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Studies on Some Transition Metal Complexes with Bidentate Schiff Bases

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Studies on Some Transition Metal Complexes with Bidentate Schiff Bases

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ABSTRACT

Metal complexes of the types [M(HSB-Cl)₂benen] and [M(HSB-Br)₂benen] where M = Mn(II), Co(II), Ni(II), Cu(II), and Cd(II); HSB-Cl = 5-chlorosalicylidene-*p*-anisidine, HSB-Br = 5-bromosalicylidene-*p*-anisidine and benen = *bis*(benzylidene)ethylenediamine have been prepared and characterized on the basis of elemental analyses, magnetic susceptibility measurements, electronic and infrared spectra. An octahedral structure has been assigned to all of the prepared complexes. The complexes show higher antimicrobial activity against the *Fasarium oxysporum* (fungi), *Salmonella typhi* (bacteria), *Saccharomyces cerevisiae* (yeast), *Lasiodiplodia theobromae* (fungi) as compared to the ligands, metal salt, and control.

Key Words: Metal complexes; Mixed-ligand complexes; Schiff base; Antimicrobial activity.

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INTRODUCTION

Mixed-ligand complexes are well known to play an important role in biological systems.^[1] In continuation of our earlier work,^[2] we report here the preparation, spectroscopic, and biocidal studies of Mn(II), Co(II), Ni(II), Cu(II), and Cd(II) complexes with 5-chlorosalicylidene-*p*-anisidine (HSB-Cl) or 5-bromosalicylidne-*p*-anisidine (HSB-Br) and *bis*(benzylidene)ethylene-diamine (benen) as a secondary ligand (Fig. 1).

EXPERIMENTAL

Materials and Measurements

The IR spectra were recorded on a FT-IR Nicolet 400 D spectrophotometer in KBr pellets. The elemental analyses were carried out using a model 240 Perkin–Elmer elemental analyzer. Metal contents were determined



5-Chlorosalicylidene-*p*-anisidine (HSB-Cl) (X = Cl);
5-Bromosalicylidene-*p*-anisidine (HSB- Br) (X = Br)



Bis(benzylidene)ethylenediamine (benen)

Figure 1. Structure of the ligands.





by EDTA titration. Magnetic susceptibility measurements were done on a model 7300 vibrating sample magnetometer. The electronic spectra were recorded on a Beckman DK-2A spectrophotometer using MgO as a reference. The melting points of the ligands and complexes were recorded in open capillaries in a capillary melting point apparatus (Tempo Instruments Co. Mumbai).

Preparation of the Schiff Bases

The preparation of HSB-Cl or HSB-Br was carried out by refluxing a solution of 10 mmol (1.23 g) of *p*-anisidene in 100 mL ethanol with a solution of 10 mmol of 5-chlorosalicylaldehyde (1.56 g) or 5-bromosalicylaldehyde (2.00 g) in 100 mL ethanol for 2-3 hr. The reaction mixture was then allowed to cool to room temperature. The yellow crystals that formed were collected and dried in air.

The preparation of benen was carried out by refluxing a solution of 2 mmol (0.212 g) of benzaldehyde in 100 mL ethanol and a solution of 2 mmol (0.06 g) of ethylenediamine in 100 mL ethanol for 2-3 hr. The reaction mixture gave the Schiff base in the form of yellow crystals. The product was filtered and washed with ether and dried in air. Yield, 0.15 g (75.0%).

Preparation of the Complexes

Bis(5-chlorosalicylidene-*p*-anisidine)diaquoM(II) or *bis*(5-bromosalicylidene-*p*-anisidene)diaquoM(II), [M = Mn(II), Co(II), Ni(II), Cu(II), andCd(II)] were synthesized by a published procedure.^[3] The preparation of themixed-ligand complexes of*bis*(5-chlorosalicylidene-*p*-anisidine)benenNi(II) or*bis*(5-bromosalicylidene-*p*-anisidine)benenNi(II) was carried out by refluxinga solution of 2 mmol of*bis*(5-chlorosalicylidene-*p*-anisidine)diaquoNi(II)(1.22 g) or*bis*(5-bromosalicylidene-*p*-anisidine)diaquaNi(II) (1.42 g) in 100 mLdimethylformamide with a solution of 2 mmol of*bis*(benzylidene)ethylenediamine (0.47 g) in 100 mL dimethylformamide for an hour. The solution wasthen concentrated to 15 mL, scratched in the flask and cooled in a refrigeratorovernight. The obtained crystals were collected and recrystallized fromchloroform. Finally, the crystals were dried in air. The complexes of Mn(II),Co(II), Cu(II), and Cd(II) were prepared similarly.

