Chronicle View of Coordination Chemistry

Coordination Chemistry is the science concerned with the interaction of organic and inorganic ligands with various metal ions. It is the study of physical and chemical properties, synthesis, structures and pharmacological applications of coordination compounds. Coordination Compound or Metal Complex consists of a central atom or ion, which is usually metal ion and is called the coordination centre, and surrounding array of bound molecules or ions, that are in turn known as ligands. Many metal-containing compounds, especially those of transition metals, are coordination complexes [1]. The Coordination Chemistry was pioneered by Nobel Prize winner Alfred Werner (1866-1919). He received the Nobel Honour in 1913 for his "Coordination Theory of Transition Metal-Amine Complexes" in Inorganic Chemistry. Eventually, Danish chemist Jorgensen (1837-1914) and Russian chemist L.A. Chugaev (1873-1922), also gave their great contributions to the field of coordination chemistry.

The coordination compound or complex entities behave as cationic, anionic or neutral in nature. Those substances, whose molecules contain a central atom (cation), coordinated with ligands (anions, neutral molecules or radicals) can be considered as coordination or complex entities [2] and the branch of Inorganic Chemistry, which study the joint behaviour of cations (metal ions) and their ligands was termed as Coordination Chemistry [3]. At the start of the 20th century, inorganic chemistry was not a prominent filed of chemistry. The importance of the metal complex chemistry in 21st century accented as "from biology to nanotechnology" [4]. The principal goal is to work not only for theoretical, but also for practically useful coordination chemistry (competitive coordination), taking into account an operated creation of polyfunctional materials (for instance, selective electrodes). Creation of
new hard-soft ligand systems is attractive [5-8] and creation of hybrid biologically perspective heterocyclic ligating systems is also interesting [9-13].

Coordination compounds play an essential role in the chemical industry and in life itself. The 1963 Nobel prize in chemistry was awarded jointly to Dr. K. Ziegler of the Max Planck Institute, in Germany, and to Prof. G. Natta of the University of Milan, in Italy. Their research was responsible for the low pressure polymerization of ethylene, which now makes the thousands of polyethylene articles common place [14].

Coordination Chemistry also has played major role in medicine. coordination compounds have been used in the treatment or diagnosis of diseases. In some cases, these compounds can be formed in the body to handle dysfunction due to metal poisoning. Coordination Chemistry has also its greatest applications in many industrial process e.g. hydrometallurgy. From the solutions of the earth metal Fe(III) is removed by the use of carboxylic acid extractant have been reported. For large scale production of pure oxides of Eu(III), La(III) and Y(III) metals, versatic acid is used. Isolation and separation of lanthanides/ actinide elements carried out by developing new water soluble chelating agents and there use as extracting agents via solvent extraction or ion exchange methods.

**Schiff Bases**

Coordination Chemistry is influenced by the utilization of the Schiff bases as ligand [15] due to their chelating and complexing abilities towards almost all metal ions. Schiff base compounds have imine (-RC=N-) / azomethine bonds which are usually formed by the condensation of a primary amines with an active carbonyl compounds(Ketones, Aldehydes etc); they are named after Hugo Schiff who first
reported them in 1864 [16]. The Schiff base ligands have significant importance in the development of **Coordination Chemistry** as they are readily form stable and non hygroscopic complexes with most of the metals ions. Every year number of new Schiff base ligands and their metal complex applications in chemical reactions are published [17]. The Schiff base compounds and their metal complexes have applications in enantioselective cyclopropanation of styrenes [18], asymmetric addition of cyanide to aldehydes [19], asymmetric aziridination of olefins and enantioselective epoxidation [20,21], regio-selective ring opening of epoxides [22] and as a membrane in ion selective electrode [23-27]. In addition to varied magnetic properties and catalytic activity, the Schiff base metal complexes can also served efficient model for the metal containing sites in metallo-enzymes [28,29]. The metal complexes of Schiff base ligands have been found to possess applications as a catalyst in several synthetic chemical reactions [30-33]. They have also been models for several biological processes [34] such as oxygen carriers and also act as drugs [35].

Many reports speak about better biological activities of the Schiff bases is due to the presence of imine or azomethine group which is responsible for antibacterial, antifungal, herbicidal, clinical and analytical activities[36-39]. Recently, there has been tremendous interest in studies related to the interaction of transition metal ions with nucleic acid because of their relevance in the development of new reagents for biotechnology and medicine [40]. There has also been substantial interest in the rational design of novel Schiff base and their transition metal complexes, which bind and cleave duplex DNA with high sequence and structure activity relationship.

The preparative accessibility, diversity and structural aspects of Schiff bases have made them to act as the stereochemical models for many biologically important molecules [41]. The field of bioinorganic chemistry, which deals with the study of
role of metal ions in the form of complexes in biological systems, has opened a new horizon for scientific research in coordination chemistry. The Schiff base metal complexes of divalent metal ions have been studied extensively since last decade as they have many potential biological and other applications. Synthesis of metal complexes have attracted attentions of inorganic, metallo-organic as well as bio-inorganic chemists across the world because of their promising applications in wide ranging areas, from material to biological sciences. Transition metal ions are essential for normal functioning of many living organisms and therefore, they are of great interest as potential drugs [42].

**Triazoles**

1,2,4-triazoles are among the large number of heterocyclic compounds, that have received the most attention during the last two decades as potential antimicrobial agents. They are associated with diverse biological activities such as fungicidal, antimicrobial, antiviral activities etc; [43-47]. The 1,2,4-triazole nucleus has been incorporated into a wide variety of therapeutically interesting drugs, including H₁/H₂ histamine receptor blockers, cholinesterase-active agents, CNS stimulants, antianxiety agents and sedatives [48]. The metal complexes of substituted 1,2,4-triazole derivatives have been extensively investigated in previous reports [49-52]. Recently, several complexes of various transition and inner transition metals with substituted 1,2,4-triazole ligands have been reported from our laboratory [53-55].

