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GREEN AND SUSTAINABLE MICROWAVE STRATEGIES FOR THE SYNTHESIS OF BENZIDINE, DIAZENE, AND DIAMINE SCHIFF BASE METAL COMPLEXES

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Abstract:

The synthesis of Schiff base metal complexes using green and sustainable techniques has gained significant attention due to the growing demand for eco-friendly and energy-efficient chemical processes. This review focuses on the microwave-assisted synthesis of Schiff base derivatives, specifically targeting the preparation of benzidine, diazene, and diamine Schiff base metal complexes. Microwave-assisted techniques have proven to be effective in reducing reaction times, improving yields, and minimizing environmental impact, making them an ideal solution for sustainable chemistry. The review provides an overview of various green solvents, including water, ionic liquids, and supercritical carbon dioxide ($scCO_2$), which contribute to the sustainability of Schiff base synthesis. The article also discusses the biological activities of the synthesized metal complexes, highlighting their potential applications in pharmaceutical and industrial fields. This study consolidates recent advancements in the field and explores the challenges and future directions for developing green and sustainable microwave strategies for Schiff base synthesis.

Keywords:*Schiff Bases, Green Chemistry, Microwave-Assisted Synthesis, Transition Metal Complexes, Sustainable Chemistry, Biological Activity,Benzidine, Diazene, Diamine Complexes.*

Introduction:

Schiff bases are a class of compounds characterized by the imine group (-C=N-) formed through the condensation of primary amines with carbonyl compounds. These versatile compounds are widely used in various applications, including pharmaceuticals, catalysis, and materials science. The synthesis of Schiff base metal complexes has attracted considerable attention due to their ability to form coordination bonds with transition metals, which enhances their biological and catalytic properties (Shri & Mehdi, 2021). Traditionally, the synthesis of Schiff bases and their metal complexes have involved the use of hazardous solvents and energy-intensive methods, raising concerns regarding their environmental impact and sustainability (Fahmi et al., 2022).

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In recent years, the demand for greener and more sustainable methods has led to the exploration of microwave-assisted synthesis as a promising alternative. Microwave irradiation provides several advantages, including rapid heating, high yields, and minimal solvent use, making it an energy-efficient and eco-friendly approach (Manjare et al., 2022). Additionally, the use of green solvents, such as water, ionic liquids, and supercritical carbon dioxide, further enhances the sustainability of the synthesis process (Shashi & Gandhi, 2021). This review aims to provide a comprehensive understanding of the green and sustainable microwave strategies for the synthesis of benzidine, diazene, and diamine Schiff base metal complexes, focusing on their synthesis, characterization, and biological applications.

Objectives of Study:

1) To examine the role of microwave-assisted techniques in enhancing the efficiency, yield, and sustainability of Schiff base synthesis.
2) To explore the application of green solvents, such as water, ionic liquids, and supercritical carbon dioxide, in Schiff base synthesis.
3) To evaluate the biological activities of Schiff base metal complexes, particularly their antimicrobial and antioxidant properties.
4) To identify research gaps and future directions for the development of greener methods for Schiff base metal complex synthesis.

Green and Sustainable Microwave Strategies for the Synthesis of Benzidine, Diazene, and Diamine Schiff Base Metal Complexes:

Microwave-assisted synthesis (MAS) has demonstrated wide practical applications in the realm of green chemistry as a substantial global move to minimize and also offset both cost and environmental consequences in Schiff bases and their anti-cancer metal complexes. It is a reaction-drastically enhanced in rate and time efficiency, for it brings about changes that would have been unnoticed by convection in a microsecond while resting for hours on end otherwise. Yield is greatly increased in theory, simultaneously downsizing energy consumption (Soni et al., 2022). For example, microwave-assisted condensation of benzidine with diazene and some diamines clearly had a chemical benefit due to the drastic reduction in condensation times for the end product. The Schiff bases undergo a further improvement in anti-microbial, antifungal, and anti-cancer actions upon coordination with copper, cobalt, or iron metal (Fahmi et al., 2022).

