EUROPEAN JOURNAL OF BIOMEDICAL AND PHARMACEUTICAL SCIENCES
http://www.ejbps.com

ISSN 2349-8870
Coden USA: EJBPBO
Volume: 13
Issue: 7
Year: 2026

MICROWAVE ASSISTED GREEN SYNTHESIS OF SCHIFF BASE LIGANDS AND THEIR TRANSITION METAL COMPLEXES: DESIGN, CHARACTERIZATION AND APPLICATIONS

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DOI: https://doi.org/10.5281/zenodo.21153143

How to cite this Article: Vishakha S. Kukade[1], Meghasham N. Narule*. (2026). Microwave Assisted Green Synthesis Of Schiff Base Ligands And Their Transition Metal Complexes: Design, Characterization And Applications. European Journal of Biomedical and Pharmaceutical Sciences, 13(7), 277–282.

Article Received on 05/06/2026 **Article Revised on 25/06/2026** **Article Published on 03/07/2026**

ABSTRACT

Study reports the microwave-assisted green synthesis of Schiff base ligands derived from aromatic diamine systems, followed by their coordination with transition metal ions. The synthetic approach significantly reduces reaction time and solvent usage while improving yield and purity. The synthesized ligands, containing azomethine (–C=N–) and azo (–N=N–) functionalities, exhibit strong chelating ability toward transition metals such as Cu(II), Co(II), Ni(II), and Zn(II). Structural characterization was performed using Fourier Transform Infrared (FTIR), Nuclear Magnetic Resonance (NMR), Ultraviolet–Visible (UV–Vis) spectroscopy, and mass spectrometry. Thermal stability and decomposition behavior were analyzed using thermo gravimetric analysis (TGA) and differential scanning calorimetry (DSC), while crystallographic studies were conducted using X-ray diffraction techniques. The results confirm the formation of stable coordination complexes with defined geometries. The metal complexes demonstrate enhanced biological activity compared to free ligands, indicating their potential applications in medicinal chemistry, catalysis, and materials science.

KEYWORDS: Schiff base; Microwave-assisted synthesis; Green chemistry; Transition metal complexes; Spectroscopy; Coordination chemistry.

1. INTRODUCTION

The development of sustainable and environmentally friendly[1-6] synthetic methods has become a major focus in modern chemistry. Green chemistry emphasizes the reduction of hazardous substances, energy efficiency, and waste minimization. Schiff base ligands, characterized by the presence of heterocyclic[7-15] azomethine (–C=N–) groups, are among the most extensively studied compounds in coordination chemistry due to their ease of synthesis, structural versatility and wide range of applications. These ligands exhibit strong coordination ability with transition metal ions through donor atoms such as nitrogen and oxygen. The resulting metal complexes[16-19] possesses diverse organometallic[17-23] geometries and enhanced physicochemical properties, making them valuable in catalysis, pharmaceuticals, and materials science. Microwave-assisted synthesis has emerged as an efficient alternative to conventional heating methods. This technique utilizes dielectric heating, enabling rapid and uniform energy distribution within the reaction medium. Compared to traditional methods, microwave synthesis offers shorter reaction times, higher yields, improved purity, and reduced environmental impact.

The basic reaction that takes place in the formation of Schiff bases can be explained as:
Primary Aromatic Amine + Aromatic Aldehyde → Azomethine (Schiff Base) + Water

For example:

- Benzidine + Salicylaldehyde → N,N′-bis(salicylidene)benzidine
- o-Phenylenediamine + Benzaldehyde → N,N′-bis(benzylidene)o-phenylenediamine

- Diazene-based amine + Aromatic aldehyde → Azo-Schiff Base Ligand

The ligands have several nitrogen donor groups and extended conjugation, which improves their coordination capacity with transition metal ions. Figure 1 indicates Schiff Base Formation Reaction.

