods

**1. Top down Approach: -**

Top-down approach involves the breaking down of the bulk material into Nano sized structures or particles. Top-down synthesis techniques are extension of those that have been used for producing micron sized particles. Top-down approaches are inherently simpler and depend either on removal or division of bulk material or on miniaturization of bulk fabrication processes to produce the desired structure with appropriate properties. The biggest problem with the top-down approach is the imperfection of surface structure. For example, nanowires made by lithography are not smooth and may contain a lot of impurities and structural defects on its surface. Examples of such techniques are high-energy wet ball milling, electron beam lithography, atomic force manipulation, gas-phase condensation, aerosol spray, etc.

**2. Bottom Up Approach: -**

The alternative approach, which has the potential of creating less waste and hence the more economical, is the 'bottom-up'. Bottom-up approach refers to the build-up of a material from the bottom: atom-by-atom, molecule-by-molecule, or cluster-by cluster. Many of these techniques are still under development or are just beginning to be used for commercial production of Nano powders. Organometallic chemical route, reverse-micelle route, sol-gel synthesis, colloidal precipitation, hydrothermal synthesis, template assisted sol-gel, electrode position etc., are some of the well-known bottom-up techniques reported for the preparation of luminescent nanoparticles. Using nanotechnology, materials can effectively be made stronger, lighter, more durable, more reactive, more sieve-like, or better electrical conductors, among many other traits. Nanotechnology has already been embraced by industrial sectors, such as the information and communications sectors, but is also used in food technology, energy technology, as well as in some medical products and medicines. nanomaterial's may also offer new opportunities for the reduction of environmental pollution.

**Nanotechnology for energy:**

India is using nanotechnology to develop more efficient solar cells and batteries. For example, the Indian company Tata Chemicals has developed a nanotech-based energy storage system that can store energy from renewable sources such as wind and solar. There are four main types of intentionally produced nanomaterials: carbon-based, metal-based, dendrimers, and nanocomposites. Carbon-based nanomaterials are intentionally produced fullerenes.

Nanotechnology tools and instruments are the hardware, software and supplies used to measure and manipulate structures on the Nano scale. They include microscopes, probes, lithography systems, manipulation and fabrication systems, software and other accessories. Rarely are these instruments unique to nanotechnologies. Metal-Based Nanoparticles. The metal-based nanoparticles such as silver, gold, copper, iron, zinc, platinum, and so on, received much attention in medicine. Faraday (1857) showed the metal nanoparticles can exist in solution. Their ability to slip through the cell's membrane makes gold nanoparticles ideal delivery devices for medications to healthy cells or fatal doses of radiation to cancer cells. However, a single milligram of gold nanoparticles currently costs about \$80 (depending on the size of the nanoparticles)

Nano biotechnology has immense potential in diagnosis, treatment, and prevention of disease; however, more substantial research needs to be conducted to eliminate the potential challenges in clinical trials, regulatory issues, and toxicity in order to advance medical science and healthcare in the future. Nanotechnology has direct beneficial applications for medicine, Foods and beverages, Information Technology, Energy, Appliances and textiles, Household and cosmetics etc.

**Medicine: -**

Cancer treatment, Bone treatment, Drug delivery, Appetite control, Drug development, Medical tools, Diagnostic tests, Imaging etc.

**Foods and beverages: -**

Advanced packing materials, sensors and lab-on-chips for food quality testing etc.

**Information Technology: -**

Smaller, Faster, More Energy efficient and powerful computing, other IT- based systems.

**Energy: -**

More efficient and effective technologies for energy production- solar cells, Fuel cells Batteries etc.

**Appliances and textiles: -**

Stain proof, water proof and wrinkle free textiles etc.

**Household and cosmetics: -**

Scratch free products, paints and better cosmetics.

**Potential risks of Nanotechnology: -**

**Health issues: -**

**A.** Nano particles could be inhaled, swallowed, absorbed through skin

**B.** They trigger inflammation and weaken the immune system and interfere with regulatory mechanism of enzymes and proteins.

**Environmental issues: -** Nano particles could accumulate in soil, water and plants

Finally it is clear that there are a lot of obstacles for to develop and construct the billionth of metre size particles from the various elements and to study their characteristics for the applications of welfare of living organism. These particles are not user-friendly, not seen by naked eyes or regular microscope they require specific tools so unknowingly they may be harmful to for living organism as well the various ecosystems.

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## Chapter -29

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### **Cestodes (Tapeworm) Parasitic Disease in Marine Fishes**

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#### **Abstract**

Parasites have a wide range of distribution in all groups of animals. They are more abundant than free-living animals, Parasitism is a natural way of life among parasitic diseases are the major public health problems leading to morbidity and mortality in tropical countries including India. Several types of parasitism are recognized. Beside intestinal parasites various types including cestodes, nematodes and trematodes. Helminth infections are major parasitic diseases in India, which causes anaemia, dysentery, diarrhoea and illness. They adversely affect not only the general health and productivity of adults but also mental and physical growth of children, especially suffering from malnutrition. The tapeworm causes great suffering to the host i.e. fresh water as well as marine water fishes. Therefore the study of cestodes is great antiquity and primitive, people knew many of the worms. The fishes are economically beneficial to human population but the tapeworms present in them cause considerable damage. This chapter summarizes knowledge on parasitological research associated with Cestodes marine fish species

#### **Introduction**

Most parasite species rarely cause problems in the natural environment but in aquaculture, parasites often cause serious outbreaks of disease. The tapeworms (Cestodes) when live in the intestine of hosts, they utilize food from the gastrointestinal tract. Metabolism of these cestodes depends on the feeding habits and the rich nourishment available in the gut of the host. These worms use this nourishment for their normal development and growth. The metabolic and in vitro studies suggest that a complex nutritional relationship occurs between a cestode and its host.

It has been observed in some cestodes that they are capable of fixing CO<sub>2</sub>. Thus, it is clear that the parasites use the waste metabolic materials from the hosts intestinal mucosa very efficiently, where as these are capable of taking the nutritional material by direct contact with the mucosal wall [01].

It has been known more than hundred years that parasitic worms contains polysaccharides, the metabolism of intestinal worms are characterized by the fermentation of Carbohydrates, It is obvious that many endoparasites have a pronounced carbohydrate metabolism[2]. The literature we conclude that the Carbohydrate play an eminent role in cestode than in the most other parasitic worms which are distinguished by different growth patterns. These carbohydrates are utilized exogenously, but their mechanism is not known. The cestodes *Hymenolepis diminuta* [3] *Taenia taeniaeformis*[4] and *Acanthocephalan Polymorphus minutus* [5] absorb glucose against concentration gradients. These are further more typical inhibitors of active transport e.g. phloridzin interfere effective with the glucose uptake of cestodes [3] and at least in *Taeniaeformis* glucose absorption has an absolute sodium requirement [6] clearly corresponding closely to the sodium pump of vertebrate tissues.

Tapeworms (Cestoda) represents a species rich (about 5000 species) group of flatworms (Neodermata) parasitizing all groups of vertebrates including humans, with about 1000 species parasitizing elasmobranchs and almost 500 occurring in teleosts as adults. They are common parasites of cultured fish, both as adults and larvae (metacestodes), but only few adult tapeworms are actually pathogenic for their fish hosts. In contrast, cestode larvae can be harmful for fish, especially plerocercoids migrating throughout their tissue and internal organs. Current knowledge of host-parasite relationships, including immune response of fish infected with tapeworms, is still insufficient to enable adequate control of cestodoses, and most data available were obtained several decades ago. Treatment of fish infected with adult tapeworms is effective, especially with praziquantel, whereas the treatment of metacestodes is problematic. Control measures include interruption of the complex life cycle and prevention of transport of uninspected fish to new region.[7]

Cestodes also called tapeworms are ribbon like flat worms. They infect the alimentary tract, muscle or other internal organ of fish. The clinical sign when fish is affected by cestode parasite are variable degree of dropsy, distended abdomen and reduced in activity. Cestoda are all endo parasites of vertebrates with over 5000 species so far described. Most of them require at least one intermediate host and complete their life cycle as adults in the definitive hosts. Two life cycle stages are represented in fish: adults inhabit the intestine, and plerocercoid larvae of the same or different species are found in the viscera and musculature; the first-stage larvae (procercooids) are generally found in aquatic crustacean [8]

Pathological and physiological changes caused by fish tapeworms include alterations in growth rate, effects on enzyme activity, reduction in reproductive capacity, liver dysfunction and alterations of several blood parameters [9]. Because of large size, adult tapeworms may reduce the lumen of the gut in heavy infections and thus affect movement of food through the intestine [10]

The Asian fish tapeworm causing bothriocephalosis has been associated with reduction in body and organ weight and caused mortalities of juvenile fish, with up to 100% mortality in hatchery ponds [11]. Heavy tapeworm burdens cause blockage of the intestine and severe pathological changes, leading to anorexia, emaciation, signs of anaemia, reduced growth, condition and survival [12]

Fish serving as intermediate hosts are infected with cestode larvae, that is histozoic metacestodes, which migrate through, and eventually encyst in the tissues, or may remain free (unencysted) in the peritoneal cavity. This injurious interaction often evokes an inflammatory response, accompanied by cell proliferation and followed by fibrosis [13] The extent of injury depends on a number of factors. Because of their size, adult tapeworms can cause obstruction of the intestinal lumen (ileus verminosus), especially in fish fry, which may lead to mortality of heavily parasitized fish. The causes of mortality include destruction of the tissue-water interface, extensive haemorrhages, necrosis, fibrosis, oedema and discoloration [14].

Whereas such serious infections are uncommon, attachment of adult cestodes in the fish gut often elicits inflammatory reaction with necrotic changes.[13]

Borucinska (2008) reported in their chapter on cestodes in fish, presented a very non-orthodoxical recommendation how to avoid problems related to cestode infections in aquaculture: 'It might be thus wiser to abandon man-made "inventions" like intensive aquaculture, thus eliminating the unnaturally harmful parasite-host relationships created by such artificial habitats.[15]

## **Conclusion**

As a Chapter described that large number of parasites infects fish some of these infections are highly pathogenic. Most of the existing knowledge of host-parasite relationships including pathology of tapeworm infections and immune response of infected fish hosts was accumulated several decades ago. In contrast, little attention is currently paid to diseases of cultured fish caused by these parasites. However, continuous development of aquaculture, global trade and climate change may pose new challenges. Considerable effort is especially required to better understand the fish immune system and its function during infections with tapeworms. A better knowledge of the drugs, their efficacy and mechanisms of action is also much needed. There should be a clear nationwide policy and strategy concerning fish disease prevention and control at large and endo and ecto-parasites in particular.

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## Chapter -30

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### **GC-HRMS analysis of some of the bioactive constituents in *Albizia procera* (Roxb.) Benth. methanolic leaf and bark extract.**

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#### **ABSTRACTS**

*Albizia procera*(Roxb.) Benth.is a large deciduous unarmed tree, 18-24 m in height and 1-8 m over in girth found along the streams in deciduous forest. It is occur in Central and E. Himalayas, Bihar, WestBengal, Satpura range, Gujrat, South India and Andaman. The plant is used for stomach and intestinal diseases, and during pregnancy. All parts of the plant are reported to show anti-cancerous activity and decoction of bark is given in rheumatism and haemorrhage. It is given to the water buffalo with salt as medicine and act as insecticidal. It has potential analgesic and CNS depressant activity. Apart from this bark extract has anti-arthritic and antioxidant activity and gum is obtained from the plant. Present investigation was designed to confirm the presence of bioactive constituents in leaf and bark methanolic extract of the plant through GC-HRMS analysis. This study revealed that bark extract of the plant contain three compound 1-Butanamine, N-butylidene; Acetic acid, diethyl and Myo-inositol,4-C-methyl. Leaves extract of the plant showed that it contain 10 compounds i.e.  $\alpha$ -Ethylcaproic acid; Pentadecanecarboxylic acid; Carbamic acid, 2-chloroethyl ester; 3-O-Methyl-  $\alpha$ -D-glucose; Hexanoic acid, 2-ethyl-; n-Hexadecanoic acid; Bromacetocarbamide; 9,12,15-Octadecatrienoic acid, methyl ester; 1-Docosene and Hexyl orthoborate. This GC-HRMS profiling can be used for biochemical marker and valuable tool for identification of this medicinally important plant.

**Keywords** – *Albizia procera*(Roxb.) Benth., GC-HRMS, medicinal plant.

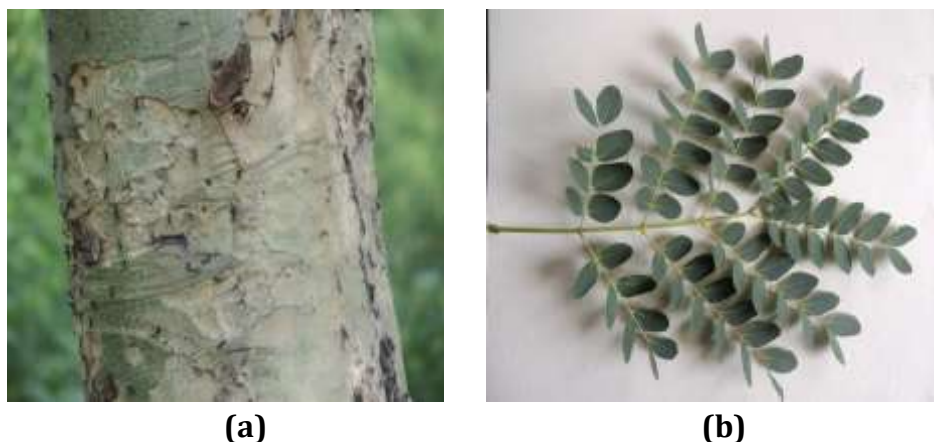
## **INTRODUCTION:-**

Medicinal plants represent the most ancient form of medication, used for thousands of years in traditional medicine in many countries around the world. The empirical knowledge about their beneficial effects was transmitted over the centuries within human communities (1). These medicinal plants consider as a rich resources of ingredients which can be used in drug development and synthesis. Besides that these plants play a critical role in the development of human cultures around the whole world (2). The use of medicinal plants for the treatment of diseases dates back to the history of human life, that is, since human beings have sought a tool in their environment to recover from a disease, the use of plants was their only choice of treatment (3). In the recent years, research on medicinal plants has attracted a lot of attentions globally. Large body of evidence has accumulated to demonstrate the promising potential of Medicinal Plants used in various traditional, complementary and alternate systems of treatment of human diseases.

*Albizia procera* (Roxb.) Benth. is a large deciduous unarmed tree, 18-24 m in height and 1-8 m over in girth found along the streams in deciduous forest (4,5,6). It isoccur in Central and E. Himalayas, Bihar, West Bengal, Satpura range, Gujrat, South India and Andaman (7). The plant is used for stomach and intestinal diseases, and during pregnancy (8). All parts of the plant are reported to show anti- cancerous activity and decoction of bark is given in rheumatism and haemorrhage (9). It is given to the water buffalo with salt as medicine and act as insectisidal (10). It has potential analgesic and CNS depressants activity. Apart from this bark extract has anti-arthritis and antioxidant activity and gum is obtained from the plant. It is used as antioxidant, anti-inflammatory, anticarcinogenic and prevent diabetes, cardiovascular diseases and Alzheimer disease (11).

In the recent year advancement in of chromatographic techniques and spectral fingerprints plays an important role in the quality control of complex herbal medicines (12, 13). The present study was designed to investigate the presence of various phytochemicals and bioactive constituents' presents in the methanolic leaf and bark extract of *Albizia procera*(Roxb.) Benth with the help of the GC-HRMS technique.





**Fig.1:Showing *Albiziaprocera*(Roxb.)Benth morphology (a)Bark and(b)Leaf**

## **MATERIALS AND METHODS**

### **Collection of Plant Material**

Leaves and bark of *Albiziaprocera* (Roxb.)Benth. Was collected from Kinwat forest(19°32.479"N 078° 14.543"E) in Nanded district of Maharashtra. Specimens were identified and authenticated by Herbarium, Department of Botany, Dr.Babasaheb Ambedkar Marathwada University, Aurangabad (Accession No- 17396). Freshly collected leaves and stem bark of *Albiziaprocera* (Roxb.)Benth.was dried in shade and pulverized to coarse powder.The powder was stored in an airtight container and kept in a cool,dark, and dry place(14, 15)

### **Method of preparation of methanol extract**

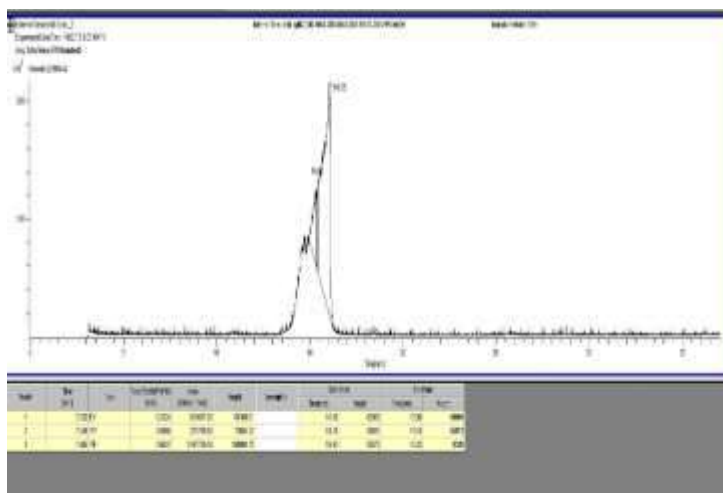
The extraction was done by hot continuous method using Soxhlet apparatus.The 25gm powder of leaves and bark were extracted using 250 ml methanol for 72 hours. The methanolic extract of bark and leaves of *Albizia procera* (Roxb.) Benth. Were used for this study(16).

