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Research Article

Solvent Free Synthesis and Characterization of Few Metal Complexes of Schiff Base Derived from 2-Amino-5, 6-dimethyl Benzimidazole and Syringaldehyde

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ABSTRACT

A solvent-free synthesis of bidentate Schiff base ligand was carried out by treating 2-Amino-5, 6-dimethyl benzimidazole with Syringaldehyde in 1:1 molar proportion in a scientific microwave oven. The excellent yield was obtained after purification. Few metal complexes of Schiff base were synthesized by using Mn(II), Fe(III), Co(II), Ni(II), Cu(II), Zn(II), Cd(II), and Ag(I) metal salts in microwave oven under solvent-free condition. All metal complexes showed distinct color at the end of the reaction, and the melting point of each complex preliminary confirmed the formation of the product. A detailed characterization of both Schiff base and its representative metal complexes was carried out by several analytical and spectral techniques, including elemental analysis, Infrared (IR) spectroscopy, ^{1}HNMR spectroscopy, liquid chromatography–mass spectrometry (LCMS), UV spectroscopy, and TGA. The spectroscopic analysis supports the predicted structure of the parent Schiff base ligand. Bioactivity of Schiff base & respective metal complexes was studied against *Escherichia coli*, *Staphylococcus aureus* and *Salmonella typhi* showing significant bioactivity of the metal complexes and Schiff base ligand.

Introduction

Schiff bases are chemically imine molecules with common structural formula $R_1R_2C=NR_3$ where R_1, $R_3 \neq H$. Hugo Schiff in 1864 synthesized such kind of molecule first time and then this class of compounds named as Schiff base.[1,2] Schiff bases further treated with many metal salts yields metal complexes, and both these Schiff bases and their metal complexes have vast applications in drug synthesis.[3] The azomethine group present in Schiff base can form highly stable complexes with transition metal ions. The 'N' atom in the azomethine group has lone pair of electrons, which enables stable transition metal complexes by occupying vacant '*d*' orbitals of metal ions.[4,5] Present work fundamentally focuses on solvent free synthesis of Schiff base and its metal complex using the scientific micro oven. It is observed that Microwave-assisted synthesis is low cost, less pollutant emitting, and time-consuming method.[6,7] Microwave-assisted synthesis also has the advantage of higher yield, low accident probability, and environmentally safe.[8-10] Schiff base and their metal complexes show significant biological activities like antiviral, antipolio, antibiotic, anticancer, antituberculastic, fungicidal, insecticidal, etc.[11-15] The principal pharmaceutical advantage of the present study is that the heterocyclic Schiff bases and their metal complexes can be structurally modified to

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anticancer agent, characteristic antimicrobial agents being remarkable pharmacophores and precursors of drug molecules.

Materials and Methods

Material Methods

All chemicals were purchased from Sigma Alderich, Loba Chem, and Merck. In addition, 2-Amino-5, 6-dimethyl benzimidazole, syringaldehyde were purchased from Sigma Alderich and metal nitrates from Loba Chem and Merck. All chemicals were used as received. Parent Schiff base ligand was synthesized by condensing 2-Amino-5, 6-dimethyl benzimidazole, and syringaldehyde in the 1:1 molar ratio in a scientific microwave oven. After ligand synthesis, metal complexes were synthesized by treating ligand with transition metal salts in a same scientific microwave oven.

Techniques

All syntheses were carried out in a scientific oven, 2450 MHz frequency, 800 W. All melting points were recorded on digital melting point apparatus. The electronic absorption spectra were recorded in the wavelength range 200 to 800 nm in DMSO solution using UV-VIS spectrophotometer. The IR spectra were recorded on a Schimadzu Dr-8031 instrument. ^{1}HNMR spectrum was recorded on Bruker's 400 MHz instrument in DMSO-d6. The mass spectrum was recorded by LC-MS spectrophotometer. The TGA was carried out in dynamic nitrogen atmosphere (30 mL/min) with a heating rate of 10/min using Shimadzu TGA–50H thermal analyser. Thin layer chromatography (TLC) analyses were performed on pre-coated aluminium plates with silica gel. TLC spots were visualized in UV chamber.