Among the very aspects accepted by practitioners in making MOAS, the use of green solvents-notably the choices of water, ionic liquids, or supercritical carbon dioxide (scCO2)is a non-toxic, non-flammable, and sustainable alternative to most solvents. Green solvents have successfully reduced the necessity of toxic chemicals and solvents, paving the way to sustainability. For instance, Water turned out to be an effective solvent for Schiff base synthesis with appreciable yields and without the need to add extra

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catalysts (Shashi & Gandhi 2021). scCO(2), as well, offers a clean, residue-free synthesis medium, which adds further viability to the ecological friendliness of the synthesis (Manjare et al. 2022). The green solvents in which the synthesis is carried out, when coupled with microwave irradiation, help to give very favorable results due to the best yields and reaction duration when compared with the conventional methods, lessening (a) the waste, (b) the energy, and (c) the environmental impacts.

1. Overview of Schiff Base Synthesis via Microwave-Assisted Methods:

One frequently finds the term "imines" associated with Schiff bases. Schiff bases stand for an organic group of compounds that possess a general formula of R2C=NH, where R is an alkyl or aryl group. Schiff bases generally arise from dissolving amines and carbonyl compounds, such as aldehydes and ketones, in concert at the primary sites. It is also a reason why Schiff bases have numerous utilities in various fields like catalysis, drug sciences, and pharmaceuticals: Antimicrobial, anticancer, anti-inflammatory, DNA-modifying enzyme inhibitors, or other reactions that cause biological activities greatly arise from these Schiff base metal complexes (Shri and Ahmad, 2021; Fahmi et al., 2022).

In the past, through the enabling of a refluxing reaction with aldehydes and anilines, various chemists had managed to make bio-based Schiff base synthesis and hence to apply ethanol or methanol. A catalyst-made continuous condensation reaction is the cause of the reaction. Give an old-fashioned way of doing things, and behold, some malaise that comes with it. Naturally, the primary con to this method, with the long reaction duration, often of several hours, reactions would undergo less condensation and result in the last synthetically produced product with low yields. Does the third point of the argument actually make sense? Considering that all these procedures are poisoning the biota, pointing at their user friendliness makes quite weak sense. The aryl ether is often associated with a relatively large amount of total liquid waste that is generated when operating in some kind of Eco-horror process.

The new era of microwave-assisted methodologies certainly offers itself as much efficient and sustainable, although over the traditional practices. The method employs microwave radiation for the direct heating of the reaction mixture, hence lending implicit and entire uniformity of energy and speeding up the reactions. Thus, the microwave-assisted method for Schiff Base synthesis renders unanswerably shorter synthetic time compared with traditional methods, possibly even achieved in minutes instead of hours. The microwave irradiation is very useful in keeping the energy input to a minimum while augmenting the yield of the product (Shashi and Gandhi, 2021). Microwave promotion seems to be quite commendable as an effective platform from which microwave-assisted synthesis can expressively evade the use of hazardous organic solvents, other than safe and eco-friendly chemicals, thus furthering the principles of green chemistry for there to be safe and sustainable chemistry processes (Soni et al., 2022).

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2. Detailed Discussion on the Synthesis of Benzidine, Diazene, and Diamine Schiff Bases:

Reaction rates are increased by several orders of magnitude upon microwave-assisted heating in the Schiff base formation with benzidine, diazene, diamines, and similar reactions from primary amines condensing with a carbonyl compound. The most significant benefit granted here is that the overall reaction sluggishly makes its way to completion for some days and minutes, depending on the extent to which the matter goes under test. For instance, the increased reaction rate might give diamine of benzidine by the methylation of salicylaldehyde using benzaldehyde, and that run-time seemed rather costly forever or millennia by comparison with microwave irradiation in hours or an evening, depending on whether it is awkward due to temperature under microwave action (Hualpa, 2014). Microwave processing in benzaldehyde or salicylaldehyde reactions in response to lengthy days long hours of reflux heaters gave a second change by quick, uniform heating mode.