### **Gas chromatography-Higher solution spectroscopy.(GC-HRMS)**

It was carried out from sophisticated analytical instrument facility, Indian Institute of Technology, Pawai. 1 mg/ml extract dissolved in methanol was used for the analysis of *Albizia procera*(Roxb.) Benth. Were used for this study(17).

**GC-HRMS of *Albizia procera* Benth. Bark extract.**

GC-HRMS chromatogram detected the presence of three peaks in *Albizia procera* Benth. bark extract. This peaks were obtained at 15.32, 15.40 and 16.05 min. this chromatogram also provided the area and height along with the starting point and ending point of height in terms of the min. of each peak (Fig. No. 2). This data was obtained through the gas chromatography high resolution mass spectroscopy instrument. It was used for the identification of the compound in the extract.

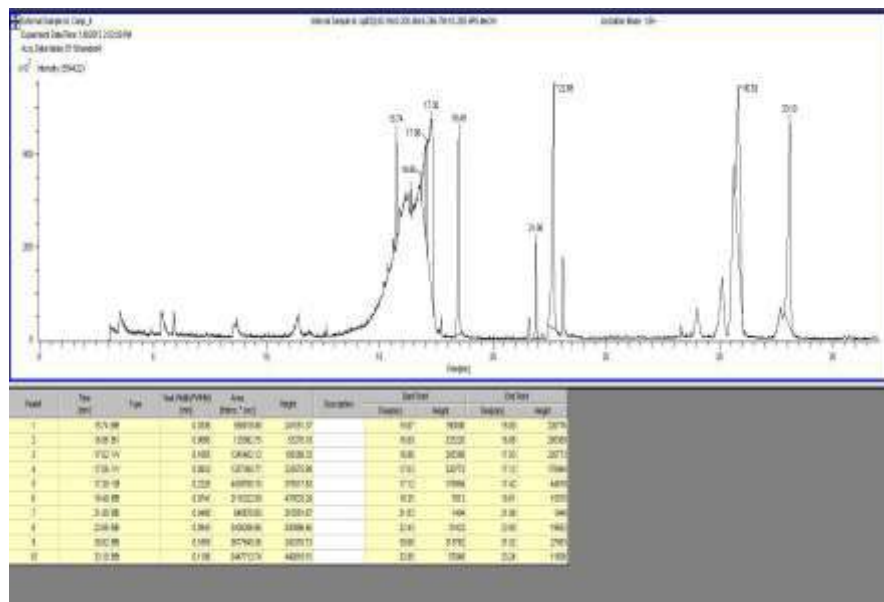


**Fig No. 2 - GC-HRMS chromatogram of *Albizia procera* Benth. Bark extract.**

**GC-HRMS of *Albezia procera* Benth. leaves extract.**

GC-HRMS chromatogram detected the presence of ten peaks in *Albizia procera* Benth. Leaves extract. This peak was obtained at 15.74, 16.86, 17.02, 17.6, 17.30, 18.49, 21.90, 22.68, 30.82 and 33.10 min. This chromatogram also provided the area and height along with the starting point and ending point of height in terms of the min. of each peak (Fig. No.3). This data was obtained through the high resolution mass spectroscopy instrument which is coupled with the GC-HRMS. It

was used for the identification of the compound in the extract.



**Fig No. 3 - GC-MS chromatogram of *Albezia procera* Benth. leaves extract.**

**Compound present in *Albezia procera* Benth.**

Peaks obtained through GC-MS were search for the compound identification with the help National Institute of Standards and Technology (NIST). This study revealed that bark extract of the plant contain three compounds 1-Butanamine, N-butyridene; Acetic acid, diethyl; Myo-inositol,4-C-methyl. Leaves extract of the plant showed that it contain  $\alpha$ -Ethylcaproic acid; Pentadecanecarboxylic acid; Carbamic acid, 2-chloroethyl ester; 3-O-Methyl- d -glucose; Hexanoic acid, 2-ethyl-; n-Hexadecanoic acid; Bromacetocarbamide; 9,12,15-Octadecatrienoic acid, methyl ester; 1-Docosene and Hexyl orthoborate this compound (Table No. 42).

**Table No. 1 - Compound present in *Albezia procera* Benth.**

Sr. No.	Bark	Leaves
1	1-Butanamine, N-butylidene	$\alpha$ -Ethylcaproic acid
2	Acetic acid, diethyl	Pentadecanecarboxylic acid
3	Myo-inositol,4-C-methyl	Carbamic acid, 2-chloroethyl ester
4		3-O-Methyl- d -glucose
5		Hexanoic acid, 2-ethyl-
6		<u>n-Hexadecanoic acid</u>
7		Bromacetocarbamide
8		9,12,15-Octadecatrienoic acid, methyl ester
9		1-Docosene
10		Hexyl orthoborate

Different compound in the *Albizia* species and other leguminosae plants were identified by several workers through the GC-MS technique. GC-MS analysis of *Albizia procera* Benth. ethanolic extract of aerial part showed the presence of twelve compounds. These twelve compounds were as follow 3-O-Methyl-d-glucose, 13-Tetradecene-11-yn-1-ol, 3-chloro-N-(4-methoxyphenyl)-, Squalene, 6,9,12 Octadecatrienoic acid, phenylmethylester, (Z,Z,Z),9,12- Octadecadienoic acid (Z,Z)-, phenylmethyl ester, Phytol, 1,10-Decanediol, 3-Pentanol, 2,3-dimethyl-, Decanoic acid, ethyl ester, 1-Undecyne and Didodecyl phthalate, Benzo[b]thiophene-2 carboxamide (18). GC-MS analysis of *Albizia saman* oil extract showed the presence of fatty acids (69.1%), oxygen containing monoterpenes (16.8%), monoterpene hydrocarbons (4.0%), sesquiterpene hydrocarbons (3.6%), aliphatic compounds (3.5%), oxygen containing sesquiterpenes (2.7%) (19). 18 compounds from *Desmodium gyrans* leaves ethanolic extract and 10 quinolizidine alkaloids from aerial parts of *Genista sandrasica* Hartvig & Strid were identified by GC-MS(20,21). Methanolic root extracts of *Pseudarthria viscida* Wight and Arn and *Desmodium gangeticum* (Linn) DC showed

the presence of 43 and 18 compounds respectively (22).

## **CONCLUSION**

Through GC-HRMS different important biologically active phytoconstituents were identified from the leaves and bark methanolic extracts. GC-HRMS profiling can be used for biochemical marker and valuable tool for identification of this medicinally important plants. The presence of various chemical compounds confirms the application of *Albizia Procera*(Roxb.)Benth. for various remedies by traditional practitioners.

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## Chapter -31

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### Synthesis, Characterization and Antimicrobial activity of metal complexes novel 1,3-diones

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#### Abstract:-

The synthesized 1,3-dione have many uses such as important intermediates for the synthesis of core heterocycles compounds such as pyrazole, isoxazole, and triazole in medicinal chemistry. Each metal complexes synthesized by metal nitrate with 1, 3 dione (L) in the ratio 1:2 stoichiometry. The characterization was carried out by elemental analysis, UV-Visible spectroscopy, infrared spectroscopy,  $^1\text{H-NMR}$ ,  $^{13}\text{C-NMR}$ , magnetic susceptibility, molar conductance and TGA for structural formulae study. The synthesized 1, 3-dione and their transition metal complexes have been screened for in vitro antibacterial, and antifungal activity using Resazurin 96 well plate method. It achieves more accurate minimum inhibitory concentration (MIC).

**Keywords:-** 1, 3-dione, Metal-complexes, Magnetic susceptibility, TGA, Antimicrobial activity

#### Introduction:-

1,3-diketones are used in synthesis of various heterocycle compounds such as pyrazol [1], isoxazole [2], triazole [3], flavones [4], benzodiazepine [5] and pyrimidine [6]. The complexes of Europium (III) have excellent luminescent property, [7] as chelating agent and extractants for lanthanide ions.[8-9]The 1,3-diones are very important molecules showing anti-sunscreen activity, antibacterial, antiviral, insecticidal, antioxidant, potential prophylactic antitumor and more important they proven to be an important pharmacophore of HIV-1 Integrase (IN) inhibitors. A characteristic of metals is that they easily lose electrons from metal to form positively charged ions, which tend to be soluble in biological fluids.

The metal ions are electron deficient, most biological molecules such as proteins and DNA are electron rich. The attraction of these opposite charges leads to a general tendency for metal ions to interact with biological molecules. In hemoglobin, an iron ion's strong affinity to bind with oxygen in the respiration process. Zn ions provide the structural framework for the zinc fingers that regulate the function of genes in the nuclei of cells also zinc is a natural component of insulin, to regulate sugar metabolism. Metals such as copper, zinc, iron, and manganese are important in catalytic proteins which initiate chemical reactions needed for life.

### **General Scheme Synthesis of 1,3-diones and its metal complexes:-**

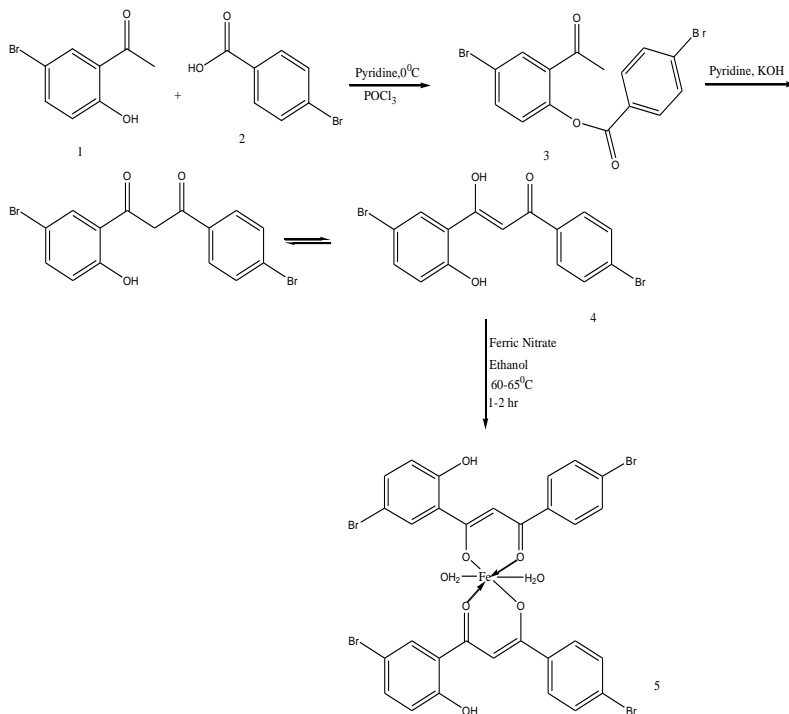
The compound substituted benzoic acid (0.01 mol) and substituted 2-hydroxy acetophenone (0.01 mol) was dissolved in 20 ml dry pyridine cool the mixture below 5°C add phosphorous oxychloride (1-2 ml) drop wise and continuously stirrer for 5-6 hrs form ester. Then using Baker Venkataraman rearrangement, ester dissolve in pyridine and add powdered KOH and stirrer for about 2-3 hrs. Then it was poured over crushed ice and acidified with dil. HCl. The resulting solid was crystallized from ethanol. The yield of 1,3-diones is 65-70%.

### **Synthesis of metal complexes of 1,3 dione:-**

A Mixture of 1,3 dione (L) (5 mmol) and 2.5 mmol of appropriate metal nitrate added in anhydrous 30 ml ethanol and the resulting mixture was refluxed at 60-65°C for 2-3 hour whereupon the complex precipitation occurs after the addition of alcoholic ammonia. The precipitated colored solid complex washed with ethanol and crystallized by using dichloromethane. The yield of complexes near about 62- 72%.



## General scheme of reaction



## A) IR Spectra:-

The characteristic infrared spectral data of 1,3 dione (L) and their metal complexes are reported. The carbonyl group ( $>C=O$ ) stretching frequency of ligand (L) appearance at 1600-1620  $\text{cm}^{-1}$ . The appearance of frequency at 1575-1593  $\text{cm}^{-1}$  due to ( $-C=C-$ ) double bond and the bond (C-O) appear at 1200-1225  $\text{cm}^{-1}$ . The metal complexes of ligand (L) show IR frequency of carbonyl group ( $>C=O$ ) at 1591-1610  $\text{cm}^{-1}$  which were lower than IR frequency of 1,3 dione (L). This lowering stretching frequency indicates that ligands coordinated with the transition metal ions. In addition, new band at 509-534  $\text{cm}^{-1}$  observed due to metal-oxygen (M-O) bond vibrations in metal complexes which were absent in ligands. This confirms of metal ions coordinate with ligand via oxygen.

B) <sup>1</sup>H NMR and <sup>13</sup>C NMR Spectra:-

The <sup>1</sup>H NMR spectral data of the 1,3-dione (L) shows singlet at  $\delta$  15.00 ppm due to enolic proton, a singlet at  $\delta$  11.94-12.00 ppm due to

phenolic proton adjacent to the carbonyl group which confirms the formation of 1,3-dione.

In the  $^{13}\text{C}$  NMR of 1,3-diones (L) peak appeared at  $\delta$  190.0 - 194.86 ppm corresponds to carbonyl carbon (C=O) and enolic carbon (C-O) at  $\delta$  178.47-179 ppm. The signal at  $\delta$  98.10 ppm appeared shows methine linkage.

### C) Magnetic Susceptibility and Molar Conductance:-

The molar conductance values were obtained in  $\Omega^{-1}\text{cm}^2\text{mol}^{-1}$  at room temperature using DMSO as a solvent and results are recorded. The molar conductance values were obtained in the range 13-20  $\Omega^{-1}\text{cm}^2\text{mol}^{-1}$ . The conductance values show metal complexes non-electrolytic in nature.[10] All metal complexes were paramagnetic in nature except Zinc complexes were diamagnetic due to non-availability of unpaired electrons

### D) UV-Visible Spectroscopy:-

The electronic spectra of the 1,3 dione exhibited bands in the regions of 259.5 and 361.5 nm, which can be assigned to intramolecular  $\pi \rightarrow \pi^*$  and  $n \rightarrow \pi^*$  electronic transitions due to the aromatic and carbonyl groups. In all metal complexes intramolecular  $\pi \rightarrow \pi^*$  and  $n \rightarrow \pi^*$  electronic transitions occur but weak d-d transition not found. All metal complexes have center of symmetry the 3d orbital cannot mix with the 3p orbital in such mixing being symmetry forbidden. The octahedral complexes with center of symmetry so  $T_{2g}-E_g$  transitions were very weak not observed.

### E) Thermo Gravimetric Study of Some Metal Complexes:-

The TG/DT analysis of some metal complexes, the temperature range usually selected  $25^\circ\text{C}$  to  $600^\circ\text{C}$  at the rate  $10^\circ\text{C}/\text{Min}$  in Nitrogen atmosphere using  $\alpha\text{-Al}_2\text{O}_3$  as reference. The thermogram curve of L-Fe Complex shows weight loss 6.018% up to  $225^\circ\text{C}$  clearly indicate removal of surface two coordinated water molecules.[11-12] A sudden weight loss (48.05%) from  $225^\circ\text{C}$  to  $475^\circ\text{C}$  was due to loss of one phenyl ring with two hydroxy and one carbonyl group. Further, the weight loss (29 %) from  $475^\circ\text{C}$  to  $566^\circ\text{C}$  corresponds to the

decomposition of two phenyl ring and a propane-1, 3-dione moiety. On further heating above 566°C the weight remained constant corresponding the formation of Ferrous oxide. In DTA graph small endothermic peak observed at 123°C which indicates dehydration process and strong exothermic at 501°C indicates thermal decomposition of L-Fe Complex.

**F) Antimicrobial Activities:-**

The antimicrobial activity carried out by Resazurin method which is developed by Drummond and Waigh in 2000.[13] This method is simple, sensitive, rapid, and reliable and achieves more accurate minimum inhibitory concentration (MIC). In this method Resazurin used as an indicator and it was prepared by dissolving 270 mg tablet in 40 ml of sterile distilled water. The color changes from purple to pink or colorless were recorded as positive. MIC values taken as color change occur at lowest concentration. The average of three values of MIC for the test material and bacterial strain has been considered.

**MIC (µg/ml) values of 1-(5-bromo-2-hydroxyphenyl)-3-(4-bromophenyl)-propane- 1,3-dione (L) and its metal complexes using modified Resazurin assay method.**

Compounds	Antibacterial activity				Antifungal activity	
	Gram positive		Gram negative		C.albicans	S.cerevisiae
	B.subtilus	S.aureus	E.coli	P.aerugenosa		
L	<50	<50	100	50	150	150
L- Fe	100	<50	100	100	150	100
L- Co	100	<50	50	50	150	100
L-Ni	50	<50	100	50	150	100
L-Cu	50	<50	100	50	100	50
L-Zn	100	<50	100	100	100	50
Tetracycline	2	1	4	1	-	-
Amphotericin B	-	-	-	-	1.25	1.25

**Conclusions:-**

The above chapter present research work shows 1,3-dione and its transition metal complexes reveals that 2:1 stoichiometry ratio for all the prepared metal complexes. These complexes were characterized by various physicochemical and spectral analyses. It shows non-electrolytic nature and octahedral geometry with center of symmetry. The thermal stability was evaluated by TG method whose results revealed good thermal stability for the synthesized metal complexes. As per results, it can be seen that the 1,3-dione and its metal complexes shows considerable antimicrobial activity against all tested bacteria and fungi compared with antibiotics Tetracycline and Amphotericin B.

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## Chapter -32

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### Thermoelastic Problem on of A Steady State Condition on Thin Circular Plate

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#### ABSTRACT

This paper is concerned with the direct steady-state and transient thermoelastic problem in which we need to determine the temperature, displacement and stress functions of a thin circular plate. The finite Hankel transform and Laplace transform techniques are used.