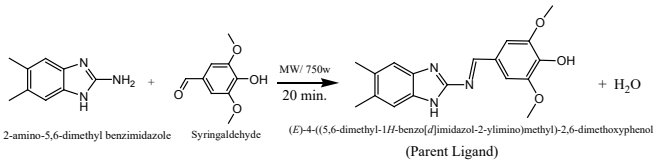

Fig. 1: Synthesis route of parent ligand

Synthesis of New Parent Schiff Base Ligand

The parent Schiff base ligand was prepared by the reaction between 2-amino-5, 6-dimethyl benzimidazole [1.62gm, 0.01 mol], and syringaldehyde [1.82 gm, 0.01mol] under solvent-free condition. The reaction mixture was first mixed in a grinder and then irradiated about 20 minutes periodically at 750 W in a microwave oven. The irradiated microwave product was then kept at room temperature and then washed with dry ether. The final product was then recrystallized using absolute ethanol to give brown crystals. The yield obtained was 3.10 gm (95%) and the melting point recorded was 120. The product's advancement and pureness was examined using TLC, and a solvent mixture used was n-hexane + ethyl acetate (7:3) (Table 1).

Syntheses of Metal Complexes

The metal complexes were also prepared by the same microwave method under solvent free conditions. First, the appropriated metal salt was mixed with the required amount of parent ligand, (*E*)-4-((5,6-dimethyl-1-*H*-benzo[*d*] imidazole-2-yliino) methyl)-2, 6-dimethoxyphenol, in a grinder to mix thoroughly. Then the mixture was irradiated for few seconds to minutes at 750 W. The product obtained was washed and recrystallized with hot ethanol, then dried at room temperature. The melting point of each complex was recorded. The metal salts used were $MnCl_2$, $Fe(NO_3)_3.9H_2O$, $Co(NO_3)_2.6H_2O$, $Ni(NO_3)_2.6H_2O$, $Cu(NO_3)_2.3H_2O$, $Zn(NO_3)2.6H_2O$, $Cd(NO_3)_2.4H_2O$ and $AgNO_3$. (Fig. 1 & Table 2).

Result and Discussion

A few important facts were observed at the end of the microwave-assisted solvent-free synthesis of Schiff

Table 1: The elemental analysis (CHNO) data for new parent Schiff base ligand

Compound	*Empirical formula*	*Molecular weight*	*% C Found (calculated)*	*% H Found (calculated)*	*% N Found (calculated)*	*% O Found (calculated)*
Parent Schiff base ligand	$C_{18}H_{19}O_3N_3$	325.27	62.56 (66.4)	6.14 (5.84)	12.52 (12.91)	18.78 (14.85)

Table 2: Details of physical properties of the new ligand and its metal complexes

Sr. No.	*Molecular Formula*	*Colour*	*M.P. (°C)*	*Time*	*Yield (%)*
1	$C_{18}H_{19}O_3N_3$	Brown	120	20 minutes	95
2	$[(C_{18}H_{19}O_3N_3)_2(H_2O)_2]Fe$	Dark green	210	30 seconds	84
3	$[(C_{18}H_{19}O_3N_3)_2(H_2O)_2]Mn$	Brownish yellow	77	120 seconds	90
4	$[(C_{18}H_{19}O_3N_3)_2(H_2O)_2]Co$	Brown	149	60 seconds	81
5	$[(C_{18}H_{19}O_3N_3)_2(H_2O)_2]Ni$	Greenish	310	30 seconds	80
6	$[(C_{18}H_{19}O_3N_3)_2(H_2O)_2]Cu$	Dark brown	110	30 seconds	91
7	$[(C_{18}H_{19}O_3N_3)_2(H_2O)_2]Zn$	Reddish brown	182	40 seconds	88
8	$[(C_{18}H_{19}O_3N_3)_2(H_2O)_2]Cd$	Brownish	323	90 seconds	87
9	$[(C_{18}H_{19}O_3N_3)_2(H_2O)_2]Ag$	Yellow	143	60 seconds	95

base and its metal complexes, like shorter reaction time and higher yield. In this method, homogeneousness of the reaction mixture was raised by a rotating reactor keeping tray. The results obtained were confirmed by repeating the procedure twice. The entire procedures were completed within a few seconds to a few minutes, and higher yield nearly and above 90%. The metal complexes obtained show absolute color and sharp melting points. All the synthesized complexes were found stable and in a solid-state at room temperature. The synthesized complexes were found insoluble in a common organic solvent but were soluble in Dimethylsulfoxide (DMSO) and Drug Master File (DMF). The parent ligand and its eight metal complexes exhibit significant biological activity.