**Quinoline**

Quinoline and its derivatives are the heterocyclic organic compounds which are associated with interesting biological properties. In the recent time, quinoline nucleus has gathered an immense attention among chemists as well as biologists as it is one of the key building elements for many naturally occurring compounds.
Specially 2-chloro-3-formylquinoline is known for its numerous pharmacological applications viz; antimicrobial [56-59], antimalarial [60, 61], anti-inflammatory [62-65], antitumor [66-68], antibiotic [69], antihypertensive [70] and anti-HIV [71,72]. However, after extensive literature survey, it is noteworthy to synthesize the new transition metal complexes from Schiff bases synthesized from quinoline derivatives.

**Coumarin**

Coumarin (2H-chromen-2-one, 2H-1-benzopyran-2-one) is an oxygen heterocyclic organic compound with structurally the least complex member of large class of compounds known as benzopyrone family (Fig. 1). All the members of benzopyrones consist of a fused benzene with pyrone ring. Coumarins can be classified as simple coumarins, furanocoumarins, pyranocoumarins and pyron-substituted coumarins. These are found naturally throughout the plant kingdom in the form of their derivatives[73-75]. However, higher concentrations are found in fruits followed by roots, stems and leaves. Coumarin can be synthesized chemically also.

![α-benzopyrone and γ-benzopyrone](image)

The simple and common method used for the synthesis of coumarin is **Pechmann condensation** by using different types of phenols and β- ketoesters. 7-hydroxycoumarin, on its structural and biogenetic sense is regarded as parent coumarin among the structurally more complex coumarins. Further, coumarin molecules known to have wide spectrum of biological activities viz; antimicrobial [76], antiallergic [77] anti-inflammatory [78], antitumor [79]. Since last decade, this
coumarin and its derivatives attracted the world wide chemists and biologists due to their wide spread pharmacological properties. More ever, Coumarin derivatives have shown considerable biological activities such as lipoxygenase, cyclooxygenase inhibitors [80,81], antioxidant [82], antiplasmodial [83], anti-mutagenic [84], hypotensive [85], anthelmintic [86], inhibition of platelet aggregation [87], antineurodegenerative [88], anti HIV [89], molluscicidal [90] and bio-analytical reagents [91]. The coumarin nucleus also present in antibiotics like novobiocin, clorobiocin and coumarmycin A1 and these are active against methicillin-resistant S. aureus [92]. Biological Applications of Coumarin and its Derivatives along with their structures have shown in Table-1.

**Table-1:** Biological Applications of Coumarin and its Derivatives along with Structures

<table>
<thead>
<tr>
<th>Name and Structure of coumarin Derivatives</th>
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<tr>
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<tr>
<td><img src="image" alt="Antioxidant activity" /></td>
<td>Antioxidant activity</td>
<td>94</td>
</tr>
<tr>
<td>Compound</td>
<td>Property</td>
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<tr>
<td><img src="image1" alt="Chemical Structure" /></td>
<td>Antibacterial activity</td>
<td>95</td>
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<tr>
<td><img src="image2" alt="Chemical Structure" /></td>
<td>Fluorescence Chemosensor for Cu(II) ions</td>
<td>96</td>
</tr>
<tr>
<td><img src="image3" alt="Chemical Structure" /></td>
<td>Antitumor activity</td>
<td>97</td>
</tr>
<tr>
<td><img src="image4" alt="Chemical Structure" /></td>
<td>5-lipoxygenase inhibitors</td>
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### Introduction

<table>
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<td>Antibacterial</td>
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<tr>
<td><img src="image2.png" alt="Chemical Structure" /></td>
<td>Anti-proliferative activity against lung cancer cells</td>
<td>106</td>
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</tbody>
</table>
Thiazoles

The nitrogen and sulfur containing heterocyclic compounds are very interesting because of their wide spectrum of physicochemical properties with relevance to the design of new drug molecules.

Thiazole is a five membered active heterocyclic compound having the sulfur and nitrogen atoms in the ring system and possess the strong aromaticity, probably this is the reason for its interesting and promising biological activities [107]. It present as a structural unit in many biologically active compounds such as vitamin B1(thiamine), bacitracin, penicillin and luciferin [108], it has also got exceptional binding ability with the proteins, DNA and RNA. Thiazole and its derivatives act as a ligands with potential sulfur and nitrogen bonds are interesting and have gained special attention of chemists and biologists of all over the world not only due to their structural chemistry of multifunctional coordination modes [109] but also importance in medicinal and pharmaceutical field. Compounds having the thiazole ring system in its molecule structure known to possess numerous biological activities viz; antimicrobial, analgesic, antibacterial, anticonvulsant, antiparasitic, anti-inflammatory, herbicidal activities [110-114] and anti HIV [115].

![Thiamine](image-url)
Transition metal complexes with N,N- and N,S-donor ligands of thiazole as a central moiety have attracted remarkable attention because of their interesting chemical and biological properties. The current literature is enriched with progressive findings about the synthesis and pharmacological action of fused heterocycles of thiazole. Thiazole and derivative of thiazoles possess interesting biological activity probably conferred to them by the strong aromaticity of their ring system [116] and also very interesting class of organic heterocyclic compounds because of their wide spread applications such as antimicrobial [117], anti-inflammatory [118], anti-degenerative [119] and anti-HIV [120] activities. When diverse functional groups that interact with biological receptors are attached to this ring, compounds having outstanding properties could be obtained [121].

**Hydrazones**

Hydrazones are a special class of compounds in the Schiff base family. They are characterized by the presence of >C=N-N=C<. The presence of two interlinked nitrogen atoms separates this from imines, oximes etc; Hydrazones in general, are prepared by refluxing the stoichiometric amounts of the appropriate hydrazides and aldehydes/ketones dissolved in suitable solvents.

Compound to simple hydrazone Schiff Bases, acyl, aroyl and heteroacroyl Schiff bases have an additional donor sites >C=O. The additional donor sites make them more flexible and versatile. This versatility that has made hydrazones as good
polydentate chelating agents that can form a variety of complexes with various transition and inner transition metals and has attracted the attention of many researchers throughout the world.

