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PREPARATION, MAGNETIC, SPECTRAL, AND BIOCIDAL STUDIES OF SOME TRANSITION METAL COMPLEXES WITH 3,5-DIBROMOSALICYLIDENEANILINE AND NEUTRAL BIDENTATE LIGANDS

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ABSTRACT

The mixed-ligand complexes of transition metal ions like Mn(II), Co(II), Ni(II), Cu(II) and Cd(II) with 3,5-dibromosalicylideneaniline (HSB) and 2,2′-bipyridylamine (Bipy-amine) or bis(benzylidene)ethylenediamine (Benen) as the secondary ligand were prepared and characterized using elemental analyses, magnetic measurements, electronic and infrared spectra. They were also tested for their antimicrobial activities against bacteria, yeast and fungi.

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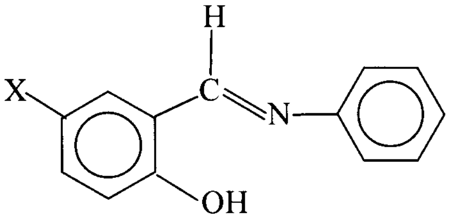

Figure 1. 3,5-Dibromosalicylideneaniline (HSB).

INTRODUCTION

Mixed-ligand complexes of Schiff bases are used as biological models and are known to possess tuberculostatic[1] fungicidal, bacteriostatic, antitumour and antiviral activities.[2] Schiff bases containing polyfunctional groups have produced stable metal complexes with transition, lanthanide and actinide metal ions.[3] In continuation of earlier work,[4,5] we prepared new mixed-ligand complexes of the types [M(SB)$_2$Bipy-amine] and [M(SB)$_2$Benen], where M = Mn(II), Co(II), Ni(II), Cu(II) and Cd(II). They were characterized on the basis of elemental analyses, magnetic measurements, electronic spectra, infrared spectra and antimicrobial activities. The structures of the ligands are shown in Figs. 1 and 2.

EXPERIMENTAL

Preparation of the Schiff Bases

The preparation of 3,5-dibromosalicylideneaniline was carried out by refluxing an ethanolic solution (100 mL) of aniline (0.1 mol, 0.93 g) with an

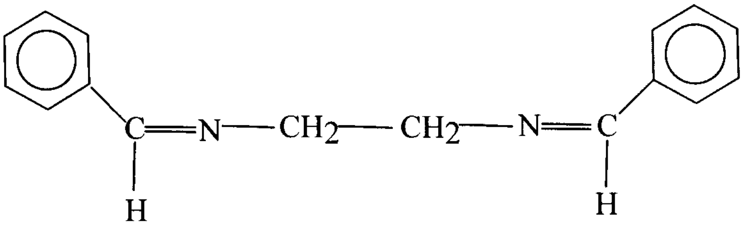

Figure 2. Bis(benzylidene)ethylenediamine (Benen).

ethanolic solution (100 mL) of 3,5-dibromosalicylaldehyde (0.1 mol, 2.80 g) for 2–3 h. The mixture was then allowed to cool to room temperature overnight. The formed yellow crystals were collected and dried in air. Yield, 2.79 g (75%).

The preparation of bis(benzylidene)ethylenediamine was carried out by refluxing an ethanolic solution (100 mL) of benzaldehyde (0.1 mol, 1.06 g) with an ethanolic solution (100 mL) of ethylenediamine (0.1 mol 0.60 g) for 2–3 hours. The solution was then allowed to cool to room temperature overnight. The formed yellow crystals were collected and dried in air. Yield, 1.16 g (70%).

Preparation of the Complexes

Bis(3,5-dibromosalicylideneaniline)diaquoM(II) [M = Mn(II), Co(II), Ni(II), Cu(II) and Cd(II)] were synthesized by a published procedure.[6] The preparation of the mixed-ligand complexes of bis(3,5-dibromosalicylideneaniline)Bipy-amineNi(II) was carried out by refluxing a dimethylformamide (100 mL) solution of bis(3,5-dibromosalicylideneaniline)diaquoNi(II) (0.02 mol, 1.48 g) with a dimethylformamide (100 mL) solution of 2,2′-bipyridylamine (0.02 mol, 0.34 g) or bis(benzylidene)ethylenediamine (0.02 mol, 0.47 g) for an hour. The solution was then concentrated to 15 mL, scratched and cooled in a refrigerator overnight. The obtained crystals were collected and recrystallized from chloroform. Finally, the crystals were dried in air. The complexes of Mn(II), Co(II), Cu(II) and Cd(II) were prepared similarly.

