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Environmental and greener synthesis: Characterization and biological significance of some transition metal complexes

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Abstract

Modern industrialization has led to significant environmental degradation. Microwave-assisted synthesis, a key approach in green chemistry, offers sustainable alternatives for various applications. This technique finds utility across multiple fields, including biotechnology, pharmaceuticals, petrochemicals, plastics, chemicals, and more. Its main uses have been found in analytical chemistry and chemical synthesis. Since microwave dielectric heating has been used successfully in industrial instruments, it is now being used more and more in chemical reactions. Coordination complexes of Co(II), Ni(II), and Cu(II) with the Schiff base obtained from 3-nitrobenzaldehyde and thioacetamide (SB), have been synthesized using both conventional and microwave-assisted methods. These compounds have been characterized through various analytical and spectral techniques such as- elemental analysis, FT-IR spectroscopy, molar conductance measurements, electronic spectra, ESR, magnetic susceptibility, and thermal analysis. The complexes are colored and stable under atmospheric conditions. Analytical data indicate that all the complexes demonstrate a 1:2 metal-to-ligand ratio. Thermal analyses further reveal their degradation patterns and stability. The Schiff base and its metal complexes display significant activity against bacteria such as *S. typhi, E. coli,* and *B. subtilis*, as well as fungi including *A. niger, F. oxysporum* and *C. albicans*. Antimicrobial assessments further suggest that the metal complexes possess superior antimicrobial efficacy compared to the Schiff bases ligand.

Keywords: Microwave Assisted Synthesis; ESR; Thermal Analysis; Antimicrobial Activities

1. Introduction

The chemistry of transition metal ions interacting with living molecules is one of the most interesting parts of coordination chemistry. Inorganic chemistry researchers are still very interested in coordination chemistry, which is the study of how Schiff base ligands combine with metal ions. Coordination chemicals connect molecules that are organic and molecules that are inorganic. It is well known that nitrogen (N) and sulfur (S) atoms play a crucial role in coordination chemistry [1-4].

Increasing industrialization has intensified environmental concerns, highlighting the need for greener synthesis methods. Microwave-assisted synthesis, a branch of green chemistry, offers a much faster reaction rate and higher yields compared to conventional methods. In traditional synthesis, the yield is generally lower, whereas microwave irradiation enhances the reaction rate and yield through effects that are not purely thermal. The salient features of the microwave approach include shorter reaction times, simple reaction conditions, and improved yields [5-9].

In the present study, a novel Schiff base ligand was synthesized from 3-nitrobenzaldehyde and thioacetamide, introducing both electron-withdrawing and sulfur functionalities. The ligand was coordinated with Co(II), Ni(II), and

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Cu(II) ions using both conventional reflux and microwave-assisted techniques to compare efficiency, yield, and structural features. The ligand and its complexes were characterized to confirm their structures and coordination modes. The antimicrobial activity of the synthesized complexes was evaluated against the bacteria *Salmonella typhi, Escherichia coli,* and *Bacillus subtilis,* as well as the fungi *Aspergillus niger, Fusarium oxysporum,* and *Candida albicans.* The results show that these complexes hold potential for both pharmaceutical and environmental remediation.

2. Materials and methods

2.1. Experimental

All the solvent and chemicals were from analytical grade. All compounds were purchased from Sigma Aldrich. Elemental analyses were conducted on Elemental Vario EL III Carlo Erba 1108 analyzer. Electronic spectra in methanol were recorded on Perkin Elmer Lambda-2B Spectrphotometer at Dr. H.S.G.V.V., Sagar (M.P.). Molar conductance measurements were recorded on Elico-CM 82 Conductivity Bridge at room temperature by using 10^{-3} M solutions of the complexes in methanol. Magnetic susceptibility measurements were carried out at room temperature on a Gouy balance by using $Hg[Co(SCN)_4]$ as a calibrant. FT-IR spectra were recorded in KBR pellet on a Perkin Elmer RX1 spectrophotometer at SAIF, CDRI, Lucknow (U.P.) in wave number 4000-400 cm^{-1}. X-band EPR spectra were recorded at room temperature on a Varian E-112 spectrometer by using TCNE as the internal standard at SAIF, IIT Mumbai. Under atmospheric condition at 10°C min^{-1}, a TGA Q500 universal V4.5A TA instrument performed thermogravimetric analysis. For microwave aided synthesis in an open glass vessel, we used a customized microwave oven model 2001 ETB with a rotating tray and 230 V, electricity. At 2450 MHz, 800W of microwave energy was generated. A thermocouple device was used to check the temperature inside the microwave. Microwave procedures were turned on and off to adjust temperature.

