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SYNTHESIS AND SPECTROSCOPIC STUDIES OF SCHIFF BASE COPPER (II), NICKEL (II), COBALT (III) AND ZINC (II) COMPLEXES

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Abstract: A series of new metal complexes containing Cu(II), Ni(II), Co(III) and Zn(II) with Schiff base ligand derived from the condensation of 1,2-bis(p-aminophenoxy)ethane and 1-amino-2-naphthaldehyde were synthesized and characterized by means of physico-chemical procedures including elemental analysis, Conductivity measurements, IR, UV-VIS, 1H NMR, 13C NMR and magnetic moments data. On the basis of above studies, tetra coordinated square-planar as well as tetrahedral geometry and a six coordinated octahedral geometry for all the complexes have been suggested. Azomethanic nitrogens are the four binding sites of ligand. Antibacterial evaluation revealed that metal (II) complexes shown powerful antibacterial activity.

Index Terms **- Antibacterial, Physico-chemical, Tetra coordinated**

I. INTRODUCTION:

Schiff base ligand and their metal complexes have been explored widely since these compounds are indispensable in chemistry and having various applications [1]. These types of ligands are recognized to coordinate to the metal atom in different mode under various reaction conditions [2]. Major area of research of Schiff base metal complexes is biological activity of that complexes with aim to target discovery of effective and safe therapeutic agents for treatment of cancers and bacterial infections. Co, Ni, Cu and Zn are life-essential elementals and shows a great biological activity whenever it associated with metallic proteins taking part in storage of Iron or electron transfer reaction and oxygen transport. This has created extensive attraction in the study of complexes containing these metals [3-15]. In existing paper Cu (II), Ni(II), Co(III) and Zn(II) Schiff base complexes derived from the condensation of 1-amino-2-naphthaldehyde and 1,2-bis(p-aminophenoxy)ethane are reported. On the basis of physico-chemical data of the complexes and adducts the suitable structure for the complexes are suggested. .

II. EXPERIMENTAL:

All chemicals which are used in this research work without further purification were purchased from Sigma-Aldrich. Electronic Spectra of complexes in UV-VIS region were noted with DMF solutions using Shimatzu 160 Model UV-VIS spectrophotometer. IR spectral data of complexes were noted by the help of Midac 1700 instrument in KBr pellets. 13C NMR and 1H NMR spectral data was recorded on Bruker Gmb H DPX-400 MHz FT-NMR digital spectrometer with DMSO-d6, magnetic susceptibilities of complexes were measured with a Sherwood scientific magnetic susceptibility balance (Model Number- MK1) at 25°C(room temperature) using $Hg[Co(SCN)_2]$ as calibrant, diamagnetic corrections were measured by Pascal's constant.

a) **SYNTHESIS OF LIGAND**:- N,N'-bis(1-amino-2-naphthaldehyde)-1,2-bis(p-aminophenoxy)ethane(L) was obtained from equimolar amounts of 1,2-bis(p-aminophenoxy) ethane (2.44g, 10 m mol) and 1-Amino-2-naphthaldehyde (3.44g, 20 m mol) in 100 ml absolute ethyl alcohol refluxing for two hours. Crystals of Schiff base which separated over cooling were recrystallized by DMF.

b) **SYNTHESIS OF METAL COMPLEXES-** Metal acetate (20 m mol) solution in DMF was mixed with Schiff base ligand (20 m mol) in DMF (dimethyl formamide) 1:1 (M:L) ratio and the content was refluxed with 150 ml DMF on oil-bath till two-three hours. Solid complexes which separated out was filtered, washed with ethyl alcohol and dried in desiccator on to silica gel. They decomposed on 275-300°C and approximately insoluble in water but sparingly soluble in polar solvents (DMSO and DMF).