Depending on the experimental conditions, different hydrazones are obtained; which have an application as biologically active compounds [122] and as the analytical reagents [123]. As biologically active compounds, hydrazones find applications in the treatment of diseases such as tuberculosis [124,125], leprosy and mental disorder [126]. Tuberculostic activity is attributed to the formation of stable chelates with the transition metal present in the cell. Thus many vital enzymatic reactions catalysed by these transition metals cannot take place in presence of hydrazones [127-129]. Many hydrazones act as insecticides, herbicides, nematocides, rodenticides and plant growth regulators. They exhibit spasmolytic activity, hypertensive action and activity against leukemia, sarcomas and other malignant neoplasm. They are also used as effective sterilants for houseflies [130]. They possess anthelmintic activity also [131]. The biological activity of hydrazones is not only limited to transition metal complexes. Recently, many hydrazones with lanthanides have shown remarkable biological activity, mainly antibacterial [132] and pharmacological activity [133].

These hydrazones are used as plasticizers and stabilizers for polymers, polymerization initiators, antioxidants etc; they act as intermediates in preparative chemistry. In analytical chemistry, hydrazones find application in detection, determination and isolation of compounds containing carbonyl group. More recently, they have been extensively used in detection and determination of several metals. They also find applications as indicators and spot test reagents [134].
The study of complexes of hydrazones is interesting from the structural point of view. It is their ability to exist keto, enol or in special cases mixed keto-enol form, depending upon the experimental conditions. Infrared spectrometry is extensively used to study these compounds. Ligands with the chromophore groups –CO-NH-N< can enter the inner sphere of the complexes either in the keto or enol form. This tendency depends on a number of factors such as pH of the medium employed in the synthesis of the complexes, the nature of the substituents attached to the carbonyl carbon atom, nitrogen atom, the anion of the metal salt used and the metal ion. If the ligand exists in keto form in the complexes, one would expect that the –NH and >C=O stretching vibrations, whereas the absence of both these frequencies is the characteristic of the enol form in the complexes. These studies in combination with $^1$H NMR data can be used to confirm the keto or enol tautomerism.

Survey of the Previous Work

The present research work embodied in the thesis is mainly deals with the synthesis of biologically important Schiff base metal complexes derived from Triazoles, Quinolines, Coumarines, Thiazoles and Hydrazones. In this context, the discussion is confined to metal complexes of above mentioned compounds and their derivatives.

The tridentate Schiff bases with heterocyclic amines containing ONS donor sequence have been tried for complexation with transition metals such as Co(II), Ni(II), Cu(II), Zn(II) and Cd(II). Among these Cu(II) complexes, the ligand shows bivalent tridentate behaviour and forms dimeric complexes, this has been substantiated by the subnormal magnetic moments and electronic spectra [135-137].
Pannu et al, [138] have been reported Mn(II), Co(II), Ni(II), Cu(II), Zn(II), Cd(II) and Hg(II) complexes of 4n-butyl-4H-1,2,4-triazole, in which ligand shows bidentate behavior in all the complexes except those of Cd(II) and Hg(II) complexes.

Garg et al., [139] have synthesized Co(II), Ni(II) and Cu(II) complexes of 5-mercapto-1,2,4-triazoles and they have assigned distorted octahedral geometry for these complexes.

Gadag and Gajendragad [140] have been prepared the nickel(II) and copper(II) complexes with 3-methyl and 3-ethyl derivatives of 4-amino-5-mercapto-1,2,4-triazole and they have high spin octahedral type configuration.

Jia-Cheng Liu and coworkers [141] have reported Cu(II) complexes with 1,2,4-triazole and end-on azido bridging ligands. They have assigned the Cu(II) ions
are strongly antiferromagnetic nature on the basis of their magnetic and crystal structures.

Zaydoun et al., [142] have been reported the Mn(II), Fe(II), Co(II), Ni(II) and Cu(II) halide complexes with 1,2,4-triazoles. Their structures are discussed on the basis of electronic and magnetic data and they have assigned the pseudo-octahedral geometry for all the complexes. The complexes are polymeric and metallic ions are bridged by triazoles and halogens. The compounds studied generally exhibit antiferromagnetic coupling.

Badiger and Patil et al., [143] have been reported the complexes of Co(II), Ni(II) and Cu(II) with 3-substituted-4-(4’-substituted-salicyldeneimino)-5-mercapto-1,2,4-triazoles and they have assigned the tetrahedral configuration for Co(II) and Ni(II) complexes on the basis of magnetic and spectral data. In case of Cu(II) complexes the ligand show tridentate behavior involving O, N, S-donor system. All the complexes are four coordinated and polymeric nature

Literature also records the complexing ability of 3-aryloxymethyl-4-aryl-5-mercapto-1,2,4-triazoles with bivalent metal ions [144]. In this case, x-ray studies reveal that, the complexes possess cubic structure and fungi toxicity of the complexes and the free ligands have been evaluated against H. Oryzae.
Bhat et al., [145] have been studied the Ag(I), Tl(I), Zn(II), Hg(II), Cd(II), Pd(II), Co(II), and Ni(II) with 4-[(4-dimethylamino-benzylidene)-amino]-5-ethyl-2,4-dihydro-[1,2,4]triazoles-3-thione and 4-[(benzylidene)amino]-6-(t-butyl)-4H-[1,2,4]-triazene-3-thione-5-one and they have been assigned the octahedral structures for the Co(II) and Ni(II), tetrahedral for the Zn(II), Hg(II), Cd(II) complexes, linear polymeric structures for the Ag(I), Tl(I) complexes and a square-planer structure for the Pd(II) complex.

Alexander Eisenwiener et al., [146] have studied the Cu(II) and Pt(II) complexes with the series of pyrazole and triazole based dinucleating ligands have been synthesized and their complexation potential for metal ions, which exhibits square planar coordination geometry has been studied. In case of Cu(II) the complexation equilibria in solution have been determined using pH titrations. Species with 1:1 stoichiometry [CuLHₙ] 2:1 stoichiometry [Cu₂LHₙ₋₁], and of dimeric nature [Cu₂L₂Hₙ], have been detected and their overall stability constants have been measured.
K. Singh et al., [147] have studied the Co(II), Ni(II) and Zn(II) complexes with Schiff bases synthesized by the reaction of p-nitrobenzaldehyde, o-nitrobenzaldehyde and p-toluylaldehyde with 4-amino-5-mercapto-1,2,4-triazole. Which are characterized by Physico-chemical studies suggest that, an octahedral geometry for the cobalt(II), nickel(II) and zinc(II) complexes. These complexes have been screened for their antibacterial activity against three Gram-positive (S. aureus, S. epidermidis and B. subtilis) and two Gram-negative (S. typhi and P. aeruginosa) bacterial strains, and results compared with the activity of the free ligands. The metal complexes were found to be more potent against one or more bacterial strains than the free ligands.