The synthesis of diazene Schiff bases has been improved using microwave irradiation. The conventional method of forming diazene Schiff bases necessitates extended heating time during the reaction of amines with diazonium salts. Microwave heating, nonetheless, did the job in just 5–10 minutes. This quick rate of synthesis under microwave irradiation minimizes undesired by-products and thereby proves a more sustainable and efficient process according to the authors (Shashi & Gandhi, 2021). As against some previous methods, the reaction of diamines has been expedited with microwave assistance. For the established synthesis, rapid microwave benches are set at mere 10–20 minutes, ensuring even heating and enhanced yields (Soni et al., 2022). The microwaved form of synthesis has led to a less-simulated route of products, as represented in various methods involving solvent-free or minimal-solvent reactions, which is a step toward environmental sustainability (Shashi & Gandhi, 2021). As well, this seems to be the highest kind of synthesis, where microwave methods may outperform the otherwise caustic methods for their yield, effectiveness, environment, and large-scale production of Schiff base derivatives according to the principles. This also reduces energy consumption and waste (Fahmi et al., 2022).

3. Importance of Transition Metal Ions (Cu, Co, Fe, Ni) in the Formation of Schiff Base Metal Complexes:

The lopper (Cu), cobalt (Co), iron (Fe), and nickel (Ni) transition metals in the first row are critically important in coordination chemistry since they contribute greatly, helping in Schiff base a metal complex formation. These are metals that occupy free d-orbitals, which deal with coordination bond formation mostly through the electron donor ligands such as Schiff bases. Building up electron-donating ligands of ion, like the Schiff bases, are of further importance to have the imine group (-C=N-) as the first-rank ligands since the imine nitrogen can donate electrons to the nickel, and in some cases, could

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potentially donate electrons by other functional groups such as hydroxyl-like oxygen (Shri & Mehdi, 2021). Variation in metals used with different oxidation states and coordination geometries enables them to form stable complexes with Schiff bases and, thus, make them of a good variety of potentials for multiple applications, especially in catalysis, materials science, and medicinal chemistry (Fahmi et al., 2022).

In Schiff base complex formation, copper, cobalt, iron, and nickel are the best metal options because of their pharmacological and physiochemical properties. The oxidation state is what makes these choices suitable. With impressive oxidative uses, Cu(I)/Cu(II) forms both metal complexes with a wide range of ligands, xanthochromatic, in particular. Cobalt and iron may serve as the second choice. Cobalt mainly exists in oxidation states +2 and +3, which are used ubiquitously for biological systems. This is, in some cases, incorporated into enzymes as essential cofactors. A big attribute of nickel is its ability to stabilize +2 oxidation states and is a relatively cheap metal with which to work. All metals nickel has been largely the preferred in industrial applications due to the stability of Schiff base complexes, the catalytic activity they retain while in complexation (Shashi & Gandhi, 2021).

The formation of Schiff base-metal ion coordination involves the migration of an electron pair by the Schiff base ligand to the metal center. Such coordination is very important for the stability and biological activity of these complexes. One example includes a Cu(II) Schiff base complex that showed excellent antimicrobial activity against Gram-negative bacteria (Fahmi et al., 2022). The transition metal ion also becomes crucial for the whole complex arising from electrons moving involving Schiff bases in catalysis, necessary for organic synthesis and environmental remediation (Soni et al., 2022). In general, chelation of transition metals by Schiff bases flips the stability and biological effectiveness coin, creating profound impacts in the fields of therapeutic and industrial.

4. Benefits of Microwave-Assisted Synthesis: Shorter Reaction Times, Higher Yields, Minimal Solvent Use:

Microwave-assisted synthesis is highly advantageous because of quick reaction time, increased yield and reduction of solvents. One of the bonuses of microwave-assisted synthesis is that reaction times are shortened almost to the minimum as compared to traditional methods with the same temperatures: it would take several hours for a reflux reaction to complete but a few short minutes would suffice for a microwave-assisted reaction (Shashi & Gandhi, 2021; Soni et al., 2022); reduced reaction times are scaled up to become compatible for large industrial use, as reaction times are sometimes very critical and can slow down the process of completion.

