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Eco-friendly, Synthesis, Characterization, and Antimicrobial Studies of Transition Metal Complexes of Schiff Base Ligand Derived from 1H-indole-3-carbaldehyde with 4-nitrobenzoydrazide

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Abstract: ***Rapid, efficient, clean and environmentally benign exclusive synthesis of Schiff base ligand and their transition metal complexes have been developed using condensation of 1H-indole-3-carbaldehyde with 4-nitrobenzoydrazide efficiently in an alcoholic suspension medium using alkali catalyst with excellent yields under microwave irradiation. The present work, microwave assisted green synthesis of novel Schiff base ligand (E)-N'-((1H-indol-3-yl)methylene)-4-nitrobenzohydrazide and its transition metal complexes of Co(II), Ni(II), Cu(II), Zn(II),and Ag(I) were prepared under microwave irradiation as a green approach method. The novel Schiff base ligand was known by melting point and thin layer chromatography. Characterization was by elemental analysis, Infrared spectra, ^{1}HNMR spectra and mass spectroscopy. The metal complexes were recognized by melting point, thin layer chromatography and their distinct colour. Metal complexes were characterized by Infrared spectra, UV-visible spectra and thermogravimetric analysis. The synthesized Schiff base ligand and its metal complexes were screened for their antibacterial activity against bacterial species Escherichia coli, Staphylococcus aurous and Salmonella Typhi.***
Keywords: Microwave synthesis, 4-nitrobenzoydrazide, Thermal Study, Schiff Base Ligand, Antibacterial Activity.

I. INTRODUCTION

In the field of coordination chemistry Schiff base ligands have been widely studied mainly due to their facile syntheses, easily availability, electronic properties and good solubility in common solvents and they easily form stable complexes with most transition metal ions [1-2].

The chemistry of metal complexes with Schiff base ligands and their application have used broad attention, mainly because of preparative accessibility, diversity and structural variability [3]. In coordination chemistry play an important role of Schiff base ligands containing O and N donor atoms related to catalysis and enzymatic reaction, magnetism and molecular architecture. Metal complexes with Schiff base ligands containing 1H-indole-3-carbaldehyde and its nitrobenzoydrazide derivatives; have been extensively studied.

Metal complexes with such ligands are fairly common and also reveal their superficial synthesis, accessibility of diverse structural modifications and inclusive applications in different fields, such as catalysis, biological systems and material chemistry [4-5].Microwave reactions under solvent free and less solvent conditions are attractive offering reduced pollution, low cost and offer high yields together with simplicity in processing and handling [6-7]. The salient features of microwave approach are shorter reaction times, simple reaction conditions and enhancements in yields [8-9]. Schiff bases belong to a broadly used group of organic intermediates important for manufacture of specialty chemicals, e.g. pharmaceuticals, or rubber add itives [10-11] and as amino protective group's inorganic synthesis [12-13]. They also have uses as liquid crystals [14] and in analytical [15] medicinal [16] and polymer chemistry [17]. Such as antifungal, antibacterial, antitumor, anti-inflammatory and antipyretic among others. Some of them have been used as complexing agents [18] and powerful corrosion inhibitors [19].

II. MATERIALS AND METHODS

All the analytical grades, reagents, and chemicals were used without further purification. According to the literature method solvents were purified and dried. All chemicals were obtained from Sigma-Aldrich and used without purification.1H-indole-3-carbaldehyde with 4-nitrobenzoydrazide; the remaining all chemical solvents were purchased from Spectrochem Ltd.

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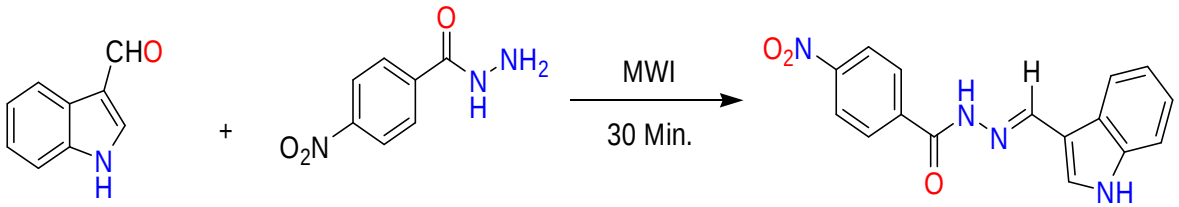
A. Physical Measurements

