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Synthesis, Characterisation and Antimicrobial Studies of Hg(II) and Pb(II) Complexes with Schiff Base Ligand

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Abstract - Metal Complexes of Hg(II) and Pb(II) with Schiff base derived from condensation of 2-hydroxy-1-naphthadelyde with 1,8- diaminonaphthalene and 2-hydroxybenzaldehyde were synthesized and characterized by elemental analysis, m.pt, IR. UV- visible and HNMR Spectroscopy. The ν(C = N) stretching vibration was observed at 1635 cm^{-1} in the spectra of the ligand the band shifted to a higher frequency in the complexes compared to the ligand indicating an increase in the C = N bond order due to the coordination of the metal with the imine nitrogen lone pair. In HNMR the phenolic O-H signal appeared in the spectrum of (HL) at δ10.08 ppm as a broad singlet peak. The molar conductivity of the complexes showed low conductance values this indicates that the compounds are non-electrolyte. The Schiff base ligand and the metal complexes were screened against four bacteria stain (Staphylococcus aureus, Streptococcus pyogene, Escherichia coli and Klebsielia pneumonia) and two fungi (Candida albicans and Aspergilllus niger). The results of the microbial screening showed that Hg(II) complex found to be the most active against all isolates.

Key words: Synthesis, Spectroscopy, Antimicrobial

I. INTRODUCTION

Schiff bases have been in chemical analogue for over 150 years [1-2]. Literature survey shows that the study of these diverse ligands system is linked with transition metal complexes. Schiff bases offer many flexible series of ligands capable of binding with various metal ions to give complexes with suitable properties for theoretical and practical applications. These various classes of Schiff bases can be prepared by condensation of different types of amine and carbonyl compounds [3]. The presence of two nitrogen and two oxygen atoms, cause the coordination to take place with one metal ion through the four coordination sites leading to the formation of three chelate rings. The double bond attached to the nitrogen atoms contributes in enhancing the basicity of both nitrogen atoms, which leads to increase in stability of the complexes formed [4]. They also exhibit a variety of biological activities with substituted aromatic compounds having higher activities [5-7].

Various biological aspects of the metal based drugs/ligands entirely depend on the ease of cleaving the bond between the metal ion and the ligand. As a consequence, it is essential to understand the relationship between ligand and the metal in biological systems. Several metal complexes are known to accelerate the drug action and the efficacy of the organic therapeutic agent. The efficacy of the various organic therapeutic agents can often been enhanced upon coordination with a suitable metal ion. The pharmacological activity of metal complexes is highly dependent on the nature of the metal ions and the donor sequence of the ligands because different ligands exhibit different biological properties [8].

The complexes of mercury are used for agricultural and industrial purposes, Insecticides, fungicides, bactericides and herbicides which are used for agrochemicals containing mercury compounds. Mercury forms useful amalgams with many metals. These amalgams have various applications in diverse fields such as Ag-Hg in dental fillings, Zn-Hg as a reducing agent in chemical synthesis and Cd-Hg in the Weston cadmium cell. Mercury also plays an important role in biological and chemical processes as a mineral element [9-10].

Mercury is one of the most toxic heavy metals on earth. For example, methylmercury is more toxic than

elemental mercury and other inorganic mercury compounds. Mercury containing ligands are known to form stable complexes with metal ions, such as gold (I) and Se (II), because mercury is considered to be a soft Lewis base [11-12]. The coordination chemistry of mercury (II) differs from most other transition metals due to its large size and d^{10} configuration. Its interference in biological system and its potential as a toxin or as a medicine require a better understanding of its coordination properties [13].

Lead and its salts had known to act as a barrier to X-rays, within a few months after their discovery, it was found that X-rays were stopped more effectively by lead than any other common metal. Lead complexes are generally considered to have cooling properties. Ordinary lead compounds have, however, not been used for many years because of their toxicity. The availability of lead EDTA complex is water-soluble and apparently non-toxic, suggested its use in diagnostic radiology. The complex may prove useful in radiological diagnosis for visualization of the alimentary canal and in hepatorenograph and other techniques; also for example as a test for renal function, but further work needs to be done to determine the largest effective doses that can be given by injection [14].