Analytical Procedures

Elemental analyses for carbon, hydrogen and nitrogen were carried out on a model 240 Perkin-Elmer elemental analyzer. Metal contents were determined by EDTA titration. The infrared spectra were measured on a FT-IR Nicolet 400D spectrophotometer in KBr pellets. The electronic spectra of the complexes were recorded on a Beckman DK-2A spectrophotometer using MgO as the reference. The melting points of the ligands and complexes were recorded in open capillaries in a capillary melting point apparatus (Tempo Instruments Co. Mumbai). The magnetic moments have been obtained by a vibrating sample magnetometer (model 7300 LakeShore 735 Controller, 450 Gaussmeter).

RESULTS AND DISCUSSION

The analytical data, colours, yields and melting points of the ligands and complexes are presented in Table I. The complexes were synthesized following the general procedures outlined by the following equations.

$$MCl_2{\cdot}nH_2O + 2HSB \longrightarrow [M(SB)_2(H_2O)_2] + 2HCl \quad (1)$$

$$[M(SB)_2(H_2O)_2] + \text{Bipy-amine} \longrightarrow [M(SB)_2\text{Bipy-amine}] + 2H_2O \quad (2)$$

$$[M(SB)_2(H_2O)_2] + \text{Benen} \longrightarrow [M(SB)_2\text{Benen}] + 2H_2O \quad (3)$$

Magnetic Measurements

The magnetic moment values of the complexes are listed in Table I. 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 consistently a considerable orbital contribution and the effective magnetic moment lies between 4.7–5.2 B.M. In the present case, the magnetic moment values (4.74 and 5.0 B.M) suggest an octahedral geometry[7] for the Co(II) complexes in the high-spin state. The magnetic moment values of the Cu(II) complexes are 1.72 and 1.70 B.M., respectively, which are very close to the spin-only value (1.73 B.M.) expected for one unpaired electron which offers the possibility of an octahedral geometry.[8] The magnetic moment values of the Mn(II) complexes (6.01 and 6.05 B.M.) are slightly greater than 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 high-spin and six-coordinated.[9] The magnetic moment values of the Ni(II) complexes are 2.74 and 2.98 B.M. indicating that these complexes are octahedral.[10] The magnetic moment determination shows that the Cd(II) complexes are diamagnetic.

Electronic Spectra

The electronic spectra provide the most detailed information about the electronic stucture. The electronic spectra of the metal chelates have been studied in solid state between 350 nm and 1700 nm. The electronic spectral data are all compatible with an octahedral geometry for the Ni(II)

Table I. Analytical Data of the Ligands and Complexes[a]

Complexes Empirical Formula	Mol. Wt.	Colour	% Found (Calculated)				M. p. (°C)	μ_{eff} (B.M.)	Yield %
			C	H	N	Metal			
HSB $C_{13}H_9Br_2ON$	354.80	Yellow	43.95 (43.96)	2.50 (2.53)	3.92 (3.94)	–	110	–	75.0
Benen $C_{16}H_{16}N_2$	236.00	Yellow	81.30 (81.35)	6.75 (6.77)	11.80 (11.86)	–	235	–	70.0
[Mn(SB)$_2$Bipy-amine] $C_{36}H_{25}Br_4MnO_2N_5$	933.54	Yellowish Brown	46.30 (46.27)	2.62 (2.67)	7.54 (7.49)	6.90 (6.88)	290	6.01	65.4
[Co(SB)$_2$Bipy-amine] $C_{36}H_{25}Br_4CoO_2N_5$	937.54	Light Brown	46.10 (46.07)	2.70 (2.66)	7.50 (7.46)	7.35 (7.34)	290	4.74	38.7
[Ni(SB)$_2$Bipy-amine] $C_{36}H_{25}Br_4NiO_2N_5$	937.31	Green	46.04 (46.08)	2.60 (2.64)	7.50 (7.45)	7.35 (7.31)	285	2.74	57.1
[Cu(SB)$_2$Bipy-amine] $C_{36}H_{25}Br_4CuO_2N_5$	942.15	Greenish brown	45.80 (45.85)	2.70 (2.65)	7.46 (7.42)	7.90 (7.87)	285	1.72	38.0
[Cd(SB)$_2$Bipy-amine] $C_{36}H_{25}Br_4CdO_2N_5$	991.00	Yellow	43.60 (43.59)	2.50 (2.52)	7.02 (7.06)	13.18 (13.13)	285	–	62.3
[Mn(SB)$_2$Benen] $C_{42}H_{32}Br_4MnO_2N_4$	998.54	Yellowish Brown	50.50 (50.47)	3.18 (3.20)	5.62 (5.60)	5.55 (5.50)	280	6.05	70.1
[Co(SB)$_2$Benen] $C_{42}H_{32}Br_4CoO_2N_4$	1002.54	Light Brown	50.27 (50.27)	3.19 (3.20)	5.53 (5.58)	5.89 (5.87)	285	5.00	68.9
[Ni(SB)$_2$Benen] $C_{42}H_{32}Br_4NiO_2N_4$	1002.31	Green	50.30 (50.28)	3.15 (3.19)	5.60 (5.58)	5.90 (5.85)	295	2.98	72.1
[Cu(SB)$_2$Benen] $C_{42}H_{32}Br_4CuO_2N_4$	1007.15	Greenish Brown	50.02 (50.04)	3.20 (3.17)	5.59 (5.56)	6.32 (6.30)	280	1.70	58.8
[Cd(SB)$_B$enen] $C_{42}H_{32}Br_4CdO_2N_4$	1056.00	Yellow	47.70 (47.72)	3.00 (3.03)	5.28 (5.30)	10.62 (10.64)	285	–	50.0

