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PREPARATION, CHARACTERIZATION, AND ANTIMICROBIAL STUDIES OF Co(II), Mn(II), Cu(II), AND Zn(II) COMPLEXES OF A NOVEL SCHIFF BASE LIGAND

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ABSTRACT

A new series of air-stable binuclear Co(II), Mn(II), Cu(II) and Zn(II) complexes of Schiff base derived from 2,3-dimethoxybenzaldehyde and 1,4-diaminobutane have been synthesized and characterized by analytical, IR, UV-Vis, ^{1}H & ^{13}C NMR and Powder XRD studies. The analytical data shows that the stoichiometry of the complexes is 2:1:2. Spectroscopic data suggested tetrahedral geometry for Co(II), Mn(II) and Zn(II) complexes, distorted tetrahedral geometry for Cu(II) complex respectively. Antimicrobial activity of the ligand and its corresponding metal(II) complexes were investigated against various microorganisms by the agar well diffusion method. Cu(II) complex is found to be more active than other complexes and free ligand. The anti-inflammatory activity of the synthesized compounds has been done by using the HRBC membrane stabilization method. In different concentrations, Cu(II) complex exhibits the high inhibition of haemolysis compared to the free ligand and Co(II), Mn(II), and Zn(II) complexes.

KEYWORDS: Schiff base, NMR, Antimicrobial

INTRODUCTION

The chemistry of coordination compounds with Schiff base ligand which contains azomethine group RCH=N-R[1], framed by the buildup of the condensation of primary amines with aldehyde and ketones, first pronounced by Hugo Schiff in 1864[1]. Recently, the synthesis and characterization of mono and binuclear it can also be homo or hetero atom plenty efforts in the manufacturing of medicines and drugs and have some biological undertaking such as anti-fungal agent, antibiotics and anti-cancer agent and for their utilization as models of mettalo-enzyme active sites[3]. They additionally advancement of new metal-based chemotherapeutic agent [4]. Recently, the synthesis and characterization of mono and binuclear it can also be homo or hetero atom plenty efforts in the manufacturing of medicines and drugs and have some biological undertaking such as anti-fungal agent, antibiotics and anti-cancer agent and for their utilization as models of mettalo-enzyme active sites[3]. They additionally advancement of new metal-based chemotherapeutic agent [4].

EXPERIMENTAL SECTION

All the chemicals and solvents used for the syntheses were of commercially available reagent grade and were used without further purification.