**KEYWORDS:-** Circular plate, direct thermoelastic problem, Hankel transform and Laplace transform.

#### INTRODUCTION:-

Nowacki (1957) has determined steady-state thermal stresses in a circular plate subjected to an axisymmetric temperature distribution on the upper face with zero temperature on the lower face and the circular edge. Further Roy Chaudhari (1972) has succeeded in determining the quasi-static thermal stresses in a thin circular plate subjected to transient temperature along the circumference of a circular plate over the upper face with lower face at zero temperature and fixed circular edge thermally insulated. Wankhede (1982) has determined the quasi-static thermal stresses in a thin circular plate subjected to arbitrary initial temperature on the upper face with lower face at zero temperature. Ishihara et al (1997) considered a circular plate and discuss the transient thermo elasto plastic bending problem making use of the strain increment theorem.

Recently Meshram, Deshmukh and Wankhede (2002) has determined the transient temperature, displacement and stress function on the upper face of a thin circular plate with boundary condition that the temperature is maintained at  $f(r)$  on upper face

while zero temperature on lower face and heat flux is maintained on outer curved surface. In the present problem, an attempt is made to study the direct steady-state and transient thermoelastic problem to determine the temperature, displacement and stress functions of a thin circular plate of thickness  $h$  occupying the space  $D: 0 \leq r \leq a, 0 \leq z \leq h$  with the known boundary conditions. The finite Hankel transform and Laplace transform techniques are used to find the solution of the problem. Numerical estimate for the temperature distribution is obtained and depicted graphically.

**MATERIAL AND METHODS**  
**FINITE HANKEL TRANSFORM**

If  $f(x)$  satisfies Dirichlet's conditions in the interval  $(0,a)$  then it's finite Hankel transform in that rang is defined to be

$$\bar{f}_\mu(\xi_i) = \int_0^a x f(x) J_\mu(x\xi_i) dx$$

Where  $\xi_i$  is the root of the transcendental equation  $J_\mu(a\xi_i) = 0$

Then at any point of  $(0,a)$  at which the function  $f(x)$  is continuous

$$f(x) = \frac{2}{a^2} \sum_i \bar{f}_\mu(\xi_i) \frac{J_\mu(x\xi_i)}{[J'_\mu(a\xi_i)]^2}$$

Where the sum is taken overall the positive roots of the equation

**1.PROERTIES OF TH HANKEL TRANSFORM**

If  $f(x)$  satisfies Dirichlet's conditions in the close interval  $[0,a]$  then

1.Finate Hankel transform of  $\frac{\partial f}{\partial x}$ , i.e.

$$H_\mu \left[ \frac{\partial f}{\partial x} \right] = \int_0^a \frac{\partial f}{\partial x} x J_\mu(x\xi_i) dx$$

$$= \frac{\xi_i}{2\mu} \left[ (\mu - 1) H_{\mu+1} \{f(x)\} - (\mu + 1) H_{\mu-1} \{f(x)\} \right]$$

2.  $H_\mu \left[ \frac{\partial^2 f}{\partial x^2} + \frac{1}{x} \frac{\partial f}{\partial x} \right] = \frac{\xi_i}{2} \left[ -H_{\mu-1} \left( \frac{\partial f}{\partial x} \right) + H_{\mu+1} \left( \frac{\partial f}{\partial x} \right) \right]$

If  $f(x)$  satisfies Dirichlet's conditions in the range  $b \leq x \leq a$  and if its finite Hankel transform in that range is defined to be

$$H[f(x)] = \bar{f}_\mu(\xi_i) = \int_b^a x f(x) [J_\mu(x\xi_i)G_\mu(a\xi_i) - J_\mu(a\xi_i)G_\mu(x\xi_i)] dx$$

In which  $\xi_i$  is the root of transcendental equation

$$[J_\mu(\xi_i b)G_\mu(\xi_i a) - J_\mu(\xi_i a)G_\mu(\xi_i b)] = 0$$

Then at which the function is continuous,

$$f(x) = \sum_i \frac{2\xi_i^2 J_\mu^2(\xi_i b) \bar{f}_\mu(\xi_i)}{J_\mu^2(a\xi_i) - J_\mu^2(b\xi_i)} [J_\mu(x\xi_i)G_\mu(a\xi_i) - J_\mu(a\xi_i)G_\mu(x\xi_i)]$$

**PROPERTY OF THE HANKEL TRANSFORM**

$$\begin{aligned} & \int_a^b \left[ \frac{\partial^2 f}{\partial x^2} + \frac{1}{x} \frac{\partial f}{\partial x} \right] [J_\mu(x\xi_i)G_\mu(a\xi_i) - J_\mu(a\xi_i)G_\mu(x\xi_i)] dx \\ &= -\xi_i^2 \bar{f}_\mu(\xi_i) + a [J_\mu(x\xi_i)G_\mu(a\xi_i) - J_\mu(a\xi_i)G_\mu(x\xi_i)]_{x=a} \\ & \quad + b [J_\mu(x\xi_i)G_\mu(a\xi_i) - J_\mu(a\xi_i)G_\mu(x\xi_i)]_{x=b} \\ &= -\xi_i^2 \bar{f}_\mu(\xi_i) \end{aligned}$$

**2. STATEMENT OF THE PROBLEM**

Consider a thin circular plate of thickness  $h$  occupying the space  $D : 0 \leq r \leq a, 0 \leq z \leq h$ . The differential equation governing the displacement function  $U(r,z)$  as [3] is

$$\frac{\partial^2 U}{\partial r^2} + \frac{1}{r} \frac{\partial U}{\partial r} = (1 + \nu) a_t T \tag{2.1}$$

with  $U_r = 0$  at  $r = a$  (2.2)

where  $\nu$  and  $a_t$  are the Poisson's ratio and the linear coefficient of thermal expansion of the material of the plate and  $T$  is the temperature of the plate satisfying the differential equation

$$\frac{\partial^2 T}{\partial r^2} + \frac{1}{r} \frac{\partial T}{\partial r} + \frac{\partial^2 T}{\partial z^2} = 0 \tag{2.3}$$

subject to the boundary conditions

$$\left[ T(r, z) + \frac{\partial T(r, z)}{\partial z} \right]_{z=h} = f(r) \tag{2.4}$$



$$\left[ T(r, z) + \frac{\partial T(r, z)}{\partial z} \right]_{z=0} = 0 \quad (2.5)$$

$$T(a, z) = 0 \quad (2.6)$$

The stress functions  $\sigma_{rr}$  and  $\sigma_{\theta\theta}$  are given by

$$\sigma_{rr} = -2\mu \frac{1}{r} \frac{dU}{dr} \quad (2.7)$$

$$\sigma_{\theta\theta} = -2\mu \frac{d^2U}{dr^2} \quad (2.8)$$

where  $\mu$  is the Lamé's constant, while each of the stress functions  $\sigma_{rz}$ ,  $\sigma_{zz}$ ,  $\sigma_{\theta z}$  are zero within the plate in the plane state of stress. The equations (2.1) to (2.8) constitute the mathematical formulation of the problem under consideration.

**3. RESULT AND DISCUSSION:-**Applying finite Hankel transform defined in [5] to the equations (2.3) to (2.5) and using (2.6) one obtains

$$\frac{d^2 \bar{T}}{dz^2} - \lambda_n^2 \bar{T} = 0 \quad (3.1)$$

$$\left[ \bar{T}(\lambda_n, z) + \frac{d\bar{T}(\lambda_n, z)}{dz} \right]_{z=h} = \bar{f}(\lambda_n) \quad (3.2)$$

$$\left[ \bar{T}(\lambda_n, z) + \frac{d\bar{T}(\lambda_n, z)}{dz} \right]_{z=0} = 0 \quad (3.3)$$

where  $\bar{T}$  denotes the finite Hankel transform of  $T$  and  $\lambda_n$  is the Hankel transform parameter.

The equation (3.1) is a second order differential equation whose solution is given by

$$\bar{T}(\lambda_n, z) = Ae^{\lambda_n z} + Be^{-\lambda_n z} \quad (3.4)$$

where  $A, B$  are constants.

Using (3.2) and (3.3) in (3.4) we obtain the values of  $A$  and  $B$ . Substituting these values in (3.4) and then inversion of finite Hankel transform leads to

$$T(r, z) = \frac{2}{a^2} \sum_{n=1}^{\infty} \frac{\bar{f}(\lambda_n)}{1 - \lambda_n^2} \left( \frac{J_0(\lambda_n r)}{J_1^2(\lambda_n a)} \right) \left[ \frac{\sinh(\lambda_n z)}{\sinh(\lambda_n h)} - \frac{\lambda_n \cosh(\lambda_n z)}{\sinh(\lambda_n h)} \right] \quad (3.5)$$

where  $\bar{f}(\lambda_n) = \int_0^a r f(r) J_0(r) dr$

and  $\lambda_n$  are the positive roots of the transcendental equation  $J_0(\lambda_n a) = 0$ .

Equation (3.5) is the desired solution of the given problem.

#### 4. DETERMINATION OF THERMOELASTIC DISPLACEMENT

Substituting the value of  $T(r, z)$  from equation (3.5) in (2.1) we obtain the thermoelastic displacement function  $U(r, z)$  as

$$U(r, z) = -\frac{2(1+\nu)a_t}{a^2} \sum_{n=1}^{\infty} \frac{\bar{f}(\lambda_n)}{\lambda_n^2(1-\lambda_n^2)} \left( \frac{J_0(\lambda_n r)}{J_1^2(\lambda_n a)} \right) \left[ \frac{\sinh(\lambda_n z)}{\sinh(\lambda_n h)} - \frac{\lambda_n \cosh(\lambda_n z)}{\sinh(\lambda_n h)} \right] \quad (4.1)$$

#### 5. DETERMINATION OF STRESS FUNCTIONS

Using equation (4.1) in (2.7) and (2.8) the stress functions are obtained as

$$\sigma_{rr} = \frac{4\mu(1+\nu)a_t}{a^2 r} \sum_{n=1}^{\infty} \frac{\bar{f}(\lambda_n)}{\lambda_n(1-\lambda_n^2)} \left( \frac{J_1(\lambda_n r)}{J_1^2(\lambda_n a)} \right) \left[ \frac{\sinh(\lambda_n z)}{\sinh(\lambda_n h)} - \frac{\lambda_n \cosh(\lambda_n z)}{\sinh(\lambda_n h)} \right] \quad (5.1)$$

$$\sigma_{\theta\theta} = \frac{4\mu(1+\nu)a_t}{a^2} \sum_{n=1}^{\infty} \frac{\bar{f}'(\lambda_n)}{(1-\lambda_n^2)} \left( \frac{J_1'(\lambda_n r)}{J_1^2(\lambda_n a)} \right) \left[ \frac{\sinh(\lambda_n z)}{\sinh(\lambda_n h)} - \frac{\lambda_n \cosh(\lambda_n z)}{\sinh(\lambda_n h)} \right] \quad (5.2)$$

#### 6. SPECIAL CASE AND NUMERICAL RESULTS

Set  $f(r) = [r^2 - ra][h^3 + 5h^2 + 4h]$ ,  $\alpha = \frac{2a}{h^3 + 5h^2 + 4h}$ ,  $a = 2$ , and  $h = 1$  in

(3.5) to obtain

$$\frac{T(r, z)}{\alpha} = \sum_{n=1}^{\infty} \left( \frac{J_0(\lambda_n r)}{J_1^2(2\lambda_n)} \right) \left[ \frac{J_3(2\lambda_n) - J_2(2\lambda_n)}{\lambda_n(1-\lambda_n^2)} \right] \times \left[ \frac{\sinh(\lambda_n z)}{\sinh(\lambda_n)} - \frac{\lambda_n \cosh(\lambda_n z)}{\sinh(\lambda_n)} \right] \quad (6.1)$$

## **7. CONCLUSION:-**

The temperature, displacement and thermal stresses have been obtained , when the boundary conditions are known, with the aid of finite Hankel transform and Laplace transform techniques. The results are obtained in terms of Bessel's function in the form of infinite series. The series solutions converge provided we take sufficient number of terms in the series. Since the thickness of the plate is very small, the series solution given here will be definitely convergent.

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## Chapter -33

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### Helminth parasites infections in freshwater fish

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#### **Abstract:-**

Fisheries play a key role in the national economy. Helminth infections in fish have a major impact on public health and sustainable fish production. Globally, helminth infections have a major impact on the fish industry due to the pathogenic effects of several species affecting productivity, as well as because of the zoonotic potential of many species. The potential impact of fish parasites on public health and fish performance, as well as the importance of the fish industry and consumption, a recent summary of the knowledge on the helminths in fish is important to understand the distribution of parasites in the country, and to design effective preventive and control measures. Aim of this chapter the latest published literature on the occurrence, prevalence and incidence of helminth infections in fish.

**Keyword:** Survey, Helminth parasites, Environmental factor, Freshwater fishes,

#### **Introduction :-**

Fish constitute a major component of diet for the people of Northeast India and they are extensively used as a protein-rich food for human consumption[01]. The major parasitic groups found in freshwater fishes are trematodes, cestodes, acanthocephalans and nematodes that complete their life cycles through intermediate hosts like piscivorous birds. The need to assess the parasitic infection arises because the fish suffering from parasitic infection or disease result into severe damage to fisheries industry. For successful prevention and elimination of such infections, it is extremely important to achieve early and correct diagnosis of the larval stages of the parasites for which fish

constitute the final host [02]. The introduction of parasites can lead to novel host–parasite relationships changing the structure of preexistent communities [03]. Helminths are an important group of pathogens, which cause infection and diseases of fish both in freshwater and marine environments, their importance being related directly to the fish that may affect the general public health. Fish parasites directly or indirectly related Human and animals health because several Helminth parasites can be transmitted to humans and domestic animals only through fish. Keeping view in mind about importance of fishes for human, records of helminths reported in alien fishes in Patagonia were compiled from scientific literature. Records from theses and scientific meetings, congress summaries and technical reports were excluded as they do not constitute formal publications. The fish species *Percichthys colhuapiensis* MacDonagh and *Percichthys laevis* (Jenyns) are considered morphotypes of *P. trucha*[04] Fish are intermediate or paratenic hosts for these parasites and serve as trophic ways to reach the birds that are the definitive hosts. Helminths display reduced host specificity in fish, which increases their chances of reaching the definitive host [05]. The occurrence of this helminth in alien fish is a natural consequence of their distribution in these Patagonian aquatic environments.[06] The time elapsed since the colonization of the salmonids, together with their success in establishing themselves as invasive fish species, have favored their chances of being colonized by native parasites. The only introduced helminth parasites that currently infect native fishes are the cestodes *D. latus* and *D. dendriticus*[07] In Patagonia, *Contracaecum* sp. was also the most generalistic parasite, which was found in nine alien fish species. The number of known hosts is not the ideal measure of a parasite's host-specificity. Parasites may show higher growth rates and fecundity on some hosts than on others, i.e. some hosts may be 'better' hosts than others [08] The infection of parasites interferes with nutrition, metabolism and secretory function of the alimentary canal, damages nervous system[09]. which may also lead to gastrointestinal abrasions and facilitate the invasion by opportunistic microorganisms.Unfavorable environmental conditions contribute to stress which also weakens the immunity and opens the pathway to pathogens[10].

The study of diversity and distribution of helminthes started in the middle of the 19th century in India and numerous works has been done in different parts of India. The distribution of helminths is not only affected by seasons but also by host age, size, diet, abundance of fishes and an independent number of parasites within the fish. Change in climatic conditions is predicted to affect the prevalence of parasites in freshwater and marine ecosystems. A study of Chubb [11]. showed the seasonal occurrence of helminths of freshwater fishes from different climatic zones. Two main categories of factors may be held responsible for the seasonal variations in host infectivity, those linked to the host and other linked to the parasites. Ibiwoye et al., [12] observed that susceptibility to infections in fishes are generally due to weakened body after hibernation. According to Bhuiyan et al.[13] decrease in water volume during dry seasons results in imbalanced nutritional conditions also make fishes vulnerable to the infections. The authors also concluded that decreased water temperature also made the hosts susceptible to infections by weakening immune systems[14]. So many other parasite associated factors are also held responsible for the development of parasites such as high temperature and explained that feeding habits of the host, availability of infective host and parasite maturation are also responsible for influencing the parasitic infections. Recently, Sheema et al., [15] have suggested that abundance of helminthes increase with the rising temperature in summer and slow down during winter. The development of intermediate hosts of helminthes during summer season also leads to better availability of infective stages resulting in higher helminths prevalence in summer. [16]

Fishes are hosts to a number of parasites. Helminths are one of the major groups of fish parasites and cause a severe loss in the fish production [17].Fishes are infected with three major groups of helminths: the Platyhelminthes (flat worms), Nematoda (round worms), and Acanthocephala (spiny headed worms). About 20,000 to 30,000 helminth species have been reported worldwide, which cause heavy losses to the fish industry [18].Dhar [19] reported 31 species of helminth parasites from Kashmir valley which cause severe damage to the fish production and population.

Globally, helminth infections have a major impact on the fish industry due to the pathogenic effects of several species affecting productivity, as well as because of the zoonotic potential of many species. Humans acquire fish-borne helminth zoonoses via the consumption of raw or undercooked fish containing infective parasite larvae [20]. Because of the potential impact of fish parasites on public health and fish performance, as well as the importance of the fish industry and consumption in India

### **Conclusion :-**

The study explores the diversity and seasonal variations of helminthic parasites in Fish. These types of studies will lead to the better understandings of host-parasite interactions what will be beneficial for the improvement of infectious diseases management and also contribute to the increase in fish production.