[a] HSB = 3,5-Dibromosalicylideneaniline.

complexes. Thus, the three d-d transition bands in the spectra occuring at $\sim$10,500, $\sim$16,900, $\sim$23,800 cm^{-1} may be assigned to $^3A_{2g}(F)\rightarrow{}^3T_{2g}(F)(\upsilon_1)$, $^3A_{2g}(F)\rightarrow{}^3T_{1g}(F)(\upsilon_2)$ and $^3A_{2g}(F)\rightarrow{}^3T_{1g}(P)(\upsilon_3)$ transitions, respectively, assuming octahedral geometry.[11] The Cu(II) complexes exhibit a broad band at $\sim$15,000 cm^{-1} assigned to the $^2E_g\rightarrow{}^2T_{2g}$ transition in accord with an octahedral geometry.[12] The electronic spectra of the Co(II) complexes exhibit three absorption bands at $\sim$9,300, $\sim$18,000 and $\sim$19,200 cm^{-1} which may be assigned to the $^4T_{1g}(F)\rightarrow{}^4T_{2g}(F)(\upsilon_1)$, $^4T_{1g}(F)\rightarrow{}^4A_{2g}(\upsilon_2)$ and $^4T_{1g}(F)\rightarrow{}^4T_{1g}(P)$ (υ_3) transitions.[13] The Mn(II) complexes exhibit three weak bands, which may be attributed to the transitions $^6A_{1g}\rightarrow{}^4T_{1g}$ (^{4}G) at $\sim$14,500 cm^{-1}, $^6A_{1g}\rightarrow{}^4T_{2g}$(^{4}G) at $\sim$19,000 cm^{-1} and $^6A_{1g}\rightarrow{}^4T_{1g}$, ^4E_g (^{4}G) at $\sim$25,000 cm^{-1}) for an octahedral geometry.[14] The values of the ligand field splitting energy (Dq), Racah interelectronic repulsion (B), Nephelauxetic ratio (β) and ratio υ_2/υ_1 for the Ni(II) and Co(II) complexes are presented in Table III.

Infrared Spectra

The infrared spectra of the metal chelates, in comparison with those of the free ligands, display certain changes which give an idea about the types of bonds and their structures. The important infrared spectral frequencies are given in Table II. 3,5-Dibromosalicylideneaniline exhibits a medium-intense band at 2700 cm^{-1} due to intramolecularly hydrogen bonded υ(OH).[15] The

Table II. Infrared Spectral Data of the Ligands and Complexes[a]

