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Preparation and Thermal, Spectroscopic, and Antibacterial Studies of Some Mixed-Ligand Complexes

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ABSTRACT

The present investigation reports the synthesis, characterization, and antibacterial activities of some mixed-ligand complexes of the type [ML(SB)H_2O], where M = Mn(II), Co(II), Ni(II), Cu(II), Zn(II), and Cd(II), SB^1 = *bis*(benzylidene)ethylenediamine (benen) or SB^2 = *bis*(acetophenone)ethylenediamine (acphen) and KHL = potassium salt of salicylideneglycine (salgly). Their structures have been elucidated on the basis of elemental analyses, thermogravimetric analyses, magnetic measurements, reflectance spectra, and IR spectra. The results are in accord with an octahedral environment around the central metal ion. The mixed-ligand complexes of Mn(II), Co(II), Ni(II), and Cu(II) are paramagnetic while the Zn(II) and Cd(II) complexes are diamagnetic. The mixed-ligand complexes have been tested against bacteria and the

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results are compared with a standard drug (tetracycline) and control (dimethyl sulfoxide).

Key Words: Mixed-ligand complexes; Schiff bases; Tetracycline; Dimethyl sulfoxide.

INTRODUCTION

The coordination chemistry of amino acid-Schiff base ligands is of considerable interest due to their biological importance.[1,2] Especially, because it has been reported that the mixed-ligand complexes of amino acid-Schiff base with transition elements are used as radiotracers in nuclear medicine[3] and as antitumor[4] agents. Moreover, in many cases, it has been suggested that some biological activities of organic compounds can be increased by coordination with transition elements. In continuation of earlier work[5] we report here the mixed-ligand complexes of Mn(II), Co(II), Ni(II), Cu(II), Zn(II), and Cd(II) with two Schiff bases. The structures of the Schiff bases are shown in Fig. 1.

EXPERIMENTAL

All the chemicals and solvents used were of analytical grade. Metal chlorides, salicylaldehyde, benzaldehyde, acetophenone, ethylenediamine, and glycine were purchased from the E. Merck (India) Limited, Mumbai. The organic solvents were purified by recommended methods.[6]

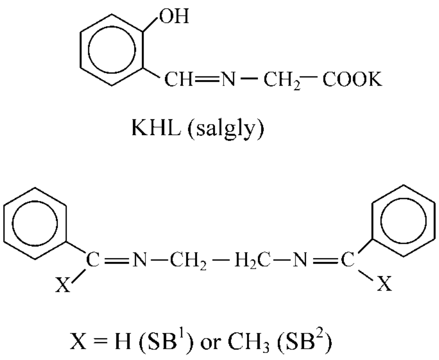

Figure 1. Structure of the Schiff bases.

Preparation of Schiff Bases

Potassium Salt of Salicylideneglycine (Salgly)

A solution of potassium hydroxide (10 mmol, 0.56 g) in 30 mL absolute alcohol was added to crystalline glycine (10 mmol, 0.75 g). The mixture was stirred until it had completely dissolved in the ethanol–KOH solution. Then an ethanolic solution (100 mL) of salicylaldehyde (10 mmol, 1.22 g) was added quickly. The reaction was allowed to come to completion by stirring at 50 °C for 1 hr. The product was crystallized by diffusion of excess absolute diethyl ether into the solution. The resulting product was filtered and dried in a vacuum desiccator. Yield: 1.59 g (63%), M.p.: 207 °C.

Bis(acetophenone)ethylenediamine (Acphen)

An ethanolic solution (100 mL) of ethylenediamine (10 mmol, 0.60 g) and an ethanolic solution (100 mL) of acetophenone (20 mmol, 2.40 g) in the mole ratio 1 : 2 was mixed with constant stirring at room temperature. Refluxing was carried out for 5 hr at 80 °C and then the mixture was cooled in a refrigerator at 0–2 °C for overnight. A fine, yellow product was formed. The product was filtered and dried in air. Yield: 2.28 g (76%), M.p.: 260 °C.