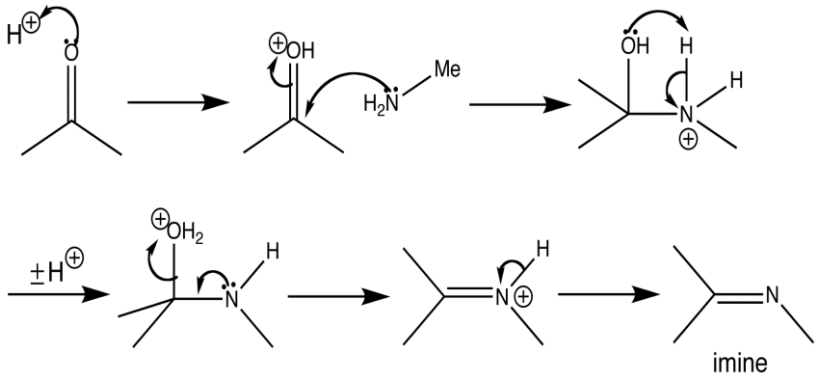
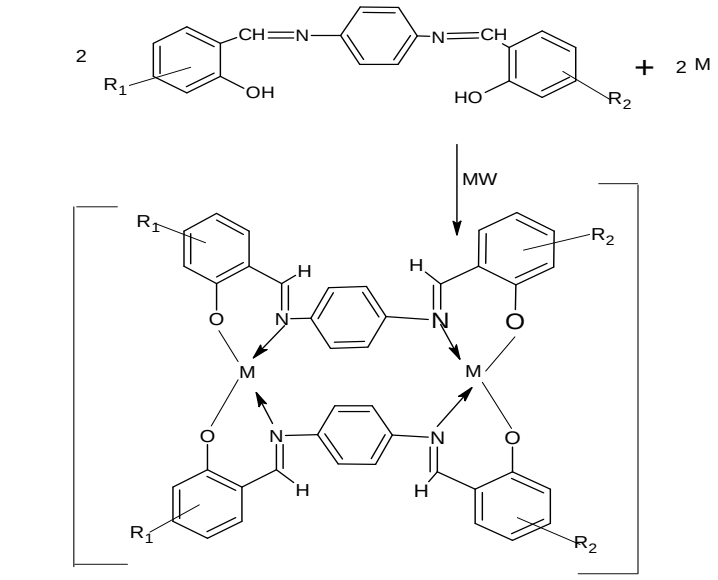


Fig. 1: Schiff Base Formation Reaction.

2. Design and Preparation of Based Schiff Base Scaffolds

Benzedrine-based Schiff base scaffolds are among the significant classes of organic compounds that have received significant interest in several other areas such as medicinal, coordination and material science. Preparation of the Schiff bases is normally done through condensation reaction between primary amines and carbonyl-containing compounds, e.g., aldehydes or ketones. As a starting material to further form symmetrical or asymmetrical Schiff base ligands, benzidine that is an aromatic diamine in which two primary amines have been attached to a biphenyl core can be utilized. Two main amine groups embedded in benzamine make it possible to create bis-Schiff base ligands, which are characterized by an increased number of structural and functional stability. It has also been known that these ligands have diverse biological activities such as; antimicrobial, antiviral, anticancer and anti-inflammatory. Moreover, benzamine-based Schiff bases are conjugated, which means that they have coordination capacity with metal ions, and more powerful metal complexes are formed.



Where,

R_1,R_2=H, OH, NO_2, Cl, OCH_3, (OCH_3), $N(CH_3)_2$, Br,

M(Metals)= Verious Metals

Scheme-I

In this case, both amino groups of benzidine are able to react with the carbonyl group of the aldehyde or ketone, resulting in bis-imine formation, as long as two moles of aldehyde are present. However, the formation of the imine group can also be reversible; hence, the conditions of the reaction are crucial in completing the formation of the Schiff base.

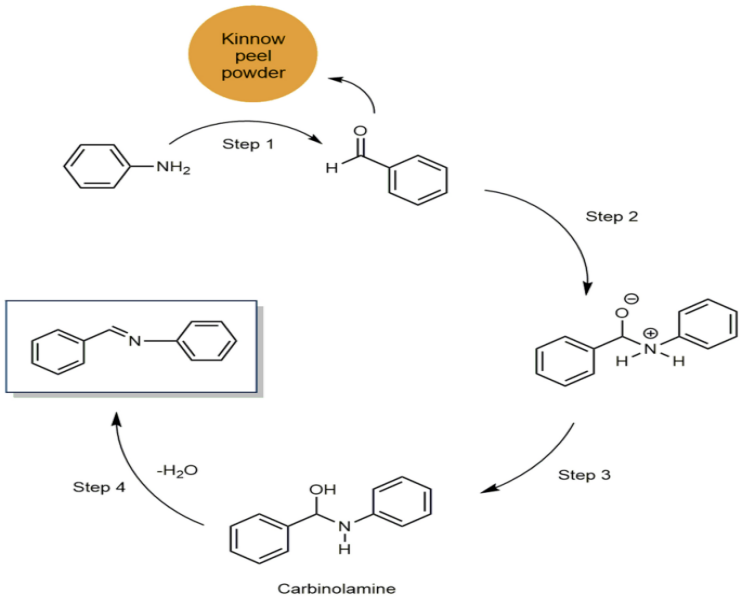


Fig. 2: General mechanism of Schiff base formation from benzidine and aldehydes.

