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Synthesis, Characterization, Thermal and Ion-Exchange Studies of Benzophenone Ethane Diol Resin and its Polychelates

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The resin (ligand) poly[(2,4-dihydroxy benzophenone) ethylene] H(DHB-ED) forms 1:2 metal:ligand chelates with Cu(II), Ni(II), Co(II), Mn(II) and Zn(II). The resin (ligand) and polychelates were characterized by the elemental analyses, IR-spectroscopy, NMR and Number Average Molecular Weight (\bar{M}_n) was determined by Vapor Pressure Osmometry (VPO). The stereochemistry, in the case of the Cu(II) polychelate, is square-planar, tetrahedral for Zn(II) and octahedral for Ni(II), Mn(II) and Co(II). The stereochemistry in each chelate is proposed on the basis of magnetic susceptibilities. IR spectra shows that the bidentate ligand coordinates through the oxygen atom of the carbonyl group and oxygen atom of the phenolic group with replacement of hydrogen by metal ions. All the chelates are paramagnetic in nature except Zn(II) chelate which is diamagnetic. Thermogravimetric and ion-exchange studies at different electrolyte concentration, pH and rate have been carried out for selected metal ions Cu^{2+} and Ni^{2+} . It is found that, Cu^{2+} has higher binding capacity than Ni^{2+} .

Keywords: benzophenone ethane diol resin; polychelates; polycondensation; thermal study; ion exchange

1 Introduction

Chelating polymers have attracted more interest, due to their applications in waste water treatment, metal recovery from diluted solution, as protective coating on metal surfaces or as a priming layer, coating on paper, fiber and fabrics, selective binding of enzymes (1–7). The study of polymeric ligands and their metal complexes are very useful in metal separation, as a catalyst and in bio-inorganic chemistry (8). In a polymeric ligand or polychelates, the function of a ligand is often only a small portion of three dimensional polymer support, hence it is subject to a number of structural variables compared to a low molecular weight ligand. This indicates a definite dependence on the complexing abilities of the insoluble polymeric ligands and physicochemical properties of the derived polychelates (9). Recently, several coordination polymers have been prepared from aromatic and aliphatic polymers containing pendant functional groups which act as a chelating group in binding polyvalent metal ions (10). The reaction of trimethylolpropane, triacrylates with benzophenone as an initiator (11) in a UV acrylic coating shows excellent curing speed. The platinum(II)-benzophenoimine chelates and crystal structure of trans-(N,N)-(benzophenoimine)-chloro-[2-(1-imino-1-phenyl

methyl) phenylido] platinum (III) acetone have been studied (12). The synthesis of novel soluble polyamide based on alkyldiamino-benzophenone and their applications for the alignment of films have been studied (13). Transition metal chelates have been found to stabilize free radical decomposition of polymer chains (14). Metal chelates, particularly of nickel, were reported as effective in stabilizing polyolefins by quenching excited state in polymers, thus reducing the likelihood of free radical formation, discoloration and embrittlement. The efficiency of quenching is related to the spatial configuration of the ligands in the complexes (15). Metal complexes involving Ni(II), Cu(II), and Fe(III) have been reported as having light stabilizing efficiency based on ultraviolet screening and free radical scavenging (16). In the present work, the preparation and characterization of Cu(II), Ni(II), Co(II), Zn(II) and Mn(II) polychelates with poly[(2,4-dihydroxy benzophenone)ethylene] have been carried out. It is found that the polychelates are thermally less stable than the resin (ligand). Ion-Exchange property of the synthesized resin towards Cu^{2+} and Ni^{2+} has been studied at different electrolyte concentrations, pH and rate.