Bis(benzylidene)ethylenediamine (Benen)

The *bis*(benzylidene)ethylenediamine (benen) was synthesized by a published procedure.[7]

Synthesis of the Mixed-Ligand Complexes

The preparation of mixed-ligand complexes was carried out by mixing an aqueous solution (100 mL) of metal chloride (10 mmol) and a hot methanolic solution (100 mL) of the potassium salt of salgly (10 mmol, 2.17 g) and benen (10 mmol, 2.36 g) or acphen (10 mmol, 2.64 g) in 1 : 1 : 1 mole ratio. The mixed-ligand complexes were formed by heating the mixture in a water bath for 1–2 hr at 50 °C. The mixture was kept overnight at room temperature. A fine, coloured product was formed. The obtained crystals were collected by filtration, washed with water and with ethanol, and dried in air.

Physical Measurements

The magnetic moments have been determined by a model 7304, Vibrating Sample Magnetometer, Lake Shore, USA. Infrared spectra were recorded on a FT-IR Nicolet 400D spectrophotometer. Carbon, hydrogen, and nitrogen were analyzed with a model 240 Perkin–Elmer elemental analyzer. The metal contents of the mixed-ligand complexes were analyzed by EDTA titration[8] after decomposing the organic matter with a mixture of perchloric, sulfuric, and nitric acid (1 : 1.5 : 2.5). Thermogravimetric analyses have been obtained with a model 5000/2960 SDTA, TA Instruments, USA instrument. The reflectance spectra of the mixed-ligand complexes were recorded in the range 1700–350 nm (as MgO discs) on a Beckman DK-2A spectrophotometer.

RESULTS AND DISCUSSION

The elemental analytical results and some physical properties of the mixed-ligand complexes are listed in Table 1. The elemental analyses indicate that the mixed-ligand complexes have the general formula [ML(SB)H_2O]. All the mixed-ligand complexes are insoluble in methanol, diethyl ether, and dimethyl formamide but soluble in dimethyl sulfoxide. The mixed-ligand complexes were synthesized following the general procedures outlined by the following equations.

$$\begin{aligned}&\mathrm{MCl_2\cdot}n\mathrm{H_2O + KHL + benen}\\&\qquad\longrightarrow \mathrm{[ML(benen)H_2O] + KCl + HCl} + (n-1)\mathrm{H_2O}\\&\mathrm{MCl_2\cdot}n\mathrm{H_2O + KHL + acphen}\\&\qquad\longrightarrow \mathrm{[ML(acphen)H_2O] + KCl + HCl} + (n-1)\mathrm{H_2O}\end{aligned}$$

Magnetic Measurement

The magnetic moment values of the Mn(II) complexes (5.90 and 5.93 B.M.) are as expected for a high-spin $3d^5$ system. These values are very close to the spin-only value, which is 5.92 B.M., but within the limits of the spin-free value for five unpaired electrons indicating that the complexes are six coordinated with an octahedral geometry around the Mn(II) ion.[9] The magnetic moment values for the Co(II) complexes have been used as criteria to determine the type of coordination around the metal ion. Due to the intrinsic orbital angular momentum in the ground state, there is

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Table 1. Analytical data of the compounds.