Elemental Composition Analysis and Physical Properties

Infrared Spectra Analysis

Analysis of parent Schiff base ligand: The IR spectra of parent Schiff base ligand showed a most characteristic band at 1674.21 cm^{-1} due to azomethine, υ(C=N),[16] stretching. Also, a band at 3452.58 cm^{-1} showed due to NH stretching of benzimidazole moiety.[16,17] The parent ligand spectrum showed bands at 3774.69 cm^{-1} and 1348.24 cm^{-1} due to stretching and deformation of phenolic OH, whereas the band at 1126.43 cm^{-1} showed due to phenolic υ(C-O).[17,18] These bands confirmed the formation of the parent ligand.

Analysis of metal complex (L-Ni): The IR spectrum of L-Ni complex showed a shift in frequency of azomethine, υ(C=N), stretching from 1674.21 to 1681.93 cm^{-1} as compared to parent ligand.[19-21] Also, band due to phenolic OH stretching shifted from 3774.69 to 3640 $cm^{-1,}$ and phenolic υ(C-O) stretching shifted from 1126.43 to 1033.85 cm^{-1}. The most characteristic bands of this metal complex are that of M-N. The M-N band appeared at 482.2 cm^{-1}. These bands confirmed the formation of a stable (L-Ni) metal complex.[22-23] The weaker bands observed at 709.8 cm^{-1} and 825 cm^{-1} are due to OH wagging mode of vibrations, suggesting coordinated water (OH_2) molecules in the metal complex.[28-30,38] These bands are absent in IR spectrum of the parent ligand.

Analysis of metal complex (L-Zn): The IR spectrum of L-Zn complex exhibited shift in frequency of azomethine, υ(C=N), stretching from 1674.21 to 1753.29 cm^{-1} as compared to parent ligand.[19-21] Also band owing to phenolic OH stretching moved from 3774.69 to 3610.74 cm^{-1} and phenolic υ(C-O) stretching moved from 1126.43 to 1033.85 cm^{-1}. The most distinguishing bands of this metal complex are that of M-N. The M-N band was detected at 495.71 cm^{-1}. These bands confirmed the formation of stable (L-Zn) metal complex.[24-27] The weaker bands detected at 779.24 and 842.89 cm^{-1} are caused by OH wagging mode of vibrations suggesting the presence of coordinated water (OH_2) molecules in the metal complex.[28-30,38] These bands are lacking in IR spectrum of the parent ligand.

The IR data of both metal complexes showed the bidentate nature of parent ligand. The IR data of both parent Schiff base ligand and its metal complexes is summarized in Table 3.

^{1}HNMR Spectral Studies

The peaks observed in ^{1}HNMR spectra of parent Schiff base ligand are as follows. The most characteristic peak at 8.99 ppm (s, 1H, N=CH) was observed due to H-from azomethine group. The peaks observed at 6.96–7.68 ppm showed owing to H-from both aromatic rings. The peak observed at 5.01 ppm (s, 2H, OH, and NH) attributable to H from phenolic -OH and imidazole –NH. The peak was observed at 3.33 ppm (s, 6H) due to two methoxy methyl groups, whereas peaks were observed at 2.51 ppm (s, 6H) caused by two methyl groups attached to the aromatic ring. The ^{1}HNMR spectrum data of parent Schiff base ligand is summarized in Table 4.

Mass Spectral Studies

The mass spectrum study of the parent Schiff base ligand showed a significant peak at m/z 327 (M^{+2}), which corresponds to the molecular weight of the parent Schiff base ligand, i.e., 325.

Electronic Spectra

The electronic spectrum of both metal complexes (L-Ni, L-Zn) was recorded in the wavelength region 200 nm to 800 nm in DMSO solution. The electronic spectral data of both complexes are summarized in Table 5.

Table 3: Selected Infrared Frequencies (cm^{-1}) of parent ligand and its complexes

Ligand / Complex	*υ(C=N) Azomethine*	*υ(OH) Phenolic*	*υ(C-O) Phenolic*	*υ(M-N)*	*υ(H_2O) wagging*
$C_{18}H_{19}O_3N_3$	1674.21	3774.69	1126.43	–	–
$[(C_{18}H_{19}O_3N_3)_2(H_2O)_2]Ni$	1681.93	3640	1033.85	482.20	709.2, 825
$[(C_{18}H_{19}O_3N_3)_2(H_2O)_2]Zn$	1753.29	3610.74	1033.85	495.71	779.24, 842.89