2 Experimental

2.1 Synthesis of Resin

All chemicals used were of Analar grade. 2,4-dihydroxy benzophenone (DHB) (6.42 gm, 0.03 mole) and ethanediol (ED)

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(5.76 ml, 0.03 mole) were mixed and refluxed at 115°C for 12 h in the presence of 40 gms polyphosphoric acid (PPA) as catalyst. Afterwards, the reaction mixture was poured into crushed ice and left overnight. The separated solid product was filtered, washed with acetone, and dried at 60°C for 24 h. Yield: 4.2 gms (85%). The structure of 2,4-dihydroxy benzophenone and poly[(2,4-dihydroxy benzophenone)ethylene] as shown in Figure 1.

2.2 Preparation of Polychelates

All polychelates were synthesized by using the following general method. Metal acetates of (BDH), Analar grade, were used in the preparation. The ligand (2.14 gms, 0.01 mole) was dissolved in DMSO (50 mL). The metal acetate (0.005 mole) was also dissolved in DMSO (25 mL). The hot and clear solution of the metal acetate was added with constant stirring to the hot and clear solution of the ligand. A dark-colored product separated out immediately. The suspension was digested on a water bath at 90°C for 2 h, and then filtered. The solid was washed with hot DMSO to remove unreacted metal acetate. Finally, the product was washed with acetone and dried at 60°C for 24 h. The yield of each polychelate was between 82 and 90%.

2.3 Analytical Procedures

Carbon and hydrogen were analyzed with a Coleman C, H, N analyzer. The metal content was determined by titration with standard EDTA after decomposing the polychelates with a mixture of concentrated hydrochloric acid, sulfuric acid and perchloric acid in a 5:2:3 mL ratio, respectively. Magnetic susceptibilities were measured by the Gouy method at room temperature. The IR spectra of the samples in KBr pellets were recorded on a model 938 Perkin-Elmer spectrophotometer. Thermal measurements were performed using a DuPont thermal analyzer at 10°C min⁻¹ heating rate. ¹H-NMR spectra were determined in CDCl₃ with a FT-NMR spectrophotometer using TMS as an internal reference. The Number Average Molecular Weight (\bar{M}_n) of polymeric

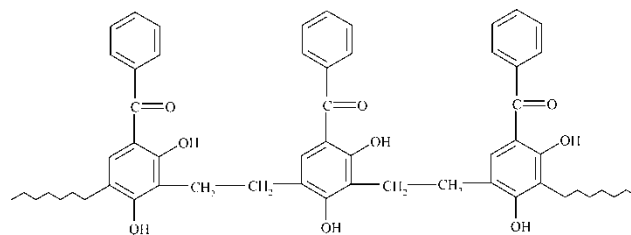


Fig. 2. Structure of the polymer.

ligand (resin) sample was measured with a Knauer Germany (VPO) using DMF as solvent at 90°C and polystyrene (PS) as a calibrant.

3 Results and Discussion

3.1 Infrared Spectra

On the basis of the reactivity of the 3 and 5 position in 2,4-dihydroxy-benzophenone (DHB), the resin has the general structure as shown in Figure 2. Accordingly, each of the phenyl rings (except the end groups) of the chain would be 1,2,3,4,5-penta substituted containing only one hydrogen atom on the benzene ring.

The end group will be of the type A or B as shown in Figure 3, although the likelihood for A is higher because *para* substituted relative to phenolic O-H is preferred to *ortho* substituted in such types of polymeric reactions.

The other possible type of end group B in polymer chain contains two adjacent H-atoms. The bands around 825 and 790 cm⁻¹ indicate 1,2,3,4 -tetra substituted phenyl rings (17). Thus, the phenyl rings in the dihydroxy monomer unit of the chain and group A of the polymer chain contain only one isolated H-atom and 1,2,4,5 tetra substituted phenyl rings. The strong out of plane C-H bending band around 900 cm⁻¹ is due to penta-substituted and 1,2,4,5 tetra substituted phenyl rings (18). These results strongly suggest that both possible types of end groups (Figure 3) may be present. The structure of the title ligand is as shown in Figure 4.