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## Chapter -34

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### **Antifungal activity of Coumarin Against *Aspergillus niger***

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#### **Introduction: -**

*Aspergillus niger* is a filamentous fungus commonly found as a sporophyte growing on dead leaves, stored grain, compost piles and other decaying vegetation [1]. It is a common contaminant of food and a plant pathogen, causing a black mould on certain types of fruit and vegetables such as peanuts, onions, grapes, mangoes and tomatoes. Besides, it causes skin and pulmonary infections. This pathogen is worldwide identified to be difficult to control. In order to avoid fungal contamination, extensive search has been made. *Aspergillus niger* is one of the most important microorganisms used in biotechnology, *A. niger* is used for bio transformations and waste treatment. In the last two decades, *A. niger* has been developed as an important transformation host to over-express food enzymes. Being pre-dated by older names, the name *A. niger* has been conserved for economical and information retrieval reasons and there is a taxonomical consensus based on molecular data that the only other common species closely related to *A. niger* in the *Aspergillus* series Nigri is *A. tubingensis*. *A. niger*, like other filamentous fungi, should be treated carefully to avoid the formation of spore dust. However, compared with other filamentous fungi, it does not stand out as a particular problem concerning allergy or mycopathology. A few medical cases, e.g. lung infections, have been reported, but always in severely immune compromised patients. In tropical areas, ear infections (otomycosis) do occur due to *A. niger* invasion of the outer ear canal but this may be caused by mechanical damage of the skin barrier. [2] *A. niger* is generally regarded as a non-pathogenic fungus widely distributed in nature.

Humans are exposed to its spores every day without disease becoming apparent. Only in few cases has *A. niger* been able to colonise the human body as an opportunistic invader and in almost all these cases the patients have a history of severe illness or immunosuppressive treatment.

Coumarins are classified as a member of the benzopyrone family, all of which consist of a benzene ring joined to a pyrone ring. The benzopyrones can be subdivided into the benzo- $\alpha$ -pyrones to which the coumarins belong and the benzo- $\gamma$ -pyrones, of which the flavonoids are principal members. Umbelliferone, esculetin and scopoletin are the most widespread coumarins in nature. During the synthesis of these compounds ortho-hydroxylation should respectively take place on *p*-coumaric, caffeic and ferulic acid. The coumarins are of great interest due to their pharmacological properties. In particular, their physiological, bacteriostatic and anti-tumor activity makes these compounds attractive backbone derivatisation and screening as novel therapeutic agents [3]. In view of the established low toxicity, relative cheapness, and presence in the diet, Coumarins and their derivatives have been found to exhibit a wide range of biological and pharmacological activities. Coumarin and its derivatives considered as one of the most active groups of compounds having an extensive range of biological actions, including, antimicrobial, anti-inflammatory, antioxidant, anticoagulant, and anticancer activities[4]. In addition, several coumarin derivatives were reported to display antifungal capacity against *A. niger*. [5]

Pyridines and Coumarins are the promising class of naturally occurring bioactive heterocycles with important physical and chemical properties. Compounds containing the coumarin framework possess a different range of pharmacological, biological, and physiological activities, which makes them important for application in medicine, the food industry and agriculture. Among all coumarins' properties, it was found that coumarins may prevent fungal growth, depending on substituents linked to the coumarin core. Therefore, many coumarin derivatives have been investigated as potentially powerful agents in preventing and controlling fungal pathogens. [6]

Novel coumarin based 1,2,3-triazoles derivatives were synthesized by trivedi [7] and coworkers from 4-hydroxy coumarin and various substituted azides using click chemistry approach. Triazole compounds [N-(substituted phenyl)-2-(4-(((2-oxo-2H-chromen-4-yl)oxy)methyl)-1H-1,2,3-triazol-1-yl)acetamide] were synthesized. These triazole compounds were shown good antifungal activity against fungi species *A. niger*. The inhibition of chitinase having an important role in developing antifungal agents. Therefore, Batran and co workers studied chitinase inhibitory properties of 4-hydroxycoumarin derivatives containing Schiff base moiety in the coumarin. Additionally, they studied the antifungal activity of these compounds against *Fusarium solani*, *Fusarium oxysporium*, and *A. niger*, as well as pathogenic yeast *C. albicans*, *Candida tropicalis*, and *Candida krusei*. In contrast, the active antifungal compounds were used for chitinase inhibition activity determination. Prepared Compound showed the best antifungal activity against most tested species and exhibited the highest chitinase inhibition effects [8]. Moreover, Mote and co-workers investigated antifungal activities of different Schiff bases of 3-acetyl-4-hydroxy-coumarin and amino pyridines using *A. niger*, *Penicillium chrysogenum*, *Fusarium moneliforme*, and *A. flavus* as tested fungal species. The study showed that all tested compounds possess better antifungal activities towards *Aspergillus* species than other cultures.[9] Waheed and coworkers Synthesized the eight novel naphthalene-derived coumarin composites from 6-amino-7-phenolchloro-2-naphthol. The antifungal evaluation of the synthesized naphthalene-derived coumarin composites have very strong fungicidal influences. along with their good oral absorption profiles, low penetration across the blood brain barrier, and low toxicity towards normal flora, these new composites could provide a valuable platform for the scanning of new fungicidal drugs.[10]

A new series of coumarin-6-sulfonamide derivatives have been synthesized by Abo-Salem et.al. via simple and convenient coupling reaction of coumarin-6-sulfonyl chloride with different sulfa drugs and various amino-heterocycles. The synthesized compounds show potent antifungal activity against *C. albicans* and *A. niger* [11]

Mixed ligand metal complexes of Co(II), Fe(III), Cu(II), Zn(II), Cd(II) and Hg(II) metal ions with azo coumarin and thiosemicarbazone derivative have been synthesized by Borhade et.al. Synthesized ligands and their metal complexes displayed modest to excellent antimicrobial potential antifungal activity against *A. niger* [12]. the coumarin derivative 4-acetatecoumarin (Cou-UMB16) has antifungal activity which inhibits virulence factors in the *Aspergillus* species[13]. Newly synthesized coumarins 4-((5-mercapto-4-phenyl-4*H*-1,2,4-triazol-3-yl)-methoxy)-2*H*-chromen-2-one and 4-((5-(phenylamino)-1,3,4-thiadiazol-2-yl)-methoxy)-2*H*-chromen-2-one were showed significant antifungal activities as compared with standard fluconazole against fungal spices *Aspergillus niger* and *Candida albicans*. [14]

A series of metal complexes of the type  $ML \cdot 2H_2O$  [M = Co(II), Ni(II), and Cu(II)] have been synthesized by Patil et.al.[15] with newly synthesized Schiff bases derived from 1,8-diaminonaphthalene and 8-formyl-7-hydroxy-4-methylcoumarin/8-acetyl-7-hydroxy-4-methylcoumarin.the compounds show the metal complexes to be more effective antifungal activity as compared with the uncomplexed Coumarins against *Aspergillus niger*, *Aspergillus flavus*, and *Cladosporium*.

### **Conclusion: -**

Many coumarin derivatives are proven to possess significant antifungal activity, both natural and synthetic. It is well established that plants biosynthesize Coumarins as a defense against different infections. It was Coumarins antifungal potency varies depending on coumarin core substituent's and those with phenolic, hydroxyl, and the carboxylic group have been proven as strong antifungal agents. Halogens as substituent's can also contribute to significant antifungal properties of Coumarins. In addition, coumarin hybrids with different heterocyclic compounds, such as pyridine moiety, contribute to different properties than the initial material. Considering these pharmacological properties of Coumarin, This study demonstrates that several Coumarins exhibit strong antifungal activities.

These results call for further studies, where these Coumarins can be examined as potential lead compounds for developing novel antifungal agents against *A.Niger*.

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## Chapter -35

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### Zeolites: Its Properties and Catalytic Applications

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#### **Abstract:**

Zeolites are crystalline aluminosilicates with a unique porous structure, which makes them attractive for a wide range of catalytic applications. Due to their high surface area and well-defined pore structure, zeolites can selectively adsorb and catalyze various chemical reactions. This chapter provides an overview of the properties and catalytic applications of zeolites, including their synthesis, and modification techniques. The applications of zeolites in various fields, including petrochemicals, fine chemicals, and environmental protection, are also discussed. The unique properties and versatility of zeolites make them an essential component in the development of efficient and sustainable catalytic processes.

**Keywords:** Zeolites, aluminosilicates, MCM-41, ZSM-5.

#### **1. Introduction:**

Zeolite is a microporous, three-dimensional crystalline solids aluminosilicate materials. Zeolites have small opening of fixed size in them, which allow small molecules to pass through them easily. But also large molecules cannot pass through them; because they have sometimes called molecular sieves. Structurally ordered aluminosilicate based on a network of tetrahedra (TO<sub>4</sub>) where the T atom can either be a silicon or aluminum atom. Zeolite are commonly used as adsorbents and catalysts.<sup>1</sup> Zeolite are mainly consist of silicon, aluminium, oxygen and there general formula is  $M^{n+1/n}(AlO_2)-(SiO_2)_x.yH_2O$  where  $M^{n+1/n}$  is either a metal ion or H<sup>+</sup>. These positive ions can be exchanged for others in a containing electrolyte solution. H<sup>+</sup> exchanged zeolites are particularly useful for heterogeneous solid acid catalysts.<sup>2</sup> Zeolite

consists of tetrahedral arrangement of silicon ion ( $\text{Si}^{4+}$ ) and aluminum ion ( $\text{Al}^{3+}$ ) that are surrounded by four oxygen anions ( $\text{O}^{2-}$ ).<sup>3-5</sup> Each oxygen ion within Si-O and Al-O bonds connect with two cations and which are share in two tetrahedron structure.<sup>6-7</sup> The general formula of zeolite denoted by M represents an alkali or alkaline Earth cation, n represents the valence of the cation, z is the number of water molecule per unit cell, and x and y are the total numbers of tetrahedral per a unit cell.

The term “zeolite” was first discover by in year 1756 by Swedish Mineralogist, Axel Fredrik Cronstedt. They observed that rapidly heating a material to have stilbite, produced large amount of steam from water that had been adsorbed by the materials. Zeolite is derived from Greek word *zeo* meaning “to boil” and *lithos* meaning “stone”.<sup>4,8,9</sup>

Zeolites are classified into two groups such as natural and synthetic zeolites.<sup>10-11</sup> Natural zeolites are mostly formed from volcanic and sedimentary rocks such as chabazite, clinoptilolite, and mordenite. Synthetic zeolites are prepared through heating of china clay, soda ash and also other sources.<sup>12</sup>

## **2. Properties of Zeolites**

Zeolites are white solids nature with ordinary handling properties, its contains aluminosilicate minerals, e.g. feldspar. They have general formula  $\text{MAlO}_2(\text{SiO}_2)_x(\text{H}_2\text{O})_y$  Where  $\text{M}^+$  is usally  $\text{H}^+$  and  $\text{Na}^+$ . the Si/Al ratio is variable, zeolites with Si/Al ratios higher than about 3 are classified as high-silica zeolites, they also shows the more hydrophobic. The  $\text{H}^+$  and  $\text{Na}^+$  can be replaced by diverse cations, because zeolites have ion exchange properties. Zeolites have microporous structure with diameter 0.3-0.8 nm. Like most aluminosilicate materieals the framework is formed by linking aluminum and silicon atoms by oxides. This linking also shows the three-dimensional network Si-O-Al, Al-O-Al and Si-O-Si linkages. The aluminum centers are negatively charged, which requires for cation. These cations are hydrated during the formation of materials. The hydrated cations interrupt the otherwise dense network of Si-O-Si, Si-O-Al and Ai-O-Al linkage, leading to regular water-filled cavities. Because of the porosity of zeolite, water can be exit the materials through channels.



Zeolites can be used as catalysts. They possess high physical and chemical stability due to the large covalent bonding contribution. They show the hydrophobic character due to adsorption of bulky, hydrophobic molecules such as hydrocarbons. Natural zeolites are also used as solid acid catalysts. The acidity is strong enough to protonate hydrocarbons and high silica zeolites are used in acid catalysis processes such as fluid catalytic cracking in petrochemical industry process to break large hydrocarbon molecules into gasoline, diesel, kerosene, waxes and all kinds of other byproducts of petroleum.<sup>13</sup>

### **3. Structure of Zeolites:**

Zeolites are crystalline substance with a structure characterized by a framework of linked tetrahedra, each consisting of four O atoms surrounding a cation. This framework contains open cavities in the form of channels and cages. These are usually occupied by water molecules and extra-framework cations that are commonly exchangeable. The channels contain large enough to allow the passage of guest species. The framework may be interrupted by (OH, F) groups; these occupy a tetrahedron apex that is not shared with adjacent tetrahedra.<sup>14</sup>

#### **3.1 Primary and Secondary Building Units:**

Zeolites are normally categorized in primary building units (PBUs) and secondary building units (SBUs). The primary building units are the  $(\text{SiO}_4)^{4-}$  and  $(\text{AlO}_4)^{5-}$  tetrahedra. These combine by sharing oxygens with adjacent tetrahedra to form a special arrangement of simple geometric forms. Secondary building units contain single rings, double rings, polyhedral or even more complex units which are linked together in a variety of ways to produce a unique system of channels and cages. Zeolite unit cell always contains an integral number of secondary building units. It contains 23 different types of secondary building units known to exist. Figure 15.

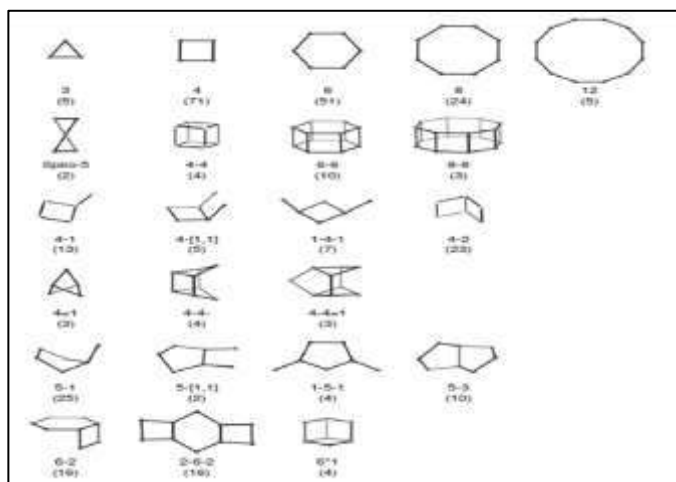


Figure 1 secondary building units and their Symbol

#### 4. Porosity of Zeolites:

This is due to a very regular pore structure of molecular dimensions. Zeolites shows the maximum size of the molecular or ionic species that can enter the pore of zeolites is controlled by the dimensions of the channels. These are conventionally defined by the ring size of the aperture, where for e.g. the term “eight-ring” refers to a closed-loop that is built from eight tetrahedrally coordinated silicon or aluminium atoms and eight oxygen atoms. These rings are not always perfectly symmetrical due to a variety of causes, including strain induced by the bonding between units that are needed to produce the overall structure or coordination of some of the oxygen atoms of the rings to cations within the structure. Therefore, the pores in many zeolites are not cylindrical in nature.

#### 5. Acidity of Zeolites :

Zeolites shows the most important properties with respect to their use as catalysts in their surface acidity. Zeolites contains nature of acid sites (i.e. Brønsted vs. Lewis acidity) it contains density or concentration of these sites and its also contains the precise location of the acid sites.

## **6. Natural Zeolites Occurrence:**

Some of the more common mineral zeolites are analcime, chabazite, clinoptilolite, heulandite, natrolite, phillipsite and stilbite. An example of the mineral formula of a zeolite is a  $\text{Na}_2\text{Al}_2\text{Si}_3\text{O}_{10}\cdot 2\text{H}_2\text{O}$ . natural zeolites occur from volcanic rocks and ash layers reacts with alkaline ground water. Naturally occurring zeolites are rarely pure and are contaminated to varying degrees by other minerals, metals, quartz or other zeolites. Zeolites transform to other minerals under weathering, hydrothermal alteration or metamorphic conditions. Some example are sequence of silica-rich volcanic rocks commonly progresses from clay-quartz-mordenite-heulandite-epistilbite-stilbite-thomsonite-mesolite-scolecite-chabazite-calcite. The sequence of silica-poor volcanic rocks commonly progresses from cowlesite-levyne-offretite-analcime-thomsonite-mesolite-scolecite-chabazite-calcite.



**Figure 2 Image of naturally occurring Zeolite**

## **7. Catalysis of Zeolites:**

Synthetic zeolites like mesoporous materials e.g. MCM-41 are also used as catalysts in the petrochemical industry, such as fluid catalytic cracking and hydrocracking. Acidic zeolites are important for solid acid heterogeneous catalyst. They are important for isomerization, alkylation and cracking. Catalytic cracking uses a reactor and regenerator. Zeolites containing cobalt nanoparticles have application in the recycling industry as a catalyst to break down polyethylene and polypropylene, two also used plastics into propane.

## **Conclusion**

Zeolites are classified into two types natural and synthetic zeolites. Natural zeolites which are found in earth and volcanogenic sedimentary rocks. Synthetic zeolites can be made in laboratory. In natural zeolites at least 60 species are known to exist, occurring naturally in soils, sediments and rock. Synthetic zeolites 200 species have been reported. The most common synthetic zeolites are zeolites A, X, Y and ZSM-5. Advantages of zeolites in providing new low cost, eco-friendly green catalyst and applicable in agriculture and medicinal uses. The future of zeolites in catalysis looks promising, as new and improved synthesis and modification techniques continue to emerge, allowing for greater control over their properties and catalytic activity. Overall, zeolites represent a valuable class of materials for catalytic applications, with the potential to play a significant role in addressing current societal and environmental challenges.

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## Chapter -36

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### Importance and Application of Microwave-Assisted Method in organic Synthesis

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#### Abstract:

Nowadays, Microwave assisted method is widely used as a convenient source of heating in organic synthesis. The heating is instant and very specific. Microwave method organic, inorganic synthesis could be considered in which all of the previously heated reactions could be performed using this technique. The benefits of microwave-assisted organic synthesis are progressively making the technique more extensively established. This technique is simple, clean, fast, efficient, and economic features for the synthesis of a large number of organic molecules. Microwave-assisted method is environmental-friendly and therefore, in agreement with the principles of green chemistry. This form of energy has been employed extensively and successfully in organic synthesis also in the case of metal-catalyzed synthetic procedures. As microwave irradiation has been showing its utility as both a time-saving procedure and an alternative way to carry on tricky transformations, its use can help inorganic chemists, too. This is often complemented by a more limited risk of decomposition of reagents or products by an increase in yield, purity, and sometimes selectivity. In the present chapter, an attempt was made to focus on the importance and application of the microwave assisted method.

