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Preparation, Characterization, and Antimicrobial Activities of Some Mixed-Ligand Complexes of Mn(II), Co(II), Ni(II), Cu(II), and Cd(II) with Monobasic Bidentate (ON) Schiff Base and Neutral Bidentate (NN) Ligands

Prakash P. Dholakiya [a] & M. N. Patel [a]
[a] Department of Chemistry , Sardar Patel University , Vallabh Vidyanagar, 388 120, Gujarat, India
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SYNTHESIS AND REACTIVITY IN INORGANIC AND METAL-ORGANIC CHEMISTRY
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Preparation, Characterization, and Antimicrobial Activities of Some Mixed-Ligand Complexes of Mn(II), Co(II), Ni(II), Cu(II), and Cd(II) with Monobasic Bidentate (ON) Schiff Base and Neutral Bidentate (NN) Ligands

Prakash P. Dholakiya and M. N. Patel*

Department of Chemistry, Sardar Patel University,
Gujarat, India

ABSTRACT

Complexes of Mn(II), Co(II), Ni(II), Cu(II), and Cd(II) with 3,5-dibromosalicylidene-*p*-anisidene (HSB) and 2,2′-bipyridylamine (bipyamine) or *bis*(benzylidene)ethylenediamine (benen) or *bis*(acetophenone) ethylenediamine (acphen) have been prepared. They were characterized by elemental analyses, magnetic measurements, electronic and infrared spectra. All the ligands, metal salts, and their complexes were screened for

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*Correspondence: M. N. Patel, Department of Chemistry, Sardar Patel University, Vallabh Vidyanagar, 388 120, Gujarat, India; E-mail: jeenenpatel@yahoo.co.in.

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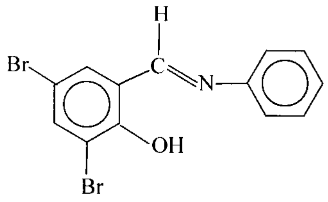
their antimicrobial activities against bacteria, yeast, and fungi and the results are discussed.

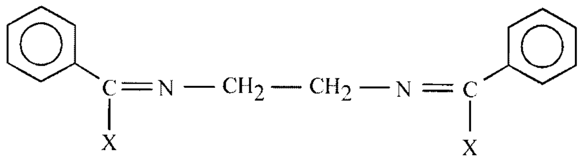
Key Words: Ligands; Metal salts; Schiff bases; Antimicrobial activities.

INTRODUCTION

In order to emulate the biological activity of proteins and enzymes,[1] various complexes containing Schiff bases have been prepared.[2] A lot of work on the synthesis and biological activity of Schiff base metal complexes has been done. These investigations show that these compounds are active against bacteria, viruses, and cancers. In continuation of earlier work[3,4] the present study describes the synthesis and characterization of the 3,5-dibromosalicylidene-*p*-anisidene complexes of Mn(II), Co(II), Ni(II), Cu(II), and Cd(II) with the HSB and their ligand replacement reactions with the bidentate nitrogen donor ligands dipy-amine, benen, or acphen (Fig. 1). The compounds were evaluated for their antimicrobial activities.



3,5-Dibromosalicylidene-*p*-anisidine (HSB)



Bis(acetophenone)ethylenediamine (acphen) X = CH_3 or

Bis(benzylidene)ethylenediamine (benen) X = H

Figure 1. Structures of the ligands.

Analytical Procedure

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 Nicolet 400 D FT-IR 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 7304 LakeShore 735 Controller, 450 Gaussmeter).

EXPERIMENTAL

Materials

All chemicals used in the synthesis were of A. R. grade. 2,2′-Bipyridyamine was obtained from Lancaster (UK). Stock solutions of Mn(II), Co(II), Ni(II), Cu(II), and Cd(II) chlorides were prepared and standardized by complexometric titration.[5] Conductivity water was used in the synthesis of the complexes.

Preparation of the Schiff Bases

Bis(benzylidene)ethylenediamine (benen) was synthesized by a published procedure.[6] The preparation of 3,5-dibromosalicylidene-*p*-anisidine was carried out by refluxing a solution of 10 mmol of *p*-anisidine (1.23 g) in 100 mL ethanol with a solution of 10 mmol of 3,5-dibromosalicylaldehyde (2.79 g) in 100 mL ethanol for 2–3 hr, and then allowing the reaction mixture to cool to room temperature overnight. The yellow crystals formed were collected, dried in air, and recrystallized from ethanol, yield: 3.01 g (75.0%), melting point: 101 °C. anal. calcd for $C_{13}H_9Br_2N_1O_1$ (355): C, 43.54%; H, 3.06%; N, 3.62%. Found: C, 43.50%; H, 3.02%; N, 3.60%.

