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First-Row Transition Metal Complexes with Schiff Base Ligands: A Review on Recent Advancements

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Abstract : Schiff base ligands exhibit broad range of application in medicine, pharmacy, coordination chemistry, biological activities, industries, food packages, dyes and polymer and also used as an O_2 detector. In recent years, the researchers have attracted enormous attention toward Schiff bases and their metal complexes owing to numerous applications in pharmacology such as antiviral, antifungal, antimicrobial, antimalarial, antituberculosis, anticancer, anti-HIV, catalytic application in oxidation of organic compounds, and nanotechnology. The unique coordination behaviour of these ligands, coupled with the diverse electronic and structural properties of transition metals, makes them promising candidates for various industrial and biomedical applications. Schiff base complexes with first-series transition metals have been widely recognized due to their versatile applications in catalysis, bioactivity, and material sciences. This review studies the recent advancements in the synthesis, structural characterization, and functional applications of Schiff base complexes with first-row transition metals. Particular focus is given to their catalytic performance in oxidation-reduction reactions, highlighting their significance in sustainable catalysis. In addition, their biological activities, including antimicrobial, anticancer, and antioxidant properties, are thoroughly analyzed, with an emphasis on structure-activity relationships. Despite their potential, challenges such as solubility, stability, and selectivity persist, necessitating further research. Future perspectives are discussed, emphasizing innovations innovel mechanistic approaches to enhance functionality. By consolidating recent findings, this review aims to provide a strong foundation for future research and technological advancements in Schiff base transition metal chemistry.

(Keywords : Schiff base complex, transition metal, catalysis, biological activity, Coordination chemistry).

Introduction

Schiff base, first characterized and introduced by Hugo Schiff (a German Scientist and Novel Laureate) almost 160 years ago, played a pivotal role in coordination chemistry since their discovery, offering a versatile framework for metal complexation[1]. Structurally, a Schiff base (imine or azomethine) is a ketone or aldehyde analogue where the carbonyl group (-C=O) is replaced by an imine (-C=N-) group (fig.1). The ability of these ligands to stabilize transition metals in various oxidation states has made them indispensable in catalysis[2], pharmaceutical[3,4], and material science[5]. First-row transition metals, including manganese, iron, cobalt, nickel, copper, and zinc, exhibit unique redox properties, making them ideal candidates for catalytic and biological applications. The coordination chemistry of Schiff bases with these metals enables the fine-tuning of electronic properties, influencing their catalytic efficiency and bioactivity.

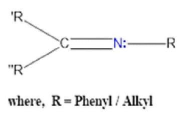


Fig 1: Schiff base

Recent studies have demonstrated that Schiff base-metal complexes can function as efficient catalysts in organic synthesis, including oxidation, reduction, and cross-coupling reactions. Their role in bioinorganic chemistry has also expanded significantly, with emerging evidence supporting their potential as antimicrobial, anticancer, and antioxidant agents. Understanding the relationship between ligand design, metal coordination, and functional properties is crucial for advancing these complexes in real-world applications.

This review aims to provide a critical evaluation of recent advancements in Schiff base complexes with first-row transition metals, focusing on their synthesis, catalytic applications, bioactivity, and structural insights. By addressing current challenges and future research directions, this work seeks to underscore the transformative potential of Schiff base-metal chemistry in modern scientific and industrial domains.

1. Synthesis and Characterization of Schiff Base Complexes

Schiff base ligands are synthesized from the condensation reaction of aromatic/aliphatic aldehydes and amines and form stable complexes with various transition metal as central ions[6-8]. The process generally involves mixing equimolar amounts of an amine and an aldehyde or ketone in an appropriate solvent, such as ethanol or methanol, under reflux conditions. Schiff bases and their metal complexes are promising for synthetic and structural research due to their easy synthesis, structural diversity, and broad chemical applications[9-12]. Schiff bases are usually formed by condensation of an aldehyde or ketone with a primary amine in various solvents, typically methanol or ethanol, under room temperature or reflux conditions as shown in (fig. 2). Dehydrating agents like magnesium sulfate enhance formation, while water removal via a Dean-Stark apparatus aids into tolune or benzene. Though generally stable, Schiff bases may degrade during purification, making crystallization preferable to chromatography. Their structural versatility allows for the design of mono-, di-, tri-, and multi-dentate chelating ligands tailored to metal ion binding.