**Keywords:** Microwave technology, Organic synthesis, Chemical industry, Application.

## **1. Introduction:**

Microwave chemistry is the science of applying microwave radiation to chemical reactions [1-2]. Microwave synthesis represents a major invention in the synthetic chemistry methodology; a dramatic change in the way chemical synthesis is performed [3-4]. Microwave-assisted organic synthesis has transformed organic synthesis. Microwave heating has thus been found to be a very convenient thermal source not only in the kitchen but also in a chemical laboratory [5-10]. Chemists have explored the possibility of the application of a conventional microwave oven to carry out chemical reactions [11-18]. In the last twenty years the application of microwave irradiation to activate and accelerate organic reactions has taken a new dimension and has experienced exponential growth. Microwave chemistry is becoming increasingly popular both in industry and in academia. Microwaves can accelerate the rate of reaction, provide better yields and higher purity, uniform and selective heating with lower energy usage, achieve greater reproducibility of reactions and help in developing convenient and cleaner synthetic routes.

In recent year's attention microwave heating for carrying out reactions on solids has also attracted [19-24]. For such 'dry media' reactions, solid supports such as alumina, silica, montmorillonite clay and zeolites have been investigated. Application of microwave irradiation in chemical synthesis involves its use in the acceleration of chemical synthesis. Microwave-enhanced synthesis results in faster reactions, higher yields, and increased product purity. In addition, due to the availability of high-capacity microwave apparatus, the yields of the experiments have now easily scaled up from milligrams to kilograms, without the need to alter reaction parameters. Microwave-assisted synthesis can be suitably applied to the drug discovery process [25]. Microwave-assisted organic synthesis has been the foremost and one of the most researched applications of microwaves in chemical reactions [26]. Literature survey reveals that scientists have successfully conducted a large range of organic reactions. The following examples are impressive and provide a good insight into the field of microwave assisted organic synthesis [27-28]. The main advantages of microwave assisted organic synthesis are:

**1) Faster Reaction:**

It has been found that microwave-enhanced chemical reaction rates can be faster than those of conventional heating methods based on experimental data [29]. The microwave can use higher temperatures than conventional heating system, and subsequently the reactions are completed in few minutes instead of hours, for instance, synthesis of fluorescein, which usually takes about 10 hours by conventional heating methods, can be conducted in only 35 minutes by means of microwave heating.

**2) Better yield and higher purity:**

Less formation of side products are observed using microwave irradiation, and the product is recovered in higher yield. Consequently, the purification step is faster and easier [30]. For example, microwave synthesis of aspirin results in an increase in the yield of the reaction, from 89% to 98%.

**3) Energy saving:**

Heating by means of microwave radiation is a highly efficient process and results in significant energy saving [31]. This is primarily because microwaves heat up just the sample and not the apparatus, and therefore energy consumption is less.

**4) Uniform and selective heating:**

In conventional heating, the walls of the oil bath get heated first, and then the solvent. As a result of this distributed heating in an oil bath, there is always a temperature difference between the walls and the solvent. In the case of microwave heating, only the solvent and the solute particles are excited, which results in uniform heating of the solvent [32]. Selective heating is based on the principle that different materials respond differently to microwaves. Some materials are transparent whereas others absorb microwaves.

**5) Green synthesis:**

Reactions showed using microwaves are cleaner and more eco-friendly than conventional heating methods [33]. Microwaves heat the compounds directly; therefore, usage of solvents in the chemical reaction can be reduced or eliminated. Synthesis without solvent, in which reagents are absorbed on mineral support, has a great potential as it offers an eco-friendly green protocol in synthesis. The use of



microwaves has also reduced the amount of purification required for the end products of chemical reactions involving toxic-reagents.

### **6) Reproducibility:**

Reactions with microwave heating are more reproducible compared to the conventional heating because of uniform heating and better control of process parameters [34]. The temperature of chemical reactions can also be easily monitored. The routine synthetic transformations are now being carried out by microwave heating. Microwave-assisted synthesis provides clean synthesis with the advantage of enhanced reaction rates, higher yields, greater selectivity, and economic for the synthesis of a large number of organic molecules, have provided the momentum for many chemists to switch from conventional heating method to microwave assisted chemistry. Microwave-assisted synthesis is rapidly becoming the method of choice in modern chemical synthesis and drug discovery. Microwave assisted synthesis has revolutionized chemical synthesis. Small molecules can be built in a fraction of the time required by conventional methods. In conventional heating methods oil bath or hot plate are used as a source of heat to a chemical reaction. Microwave irradiation is widely used as a source of heating in chemical synthesis. The basic mechanisms observed in microwave-assisted synthesis are dipolar polarization and conduction [35]. Conventional methods of organic synthesis usually need longer heating time, tedious apparatus setup, which result in higher cost of process and the excessive use of solvents/reagents. During these processes there are many problems of health and safety for workers in addition to the environmental problems.

## **2. Applications of Microwave technology**

### **2.1 Microwave technique in organic synthesis applications:**

The microwave radiation is used in organic synthesis reaction, of electromagnetic pulse to the acrylic acid, acrylic acid, methacrylic acid polymerization [36]. (1) usually microwave-assisted organic reaction mechanism is divided into two kinds, one for the thermal effect (1), the microwave-assisted organic synthesis reaction mechanism, the microwave-assisted organic synthesis reaction mechanism,: Microwave heating speed and uniform, showing no temperature gradient, no

hysteresis effect. The microwave may cause the instantaneous hyperthermization of the solvent, and the liquid may reach a higher temperature before boiling. Microwave can effectively accelerate the chemical reaction, which can be polar organic heating; the other for the non-heating effect: the microwave will accelerate the molecular motion of the reactants, the reactant temperature will rise rapidly, during which ions and polar molecules Lorentz force to promote the special movement between particles, resulting in non-heating effect. (2) microwave organic synthesis reaction technology[37]: This technology under the different organic reactions, microwave response to specific equipment and reaction technology to achieve, usually microwave organic synthesis reaction technology for the microwave closed synthesis technology, in 1986 some scholars use microwave technology Organic synthesis of the way is closed synthesis technology. Microwave closure synthesis reaction technology emphasizes the reactant sealing reaction device placed in the microwave source, when the reaction is completed, the reactor cooling product purification, the technology is a high temperature, high pressure reaction of a technology. Microwave heating solution in the device can be refluxed; PTFE tube with the reactor connected to each other, and then the pipeline to provide an inert gas for the reaction, then can effectively protect the reaction system.

## **2.2 Microwave technique in the synthesis of materials:**

There are many microwave methods for synthesizing chemical materials, such as microwave homogeneous precipitation method and microwave precipitation method, microwave hydrothermal method and microwave sintering method [38]. Often olivine-type structure of lithium iron phosphate lithium-ion battery cathode material, which has a high theoretical capacity, the voltage platform is also reasonable and realistic, good cycle performance, to achieve low prices, material safety and environmental protection. Microwave synthesis of lithium-ion battery cathode material, can effectively shorten the synthesis time, low energy consumption, cadmium sulfide for semiconductor materials, is widely used in solar energy conversion and non-linear optics, photoelectron chemical batteries and other materials synthesis.

### **2.3 Microwave technique in the sample analysis of the application:**

The microwave technique in the sample analysis can be divided into three aspects: microwave drying, microwave digestion and microwave extraction: (1) Microwave drying is the internal heating mode, that is, microwave heating in microwave drying, Water molecules will change with the alternating high-frequency electric field to achieve interactive changes, then there will be violent collision and friction, to promote the local microwave energy into molecular kinetic energy, the form of heat to the performance of water temperature also shows that the material will Leave, then you can achieve the purpose of drying. After the microwave is dried and absorbed, the internal energy of the dielectric material is converted to heat. Microwave drying is the electromagnetic wave as the heating source, and the need to dry the material is the heating element [39]. Microwave drying speed, the short time required, high thermal stability, uniform heating, high product quality, and sterilization effect of a good insecticide. In order to make full use of microwave drying and the characteristics of the existing drying technology, microwave hot air drying machine microwave vacuum drying has been developed and widely used. (2) Microwave digestion is a mixture of acid and sample polarity of the molecules in the microwave under the electromagnetic field will change the polarity, then it will have a microwave digestion.

The contact with the heated sample of water and acid will produce more heat than the sample surface, then there is a strong thermal convection to achieve mixing and removal of sample particles have been dissolved surface of the inactive surface layer, the sample And the acid contact surface will be quickly updated to achieve the purpose of sample dissolution acceleration. Microwave digestion has been used in many fields, such as biotechnology, cosmetics, environmental protection, and materials synthesis. Some scholars to microwave digestion milk samples, while optimizing the microwave digestion conditions, the test available: microwave digestion of milk powder in the relative standard deviation of copper, spike recovery, this approach is simple and convenient, applicability, usually in this way the analysis of milk And the amount of copper contained in dairy products; (3) Microwave extraction is the use of microwave heating can

be used to extract the microwave energy, which can quickly improve the extraction efficiency. There are different substances in the microwave field, the dielectric constant is different, the degree of absorption of microwave energy is also different, the resulting heat there is a certain difference, the heat can be transmitted is different, these differences in the extraction system The local composition and the matrix material appear area uneven heating, and this non-uniformity will cause the extract in the matrix separation. Microwave extraction has been used in environmental samples, soil, plants and animals in the extraction of active ingredients, such as microwave extraction can be within 20 seconds in the days of tincture extraction of flavonoids, the total extraction up to 1.883%; or in marine sediments in the half 12 hours within the sample processing, while the traditional technology is only 16 hours to deal with one, showing that the microwave extraction technology is fast, and its extraction efficiency, the resulting product quality, low cost. Microwave technology has been used for derivatization, regeneration, analysis, sample online processing.

#### **2.4 Microwave technique in wastewater treatment applications:**

Microwave heating does not require heat transfer, or internal and external heat, heat transfer will not be heat loss, this advantage can be used to deal with microwave technology, sludge or organic pollutants [40]. Application of microwave technology in wastewater treatment is: (1) Microwave-induced oxidation, the oxidation reaction is a strong absorption of microwave sensitizers to microwave energy is not directly absorbed in the microwave absorption of organic compounds, and thus the catalytic reaction, The technical realization is in the microwave induced oxidation process preparation of the appropriate catalyst. Some scholars use ferric oxide as a catalyst to treat wastewater, the results show that about 50% COD and 80% of the color water can be removed; some scholars to self-made Fe-Zr as a catalyst, and H<sub>2</sub>O<sub>2</sub> as an oxidant treatment of n-butyric acid (2) Microwave-induced UV treatment of wastewater, some scholars to sodium hypochlorite as oxidant, and then to the wastewater treatment, the use of micro-wave technology can be effective in the treatment of

waste water, the TOC removal rate of 95% The results show that the effect of wastewater treatment is better if the dosage of sodium hypochlorite is increased, but the dosage of sodium hypochlorite is strictly controlled; (3) The microwave irradiation regeneration, in this way is to achieve the adsorption of activated carbon or adsorbent pollutants, and then saturated adsorbent in the microwave field deep radiation, the pollutants fully degraded, microwave radiation regeneration wastewater treatment costs less, and the adsorbent is renewable ; (4) Direct microwave radiation is directly to the microwave radiation containing pollutants adsorbate, the water pollutants fully removed, this way with chemical oxidation, photo catalysis and other applications in order to effectively improve the pollutant removal effect.

### **3. The development prospects of microwave technology:**

Microwave technology is widely used in chemical and chemical industry, the microwave heating in chemical synthesis is very superior, and its heating speed, can improve the reaction rate; heat source and reactants, solvents cannot direct contact, the reaction parameters can be effectively controlled, Safety is also controlled; this technique can be selectively heated to selectively heat the microwaves if the compounds present in the reaction mixture have different absorption microwave capabilities to ensure that the yield is increased and the incidence of side reactions is also reduced [41]. The level of automation with the rapid development of science and technology to enhance productivity is also rising. Microwave technology is fast and efficient, low energy consumption, and little pollution to the environment, for the green synthesis method, can stimulate the high energy, background radiation than the traditional low, can be widely used in various fields. However, there are some limitations, such as the high cost of the microwave reactor, the shorter depth of the microwave radiation through the liquid medium, the large size of the reactor, and the difficulty of the experiment.

### **4. Conclusions**

Microwave technology also will be rapid development, the technique heating speed, high efficiency, and the cost is not much, with

the rapid development of modern society and economy, the impact on the natural environment is small, the technology has been used for navigation, Communications, military, weapons and other fields. In this chapter, the development of microwave technology, the application of microwave technology, the development prospects of microwave technology, as a microwave technology in the chemical and chemical industry to provide a reference basis. Microwave irradiation has been proving its value as a useful synthetic tool within the coordination and organometallic chemistry community but probably with results that are less captivating with respect to other fields. It is that Microwave pushes reactions to completion more rapidly than conventional heating. In all examples, there is a change in the timescale, roughly speaking, from hours to minutes. The efficient transfer of energy into the reaction medium contributes to the rapid heating, resulting in a uniformly reached temperature in seconds.

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## Chapter -37

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### **Synthesis, structural characterization and biological evolution of the Schiff base and their transition metal complexes derived from (20E)-N'-((4-oxo-4H-chromen-3-yl)methylene)thiophene-2-carbohydrazide**

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#### **ABSTRACT**

A novel Schiff base(20E)- N'- (( 4 - oxo - 4H - chromen - 3 -yl) methylene)thiophene-2-carbohydrazide obtained by the condensation of 4 - oxo - 4H - chromene - 3 - carbaldehyde and Thiophene - 2 - carbohydrazide and and the synthesized schiff base was characterized by various analytical technique such as I.R., <sup>1</sup>H-NMR, <sup>13</sup>C-NMR and. Further it used for the complexation with different transition metal ions such as Fe(III), Co(II), Ni (II), Cu(II), And Zn(II) by using molar ratio of metal to ligand as 1: 2. The prepared metal complexes were identified with the help of FT-IR, elemental analysis, and TGA methods. The spectral data reveal that the ligand acts as bidentate, tridentate in ML complexes. The effect of these metal complexes on bacterial and fungal species was studied and compared with those of free ligand. The results of antimicrobial studies show enhanced activity in comparison to the free ligand.

**Keywords:** Spectroscopic analysis; antimicrobial activity, elemental analysis

#### **Introduction**

Hydrazones and their metal complexes have increasing significance like anti-inflammatory, anti-microbial, anti-tubercular, anti-fungal, anti- HIV and anti-cancer activities [1-5].

Numerous researchers have synthesized these compounds as target structures and evaluated their biological properties. In several cases it was reported that metal complexes have superior biological properties than their corresponding ligands [6-8]. The biological property of hydrazones may be due to the availability of multidentate coordination sites and their capability to form stable complexes with critical metal ions which organisms require in their metabolic activity [9-13]. Hydrazones having an azomethine -NH-N=CH- proton comprise an important class of compounds for development of new therapeutic agents. Pinheiro et al. synthesized a series of hydrazone derivatives with significant antitubercular activity [14]. B. C. Raju et al. synthesized potential anti- mycobacterial and anticancer agents [15].

The first row transition metal play an imperative role in the synthesis of numerous coordination complexes due their variable oxidation states which facilitate their structural, stereo chemical, electrochemical and spectroscopic properties. Among the diverse transition metals, the current study is focused on nickel, copper and zinc metal ions based on their extensive biological applications.

## **2. Experimental**

### **2.1 | Materials**

All the purchased chemicals were analytical grade and used without further purification. Solvents were purified and dried according to literature method [16]. All chemicals were obtained from Sigma–Aldrich chemical used without purification. They included 4-oxo-4H-chromene – 3 - carbaldehyde and Thiophene – 2 - carbohydrazide, remaining all chemical solvents were purchased from spectrochem ltd.

### **2.2. Physical measurements**

Elemental analysis (C, H, N,) was performed using Perkin Elmer CHN analyzer. IR spectra of the ligands and their metal complexes were recorded on Bruker spectrometer within the range of 4000–400  $\text{cm}^{-1}$ . Thermal studies of the complexes were carried out on a Perkin Elmer diamond TGA instrument.  $^1\text{H-NMR}$  and  $^{13}\text{C-NMR}$  spectra of the ligands were recorded on Bruker spectrometer using DMSO-d<sub>6</sub> as a solvent and

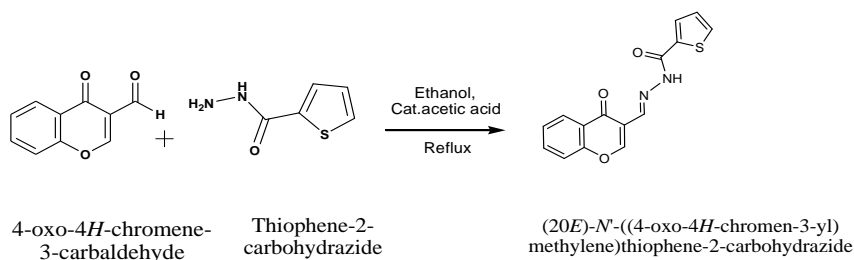
TMS as internal standard. Mass spectra were recorded on water, Qt of micromass (ESI-MS).

### 2.3 | Synthesis of the Schiff base ligand

The Schiff base ligand was prepared by condensation of 4-oxo-4H-chromene-3-carbaldehyde (1.00 mmole) and Thiophene-2-carbohydrazide (1.00 mmole) in absolute ethanol (20 ml), 2-6 drops of acetic acid. The mixture was refluxed for 6-8 hr with continuous stirring. The progress of reaction was monitored by TLC and after completion of the reaction; the mixture was poured on crushed ice and filtered off. The obtained product was recrystallized in absolute Methanol.