Elemental analysis (C, H, N) was implemented using the Perkin Elmer CHN analyzer. IR spectra of the ligands and their metal complexes were recorded on Bruker spectrometer within the range of 4000-400 cm^{-1}. Thermal studies of the complexes were carried out using a Perkin Elmer diamond TGA instrument. ^{1}H-NMR spectra of the ligands were recorded on Bruker spectrometer using DMSO-d6 as a solvent and TMS as an internal standard. Mass spectra were recorded on water, Qt of micromass (ESI-MS).

B. Synthesis of Schiff base ligand.

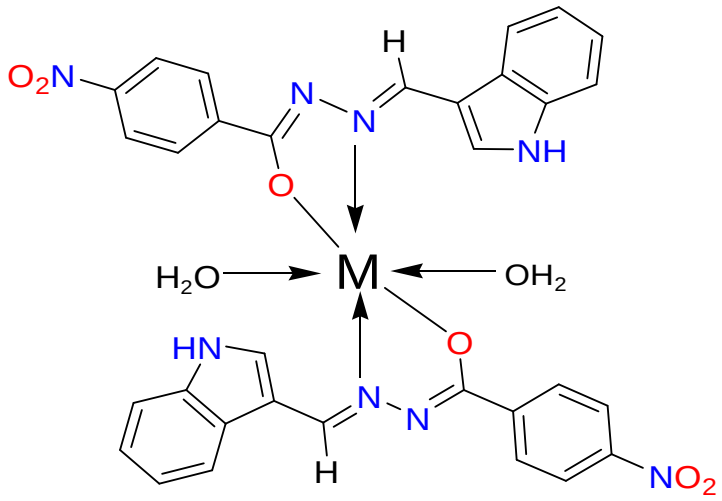
The Schiff base ligand has been synthesized by reacting 1H-indole-3-carbaldehyde (1.45gm) (1.00 mmole) and with 4-nitrobenzoydrazide (1.81 gm) (1.00 mmole). The reaction was carried out in a microwave oven for 15-18 minutes. The microwave irradiated product was washed with dry ether and filtered. The final product was recrystallized from ethanol to give yellow crystals. The product's purity was monitored by using TLC, n-hexane, and ethyl acetate (7:3) (Scheme 1).



1*H*-indole-3-carbaldehyde 4-nitrobenzohydrazide (*E*)-*N*'-((1*H*-indol-3-yl)methylene)-4-nitrobenzohydrazide

C. Synthesis of Metal Complexes

The complexes were synthesized by mixing the appropriate metal nitrate with the required amount of the ligand in a 1:2 metal to ligand ratio. The reaction mixture was irradiated in a microwave oven at 750 W for 120-183 second. The final products were washed with hot ethanol, filtered and dried at room temperature. The metal salts used were Co $(NO_3)_2$.$6H_2O$, Ni $(NO_3)_2$.$6H_2O$, Cu $(NO_3)_2$.$3H_2O$, Zn $(NO_3)_2$.$6H_2O$ and Ag $(NO_3)_2$.$4H_2O$.



III. RESULT AND DISCUSSION

All transition metal complexes and novel Schiff base ligand are colored, solid and stable at room temperature. They possess sharp melting point. The complexes are soluble in dimethyl formamide and dimethyl sulfoxide but insoluble in common organic solvents.

A. Elemental Analysis

The elemental analysis of Schiff base ligand (Found: C= 62.23; H = 3.48; O=15.56; N= 18.20%); (Calculated: C = 62.33; H = 3.92; O=15.57; N =18.17; %) indicated that the ligand has the molecular formula i.e. $C_{16}H_{12}N_4O_3$.