Compounds; empirical formula	Formula weight (g)	Colour	% Found (calcd.) C	H	N	Metal	M.p. (°C)	μ_{eff} (B.M.) (calcd.)	Yield (%)
KHL; $C_9H_8NO_3K$	217.18	Orange yellow	49.78 (49.76)	3.65 (3.68)	6.43 (6.44)	—	207	—	63
benen (SB^1); $C_{16}H_{18}N_2$	236.16	Yellow	81.32 (81.36)	7.60 (7.62)	11.80 (11.85)	—	235	—	72
acphen (SB^2); $C_{18}H_{20}N_2$	264.18	Yellow	81.80 (81.83)	7.55 (7.57)	10.64 (10.60)	—	260	—	76
[Mn(L)(benen)H_2O]; $C_{25}H_{25}MnN_3O_4$	486.18	Yellow	61.71 (61.75)	5.10 (5.14)	8.60 (8.63)	11.28 (11.29)	>360	5.90 (5.92)	35
[Co(L)(benen)H_2O]; $C_{25}H_{25}CoN_3O_4$	490.18	Brown	61.27 (61.25)	5.14 (5.10)	8.60 (8.56)	12.03 (12.02)	>360	4.10 (3.87)	35
[Ni(L)(benen)H_2O]; $C_{25}H_{25}N_3NiO_4$	489.94	Reddish yellow	61.32 (61.28)	5.15 (5.10)	8.53 (8.57)	11.96 (11.98)	330	3.18 (2.83)	40
[Cu(L)(benen)H_2O]; $C_{25}H_{25}CuN_3O_4$	494.79	Green	60.63 (60.68)	5.00 (5.05)	8.49 (8.48)	12.88 (12.84)	190	1.96 (1.73)	34
[Zn(L)(benen)H_2O]; $C_{25}H_{25}N_3O_4Zn$	496.64	Yellow	60.40 (60.45)	4.98 (5.00)	8.42 (8.49)	13.18 (13.16)	>360	—	33
[Cd(L)(benen)H_2O]; $C_{25}H_{25}CdN_3O_4$	543.66	Yellow	55.29 (55.22)	4.62 (4.59)	7.75 (7.72)	20.72 (20.67)	288	—	38

(*continued*)

Table 1. Continued.

Compounds; empirical formula	Formula weight (g)	Colour	% Found (calcd.) C	H	N	Metal	M.p. (°C)	μ_{eff} (B.M.) (calcd.)	Yield (%)
[Mn(L)(acphen)H_2O];	514.20	Yellow	63.01	5.69	8.18	10.67	>360	5.93	44
$C_{27}H_{29}MnN_3O_4$			(63.06)	(5.64)	(8.16)	(10.68)		(5.92)	
[Co(L)(acphen)H_2O];	518.20	Brown	62.60	5.63	8.12	11.40	>360	4.09	35
$C_{27}H_{29}CoN_3O_4$			(62.57)	(5.59)	(8.10)	(11.37)		(3.87)	
[Ni(L)(acphen)H_2O];	517.96	Reddish yellow	62.64	5.57	8.14	11.38	332	3.08	41
$C_{27}H_{29}N_3NiO_4$			(62.60)	(5.59)	(8.10)	(11.33)		(2.83)	
[Cu(L)(acphen)H_2O];	522.18	Green	62.00	5.58	8.00	12.11	190	1.99	46
$C_{27}H_{29}CuN_3O_4$			(62.02)	(5.54)	(8.03)	(12.15)		(1.73)	
[Zn(L)(acphen)H_2O];	524.66	Yellow	61.76	5.50	7.96	12.43	>360	—	38
$C_{27}H_{29}N_3O_4Zn$			(61.80)	(5.52)	(8.00)	(12.46)			
[Cd(L)(acphen)H_2O];	571.68	Yellow	56.69	5.05	7.31	19.60	288	—	41
$C_{27}H_{29}CdN_3O_4$			(56.72)	(5.07)	(7.34)	(19.66)			

Note: KHL = potassium salt of salicylideneglycine; benen = *bis*(benzylidene)ethylenediamine; acphen = *bis*(acetophenone)ethylenediamine.

consistently a considerable orbital contribution and the effective magnetic moment values of the Co(II) complexes (4.10 and 4.09 B.M.) are slightly greater than the spin-only value (3.87 B.M.), which suggest an octahedral geometry for the Co(II) complexes[10] in the high-spin state. The magnetic moment values of the Ni(II) complexes are 3.18 and 3.08 B.M., which are close to the spin-only value (2.83 B.M.) for octahedral Ni(II) complexes.[11] The magnetic moment values of the Cu(II) complexes are 1.96 and 1.99 B.M., which are close to the spin-only value (1.73 B.M.) expected for one unpaired electron, which offers the possibility of an octahedral geometry.[12] The magnetic moment determination shows that the Zn(II) and Cd(II) complexes are diamagnetic.