### 2.4 Spectral data of ligand:

**Color:** Spongy white , **Yield:** 82 % , **M.P.:** 280 °C, Selected IR bands (KBr,  $\text{cm}^{-1}$ ) : 3188.46  $\nu$  (NH), 1664  $\nu$ (C=O chromone), 1635  $\nu$  (C=O hydrazonic), 1568  $\nu$ (C=N );  $^1\text{H}$ - NMR (DMSO- $d_6$ ,  $\delta$  ppm ) 12.30 (1H,s, iminolic -OH); 7.96 (s, 1H, HC=N), 6.80-8.32 (m, 4H, Ar-H); 2.86 (s, 3H, -CH<sub>3</sub>);  $^{13}\text{C}$ - NMR (DMSO- $d_6$ ,  $\delta$  ppm) 160.60 (C=O chromone), 152.40 (C=O hydrazone ), 151.90 (-HC=N), 14.39(-CH<sub>3</sub>);

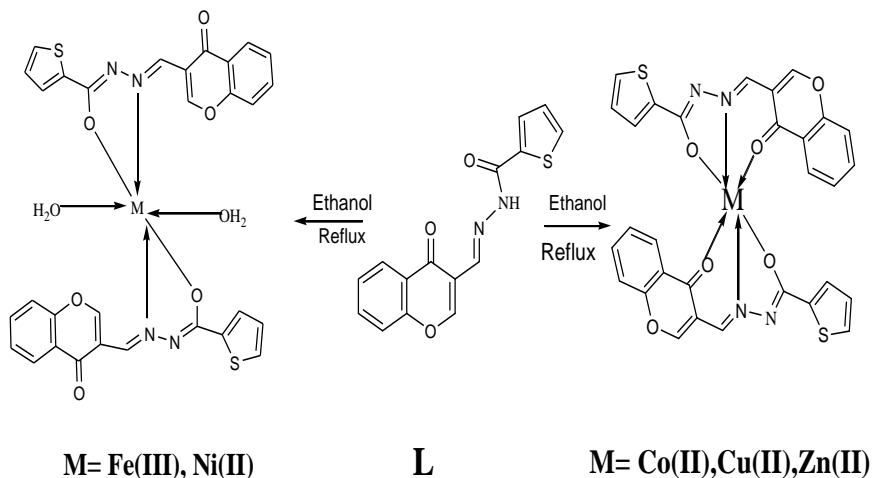


**Scheme:** The schematic route for synthesis of Schiff base (L)

### 2.5 Synthesis of metal complexes

The Schiff base ligand L (0.01 mol) is dissolved in hot methanol solution of corresponding salts (0.005 mol) M [where M=[Fe(III), Co(II), Ni (II), Cu(II), And Zn(II)] were mixed together and refluxed with constant stirring for 6–8h at refluxing temperature. On cooling colored solids were precipitated out. The products were filtered,

washed with cold methanol, dried in air and in desiccator over anhydrous CaCl<sub>2</sub> and stored in an airtight sample bottle. All the compounds are colored and are stable to air and moisture.



**Scheme:** The schematic route for synthesis of Metal Complexes(M)

### 3. RESULTS AND DISCUSSION

The analytical data and physical properties of the ligand and its metal complexes are listed in Table 1. The Schiff base ligand (L) is soluble in common organic solvents. The resultant Schiff base complexes are partially soluble in MeOH and CHCl<sub>3</sub> but freely soluble in DMF and DMSO. The analytical data indicate that the metal to ligand ratio is 1:2 in all the metal complexes.

**Table-1: Physical and analytical data of L and its metal complexes.**

Compound	Mol. Formulae (F.W.)	M.P.°C	Colour	Elemental analysis found (calculated.)%			
				% C (cal.)	% H (cal.)	% N (cal.)	% M (cal.)
Ligand (L)	C <sub>15</sub> H <sub>10</sub> N <sub>2</sub> O <sub>3</sub> S (298)	280°C	Yellow	59.44 (60.39)	2.98 (03.38)	08.88 (09.39)	-
[Fe(L) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]	C <sub>30</sub> H <sub>18</sub> FeN <sub>4</sub> O <sub>6</sub> S <sub>2</sub> (650.50)	>280°C	Green	54.33 (55.39)	2.64 (02.79)	15.10 (15.54)	7.98 (08.59)
[Co(L) <sub>2</sub> ]	C <sub>30</sub> H <sub>18</sub> CoN <sub>4</sub> O <sub>6</sub> S <sub>2</sub>	>280°C	Brown	54.90	2.22	14.78	08.24

	(653.60)			(55.13)	(02.78)	(15.53)	(09.02)
[Ni(L) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]	C <sub>30</sub> H <sub>22</sub> NiN <sub>4</sub> O <sub>8</sub> S <sub>2</sub> (689.30)	>280°C	Coffee	52.10 (52.27)	02.66 (03.22)	08.38 (08.13)	7.96 (08.51)
[Cu(L) <sub>2</sub> ]	C <sub>30</sub> H <sub>18</sub> CuN <sub>4</sub> O <sub>6</sub> S <sub>2</sub> (658.20)	>280°C	Brown	54.05 (54.75)	2.26 (02.76)	08.14 (08.51)	8.88 (09.66)
[Zn(L) <sub>2</sub> ]	C <sub>30</sub> H <sub>18</sub> ZnN <sub>4</sub> O <sub>6</sub> S <sub>2</sub> (689.30)	>280°C	Brown	54.20 (54.59)	02.10 (02.75)	08.28 (08.49)	8.78 (09.91)

### 3.1 FT-IR spectra

The IR spectra containing relevant vibrational bands of the ligands and their metal complexes are listed in Table 2.

**Table-2: The selective Infrared frequencies of ligand (L) and its metal complexes**

Compound Name	$\nu(\text{C}=\text{O})$ Chromone	$\nu(\text{C}=\text{O})$ hydrozonic	$\nu(\text{C}=\text{N})$	$\nu(\text{M}-\text{O})$	$\nu(\text{M}-\text{N})$
L	1664	1635	1568	-	
[Fe(L) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]	1652	1618	1544	574	436
[Co(L) <sub>2</sub> ]	1650	1614	1534	556	445
[Ni(L) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]	1645	1608	1562	542	440
[Cu(L) <sub>2</sub> ]	1642	1606	1568	530	455
[Zn(L) <sub>2</sub> ]	1638	1602	1540	540	428

The ligands showed a band at 1664 cm<sup>-1</sup> which is due to  $\nu(\text{C}=\text{O})$  group of the chromone moiety. This band was shifted to lower wave number region 05–10 cm<sup>-1</sup> in their corresponding metal complexes, indicating the coordination of oxygen atom of carbonyl group of the chromone moiety. The stretching vibration of the azomethine group (C=N) was observed at 1568 cm<sup>-1</sup> in the ligand. This band was shifted to lower wave number region 20-40 cm<sup>-1</sup> in their metal complexes, indicating the participation of nitrogen atom of azomethine group in coordination to the metal ion. An appearance of new broad band in the region 3266-3365 cm<sup>-1</sup> indicates the presence of coordinated water in

Fe(III),Ni(II), metal complexes. The coordination of nitrogen and oxygen atoms was supported by the appearance of a non-ligand bands in the range 500-570 $\text{cm}^{-1}$  and 416-455  $\text{cm}^{-1}$  region due to the  $\nu(\text{M-O})$  and  $\nu(\text{M-N})$ , respectively. From the above spectral data, it was concluded that schiff base ligand acts as bidentate in Fe(III) and Ni(II) metal complexes due to coordination of two water molecule and tridentate ligands in Cu(II), Co(II), and Zn(II) metal complexes with ONO donor sites.

### 3.2 $^1\text{H-NMR}$ spectra

The  $^1\text{H-NMR}$  and  $^{13}\text{C-NMR}$  spectrums of ligand was recorded in DMSO- $d_6$   $^1\text{H-NMR}$  (DMSO- $d_6$ ,  $\delta$  ppm ) 12.36 (S, 1H, NH); 8.47 (S, 1H, HC=N), 6.94-8.26 (m, 4H, Ar-H); 2.98 (S, 3H,  $-\text{CH}_3$ );  $^{13}\text{C-NMR}$  (DMSO- $d_6$ ,  $\delta$  ppm) 163.63 (C=O chromone), 160.38 (C=O hydrazone ), 155.98 ( $-\text{HC=N}$ ), 15.39( $-\text{CH}_3$ );

### 3.4 Antimicrobial activity

The in vitro antimicrobial screening of synthesized ligand and metal complexes was tested against four bacteria (*S. Aureus*, *S. Pyogenes*, *E. Coli* & *S. Typhi*) and two fungi (*C. Albicans* & *T. Rubrum*) by petri-plate containing 30 ml potato dextrose agar and nutrient agar medium, the plates were incubated for 20-24 hr and 24-48 hr for bacteria and fungi stains, respectively. The activities were measured in terms of zone of inhibition in mm. Cefotaxime, Azithromycin and Clotrimazole were used as standard drugs for bacteria and fungi, respectively at 500 ppm concentration of sample as well as drugs. The results of antimicrobial activity of ligand and metal complexes are shown in Table 3.

The metal complexes exhibit higher inhibition against tested microorganism compared to the free ligand[17]. The value in the above table indicates that the activity of the Schiff base ligand became more pronounced when coordinated with the metal ions. The presence of azomethine moiety and chelation effect with central metal enhances the antibacterial activities. This enhancement in antibacterial activity of these metal complexes can be explained based on the chelation theory .

When a metal ion is chelated with a ligand, its polarity will be reduced to a greater extent due to the overlap of ligand orbital and the partial sharing of the positive charge of the metal ion with donor groups. Furthermore, the chelation process increases the delocalization of the  $\pi$ -electrons over the whole chelate ring, which results in an increase in the lipophilicity of the metal complexes. Consequently, the metal complexes can easily penetrate into the lipid membranes and block the metal binding sites of enzymes of the microorganisms. These metal complexes also affect the respiration process of the cell and thus block the synthesis of proteins, which restrict further growth of the organism. The results of antimicrobial activity of ligand and metal complexes are shown in factors. They are the chelate effect, nature of coordinated ligand, total charge of complex, nature of the ion neutralizing the ionic complex and nuclearity of the metal center in the complex [18]. The increased activity of the metal complex than the free ligand can also be explained on the basis of chelation theory.

**Table 3: Results of antimicrobial activity of synthesized compounds**

Compounds	Zone of Inhibition in mm					
	Gm +ve bacteria		Gm -ve bacteria		Antifungal activity	
	S. Aureus	S. Pyogenes	E. Coli	S. Typhi	C. Albicans	T. Rubrum
Ligand (HL)	7	6	8	9	-	-
[Co(HL) <sub>2</sub> ]	14	14	9	8	14	16
[Ni(HL) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]	-	-	11	12	-	-
[Cu(HL) <sub>2</sub> ]	16	14	10	08	14	16
[Zn(HL) <sub>2</sub> ]	10	12	12	14	12	10
Cefotaxime	-	-	26	22	-	-
Azithromycin	26	24	-	-	-	-
Clotrimazole	-	-	-	-	16	15

#### 4. CONCLUSION

In the present work, Fe(III), Co(II), Ni(II), Cu(II), and Zn(II) complexes were prepared from (20E)-N'-((4-oxo-4H-chromen-3-yl)methylene)thiophene-2-carbohydrazide. These Schiff base are characterized using various spectral techniques. IR spectra revealed coordination of Schiff base ligand with metal ion through azomethine nitrogen, carbonyl oxygen of chromone moiety and carbonyl oxygen of hydrazide moiety. The structural elucidation studies by various spectral techniques (IR, and <sup>1</sup>H NMR) suggested the nature of ligand is bidentate in Fe(III) and Ni(II) metal complexes due to coordination of two water molecule and tridentate ligands in Cu(II), Co(II), and Zn(II) metal complexes with ONO donor sites tridentate and geometry of the metal complexes are octahedral. Antimicrobial studies suggest that Schiff base and its complexes play a vital role in developing a new class of antibiotics.

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## Chapter -38

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### Green Hydrogen Energy and Its Importance

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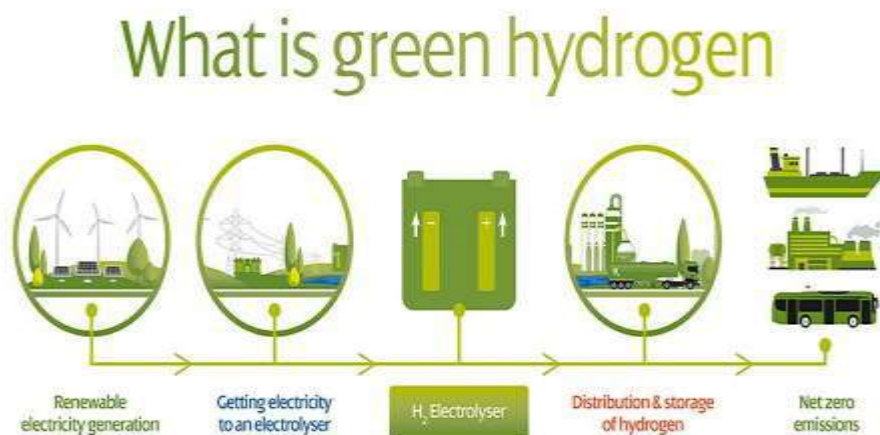
#### ABSTRACT:

Hydrogen name made from the Greek 'hydro' and 'genes' meaning water forming. A colorless, odorless gas. It has the lowest density of all gases. Hydrogen is element easily available in nature as it is clean energy source with zero emission of toxic gases and only emits water vapors on burning. Types of hydrogen are Green hydrogen, blue hydrogen, brown hydrogen and even yellow hydrogen, turquoise hydrogen and pink hydrogen. They're essentially color codes, or nicknames, used within the energy industry to differentiate between the types of hydrogen. Grey hydrogen can turn "blue" when most of these carbon emissions are captured and, for example, sequestered underground. Grey hydrogen is currently the most common, and the cheapest, form of hydrogen production. It is used as a fuel and doesn't generate greenhouse gas emissions itself, but its production process does. Green hydrogen is more expensive to produce, but it can be manufactured with zero emissions using renewable electricity to split water into oxygen and hydrogen.

#### 1. Introduction

In 1776 Hydrogen was first identified as a distinct element by British scientist Henry Cavendish after he evolved hydrogen gas by reacting zinc metal with hydrochloric acid. In a demonstration to the Royal Society of London, Cavendish applied a spark to hydrogen gas yielding water[1-4]. This gas has been used to fuel cars, airships and spaceships since the beginning of the 19th century. Belgium. Johan Martens, Tom Bosserez and Jan Rongé have invented a solar panel that produces clean hydrogen gas from sunlight and ambient moisture,

potentially providing an alternative source of green energy for buildings around the world[5-8].



Electrolysis is THE technique used to produce hydrogen that consists of “breaking” the water molecules using an electric current in an electrolyzer in order to extract the dihydrogen H<sub>2</sub>. Hydrogen is currently used in industrial processes, as rocket fuel, and in fuel cells for electricity generation and powering vehicles. There are four main sources for the commercial production of hydrogen: natural gas, oil, coal, and electrolysis; which account for 48%, 30%, 18% and 4% of the world’s hydrogen production respectively. Fossil fuels are the dominant source of industrial hydrogen[8-12].

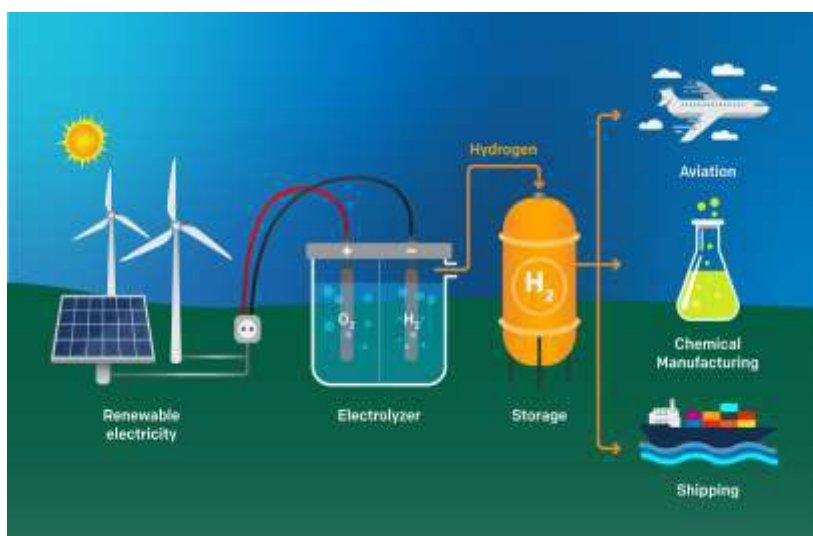
Hydrogen is a powerful, transportable energy carrier that can produce electricity, power industry, and enable transportation. Unlike fossil fuels, when hydrogen is burned, it generates only water as a by product, meaning no harmful greenhouse gas emissions. For this reason, it is an attractive fuel for the future.

### **Methods of Production of Green Hydrogen:**

Electrolysis is THE technique used to produce hydrogen that consists of “breaking” the water molecules using an electric current in an electrolyzer in order to extract the dihydrogen H<sub>2</sub>. The electricity must itself be carbon-free in order to consider this hydrogen as green or renewable

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Renewable electricity > Electrolyzer > Hydrogen Storage > Use as energy source for various purpose in industry.



The cost of manufacturing green hydrogen, which is made using renewable energy rather than power derived from fossil fuels, in India is currently at about 300 rupees per kg.



### **Top Companies Producing Green Hydrogen**

Plug Power Inc, Adani Green Energy Ltd., Air Products and Chemicals Inc., Sinopec., Air Liquide S.A., Linde plc., Shell plc., Reliance Industries Ltd.