The preparation of *bis*(acetophenone)ethylenediamine was carried out by refluxing a solution of 10 mmol of acetophenone (1.20 g) in 100 mL ethanol with a solution of 10 mmol of ethylenediamine (0.60 g) in 100 mL ethanol for 2–3 hr. The reaction mixture then was allowed to cool to room temperature overnight. The yellow microcrystals were collected, dried in air, and recrystallized from ethanol, yield: 2.26 g (75.0%), melting point: 260 °C, anal. calcd. for $C_{18}H_{20}N_2$ (264): C, 81.81%; H, 7.57%; N, 10.60%. Found: C, 81.79%; H, 7.60%; N, 10.54%.

Preparation of the Complexes

The *bis*(3,5-dibromosalicylidene-*p*-anisidine)diaquaM(II) complexes [M = Mn(II), Co(II), Ni(II), Cu(II), and Cd(II)] were synthesized by a published procedure.[7] The preparation of mixed-ligand complexes of *bis*(3,5-dibromosalicylidene-*p*-anisidine)acphenNi(II) or *bis*(3,5-dibromosalicylidene-*p*-anisidine)benenNi(II) was carried out by refluxing a solution of 2 mmol of *bis*(3,5-dibromosalicylidene-*p*-anisidine)diaquaNi(II) (0.37 g) in 100 mL dimethylformamide with a solution of 2 mmol of 2,2′-bipyridylamine (0.34 g) or *bis*-(benzylidene)ethylenediamine (0.47 g) or *bis*(acetophenone) ethylenediamine (0.26 g) for an hour. The solution was then concentrated to 15 mL by evaporation, scratched, and cooled in a refrigerator overnight. The obtained crystals were collected and recrystallized from chloroform and, finally, dried in air. The complexes of Mn(II), Co(II), Cu(II), and Cd(II) were prepared similarly.

RESULTS AND DISCUSSION

The percentage yields, colors, melting points, analytical and magnetic moment data of the complexes are compiled in Table 1.

The compounds in this work were synthesized following the general procedures outline in the following equations.

$$\mathrm{MCl_2 \cdot \mathit{n}H_2O + 2HSB \xrightarrow{H_2O} [M(SB)_2(H_2O)_2] + 2HCl + (\mathit{n}-2)H_2O}$$

$$\mathrm{[M(SB)_2(H_2O)_2] + bipy\text{-}amine \xrightarrow[1\ hr]{EtOH,\ Reflux} [M(SB)_2bipy\text{-}amine] + 2H_2O}$$

$$\mathrm{[M(SB)_2(H_2O)_2] + benen \xrightarrow[1\ hr]{EtOH,\ Reflux} [M(SB)_2benen] + 2H_2O}$$

$$\mathrm{[M(SB)_2(H_2O)_2] + acphen \xrightarrow[1\ hr]{EtOH,\ Reflux} [M(SB)_2acphen] + 2H_2O}$$

The magnetic moment values of the Cu(II) complexes are 1.74, 1.79, and 1.71 B.M. and thus very close to the spin-only value (1.73 B.M.) expected for one unpaired electron which is consistent with an octahedral geometry.[8] The magnetic moment values of the Ni(II) complexes are 2.74, 2.98, and 2.74 B.M. and are indicating that these complexes are also octahedral.[9] The magnetic moment values of the Co(II) complexes are 4.00, 4.30, and 4.05 B.M. and are in good agreement with a high-spin octahedral geometry.[10] The magnetic moment values of the Mn(II) complexes are 6.00, 6.08, and 6.00 B.M., respectively, but are within the limits of spin-free values for five unpaired

Table 1. Analytical data of the complexes.[a]