III. RESULT AND DISCUSSION;

Analytical data for all the complexes are given in table -1. Schiff base ligand was formed by the condensation of 1-amino-2-naphthaldehyde with 1,2-bis(p-aminophenoxy)ethane in absolute ethyl alcohol. Metal to ligand ratio of Ni(II), Co(III), Cu(II) and Zn(II) complexes was found to be 1:1 ratio in adding Ni(II), Cu(II) and Zn(II) complexes were existing one extra molecule of water for crystallization but Co(III) complex has two extra coordinated molecules of water and hydroxo. Their formation may be represented by following common reactions-

$$L + Cu(CH_3COO)_2H_2O \rightarrow [CuL]H_2O + 2CH_3COOH$$

$$L + Ni(CH_3COO)_24H_2O \rightarrow [NiL]H_2O + 2CH_3COOH + 3H_2O$$

$$L + Zn(CH_3COO)_22H_2O \rightarrow [ZnL]H_2O + 2CH_3COOH + H_2O$$

$$L + Co(CH_3COO)_24H_2O + {}^1/_2\,O_2 \rightarrow [CoLH_2O(OH)] + 2CH_3COOH + 2H_2O + H_2O_2$$

A. **CONDUCTIVITY MEASUREMENTS-** Complexes of Ni(II) , Zn(II), Cu(II) and Co(III) are non-electrolytes as exhibited by the molar conductivity measurements with DMF. Which are under range 2.7- 5.8 ohm^(-1) cm^(2) mol^(-1).

B. **MAGNETIC SUSCEPTIBILITY-** Cu(II) complex is paramagnetic and it's magnetic susceptibility is 1.72 BM. Therefore it's 1H NMR spectra can not be obtained. Zn(II), Ni(II) and Co(III) complexes are diamagnetic. Therefore these 13C NMR and 1H NMR spectrum were obtained.

C. **IR SPECTRA**-IR bands of Schiff base ligand and their complexes are represented in table 2.Broad bands and weak bands are taking place around 3400cm^(-1) that can be assigned to OH vibration of H2O molecules in complexes. Band on 1285 cm^(-1) in IR spectra of ligand is attributed to phenolic C-O stretching vibration. The band is found under the region 1280-1283 cm^(-1) in IR spectrum of complexes proposed amines of the Schiff base component has taken part in formation of complexes. Coordination through imine nitrogen is inferred with 1616cm^(-1) and Schiff base ligand from 1613-1620 cm^(-1) of complexes. Conclusive proof of bonding is exhibited by observation that's latest bands in spectrum of metal complexes occur on 500-557 cm^(-1) designated to M-N stretching vibration which is not found in spectrum of ligand

D. **ELECTRONIC SPECTRA: -** Electronic spectra of all complexes were noted in 10^(-3) M DMF at 25°C(room temperature). Spectra of Schiff base show two absorption bands on 265-275 nm and 318-326 nm. Bands are ascribed to π→π* transitions, in which first transition for benzene ring and second for imino group. In complexes π→π* transition of imino group is shifted to longer wave length as a result of coordination. While binding with metal proving the formation of the Schiff base metal complexes. Electronic spectra of Ni(II) complex exhibits absorption band on 444 nm(ϵ = 405 L mol^(-1) cm^(-1)) assigned to $^1A_1g \rightarrow {}^1B_1g$ transition, which is suitable with the complex having squar-planar geometry. Electronic spectra of Cu(II) complex exhibits absorption band on 600 nm (ϵ= 290 L mol^(-1) cm^(-1)) assigned to $^2T_2g \rightarrow {}^2Eg(G)$ transition which is suitable with complex having square-planar geometry. Electronic spectra of Co(III) complex exhibits absorption bands on 424 nm (ϵ = 600 L mol^(-1) cm^(-1)) and 458 nm (ϵ = 400 L mol^(-1) cm^(-1)) assigned to $^4T_1g(F) \rightarrow {}^4T_1g(P)$ and $^4T_1g(F) \rightarrow {}^4A_2g(F)$ transition, which is suitable with the complex having octahedral geometry. Electronic spectra of Zn(II) complex exhibits absorption band on 444 nm (ϵ = 1395 L mol^(-1) cm^(-1)) assigned to L- M (charge transfer) transition, which suitable with the complex having tetrahedral geometry.