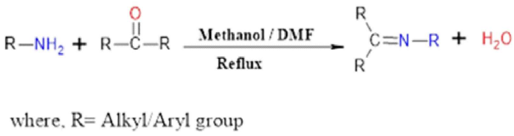


Fig 2: Formation of Schiff base by condensation of reaction

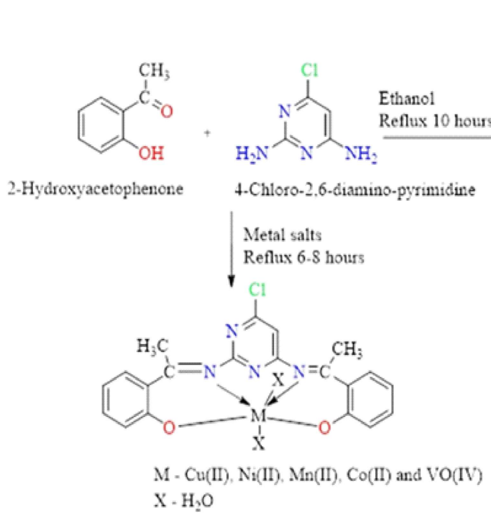
The coordination chemistry of Schiff base ligands with first-row transition metals (e.g., Mn, Fe, Co, Ni, Cu, and Zn) is an essential area of study due to their potential applications. Metal complexation typically occurs via the lone pair on the nitrogen of the imine group and additional donor atoms, such as oxygen from hydroxyl or carboxyl groups, within the ligand framework. The preparation of Schiff bases and their complexes can be carried out by the following methods:

(a) In Situ Metal Complexation : This method is widely used in coordination chemistry due to its efficiency, simplicity, and applicability to a broad range of transition metals. Schiff base ligands are synthesized and complexed with metal salts in a single-pot reaction[13]. The metal salt and precursor molecules (amine and carbonyl compound) react in the same reaction condition under reflux or at room temperature, depending on the nature of the ligand and metal. The presence of metal ions influences the formation of Schiff base ligands, often directing the final geometry of the complex. Polar solvents are commonly used to facilitate solubility and reaction kinetics. Since the ligand and metal interact simultaneously, unwanted side reactions are minimized, leading to higher product yield.

(b) Pre-formed Ligand Complexation : The Schiff base is synthesized separately and then mixed with metal salts in an appropriate solvent under controlled conditions (reflux) to form the desired complex[14].

(c) Template Synthesis: In this method, complexes are synthesized in a single-step reaction by directly reacting the aldehyde, amine, and metal compound without isolating the Schiff base. Template processes are classified as thermodynamic and kinetic. In thermodynamic processes, the template binds to a reactant, shifting the equilibrium toward product formation. In kinetic processes, templates stabilize transition states under irreversible conditions, ensuring selective product formation. In many kinetically controlled reactions, the template remains strongly bound to the final product, functioning as both a kinetic and thermodynamic template. Distinguishing between kinetic and thermodynamic control in template reactions is often challenging in practice.

Sakthilatha and Rajavel (2013)[15] synthesized a novel class tetradentate Schiff base ligand 2,2'-(1E,1'E)-1,1'-(6-chloropyrimidine-2,4-diyl) bis(azan-1-yl-1-ylidene) bis(ethan-1-yl-1-ylidene) diphenol (L) by the reaction of 2-hydroxy acetophenone and 4-chloro-2,6-diamino-pyrimidine. The structure was elucidated by elemental analysis, IR, ^{1}H-NMR and electronic spectra. The complexes were synthesized by the template effect of 2-Hydroxy acetophenone, 4-chloro-2,6- diaminopyrimidine and $(CH_3COO)_2Cu.H_2O$, $(CH_3COO)_2Ni.4H_2O$, $(CH_3COO)_2Co.4H_2O$, $(CH_3COO)2Mn.4H_2O$ and $VOSO_4.5H_2O$ respectively.