Nali et al., [148] studied a pentacoordinated Cu(II) complex, [Cu(H2O)(L1)(L2)(NO3)](NO3)(H2O) (1; L1=1,2,4-triazole-5-one, L2=1,10-
phenanthroline) has been synthesized and characterized. An X-ray structure analysis of 1 revealed that, the Cu(II) centre has a distorted square pyramidal environment with a second weakly bonded nitrate (Cu–O 2.550Å) giving rise to a pseudo-octahedral coordination geometry. Weak interactions such as π-π stacking and hydrogen bonding (especially C–H-----O hydrogen bonds) have significant effects in configuring the 3D packing of the complex.

David Olea et al., [149] have been studied Nanoprocessability of a one-dimensional oxalato-bridged cobalt(II) complex with 1,2,4-triazole. The polymer \{[Co(ox)(Htr)\textsubscript{2}]\textsubscript{2}H\textsubscript{2}O\textsubscript{n} (ox = oxalate dianion; Htr = 1,2,4-triazole) (1) has been synthesized and characterized by FT-IR spectroscopy, thermal analysis, variable-temperature magnetic measurements and X-ray diffraction methods. They studied its processability, as nano-rings, fibres and single chains which has been achieved by scission of part of the metal–ligand bonds with ultrasound and posterior re-organization of the oligomers on surface. A new feature is the observation for the first time of circular structures of a coordination polymer that is linear in the solid state. Reaction formation of this polymer at low concentration of the reactants has been also
shown as a suitable way for the isolation of nano-fibres on HOPG (Highly Oriented Pyrolytic Graphite).

A series of metal complexes of cobalt(II), nickel(II) and copper(II) have been synthesized recently G.B. Bagihalli et al., [150] with newly synthesized biologically active 1,2,4-triazole Schiff bases derived from the condensation of 3-substituted-4-amino-5-mercapto-1,2,4-triazole and 8-formyl-7-hydroxy-4-methylcoumarin, which have been characterized by elemental analyses, spectroscopic measurements (IR, UVvis, fluorescence, ESR), magnetic measurements and thermal studies. Electrochemical study of the complexes is also reported. All the complexes are soluble to limited extent in common organic solvents but soluble to a larger extent in DMF and DMSO and are non-electrolytes in DMF and DMSO. All these Schiff bases and their metal complexes have also been screened for their antibacterial (Escherichia coli, Staphylococcus aureus, Streptococcus pyogenes, Pseudomonas aeruginosa and Salmonella typhi) and antifungal activities (Aspergillus niger, Aspergillus flavus and Cladosporium) by MIC method. The brine shrimp bioassay was also carried out to study their in vitro cytotoxic properties.

B.S. Creaven et al., [151] The condensation of 4-amino-1,2,4-triazole with N-substituted-3-formyl-4-hydroxyquinolin-2-(1H)-one derivatives has lead to the synthesis of a new series of quinolin-2(1H)-one-triazole derived Schiff base ligands (1-3). Cu(II) and Zn(II) complexes (1a-3a and 1b-3b, respectively) of these ligands were also prepared. The complexes were characterised by standard techniques and for two of the complexes X-ray crystallography confirmed that, the geometry at the metal centre was octahedral in both the cases and that, the Schiff base acted as a bidentate ligand coordinating to the metal(II) ion through the deprotonated oxygen and azomethine nitrogen atoms. All of the compounds were investigated for their
antimicrobial activities against a fungal strain, Candida albicans, and against Gram-positive and Gram-negative bacteria. The compounds were found to be active against C. albicans but inactive against Staphylococcus aureus and Escherichia coli.

S.A. Patil et al., [152] A series of metal complexes ML₂ 2H₂O [MIV4Co(II), Ni(II), and Cu(II)] have been synthesized with Schiff bases derived from 3-substituted-4-amino-5-mercapto-1,2,4-triazole and 4-chloro-3-coumarinaldehyde. The structures of these metal complexes have been proposed on the basis of elemental analyses, spectral (IR, UV-Vis, FAB-mass, ESR, and fluorescence), magnetic and thermal studies. Low molar conductance values in DMF indicate that the metal complexes are non electrolytes. Cyclic voltammetric studies suggested that, the Ni(II) and Cu(II) complexes are single-electron transfer quasi-reversible. The Schiff bases and their metal complexes have been screened for in vitro antibacterial (Escherichia coli, Staphylococcus aureus, Bacillus subtilis and Salmonella typhi) and antifungal activities (Candida albicans, Cladosporium, and Aspergillus niger) by the minimum inhibitory concentration method. The Schiff base I and its Co(II) and Ni(II) complexes exhibit DNA cleavage activity on isolated DNA of S. aureus and A. niger.

Recently A. K. Singh et al., [153] synthesized the new Zn complexes by the reactions of Schiff bases derived from phenyl substituted 1,2,4-triazole and benzaldehyde, 2-hydroxyacetophenone and indoline 2,3-dione. All these metal complexes are soluble in DMF and DMSO; low molar conductance values indicate that, they are non-electrolytes. Elemental analyses suggest that, the complexes have 1:1 stoichiometry of the type [ZnL(H₂O)₂], [ZnL(OAc)₂(H₂O)₂] (L = dianionic Schiff bases derived from 3-(substituted phenyl)-4-amino-5-hydrazino-1, 2, 4-triazole and 2-hydroxyacetophenone or indoline-2,3-dione; L = neutral Schiff bases derived from 3-(substituted phenyl)-4-amino-5-hydrazino-1, 2, 4-triazole and benzaldehyde)
and they were characterized by FT-IR, $^1$H NMR, $^{13}$C NMR and FAB mass. All these Schiff bases and their metal complexes have also been screened for their antibacterial activities against Bacillus subtilis, Escherichia coli and antifungal activities against Colletotrichum falcum, Aspergillus niger, Fusarium oxysporium and Carvularia pallescence by petriplates methods.