II. MATERIALS AND METHODS

Materials and Solvents

All the chemicals and solvents used were of analar grade and were used without further purification. Hg(II) and Pb(II) salts were used as chloride. Thermo Flash EA CHNS-O elemental analyser. Griffin melting point apparatus was used to determine the melting points. Fourier Transform Infrared Spectroscopy (FTIR). UV/Visible Spectrophotometer Model 721. The ^{1}HNMR Brucker/Top Spin 3.2T-D-65536 spectrophotometer and Estick(R) Series Model EC 500/Conductivity and Temperature Meter were used for the study.

Methods

Synthesis of the Ligand (HL) in 1:1:1 (aldehyde: amine: aldehyde) Molar Ratio

The method employed for the synthesis of the ligands in 1:1:1 (aldehyde: amine: aldehyde) molar ratio is similar to the method described previously. A typical procedure for the preparation of the Schiff base ligand is as follows: a 30 ml ethanolic solution of 2-hydroxy-1-naphthadelyde (10 mmol, 1.722g) was mixed with stirring with 30 ml ethanolic solution of 1,8- diaminonaphthalene (10 mmol. 1.582 g) and 2-hydroxybenzaldehyde (10 mmol, 1.22g) after which 2-3 drops of conc. H_2SO_4 was added. The mixture was refluxed for 3hrs in a quick fit conical flask. After refluxing, the mixture was left to stand for 2-3 days. The solid product obtained was filtered, washed with ethanol and dried in a desiccator over calcium chloride ($CaCl_2$).

Preparation of the complexes are presented below

The metal complexes were prepared according to literature procedures [15-17] .Metal salts of Hg(II) and Pb(II) were added to the Schiff base ligands in a mole ratio 1:1 (metal: ligand). A mixture of the Schiff base (HL) under investigation (1 mmol, 0.4165g) in 30 ml ethanolic solution and 30 ml ethanolic solution of Hg(II) salt (1 mmol, 0.271g) were refluxed for 2 hours, after which it was cooled, filtered and the product obtained were dried in a desiccator containing calcium chloride ($CaCl_2$) as a desiccant. The same procedure was applied to Pb (II) metal salt.

Equation for the Reaction

$$M(X)_2.nH_2O + 2HL \longrightarrow ML_2X_2 + nH_2O$$

where M = Hg (II) and Pb(II), HL = Schiff base Ligands, n = 1, 2, 3, 4 or 6 and X = Cl

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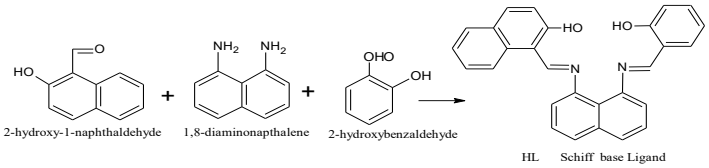

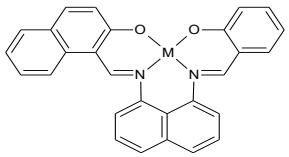

Metal complex
where M=Hg(II), Pb(II)

Study on Antimicrobial Activity

Antibacterial activities of the ligands and metal complexes were screened against two strains of Gram positive bacteria *(Staphylococcus aureus, Streptococcus pyogenes)* and two Gram negative *(Escherichia coli*, *Klebsiella pnuemonia).* Pure clinical bacterial strains were collected from the Department of Medical Microbiology, University of Maiduguri Teaching Hospital (UMTH) Nigeria. A known concentration of the isolates was prepared by suspending into sterilized nutrient broth for bacteria and sabouroid-dextrose agar for fungi. Different concentrations (400, 300, 200, mg/ml) of the metal complexes and the Schiff bases were placed on the surface of the culture media and incubated at 37^0C for 2 h. After incubation, the average of the inhibition zones was measured and recorded. Metal complexes that show 7 mm zone of inhibition and above were further assayed for minimum inhibition concentration (MIC) and minimum bacterial concentration (MBC) using samples concentration of (100, 50, 25, mg/ml) with same bacterial species [18].