E. **1H NMR AND 13C NMR SPECTRA**- 1H NMR Spectra (in DMSO-d6) of ligand L shown signals on 3.49 (s, 4H) Ar-NH2, 4.42(s, 4H) O-CH2, 7.04(d, 2H, J. 9.12 Hz), 7.14(d, 4H, J; 8.92 Hz), 7.35(t,2H, J; 7:36Hz), 7.54(t, 2H, J; 7.21 Hz), 7.64(d, 4H, J; 7.21 Hz), 7.80(d, 2H, J; 7.72 Hz), 7.92(d, 2H, J; 9.16 Hz), 8.51(d, 2H, J; 8.52 Hz) Ar-H, 9.66(s, 2H) HC=N ppm. 1H NMR spectra (in;DMSO- d6) of Zn(II) complex shown signals on 3.40(br,2H)H2O, 3.49(s, 4H)Ar-NH2, 4.42(s, 4H) O-CH2, 7.04(d, 2H, J; 9.18 Hz), 7.14(d, 4H, J;8.84 Hz), 7.35(t, 2H, J;7.38 Hz), 7.55(t, 2H, J; 7.91 Hz), 7.64(d,4H, J;8.80 Hz), 7.80(d, 2H, J;7.96 Hz), 7.92(d, 2H, J; 9.12 Hz), 8.51(d, 2H, J; 8.50 Hz) Ar-H, 9.66(s, 2H)HC=N ppm.1H NMR spectra (in DMSO- d6) of Ni(II) complex shown signals on 3.45(br, 2H)H2O, 3.49(s, 4H) Ar-NH2, 4.42(s, 4H)O-CH2, 704(d, 2H, J; 9.15 Hz), 7.14(d, 4H, J; 8.88 Hz), 7.35(t, 2H, J; 7.38 Hz), 7.55(t, 2H, J; 7.92 Hz), 7.92(d, 2H, J; 9.13 Hz), 8.51(d, 2H, J; 8.50 Hz) Ar-H, 9.66(s, 2H)HC=N ppm. 13C NMR Spectra (in DMSO-d6) of ligand L shown signals on 109.47, 116.37, 121.23, 122.63, 122.91, 124.17, 127.58, 128.79, 129.82, 133.91, 136.91, 138.54, 155.68, 158.15 ppm.13C NMR spectra (in DMSO-d6) of Zn(II) complex shown signals on 109.47, 116.37, 12.23, 122.63, 122.91, 124.18, 127.58, 128.79, 129.82, 133.92, 136.92, 138.51, 155.66, 158.15 ppm.

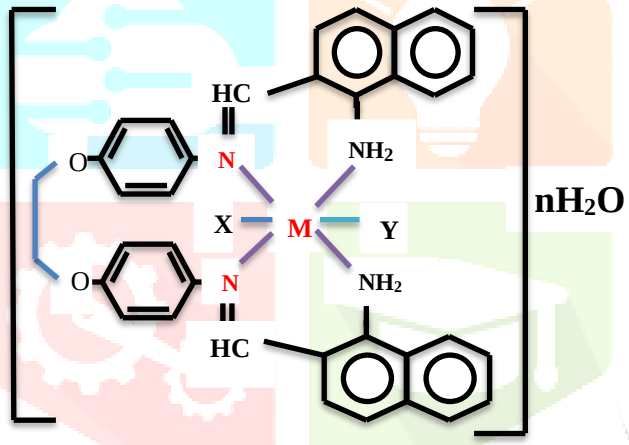
F. **ANTIBACTERIAL ACTIVITY**-Research work has revealed that the coordination of ligand with metal having potential to inhibit the growth of bacteria. Ni (II) complex had a great potential against P. aeruginosa and E. Coli while Zn(II) complex was moderately active against E. Coli. Overall these complexes can be used as anti-bacterial agents for treatment of common deceases caused by E.Coli and P.aeruginosa.

