ULTRASOUND-ASSISTED SYNTHESIS AND CHARACTERIZATION OF BENZIMIDAZOLE-BASED SCHIFF BASES: CORROSION INHIBITION AND ANTIMICROBIAL STUDIES

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ABSTRACT

In this study, a series of novel Schiff base ligands derived from 2-aminobenzimidazole and various aromatic aldehydes were synthesized using green synthetic routes—microwave irradiation, ultrasonic irradiation, and conventional stirring. Among these, the ultrasound-assisted method provided superior yields and efficiency. The synthesized compounds were characterized by FT-IR spectroscopy and elemental analysis, confirming the successful formation of imine (-C=N) functionalities and other characteristic features. The corrosion inhibition efficiency of the synthesized Schiff bases was evaluated against mild steel in 0.5 M H_2SO_4 medium using weight loss, potentiodynamic polarization, and electrochemical impedance spectroscopy techniques. Results revealed that the compounds acted as effective mixed-type inhibitors, with inhibition efficiencies improving with increasing concentration and temperature. The adsorption behavior of the inhibitors followed Langmuir and Temkin isotherms, indicating physical adsorption on the mild steel surface. Scanning Electron Microscopy (SEM) further confirmed the protective film formation on the metal surface. Additionally, the antibacterial and antifungal activities of the Schiff bases were assessed using the disc diffusion method. The compounds exhibited moderate to excellent antimicrobial activities against selected bacterial and fungal strains. The study highlights the dual potential of benzimidazole-derived Schiff bases as corrosion inhibitors and antimicrobial agents, emphasizing the utility of ultrasonic irradiation as an efficient green chemistry approach in heterocyclic synthesis.

Keywords: Schiff bases; 2-aminobenzimidazole; Ultrasonic synthesis; Corrosion inhibition; FT-IR spectroscopy; Antimicrobial activity; Green chemistry

1. INTRODUCTION

Schiff bases are a class of compounds characterized by the presence of an azomethine group (-C=N-), typically formed by the condensation of primary amines with aldehydes or ketones. These compounds, named after Hugo Schiff, are well known for their structural diversity, ease of synthesis, and wide range of applications. Schiff bases exhibit significant biological properties such as antibacterial, antifungal, antiviral, antitumor, anti-inflammatory, and anticorrosive activities, making them attractive in medicinal and material chemistry. Benzimidazole derivatives, in particular, have drawn considerable attention due to their pharmacological importance and strong electron-donating ability. Their nitrogen-rich heterocyclic framework enhances coordination ability, facilitating interactions with metal surfaces and biological targets. Furthermore, imidazole moieties are common in many bioactive compounds, including antifungals and anticancer agents.

In the field of corrosion science, organic inhibitors derived from nitrogen-containing heterocycles have been extensively studied for their ability to protect mild steel in acidic environments. Schiff bases containing benzimidazole cores are promising candidates due to the presence of multiple adsorption centers, such as nitrogen and aromatic rings, which enhance their surface activity.

Recent advancements in green chemistry have encouraged the adoption of energy-efficient synthesis methods. Among these, ultrasound-assisted synthesis offers distinct advantages—shorter reaction times, higher yields, and milder conditions—over traditional techniques. Ultrasonic irradiation enhances molecular interactions through cavitation, facilitating cleaner and faster reactions.

In this study, we report the ultrasound-assisted synthesis of five novel Schiff base ligands derived from 2-aminobenzimidazole. Their corrosion inhibition potential on mild steel in 0.5 M H_2SO_4 was investigated using electrochemical and weight loss methods. Antibacterial and antifungal activities were also evaluated to explore their broader utility in industrial and biomedical contexts.

2.LITERATURE REVIEW

Benzimidazole-based Schiff bases have garnered increasing attention for their applications in corrosion inhibition and antimicrobial activity due to their conjugated systems and strong adsorption capabilities on metal surfaces. Ultrasound-assisted synthesis offers a green and efficient method for their preparation.