As the rate of reaction is increased with microwave radiation, greater molecule collisions occur and the deactivation energy is decreased, which results in faster product formation. The microwave-enhanced processes provide 80–90& yield of Schiff bases and

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metal complexes compared to a mere 60–70% in the traditional mode (Fahmi et al., 2022). It thus reduces the cost of production and hastens the entire duration of the reaction.

Secondly, the removal or reduction of the solvent relies on microwave energy, which is used in all conventional practices. However, by using the solvent-free methods or decreasing the amount of solvent, the conditions, in compliance with green chemistry principles, become fairer and minimize the waste and potential hazards associated with the toxic solvents (Shashi & Gandhi, 2021). Thus, the microwave-assisted synthesis may be deemed the cheapest and most environmentally acceptable method.

5. Advantages of Using Green Solvents Such as Water, Ionic Liquids, and Supercritical CO_2 in Schiff Base Synthesis:

Sustainable chemistry is about being at ease with the environment; water contributes to a reduced environmental risk and health hazards. In fact, water is regarded as an extremely "green" solvent compared with the volatile organic solvents it replaces. The moth-making synthesis of Schiff bases, a highly important area in organic chemistry (Shashi & Gandhi, 2021; Raja et al., 2022), provides significant benefits. Due to its non-toxic, non-flammable, and readily available characteristics, water is the most widely used green solvent Known for its exceptional capacity to grant high-yield reactions with relatively small environmental repercussions imbibed in a solvent-less system even in the synthesis of Schiff base, at least unless the reactants are lipophilic in nature, which may involve elevated temperatures or longer reaction times (Soni et al., 2022).

Ionic liquids are being considered as a new departure in clean solvents, notable for their low volatility and tunable characteristics. Their high solvating ability allows reactants to readily dissolve, thus affecting reaction rate and yield favorably. Furthermore, ionic liquids can be recuperated and, in turn, play a role in waste minimization, in the process also helping to decrease the costs of using these solvents on commercial levels. The flip side to these ionic liquids is the high cost and the likelihood of being toxic. (Shashi & Gandhi, 2021).

Supercritical CO_2 is a solvent that is innocent, non-flammable, and nontoxic. It also has solvent properties with excellent reaction conditions inferior to the environmental toxicity of the organic solvents. Its ease of removal and redistribution from the reaction site make it perfect for avoiding waste disposal and for increasing the efficiency of the reaction since heat and mass migrations are simplified (Manjare et al., 2022). Its introduction into the industry for some applications is further limited by the absence of a source of high-pressure density and equipment in operation (Sinkar, 2021).

One of the frequently used strategies to increase the sustainability of the Schiff base synthesis is the use of green solvents. They affect a great deal of growth with fewer health threats and effort toward a hazardous path (Soni et al., 2022).

6. Examples of Schiff Base Complexes Synthesized via Microwave-Assisted Methods and Their Industrial Applications

Schiff base metal complex fragments are popular due to their quick and microwave-assisted condensation, and they represented the systems most suitable for the microwave sensors used in the studies. For the latter, complexes of Schiff bases with metal(II) (Cu) and metal(III) (Co) were fashioned by microwave methods that sped up the reaction to approximately 15 min per synthesis chore, occurring much quicker than the hours the conventional process requires (Soni et al., 2022). It was confirmed through suitable identification techniques that the complexation of Schiff base ligands had occurred successfully with the metals and that FT-IR, NMR, UV-Vis, and magnetic susceptibility measurements were applied on the synthesized metals.

This change in time of the microwave-bearing system for metal might produce some promising, quick diagnoses where rapidity and quality of results under acoustic pressurefetch the pharmaceutical application somewhat further.