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B. Physical Properties

Physical properties of the novel ligand and metal complexes summarized in Table-1

Sr. No	Molecular formula	Colour	M.P.(^{0}c)	Time	Yield %
1	$C_{16}H_{12}N_4O_3$ (Ligand)	Orange	326	30 min	92
2	$[(C_{16}H_{12}N_4O_3)_2(H_2O)_2]Co$	Yellow	264	132 sec.	94
3	$[(C_{16}H_{12}N_4O_3)_2(H_2O)_2]$ Ni	Greenish	263	178 sec.	87
4	$[(C_{16}H_{12}N_4O_3)_2(H_2O)_2]$ Cu	Light Green	273	128 sec.	91
5	$[(C_{16}H_{12}N_4O_3)_2(H_2O)_2]$ Zn	Green	258	157 sec.	89
6	$[(C_{16}H_{12}N_4O_3)_2(H_2O)_2]$ Ag	Yellow	276	164 sec.	90

C. Infrared Spectral Analysis

The IR spectrum of novel Schiff base ligand show the characteristics band at 1648 cm^{-1} which assigned to (C=N) stretching vibration, which indicates the presence of azomethine group in the ligand [20]. The spectrum show vibrational band at 3375 cm^{-1} indicates (N-H) stretching in the ligand. The stretching at 1577cm^{-1} corresponds to aromatic (C=C) bonding in the ligand. The band observed at 2996 cm^{-1} indicates aromatic (C-H) stretching. Ni complex the band observed at 1618 cm^{-1} attributed to the stretching vibration of azomethine (C=N) group, whereas in spectrum of ligand same band is observed at 1618cm^{-1} [21]. The bands observed at 2962 cm^{-1}, 3442 cm^{-1} and 1542cm^{-1} were assign to aromatic (C-H) stretching, (N-H) stretching vibration and aromatic (C=C) stretching respectively, The most characteristics bands appeared at 480 cm^{-1} was due to (M-N) stretching, which confirms the formation of metal ligand bonding . The weak bands observed at 1030 cm^{-1} and 895 cm^{-1} were due to OH wagging mode of vibration, indicating coordination of water molecule in metal complex [22].

whereas same bands are observed of Cu Complex at 2968 cm^{-1}, 3420 cm^{-1} and 1556 cm^{-1} in spectrum of ligand respectively. The most characteristics bands appeared at 475 cm^{-1} was due to (M-N) stretching, which confirms the formation of metal ligand bonding [23]. The weak bands observed at 1050 cm^{-1} and 890 cm^{-1} were due to OH wagging mode of vibration, indicating coordination of water molecule in metal complex [24]. Above bands which are appeared in spectrum of complex are not appeared in spectrum of ligand that confirm the formation of metal complex with stable metal ligand bonding [25].

Table 2: Selected Infrared Frequencies (cm^{-1}) of ligand and its complexes

Ligand/ Complexes	υ(C=N) Azomethine	Aromatic C-H cm^{-1}	υ(N-H) cm^{-1}	Aromatic C=C cm^{-1}	υ(M-N) cm^{-1}
$C_{16}H_{12}N_4O_3$ [L]	1648	2996	3375	1577	-
$[Ni(L)_2]$	1618	2962	3442	1542	480
$[Cu(L)_2]$	1620	2968	3420	1556	475

D. ^{1}HNMR spectral studies

Observed ^{1}H NMR peaks (ppm) of novel Schiff base ligand shows different peaks. The characteristic peak observed at 8.60 ppm is due to H from azomethine group [26]. Peak observed at 11.40 ppm is due to H-from of indole ring. The peak observed at 11.70 ppm is due to H-from of CO-NH group. The peaks observed at 8.26 -7.97 ppm are due to H-from aromatic rings.

E. Mass Spectral Studies

The mass spectrum study of novel Schiff base ligand showed a peak at m/z. 310 (M+2) that corresponds to the molecular weight of the Schiff base ligand 308.

F. Electronic Spectra.

Electronic Spectra UV-Vis spectrum of both metal complexes Ni(II), Cu(II) recorded in the wavelength region 200nm to 400nm in DMSO solution[27]. UV-Vis spectral data of Ni: Electronic spectrum of Ni(II) complex shows absorption maxima at 41666 (240), 43859 (228) and 46296 (216) assign to $^3A_2g \rightarrow {}^3T_2g(F)$, $^3A_2g \rightarrow {}^3T_1g(F)$ and $^3A_2g \rightarrow {}^3T_1g(P)$ transitions respectively indicating that complex possess octahedral geometry [28]. UV-Vis spectral data of Cu: Electronic spectrum of Cu (II) complex shows absorption maxima at 42016 (238) and 44642 (224) assign to $^2B_1g \rightarrow {}^2A_1g$,
$^2B_1g \rightarrow {}^2B_2g$ and $^2B_1g \rightarrow {}^2Eg$ transitions showing that complex possess octahedral geometry [29].