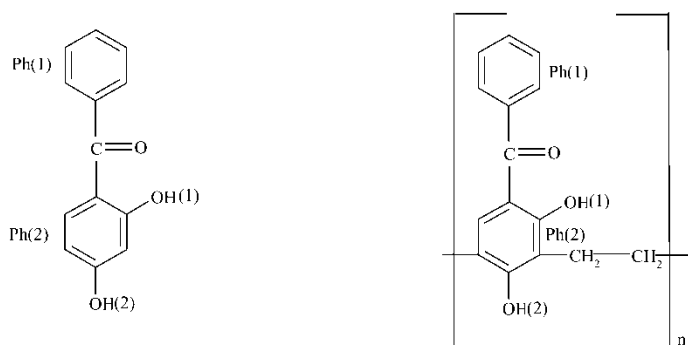


Fig. 1. Structure of 2,4-dihydroxy benzophenone and poly[(2,4-dihydroxy benzophenone)ethylene].

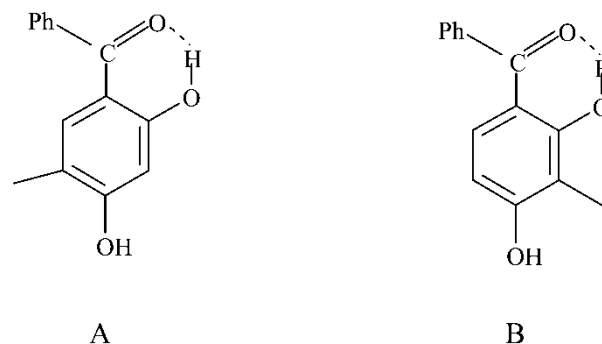


Fig. 3. Possible end group of the polymeric chain.

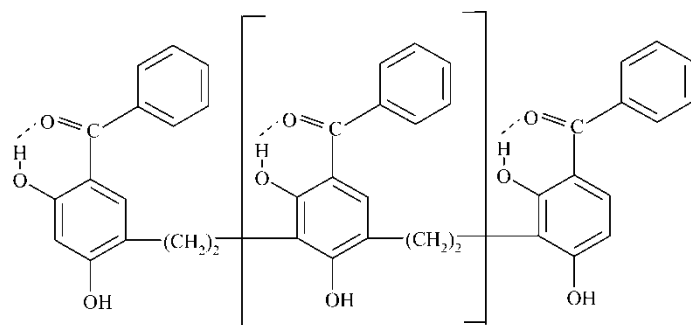


Fig. 4. Structure proposed for the ligand.

All of the polychelates are reddish brown, amorphous powders which are insoluble in water and common organic solvents. The analytical data of the ligand and its polychelates are presented in Table 1. These results suggest that the polychelates possess a 1:2 metal ligand ratio. The important IR frequencies of the polymeric ligand and its polychelates are presented in Table 2. In the polychelates, the band observed around 455 and 560 cm^{-1} indicates the M-O bond and suggests that the phenolic and carbonyl groups are involved in bond formation with the metal ion. The C=O stretching frequency in the resin is observed around 1630 cm^{-1} and appears at a lower frequency by 20 to 30 cm^{-1} in all of the polychelates, which suggests C=O \rightarrow M coordination (19). The broad band at 3240–3620 cm^{-1} is due to the O-H stretching vibration. Bands around 825, 1170, 1260, and 1470 cm^{-1} correspond to an isolated hydrogen atom on the benzene ring (20), C-O vibration, O-H vibration and C=C skeletal vibration, respectively as shown in Figure 5.

The above study suggests that chelation take place between two chelating groups in the same chain as shown in Figure 6.

4 Magnetic Measurements

To obtain information about the geometry of the polychelates, magnetic measurements were carried out. The magnetic moments and stoichiometry of the polychelates suggests octahedral geometry for Ni(II), Co(II) and Mn(II), a square planar for Cu(II) and tetrahedral for Zn(II). The magnetic susceptibility values of the polychelates are given in Table 1.