Compounds	υ(C-O)	υ(C=N)	υ(C-N)	υ(C-C)	υ(M-O)	υ(M-N)
HSB	1525 s	1640 s	—	1575 s	—	—
Bipy-amine	—	1585 s	1165 s	—	—	—
Benen	—	1642 sh	1160 s	1575 s	—	—
[Mn(SB)$_2$Bipy-amine]	1530 s	1625 sh	1175 s	1590 s	410 m	460 w
[Co(SB)$_2$Bipy-amine]	1530 s	1625 sh	1175 s	1585 s	410 w	460 w
[Ni(SB)$_2$Bipy-amine]	1540 s	1629 s	1175 s	1575 sh	420 m	450 m
[Cu(SB)$_2$Bipy-amine]	1540 s	1629 s	1165 s	1575 sh	420 w	450 w
[Cd(SB)$_2$Bipy-amine]	1540 s	1629 s	1165 s	1590 sh	420 m	440 w
[Mn(SB)$_2$Benen]	1530 s	1629 s	1165 s	1590 sh	410 m	440 w
[Co(SB)$_2$Benen]	1530 s	1629 s	1160 s	1590 m	410 w	460 w
[Ni(SB)$_2$Benen]	1535 s	1625 s	1170 s	1590 m	420 m	460 w
[Cu(SB)$_2$Benen]	1535 s	1625 s	1175 s	1585 s	420 m	450 w
[Cd(SB)$_2$Benen]	1535 s	1625 s	1160 s	1585 s	420 w	450 w

[a] b = broad, m = medium, s = strong, sh = shoulder, w = weak.

Table III. Electronic Spectral Data of the Co(II) and Ni(II) Complexes[a]

	Observed Bands (cm^{-1})							
Complexes	υ_1	υ_2	υ_3	υ_2/υ_1	B	β	β^0	Dq
[Co(SB)$_2$Bipy-amine]	93,00	18,100	19,030	1.954	724.568	0.746	25.37	10,42.0
[Ni(SB)$_2$Bipy-amine]	10305	18,180	23,250	1.763	700.454	0.680	32.00	1,031.0
[Co(SB)$_2$Benen]	9302	18,181	19,047	1.954	724.568	0.746	25.37	10,42.0
[Ni(SB)$_2$Benen]	10.310	17,390	19,000	1.640	676.260	0.666	34.40	1,042.0

[a] HSB = 3,5-Dibromosalicylideneaniline.

absense of this band in the complexes indicates the deprotonation of the phenolic groups and coordination of the oxygen atom to the metal ion. In the free ligand a strong band at 1525 cm^{-1} due to υ(C-O) (phenolic) shifts to higher energy by 5–15 cm^{-1} in the complexes indicating the coordination of the phenolic oxygen atom to the metal ion. This shift to higher energy is expected due to the maintenance of ring currents arising from the electron delocalization in the chelate ring. The υ(C=N) frequency of 2,2′-bipyridylamine occurs at 1585 cm^{-1}. This band shifts to higher energy by 30–35 cm^{-1} in the complexes indicating the bidentate (N,N-dipyridyl nitrogens) coordination of this heterocydic ligand. The υ(C=N) frequency of bis(benzylidene)ethylenediamine occurs at 1640 cm^{-1}. This band shifts to lower energy by 10–20 cm^{-1} in the complexes indicating coordination through the azomethine nitrogen.[16] The frequencies in the range 450–460 cm^{-1} and 410–420 cm^{-1} are attributed to υ(M-N)[17] and υ(M-O),[18] respectively.

The probable structures of the complexes are shown in Figs. 3 and 4.

Antimicrobial Activity

The antimicrobial activities of the ligands, metal salts and their corresponding complexes were tested against *Salmonella typhi* (bacteria), *Saccharomyces* c*eresuiae* (yeast) and *Lasiodiplodia theobromae, Fusarium oxysporum* (fungi). The antimicrobial activities of the compounds were tested on *Salmonella typhi* (bacteria) and *Saccharomyces ceresuiae* (yeast) using the "Agar Diffusion Method".[19] The fungal activity of the compounds has been screened against *Lasiodiplodia theobromae*, *Fusarium oxysporum* (fungi) using the "Potato Dextrose Agar Method".[20] All of the compounds have significant antibacterial activity at 5×10^{-4} g mL^{-1} against bacteria, yeast and fungi. The growth was compared with

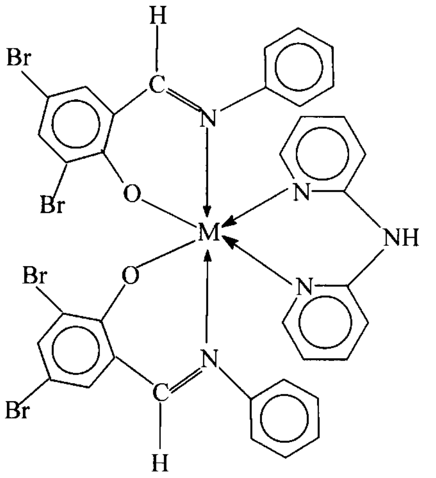

Figure 3. Suggested structure of the complexes [M(SB)$_2$Bipy-amine]. M = Mn(II), Co(II), Ni(II), Cu(II) and Cd(II); HSB = 3,5-Dibromosalicylineaniline.