G. Thermo Gravimetric Analysis

Thermal analysis was used mainly for the confirmation of the water molecule or solvent associated with being in the sphere of coordination or in the outer sphere of the complex [30] and the information about its properties, the nature of the product's intermediate and final thermal decomposition. From the TGA curves, the weight loss was calculated for the different steps and compared with the theoretically calculated weight for the suggested formulas based on the results obtained from the elemental analyses [31]. Thermal stability of the synthesized metal complexes was done up to 600°C at a heating rate of 10°C/min in a nitrogen atmosphere.

Metal complexes exhibit similar decomposition patterns, as is evident from their TGA graphs. TGA graph shows that decompose of Ni metal complex in three steps within temperature range of 10-600 °C. First step corresponds to the loss of two coordinated water molecules (Found 4.10%, calcd. 4.62 %) in temperature range of 10-190 °C. The second step corresponds to the loss of $(C_{12}H_9N_3O_3)_2$ (Found 69.78 %, calcd. 73.80 %) in temperature range of 200 - 445 °C. A peak corresponding to mass loss of (20.58% calcd.21.02 %) at 445-600 °C was due to the loss of $(C_4H_3N)_2$ the third step and as a final product, it leaves NiO as residue [32].

The TGA graph shows the decomposition of the Cu metal complex in three steps within a temperature range of 10-650°C. The first step corresponds to the loss of two coordinated water molecules (Found 3.23%, calc.4.03 %) in the temperature range of 10-80°C. The second step corresponds to the loss of $(C_{12}H_9N_3O_3)_2$ (Found 69.06 %, calc. 74.82 %) in the temperature range of 200-430°C. A peak corresponding to mass loss of (19.08% calcd.20.28 %) at 430-590°C was due to the loss of $(C_4H_3N)_2$ in the third step, and as a final product, it leaves CuO as residue [33].

H. Antimicrobial Activity

The in vitro antimicrobial screening of synthesized ligand and metal complexes was tested against three bacteria S. Aureus, E. Coli and S. Typhi

The metal complexes exhibit higher inhibition against tested microorganisms compared to the free ligand. The value in the above table indicates that the activity of the Schiff base ligand became more pronounced when coordinated with the metal ions. The presence of azomethine moiety and chelation effect with central metal enhances the antibacterial activities [34-35]. This enhancement in the antibacterial activity of these metal complexes can be explained based on the chelation theory.

Ni(II) ,Zn(II) complexes are shows better antibacterial activity on E.coli and Co(II), Cu(II), Ag(I) good antibacterial activity on E.coli as compared to rest of metal complexes and parent ligand [36]. Cu(II) complex shows excellent antibacterial activity on S.Aureus as compared to rest of metal complexes and parent ligand. Co(II) and Zn(II) shows better antibacterial activity on S.Typhi as compared to rest of metal complexes and parent ligand [37].

The antimicrobial activity of ligand and metal complexes are shown in Table 3.

Table 3: Antibacterial activity of Novel ligand and their metal complexes

Sr.No.	Compounds	Minimal inhibition Concentration (µg/ml)		
		E.Coli	S.Aureus	S.Typhi
1	Ligand	100	50	75
1	Co(II) Complex	150	250	50
2	Ni(II) Complex	50	100	125
3	Cu(II) Complex	100	25	250
4	Zn(II) Complex	50	100	50
5	Ag(I) Complex	75	125	100

IV. CONCLUSION

Metal complexes with Co(II), Ni(II), Cu(II) ,Zn(II), and Ag(I) ions were synthesized using the resynthesized ligand (E)-N'-((1H-indol-3-yl)methylene) -4-nitrobenzohydrazide. The ligand (L) and its metal complexes were analyzed using TLC, melting point determination, FT-IR spectroscopy, 1H-NMR spectroscopy, UV-Vis spectroscopy, and thermo gravimetric analysis. Complexes exhibit octahedral stereochemistry based on UV-Vis spectroscopy.