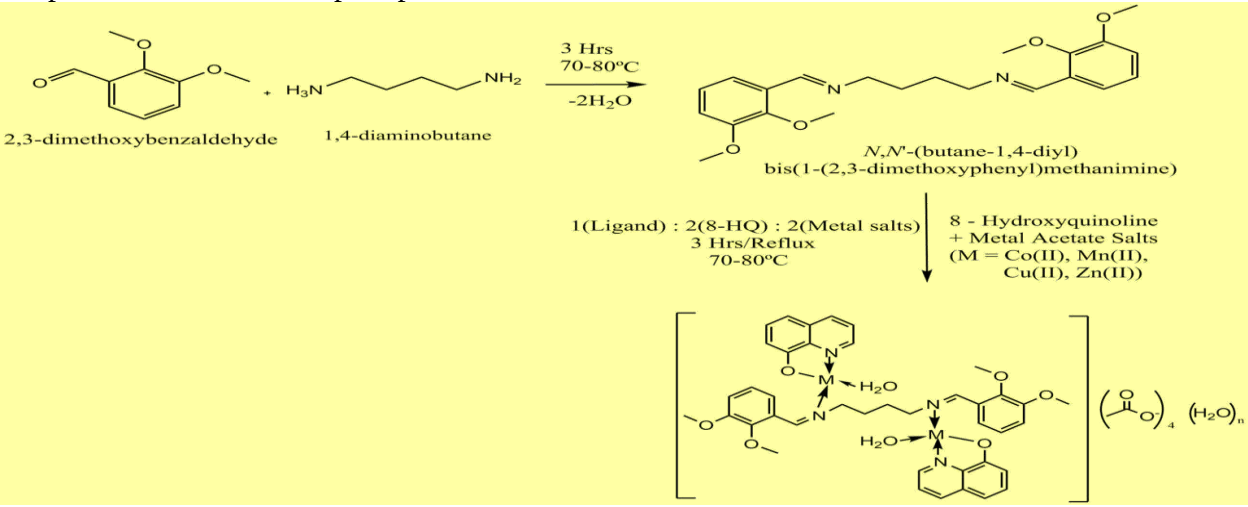
PREPARATION OF SCHIFF BASE LIGAND AND ITS COMPLEXES

Synthesis of the ligand

The ligand N, N′- (butane-1,4-diyl) bis (1-(2,3-dimethoxyphenyl)methanimine) prepared from 2,3 – dimethoxy benzaldehyde (0.664g, 4 mmol) dissolved in 10ml of methanol, then added dropwise to 1,4-diaminobutane (0.2ml, 2 mmol) in 10ml methanol solution, with continuous stirring on magnetic stirrer and the reaction solution was refluxed for 3 hours at 75-80ºC. the resulting yellow color solution was allowed to cool. The yellow color product was obtained.

Synthesis of the metal complexes

The metal complexes were prepared by the addition of ligand and 8-hydroxyquinoline with metal acetate salts. The methanolic solution of 8-hydroxyquinoline was added to the methanolic of Schiff base ligand [N,N'-(butane-1,4-diyl)bis(1-(2,3-dimethoxyphenyl) methanimine)] in the RB flask. Metal acetate salts like (Co, Mn, Cu, and Zn) were dissolved in 10ml methanol added drop wise to the above mixture. The metal-ligand – 8-hydroxyquinoline molar ratio taken was (2:1:2). The mixture solution stirrer for 1 hour in the magnetic stirrer then refluxed for 3 hours. The solution was cooled in the room temperature, the obtained precipitate was filtered and dried in a desiccators.



RESULTS AND DISCUSSION

Molar conductance

Molar conductance used to be measured at 1×10^{-3} M of each complex in DMF at room temperature. The binuclear metal complexes exhibited high value in the vary of 120-140 ohm^{-1} cm^2 mol^1 indicate that the complexes are electrolytic in nature with the presence of anions outside the co-ordination sphere shown in table 1.

Table 1 Analytical data of the Schiff base ligand and its mononuclear metal complexes

Compound	Molecular formula	Molecular weight	Elemental Analysis Found(cal.)%				Colour	Molar conductance Ω^{-1} cm^2 mol^{-1}
			C	N	O	M		
A	$C_{22}H_{28}N_2O_4$	384.48	68.73	7.29	16.64	-	Light Yellow	-
B	$Co[C_{48}H_{68}N_4O_{22}]$	1170.82	49.24	4.78	30.06	10.07	Light brown	129
C	$Mn[C_{48}H_{68}N_4O_{22}]$	1164.97	49.57	4.82	30.27	9.45	Red brown	134
D	$Cu[C_{48}H_{56}N_4O_{16}]$	1072.23	53.78	5.23	23.88	11.85	Dark Green	122
E	$Zn[C_{48}H_{62}N_4O_{18}]$	1113.68	51.86	5.04	25.90	11.76	Brown	138

Infrared spectra

The most valuable IR bands of the Schiff base ligand and their binuclear metal complexes were listed in table 2 and shown in fig 2a-e. The imino stretching frequency of Schiff base ligand exhibit a band at 1590cm^{-1}. The band is shifted to lower frequencies in metal complexes[11]. The lower value may be explained as the drift of lone pair density nitrogen towards the metal atom[12]. However, broadband at 3399-3439cm^{-1} was observed in the spectra of all complexes, which indicate the υ(OH) of water coordinated to the metal complexes[15]. The new bands appear in the region 635-667cm^{-1} and 498-522cm^{-1} which are assigned to υ(M-N) and υ(M-O) respectively, it is supported by the coordination of the metal and ligand in the complexes[16].