2.2. Conventional synthesis of Schiff base ligand (SB)

The ligand (SB) has been derived by adding the methanolic solution of 3-nitrobenzaldehyde with methanolic solution of thioacetamide in equimolar ratio. The reaction product was refluxed on a water bath for about 6 hrs. The condensation product was filtered, thoroughly washed with ethanol and ether, recrystallized and dried in vacuo. The light brown coloured product is recrystallized with ethanol and petroleum ether and purity is checked by TLC using silica gel G. (M.P. 82°C; yield: 65%)

2.3. Microwave method for the synthesis of Schiff base ligand (SB)

The 1:1 ratio of 3-nitrobenzaldehyde and thioacetamide were mixed in a microwave grinder. A microwave oven heated the reaction mixture after adding 3 mL solvent. The reaction was completed in 6 min (short time). The resulting solid was recrystallized with ethanol and dried over anhydrous CaCl2 under reduced pressure. It was kept in a vacuum environment. The progress of the reaction was checked by TLC (yield: 85%).

2.4. Conventional synthesis of Metal Complexes with Schiff base ligand (SB)

Metal complexes were synthesized by adding a methanolic solution of the appropriate metal salt, $MCl_2 \cdot XH_2O$, to a methanolic solution of the Schiff base 3-nitrobenzylidene-thioacetamide (SB) in a 1:2 metal-to-ligand ratio. The mixture was refluxed on a water bath for approximately 7–10 hours and then left to stand overnight. The resulting-colored precipitate was washed sequentially with ethanol and petroleum ether, and dried under reduced pressure over anhydrous $CaCl_2$ in a desiccator. The progress of the reaction was monitored by TLC method (yield: 57–61%).

2.5. Microwave-Assisted Synthesis of Metal Complexes with Schiff base ligand (SB)

The ligand (SB) and the metal salt were thoroughly mixed in a 1:2 (metal-to-ligand) ratio using a grinder. The reaction mixture was then irradiated in a microwave oven in the presence of 4 mL of solvent. Completion of the reaction was achieved within 8–10 minutes. The resulting product was recrystallized from ethanol and ether, and subsequently dried under reduced pressure over anhydrous $CaCl_2$ in a desiccator. Reaction progress and product purity were monitored by thin-layer chromatography (TLC) using silica gel G (yield: 78–82%).

2.5.1. Biological screening: antimicrobial studies

The biological efficacy of a newly synthesized Schiff base ligand (SB) and its metal complexes with Co(II), Ni(II), and Cu(II) ions. The study was carried out to assess their antibacterial and antifungal activities against a panel of microbial strains, including *Salmonella typhi, Bacillus subtilis, Escherichia coli, Fusarium oxysporum, Aspergillus niger,* and *Candida albicans*. The bioassays were conducted at two concentrations: 25 µg/mL and 50 µg/mL, and the results are expressed

in terms of zone of inhibition (mm). Streptomycin and Griseofulvin were used as standard references for antibacterial and antifungal comparisons, respectively.

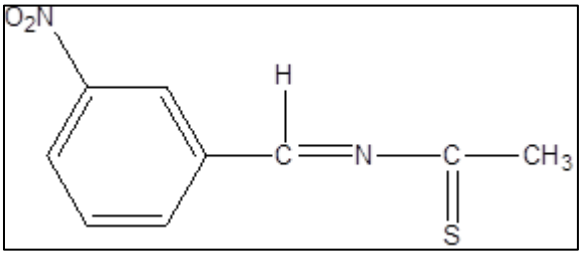

Figure 1 Structure of Schiff Base Ligand

3. Results and discussion

The molar conductance values; Magnetic moment; C, H, N percentage values; Reaction period and Yield percentage of the compounds {Conventional (CM); Microwave Synthesis (MM)} are given in the Table 1.

In microwave-assisted synthesis, the process was completed faster and with higher yields than with the conventional method. It was possible to make the reaction mixture more homogeneous in the microwave method by the rotation of reaction platform tray. It was also checked that the results were correct by the repeating of the process. Comparative studies between the microwave-irradiated synthesis and conventional reflux method show that while the traditional method required 6-10 hours to complete, microwave-assisted syntheses completed within 6-10 minutes with improved yields from 57-65% to 78-85%.

At room temperature all the complexes are coloured, solid, and stable towards air and moisture. They decompose on heating at high temperature and more or less soluble in common organic solvents. Analytical data show that metal complex has 1:2 (metal-to-ligand) stoichiometry. The molar conductance in methanol suggests the Co(II) and Cu(II) non-electrolytic in nature while the Ni(II) complex exhibits electrolytic nature [10, 11].