Infrared Spectra

All IR spectra exhibit a broad band in the region 3400–3000 cm^{-1}, suggesting the presence of coordinated water molecules.[13] The weak bands around 850 and 710 cm^{-1} are assigned as ν(OH) rocking and wagging vibrations, respectively.[14] The formation of the Schiff bases is confirmed by the presence of a band specific for ν(C=N) and the subsequent disappearance of bands due to aldehydic and amino groups. IR spectra of the Schiff bases exhibit a band at ~1645 cm^{-1} due to the azomethine group which undergoes a shift to a lower frequency 10–15 cm^{-1} on complexation, indicating the coordination of the azomethine nitrogen to the metal atom.[15] The two absorption bands occurring near 1595 and 1400 cm^{-1} in the Schiff base corresponding to the asymmetric and symmetric stretching vibration bands of the ionic carboxy group ν(COO) in the complexes are shifted to lower frequencies ~1585 and ~1390 cm^{-1}, respectively.[16] The ν(O–H) band originally found in the Schiff base disappeared on complexation indicating deprotonation of the phenolic hydroxyl group and coordination of phenolic oxygen to the metal. This is further supported by the shift in the stretching frequency of the phenolic ν(C–O)[17] at 1530 cm^{-1} to the higher side by ~10–20 cm^{-1}. In the low-frequency region, the bands observed in the region of ~525–535 cm^{-1} are attributed to ν(M–N),[18] in the region of ~460–470 cm^{-1} to $\nu(\mathrm{M–O})_{\mathrm{phenolic}}$, and in the region of ~415–420 cm^{-1} to $\nu(\mathrm{M–O})_{\mathrm{carboxy}}$.[19] The IR data suggest that the metal ion is bonded to the Schiff bases through the carboxylate oxygen, the phenolic oxygen, and azomethine group as well as the water molecule. The sharp bands in the range 750–780 and 1525–1535 cm^{-1} are due to aromatic ν(C–H)[20] and ν(C=C),[21] respectively. The bands in the range 1475–1480 cm^{-1} and 1370–1380 cm^{-1} are due to ν(C–H) of alkane and ν(C–CH$_3$), respectively.

Electronic Spectra

The ground state of the Mn(II) is $^6A_{1g}$. Mn(II) complexes in an octahedral[22] field should have three transitions corresponding to $^6A_{1g} \rightarrow {}^4T_{1g}$ (ν_1 $\sim$15,000 cm^{-1}), $^6A_{1g} \rightarrow {}^4T_{2g}$ (ν_2 $\sim$19,800 cm^{-1}), $^6A_{1g} \rightarrow {}^4E_g$, $^4A_{1g}$ (ν_3 $\sim$24,300 cm^{-1}) in increasing order of energy. The Co(II) complexes exhibit three spin-allowed bands, which can be assigned to $^4T_{1g} \rightarrow {}^4T_{2g}$ (ν_1 $\sim$9,400 cm^{-1}), $^4T_{1g} \rightarrow {}^4A_{2g}$ (ν_2 $\sim$17,800 cm^{-1}), and $^4T_{1g} \rightarrow {}^4T_{1g}(P)$ (ν_3 $\sim$19,100 cm^{-1}) transitions, respectively. The electronic spectra of the complexes exhibited three bands and their position indicates an octahedral geometry around Co(II).[23] The electronic spectra of the Ni(II) complexes are composed of three absorption bands: the first band appeared around ν_1 $\sim$10,550 cm^{-1} and is assigned to the $^3A_{2g} \rightarrow {}^3T_{2g}$ transition, the second is observed at ν_2 $\sim$17,400 cm^{-1} is attributed to the $^3A_{2g} \rightarrow {}^3T_{1g}$ transition, and the third one is located at ν_3 $\sim$25,100 cm^{-1} and is due to the $^3A_{2g} \rightarrow {}^3T_{1g}(P)$ transition, respectively, in an octahedral[24] environment. The ligand field splitting energy (10 Dq), interelectronic repulsion parameter (B), and covalency factor (nephelauxetic ratio) (β) for the Co(II) and Ni(II) complexes have been calculated using the secular equations given by König.[25]