The magnetic moments of the copper (II) polychelate is 1.87 B.M. which is very close to the spin-only value for one unpaired electron, suggesting that the orbital contribution is quenched by the crystal field (21). The Ni(II) polychelate is paramagnetic because it exhibits a magnetic moment value of 3.16 B.M., consistent with distorted or regular octahedral stereochemistry (22). The Co(II) polychelate exhibits a magnetic moment of 4.32 B.M. which is in agreement with Co(II) in an octahedral geometry. The Zn(II) polychelate is diamagnetic, due to a $3d^{10}$ configuration. The magnetic moment of the Mn(II) polychelate is 5.35 B.M. the low magnetic moment of the Mn relative to the spin-only value (5.92 B.M.) may be due to air oxidation of manganese during synthesis (23).

4.1 ^1H NMR Spectra

The ^1H -NMR spectrum of H(DHB) in Table 3 shows that signals at δ 12.70 and 11.60 ppm are due to presence of -OH(1) proton *ortho* to (Ar-C=O) and -OH(2) proton *para* to (Ar-C=O) groups, respectively. Aromatic ring protons appear as two multi-plates between δ 7.50–8.30 and 6.35–6.75 ppm (24). H(DHB-ED) shows two signals at δ 12.50 and δ 11.40 ppm due to presence of -OH(1) protons *ortho* to (Ar-C=O) and -OH(2) proton *para* to (Ar-C=O) groups, respectively. An aromatic ring proton appears as two multiplates

Table 1. Analytical and magnetic moment data for the polymeric ligand^a and the chelates

Compound	M.W. of repeating unit	Yield (%)	Elemental analysis found (Calcd) %			D.P. ^b °C	μ_{eff} (B.M.)
			M	C	H		
[L] _n	240	90	—	74.88 (75.00)	5.02 (5.00)	>400	—
[C ₃₀ H ₂₂ CuO ₆] _n	542	88	11.71 (11.62)	66.50 (66.42)	4.01 (4.05)	>300	1.87
[NiL ₂ (H ₂ O) ₂] _n	573	87	10.38 (10.29)	62.70 (62.82)	4.60 (4.53)	>350	3.16
[C ₃₀ H ₂₆ NiO ₈] _n	573	82	10.21 (10.29)	62.74 (62.82)	4.55 (4.54)	>350	4.32
[MnL ₂ (H ₂ O) ₂] _n	569	92	9.62 (9.66)	63.21 (63.26)	4.68 (4.56)	>350	5.35
[C ₃₀ H ₂₆ MnO ₈] _n	543	90	12.03 (11.97)	66.18 (66.29)	4.11 (4.05)	>400	Diamagnetic

^aL = poly[(2,4-dihydroxy benzophenone) ethylene].

^bDecomposition point.

Table 2. Infrared spectra of the polymeric ligand and its polychelates (cm^{-1})^a

Compound	ν (-O-H)	ν (Aromatic CH ₂)	-CH ₂ Bridge	Intramolecular -H- bond	Chelated-C=O	Pentasubstituted -Ph ring	Tetrasubstituted -Ph ring	Two adjacent H on Ph ring	ν (M-O)
[L]	3300m 3600b	2970w	2930w	2725m	1625s	900m	860m	825m	—
[CuL ₂] _n	3300m 3600b	2975w	2920w	—	1630s	890m	860m	825m	457w
∞ [NiL ₂ (H ₂ O) ₂] _n	3300m 3600b	2970w	2920w	—	1625m	900m	860m	825m	475w
[CoL ₂ (H ₂ O) ₂] _n	3300m 3600b	2965w	2925w	—	1620s	890m	860m	825m	475w
[MnL ₂ (H ₂ O) ₂] _n	3300m 3600b	2970w	2920w	—	1625s	890m	860m	825m	475w
[ZnL ₂ (H ₂ O) ₂] _n	3300m 3600b	2975w	2920w	—	1630s	900m	860m	825m	475w

^ab = broad, m = medium, s = strong, w = weak; L = poly[(2,4-dihydroxy benzophenone)ethylene].