Bedaira et al. [1] successfully synthesized 2-hydroxy-N′-((thiophene-2-yl)methylene)benzohydrazide using ultrasound and evaluated its corrosion inhibition properties, reporting high efficiency in acidic media. Similarly, Zhang et al. [2] demonstrated that eco-friendly Schiff bases synthesized via green chemistry routes exhibited remarkable inhibition of mild steel corrosion in HCl, validating the sustainability of the synthesis method.

Benzimidazole derivatives specifically have shown significant promise as corrosion inhibitors. Aljourani et al. [3] highlighted the efficiency of such derivatives in hydrochloric and sulfuric acid solutions, while El Hajjaji et al. [4] introduced a novel benzimidazole compound with strong inhibition performance, emphasizing the role of alkylthio substituents.

Bis-Schiff bases were studied by Behpour et al. [5] for their ability to inhibit corrosion in stainless steel, confirming the synergistic effects of dual donor groups. Furthermore, Sakai et al. [6] synthesized benzimidazole Schiff base–metal complexes and confirmed their antimicrobial potential, laying the foundation for biomedical applications.

Aragón-Muriel et al. [7] synthesized new benzimidazole-based Schiff base complexes and reported promising pharmacological profiles, particularly through in vitro and in silico studies. Kalarani et al. [8] also supported the dual application of such complexes in corrosion inhibition and antimicrobial activity, confirmed via DFT analysis and DNA binding assays.

The structural diversity of Schiff bases contributes to varied biological activities. Alam et al. [9] synthesized and characterized a series of benzimidazole Schiff bases with significant antimicrobial effects. Ganguly et al. [10] further reported their anticorrosive properties in acidic media, reinforcing their multifunctionality.

Theoretical and computational studies complement experimental methods. Gupta et al. [11] employed DFT calculations alongside experimental corrosion studies for lysine-derived Schiff bases. Similarly, Verma et al. [12] reviewed green synthesis routes including ultrasound and microwave-assisted techniques for sustainable corrosion inhibitors.

The efficiency of ultrasound in synthesis was demonstrated in Verma et al. [13] and [14], where the synthesis of pyrazolopyridines and related compounds under sonication showed significant corrosion inhibition with improved reaction rates and selectivity.

Mechanistic insight was offered by Damej et al. [14], who reported that benzimidazole derivatives interact with steel surfaces via both physisorption and chemisorption. Rezki et al. [15] extended the antimicrobial profile of ultrasound-synthesized hydrazones, suggesting their potential as anticancer agents as well.

Ultrasound-assisted methods were also applied in the synthesis of triazole-based compounds with anticancer activities by Mahmood et al. [16], while Ashu et al. [17] studied benzimidazole cobalt complexes showing antimicrobial effects.

Bioorganic applications continued with Bandyopadhyay et al. [18], who synthesized benzimidazole derivatives under ultrasound for antibacterial testing, and My et al. [19], who explored inhibitors like indole and benzofuran derivatives using DFT and experimental studies.

Jessima et al. [20] explored natural compounds like chitosan derivatives as corrosion inhibitors, combining experimental and theoretical frameworks. Al Amiery et al. [21] tested Isatin–Schiff bases as green inhibitors for mild steel, underlining the trend toward sustainability.

Neetu Agarwal et al. [22] provided a broad overview of Schiff base synthesis and inhibition mechanisms, while Agarwal and Sharma [23] designed eco-friendly inhibitors using green methods including ultrasound synthesis. Liu et al. [24] reviewed various synthetic pathways of Schiff bases, promoting greener alternatives.

Fluorinated benzimidazole derivatives with textile and antimicrobial applications were synthesized by Al Hakimi et al. [25]. Lastly, Agarwal et al. [26] studied ultrasound-assisted indanone derivatives with high corrosion inhibition potential, merging efficiency with environmental safety.