### **Uses of Green Hydrogen:**

Green Hydrogen is considered a promising alternative for enabling this transition. Hydrogen can be utilized for long-duration storage of renewable energy, replacement of fossil fuels in industry, clean transportation, and potentially also for decentralized power generation, aviation, and marine transport.

### **Limitations of Green Hydrogen:**

Although it is 100% sustainable and versatile, green hydrogen is expensive to produce due to the cost of energy from the renewable sources that are key to generating green hydrogen through electrolysis. It requires more energy than other fuels to produce any kind of hydrogen, green in particular.

Hydrogen is highly volatile and flammable because of this the risk of leakage explosions so extensive safety measures are needed.

### **CONCLUSION:**

There is few renewable green energy source present but there use is limited and the overall working in practical daily life is not that much because we have not targeted these energy source. In fact we have not investing our best for the use of these energy. We have to focus on these types of energy and develop the needful and practical useable technology to convert these energy for the daily use. Energy like wind energy, solar energy, green hydrogen, biogas, hydropower, geothermal heat these are sources on which we have to focus. In this book we are discuss on the availability of energy sources, recent need of energy and actual production and supply of energy. In this proportion the green energy production and actual use of energy as per need. By the increasing production and supply chain we can use the maximum overall need so it can reduce the pollution.

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## Chapter -39

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### Pharmacological Effects of Imidazole Derivatives

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#### Abstract:-

Imidazole derivatives is having Broad Spectrum application in medicinal chemistry. Imidazole is a nitrogen-containing heterocyclic ring; it has two equivalent forms; hydrogen atom may be located on any of two nitrogen atoms. Imidazole ring may interact with various cations and anions, as well as with biomolecules by different reactions; the presence of various groups in the nitrogenous heterocycle structure makes it possible to identify substances with a broad spectrum of pharmacological effects. Imidazole drugs have broadened scope in remedying various dispositions in clinical medicines. Numerous methods for the synthesis of imidazole and also their various structure reactions offer enormous scope in the field of medicinal chemistry. Antibiotic resistance is a phenomenon where microorganisms acquire or innately possess resistance to antimicrobial agents. New materials offer a promising antimicrobial strategy as they can kill or inhibit microbial growth on their surface or within the surrounding environment with superior efficacy, low toxicity and minimized environmental problems. The present chapter focuses on classification of antimicrobial materials, surface modification and design requirements, their mode of action, antimicrobial evaluation tests and clinical status.

**Keywords-** Imidazole, antibacterial, antifungal, heterocyclic, biological active

#### Introduction :-

One of the modern objectives of medicine is to search for new biologically active substances with high efficiency and low toxicity to the human body.

Currently, the complex organic molecule directed synthesis method development studies are carried out in the modern synthetic organic chemistry to obtain physiologically active substances with the selective effect [01]

Imidazole is an entity which is being synthesized in many of its derivative form from past few years; the entity is major source of interest for many of medicinal chemist to explore its various pharmacological potentials. In present Chapter we review the chemistry of imidazole and its pharmacological actions as antihelmintics, anticancer, antifungal and anti-inflammatory agent by studying its various synthesized derivatives. [02] Imidazole-containing drugs have a broader spectrum of application in clinical medicine. Imidazole component presents in some pharmacologically important drugs such as Metronidazole, Pretomanid, Ketoconazole, Clotrimazole and Miconazole[03]

### **Pharmacological effects**

Imidazole is better known due to its broad range of chemical and biological properties. Imidazole has become an important synthon in the development of new drugs. The derivatives of 1, 3-diazole show different biological activities such as antibacterial, antimycobacterial, anti-inflammatory, antitumor, antidiabetic, anti-allergic, antipyretic, antiviral, antioxidant, anti-amoebic, antihelmintic, antifungal and ulcerogenic activities, etc. as reported in the literature. There are different examples of commercially available drugs in the market which contains 1, 3-diazole ring such as clemizole (antihistaminic agent), etonitazene (analgesic), enviroxime (antiviral), astemizole (antihistaminic agent), omeprazole, pantoprazole (antiulcer), thiabendazole (antihelmintic), nocodazole (antinematodal), metronidazole, nitroso-imidazole (bactericidal), megazol (trypanocidal), azathioprine (anti rheumatoid arthritis), dacarbazine (Hodgkin's disease), tinidazole, ornidazole (antiprotozoal and antibacterial), etc.[04]



### 1. Antifungal Activity

Different series of imidazole and triazole derivatives having an azomethine linkage to pyridine 2-carboxamidrazone were designed and synthesized by Bangi et al.[05] show good antifungal and activities *in vitro* by standard and newly developed microdilution assays.

A novel imidazole ligand was meticulously synthesized through a reaction involving a 1,3-oxazole derivative and hydroxylamine by Abdullah et.al.[06] the ligand & complexes demonstrated high inhibition activity against *Candida albicans*, and *Aspergillus flavus*. The inhibitory activity exhibited a positive correlation with the concentration of the metal complexes. As the concentration of the metal complexes increased, their inhibition activity against the tested microorganisms improved significantly the antifungal activity was also enhanced when the ligand was coordinated with metal ions, underscoring the potential of these complexes as antifungal agents.

### 2. Antibacterial Activity

Three series of 2-cyclopropyl-5-(5-(6-methylpyridin-2-yl)-2-substituted - 1H - imidazol - 4 - yl) - 6 - phenylimidazo [2,1-b] [1,3,4] thiadiazoles (13a-e, 14a-d, and 15a-f) were synthesized and their antibacterial activity Showed the strongest activity against Gram-positive and drug-resistant bacteria as well as high selectivity against Gram-negative bacteria.[07] Slassi and Co workers.[08] synthesized the newly triazole derivatives by condensation of (E)-5-((1H-1,2,4-triazol-3 - yl) diazenyl) - 2 - hydroxy - 3 - arylbenzaldehyde and N(-3-aminopropyl) imidazole antibacterial screening of triazole compounds is prepared against four pathogenic bacteria, one Gram-positive cocci *Staphylococcus aureus* and three Gram-negative bacteria *E. coli*, *Klebsiella pneumoniae* and *Pseudomonas putida* shown excellent results for antibacterial activity [08]

### 3. Anticarcinogenic Activity

Bai et.al. reported on 5-(3,4,5-trimethoxybenzoyl)-4-methyl-2-(*p*-tolyl) imidazole (BZML, **13**), a compound with potent activity against colorectal cancer cell lines that inhibits tubulin polymerization and causes DNA damage.[09]

#### 4. Antiviral Activity

A series of novel 2-substituted 7,8-dihydro-6*H*-imidazo[2,1-*b*][1,3]benzothiazol-5-ones (**3a-k**) were synthesized by cyclohexane-1,3-diones and assessed for their cytotoxicity and antiviral activity against influenza virus A/Puerto Rico/8/34 (H1N1) in MDCK cells.[10]

#### 5. Anti-inflammatory Activity

Novel small molecules of the imidazole class synthesized by Nascimento & Co. workers screen for their *in vitro* anti-inflammatory activity. eight imidazoles tested methyl 1-allyl-2-(4-fluorophenyl)-5-phenyl-1*H*-imidazole-4-acetate inhibited nitric oxide metabolites and pro-inflammatory cytokine (TNF- $\alpha$ , IL-6, and IL-1 $\beta$ ) secretion in J774 macrophages stimulated with LPS. . In this context, results demonstrate that imidazole has promising potential as a prototype for the development of a new anti-inflammatory drug to treat inflammatory conditions in which NF- $\kappa$ B and oxidative stress play a prominent role.[11]

#### 6. Antidiabetic Activity

A new series of fused carbazole-imidazoles as potential  $\alpha$ -glucosidase inhibitors was synthesized by Adib and Coworkers. They evaluated these agents for their  $\alpha$ -glucosidase inhibitory activities on  $\alpha$ -glucosidase from *Saccharomyces cerevisiae*. all of the synthesized compounds revealed more potent than acarbose (standard drug). revealed that these compounds interact with the key residues in the active site of  $\alpha$ -glucosidase.[12]

#### Conclusion:-

Considering these pharmacological properties of Imidazole, it is expected that these compounds have the effective activity. In addition, the mutual combination of Imidazole ring and various substituents may result in the effect enhancement. Based on the studies conducted in recent years and confirmed the antifungal and antibacterial activity, there are grounds for further study of Imidazole derivatives.

In perspective, the addition of new compounds may result in the development of safer and more effective compounds.

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## Chapter -40

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### Synthesis Of 3,4-Dihydro-3,3-Dimethyl-9-Arylacridin-1-Ones Using Alum ( $KAl(SO_4)_2 \cdot 12H_2O$ )

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#### ABSTRACT

Alum ( $KAl(SO_4)_2 \cdot 12H_2O$ ) catalyzed synthesis of One-pot synthesis of 3,4-dihydro-3,3-dimethyl-9-acylaridin(2H,9H,10H)-1-one by the cyclocondensation of aromatic aldehydes, aromatic amines and dimedone and use of Alum ( $KAl(SO_4)_2 \cdot 12H_2O$ ) nanocomposite heterogeneous catalyst and its reusability. The method is cost effective and eco-friendly. And use of ethanol as a solvent makes the method greener and more efficient. The method has simple workup procedure and the products are obtained in good to moderate yields.

**Key Words:** 3,4-Dihydro-3, 3-Dimethyl-9-Arylacridin-1-Ones, Alum ( $KAl(SO_4)_2 \cdot 12H_2O$ )

#### INTRODUCTION

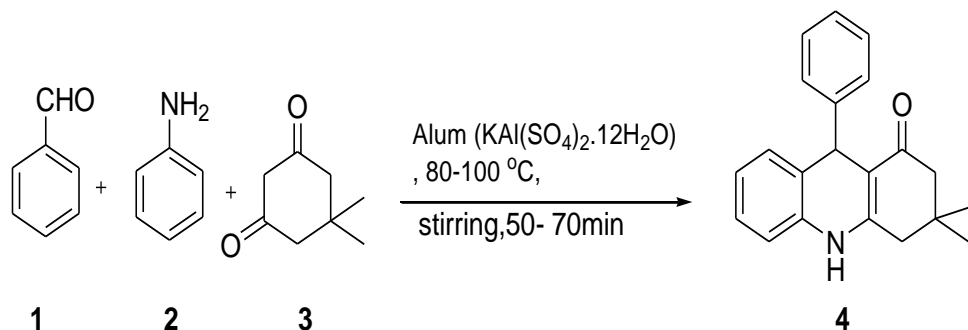
It is expected from all the chemists and co-workers while performing any chemical conversions in the laboratory. Therefore, chemists have to change the procedures or keep control of it so that the developed methodology can help to protect human health and sustain the environment unaffected<sup>1-9</sup>. Keeping these factors in view, it was thought worthiness to synthesize 3,4-dihydro-3,3-dimethyl-9-phenylacridin-1-ones.

A mild, general method still remains a challenge. Recently many organic reactions are been carried out in water which is readily available, non-toxic, inexpensive and eco-friendly solvent. Here we are interested to use alum ( $KAl(SO_4)_2 \cdot 12H_2O$ ) which is also non-toxic, easy

handling, eco-friendly and inexpensive catalyst which is previously been reported as effective catalyst for the synthesis of 5-arylidene-2,4-thiazolidinedione<sup>10</sup>, coumarins<sup>11</sup>, anthraquinone<sup>12</sup>, dihydropyrimidine<sup>11</sup> and trisubstituted imidazoles<sup>12</sup>.

### Procedure for the synthesis of 3,4-dihydro-3,3-dimethyl-9-phenylacridin-1-one derivative

Benzaldehyde (0.223 g, 0.002 mol) and aniline (0.194 g, 0.002 mol) and Alum( $KAl(SO_4)_2 \cdot 12H_2O$ ) (15 mol %) was added ethyl alcohol were stirred at for 50-70 minutes. To this solution, dimedone (0.292g, 0.002 mol) was added portion wise with continued stirring at same temperature. The progress of the reaction was monitored after interval of each 25 min by TLC. The reaction is completed after specified period of time. After completion the reaction mixture was poured on crushed ice. The obtained solid was filtered, dried and purified by recrystallization in ethanol to yield a pure product. Similar procedure was applied for the synthesis of other derivatives. All compounds were characterized by spectroscopic analysis.



**Scheme.** synthesis of One-pot synthesis of 3,4-dihydro-3,3-dimethyl-9-acylaridin-1-one

### Compound 4d:7-Chloro-3,4-dihydro-3,3-dimethyl-9-phenylacridin-1(2H,9H,10H)-one

M. P. 189-190 °C; <sup>1</sup>H NMR ( $CDCl_3$ )  $\delta$  ppm; of 10.00 (s, 1H, N-H), 7.7 (d, 2H, Ar-H,  $J = 1.9$ ), 7.2 (s, 1H, Ar-H), 6.8 - 7.00 (m, 5H, Ar-H), 4.8 (s, 1H, C-H), 3.00 (s, 4H, CH<sub>2</sub>), 2.10 (s, 6H, CH<sub>3</sub>); MS  $m/z = 336.25$ ; IR (KBr);  $\nu$  cm<sup>-1</sup> 3127 (N-H Str.), 3020 (CH<sub>3</sub> Str.), 1780 (C=O Str.), 1420 (C=C Str.), 800 (C-Cl Str.).

**Table. One pot Synthesis of 3,4-dihydro-3,3-dimethyl-9-acylaridin-1-one**

Entry	R/Aldehyde	R' /Amine	Time, min	Yield, %
<b>4a</b>	H	H	65	80
<b>4b</b>	4-Cl	H	50	83
<b>4c</b>	H	4-Cl	70	82
<b>4d</b>	4-CH <sub>3</sub>	H	70	79

## RESULT AND DISCUSSION

One pot cyclo condensation of Benzaldehyde (0.223 g, 0.002 mol) and aniline (0.194 g, 0.002 mol) to that Alum ( $KAl(SO_4)_2 \cdot 12H_2O$ ) (15 mol %) was added it was carried out under ultrasound irradiation which result into the subsequent To this solution, dimedone (0.292g, 0.002 mol) was added portion wise with continued stirring at same temperature (**4a-d**) as given in (**Table** ). We have screened various percentage Alum ( $KAl(SO_4)_2 \cdot 12H_2O$ ) and optimization using different mol percentage for the reaction which was carried out under stirring and heating up to 80 to 100°C.

## CONCLUSION

In conclusion, we have developed a simple and highly efficient method were 3,4-dihydro-3,3-dimethyl-9-acylaridin-1-one and their derivatives are synthesized using Alum ( $KAl(SO_4)_2 \cdot 12H_2O$ ) as heterogeneous catalyst which is reusable and cost-effective. The reaction is performed in ethanol as solvent under stirring and heating up to 80 to 100°C. Thus, the method is clean and efficient method. Further studies on the biological activities of the products and application of this methodology to other interesting (**4a-d**) derivatives are underway in our laboratory.

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## Chapter -41

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### Aquaponics

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#### Introduction

The combined technique of fish and plant production known as aquaponics ([Goddek et al., 2019](#)) is mainly made up of two subsystems: hydroponics and aquaculture. The basic idea is to divide and share nutrient resources between fish and plants, as well as to effectively use water to grow two crops instead of one. This integrated farming approach is widely utilized to cultivate fish and vegetables in urban settings with limited resources. Aquaponics is the culture of fish and horticultural plants. Numerous plants can be used in aquaponics systems; however, which ones are best for a given system will depend on the fish's stocking density and age.

Aquaponics is a hydroponic and aquaculture hybrid([Burnell et al., 2020](#)) agricultural method that is used to grow plants and aquatic animals. In aquaponics systems, crop plants, fish, and bacteria form a closed circuit that cycles nutrients and fish waste is converted by the system's nitrifying bacteria into nitrates for plants, which clean the fish's water.

Aquaponics systems work well with green leafy plants that have low to medium nutrient requirements, such as tomatoes, watercress, chives, basil, spinach, lettuce, and capsicum. It is essentially a Recirculation Culture System in which fish waste is routed into bio-filter troughs containing horticultural plants, and fish are fed high-quality floating pellet feed([Hitchcock, 2019](#)). The water flow rate is controlled with the aid of a timer. Aquaponics systems grow entirely organic fish and plants.

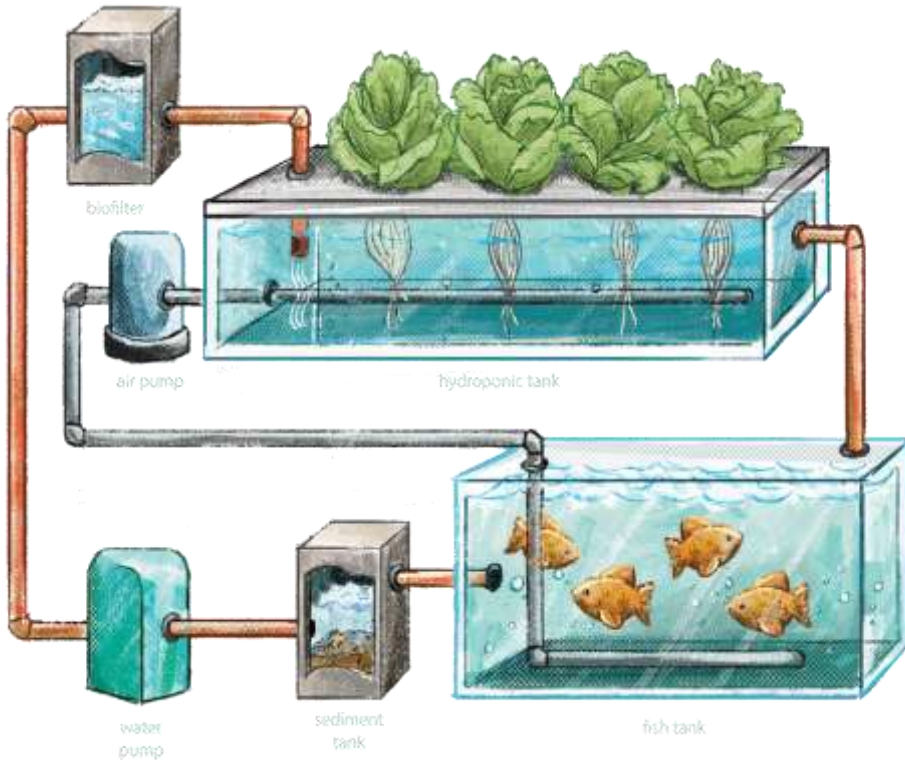
## **Back ground**

During the height of aquaculture research in the 1970s and 1980s, modern aquaponics systems emerged. The 1969-founded research centre New Alchemy Institute collaborated with Sol search Architects to design the "Ark," a residential building on Prince Edward Island that integrated fish and vegetable farming([Hitchcock, 2019](#); [Packer, 2014](#)). In the 1980s, scientists at North Carolina State University built the first successful closed-loop aquaponics circuit, and in Amherst, Massachusetts, the first commercial aquaponics facility debuted in the same decade. The idea gained increasing traction in the early years of the twenty-first century, particularly in Australia and Canada, and commercial systems and kits for backyard aquaponics were easier to get.