Schiff base metal complexes are widely utilized in various areas such as medicinal chemistry, catalysis, and material science. For example, Cu(II) Schiff base complexes exhibit considerable antimicrobial and anticancer properties. They, therefore, have strong potential for future drug development. While enzyme inhibition and DNA cleavage are other properties intrinsically present in such complexes. For catalysis, these complexes are rather good, accelerating various oxidation and polymerization processes. For such applications, chemically heterogeneous examples can be prospects in the refinery industry and fine chemical production, etc.Schiff base complexes are effectively involved in the production of surface-acting functional polymers, films, and coatings.

Many topics regarding the scalability of microwave-assisted syntheses have been extensively studied, and the process of formation of the copper(II) Schiff base complex was scaled up so as to achieve higher efficacy, lower reaction time, and hence higher yield (Kumar & Mythreyi, 2015). Being eco-friendly and complying with principles of green chemistry, this process could also become a viable option for industrial practice as an economic and sustainable tool.

Characterization Techniques Used in Reviewed Studies:

The characterization of Schiff base metal complexes is crucial to understanding their structure, coordination environment, and biological properties. Several analytical techniques have been employed to confirm the successful synthesis of these compounds and to elucidate their structural details.

1) **Fourier Transform Infrared (FTIR) Spectroscopy:**FTIR spectroscopy is widely used to identify the functional groups present in Schiff bases and their metal complexes. The FTIR spectra typically show characteristic peaks corresponding to the azomethine (-C=N-) group, which is the hallmark of Schiff base ligands. The

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coordination of the metal ion to the ligand is confirmed by shifts in the IR bands associated with the azomethine and other functional groups (Shri & Mehdi, 2021).

2) **Nuclear Magnetic Resonance (NMR) Spectroscopy:**NMR spectroscopy, including 1H-NMR and 13C-NMR, is essential for determining the chemical structure of Schiff base ligands and their metal complexes. NMR spectra provide valuable information about the connectivity of atoms, the environment of hydrogen and carbon atoms, and the coordination of metal ions (Fahmi et al., 2022).
3) **Ultraviolet-Visible (UV-Vis) Spectroscopy:**UV-Vis spectroscopy is used to study the electronic transitions in Schiff base metal complexes. The presence of specific absorption bands in the UV-Vis spectra can provide insights into the coordination of the metal ion and the electronic properties of the complex (Shashi & Gandhi, 2021).
4) **Magnetic Susceptibility Measurements:**Magnetic susceptibility is an important technique for determining the magnetic properties of metal complexes. These measurements help identify the oxidation state of the metal ion and the geometry of the complex (Soni et al., 2022).
5) **X-ray Diffraction (XRD):**XRD is employed to determine the crystallographic structure of Schiff base metal complexes. This technique is particularly useful for analyzing the arrangement of atoms within the complex and confirming its stability (Butt et al., 2022).

Research Gaps:

Despite significant advancements in microwave-assisted synthesis of Schiff base metal complexes, several research gaps remain. One notable gap is the limited exploration of Schiff base complexes with rare or less-studied metals, despite extensive research on common metals like Cu, Co, and Ni. Further studies should focus on synthesizing these complexes and assessing their unique properties. Additionally, the optimal reaction conditions for different Schiff base derivatives and metal complexes are not fully established. There is a need for more research to optimize microwave irradiation conditions, including power levels, temperature, and reaction time, to achieve the best results across various Schiff base metal complexes. Furthermore, while microwave-assisted synthesis has shown effectiveness on a small scale, research is needed to explore its scalability for industrial applications, especially when using green solvents.

Moreover, although many Schiff base metal complexes exhibit promising biological activities, comprehensive evaluations of their therapeutic potentials are still lacking. Future research should prioritize in-depth biological studies, including clinical trials, to validate these complexes' antimicrobial, anticancer, and other therapeutic effects. The adoption of microwave-assisted synthesis using green solvents, such as water, ionic liquids, and supercritical CO_2, offers significant environmental benefits and aligns with the principles of green chemistry. As this field advances, addressing these research gaps will be crucial for developing sustainable and effective Schiff base metal complexes for various industrial and pharmaceutical applications.

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