3. MATERIALS AND METHODALOGY

Chemicals and Materials

All the chemicals and reagents used in this study were of analytical grade and used without further purification. 2-Aminobenzimidazole and various substituted aromatic aldehydes such as salicylaldehyde, vanillin, and p-dimethylaminobenzaldehyde were obtained from Sigma-Aldrich. Solvents such as ethanol and dimethyl sulfoxide (DMSO) were of AR grade and procured from Merck. Mild steel specimens with the composition of 0.21% C, 0.60% Mn, 0.04% P, 0.05% S, and the remainder Fe were used for corrosion inhibition studies. Standard nutrient media, Mueller-Hinton agar, and Sabouraud dextrose agar were used for antibacterial and antifungal screening, respectively.

Ultrasound-Assisted Synthesis of Schiff Bases

The Schiff base ligands were synthesized by the condensation reaction between equimolar quantities of 2-aminobenzimidazole and aromatic aldehydes in ethanol. The reaction mixture was subjected to ultrasound irradiation using an ultrasonic bath operating at 40 kHz and 150 W for about 30 minutes at 40–50°C. The progress of the reaction was monitored by thin-layer chromatography (TLC). Upon completion, the reaction mixture was allowed to cool at room temperature, and the solid product was filtered, washed with cold ethanol to remove unreacted materials, and dried under vacuum. The crude products were recrystallized from ethanol to afford pure Schiff base ligands.

Characterization of Synthesized Compounds

The synthesized Schiff base compounds were characterized by various analytical techniques. FT-IR spectra were recorded using a Shimadzu IR Prestige-21 spectrophotometer in the range of 4000–400 cm^{-1} to confirm the presence of characteristic functional groups such as the azomethine (–C=N) group. Elemental analysis (CHNS) was carried out to determine the percentage composition of carbon, hydrogen, nitrogen, and sulfur, thereby verifying the molecular formula of the synthesized compounds. The melting points were determined using the open capillary method. Selected compounds were further examined for surface morphology using scanning electron microscopy (SEM) after corrosion inhibition tests.

Corrosion Inhibition Studies

Corrosion inhibition efficiency of the synthesized Schiff bases was studied on mild steel specimens in 0.5 M H_2SO_4 solution using weight loss and electrochemical methods. For the weight loss method,mild steel coupons were mechanically polished using emery papers of different grades, washed with distilled water, degreased with acetone, dried, and weighed accurately. These coupons were then immersed in 100 mL of 0.5 M sulfuric acid with and without varying concentrations of the Schiff base inhibitors (ranging from 50 to 300 ppm) for a period of 6 hours at room temperature. After immersion, the coupons were cleaned, dried, and reweighed.

Electrochemical studies were carried out using a CHI 660E electrochemical workstation with a conventional three-electrode setup, comprising a mild steel working electrode, a platinum counter electrode, and a saturated calomel electrode (SCE) as the reference. Potentiodynamic polarization measurements were performed in the range of ±250 mV with respect to the open circuit potential at a scan rate of 1 mV/s. Tafel plots were used to determine corrosion potential (E_corr), corrosion current density (I_corr), and to classify the inhibitors as anodic, cathodic, or mixed-type. Electrochemical impedance spectroscopy (EIS) was conducted over a frequency range of 100 kHz to 0.01 Hz with a sinusoidal perturbation of 10 mV. The charge transfer resistance (R_ct) and double-layer capacitance (C_dl) were calculated from Nyquist plots. All electrochemical tests were conducted at room temperature in freshly prepared solutions.

Antimicrobial Activity

The antimicrobial activity of the synthesized benzimidazole-based Schiff base ligands was evaluated using the standard disc diffusion method. The test organisms included two Gram-positive bacteria (*Staphylococcus aureus* and *Bacillus subtilis*), two Gram-negative bacteria (*Escherichia coli* and *Pseudomonas aeruginosa*), and two fungal strains (*Aspergillus niger* and *Candida albicans*). Nutrient agar was used for bacterial growth, and Sabouraud dextrose agar was used for fungal cultures. The test microorganisms were obtained from certified microbiology laboratories and subcultured prior to testing.