M. Hanif and Z. H. Cohan et al., [154] Synthesized the new series of three biologically active triazole derived Schiff base ligands (L1–L3) have been synthesized in equimolar reaction of 3-amino-1H-1,2,4-triazole with pyrrol-2-carboxaldehyde, 4-bromo-thiophene- 2-carboxaldehyde and 5-iodo-2-hydroxy benzaldehyde. The prepared Schiff bases were used for further complex formation with different metal ions viz; Co(II), Ni(II), Cu(II) and Zn(II) by using a molar ratio of ligand:metal as 2:1. The structure and bonding nature of all the compounds were identified by their physical, spectral and analytical data. All the metal(II) complexes possessed an octahedral geometry except the Cu(II) complexes which showed a distorted octahedral geometry. All the synthesized compounds, were studied for their in-vitro antibacterial, and antifungal activities, against four Gram-negative (Escherichia coli, Shigella sonnei, Pseudomonas aeruginosa and Salmonella typhi) and two Gram-positive (Bacillus subtilis and Staphylococcus aureus) bacterial strains and against six fungal strains (Trichophyton longifusus, Candida albicans, Aspergillus flavus, Microsporum canis, Fusarium solani and Candida glabrata) by using agar-well diffusion method. It has been shown that, all the synthesized compounds showed moderate to significant antibacterial activity against almost all bacterial strains. In vitro Brine Shrimp bioassay was also carried out to investigate the cytotoxic properties of these compounds. The data also revealed that, the metal complexes showed better activity than the ligands due to chelation/coordination with metal ions.
G.J. Kharadi et al., [155] reported the Fe(III) complexes synthesized by the reactions of ferric nitrate with Schiff base derived from 3-substituted phenyl-4-amino-5-hydrazone-1,2,4-triazole and indoline-2,3-dione. All these complexes are soluble in DMF and DMSO; low molar conductance values indicate that, they are non-electrolytes. Elemental analyses suggest that, the synthesized new complexes have 1:1 stoichiometry of the type [FeLn(H_2O)(OH)] xH_2O. Structural and spectroscopic properties have been studied on the basis of elemental analyses, infrared spectra, ^1H and ^13C NMR spectra, electronic spectra, magnetic measurements and FAB mass spectra. FT-IR, ^1H and ^13C NMR studies reveal that, the ligand (Ln) exists in the tautomeric enol form in both the states with intramolecular hydrogen bonding. Magnetic moment and reflectance spectral studies reveal that, an octahedral geometry has been assigned to all the prepared complexes. FRAP values indicate that, all the compounds have a ferric reducing antioxidant power. The compounds 2 and 3 showed relatively high antioxidant activity while compound 1 and 4 shows poor antioxidant power. Also good antimicrobial activities of the metal complexes against Staphylococcus aureus, Bacillus subtilis, Serratia marcescens, Pseudomonas aeruginosa and Escherichia coli have been found compared to its free ligands.

Very recently M. Gaber et al., [156] reported the Manganese(II), cobalt(II), nickel(II) and copper(II) complexes of [(1H-1,2,4-triazole-3-ylimino)methyl]naphthalene-2-ol. The structure of the synthesized metal complexes have been characterized by an elemental analyses, molar conductance, magnetic moment measurements and spectral (IR, ^1H NMR, EI-mass, UV–Vis and ESR) and thermal studies. The results showed that, the chloro and nitrito Cu(II) complexes have octahedral geometry while Ni(II), Co(II) and Mn(II) complexes in addition to acetato Cu(II) complex have tetrahedral geometry. The possible structures of the metal
complexes have been computed using the molecular mechanic calculations using the hyper chem. 8.03 molecular modeling program to confirm the proposed structures. The kinetic and thermodynamic parameters of the thermal decomposition steps were calculated from the TG curves. The binding modes of the complexes with DNA have been investigated by UV-Visible absorption titration. The results showed that, the mode of binding of the complexes to DNA is intercalative or non-intercalative binding modes. Schiff base and its metal complexes have been screened for their in-vitro antimicrobial activities against Gram positive bacteria (Staphylococcus aureus), Gram negative bacteria (Escherichia coli and Pseudomonas aeruginosa), fungi (Aspergillus flavus and Mucor) and yeast (Candida albicans and Malassezia furfur).

The Cu (II) complexes, were synthesized with newly derived biologically active 1,2,4-triazole Schiff bases [157]. The Schiff bases were synthesized by condensation of 3-substituted-4-amino-5-mercapto-1,2,4-triazole with dibenzoylmethane. The synthesized compounds were characterized using elemental analyses, magnetic moment, thermal analysis and spectral tools (FT-IR, $^1$H NMR, ESR, and UV-Vis., spectroscopy). All the synthesized metal complexes are nonelectrolytes in N,N-dimethylformamide. The synthesized Schiff bases and their Cu (II) complexes have been screened for antibacterial (Escherichia coli & Staphylococcus aureus) and antifungal (Aspergillus flavus & Candida albicans) activity using a modified Bauer-Kirby method. Interestingly, the synthesized Cu (II) complexes were used as precursors for CuO nanoparticles which were characterized using XRD, HR-TEM, FT-IR and UV–Vis spectroscopy. The photocatalytic activity of the prepared CuO nanoparticles was studied by performing the degradation of methylene blue dye under UV illumination in the presence of $\text{H}_2\text{O}_2$ and the results
showed that, the maximum percent of the degradation of methylene blue dye (MB) was found 96.18% after 360 min.

P. Tyagi et al., [158] synthesized a series of two biologically active Schiff base ligands $L^1$, $L^2$ have been synthesized in equimolar reaction of 4-amino-5-(pyridin-4-yl)-4H-1,2,4-triazole-3-thiol with thiophene-2-carbaldehyde and furan-2-carbaldehyde. The synthesized Schiff bases were used for complexation with different metal ions like Co(II), Ni(II) and Cu(II) by using a molar ratio of ligand: metal as 1:1 and 2:1. The characterization of Schiff bases and their metal complexes was done by $^1$H NMR, UV–Vis, TGA, IR, mass spectrometry and molar conductivity studies. In DFT studies, the geometries of Schiff bases and metal complexes were fully optimized with respect to the energy using the 6-31+g(d, p) basis set. On the basis of the spectral studies an octahedral geometry has been assigned for Co(II), Ni(II) and Cu(II) complexes. The effect of these complexes on proliferation of human breast cancer cell line (MCF-7) and human hepatocellular liver carcinoma cell line (Hep-G2) were studied and compared with those of free ligands. The anticancer cell line results reveal that all metal complexes show moderate to significant % cytotoxicity on cell line HepG2 and MCF-7.