Table 4: Observed ^{1}HNMR Peaks (in ppm) of Parent Schiff base ligand

Compound	*H-from azomethine group*	*H- from aromatic group*	*H-from phenolic and imidazole*	*H-from methoxy methyl group*	*H-from 5,6 dimethyl group*
$C_{18}H_{19}O_3N_3$	8.99	6.96-7.68	5.01	3.33	2.51

Table 5: Electronic spectral data and probable geometries for the metal complexes

Sr. No.	*Complex*	*UV Vis major bands absorption maxima cm^{-1} (nm)*	*Assignment*	*Proposed geometry*
1	$[(C_{18}H_{19}O_3N_3)_2(H_2O)_2]Ni$	32320.62 (309.4)	$^3A_{2g}(F) \rightarrow {}^3T_{2g}(F)$	Octahedral
		34411.56 (290.6)	$^3A_{2g}(F) \rightarrow {}^3T_{1g}(F)$	
		44274.79 (226)	$^3A_{2g}(F) \rightarrow {}^3T_{1g}(P)$	
		49067.71 (203.8)	Charge transfer	
2	$[(C_{18}H_{19}O_3N_3)_2(H_2O)_2]Zn$	32341.53 (309.2)	–	Octahedral
		34459 (290.2)	–	
		44326.24 (225.6)	–	
		46468.4 (215.2)	–	

Table 6: Thermogravimetric analytical data of metal complexes

$[(C_{18}H_{19}O_3N_3)_2(H_2O)_2]Ni$		$[(C_{18}H_{19}O_3N_3)_2(H_2O)_2]Zn$	
Weight loss (%)	*Temperature (°C)*	*Weight loss (%)*	*Temperature (°C)*
0	27.23	0	30.2
10	173.83	10	89.62
20	249.53	20	194.63
30	284.02	30	266.52
40	303.10	40	326.57
50	343.06	50	379.64
60	380.23	60	463.76
70	385.01	70	486.77
80	416.73	74.024 (Total wt. loss)	500
87.151 (Total wt. loss)	475	–	–

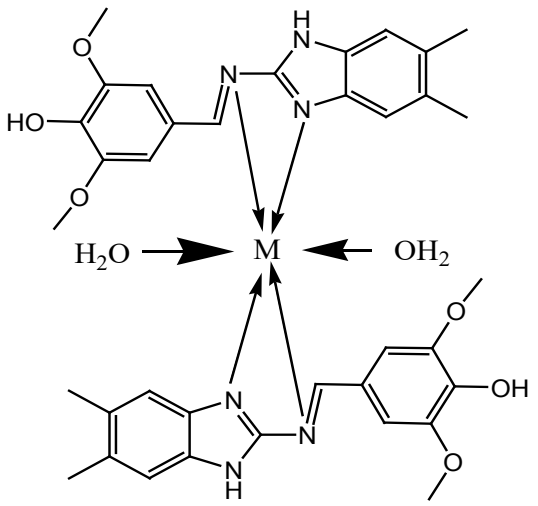

Fig. 2: Projected structure of Metal complexes
[M = Mn^{+2} / Fe^{+3} / Co^{+2} / Ni^{+2} / Cu^{+2} / Zn^{+2}/ Cd^{+2} /Ag^{+}]

The electronic spectrum of the Ni (II) complex reveals major bands at 49067.71 cm^{-1}(203.8 nm), 44274.79 cm^{-1} (226 nm), 34411.56 cm^{-1} (290.6 nm) and 32320.62 cm^{-1} (309.4 nm). The band at 49067.71 cm^{-1} is due to charge transfer proving the coordination of the ligand to Ni (II).

Table 7: Antibacterial activity of ligand and their metal complexes

Sr. No.	*Compounds*	*Minimum Inhibition Concentration (µg/ml)*		
		E. Coli	*S. Aureus*	*S. Typhi*
1	$C_{18}H_{19}O_3N_3$	100	125	125
2	$[(C_{18}H_{19}O_3N_3)_2(H_2O)_2]Fe$	100	125	250
3	$[(C_{18}H_{19}O_3N_3)_2(H_2O)_2]Mn$	250	125	100
4	$[(C_{18}H_{19}O_3N_3)_2(H_2O)_2]Co$	250	100	250
5	$[(C_{18}H_{19}O_3N_3)_2(H_2O)_2]Ni$	250	125	250
6	$[(C_{18}H_{19}O_3N_3)_2(H_2O)_2]Cu$	250	125	500
7	$[(C_{18}H_{19}O_3N_3)_2(H_2O)_2]Zn$	250	125	125
8	$[(C_{18}H_{19}O_3N_3)_2(H_2O)_2]Cd$	500	100	500
9	$[(C_{18}H_{19}O_3N_3)_2(H_2O)_2]Ag$	500	500	250