For the Co(II) complexes:

$$10\,\mathrm{Dq} = \frac{1}{2}[(2\nu_1 - \nu_3) + (\nu_3^2 + \nu_1\nu_3 - \nu_1^2)^{1/2}]$$

$$15B = \nu_3 - 2\nu_1 + 10\,\mathrm{Dq}$$

$$\beta = \frac{B}{B_0}[B_0(\text{free ion}) = 971]$$

$$\beta^0 = (1 - \beta)100$$

For the Ni(II) complexes:

$$10\,\mathrm{Dq} = \nu_1$$

$$15\mathrm{B} = (\nu_2 + \nu_3) - 3\nu_1$$

$$\beta = \frac{B}{B_0}[B_0(\text{free ion}) = 1030]$$

$$\beta^0 = (1 - \beta)100$$

The calculated electronic parameter values of the Co(II) and Ni(II) mixed-ligand complexes are presented in Table 2. The Cu(II) complexes show one band appearing around ν_1 $\sim$10,550 cm^{-1} due to the $^2E_g \rightarrow {}^2T_{2g}$ transition suggesting an octahedral structure for the Cu(II) complexes.[26]

Table 2. Electronic spectral data of the Co(II) and Ni(II) complexes.

	Observed band (cm^{-1})							
Mixed-ligand complexes	ν_1	ν_2	ν_3	ν_2/ν_1	B	β	β^0	10 Dq
[Co(L)(benen)(H_2O)]	9,025	18,050	19,035	2.00	742	0.76	24	10,145
[Ni(L)(benen)(H_2O)]	10,310	17,325	25,110	1.68	767	0.74	26	10,310
[Co(L)(acephen)(H_2O)]	9,090	18,205	19,155	2.00	746	0.77	23	10,217
[Ni(L)(acephen)(H_2O)]	10,215	17,465	21,195	1.70	801	0.77	22	10,210

Note: KHL = potassium salt of salicylideneglycine; benen = *bis*(benzylidene) ethylenediamine; acphen = *bis*(acetophenone)ethylenediamine.

Thermogravimetric Analysis

It has been observed that all the mixed-ligand complexes show a loss in weight corresponding to one water molecule in the range ~150–180 °C indicating that this water molecule is coordinated to the metal ion.[27] In the temperature range 180–600 °C the ligand molecules are lost. In all the cases the final products are metal oxides. These results are in good accordance with the composition of the mixed-ligand complexes. The suggested structure of the mixed-ligand complexes are shown in Fig. 2.

Antibacterial Activity

Disc Diffusion Method

The antibacterial activity of the control (dimethyl sulfoxide), the standard drug (tetracycline), the metal salts, the ligands, and their mixed-ligands complexes were evaluated by the disc diffusion technique.[28] A stock solution of 250 µg/mL was made by dissolving 25 mg of each compound in dimethyl

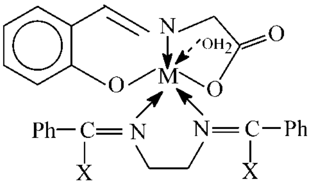

Figure 2. Structure of the mixed-ligand complexes [ML(SB)H_2O], where X = H (SB^1) or CH_3 (SB^2).

sulfoxide (100 mL). Filter paper (Whatman No. 4) discs (6 mm diameter) were soaked in solutions (20 μL) of the test compounds and after drying, placed on a nutrient agar plate containing lawn cultures of various bacteria. The plates were incubated at an optimum growth temperature of 37 °C. The zones of inhibition (in mm) around the discs were measured after 24 hr. The test bacteria included (a) *S. typhi*, (b) *E. coli*, and (c) *Serratia marcescens.* The control experiments were performed with equivalent volumes of solvents without added test compounds which were applied to the paper discs and the zone of inhibition was measured (in mm). All experiments were performed in triplicate and tetracycline was used as a standard drug. The mixed-ligand

Table 3. Antibacterial activities of the complexes.