## **System Working Method**

Aquaponics systems depend on the interaction of bacteria, plants, and fish. Simplified, the system mimics the nutrient exchanges and natural cycles of aquatic environments. Fish are kept in a tank that is connected to the crop beds via a pump in a typical aquaponics system. The water in the system needs to have a pH of between 6.8 and 7.2, which is the neutral range. Fish are at risk of dying at incorrect pH levels, and plants are unable to properly absorb nutrients([Hitchcock, 2019](#)). For freshwater aquaponics, carp, catfish, and tilapia are common fish species employed. The fish excrete waste that is high in the chemical component ammonia and ingest nitrogen-containing diet. The aquaculture component of aquaponics is used to raise aquatic animals, while the hydroponics component is used to cultivate plants. Due to the closed-system recirculation of most aquaculture systems, aquatic effluents from uneaten feed or rearing fish build in water. High amounts of the effluent-rich water are hazardous to aquatic life, but it also includes nutrients that are vital to plant growth.

While essentially made up of these two components, aquaponics systems are typically divided into a number of sections or subsystems that are in charge of efficiently removing solid waste, introducing bases to neutralize acids, or keeping the oxygenation of the water at a certain level(Hargrove, 2021; Packer, 2014).



**Flow Chart of Aquaponics**

**Typical elements consist of**

- **Rearing tanks-** fish tanks used for fish rearing and feeding(Simpson, 2017)
- **A settling basin-** is a device used to remove tiny particles and collect leftover food and biofilms.
- **A biofilter-** is an environment in which nitrification bacteria can proliferate and transform ammonia into nitrates that plants can use;

- **Hydroponics subsystem** -The section of the system where plants are grown by absorbing extra nutrients from the water is known as the hydroponics subsystem(Southern & King, 2017)  
**The sump**-is the lowest point in the system, when water is pumped back to the rearing tanks after flowing there.

The units for solids removal, biofiltration, and the hydroponics subsystem may be merged into one unit or subsystem, depending on the complexity and cost of the aquaponics system. This keeps the water from moving straight from the aquaculture portion of the system to the hydroponics portion. Sand or gravel can be used as a plant supporting medium since it has the surface area to enable fixed-film nitrification while also capturing solids. An expensive, separate biofilter is frequently not necessary for aquaponic systems because of the possibility to integrate hydroponics with biofiltration.

To function properly, an aquaponic system needs a variety of live components. Fish and other water organisms, plants, and bacteria are the three main living components. Certain systems additionally incorporate live components, such as worms.

## **Plants**

A hydroponics technique called Deep Water Culture(Bernstein, 2011; Simpson, 2017), in which plants are grown without the use of a soil medium, straight into the effluent-rich water. Because the roots do not have to spread outward to support the weight of the plant, plants can be planted closer together. Plant placed in a nutrition film technique (NFT) system's nutrient-rich water channel. Numerous plants can be used in aquaponic systems; however, which ones are best for a given system will depend on the fish's stocking density and age. These variables affect the amount of nutrients that are released into the plant roots by bacteria as well as the concentration of nutrients found in fish effluent. Chinese cabbage, lettuce, and other green leafy vegetables with low to medium nutrient requirements grow well in aquaponic systems.

## **Fish**

Because they can handle crowding, freshwater fish are the most popular aquatic animal farmed in aquaponics. Since freshwater crayfish and prawns generate nutrient-rich excrement, they are also occasionally employed. Saltwater aquaponics is a subset of aquaponics that uses saltwater fish. Both warmwater and cold water fish species are well suited to aquaculture systems. Since tilapia are a warmwater fish species that can withstand crowding and fluctuating water conditions, they are really the most preferred fish for both residential and commercial projects that aim to raise edible fish(Hamilton et al., 2023).

## **Bacteria**

One of the most crucial processes in an aquaponic system is nitrification, or the aerobic conversion of ammonia to nitrates. This process lessens the toxicity of the water for fish and enables the plants to take the resultant nitrate molecules for nutrition(Ruiz et al., 2023). As a byproduct of their metabolism, fish excrete ammonia into the water through their gills and excreta. However, higher concentrations of ammonia (usually between 0.5 and 1 ppm) can harm fish growth, cause extensive tissue damage, reduce their resistance to disease, and even kill them. As a result, the water must be filtered to remove the ammonia. While plants may take up ammonia from the water to some extent, nitrates are more readily absorbed, which effectively lowers the water's toxicity for plants(Ruiz et al., 2023).

## **Conclusion**

Aquaponics is an eco-friendly, sustainable farming method that produces food that is both nutritious and abundant, which makes it a crucial strategy for food production in the future. No matter the location or environment, aquaponics makes it possible to grow food anywhere. However, despite the fact that no method is perfect, it seems that governments and environmental groups worldwide are starting to recognize the potential of aquaponics as a solution to their nations' food scarcity concerns.

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## Chapter -42

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### **Ultrasonic Studies on Molecular Interaction of Urea Based Aqueous System at 298.15 K through Computation of Excess Parameters**

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#### **ABSTRACT:-**

Research and development in the field of Ultrasonics is growing steadily. Ultrasonic non-destructive testing is a resourceful technique that can be appropriate for the study of liquids and solutions. In the present paper, attempts have been made to estimate the Excess parameters of the binary system consisting of Urea+water at ambient temperature 298.15 K and at constant 2 MHz frequency. Experimental measurements of Ultrasonic Velocity ( $u$ ) and density ( $\rho$ ) have been carried out on aqueous solution of Urea at different concentration and at ambient temperature 298.15 K. From experimental data the excess parameters such as excess Ultrasonic Velocity ( $U^E$ ), excess density ( $\rho^E$ ), excess intermolecular free length ( $L^E$ ), excess adiabatic compressibility ( $\beta^E$ ) and excess acoustic impedance ( $Z^E$ ) have been obtained. The results have been interpreted in the light of solute-solvent molecular interaction.

**Keywords:** Excess Ultrasonic velocity, Excess Density, Excess Intermolecular free length, Excess Adiabatic compressibility, Excess acoustic impedance.

#### **1. INTRODUCTION**

Many scientists extensively used ultrasonic methods to investigate the nature of molecular interactions and discuss their physico-chemical behaviors [1-3]. The Ultrasonic technique is the most important and powerful tool in the estimation of ultrasonic velocity and related parameters which help in understanding molecular

structure and their interactions occurring in the formation of the liquid mixtures[4]. In general, the Excess properties of liquid mixtures are helpful in understanding molecular structural arrangement and interaction during bond making and bond breaking process. Acoustical parameters throw light on different kinds of associations such as formation of molecular agglomerates, molecular motion and various kinds of intermolecular interactions and their strength in the liquid mixture [5]. Many research workers have been studied different mixed solvents because they find practical applications in many chemical, biological and industrial processes [6]. From the agricultural research point of view usage of Urea is of utmost importance hence we have undertaken the study of Urea aqueous system using ultrasonic technique as a function of concentration at temperature 298.15 K.

## 2. MATERIALS AND METHODS

The fine powder of purified AR grade urea is used in the present study. The experimental part consists of weighing of Urea powder and preparation of standard stock solution. From this standard solutions, different concentrations of urea+water binary system explained in our earlier work [7]. To prepare binary liquid mixture of (urea+water) system, the deionized and double glass distilled water was used. The specific conductivity of the deionized and glass distilled water was  $\sim 10^{-6}$  Siemens-cm<sup>-1</sup>. The special air tight or sealed glassware were used for preparations and storage of solutions in a dry and clean place.

### Ultrasonic velocity measurements

In the present experimental setup the crystal ultrasonic interferometer (F-81 model supplied by Mittal Enterprises, Ltd. New Delhi) operating frequency at 2MHz has been used to measure ultrasonic velocity at 298.15 K temperature maintained by constant temperature controller water bath with accuracy  $\pm 0.1^\circ$  K. The ultrasonic interferometer was calibrated by using standard glass distilled water at 298.15K. The present calibrated experimental value is 1497.08ms<sup>-1</sup> which is in good agreement with reported literature value of 1496.69 ms<sup>-1</sup>[8].



## Density Measurements

The Density Measurements were performed with the help of specific gravity bottles of 25 ml. Measurements of density were carried out by an accuracy of  $\pm 2 \times 10^{-2}$  kg m<sup>-3</sup>. To minimize error in measurements average of ten measurements are considered. In the experiment accuracy of temperature was maintained (at  $\pm 0.001$ K) with the help of thermostatic water bath.

### 3. THEORETICAL ASPECTS AND CALCULATIONS

From the experimental values of the ultrasonic velocity( $u$ ) and density( $\rho$ ) the various acoustical parameters such as the adiabatic compressibility( $\beta$ ) and acoustic impedance( $z$ ) were calculated by using the empirical Jacobson's relations[9-13] which are as follows:

$$\text{Ultrasonic velocity: } u = n \times \lambda \text{----- (1)}$$

$$\text{Adiabatic compressibility: } \beta = 1 / \rho u^2 \text{----- (2)}$$

$$\text{Specific Acoustic impedance: } Z = u \times \rho \text{----- (3)}$$

Where  $K$  is temperature dependent constant called as Jacobson's constant.  $\rho$  and  $u$  are the density and ultrasonic velocity of binary solution.

From above experimentally measured values and various calculated acoustical parameters the excess thermodynamic properties such as  $u^E, \rho^E, \beta^E, Z^E$ , and have been calculated using following equation:

$$Y^E = Y_{mix} - [X_1 Y_1 + X_2 Y_2] \text{----- (4)}$$

Where  $Y^E$  is excess ultrasonic velocity( $u^E$ ), excess intermolecular free length( $L_f^E$ ), excess adiabatic compressibility( $\beta^E$ ), excess density( $\rho^E$ ), and excess Specific Acoustic impedance ( $Z^E$ ) and  $(x)$  represents mole fraction of the component and subscript 1 and 2 for the components 1 and 2, respectively.

### 4. RESULTS AND DISCUSSION

Excess acoustic and thermodynamic parameters were found to be very in elucidating intermolecular solute solvent interaction in aqueous binary mixtures. The experimental values of ultrasonic

velocity and density and other derived acoustical parameters were utilized to evaluate various excess parameters using equation (5) which were reported in Table 1 for Urea+water binary system at constant temperature 298.15 K.

### **i) Excess Ultrasonic velocity ( $u^E$ ):**

The values of excess ultrasonic velocity have been evaluated by using standard relation stated as above. From Table 1, it was observed that, the values of excess ultrasonic velocity are positive at all increasing concentration of solute at constant temperature 298.15 K for urea+water binary system. The increasing trend of  $u^E$  plot with increasing concentration of solute was observed. Plot shows on increasing concentration (x) of urea in liquid mixtures the values of excess ultrasonic velocity also increases. This positive and increasing trend of plot for urea+water binary system indicates that the molecular interaction between urea+water binary system was stronger as show in Fig. 1.

### **ii) Excess adiabatic compressibility ( $\beta^E$ ):**

The variation of excess adiabatic compressibility ( $\beta^E$ ) with concentration at temperature 298.15 K was given in Table 1. Fig.2 shows that the value of excess adiabatic compressibility ( $\beta^E$ ) was negative and plot shows clearly negative decreasing trend for urea+water binary system. Excess values observations support that the binary mixture has a tendency for closed packing in the mixture. Breaking of H-bond was responsible for excess ( $\beta^E$ ).

The negative value of ( $\beta^E$ ) indicates significant interaction between molecules in binary mixture forming donor-acceptor complex. As a result of complex formation there was contraction in volume resulting negative values of ( $\beta^E$ ) with concentration (x).

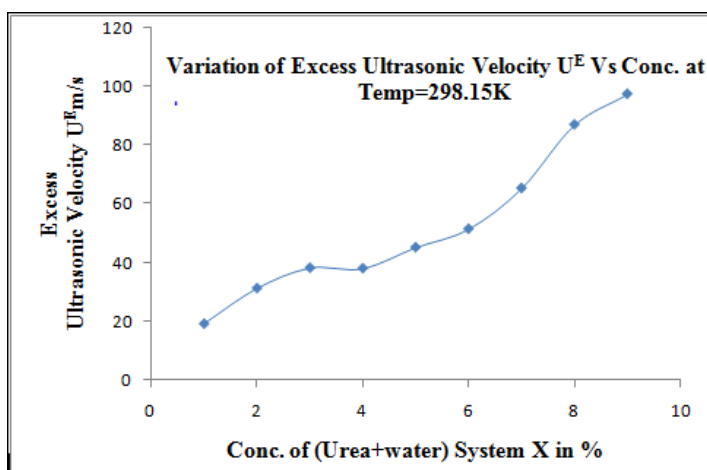
### **iii) Excess acoustic impedance ( $Z^E$ ):**

From Table 1, it was observed that the values of excess acoustic impedance ( $Z^E$ ) were positive and plot shows increasing trend as concentration of urea increases in binary system at temperature 298.15 K. The positive excess value of ( $Z^E$ ) suggest the presence of weak

interaction among unlike molecules. As size of component molecules not equal, so their molecules do not pack well into each other's structures, which results in expansion of volume and hence positive excess ( $Z^E$ ) shown in Fig. 3

**Table 1. Acoustically measured various Excess parameters of (Urea + water) system at 298.15 K.**

Conc % mole/lit	$U^E$ m/s	$\rho^E$ * $10^3$ Kg/m <sup>3</sup>	$\beta^E$ * $10^{-10}$ m <sup>2</sup> /N	$Z^E$ * $10^{-6}$ Kg/m <sup>2</sup> s
1	19.0669	0.0321	-0.2415	0.067
2	31.1483	0.0346	-0.3166	0.0831
3	38.1097	0.0369	-0.3628	0.0941
4	37.931	0.0418	-0.3871	0.1032
5	45.0689	0.0605	-0.4311	0.1392
6	51.3523	0.0729	-0.5707	0.1651
7	65.2302	0.0812	-0.6679	0.1932
8	86.8645	0.1011	-0.8361	0.2483
9	97.2822	0.1108	-0.9143	0.2743



**Fig1. Plot of  $u^E$  (Urea + water) system at 298.15 K.**

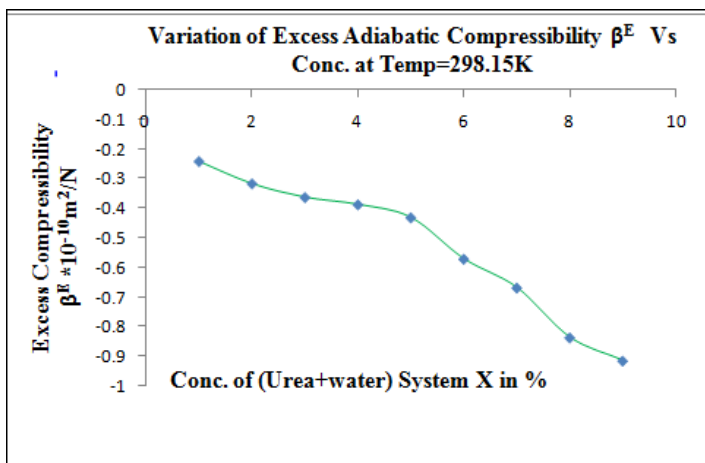


Fig2. Plot of  $\beta^E$  (Urea + water) system at 298.15 K.

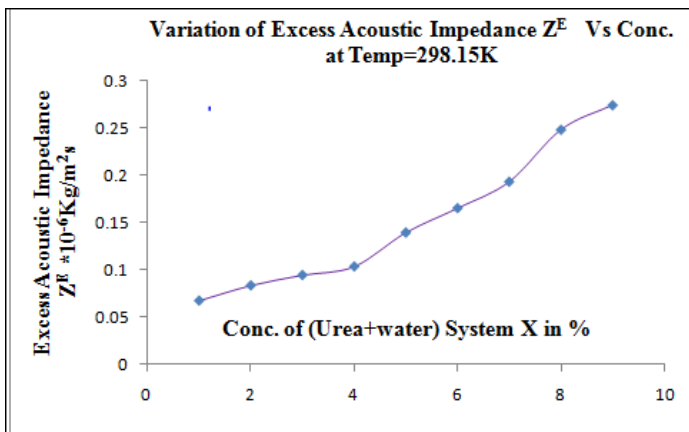


Fig.3. Plot of  $Z^E$  (Urea + water) system at 298.15 K.

## 5. CONCLUSION

In the present paper the excess thermodynamic parameters like  $u^E, \rho^E, \beta^E$  and  $Z^E$  were evaluated and reported here for urea+water binary system at temperature 298.15 K. The trend of ultrasonic velocity in given system was increasing which shows that urea was structure making nature that is it dissociates by donating H<sup>+</sup> ions. It was oxidation process. In urea+water binary system intermolecular interaction with ions were strong which is indicated by excess parameters. Hence complex formation occurs due to solute solvent interaction, dipole-dipole or ion-dipole interaction.

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