Complexes empiral formula	Formula weight	Color	% Found (calculated) C	H	N	Metal	M.p. (°C)	μ_{eff} (B.M.)	Yield (%)
[Mn(SB)$_2$ bipy-amine]	993.54	Yellowish brown	45.89	2.90	7.04	5.52	280	6.00	45.5
($C_{38}H_{29}Br_4MnN_5O_4$)			(45.94)	(2.84)	(7.02)	(5.49)			
[Co(SB)$_2$ bipy-amine]	997.54	Light brown	45.71	2.90	7.01	5.90	290	4.0	35.8
($C_{38}H_{29}Br_4CoN_5O_4$)			(45.75)	(2.92)	(6.99)	(5.95)			
[Ni(SB)$_2$ bipy-amine]	997.31	Green	45.72	2.90	7.01	6.08	290	2.74	57.1
($C_{38}H_{29}Br_4N_5NiO_4$)			(45.80)	(2.95)	(7.00)	(6.10)			
[Cu(SB)$_2$ bipy-amine]	1002.45	Greenish brown	45.50	2.89	6.98	7.32	285	1.74	38.2
($C_{38}H_{29}Br_4CuN_5O_4$)			(45.45)	(2.90)	(6.90)	(7.38)			
[Cd(SB)$_2$ bipy-amine]	1051.1	Yellow	43.38	2.75	7.04	12.27	285	—	30.0
($C_{38}H_{29}Br_4CdN_5O_4$)			(43.48)	(2.80)	(7.02)	(12.30)			
[Mn(SB)$_2$ benen]	958.54	Yellowish brown	49.88	3.48	5.29	5.73	280	6.0	69.7
($C_{44}H_{36}Br_4MnN_4O_4$)			(49.90)	(3.50)	(5.35)	(5.80)			
[Co(SB)$_2$ benen]	1067.15	Brown	49.69	3.48	5.27	5.54	290	4.3	68.0
($C_{44}H_{36}Br_4CoN_4O_4$)			(49.75)	(3.45)	(5.30)	(5.59)			

(*continued*)

Table 1. Continued.

Complexes empiral formula	Formula weight	Color	% Found (calculated) C	H	N	Metal	M.p. (°C)	μ_{eff} (B.M.)	Yield (%)
[Ni(SB)$_2$ benen] ($C_{44}H_{36}Br_4N_4NiO_4$)	1062.31	Yellowish green	49.77 (49.79)	3.38 (3.45)	5.27 (5.30)	5.52 (5.58)	290	2.98	65.1
[Cu(SB)$_2$ benen] ($C_{44}H_{36}Br_4CuN_4O_4$)	1062.54	Greenish brown	49.74 (49.75)	3.37 (3.38)	5.24 (5.28)	6.33 (6.40)	290	1.79	70.4
[Cd(SB)$_2$ benen] ($C_{44}H_{36}Br_4CdN_4O_4$)	1116	Yellow	47.31 (47.38)	3.26 (3.30)	5.01 (5.00)	10.07 (10.05)	280	—	70.0
[Mn(SB)$_2$ acphen] ($C_{46}H_{40}Br_4MnN_4O_4$)	1086.54	Yellowish brown	50.68 (50.70)	3.68 (3.70)	5.15 (5.20)	5.05 (5.00)	288	6.08	68.1
[Co(SB)$_2$ acphen] ($C_{46}H_{40}Br_4CoN_4O_4$)	1090.54	Brown	50.61 (50.65)	3.66 (3.70)	5.13 (5.20)	5.40 (5.45)	280	4.05	60.7
[Ni(SB)$_2$ acphen] ($C_{46}H_{40}Br_4N_4NiO_4$)	1090.51	Yellowish green	50.62 (50.70)	3.66 (3.60)	5.13 (5.17)	5.38 (5.40)	280	2.84	70.8
[Cu(SB)$_2$ acphen] ($C_{46}H_{40}Br_4CuN_4O_4$)	1095.15	Greenish brown	50.40 (50.35)	3.65 (3.69)	5.11 (5.15)	5.80 (5.85)	280	1.71	78.0
[Cd(SB)$_2$ acphen] ($C_{46}H_{40}Br_4CdN_4O_4$)	1144	Yellow	48.25 (48.20)	3.49 (3.45)	4.89 (5.95)	9.82 (9.85)	288	—	58.6

[a] HSB = 3,5-Dibromosalicylidene-*p*-anisidine.

electrons, indicating that the complexes are high-spin six-coordinated.[11] The magnetic moment determinations show that the Cd(II) complexes are diamagnetic and presumably octahedral.