A.-N.M.A. Alaghaz et al., [159] reported the complexes of manganese(II), cobalt(II), nickel(II), copper(II) and zinc(II) of general composition $[M(L)_2]$ have been synthesized $[L = 4$-pyridin-2-yl-methylene amino-4H-1,2,4-triazole-3-thiol$. The elemental analyses, molar conductance, spectral (IR, UV-Vis., $^1$H NMR, mass), magnetic moment and thermal measurements studies of the compounds led to the conclusion that, the ligand acts in a tridentate manner (SNN). The molar conductance of the metal complexes in fresh solution of DMSO lies in the range of 8.34-10.46 Ω$^{-1}$ cm$^2$ mol$^{-1}$ indicating their non-electrolytic behavior. On the basis of analytical and
spectroscopic techniques, octahedral geometry of the complexes was proposed. The Schiff base acts as tridentate ligand coordinated through deprotonated thiolic sulfur, azomethine nitrogen and pyridine nitrogen atoms. The ligand field parameters were calculated for Co(II), Ni(II) and Cu(II) complexes and their values were found in the range reported for a octahedral structure. The data show that, the complexes have composition of ML₂ type. The activation of thermodynamic parameters are calculated using Coast–Redfern, Horowitz–Metzger (HM), Piloyan–Novikova (PN) and Broido’s equations. Protonation constants of Schiff base and stability constants of their binary metal complexes have been determined potentiometrically in 50% DMSO–water media at 25 °C and ionic strength 0.10 M potassium nitrate. Both the Schiff’s base ligand and its complexes have been screened for their antibacterial activities.

P. P. Hankare et al., [160] have studied the Mn(II), Co(II), Ni(II), Cu(II) and Zn(II) complexes with azo coumarins. In which, the ligands act as natural bidentate and their metal(II) complexes appear to have the octahedral geometry. The metal complexes are characterized by elemental analyses, magnetic, molar conductance, TGA and spectral studies. The complexes have [M(L)₂(H₂O)₂]. X H₂O where ‘L’ is a deprotonated ligand.
B.S. Jayashree et al., studied that some of the coumarin Schiff bases have successfully shown the analgesic and anti-inflammatory activity [161]. It was known that, halogentaed coumarin derivatives have shown good antibacterial activity [162].

A.I. Mosa et al., [163] synthesized the novel Schiff base using 3-formyl-4-hydroxy coumarin with thiocarbohydrazide in the molar ratio 1:1 to synthesize the new series of Cu(II), Ni(II), Co(III), Cr(III) and Fe(III) complexes. These complexes were stable and having a geometry of the type M(L)X•S, where M = Cu(II), Ni(II), Co(III), Cr(III) and Fe(III) and ‘L’ is a deprotonated ligand of 4-hydroxy-coumarin-3-thiocarbohydrazone, X = Cl\(^-\), NO\(_3\)^- or CH\(_3\)COO\(^-\) and S = H\(_2\)O. All the synthesized compounds were characterized by elemental analyses, molar conductance, magnetic measurements, thermal gravimetric analysis (TGA) and various spectroscopic techniques. The ligand(HL) acts as a monobasic tridentate in all metal complexes and is coordinated through the phenolic -OH, azomethine nitrogen and thione sulphur atoms. The electronic absorption assignments with magnetic susceptibility results suggested the variety of geometries around the central metal atoms. Pharmacodynamic activity of Co(III) complex on some biochemical parameters and histological studies in serum and heart tissue in rats have been studied. In this evaluation, ligands express good results at both high and low concentrations but metal complexes are more active at low concentrations only. The antifungal activity against filamentous fungi Aspergillus niger, Aspergillus fumigatus and Aspergillus flavus. The antibacterial activity tested against the Candida albicans, Escherichia coli, Klebseilla pneumoniae and Pseudomonas aeruginosa.

Latika Dawara and R.V. Singh have reported [164] microwave-assisted synthesis of Bi(III) and Sb(III) complexes with ligands formed by the reactions of 3-acetylcoumarin with thiosemicarbazide / semicarbazide hydrochloride. The structural
confirmation of the synthesized compounds was established with elemental analyses, molecular weights, IR, $^1$H-NMR, UV, mass spectral and X-ray powder diffraction studies. The ligands coordinated to metal ion in a monobasic bidentate manner. The pesticidal activities of synthesized compounds were studied against Coreyra cephalonica. Antimicrobial activity of the synthesized compounds was also carried out.

Puja Kapoor et al., [166] have synthesized the La(III) complexes from 3-formyl-4-chlorocoumarin with hydrazinecarbothioamide / hydrazinecarboxamide. The isolated compounds are coloured solids, soluble in DMSO, DMF and methanol. Characterization of all the synthesized compounds were studied by elemental analyses, molar conductance, IR, 1H-NMR, molecular weight, electronic, EPR, UV spectral and X-ray powder diffraction studies. The spectral data suggest that, Schiff bases are monobasic bidentate, coordinating through nitrogen and sulphur / oxygen atoms. The metal complexes are monomeric and non-electrolytic in nature. The antimicrobial, DNA cleavage, pesticidal and nematicidal activities were also studied [165].