Table 1: Physical Properties and some Analytical Data of the Schiff Base Ligand and its Metal Complexes

S/No	Compound	Colour	Yield, g(%)	M.P. (^{0}C)	Λ_m (Scm^2 mol^{-1})
1	HL	Light brown	3.01 (72)	230	-
2	[Hg(HL)Cl]	Dark brown	0.77 (59)	260 -262	20.00
3	[Pb(HL) Cl]	Dark brown	0.40 (60)	320	15.50

Key: HL- Ligand, M.P/D.T-Melting point/Decomposition temp. Λ_m= molar Conductivity

Table 1 shows the physical properties of the ligands and their metal complexes. The results indicate that all the compounds formed are air stable. The complexes showed various colors ranging from light brown to dark brown with a good yields ranging from 59-72%. The molar conductivity of the complexes showed low conductance values. The ligands and complexes showed high melting points in the range of 230-320 ^{O}C.

Table 2: Microanalysis of the Schiff Base Ligand and the Metal Complexes

S/No	Compound	C Found (Calcd)	H Found (Calcd)	N Found (Calcd)	M % Found (Calcd)
	HL				
1	$C_{28}H_{22}N_2O_2$ (416.50)	78.67 (78.74)	5.20 (5.28)	6.81 (6.72)	- -
2	$Hg(C_{28}H_{22}N_2O_2)Cl$	49.39	3.86	4.50	30.55

	(653)	(49.61)	(3.98)	(4.28)	(30.78)
3	$Pb(C_{28}H_{22}N_2O_2)Cl$ (659)	49.46 (49.16)	3.94 (3.95)	4.02 (4.20)	31.46 (31.41)

The Microanalysis of the ligand and the metal complexes are presented above. Microanalysis (C, H, N) results indicates that the experimental data are in close agreement with the theoretical values and agrees with the formation of 1:1 (M: L) molar ratio for the complexes. Similar observations reported by [19].

Table 3: The Relevant Infrared Frequencies (cm^{-1}) for the Schiff Base Ligand and its Metal Complexes

S/No	Compounds	ν(OH)	ν (C=N)	ν(C-O)	ν(C=C)	M-O	M-N
1	HL	3406s	1635m	1462w	1558w	-	-
2	[Hg(HL) Cl]	3406s	1643m	1415b	1527s	748b	613w
3	[Pb(HL) Cl]	3402s	1639m	1411w	1581b	744b	489w

Where m = medium, b = broad, w = weak, s = sharp

The ν(OH) band was observed at 3406 cm^{-1} in the 2-hydroxy-1-naphthaldehyde and 1,8-diaminonaphthalene with 2-hydroxybenzaldehyde ($HL^{)}$ ligand due to intra-molecular hydrogen bond. Similar observation reported by [20]. The ν(C = N) stretching vibration was observed at 1635 cm^{-1} in the spectra of the ligand the band shifted to a higher frequency in the complexes compared to the ligand indicating an increase in the C = N bond order due to the coordination of the metal with the imine nitrogen lone pair [21].

The ring skeletal vibrations ν(C=C) was observed at 1558 cm^{-1} in the ligand. On coordination with the metal ion this band shifted to lower frequency at a region of 1527 cm^{-1}. In Pb(HL)Cl complex the band shifted to higher frequency at 1581 cm^{-1} attributed to the nature of the metal ion coordinated to the ligand. The low intensity band occurring in the range 748-489 cm^{-1} was assigned to ν(M-O) and ν(M-N) respectively. This indicates coordination of the ligand to metal ion through phenolic oxygen and azomethine nitrogen as in the other complexes.

Table 4: Electronic Spectra of the Schiff Base Ligand and its Metal Complexes

S/No	Compound	λ_{max} nm	λ_{max} cm^{-1}	A	Σ	Assignment
1	HL	360	27777	0.700	7000	n - π^*
		390	25641	1.121	11210	π - π^*
2	[Hg(HL)Cl]	390	25641	1.023	1023	MLCT
		400	25000	0.830	830	MLCT
3	[Pb(HL)Cl]	390	25641	1.023	1023	MLCT
		379	26642	0.949	949	MLCT

Two absorption bands in the UV region in the range 27777 - 25641 cm^{-1} due to π - π* and n - π* transitions of the aromatic ring occurred in ligand. The bands shifted to lower wavelength on coordination with the metal ion. This shift is an evidence of coordination with the metal ion. Hg(HL)Cl complex showed two absorption bands at 25641 and 25000 cm^{-1} due to charge transfer transitions. Pb(HL)Cl also showed absorption in the range of 25641-26642 cm^{-1} attributed to metal to ligand charge transfer transitions.