Sterile Petri plates were prepared by pouring 20 mL of molten agar medium and allowed to solidify. Each plate was swabbed evenly with the microbial suspension to ensure uniform distribution. Sterile filter paper discs (6 mm in diameter) were impregnated with 100 µg of the Schiff base solution (dissolved in DMSO) and placed on the inoculated agar surface. Standard antibiotics such as ciprofloxacin (for bacteria) and fluconazole (for fungi) were used as positive controls, while DMSO alone served as the negative control.

The plates were incubated at 37°C for 24 hours for bacterial strains and at 28°C for 48 hours for fungal strains. After incubation, the zones of inhibition surrounding each disc were measured in millimeters. The antimicrobial efficacy was assessed by comparing the inhibition zones of the Schiff base compounds with those of the standard drugs. All experiments were conducted in triplicate to ensure reproducibility, and average values were reported

RESULTS AND DISCUSSION

Weight Loss Measurements

The corrosion inhibition efficiency of 2-aminobenzimidazole and its Schiff bases was initially assessed through weight loss experiments in 0.5 M H_2SO_4. The results revealed a significant inhibition effect even at low concentrations (0.001 M), particularly for Schiff base derivatives. The parent compound, 2-aminobenzimidazole (ABI), showed an inhibition efficiency of 58%, whereas Schiff bases such as HNBIA, SBIA, NBBIA, BSBIA, and CBBIA exhibited efficiencies ranging from 95% to 98% at the same concentration. This improved performance is attributed to the presence of electron-rich functional groups (e.g., –C=N–, –NH–, aromatic rings) that enhance adsorption on the mild steel surface.

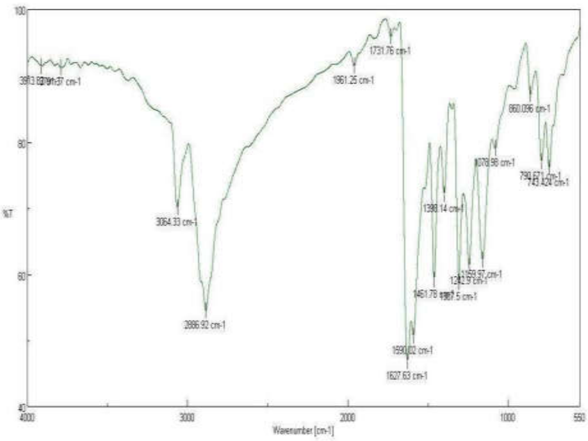

Figure 1, FT-IR spectrum of HNBIA

he FT-IR spectrum of HNBIA showed characteristic bands at 3062 cm^{-1} (N–H stretching), 1627 cm^{-1} (C=N stretching), and 1439 cm^{-1} (C=C aromatic), confirming the formation of the Schiff base linkage

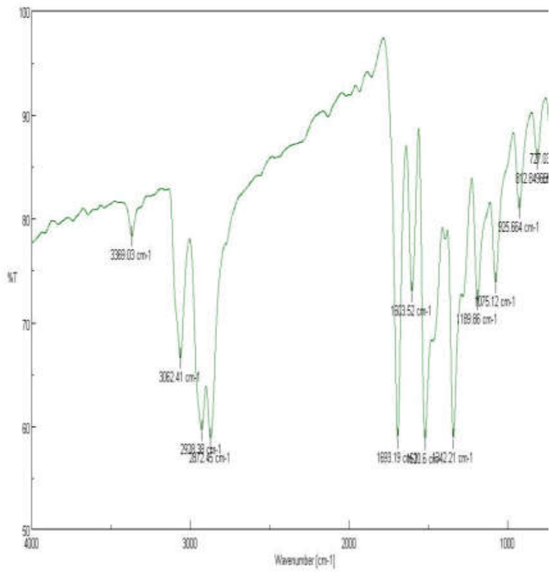

Figure 2. FT-IR spectrum of NBBIA

Here is Figure 2: FT-IR Spectrum of NBBIA, showing major peaks such as 3215 cm⁻¹ (N–H), 1562 cm⁻¹ (C=N), 1462 cm⁻¹ (C=C), and 1269 cm⁻¹ (C–N), confirming the successful formation of the Schiff base structure.