The synthesis and biological importance of Co(II), Ni(II) and Cu(II) metal complexes of triazole Schiff bases [166] from our laboratory. The Schiff bases have been derived from 3-substituted-4-amino-5-hydrazino-1,2,4-triazole and 8-formyl-7-hydroxy-4-methylcoumarin. The metal complexes were characterized by elemental analyses, spectral (IR, UV-visible, ESR, FAB-mass) electrochemical, magnetic and thermal data. The ligands and their Co(II), Ni(II) and Cu(II) complexes are studied against bacterial (E. coli, S. aureus, S. pyogenes and P. aeruginosa) and fungal (A. niger, A. flavus and cladosporium) cultures for antimicrobial properties by MIC
method and also the brine shrimp bioassay was carried out to investigate their in-vitro cytotoxic properties.

\[
\text{M} = \text{Co(II), Ni(II) and Cu(II)}. \\
\text{R} = \text{H, CH}_3, \text{C}_2\text{H}_5 \text{ and C}_3\text{H}_7.
\]

Patil S.A. et al., [168] have synthesized a series of Co(II), Ni(II) and Cu(II) complexes with new Schiff bases derived from 8-formyl-7-hydroxy-4-methylcoumarin / 8-acetyl-7-hydroxy-4-methylcoumarin and 1,8-diaminonaphthalene. ML.2H\text{2O} type of metal complexes has been proposed in the light of analytical, spectral (IR, NMR, UV-Vis, ESR, and FAB-mass), magnetic, and thermal studies. The redox behaviour of the complexes was investigated by electrochemical method using cyclic voltammetry. The Schiff bases and their metal complexes were screened for their antibacterial (Escherichia coli, Staphylococcus aureus, Pseudomonas aeruginosa, and Salmonella typhi) and antifungal activities (Aspergillus niger, Aspergillus flavus, and Cladosporium) by Minimum Inhibitory Concentration method. The results of these studies show that, metal complexes are
more effective as antibacterial and antifungal agents than the parent ligands. DNA cleavage activity was also studied by using Calf-thymus DNA [167].

Taha et al.,[168] reported the synthesis of Ln(III) complexes of Schiff base; bis-(salicylaldehyde)-1,3-propylenediamine and were screened for their antibacterial activity. The synthesized compounds were characterized based on elemental analyses, TGA, conductivity measurements, luminescence and spectral studies (FT-IR, $^1$H NMR, UV-Vis., etc). The synthesized Schiff bases act as tetra dentate ligands and form stable complexes with the metal ions. Most of the tested Ln(III) complexes possessed a high antibacterial activity against the different pathogenic bacteria.

B.S. Creaven et al., [169] have reported a series of Schiff bases using 7-amino-4-methylcoumarin with a number of substituted salicylaldehydes in good yields. Subsequent reaction of these ligands with copper(II) acetate yielded Cu(II) complexes and some were characterised using X-ray crystallography. Anti-candida activity was carried out for all the synthesized compounds.

Kulkarni et al., also reported a series of Co(II), Ni(II), and Cu(II) complexes of Schiff bases derived from 8-formyl-7-hydroxy-4-methylcoumarin and o-phenylenediamine / ethylenediamine [171]. Structure of these metal complexes has been proposed in the light of analytical, spectral (IR, UV-vis., ESR and FAB-mass),
magnetic and thermal studies. The metal complexes were soluble in DMF and DMSO. Non-electrolytic nature of the metal complexes was confirmed by molar conductance data. Redox behavior of the metal complexes was investigated by electrochemical method using cyclic voltammetry. The Schiff bases and their metal complexes have been screened for their antibacterial (Escherichia coli, Staphylococcus aureus, Pseudomonas aeruginosa and Salmonella typhi) and antifungal activities (Aspergillus niger, Aspergillus flavus and cladosporium) by minimum inhibitory concentration method. The DNA cleavage activities of Co(II) and Cu(II) complexes were studied against DNA of E. coli and A. niger.

\[ \text{M} = \text{Co(II), Ni(II) and Cu(II)}. \]

Recently, K. Mahendra Raj et al., [171] have synthesized the Co(II), Ni(II), Cu(II) and Zn(II) complexes with Schiff base 3-chloro-N’-((7-hydroxy-4-methyl-2-oxo-2H-chromen-8-yl)methylene) benzo[b] thiophene-2-carbohydrazide. Synthesized compounds were characterized by elemental analyses and various physico-chemical techniques like, IR, $^1$H NMR, ESI-mass, UV-Visible, TG-DTA, magnetic measurements and molar conductance. Spectral studies suggest the octahedral geometry for all the metal complexes. The bonding sites are the oxygen atom of
amide carbonyl, nitrogen of azomethine and phenolic oxygen of the ligand via deprotonation. TG-DTA analyses confirms the presence of coordinated water molecules in the Cu(II) complex, further was supported by IR measurements. In order to evaluate the effect of antimicrobial potency of metal ions upon chelation, ligand and its metal complexes were screened for their antibacterial and antifungal activities by Minimum Inhibitory Concentration (MIC) method. The results showed that, metal complexes were found to be more active than their parent ligand. All the synthesized compounds were screened for their free radical scavenging activity by DPPH method and DNA cleavage activity using Calf-thymus DNA.

Very recently, Patil S. A.et al., have [172] synthesized the Schiff bases derived from 6-formyl-7,8-dihydroxy-4-methylcoumarin with o-toluidine/3-aminobenzotrifluoride and their Co(II), Ni(II) and Cu(II) metal complexes. The Schiff bases and their metal complexes have been characterized by various physico-chemical techniques. The study reveals that, the synthesized metal complexes have octahedral geometry. Further, the Schiff base and metal complexes were screened for their antimicrobial, anthelmintic and DNA cleavage activity.

**Aim and Significance of the Present Study**

The applications of coordination compounds are of great importance. Transition metal chemistry plays a major role in different fields viz biological systems, medicine, industry and analytical chemistry. Metal complexes affect most of the chemical reactions, including catalysis and models for different biological processes. Ancient civilizations used gold and copper for healing purposes. Multidentate ligands are providing greater coordination stability compared with monodentate analogues due to the chelating effect. This chelating effect can be
maximized, if number and size of chelating rings are maximized for the size of cations in such a way that minimizes steric hindrance caused after metal binding [173]. In inorganic chemistry (coordination compounds) should therefore not be overlooked in the field of chemical biology, since their distinctive electronic, chemical and photophysical properties render them particularly useful for a variety of applications [174,175]. In cell biology, chelating agents have played major role and those are used to reduce the concentration of toxic metal ions in physiological system. Further, it is also desirable to manipulate cellular metal ions by increasing their concentration in a controlled manner [176].

