Resin. III. Synthesis, Characterization, and Ion-Exchange Properties of a 2,2′-Dihydroxybiphenyl–Formaldehyde Copolymer Resin

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ABSTRACT: A 2,2′-dihydroxybiphenyl–formaldehyde copolymer, synthesized by the condensation of 2,2′-dihydroxybiphenyl with CH_2O in the presence of an acid catalyst, proved to be a selective chelating ion-exchange copolymer for certain metals. The chelating ion-exchange properties of this copolymer were studied for Fe(III), Cu(II), Ni(II), Zn(II), Cd(II), and Pb(II) ions. A batch equilibrium method was employed in the study of the selectivity of metal ion uptake, involving the measurements of the distribution of a given metal ion between the copolymer sample and the solution containing the metal ion. The study was carried out over a wide pH range and in media of various ionic strengths. The copolymer showed a higher selectivity for Fe(III), Cu(II), and Ni(II) ions than for Co(II), Zn(II), Cd(II), and Pb(II) ions. © 2008 Wiley Periodicals, Inc. J Appl Polym Sci 109: 508–514, 2008

Key words: FTIR; metal-polymer complexes; polyamides; resins; step-growth polymerization

INTRODUCTION

Ion-exchange resins show selectivity toward the absorption of various metal ions and possess a high absorption capacity.[1,2] These resins possessing chelating groups may be used in chromatographic separations of certain metal ions and in the recovery and removal of traces of certain metallic ions from high concentrations of other electrolytes.[1–3] Dalal and Kapadia[4,5] reported the ion-exchange properties of phenol–furfural and phenol–acetaldehyde resins. Chelation ion-exchange properties of 2,4-dinitrophenylhydrazone of a 2-hydroxyacetophenone–formaldehyde resin have been reported.[6]

An ion-exchange resin derived from semicarbazone and oximes of 2-hydroxyacetophenone-substituted benzoic acid–formaldehyde was studied by Nayak and coworkers.[7,8] An oxyethylene copolymer with phenyl and 4,4′-biphenyl structural units in the backbone has been reported in the literature.[9] Cyclohexanone–formaldehyde and acetophenone–formaldehyde resins, modified by the incorporation of phenol, biphenol, and substituted acetophenone, have been reported in the literature.[10] The effect of annealing on structural changes in a liquid-crystalline copolymer of 6-hydroxy-2-naphthanoic acid, *p*-hydroxybenzoic acid, terephthalic acid, and 4,4′-

biphenol was investigated by Yoon et al.[11] In a continuation of our earlier work on the synthesis and characterization of chelating ion exchangers,[12–14] this communication reports the detailed characterization and ion-exchange properties of a newly synthesized resin.

EXPERIMENTAL

Materials

The chemicals were all analytical reagent or chemically pure grade. Dimethylformamide (DMF) and dimethyl sulfoxide (DMSO) were used after double distillation. 2,2′-Dihydroxybiphenyl (Lancaster, England, UK; 99% pure) and a formaldehyde solution (Merck, Mumbai, India; 35%) were used.

Preparation of the 2,2′-dihydroxybiphenyl–formaldehyde (BPhF) copolymer resin

The BPhF copolymer resin was prepared by the refluxing of 2,2′-dihydroxybiphenyl (18.6 g, 0.1 mol) with formaldehyde (17.2 mL of a 35% solution, 0.2 mol) in the presence of 2*M* HCl (100 mL) as a catalyst at 120 ± 2°C in an oil bath for 5 h.[7,13] The solid product obtained was immediately removed from the flask as soon as the reaction period was over. It was washed with cold water, dried, and powdered. The powder was repeatedly washed with hot water, which was followed by methanol to remove unreacted monomers. The copolymer then was purified by dissolution in 8% NaOH and filtered. The copoly-

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mer was then reprecipitated by the dropwise addition of 1 : 1 (v/v) concentrated HCl–water with constant stirring and filtered. The process was repeated twice. The resulting polymer sample was washed with boiling water and dried *in vacuo* at room temperature. The purified copolymer resin was finely ground to pass through a 300-mesh size sieve and kept *in vacuo* over silica. The yield of the copolymer resin was found to be 87%. The purified 300-mesh sieve resin sample was used in all the experiments carried out in the ion-exchange study.