	Zone of inhibition (in mm) (% inhibition)		
Compounds	*S. typhi*	*E. coli*	*Serratia marcesens*
DMSO (control)	9	12	15
Tetracycline	18 (100.00)	21 (75.00)	26 (73.33)
KHL	10 (11.11)	13 (08.33)	17 (13.33)
benen (SB^1)	11 (22.22)	14 (16.66)	17 (13.33)
acphen (SB^2)	11 (22.22)	13 (08.33)	16 (06.66)
$MnCl_2 \cdot 4H_2O$	10 (11.11)	13 (08.33)	16 (06.66)
$CoCl_2 \cdot 6H_2O$	10 (11.11)	13 (08.33)	17 (13.33)
$NiCl_2 \cdot 6H_2O$	11 (22.22)	14 (06.66)	16 (06.66)
$CuCl_2 \cdot 2H_2O$	11 (22.22)	13 (08.33)	17 (13.33)
$ZnCl_2$	10 (11.11)	14 (16.66)	17 (13.33)
$CdCl_2 \cdot 2H_2O$	11 (22.22)	14 (16.66)	17 (13.33)
[Mn(L)(benen)(H_2O)]	13 (44.44)	15 (25.00)	20 (33.33)
[Co(L)(benen)(H_2O)]	14 (55.55)	17 (41.66)	23 (55.33)
[Ni(L)(benen)(H_2O)]	13 (44.44)	16 (33.33)	19 (26.26)
[Cu(L)(benen)(H_2O)]	13 (44.44)	17 (41.66)	19 (26.66)
[Zn(L)(benen)(H_2O)]	11 (55.55)	18 (50.00)	21 (40.00)
[Cd(L)(benen)(H_2O)]	13 (44.44)	16 (33.33)	22 (46.66)
[Mn(L)(acphen)(H_2O)]	12 (33.33)	16 (33.33)	19 (26.66)
[Co(L)(acphen)(H_2O)]	14 (55.55)	18 (50.00)	22 (16.66)
[Ni(L)(acphen)(H_2O)]	13 (44.44)	15 (25.00)	20 (33.33)
[Cu(L)(acphen)(H_2O)]	13 (44.44)	15 (25.00)	23 (53.33)
[Zn(L)(acphen)(H_2O)]	14 (55.55)	17 (41.66)	23 (53.33)
[Cd(L)(acphen)(H_2O)]	13 (44.44)	16 (33.33)	21 (40.00)

Note: KHL = potassium salt of salicylideneglycine; benen = *bis*(benzylidene) ethylenediamine; acphen = *bis*(acetophenone)ethylenediamine.

complexes are soluble in dimethyl sulfoxide. Therefore, the growth was compared with dimethyl sulfoxide as the control, and is expressed as zone of inhibition and percentage inhibition versus control. The zone of inhibition and percentage inhibition of the complexes are recorded in Table 3. From the data it is observed that the mixed-ligand complexes show higher activity against bacteria, as compared to the control (blank), the ligands, and the metal salts. The mixed-ligand complexes show moderate activities as compared to the standard drug (tetracycline). The results suggest that the changes in structure due to the coordination in the mixed-ligand complexes decreases the growth of microorganisms relative to the metal ion, the ligands, and the control, hence, the complexation increases the antibacterial activity due to the presence of three azomethine (C═N) groups.[29] The patterns of activity are complex and no clear trend can be ascertained.

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