The electronic spectra provide the most detailed information about the electronic structure. The electronic spectra of the metal chelates have been studied in the solid state between 350 and 1700 nm. The electronic spectra of the nickel(II) complexes exhibit three bands at ~11,000, ~19,000 and ~ 23,000 cm^{-1}, which may be due to the transitions[12] ${}^3A_{2g}(\mathrm{F}) \rightarrow {}^3T_{2g}(\mathrm{F})$ (ν_1), ${}^3A_{2g}(\mathrm{F}) \rightarrow {}^3T_{1g}(\mathrm{F})$ (ν_2), and ${}^3A_{2g}(\mathrm{F}) \rightarrow {}^3T_{1g}$ (P) (ν_3), respectively. The three bands for the cobalt(II) complexes have been assigned to ${}^4T_{1g}(\mathrm{F}) \rightarrow {}^4T_{2g}$ (ν_1), ${}^4T_{1g}(\mathrm{F}) \rightarrow {}^4A_{2g}$ (ν_2), and ${}^4T_{1g}(\mathrm{F}) \rightarrow {}^4T_{1g}(\mathrm{P})$ (ν_3) at ~9100, ~18,000, and ~19,500 cm^{-1}, respectively.[13] The electronic spectra of the copper(II) complexes exhibit one band at ~15,000 cm^{-1}, which may be attributed to the transition ${}^2E_g \rightarrow {}^2T_{2g}(\mathrm{F})$. These observations suggest an octahedral geometry for these complexes.[14] The electronic spectra of the manganese(II) complexes exhibit three weak absorption bands at ~15,000, ~19,000, and ~25,000 cm^{-1}, which have been assigned to the transitions ${}^6A_{1g} \rightarrow {}^4T_{1g}$ (^{4}G) (ν_1), ${}^6A_{1g} \rightarrow {}^4T_{2g}$ (^{4}G) (ν_2), and ${}^6A_{1g} \rightarrow {}^4A_{1g}$, ${}^4E_g({}^4\mathrm{G})$ (ν_3), respectively, in an octahedral field of Mn(II) ion.[15] The value of D_q, B, β, β^0, and ratio ν_2/ν_1 for the cobalt(II) and nickel(II) complexes are presented in Table 2.

The characteristic vibrational frequencies have been identified by comparing the spectra of the complexes with those of their percursors and literature values of absorption frequencies of similar compounds.[16] The Schiff base (HSB) exhibits a medium-intense band at ~2700 cm^{-1} due to intramolecularly hydrogen bonded ν(OH).[17] The absence of this band in the complexes indicates the deprotonation of the phenolic groups and coordination of the oxygen atoms to the metal ion. In the free ligand (HSB) a strong band at 1525–1540 cm^{-1} due to ν(C–O) (phenolic) shifts to higher energy by 5–10 cm^{-1} in the complexes indicating the coordination of the phenolic oxygen atom to the metal ion. This shifts to higher energy as expected due to the maintenance of ring currents arising from the electron delocalization in the chelate ring. The Schiff base (HSB) shows the C=N stretching band at 1630 cm^{-1}. This band shifts to lower energy by 10–15 cm^{-1} in the complexes indicating coordination through the azomethine nitrogen.[17] The ν(C=N) frequency of 2,2′-bipyridylamine occurs at 1585 cm^{-1}. This band shifts to higher energy by 35–45 cm^{-1} in the mixed-ligand complexes indicating the bidentate (NN, pyridyl nitrogens) coordination of this heterocyclic ligand.[18] The Schiff bases benen and acphen show the C=N stretching band at ~1640 cm^{-1}. This band shifts to lower energy by 20–30 cm^{-1} in the mixed-ligand complexes indicating coordination through the azomethine nitrogen. The frequencies in the range 450–460 and 410–420 cm^{-1} are attributed to ν(M–N)[19] and ν(M–O),[20] respectively.

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

	Observed band (cm^{-1})							
Complexes	ν_1	ν_2	ν_3	ν_2/ν_1	B	β	β^0	D_q
[Ni(SB)$_2$bipy-amine]	10,305	18,180	23,250	1.76	700.45	0.680	32.00	1,305.00
[Co(SB)$_2$bipy-amine]	9,300	18,100	19,030	1.95	724.56	0.748	25.38	1,042.55
[Ni(SB)$_2$benen]	10,525	16,950	25,000	1.61	691.66	0.671	32.84	1,525.00
[Co(SB)$_2$benen]	9,100	18,000	19,000	2.00	738.59	0.760	23.93	1,021.01
[Ni(SB)$_2$acphen]	10,400	18,181	23,350	1.76	700.45	0.680	32.00	1,400.00
[Co(SB)$_2$acphen]	9,200	18,000	19,000	1.94	728.01	0.749	25.10	1,032.02

[a] HSB = 3,5-Dibromosalicylidene-*p*-anisidine.