Scheme 1 : Synthesis of ligand and Schiff base metal complexes

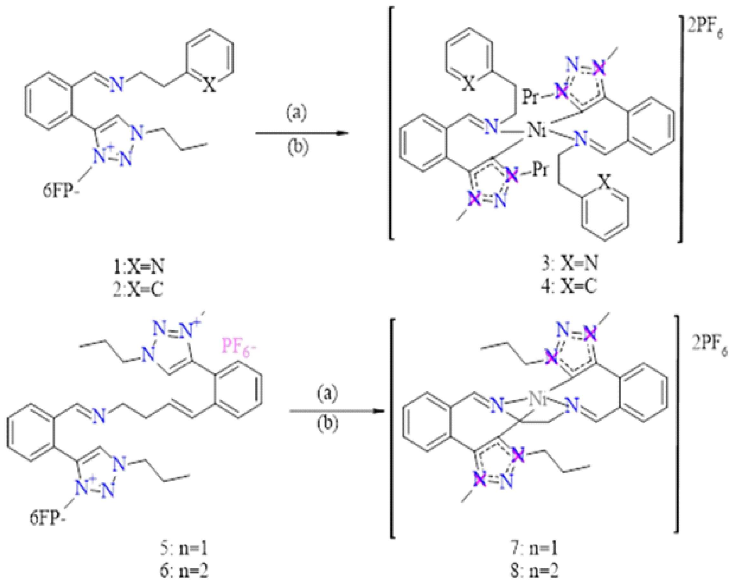
(d) **Solvent-Assisted Complexation:** In Solvent-assisted complexation, coordinating solvents, such as dimethyl formamide (DMF) or dimethyl sulfoxide (DMSO) is used to stabilize the intermediates during metal complex formation. These solvents have lone pairs on their heteroatoms (e.g., oxygen or nitrogen) that can interact with metal ions, and facilitate ligand exchange and coordination. This approach enhances solubility, prevents unwanted side reactions, and promotes the efficient formation of metal-ligand complexes. The choice of solvent significantly influences the stability, better yield, and structural characteristics of the product.

Complexation conditions, such as pH, temperature, and ligand-to-metal ratio, significantly influence the coordinating behaviour and stability of the complexes. The structural elucidation of Schiff base ligands and their metal complexes involves several spectroscopic and analytical techniques includes FTIR Spectroscopy which identifies characteristic vibrational modes, particularly the imine (-C=N-) stretching frequency, and confirms metal coordination through shifts in absorption bands[16]. ^{1}H and ^{13}C NMR provide insights into ligand backbone structure and electronic environment changes upon complexation[17]. UV-Vis Spectrophotometer analyzes electronic transitions in the ligand and metal complex which is useful for confirming coordination geometry[18]. XRD provides definitive structural information, including bond lengths, angles, and coordination environment[19]. Mass Spectrometry is used to determines molecular weight and fragmentation patterns, aiding in structural confirmation[20]. TGA/DSC evaluates thermal stability and decomposition pathways of ligands and complexes[21]. EPR Spectroscopy is used for paramagnetic metal complexes like Cu(II) and Mn(II) to studyelectronic structures and bonding characteristics[22].