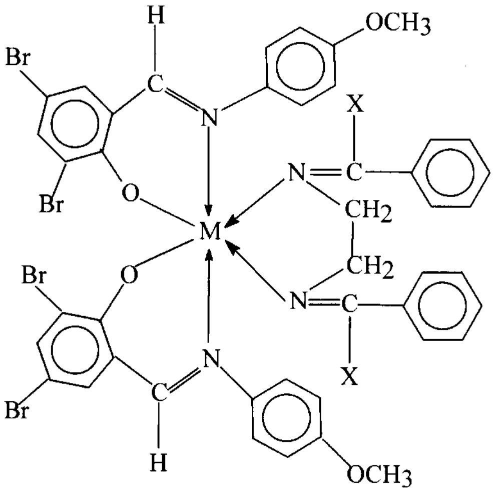

Figure 4. Suggested structure of the complexes [M(SB)$_2$Benen]. M = Mn(II), Co(II), Ni(II), Cu(II) and Cd(II); HSB = 3,5-Dibromosalicylineaniline; X = H.

Table IV. Antimicrobial Activity of the Complexes[a]

	% Inhibition			
	Bacteria	Yeast	Fungi	
Compounds	*Salmonella typhi*	*Saccharomyceh ceresaiue*	*Lasiodiplodia theobromae*	*Fusarium oxysporum*
$MnCl_2{\cdot}4H_2O$	22.22	21.05	20.00	15.30
$CoCl_2{\cdot}6H_2O$	15.16	10.52	06.60	07.70
$NiCl_2{\cdot}6H_2O$	11.11	08.26	10.30	10.30
$CuCl_2{\cdot}2H_2O$	22.22	15.78	20.00	15.30
$CdCl_2{\cdot}H_2O$	16.66	08.26	06.60	07.70
HSB	38.88	31.50	20.00	15.30
Bipy-amine	11.11	05.26	06.60	07.70
Benen	33.33	05.00	20.00	37.00
[Mn(SB)$_2$Bipy-amine]	66.66	52.63	50.00	50.00
[Co(SB)$_2$Bipy-amine]	50.00	47.36	40.00	46.00
[Ni(SB)$_2$Bipy-amine]	66.66	57.89	53.30	50.00
[Cu(SB)$_2$Bipy-amine]	61.11	52.63	26.60	61.50
[Cd(SB)$_2$Bipy-amine]	66.00	57.89	26.60	46.00
[Mn(SB)$_2$Benen]	61.11	47.36	33.30	46.00
[Co(SB)$_2$Benen]	61.11	57.89	33.30	53.00
[Ni(SB)$_2$Benen]	66.66	63.15	33.30	61.30
[Cu(SB)$_2$Benen]	66.66	63.15	46.60	46.00
[Cd(SB)$_2$Benen]	50.00	57.89	40.00	53.00

[a] HSB = 3,5-Dibromosalicylideneaniline.

dimethyformamide as the control and is expressed as percentage versus control.

The percentage inhibition of various complexes is recorded in Table III. The data represent the values of five replicates. From the data it is observed that the complexes show higher activity against bacteria, yeast and fungi as compared to the control (blank), the ligands and metal salts. The results suggest that the change in structure due to coordination in the complexes decreases the growth of microorganisms. Hence, complexation increases the antimicrobial activity. The complexes show higher activity against *Salmonella typhi* (bacteria) than *Saccharomyles cereulsae* (yeast) and *Lasiodiplodia theobromae*, *Fusariunl oxysporum* (fungi). The complexes also show higher activity against *Saccharomyces cereuisae* (yeast) than *Lasiodiplodia theobromae, Fusarium oxysporum* (fungi). The patterns of activity are complex and no clear trend can be ascertained.

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