They need to optimize the reaction conditions to obtain high yield and purity when synthesizing benzidine-based Schiff bases. The choice of solvent can have a great impact on the solubility of reactants or on the stability of intermediates. Ethanol polar protic solvents are usually used due to its ability to dissolve the benzidine and aromatic aldehydes as well as assist in the transfer of protons during the reaction. The geometry and electronic nature of the ligand structure highly determine the coordination behavior of Schiff bases. Thus, design of scaffolds made of benzidine typically takes into account the possibility of use of chelating ligands by the scaffold in a metal coordination system.

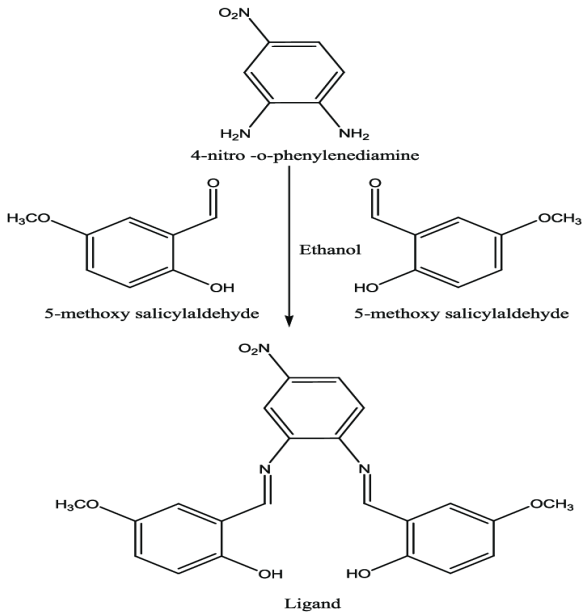


Fig. 3: Representative benzidine-based bis-Schiff base scaffold.

As a pharmaceutical, benzidine-based Schiff bases are explored in an array of biological functions because of their capacity to interrelate with biomolecules via hydrogen bonding, π -pile, and coordination bases. The biological activity can be dramatically affected by structural alterations of the scaffold, and thus, to generate useful therapeutic candidates, rational design approaches will be necessary.

2.1 X-ray Crystallography

The DSC technique is very useful in providing supporting information for the TGA technique, as it helps to determine the energy changes during thermal transformations. By using the results obtained from the TGA and DSC techniques, the thermal stability and decomposition mechanism of the metal complex can be understood.

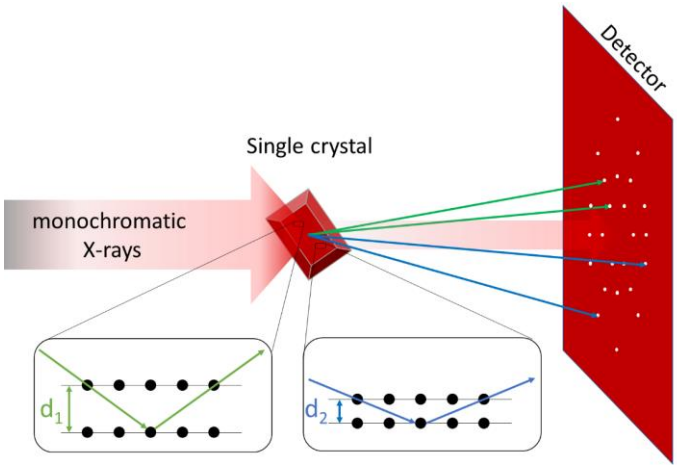


Figure 1.14: X-ray Crystallography.

X-ray crystallography is one of the most powerful instruments that can be used to establish the structure of crystalline materials. This is a process of crystallography based on the X-ray diffraction of crystalline materials. The existence of such interactions is what knowledge is required to make predictions regarding the physical and chemical properties of the complex.

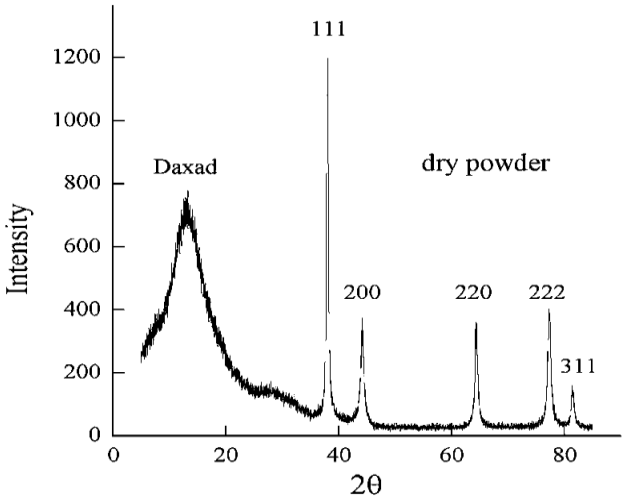


Figure 1.15: Powder X-ray Diffraction (PXRD).