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RESULTS AND DISCUSSION

Elemental analysis data, formula weights, colors, percentage yields, melting points, and magnetic moment data are given in Table 1. The compounds in this work were synthesized following the general procedures outlined in the following equations.

$$MCI_2 \cdot nH_2O + 2HSB \xrightarrow{H_2O} [M(SB)_2(H_2O)_2] + 2HCl + (n-2)H_2O$$
$$[M(SB)_2(H_2O)_2] + benen \xrightarrow{ErOH, Reflux} [M(SB)_2benen] + 2H_2O$$

Here HSB = HSB-Cl or HSB-Br.

The magnetic moment values of the Mn(II) complexes are 6.00 and 6.01 B.M., respectively, and are within the limits of the spin-free value for five-unpaired electrons, due to a high-spin d⁵ system.^[4] The electronic spectra of the Mn(II) complexes exhibit three weak absorption bands at ~14,500, ~18,000, and ~25,000 cm⁻¹ assigned to ${}^{6}A_{1g} \rightarrow {}^{4}T_{1g}({}^{4}G) (\nu_{1})$, ${}^{6}A_{1g} \rightarrow {}^{4}T_{2g}({}^{4}G) (\nu_{2})$, and ${}^{6}A_{1g} \rightarrow {}^{4}A_{1g}$, ${}^{4}E_{g}({}^{4}G) (\nu_{3})$ transitions.^[5]

The magnetic moment values of the Co(II) complexes have been used as criterion 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 4.70 and 5.20 B.M. In the present case, the magnetic moment values (4.50 and 4.14 B.M.) suggest^[6] an octahedral geometry for the Co(II) complex in the high-spin state. The complexes exhibit three bands at ~9,200, ~18,500, and ~19,500 cm⁻¹. These bands are assigned to ${}^{4}T_{1g}(F) \rightarrow {}^{4}T_{2g}(F)$ (ν_1), ${}^{4}T_{1g}(F) \rightarrow {}^{4}A_{2g}$ (ν_2), and ${}^{4}T_{1g}(F) \rightarrow {}^{4}T_{1g}(P)$ (ν_3) transitions, respectively, consistent with an octahedral geometry.^[7] The magnetic moment values of the Ni(II) complexes are 2.71 and 2.67 B.M., which are in the range usually expected for similar hexa-coordianted Ni(II) metal ions.^[8] The bands observed at ~10,500, ~18,500, and ~24,000 cm⁻¹, are assigned to ${}^{3}A_{2g}(F) \rightarrow {}^{3}T_{2g}(F)$ (ν_1), ${}^{3}A_{2g}(F) \rightarrow {}^{3}T_{1g}(F)$ (ν_2), and ${}^{3}A_{2g}(F) \rightarrow {}^{3}T_{1g}(P)$ (ν_3) transitions, respectively, which suggest an octahedral geometry.^[9]

The observed magnetic moment values of the Cu(II) complexes are 1.89 and 1.99 B.M., which are very close to the spin-only value (1.83 B.M.) expected for one unpaired electron,^[10] and the band observed at $\sim 14,000 \text{ cm}^{-1}$ is assignable to the ${}^{2}E_{g} \rightarrow {}^{2}T_{2g}(\text{F})$ transition, which also suggests an octahedral geometry.^[11] The magnetic moment determination shows that the Cd(II) complexes are diamagnetic. The value of the ligand field splitting energy (D_{q}), nephelauxetic ratio (β), and ratio ν_{2}/ν_{1} for the Ni(II) and Co(II) complexes are presented in Table 2.

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				% Found (calculated)				
Compound empirical formula	Formula weight	Color	C	Н	N	Metal	M.p. (°C)	$\mu_{\rm eff}$ (B.M.)	%Yield (g)
HSB-CI	261.00	Yellow	64.36	4.59	5.36		265		70.00
$C_{14}H_{12}CIO_2N$			(64.32)	(4.55)	(5.32)				(1.95)
HSB-Br	305.90	Yellow	54.91	3.92	4.57	I	270	I	78.00
$C_{14}H_{12}BrO_2N$			(54.90)	(3.90)	(4.52)				(2.51)
Benen	236.00	Yellow	81.35	6.77	11.86	I	235		70.00
C ₁₆ H ₁₆ N ₂			(81.30)	(6.75)	(44.81)				(0.15)
[Mn(SB-Cl) ₂ benen]	810.94	Yellowish	65.10	4.68	6.90	6.77	280	6.00	58.09
$C_{44}H_{38}Cl_2MnN_4O_4$		brown	(65.11)	(4.66)	(6.92)	(6.72)			(0.98)
[Co(SB-CI) ₂ benen]	814.94	Light	64.79	4.66	6.87	7.23	280	4.50	50.58
$C_{44}H_{38}Cl_2CoN_4O_4$		brown	(64.80)	(4.62)	(6.85)	(7.20)			(0.85)
[Ni(SB-Cl) ₂ benen]	814.71	Green	64.80	4.66	6.87	7.20	285	2.71	60.12
$C_{44}H_{38}Cl_2NiN_4O_4$			(64.75)	(4.60)	(6.86)	(7.15)			(1.02)
[Cu(SB-CI) ₂ benen]	819.55	Greenish	64.42	4.63	6.83	7.75	280	1.89	62.00
$C_{44}H_{38}CuN_4O_4$		brown	(64.40)	(4.64)	(6.82)	(7.74)			(1.05)