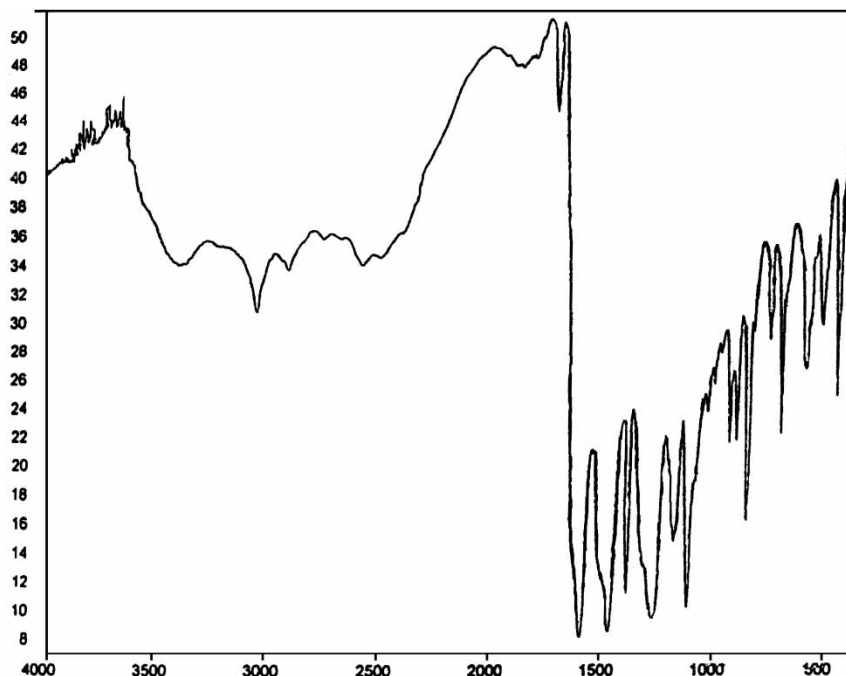


Fig. 5. IR Spectra of DHB-ED DHB-ED = [(2,4 Dihydroxy Benzophenone) Ethane Diol].

between δ 7.50–7.80 and 6.45–6.90ppm. One additional signal at δ 2.90 ppm appears, due to the presence of (Ar-C=O-Ar-CH₂-CH₂-) protons as shown in Figure 7.

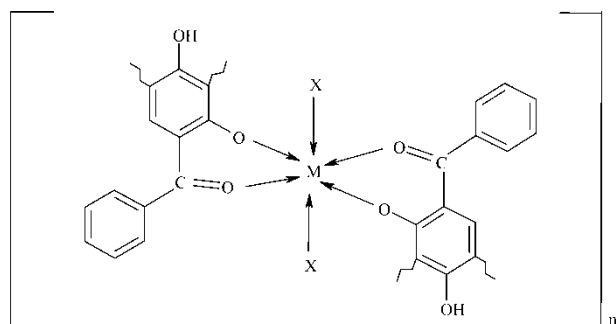
4.2 Vapor Pressure Osmometry

The Number Average Molecular Weight (\bar{M}_n) of the polymeric ligand (resin) samples were estimated by Vapor Pressure Osmometry (25) shown in Table 4. The dilute solutions of polymer samples were prepared to determine \bar{M}_n . Four concentrations 2.21, 4.42, 6.63, and 8.84 g · kg⁻¹ were prepared in DMF. A VPO experiment was carried out for

each concentration, and the corresponding bridge output reading in millivolts was noted. The plot of millivolts Vs concentration was drawn. With the help of the slope and the VPO constant K, the \bar{M}_n value of the polymer was calculated and $\bar{M}_n=1059$ g mole⁻¹ was found.