PXRD analysis is the powdered sample of many small crystallites is exposed to X-rays in PXRD analysis and a diffraction pattern is produced which reflects the general crystal structure of the material.

3. MATERIALS AND METHODS

3.1 Chemicals and Reagents

All chemicals used in this study, including aromatic diamines (benzidine, o-phenylenediamine, p-phenylene diamine), aldehydes, and transition metal salts ($CuCl_2$, Co $(NO_3)_2$, Ni $(CH_3COO)_2$, $ZnCl_2$), were of analytical grade and used without further purification.

3.2 Synthesis of Schiff Base Ligands

Schiff base ligands were synthesized via condensation reactions between aromatic diamines and aldehydes. The reaction mixture was subjected to microwave irradiation in a polar solvent such as ethanol or methanol. A few drops of acetic acid were added as a catalyst to enhance the reaction rate. The reaction was completed within minutes, yielding crystalline Schiff base products after cooling and recrystallization.

3.3 Microwave-Assisted Synthesis of Metal Complexes

The synthesized ligands were dissolved in a suitable solvent and mixed with corresponding metal salt solutions. The reaction mixture was irradiated under microwave conditions for 5–15 minutes. The resulting complexes were filtered, washed, and purified by recrystallization.

3.4 Characterization Techniques

- FTIR Spectroscopy: Identification of functional groups and coordination sites
- NMR Spectroscopy: Structural confirmation of ligands
-UV–Vis Spectroscopy: Electronic transitions and geometry analysis
- Mass Spectrometry: Molecular weight determination

-TGA/DSC: Thermal stability and decomposition analysis
- X-ray Diffraction (XRD): Crystallographic structure determination

4. RESULTS AND DISCUSSION

4.1 Synthesis and Yield

Microwave-assisted synthesis significantly reduced reaction time from several hours to a few minutes. The yields of both ligands and metal complexes were higher compared to conventional methods, with improved purity and reduced by-products.

4.2 Ligand Structure and Coordination Behavior

The Schiff base ligands exhibited strong coordination ability due to the presence of azomethine nitrogen and additional donor atoms such as phenolic oxygen. Diamine-derived ligands formed bis-imine structures, enhancing chelation through the chelate effect.

4.3 Spectroscopic Analysis

FTIR spectra showed characteristic imine (C=N) stretching bands around 1600–1650 cm^{-1}. Upon coordination with metal ions, these bands shifted to lower frequencies, indicating involvement of the nitrogen atom in bonding. NMR spectroscopy confirmed the presence of imine protons in the range of 8–9 ppm. UV–Vis spectra revealed π–π* and n–π* transitions, along with d–d transitions in metal complexes, suggesting geometries such as square planar, tetrahedral, and octahedral. Mass spectrometry confirmed molecular weights and fragmentation patterns consistent with proposed structures.

4.4 Thermal Analysis

TGA results indicated multi-stage decomposition patterns. Initial weight loss corresponded to solvent molecules, followed by decomposition of coordinated ligands at higher temperatures. Residual metal oxides were observed at elevated temperatures. DSC analysis showed endothermic peaks corresponding to solvent loss and exothermic peaks associated with ligand decomposition.

4.5 Crystallographic Studies

X-ray diffraction analysis confirmed the crystalline nature of the complexes. Structural parameters indicated coordination geometries dependent on the metal ion:
- Cu (II): square planar
- Ni (II), Co (II): octahedral
- Zn (II): tetrahedral

4.6 Ligand–Metal Binding Mechanism

Schiff base ligands act as multidentate ligands, coordinating through nitrogen and oxygen donor atoms. The formation of chelate rings enhances stability via the chelate effect. Factors such as ligand structure, metal ion properties, and solvent influence the coordination behavior.

4.7 Biological and Catalytic Applications

Metal complexes exhibited enhanced antimicrobial, antifungal, and antioxidant activities compared to free ligands. Increased lipophilicity due to metal coordination improves interaction with biological targets.

Additionally, these complexes show potential as catalysts in oxidation, polymerization, and coupling reactions. Their electronic properties also make them suitable for materials science applications.

5. CONCLUSION

Microwave-assisted synthesis provides a rapid, efficient, and environmentally friendly approach for the preparation of Schiff base ligands and their transition metal complexes. The synthesized complexes exhibit well-defined structures, enhanced stability, and improved biological activity. This study demonstrates the potential of combining green chemistry principles with advanced synthetic techniques to develop functional coordination compounds for diverse applications.

Declaration of Competing Interest

The authors declare no conflict of interest.

ACKNOWLEDGMENTS

The Director SAIF Punjab University Chandigarh is thanked by the author for supplying spectral and analytical data.

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