The last three correspond to $^3A_{2g}(F) \rightarrow {}^3T_{2g}(F)$, $^3A_{2g}(F) \rightarrow {}^3T_{1g}(F)$, $^3A_{2g}(F) \rightarrow {}^3T_{1g}(P)$ transitions respectively.[18,24-26] The electronic transition shown in the spectrum supports octahedral geometry of the complex.[25] The major bands also conform $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transitions.

The electronic spectrum of the Zn (II) complex exposes main bands at 32341.53 cm^{-1} (309.2 nm), 34459 cm^{-1} (290.2 nm), 44326.24 cm^{-1} (225.6 nm) and 46468.4 cm^{-1}(215.2 nm). However *'d'* shell of Zn (II) complex is complete and unavailable for bonding. The Zn^{+2} ions have complete d shell resulting absence of ligand field stabilization effect. The stereochemistry of the Zn (II) complex is proposed based on size, covalent bonding, and available electronic spectrum. The spectrum transitions may be recognized to charge transfer bands proving the coordination of the ligand to Zn (II). The electronic transition is shown in the spectrum also suggests the octahedral geometry of the complex.[5, 27]

Thermal Analysis of Metal Complexes

The TGA of both the metal complexes (L-Ni, L-Zn) were carried with in the temperature range from room temperature to 500. The heating was carried in the dynamic nitrogen atmosphere with a linear heating rate of 10 min^{-1}. The nitrogen flow rate was kept at 30 mL min^{-1}. The thermal data extracted from the thermogram of both complexes are summarized in Table 6.

The L-Ni complex thermogram clearly shows a total weight loss of 87.151%, which may be observed in steps shown in Table 6. At first, the water of crystallization was lost in the range of 27 to 150 and 10% weight loss was found at 173.83. This is followed by loss of methyl, methoxy, hydroxyl and remaining organic moiety resulting in total weight loss of 87.151% (cal. 91 %) up to 475. A stable curve above 475 indicates the formation of stable metal oxide (Ni-O).[25, 31- 33]

The L-Zn complex thermogram, evidently, exhibits a total weight loss of 74.024%, which may be seen in steps shown in Table 6. At first, the water of crystallization was lost in the range of 30 to 80 and 10% weight loss was found at 89.62. This is followed by loss of methyl, methoxy, and remaining organic moiety resulting in total weight loss of 74.024% up to 500. A constant curve at 500 indicates the formation of stable metal oxide (Zn-O).[33, 34]

Bioactivity Study

The biological activity was measured in terms of % of inhibition *in vitro*. The assay was carried according to the micro-assay protocol of Rieckmann and co-workers with minor modifications.[39, 40] The antimicrobial activity of synthesized Schiff base ligand and its metal complexes were screened against *Escherichia Coli*, *staphylococcus Aureus* and *Salmonella Typhi* grew at 37 overnight. The Micro Broth Dilution method measured the minimum inhibition concentration using streptomycin as a reference drug at wavelength 475 nm. The test samples were prepared using DMSO solvent in the concentration range between 4 µg/mL to 100 µg/mL.

The MIC data summarized in Table 7 clearly shows that the parent ligand and its Fe (III) complex show excellent activity against *Escherichia Coli* compared to the rest of the metal complexes. The Co (II) complex and Cd (II) complex show excellent activity against *S. Aureus* compared to the parent ligand and the rest of the metal complexes. The Mn (II) complex shows excellent activity against *Salmonella Typhi* compared to the parent ligand and the rest of the metal complexes.

Conclusion

In the present work, solvent-free syntheses of a new bidentate Schiff base ligand, (*E*)-4-((5, 6-dimethyl-1-*H*-benzo[*d*] imidazole-2-yliino) methyl)-2, 6-dimethoxyphenol, and its metal complexes were performed using the microwave method. All the synthesized compounds were characterized by different analytical and spectral methods, which support the projected structure of the new Schiff base ligand and its corresponding metal complexes. The main advantages of this method are a decrease in reaction time and an increase in yield. The method also supports the green synthesis concept and very easy to conduct.

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