4.3 Thermogravimetric Analyses

Thermogravimetric analyses (TGA) data of the resin and polychelates are presented in Table 5. These data revealed that the rate of decomposition of the polychelate is higher than that of the parent resin, suggests that there may be strong intramolecular hydrogen bonding in the polymer. The absence of such hydrogen bonding in polychelate favors the reduction in thermal stability of polychelates compared to the parent resin (26). It seems that metal ions accelerate the decomposition of the polychelates. For the copper chelate, the decomposition occurred at 250°C, which may be due to the oxidation of polymer by the catalytic action of Cu(II) ion. The thermal stability of the ligand and metal chelates is in the order: ligand > Zn(II) ≥ Ni(II) > Mn(II) > Co(II) > Cu(II). The presence of water molecules considered as a water of co-ordination. According to Nikolaev et al. (27) water eliminated above 150°C may be due to coordination to the metal ion. The nature of the water molecules observed in the complexes is water of coordination (28–30), which is supported by cumulative % weight loss and thermal data presented in Table 6.



X -- H₂O nil for M – Cu(II)

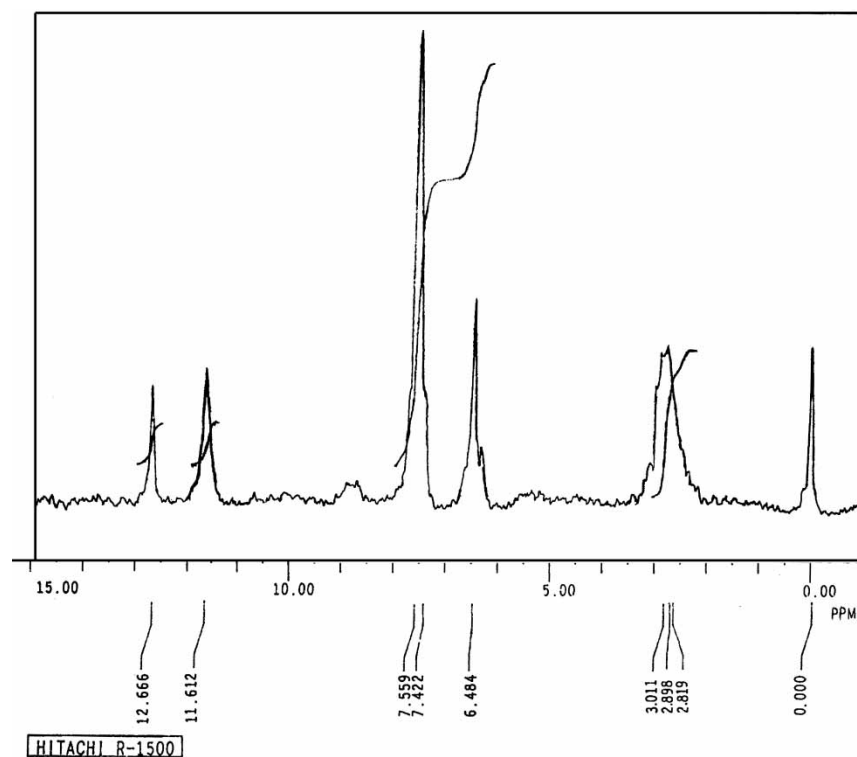
X == H₂O for M = Ni(II), Mn(II), Co(II), and Zn(II)

Fig. 6. Proposed structure of the polymeric chelate.

Table 3. $^1\text{H-NMR}$ data of starting material and polymeric ligand (δ , ppm)

Compound	Ph (1)	Ph (2)	-OH (1)	-OH (2)	Ar-CH ₂ -CH ₂ -
H[DHB]	6.35–6.75	7.55–8.30	12.70	11.60	—
H[DHB-ED]	6.45–6.90	7.50–7.80	12.50	11.40	2.70

H[DHB] = 2,4-dihydroxy benzophenone; H[DHB-ED] = poly[2,4-dihydroxy benzophenone) ethylene].