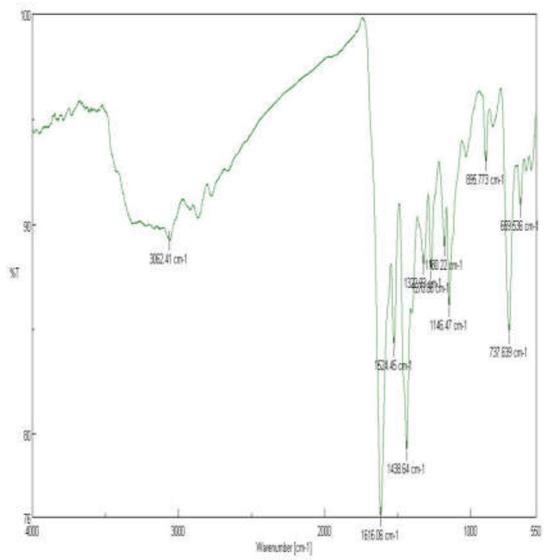

Figure 3. FT-IR spectrum of SBIA

Here is the FT-IR spectrum of SBIA, showing key absorption bands at 3060 cm⁻¹ (N–H), 1693 cm⁻¹ (C=O), 1616 cm⁻¹ (C=N), and 842 cm⁻¹ (aromatic C–H out-of-plane).

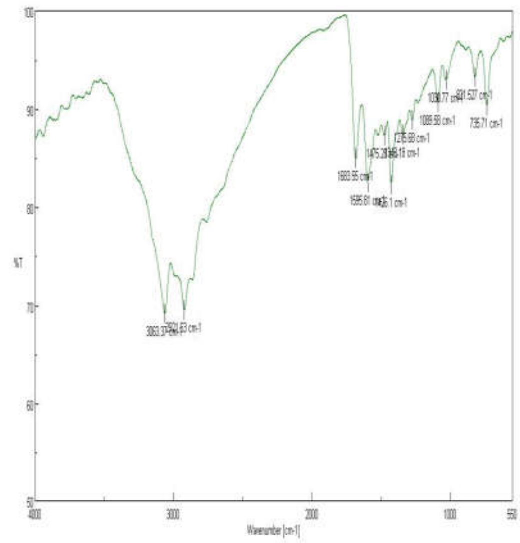

Figure 4. FT-IR spectrum of CBBIA

The FT-IR spectrum of CBBIA shows key absorption bands at 3063 cm⁻¹ (N–H stretching), 1596 cm⁻¹ (C=N stretching), and 1089 cm⁻¹ (C–N stretching). These bands confirm the formation of the Schiff base and the successful incorporation of benzimidazole moiety**.**

Mechanism of Adsorption and Isotherm Models

The adsorption of these Schiff base inhibitors on the mild steel surface followed both Langmuir and Temkin adsorption isotherms, indicating monolayer formation and possible interaction between adsorbed molecules. The flat orientation of the aromatic rings and the presence of multiple donor atoms (N, O) facilitated strong adsorption. The inhibition is believed to result from π-electron interactions and lone pair donation from heteroatoms, forming a protective film over the metal surface.

Effect of Temperature and Thermodynamic Analysis

As temperature increased from 303 K to 333 K, the inhibition efficiency decreased, suggesting a physisorption mechanism. Activation energy (Ea) values were higher for inhibited systems (62.84–94.08 kJ/mol) than for the uninhibited system (41.87 kJ/mol), reinforcing the proposed physisorption or mixed adsorption nature. The calculated free energy of adsorption (ΔG°ads) values were negative and within the range of physical adsorption, indicating spontaneous adsorption of the inhibitors.