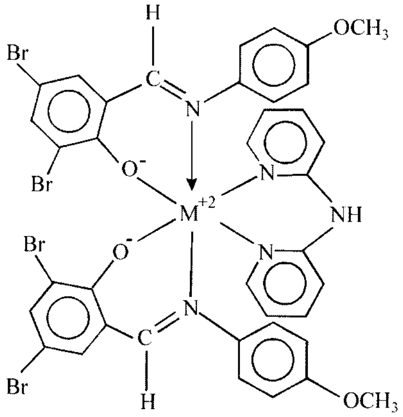


Figure 2. Suggested structure of the complexes [M(SB)$_2$bipy-amine].

The suggested structures of the complexes are shown in Figs. 2 and 3.

Antimicrobial Activity

The antimicrobial activity of the ligands, metal salts, and the corresponding complexes were assayed against *Salmonella typhi* (bacteria) and *Saccharomyces cerevisiae* (yeast) using the "Agar Diffusion Method."[21]

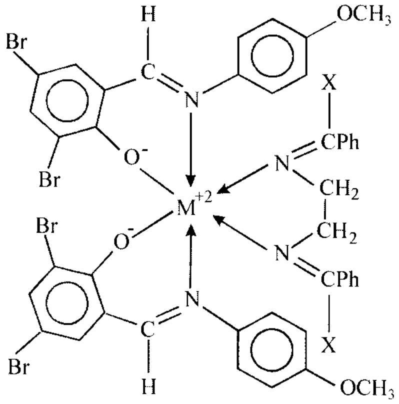


Figure 3. Suggested structure of the complexes [M(SB)$_2$benen]; (X = H) or [M(SB)$_2$acphen]; (X = CH_3).

The antifungal activity of the complexes were assayed against *Fusarium oxysporum* and *Lasiodiplodia theobromae* (fungi) using the "Potato Dextrose Agar Method."[22] All the compounds have significant antibacterial activity at $5 \times 10^{-4}\,\mathrm{g\,mL^{-1}}$ against bacteria, yeast, and fungi. The results were compared against dimethyl formamide as the control and are expressed as percentage vs. control. The percentage inhibition of various compounds are recorded in Table 3. 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 control, ligands, and metal salts. The results

Table 3. Antimicrobial activities of the complexes.[a]

	% Inhibition			
			Fungi	
Compound	Bacteria (*S. typhi*)	Yeast (*S. cerevisiae*)	*Lasiodiplodia theobromae*	*Fusarium oxysporum*
$MnCl_2 \cdot 4H_2O$	21.11	21.05	13.30	15.30
$CoCl_2 \cdot 6H_2O$	16.66	10.52	06.60	15.30
$NiCl_2 \cdot 6H_2O$	11.11	05.26	13.30	13.10
$CuCl_2 \cdot 2H_2O$	22.22	15.78	20.00	07.70
$CdCl_2 \cdot H_2O$	16.66	05.26	06.60	13.00
HSB	33.33	31.57	20.00	27.00
Bipy-amine	11.11	05.26	06.60	07.70
acphen	26.00	10.50	26.00	23.00
benen	37.00	05.00	20.00	37.00
$[Mn(SB)_2$bipy-amine]	50.00	42.10	60.00	53.00
$[Co(SB)_2$bipy-amine]	50.00	47.36	46.60	53.00
$[Ni(SB)_2$bipy-amine]	55.00	52.63	33.30	46.00
$[Cu(SB)_2$bipy-amine]	55.00	42.10	60.00	46.00
$[Cd(SB)_2$bipy-amine]	55.00	52.63	40.00	51.03
$[Mn(SB)_2$benen]	50.00	47.36	46.60	61.30
$[Co(SB)_2$benen]	66.66	47.36	40.00	61.00
$[Ni(SB)_2$benen]	61.11	52.63	33.30	53.00
$[Cu(SB)_2$benen]	55.00	42.10	33.30	46.00
$[Cd(SB)_2$benen]	50.00	42.00	60.00	53.00
$[Mn(SB)_2$acphen]	66.66	50.00	46.60	61.30
$[Co(SB)_2$acphen]	50.00	47.36	33.30	61.00
$[Ni(SB)_2$acphen]	55.00	36.84	53.60	53.80
$[Cu(SB)_2$acphen]	66.66	47.36	60.60	46.00
$[Cd(SB)_2$acphen]	44.48	42.10	40.00	61.30

[a] HSB = 3,5-Dibromosalicylidene-*p*-anisidine.

suggest that the change in the structure due to coordination in the mixed-ligand complexes decreases the growth of microorganisms. Hence, complexation increases the antimicrobial activity. The complexes show greater activity against *S. typhi* (bacteria) than *S. cereuisae* (yeast). The patterns of activity are complex and no clear trend can be ascertained.

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