2. Catalytic Applications of Schiff Base Complexes

The coordination environment in Schiff base metal complexes can be tailored by introducing various substituents to the ligand. This modification helps regulate steric and electronic properties, making it possible to fine-tune the structure and reactivity. Schiff bases can form metal complexes with first series transitions metals, which are recognized for their effectiveness as catalysts in various synthetic and chemical reactions[23-25]. **Wang et al. (1999)** documented the successful oxidation of olefins facilitated by Mn(II) amino acid Schiff base complexes[26]. Recently, both heterogeneous and homogeneous catalysts have gained significant attention from chemists due to their improved selectivity and recyclability.The number of studies on catalysis using supported Schiff base complexes has grown exponentially in recent years. However, homogeneous catalysis remains particularly relevant as it allows for a clear understanding of reaction mechanisms.

Lawal et al. (2022)[27] investigated the oxidation of styrene to benzaldehyde using Schiff base functionalized1, 2,3 triazolylidene nickel (II)



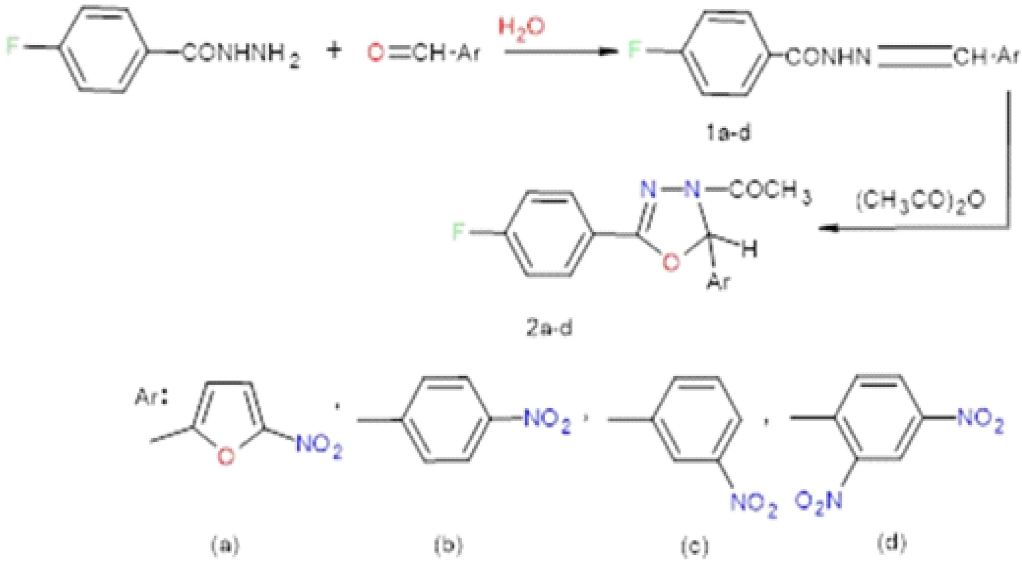
Scheme 2. Synthesis of Schiff base functionalised trizolylidene Ni(II) complexes; (a) Ag_2O, DMC, N_2 Stir, RT. 4h (b) Ni(diglyme) Cl_2, MeOH, N_2, Stir, RT, 12h, (i) functionalized mono carbene ligand frame (ii) functionalized dicarbene ligand frame.

(Ni(II) complexes as catalysts. Four novel Ni(II) complexes were synthesized via a trans-metalation route using silver oxide, followed by coordination with Ni(diglyme)Cl_2 and characterized using various spectroscopic methods viz. nuclear magnetic resonance (NMR), infrared (IR), and mass spectrometry analyses. The oxidation reaction was performed using acetonitrile as the solvent at 80°C, with H_2O_2 as the oxidant. Among the tested catalysts, complex 3 exhibited the highest conversion rate (88%) with a 70% selectivity towards benzaldehyde. Key reaction parameters were optimized and found that a 1 mol% catalyst concentration and a 1:5 ratio of styrene to H_2O_2 provided the best results. The kinetic study revealed a first-order dependence on both catalyst and oxidant concentrations with activation energy equal to 65 ± 3 kJ/mol.