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	-			% Found (6	calculated)		;		
Compound empirical formula	Formula weight	Color	С	Н	N	Metal	M.p. (°C)	$\mu_{\rm eff}$ (B.M.)	% Y ield (g)
[Cd(SB-CI) ₂ benen]	868.40	Yellow	60.80	4.37	6.44	12.94	280	I	70.00
$C_{44}H_{38}Cl_2CdN_4O_4$			(60.79)	(4.35)	(6.40)	(12.90)			(1.26)
[Mn(SB-Br) ₂ benen]	900.74	Yellowish	58.61	4.21	6.21	60.9	290	6.01	58.12
$\mathrm{C}_{44}\mathrm{H}_{38}\mathrm{Br}_{2}\mathrm{MnN}_{4}\mathrm{O}_{4}$		brown	(58.60)	(4.20)	(6.24)	(6.03)			(1.08)
[Co(SB-Br) ₂ benen]	904.74	Light	58.35	4.20	6.18	6.48	292	4.14	68.00
$\mathrm{C}_{44}\mathrm{H}_{38}\mathrm{Br}_{2}\mathrm{CoN}_{4}\mathrm{O}_{4}$		brown	(58.31)	(4.17)	(6.16)	(6.50)			(1.27)
[Ni(SB-Br) ₂ benen]	904.51	Green	58.37	4.20	6.19	6.49	280	2.67	60.58
$\mathrm{C}_{44}\mathrm{H}_{38}\mathrm{Br}_{2}\mathrm{NiN}_{4}\mathrm{O}_{4}$			(58.35)	(4.18)	(6.20)	(6.48)			(1.13)
[Cu(SB-Br) ₂ benen]	909.35	Greenish	58.06	4.17	6.15	7.02	290	1.99	75.88
$\mathrm{C}_{44}\mathrm{H}_{38}\mathrm{Br}_{2}\mathrm{CuN}_{4}\mathrm{O}_{4}$		brown	(58.08)	(4.15)	(6.18)	(7.05)			(1.29)
[Cd(SB-Br) ₂ benen]	958.20	Yellow	55.10	3.96	5.84	11.73	285		60.00
$C_{44}H_{38}Br_2CdN_4O_4$			(55.12)	(3.92)	(5.82)	(11.77)			(1.19)
^a HSB-Cl = 5 -chlorosalicylid	ene-p-aniside	ne; HSB-Br =	5-bromosal	icylidene-p-	anisidene.				

Table 1. Continued.

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	Table 2.	Electronic sl	bectral data of	f the Ni(II) and	nd Co(II) compl	exes. ^a		
	Obser	ved bands (c	m^{-1})					
Complexes	ν_1	ν_2	ν_3	ν_2/ν_1	В	β	$\beta^{\rm o}$	$D_{ m q}$
[Co(SB-CI) ₂ benen]	9,200	18,000	19,000	1.900	724.560	0.746	25.37	1042.0
[Ni(SB-CI) ₂ benen]	10,300	18, 180	23,300	1.763	700.450	0.680	32.00	1030.00
[Co(SB-Br) ₂ benen]	9,300	18,000	19,050	1.974	724.560	0.746	25.31	1042.0
[Ni(SB-Br) ₂ benen]	10,310	18,100	23,000	1.766	676.260	0.668	34.40	1031.0
^a HSB-Cl = 5-chlorosalicylide	ene- <i>p</i> -anisiden	e; HSB-Br =	= 5-bromosali	cylidene-p-ar	iisidene.			