**Fig. 7.** $^1\text{H-NMR}$ Spectra of DHB-ED in CDCl_3 DHB-ED = [(2,4 dihydroxy benzophenone) ethane diol].

4.4 Ion-Exchange Study

The purified resin sample DHB-ED was finally powdered to pass a 300 mesh screen and used in all experiments for ion-exchange study. The batch equilibration method was used. The details of the procedure for selectivity of the different metal ions by the resin, is similar to that reported earlier (31).

Table 4. Molecular weight determination of polymeric ligand by vapor pressure osmometry method

Ligand (resin)	Concentration $\text{g} \cdot \text{kg}^{-1}$	Milli volts	Slope of plot	$\bar{M}_n = K/\text{Slope}$ $\text{g} \cdot \text{mol}^{-1}$
H[DHBED]	2.21	24	10.85	1059
	4.42	48		
	6.63	72		
	8.84	101		

Polystyrene $K = 1.15 \times 10^4$; H[DHB-ED] = poly[(2,4-dihydroxy benzophenone) ethylene].

4.5 Influence of Various Electrolyte on Metal Uptake

The influence of Cl^- , NO_3^- and SO_4^{2-} at various concentrations on the equilibrium state of the metal-resin interaction has been studied. The results are presented in the (Table 7). It reveals that, the amount of Ni^{2+} and Cu^{2+} taken up by the resin is increasing with an increasing in concentration of NO_3^- and Cl^- and decreases with an increase in concentration of SO_4^{2-} . This may be due to the sulphate form rather strong chelates with Ni^{2+} and Cu^{2+} , while nitrate and chloride might form weak chelates. Therefore, the influence of NO_3^- and Cl^- is less, on the position of Ni^{2+} and Cu^{2+} chelates at equilibrium state that does sulphur.

4.6 Rate of Metal Uptake Capacity at Various pH

The results of metal uptake presented in Table 8 show the dependence of the rate of metal ion uptake on the nature of metal ion. The study was restricted up to a maximum

Table 5. Cumulative % weight loss data and thermal data of the polymeric ligand and its polychelates

Compound	% Weight loss at temperatures (°C)										
	100	150	200	250	300	350	400	450	500	550	600
[L] _n	0.50	0.60	0.70	0.85	5.80	12.25	22.00	32.60	45.30	62.00	72.75
[CuL ₂] _n	1.90	2.00	2.10	2.70	27.55	40.15	54.85	58.80	62.30	72.80	78.05
[NiL ₂ (H ₂ O) ₂] _n	1.25	1.50	1.75	2.10	14.15	28.80	37.50	52.70	58.90	67.50	76.75
[CoL ₂ (H ₂ O) ₂] _n	1.70	1.90	2.10	2.45	15.40	26.45	39.50	51.60	59.80	68.70	78.90
[MnL ₂ (H ₂ O) ₂] _n	1.30	1.45	1.65	2.10	14.90	24.40	36.40	48.70	55.70	68.20	76.70
[ZnL ₂ (H ₂ O) ₂] _n	1.10	1.35	1.60	2.15	10.85	18.30	23.80	46.40	52.80	64.70	68.20

Table 6. Water content at 25°C and cumulative weight loss data of the polymeric ligand and its polychelates at 100, 150, and 200°C