Electrochemical Studies

Electrochemicalimpedance spectroscopy (EIS) and potentiodynamic polarization studies further confirmed the inhibitive action. The Nyquist plots showed increased charge transfer resistance (R_ct) and decreased double-layer capacitance (C_dl) with increasing inhibitor concentration, suggesting the formation of a protective layer on the metal surface. Tafel polarization curves indicated that the Schiff bases acted primarily as anodic inhibitors by reducing the anodic dissolution of mild steel.

Synergistic Effect of Halide and Chromate Ions

The addition of halide ions (Cl^-, Br^-, I^-) and chromate ions showed a synergistic effect, enhancing inhibition efficiency. Among the halides, iodide ions (I^-) exhibited the most significant improvement, consistent with their higher polarizability and ability to facilitate inhibitor adsorption. Chromate ions also promoted adsorption, though to a slightly lesser extent than iodides.

Surface Morphology by SEM

SEM analysis of the mild steel surface before and after immersion in acid with inhibitors confirmed the protective effect. The uninhibited sample exhibited severe corrosion with visible pits and roughness, whereas the inhibited samples (especially those with HNBIA and CBBIA) showed smooth, dense films with minimal surface damage. This confirms the role of Schiff bases in forming adherent barrier layers that reduce corrosion activity.

Antimicrobial Activity

The Schiff base ligands were screened for antibacterial activity against *Staphylococcus aureus*, *Escherichia coli*, and *Pseudomonas aeruginosa*. Compounds SBIA and BSBIA demonstrated significant zones of inhibition against all bacterial strains, outperforming the parent compound ABI. Interestingly, only ABI exhibited antifungal activity against *Candida* and *Aspergillus* species, while the Schiff bases were inactive against fungi. The enhanced antibacterial effect of Schiff bases may be attributed to the presence of the imine group (–CH=N–), which is known to enhance lipophilicity and facilitate cell membrane penetration.

CONCLUSION

In this study, a series of benzimidazole-based Schiff base ligands were successfully synthesized using an ultrasound-assisted method, which offered improved reaction rates and yields compared to conventional approaches. Structural confirmation was achieved through FT-IR spectroscopy and elemental analysis, validating the presence of imine and benzimidazole functional groups.

The corrosion inhibition efficiency of the synthesized Schiff bases was thoroughly evaluated on mild steel in 0.5 M H_2SO_4 using both weight loss and electrochemical techniques. Results indicated that the Schiff bases significantly enhanced corrosion resistance, with inhibition efficiencies exceeding 95% at optimal concentrations. Adsorption isotherms and thermodynamic data supported a primarily physisorption-controlled inhibition mechanism. Surface morphology analysis using SEM confirmed the formation of a uniform protective film on the metal surface in the presence of inhibitors.

In addition to corrosion inhibition, the Schiff base ligands exhibited considerable antibacterial activity against both Gram-positive and Gram-negative bacteria. However, antifungal activity was limited, observed only in the parent benzimidazole compound. These dual-functional properties suggest that benzimidazole Schiff bases are promising candidates for industrial applications requiring both corrosion protection and microbial resistance.

FUTURE SCOPE

The synthesized Schiff base ligands demonstrate promising multifunctional behavior; however, further investigations could expand their applicability. Future research could focus on: Development of metal complexes of the synthesized Schiff bases to potentially enhance both corrosion inhibition and antimicrobial activity. Computational modeling such as DFT and molecular docking to predict and optimize molecular interactions with metal surfaces and microbial targets. Environmental compatibility studies to assess biodegradability and toxicity for large-scale industrial applications. Surface coatings incorporating these inhibitors into paints or polymer matrices for long-term corrosion control in marine or acidic environments. Structure–activity relationship (SAR) analysis to fine-tune substituent groups and improve biological efficacy.

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