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The infrared spectra of the HSB-Cl and HSB-Br exhibit a medium-intense band at ~2700 cm⁻¹, which may be assigned to the intramolecularly hydrogen bonded ν (OH).^[12] The absence 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 which may be assigned to ν (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.^[13] This band shift to higher energy is expected due to the maintenance of ring currents arising from the electron delocalization in the chelate ring. The infrared spectra of benen, HSB-Cl, and HSB-Br exhibit the C==N stretching band at ~1645 cm⁻¹. This band shifts to lower energy by 10–20 cm⁻¹ in the mixed-ligand complexes indicating coordination through the azomethine nitrogen.^[14]

The complexes of HSB-Cl exhibit a band at \sim 750 cm⁻¹, which may be due to ν (C–Cl).^[15] The complexes of HSB-Br exhibit a band at \sim 685 cm⁻¹ which may be due to ν (C–Br).^[16] The frequencies in the range \sim 2815–2830 and \sim 1375–1385 cm⁻¹ may be due to ν (C–OCH₃)^[17] and ν (C–CH₃),^[18] respectively. The bands observed in the range 455–465 and 420–430 cm⁻¹ may be attributed to ν (M–N)^[19] and ν (M–O),^[20] respectively.

The suggested structure of the complexes is shown in Fig. 2.

Antimicrobial Activity Study

The medicinal potency of many coordination compounds has been established by their antitumor, antiviral,^[21] and antimalarial^[22] activities. These



Figure 2. Suggested structure of the complexes $[M(SB-Cl)_2benen]$ [X = Cl] or $[M(SB-Br)_2benen]$ [X = Br].





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characteristic properties have been related to the ability of the metal ion to form complexes^[23] with ligands containing nitrogen and oxygen donor atoms. It has been observed^[24] that chelation in compounds is to a large extent responsible for the antimicrobial activity. In view of the medicinal potency of the ligands, the metal salts and the metal complexes, we evaluated the antimicrobial activity of the compounds against bacteria and yeast using the "Agar Diffusion Method." ^[25] The antifungal activity of the compounds has been screened against *Lasiodiplodia theobromae* and *Fusarium oxysporum* using the "Potato Dextrose Method." ^[26] All of the compounds have significant antimicrobial activity at 5×10^{-4} g mL⁻¹ against *Salmonella typhi*, *Saccharamyces cerevisiae*, *L. theobromae*, and *F. oxysporum*. The compounds are soluble in dimethylformamide. Therefore, the growth was compared with dimethylformamide as the control and is expressed as percentage inhibition vs. controls. The percentage inhibition of various compounds are recorded in Table 3. The

		% I	nhibition	
	D ()	N 7	Fu	ngi
Compounds	Bacteria S. typhi	S. cerevisiae	L. theobromae	F. oxysporum
$MnCl_2 \cdot 4H_2O$	22.22	21.05	20.00	15.30
$CoCl_2 \cdot 6H_2O$	15.16	10.52	6.60	7.70
$NiCl_2 \cdot 6H_2O$	11.11	8.26	10.30	10.30
$CuCl_2 \cdot 2H_2O$	22.22	15.78	20.00	15.30
$CdCl_2 \cdot 2H_2O$	16.66	8.26	6.60	7.70
HSB-Cl	22.20	15.78	6.60	23.00
HSB-Br	27.77	26.31	13.30	37.00
Benen	38.33	5.00	20.00	36.00
[Mn(SB-Cl) ₂ benen]	55.50	50.00	46.60	61.30
[Co(SB-Cl) ₂ benen]	55.00	53.63	53.30	80.00
[Ni(SB-Cl) ₂ benen]	68.66	68.40	40.00	62.00
[Cu(SB-Cl) ₂ benen]	66.66	57.89	54.00	69.00
[Cd(SB-Cl) ₂ benen]	72.22	52.63	46.00	80.00
[Mn(SB-Br) ₂ benen]	66.66	53.00	47.00	62.00
[Co(SB-Br) ₂ benen]	52.50	42.10	33.30	53.00
[Ni(SB-Br) ₂ benen]	66.66	52.63	46.00	69.00
[Cu(SB-Br) ₂ benen]	55.00	42.10	40.00	61.30
[Cd(SB-Br) ₂ benen]	56.00	53.00	47.60	61.30

Table 3. Antimicrobial activities of the complexes.^a

^aHSB-Cl = 5-chlorosalicylidene-*p*-anisidene; anisidene.

HSB-Br = 5-bromosalicylidene-p-



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data represent the average values of five replicates. From the data it is observed that the complexes show higher activity against *S. typhi*, *S. cerevisiae*, *L. theobromae*, and *F. oxysporum* compared to the control, metal salts, and ligands.

The results suggest that changes in structure due to coordination in the complexes decreases the growth of microorganisms relative to the metal ions and ligands. Hence, complexation increases the antimicrobial activity due to the presence of two halogen and four azomethine (C==N) groups. The complexes are more active against *S. typhi* and *F. oxysporum* compared to *S. cerevisiae* and *L. theobromae*. The complexes are also more active against *S. cerevisiae* compared to *L. theobromae*. The pattern of activity is complex and no clear trend can be ascertained.

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