Table No.1-The formulas and elemental analysis of ligand and complexes (calculated and found);

Compounds	Metal(in Percentage)	C(in Percentage)	H(in Percentage)	N(in Percentage)	μ_{eff} (B.M)	Conductivity (ohm^(-1) cm^(-2) mol^(-1))
Ligand(L) $C_{36}H_{30}N_4O_2$	–	78.53 (78.54)	5.43 (5.45)	10.17 (10.18)	–	–
[CuL]H_2O $C_{36}H_{32}N_4O_3Cu$	10.05 (10.06)	68.44 (68.46)	5.05 (5.07)	8.86 (8.87)	1.72	2.7
[NiL]H_2O $C_{36}H_{32}N_4O_3Ni$	9.33 (9.37)	69.00 (69.01)	5.10 (5.11)	8.91 (8.94)	Dia	4.1
[CoL H_2O(OH)] $C_{36}H_{33}N_4O_4Co$	9.20 (9.21)	67.50 (67.52)	5.12 (5.15)	8.70 (8.71)	Dia	3.0
[ZnL]H_2O $C_{36}H_{32}N_4O_3Zn$	10.34 (10.36)	68.21 (68.24)	5.02 (5.05)	8.86 (8.88)	Dia	5.8

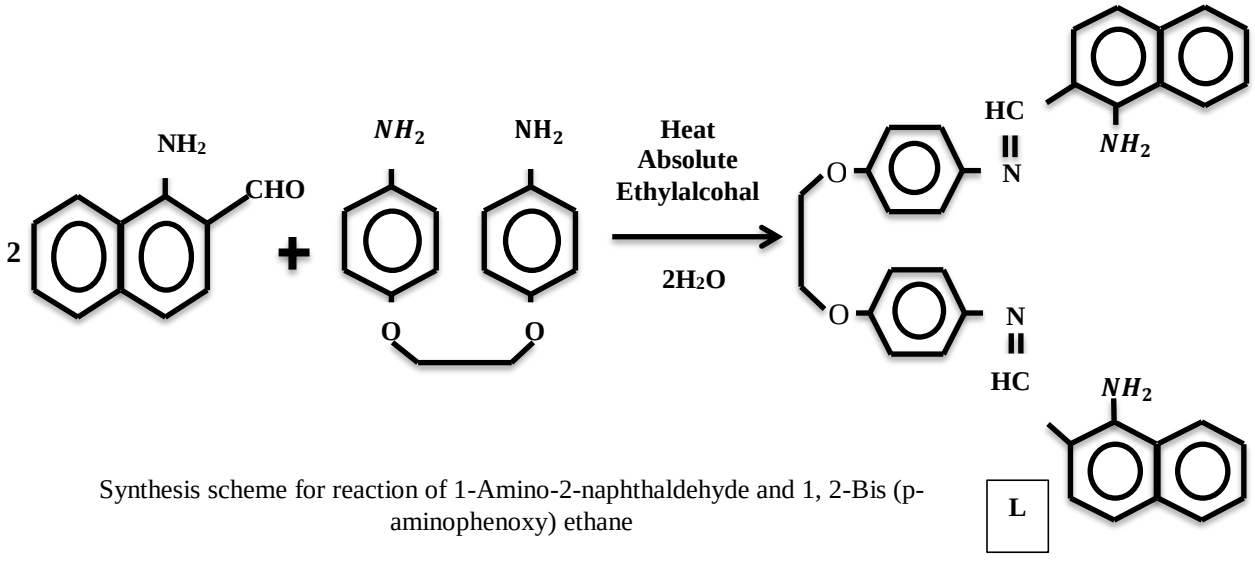
Table No.2-IR Frequency (cm^{-1}) of Schiff Base and its complexes;

Ligand(L)	**[CuL]H_2O**	**[NiL] H_2O**	**[CoL H_2O(OH)]**	**[ZnL] H_2O**	**Assingment**
-	3430 *w*	3440 *w*	3441m	3446w	H_2O
1616s	1613s	1617s	1615s	1620s	C=N stretching
1285m	1283s	1280s	1281s	1282s	C-O stretching
-	505w	557w	506w	500w	ν(M-N)



M= Co (III), Ni(II),Cu(II),Zn(II)
X= H2O — — —
n = --- 1 1 1
Y = OH — — —

Proposed structure of the tetrahedral Zn(II), Square-planar Cu(II) and octahedral Co(III) complexes of the ligand L



Synthesis scheme for reaction of 1-Amino-2-naphthaldehyde and 1, 2-Bis (p-aminophenoxy) ethane

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