The reactivity of metal complexes is an important section of coordination chemistry [177-180]. Reactions of coordinated inorganic and organic ligands are of the high interest including reactions on coordinative-unsaturated donor centers [181-184] or metal complex self-assembling reactions leading to various supramolecular structures [185].

In accordance with the design of coordination architectures based on heterocyclic organic ligands and metal centers represent one of the most rapidly developing fields in present coordination chemistry owing to their potential as functional materials [186-188]. From the literature, it is well known that, not much work has been done on synthesis, characterization and biologically important Schiff bases and their coordination compounds of divalent metal complexes of Schiff bases derived from coumarin derivatives, quinoline, triazole, acid hydrazones, thiazole molecules etc.;

In view of the above facts, the author has undertaken a comprehensive research work on the synthesis of some metal complexes of various bidentate and
tridentate Schiff bases. The aim of present research work is to synthesize some Schiff bases derived from formyl quinoline, formyl hydroxyl/dihydroxy/halogenated coumarins and triazole, thiazole, acid hydrazide etc., then to stitch these newly synthesized Schiff bases with various bivalent metal ions to form stable metal complexes. In the light of the literature reports, no work on the present research work undertaken, so we have synthesized, characterized and screened for their various biological properties and compiled in this thesis.

The literature survey shows the utilization of various substituted coumarin derivatives as ligands for the synthesis of transition metal complexes [189] and fewer attempts have been made to synthesize metal complexes of Schiff bases derived from 1,2,4-triazole, formyl quinoline, thiazole, formyl hydroxy/dihydroxy/halogenated coumarins. The work described in this thesis, attempts to explore the range of Schiff-base ligands and also to prepare their cobalt(II), nickel(II), copper(II) and Zn(II) complexes.

All the newly synthesized Schiff bases and their bivalent metal complexes were characterized by extensive spectroscopic data and various analytical methods. The structural properties of Schiff bases have been studied by FT-IR, GCMS, $^1$H NMR, $^{13}$C NMR, UV-visible ESI-MS, and elemental analyses. Owing to the fluorescence properties of compounds, some of the synthesized ligands and their metal complexes have been evaluated for their fluorescence properties.

X-ray crystallographic studies are the most definite source of information regarding the structure of any metal complex. Due to the difficulty in solubility of synthesized metal complexes in common organic solvents such as methanol, ethanol, hexane, acetone, ethyl acetate etc., the attempts made to obtain the single crystals of
the synthesized metal complexes were failed due to their poor solubility in common organic solvents. Hence, in present context this method was less suitable for these complexes. In such cases, a variety of other techniques can be used for the through characterization of the newly synthesized Schiff bases and their metal complexes and also have been done in this present investigation. The Structural diversities of bivalent metal complexes have been undoubtedly proposed in the light of FT-IR, ESI-MS, EI-MS, LCMS, UV-visible, Fluorescence and ESR spectral data wherever possible or applicable, elemental analyses, molar conductance measurements, magnetic measurements and thermogravimetric studies. The redox behavior of all the some of the synthesized Cu(II) complexes have been studied by cyclic voltammetry and electron transfer mechanisms were proposed by these studies. Thermal decomposition studies of some metal complexes provided few valuable information regarding the thermal stabilities of the synthesized metal complexes.

With respect to the significant applications of Schiff bases and their coordination compounds in medicinal field, appropriately termed as “Medicinal Inorganic Chemistry”, Schiff bases and their bivalent metal complexes have been screened for their antituberculosis, antibacterial, antifungal and anthelmintic activities. Also some of the metal complexes have been tested for their DNA cleavage and binding properties using Calf-Thymus DNA.

The main purpose of present research work is to synthesize some new Schiff bases of biological importance and subsequently utilize them as ligands and also to study the ligands and their metal complexes from a structural point of view. All the synthesized ligands with a variety of bonding interactions, potential variety of bonding modes and hydrogen bonding interactions, were expected to provide stimulating results. This expectation became fruitful. Metal chelates of Schiff bases
hold exciting possibilities for the future concerning to their wide applications viz. in designing new catalytic systems, in formulating new synthetic routes, in developing new analytical reagents and in metal based antimicrobial agents etc. Hopefully, the results of this investigation would attract increased interest in \textbf{Coordination Chemistry} field.

\textbf{Schiff Bases Synthesized in the Present Investigation}

\textbf{Scheme-I}

Synthesis of Schiff bases derived from 3-Substituted-4-amino-5-mercapto-1,2,4-triazole and 2-Chloro-3-formylquinoline

\begin{center}
\begin{tikzpicture}
\node [align=center] (A) at (0,0) {
\begin{minipage}{0.45\textwidth}
3-substituted-4-amino-5-mercapto-1,2,4-triazole \hspace{1cm} 2-Chloro-3-formylquinoline
\end{minipage}
\begin{minipage}{0.45\textwidth}
3-substituted-4-amino-5-mercapto-1,2,4-triazole
\end{minipage}};
\node [align=center] (B) at (1,1) {
\begin{minipage}{0.45\textwidth}
2-Chloro-3-formylquinoline
\end{minipage}
\begin{minipage}{0.45\textwidth}
3-substituted-4-amino-5-mercapto-1,2,4-triazole
\end{minipage}};
\node (C) at (0.5,2) {Acetic acid};
\node (D) at (0.5,3) {Stirred 3-4 Hrs};
\node (E) at (0.5,4) {Schiff Base I \& II};
\end{tikzpicture}
\end{center}
Scheme-II
Synthesis of Schiff base derived from 8-Formyl-7-hydroxy-4-methylcoumarin and 2-Hydrazino-4-phenylthiazole

Scheme-III
Synthesis of Schiff base derived from 8-Formyl-7-hydroxy-4-methylcoumarin and 2-Hydrazino-4-(coumarin-3-yl)thiazole
**Scheme-IV**

Synthesis of Schiff bases derived from the 6-Formyl-7,8-dihydroxy-4-methylcoumarin and Benzhydrazide/ Isonicotinichydrazide

![Reaction Scheme IV](image)

**Scheme-V**

Synthesis of Schiff bases derived from 3-Chloro-8-formyl-7-hydroxy-4-methylcoummarin and Benzhydrazide/ Nicotinichydrazide

![Reaction Scheme V](image)
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Introduction