Table 1 The molar conductance values; Magnetic moment; C, H, N percentage values; Reaction period and Yield percentage of the compounds {Conventional method (CM); Microwave Method (MM)}

Compounds/ Molecular Formulae Mol.Wt [Colour]	**Reaction period**		**Yield (%)**		**Elemental analysis, found (calcd.) %**				**μeff (B.M.)**	**Λ_m (Scm^2 mol^{-1})**
	CM (h.)	MM (min.)	CM	MM	C	H	N	M		
($C_9H_8N_2SO_2$) 208.0 [Orange brown]	6.0	5.6	65	85	51.44 (51.92)	3.29 (3.84)	13.61 (13.46)	-	-	-
[Co($C_9H_8N_2SO_2$)$_2$Cl$_2$]3H$_2$O 599.93 [Greenish black]	10	9.6	57	81	36.44 (36.00)	3.38 (3.66)	9.97 (9.33)	9.21 (9.82)	5.12	38.5
[Ni($C_9H_8N_2SO_2$)$_2$]Cl$_2$.3H$_2$O 599.71 [Coffee brown]	8.6	8.0	61	78	36.56 (36.01)	3.32 (3.66)	9.01 (9.33)	9.41 (9.78)	Dia.	147.0
[Cu($C_9H_8N_2SO_2$)$_2$Cl$_2$]3H$_2$O 604.54 [Brown]	7.3	8.7	60	82	37.46 (35.72)	3.49 (3.64)	9.52 (9.26)	10.24 (10.51)	1.99	32.9

3.1. IR spectra

The IR spectra of the complexes were compared with that of the free ligand to identify the coordination sites involved in chelation. Characteristic peaks in both the ligand and the complexes were examined and compared.

A sharp band at 1646 cm^{-1} in the IR spectrum of the ligand (SB) is owing to ν(C=N) group (Schiff base). This band shifts towards lower frequency side (1615$\pm$5 cm^{-1}) in complexes, on coordination through azomethine nitrogen. A band due to ν(C=S) stretching vibrations in free ligand appears at about 1540 cm^{-1}. It has shifted towards lower frequency side in

complexes by 20-30 cm^{-1} with reduced intensity. This suggests the involvement of S-atom in coordination. The ligand (Schiff base) is bidentate. The new bands at 470±10$cm^{-1}$and 403±4 cm^{-1} have been assigned to ν(M-N) and ν(M-S) modes, respectively [12-15].

3.2. Electronic spectra and magnetic moment

The nature of the ligand field surrounding the metal ion was elucidated from the electronic absorption spectra, which were recorded at room temperature using methanol as the solvent. The electronic spectrum of Co(II)-complex shows two bands at 15352 cm^{-1} and 20428 cm^{-1} which are assignable to $^4T_{1g}(F)$-$^4A_{2g}(F)(\nu_2)$ and $^4T_{1g}(F)$-$^4T_{1g}(P)(\nu_3)$ respectively. The various ligand field parameters, 10Dq, B, β, λ, ν_2/ν_1 and LFSE have been calculated and the values are as: 8277 cm^{-1}, 970 cm^{-1}, 0.86, (-)654 cm^{-1}, 2.16 and 79.08 kJ mol^{-1} respectively. The magnetic moment is 5.12 B.M. This favours the geometry to be octahedral. The Ni(II)-complex exhibits two bands at 13212 $cm^{-1}$and 18682 cm^{-1}; these are assigned to $^1A_{1g}$-^1E_g (ν_1) and $^1A_{1g}$-$^1B_{2g}$ (ν_2) transitions, respectively. Since the complex is diamagnetic, the square planar geometry has been suggested for this complex. The Cu(II)-complex gives a broad band at 14292 cm^{-1} corresponding to transition ^2E_g-$^2T_{2g}$. The value of ligand field parameters 10Dq, λ and LFSE comes to be 14292 cm^{-1}, (-)1073 cm^{-1} and 102.4 kJ mol^{-1} respectively. The magnetic moment is 1.99 B.M. These parameters suggest the octahedral geometry for this Cu(II)-complex [16-19].

3.3. ESR spectra

The X-band ESR spectra of Cu(II) complexes were recorded in the solid state at room temperature and their $g_\parallel$, $g_\perp$, Δg, g_{av} and G have been calculated. The values of ESR parameters $g_\parallel$, $g_\perp$, g_{av}, Δg and G for Cu(II) complex of SB are 2.2298, 2.1701, 0.0597, 2.1900 and 1.3551, respectively.

ESR spectra of the complexes revealed two g values ($g_\parallel$ and $g_\perp$). Since the $g_\parallel$ and $g_\perp$ values are closer to 2 and $g_\parallel > g_\perp$ suggesting a tetragonal distortion around the Cu(II) ion. The trend $g_\parallel > g_\perp > ge$ (2.0023) shows that the unpaired electron is localized in $d_{x^2-y^2}$ orbital in the ground state of Cu(II) and spectra are characteristic of axial symmetry.