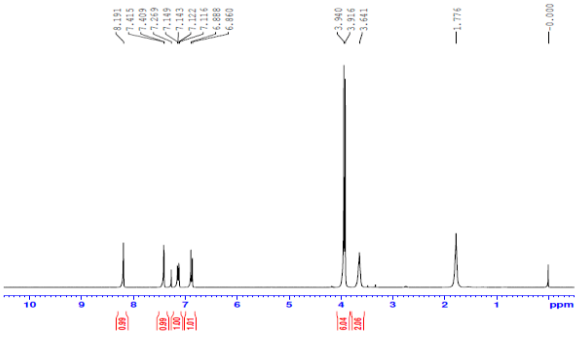
Table 2 Infrared Spectroscopic Data of the Schiff Base Ligand and its binuclear metal complex

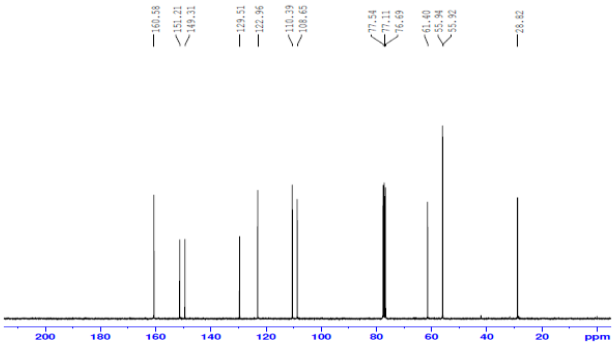
Compound	**ν(CH=N) cm^{-1}**	**ν(-OCH$_3$) (cm^{-1})**	**ν(CH$_2$=CH$_2$) cm^{-1}**	**8 Hydroxy quinoline cm^{-1}**			**ν(M-N) cm^{-1}**
				ν(M-N)	**ν(M-O)**	**ν(H$_2$O)**	
A	1590	2840	2930	-	-	-	-
B	1562	2836	2936	1511	522	3406	667
C	1576	2835	2933	1507	522	3399	637
D	1589	2842	3014	1510	498	3439	635
E	1588	2847	2940	1508	501	3365	640

^{1}H & ^{13}C NMR SPECTROSCOPY

In ^{1}H NMR spectra the chemical value for the shift aromatic protons are observed at 6.93-8.51 for Schiff base ligand [L] . The azomethine protons showed the chemical shift in the range 8.19 for ligand [L]. The methoxy proton showed the chemical shift in the range 3.91 for ligand.

In ^{1}C NMR spectra the chemical value for the shift methoxy group show the shift in the range 61.40 and 55.94 ppm for ligand. The aomethine carbon show the chemical shift in 160.5(CH=N). The aromatic carbons show the shift in the range 108– 129.29 respectively.

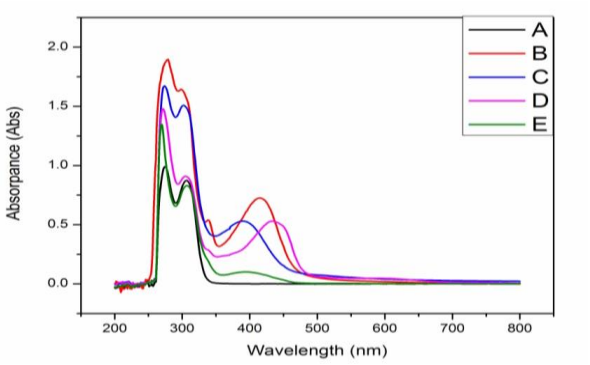




Electronic spectra

The electronic spectra of the ligand and their complexes have been measured in DMF solution between 200-800 nm at room temperature. In the spectra of the Schiff base ligand, the absorption band observed at 275 nm were assigned to π - π* transition and the band at 303 nm were assigned to n - π* transition associated with the azomethine choromophore (CH=N). The electronic spectra of cobalt, manganese, copper and zinc complexes shows absorption peaks at 399,397, 433 and 396 which corresponds to L - M transition. So from the above evidences it is suggested that cobalt, manganese and zinc complexes exhibit tetrahedral geometry and copper exhibit distorted tetrahedral geometry .