Compound	H ₂ O at 25°C		Found					
			100°C		150°C		200°C	
	g	%	G	%	g	%	g	%
[L]	—	—	—	—	—	—	—	—
[CuL ₂] _n	—	—	—	—	—	—	—	—
[NiL ₂ (H ₂ O) ₂] _n	36.00	6.28	7.16	1.25	8.59	1.50	10.02	1.75
[CoL ₂ (H ₂ O) ₂] _n	36.00	6.28	9.74	1.70	10.88	1.90	12.03	2.10
[MnL ₂ (H ₂ O) ₂] _n	36.00	6.32	7.39	1.30	8.25	1.45	9.38	1.65
[ZnL ₂ (H ₂ O) ₂] _n	36.00	6.62	5.97	1.10	7.33	1.35	8.68	1.60

^aL = poly[(2,4-dihydroxy benzophenone) ethylene]; a = polymeric ligand.

pH = 6, due to hydrolysis of metal ion at higher pH. The formation of metal hydroxide interferes with the ion-exchange process. It is found that the relative amount of metal adsorbed by the resin increases with increasing pH of the medium.

Table 7. Evaluation of different electrolytes in uptake of metal ions

Metal ions	Electrolyte (Mol · lit ⁻¹)	Metal ion uptake (meq · g ⁻¹) in the presence of electrolyte		
		NaCl	NaNO ₃	Na ₂ SO ₄
Cu ²⁺	4.5	0.05	0.40	0.31
	4.5	0.10	0.44	0.35
	4.5	0.50	0.68	0.55
Ni ²⁺	4.5	1.00	0.73	0.64
	4.5	0.05	0.29	0.37
	4.5	0.10	0.33	0.42
	4.5	0.50	0.66	0.64
	4.5	1.00	0.65	0.69

Resin, DHB-ED; M(NO₃)₂ = 0.1 mol/lit; volume of metal ion solution, 2 mL; volume of electrolyte solution, 44 mL; time, 24 h.; Temperature = 30°C.

4.7 Rate of Metal Uptake Capacity as a Function of Time

The result of the rate of metal uptake by the resin as a function of time is expressed in terms of % of metal ions adsorbed by the resin after regular time intervals with respect to 100% adsorption at the state of equilibrium. In the results presented in Table 9, the term “rate” refers to the speed of change in the concentration of metal ion in the aqueous solution, which is in contact with the resin. The result shows that the time taken for the uptake of different metal ions at given stage depends on the nature of the metal and chelating resin sample. The rate becomes slow in the first 2 h, but nearly to the equilibrium after 5 to 6 h. It is found that adsorption of Cu²⁺ is more compared to Ni²⁺.

Table 8. Effect of pH on binding capacity of resin for Cu²⁺ and Ni²⁺ metal ions

Metal ion	Metal ion uptake (m · eq/g) pH of the medium				
	3.5	4.0	4.5	5.0	5.5
Cu ²⁺	0.55	0.59	0.64	0.72	0.80
Ni ²⁺	0.52	0.56	0.58	0.76	0.82

Weight of resin, 50 mg; M(NO₃)₂ = 0.1 mol/lit, 2 mL; NaNO₃ = 0.1 mol/lit, 40 mL; Temperature = 30°C.

Table 9. Evaluation of rate of metal ions uptake

Time in hours	% Attainment at equilibrium of metal ion	
	Cu ²⁺	Ni ²⁺
1.0	40.14	26.90
2.0	48.29	31.67
3.0	58.21	43.32
4.0	69.87	52.92
5.0	81.92	67.93
6.0	89.29	76.64
7.0	93.28	86.71

Weight of resin 50 mg; M(NO₃)₂ = 0.1 mL, 2 mL; NaNO₃ = 0.1 mol/lit, 40 mL; Temperature = 30°C; Time, 24 h (equilibrium state).

5 Conclusions

From elemental analyses, IR, thermogravimetric analysis, ¹H-NMR spectra, magnetic properties and Vapor Pressure Osmometry, the proposed geometry of the polychelate is shown in Figure 6. The resin has good binding capacity for the selected metal ions (Cu²⁺ and Ni²⁺). Various conditions have been employed for the ion exchange study. It is revealed from the data obtained that the resin can be used as an ion exchanger.

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