The exchange coupling interaction between two Cu(II) ions is explained by Hathaway expression $G = (g_\parallel - 2.0023)/(g_\perp - 2.0023)$. According to Hathaway, if the value G is greater than four (G>4.0), the exchange interaction is negligible; whereas when the value of G is less than four (G<4.0) a considerable exchange coupling is present in solid complex. The G values for the Cu(II) complex are less than four indicating, considerable exchange interaction in the complexes [20,21].

3.4. Thermal Study

The thermal behavior of the metal complexes indicates that the hydrated complexes first lose molecules of hydration, followed by decomposition of the ligand molecules in subsequent steps. The TGA curve of the $[Co(C_9H_8N_2SO_2)_2Cl_2].3H_2O$ complex shows a mass loss between 90–160°C, corresponding to the removal of three lattice water molecules (remaining wt. %, Obs./Calc., 89/90.56). An inflection in the curve is observed between 230–380°C, attributable to the loss of ligand molecules (remaining wt. %, Obs./Calc., 23/16.67). Between 380–400°C, there is negligible change in weight; however, above 400°C, mass loss accelerates up to 450°C, after which a constant weight region is recorded. The final weight of the residue corresponds to a mixture of cobalt oxides (remaining wt. %, Obs./Calc., 17/13.11). The slight discrepancies between observed and calculated weight values may be due to residual carbonaceous material [22,23].

3.5. Biological Screening

The microbial resistant ability of the synthesized compounds illustrated; the free ligand (SB) demonstrated only moderate activity against all tested organisms, with inhibition zones ranging from 10 to 17 mm. However, upon coordination with transition metal ions, the antimicrobial activity was significantly enhanced. Among the complexes, the Cu(II) complex exhibited the most potent antimicrobial performance. Against bacterial strains, it showed inhibition zones up to 25 mm for *S. typh*i, 24 mm for *B. subtilis*, and 24 mm for *E. coli* at 50 μg/mL. The Co(II) complex followed closely with notable activity, while the Ni(II) complex showed relatively moderate antibacterial effects. The enhanced activity of these metal complexes, especially at higher concentration, reflects a dose-dependent response and suggests improved bioavailability upon metal coordination.

In the antifungal evaluation, the Cu(II) complex again outperformed others, showing inhibition zones of 28 mm against *F. oxysporum*, 25 mm against *A. niger*, and 25 mm against *C. albican*s. Co(II) and Ni(II) complexes also displayed considerable antifungal potential, though slightly less than Cu(II). The results were comparable to those of the standard antifungal drug Griseofulvin, which showed zones up to 32 mm (Table 2). The enhanced biological activity of the metal complexes over the free ligand can be attributed to the chelation effect, which reduces the polarity of the metal ion

through partial sharing of its positive charge with donor atoms, thereby increasing the lipophilicity of the complex and its ability to penetrate microbial cell membranes.

The bactericidal and fungicidal investigation data of the compounds are summarized in Table 2. The results of the investigations account for the antipathogenic behavior of the compounds and this efficacy is positively modified on complexation. Overtone's Concept and Chelation Theory explain well this drug action [24-27].

Table 2 Antimicrobial screening data for the ligand and its metal complexes (*concentration in ppm)

Compound	**Antibacterial**						**Antifungal**					
	S. typhi		*B. subtilis*		*E. coli*		*F. oxysporum*		*A. niger*		*C. albicans*	
	25*	50*	25*	50*	25*	50*	25*	50*	25*	50*	25*	50*
SB	12	16	10	13	10	15	12	17	11	13	10	14
Co (II)	19	24	16	22	15	20	22	30	18	24	20	26
Ni (II)	17	21	16	20	12	16	20	25	17	22	16	24
Cu (II)	20	25	18	24	13	18	23	28	20	25	17	25
Streptomycin	22	28	20	27	14	22	-	-	-	-	-	-
Griseofulvin	-	-	-	-	-	-	26	32	24	28	22	29

4. Conclusion

The present study reports the successful synthesis and characterization of Co(II), Ni(II), and Cu(II) coordination complexes with a Schiff base (SB) derived from 3-nitrobenzaldehyde and thioacetamide, using both conventional reflux and microwave-assisted methods. From an environmental perspective, microwave-assisted synthesis offers significant advantages. Comprehensive physicochemical and spectroscopic analyses confirmed the formation of stable, colored complexes with a 1:2 metal-to-ligand stoichiometry. Thermal analysis revealed the stability and degradation patterns of the complexes. Biological screening (antimicrobial studies) demonstrated that metal complexation markedly enhanced the bioactivity of the ligand, with the Cu(II) complex showing the highest antibacterial and antifungal efficacy, often comparable to that of standard drugs. These findings highlight the potential of Schiff base metal complexes, particularly Cu(II), as promising compound in the development of effective antimicrobial agents.

Compliance with ethical standard

Acknowledgments

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Disclosure of conflict of interest

Authors have no conflict of regarding this paper.

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