Table 5: ^1H Nuclear Magnetic Resonance Data of the Ligand in (ppm)

Compounds	Molecular Formula (Molar Mass)	Phenolic proton δ (OH)	Azomethineδ (-HC=N)	Aromatic protonδ (C – H)	Solvent (DMSO)

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HL	$C_{28}H_{24}N_2O_4$ (451.50)	10.08	8.78	6.30 - 7.83	2.50 -3.32

The phenolic O-H signal appeared in the spectrum of (HL) at δ10.08 ppm as a broad singlet peak [22] reported a peak at 10.66 attributed to hydrogen-bonded phenolic proton. The singlet peak at δ8.78 ppm is due to -HC=N moiety. The aromatic ring proton showed a multiple signals in the range of 6.30-7.83 ppm respectively. The doublet signals occur in the range of 2.50-3.32 ppm corresponding to solvent proton.

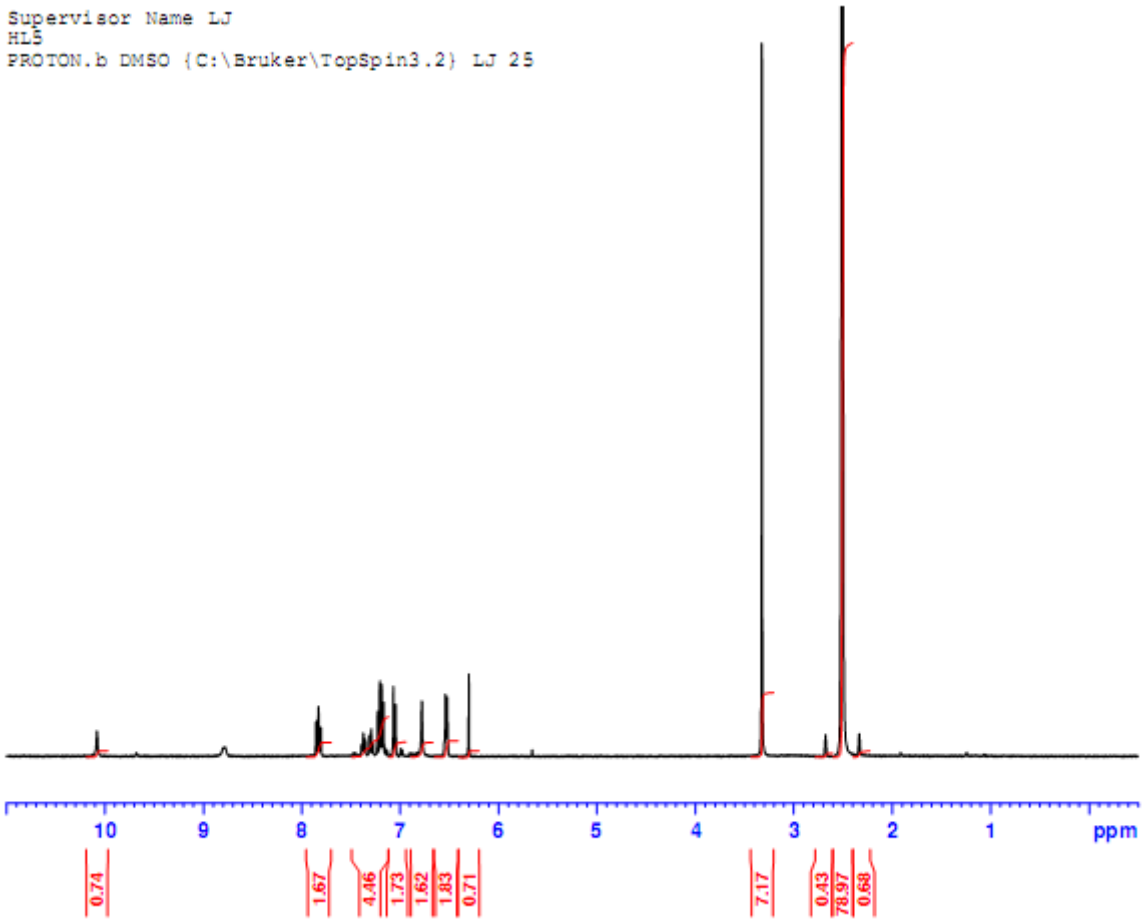

Table 6: Antimicrobial Activities of the Schiff Base Ligand (HL) and its Metal Complexes