Schiff base complexes with first-row transition metals are emerging as efficient catalysts for green and sustainable chemistry due to their versatility, stability, and ability to facilitate diverse reactions. They play a crucial role in oxidation, polymerization, and hydrogenation, utilizing eco-friendly oxidants like H_2O_2 and operating under mild conditions. Recent advancements focus on water-soluble ligands, solvent-free synthesis, and minimizing hazardous byproducts.

1. Bioactivity of Schiff Base Complexes

Schiff base metal complexes of first-series transition metals exhibit potent antimicrobial, antifungal, anticancer, anti-inflammatory and antioxidant activities by interacting with biomolecules and disrupting cellular processes. Their metal coordination enhances bioavailability, redox modulation, and enzyme inhibition, making them effective therapeutic agents. Notably, Cu(II) and Ni(II) complexes has antibacterial and antifungal properties and it induce apoptosis and thus

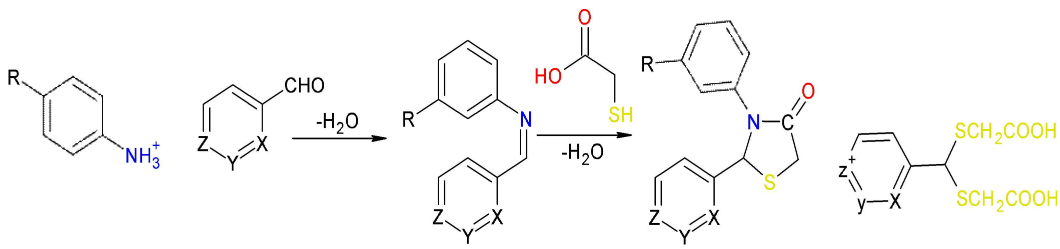


Scheme 3. Scheme of Synthesis a series of hydrazide hydrazones.

shows anticancer properties. Zn(II) and Co(II) complexes show strong anti-inflammatory and antiviral properties. These complexes hold significant potential for drug development due to their stability, selectivity, and pharmacological efficacy.

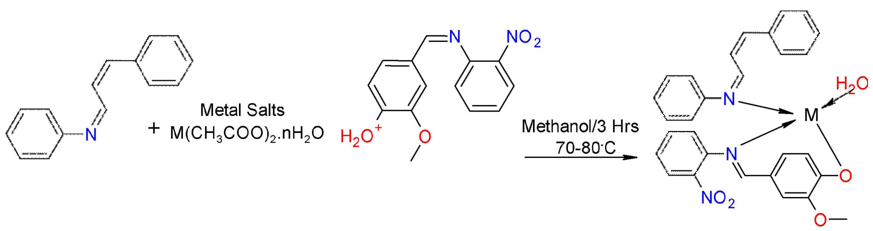
Sevim and colleagues (2002) synthesized and analyzed a series of hydrazide-hydrazones and 1,3,4-oxadiazolines derived from 4-fluorobenzoic acid and hydrazide**(Scheme 3)** as potential antimicrobial agents. These compounds were evaluated for their antibacterial and antifungal properties against Staphylococcus aureus, Escherichia coli, Pseudomonas aeruginosa, and Candida albicans. Among them, 4-fluorobenzoic acid [(5-nitro-2-furanyl) methylene] hydrazide (1a) exhibited antimicrobial activity comparable to ceftriaxone against Staphyl ococcus aureus[28].

Vazzana et.al (2004) prepared two small groups of aromatic Schiff bases and 2,3-diaryl-1,3-thiazolidin-4 one derivatives (Scheme 4), and evaluated for their anti inflammatory and antinociceptive properties. The thiazolidinone derivatives were derived from azomethines through a reaction with α-mercaptoacetic acid. Both categories of compounds demonstrated significant effectiveness in reducing carrageenan -induced edema in the hind paws of rats, whereas their activity in the writhing test conducted on mice was moderate[29].