When a metal ion is chelated with a ligand, its polarity will be reduced to a greater extent due to the overlap of ligand orbital and the partial sharing of the positive charge of the metal ion with donor groups. Furthermore, the chelation process increases the delocalization of the π-electrons over the whole chelate ring, which results in an increase in the lipophilicity of the metal complexes.

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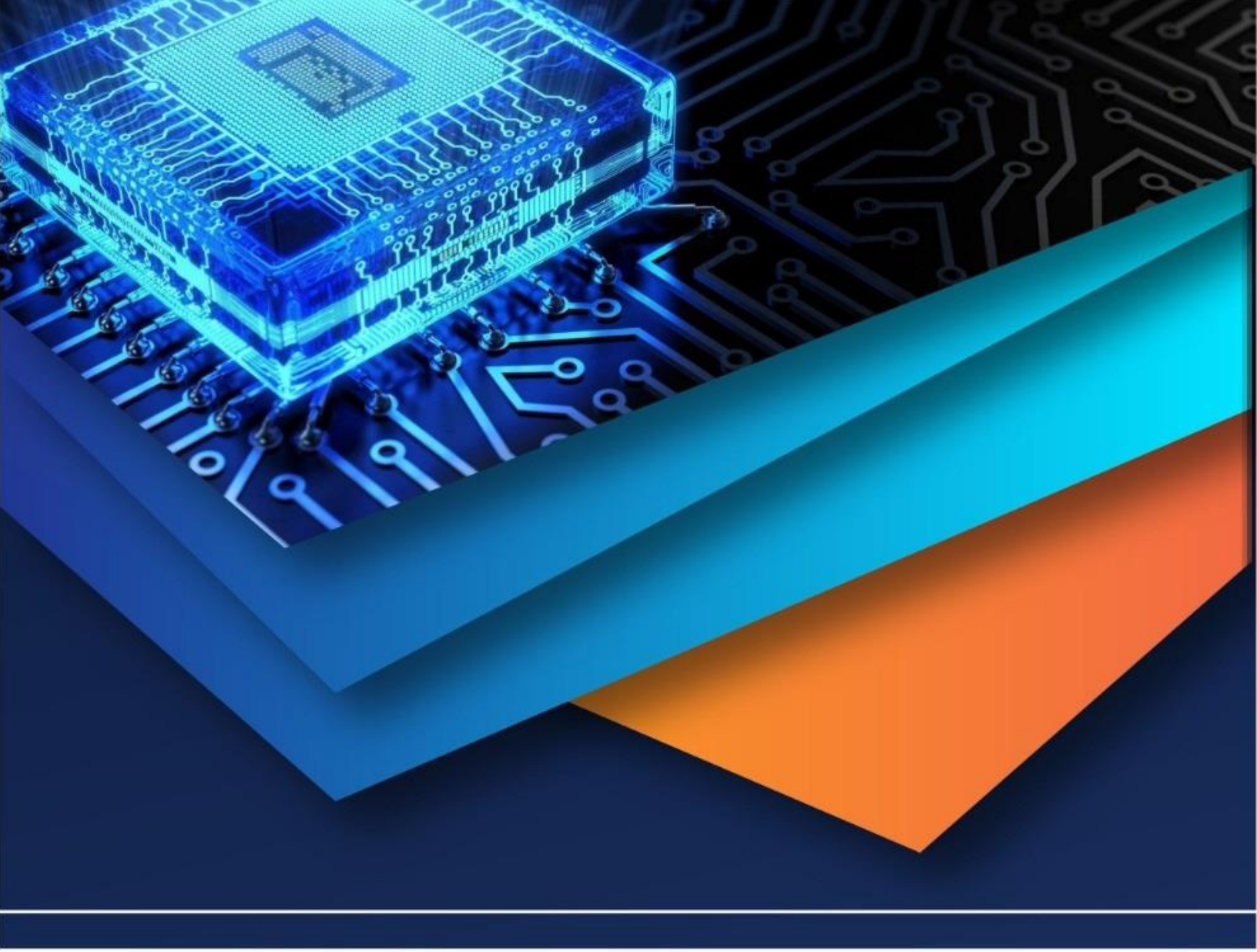

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