Compounds	Concentration (mg/ml)	Antimicrobial activity with zone of inhibition (mm)					
		Staphylococcus aureus	*Streptococcus Pyogenes*	*Escherichia coli*	*Klebsiella*	*Candida albican*	*Aspergillus niger*
HL	400	0.00±0.00[a]	0.00±0.00[a]	0.00±0.00[a]	0.00±0.00[a]	0.00±0.00[a]	0.00±0.00[a]
	300	0.00±0.00[a]	0.00±0.00[a]	0.00±0.00[a]	0.00±0.00[a]	0.00±0.00[a]	0.00±0.00[a]
	200	0.00±0.00[a]	0.00±0.00[a]	0.00±0.00[a]	0.00±0.00[a]	0.00±0.00[a]	0.00±0.00[a]
	100	0.00±0.00[a]	0.00±0.00[a]	0.00±0.00[a]	0.00±0.00[a]	0.00±0.00[a]	0.00±0.00[a]

[Hg(HL)Cl]	400	22.0±0.00[b]	23.0±0.00[b]	13.0±0.00[a]	16.0±0.00[b]	22.67±0.58[b]	16.0±0.00[b]
	300	17.67±0.58[c]	18.33±0.58[c]	10.0±0.00[b]	12.0±0.00[c]	19.33±0.58[c]	12.0±0.00[c]
	200	13.67±0.58[d]	14.0±0.00[d]	7.0±0.00[c]	0.00±0.00[a]	15.0±0.00[d]	8.0±0.00[d]
	100	11.0±0.00[e]	10.0±0.00[e]	0.00±0.00[d]	0.00±0.00[a]	11.0±0.00[e]	0.00±0.00[a]
[Pb(HL)Cl]	400	0.00±0.00[a]	0.00±0.00[a]	0.00±0.00[e]	0.00±0.00[a]	0.00±0.00[a]	0.00±0.00[a]
	300	0.00±0.00[a]	0.00±0.00[a]	0.00±0.00[a]	0.00±0.00[a]	0.00±0.00[a]	0.00±0.00[a]
	200	0.00±0.00[a]	0.00±0.00[a]	0.00±0.00[a]	0.00±0.00[a]	0.00±0.00[a]	0.00±0.00[a]
	100	0.00±0.00[a]	0.00±0.00[a]	0.00±0.00[a]	0.00±0.00[a]	0.00±0.00[a]	0.00±0.00[a]
Cipro	400	20.66±0.58[g]	35.00±0.00[h]	18.00±0.00[f]	17.33±0.58[d]	-	-
Amph. B		-	-	-	-	22.33±0.58[h]	32.00±0.00[e]

Different superscript letters along the same column are significantly (P<0.05) different.

The Schiff base ligand and the metal complexes were tested against different pathogenic microorganisms such as *Staphylococcus aureus, Streptococcus pyogene, Escherichia coli, Klebsielia pneumonia* and two antifungal *Candida albicans* and *Aspergillus niger* at concentrations of 400 mg/ml, 300 mg/ml, 200 mg/ml and 100 mg/ml. The ligand HL showed no activity with all the strains of organisms tested. Hg(II) complex was susceptible against the tested microorganisms. Inhibitions by the metal chelates was higher than that of the free ligand and the results are in good agreement with the previous findings with respect to comparative activity of free ligands and its complexes [20]. Pb(II) complex showed no activity against all strains of microorganisms tested.

III. CONCLUSION

All the compounds are air-stable and were formed in good yields (59-72%) with various colors ranging from light brown to dark brown. The compounds showed high melting point indicates that they are stable. The complexes were also soluble or slightly soluble in the solvents used. The molar conductivity of the complexes indicated that they are non-electrolytes.

From the IR results, the Schiff base ligands are tetradentate and coordinate with the metal ion through the phenolic oxygen and azomethine nitrogen atoms. The electronic spectral measurements of the complexes in methanol suggest the complexes to be either four or five coordinated. The hydrogen nuclear magnetic resonance (^{1}HNMR) was done on the Schiff base ligand only. The antimicrobial result indicate that the Hg(II) complex are potent antimicrobial against all the strain of microorganism tested.

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