Scheme 4. Aromatic Schiff bases and 2,3-diaryl-1,3-thiazolidin-4-one

Karthik et al. (2024)[30] synthesized Schiff base metal (II) complexes include Co(II), Ni(II), Cu(II), and Zn(II) metal centers derived from cinnamaldehyde-aniline (L1) and 4- hydroxy-3-methoxybenzaldehyde-o-nitroaniline (L2) (Scheme 5). Among them, the anticancer properties of Schiff base ligands (L1, L2) and Cu(II) complex were assessed against MCF-7 human breast cancer cells using the MTT assay. It exhibited the highest cytotoxicity, with an IC50 value of 24.05 ìg/mL, significantly lower than the ligands (L1: 43.51 ìg/mL, L2: 34.18 ìg/mL), indicating enhanced anticancer potency. The enhanced activity is due to chelation, which increases lipophilicity, and triggers apoptosis in cancer cells.



Scheme 5. Synthesis of Schiff base mixed ligand complexes

1. Structural and Electronic Insights

Schiff bases are a class of compounds defined by the presence of an imine (-C=N-) or azomethine (-N=N-) linkage, typically formed through the condensation of a primary amine and a carbonyl compound. These compounds have garnered significant attention across various fields, including organic synthesis, coordination chemistry, materials science, and medicinal chemistry, owing to their versatile structural and electronic properties.

Structural Insights

1. Imine Linkage and Tautomerism

Schiff bases are primarily characterized by their imine (-C=N-) functional group, which plays a crucial role in their structural and electronic properties. This imine linkage is highly susceptible to tautomerism, where the compounds can exist in either the imine or enamine form, depending on environmental conditions. Such tautomeric shifts significantly influence the compound's reactivity and stability[31].

2. Keto-Enol Tautomerism

Beyond the imine linkage, Schiff bases often exhibit keto-enol tautomerism, particularly when conjugated with carbonyl groups. This tautomerism has a profound impact on the photophysical and electronic properties of these compounds. For instance, the keto form is often associated with enhanced emission characteristics, making Schiff bases promising candidates for optoelectronic applications[31,32].

3. Crystallographic Studies

Single-crystal X-ray diffraction studies have been instrumental in elucidating the structural parameters of Schiff bases. These studies reveal that Schiff bases frequently adopt planar or near-planar geometries, stabilized by intramolecular hydrogen bonding and aromaticity. This structural planarity is vital for electronic delocalization and optical properties, enhancing their potential for various electronic applications[32,33].

4. Substituent Effects

The electronic and structural properties of Schiff bases can be finely tuned by modifying the substituents attached to the molecular framework. Electron-donating or electron-withdrawing groups significantly influence charge distribution, molecular orbitals, and optical properties. This tunability allows Schiff bases to be customized for specific applications, including catalysis, sensing, and electronic devices[31].

Electronic Insights

1. Excited-State Intramolecular Proton Transfer (ESIPT)

One of the most remarkable electronic features of Schiff bases is their ability to undergo excited-state intramolecular proton transfer (ESIPT). In this process, a proton shifts within the molecule upon excitation, leading to significant shifts in emission wavelengths. ESIPT plays a crucial role in achieving efficient luminescence in Schiff base-based materials, making them attractive for fluorescence-based applications[31,32].

2. Molecular Orbital Analysis

The electronic behavior of Schiff bases is further understood through molecular orbital (MO) analysis. The highest occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital (LUMO) define the compound's electronic transitions. The energy gap between these orbitals is a key determinant of the optical and electronic properties of Schiff bases, influencing their absorption, fluorescence, and overall electronic behaviour[31,33].

3. Charge Distribution and Electrophilicity

Schiff bases often exhibit distinct charge distribution patterns, influenced by both their molecular framework and substituents. Electrophilic and nucleophilic regions within the molecule can be mapped using molecular electrostatic potential (MEP) analysis and Mulliken charge calculations. These insights provide a deeper understanding of the compound's reactivity and its interactions with other molecules, particularly in catalytic and biological environments[31,33].