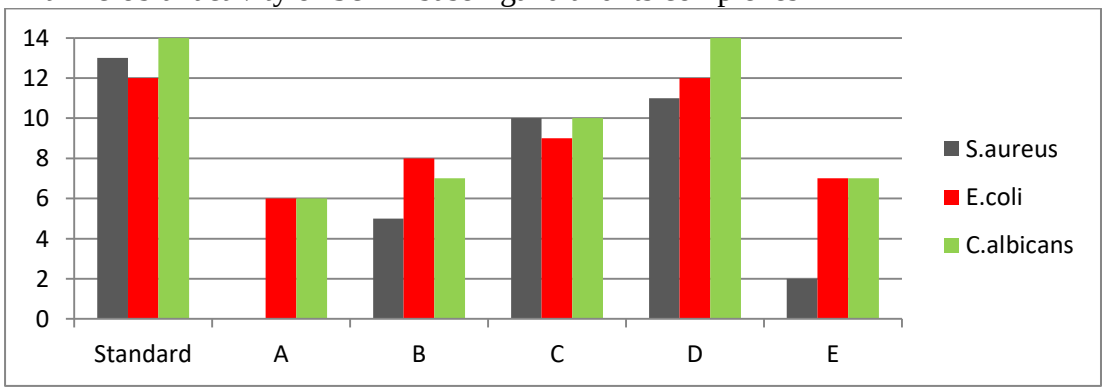
Compound	Absorption (nm)					Geometry of the complex
	π	π*	n	π*	L M	
Ligand 1	275		303		-	-
[CoL]	279		299		399	Tetrahedral
[MnL]	274		302		397	Tetrahedral
[CuL]	272		306		433	Distorted Tetrahedral
[ZnL]	269		307		396	Tetrahedral



Antimicrobial activity

The Antibacterial and Antifungal activities of the Schiff base ligand and its metal(II) complexes were tested against Gram-positive bacteria such as (S.aureus), Gram-negative bacteria such as (E.Coli), while antifungal activities of Schiff base ligand and its metal complexes were tested on fungi(Candida albicans). The disc diffusion method was used for antimicrobial activity. Tetracycline was used as a standard drug for antibacterial and Fluconazole was used as a standard drug for the antifungal test. All metal complexes show maximum inhibition zone than the free ligand. In the above results, Cu(II) complex exhibits the highest antimicrobial activity.

Antimicrobial activity of Schiff base ligand and its complexes



CONCLUSION

The Schiff base ligand and its metal complexes were synthesized. The analytical techniques such as electronic spectroscopy, IR and ^{1}H & ^{13}C NMR spectroscopy were also discussed. All the Schiff base metal complexes are coloured. They are readily soluble in DMSO and DMF. From the IR spectra, the shift in the azomethine frequency confirms the coordination of the central metal to the nitrogen and oxygen (ie M-O and M-N) atoms respectively. The electronic spectra of the Schiff base ligand and its metal complexes provide information about the geometry of the complexes and are in good agreement with the proposed Tetrahedral geometry for Co, Ni, Cu and Zn ion. The presences of azomethine group in both ligands are confirmed by NMR spectral study. The XRD analysis clearly indicated the crystalline size of the synthesized compounds. The anti-microbial study revealed that Cu(II) complexes have super active and all other complexes were more active than the free ligand.

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