4. Optical and Photonic Properties

Schiff bases are widely recognized for their outstanding optical and photonic properties, making them valuable for optoelectronic applications. Their high polarizability and hyperpolarizability contribute to nonlinear optical behavior, while their fluorescence characteristics enhance their utility in sensing and imaging technologies. These properties make Schiff bases promising candidates for use in advanced electronic and photonic materials[31,33,34].

The catalytic and biological activities of Schiff bases are directly influenced by their structural features. Metal coordination, substituent effects, and conjugation significantly impact their efficiency. Understanding the structure-activity relationship (SAR) aids in designing Schiff bases with optimized catalytic and therapeutic applications.

2. Challenges and Future Prospects

Despite significant progress in the study and application of Schiff base complexes with first-row transition metals, several challenges remain :

- i. **Stability and Solubility:** Many Schiff base complexes degrade in solution, particularly under physiological or catalytic conditions, limiting their effectiveness in biological and industrial applications.
- ii. **Selective Catalysis:** Achieving high selectivity in catalytic processes can be difficult, especially in multi-step reactions where unintended side products reduce efficiency.
- iii. **Bioavailability and Toxicity:** While these complexes show promise in biological applications, their absorption, distribution, and potential toxicity need further study to ensure safety in pharmaceutical use.
- iv. **Structural Optimization:** Fine-tuning ligand design and metal coordination to enhance functional properties is complex and requires extensive modifications.
- v. **Scalability and Cost:** Producing Schiff base complexes on a larger scale with high purity remains a challenge due to material costs and complex purification processes.

Future Prospects

To address these challenges and expand the potential of Schiff base-metal complexes, future research could focus on:

- i. **Ligand Design and Functionalization:** Modifying ligands with electron-donating or withdrawing groups may improve stability, solubility, and selectivity.
- ii. **Computational and AI-Assisted Approaches :** Using computational chemistry and artificial intelligence could streamline the design of new Schiff base complexes with optimized properties.
- iii. **Biocompatibility and Drug Delivery:** Investigating their potential in targeted drug delivery and controlled release systems could lead to new medical applications.
- iv. **Sustainable Synthesis:** Developing environmentally friendly synthetic methods, such as solvent-free or microwave-assisted techniques, could make production more efficient.
- v. **Hybrid Materials and New Applications:** Incorporating these complexes into materials like metal-organic frameworks (MOFs) or nanomaterials could open up new possibilities in sensing, energy storage, and catalysis.

With continued research, Schiff base complexes with first-row transition metals have the potential to contribute meaningfully to catalysis, medicine, and materials science, leading to new discoveries and practical advancements.

3. Conclusion

Schiff base complexes of first-row transition metals have emerged as a cornerstone of modern coordination chemistry, demonstrating remarkable versatility in catalysis, bioactivity, and material science. The structural adaptability of Schiff base ligands, combined with their ability to form stable and functionally diverse metal complexes, has driven extensive research in both fundamental and applied sciences. Their catalytic efficiency in oxidation, reduction, and coupling reactions underscores their potential for advancing green and sustainable chemistry. Additionally, their significant biological activities, including antimicrobial, anticancer, and antioxidant properties, highlight their relevance in pharmaceutical and biomedical applications. Despite these advancements, challenges remain in optimizing the stability, selectivity, and solubility of these complexes to enhance their real-world applicability. Addressing these limitations through innovative ligand modifications, advanced synthetic methodologies , and computational modelling will be essential for unlocking their full potential. Future research should focus on developing novel Schiff base-metal frameworks with enhanced electronic and structural properties, facilitating the design of next-generation catalysts and bioactive compounds.

Given the rapid progress in this field, Schiff base complexes with first-row transition metals hold immense promise for transformative applications in science and technology. A deeper understanding of their coordination chemistry and functional mechanisms will undoubtedly drive further innovation, solidifying their role as indispensable tools in catalysis, pharmaceutical chemistry, and materials science.

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