Instruments

The copolymer resin was subjected to microanalysis for C and H on a PerkinElmer (Waltham, MA) 2400 series II CHNS/O analyzer at the Sophisticated Instrumentation Centre for Applied Research and Testing (Sicart, Gujarat, India). The number-average molecular weight (M_n) was determined by conductometric titration in DMF with KOH in a 50% (v/v) DMF/alcohol mixture as the titrant. The viscosities were determined with a Tuan-Fuoss viscometer (Nagpur University, Nagpur, India)[15] at six different concentrations ranging from 0.5 to 3.0% of the copolymer in DMF at 33°C. The intrinsic viscosity ($[\eta]$) was calculated with the Huggins equation [eq. (1)][16] and the Krammer equation [eq. (2)]:[17]

$$\eta_{sp}/C = [\eta] + k_1[\eta]^2 C \qquad (1)$$

$$\ln \eta_{rel}/C = [\eta] - k_2[\eta]^2 C \qquad (2)$$

where C is the concentration (g/100 mL); η_{rel} is the ratio of the viscosity of the solution (η) to the viscosity of the solvent (η_0), that is, the relative viscosity ($\eta_{rel} = \eta/\eta_0$); and η_{sp} is a function derived from η_{rel} [$\eta_{sp} = (\eta - \eta_0)/\eta_0 = \eta/\eta_0 - 1 = (\eta_{rel}) - 1$]. k_1 and k_2 are constants for a given polymer/solvent temperature system.

The electronic absorption spectrum of the copolymer in DMF was recorded on a Shimadzu double-beam spectrophotometer in the range of 280–700 nm at the Department of Chemistry of Nagpur University (Nagpur, India). The infrared spectrum of the BPhF copolymer resin was recorded on a Nicolet Magna 550 IR spectrophotometer in KBr pellets in the region of 4000–400 cm^{-1}. The ^{1}H-NMR spectrum of the BPhF copolymer resin was scanned on a Varian VXR 300S 300 MHz ^{1}H-NMR spectrophotometer with DMSO-d_6 as a solvent.

Ion-exchange properties

The ion-exchange properties of the BPhF copolymer resin were determined by the batch equilibrium method.[18] The ion-exchange properties of the prepared BPhF resin were studied.

Determination of the metal uptake in the presence of various electrolytes and different concentrations

The copolymer sample (25 mg) was suspended in an electrolyte solution (25 mL) of a known concentration. The pH of the suspension was adjusted to the required value with either 0.1M HNO$_3$ or 0.1M NaOH. The suspension was stirred for 24 h at 25°C. To this suspension was added 2 mL of a 0.1M solution of the metal ion, and the pH was adjusted to the required value. The mixture was again stirred at 25°C for 24 h and filtered.[18] The solid was washed, the filtrate and washing were combined, and the metal ion content was determined by titration against standard ethylenediaminetetraacetic acid. The metal ion uptake of the polymer was calculated from the difference between a blank experiment without the polymer and the reading in the actual experiments.[6,18] The experiment was repeated in the presence of several electrolytes.

Evaluation of the rate of metal uptake

To estimate the time required to reach the state of equilibrium under the given experimental conditions, a series of experiments of the type described previously were carried out in which the metal ion taken up by the chelating resins was determined from time to time at 25°C (in the presence of 25 mL of a 1M NaNO$_3$ solution). It was assumed that, under the given conditions, the state of equilibrium was established within 24 h. The rate of metal uptake is expressed as a percentage of the amount of metal ions taken up after a certain time related to that at the state of equilibrium.

Evaluation of the distribution of metal ions at different pHs

The distribution of each one of the seven metal ions, that is, Cu(II), Ni(II), Co(II), Zn(II), Cd(II), Pb(II), and Fe(III), between the polymer phase and the aqueous phase was determined at 25°C and in the presence of a 1M NaNO$_3$ solution. The experiments were carried out as described previously at different pH values. The distribution ratio is defined by the following relationship:

$$\text{Distribution ratio} = \frac{\text{Weight (mg) of metal ions taken up by 1 g of copolymer}}{\text{Weight (mg) of metal ions present in 1 ml of solution}}$$

RESULTS AND DISCUSSION

The copolymer used in this investigation was prepared by the reaction shown in Figure 1.
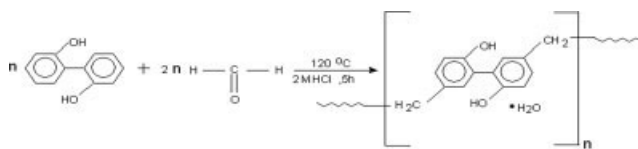
Figure 1 Suggested structure of the copolymer.

The newly synthesized purified BPhF copolymer resin was found to be faint pink in color. The copolymer resin was soluble in DMF, DMSO, and aqueous KOH and NaOH solutions and was insoluble in almost all organic solvents and concentrated acids. The resin was analyzed for the carbon and hydrogen contents [found (calcd.): C, 73.68% (73.04%); H, 6.12% (6.09%)]. The decomposition temperature of the polymer was found to be above 400°C. The chemical composition calculated for the repeat unit of the BPhF copolymer was $C_{14}H_{12}O_2 \cdot H_2O$.

Characterization of the copolymer

M_n of the copolymer was determined by nonaqueous conductometric titration in DMF against KOH in a 50% (v/v) DMF–alcohol mixture with a 50-mg sample. A plot of the specific conductance against the milliequivalents of potassium hydroxide required for the neutralization of 100 g of the copolymer was made (Fig. 2). The inspection of such a plot revealed that there were many breaks in the plot. From this plot, the first and last breaks were noted. The calculation of M_n by this method is based on the following considerations.[13] First, the first break corre-
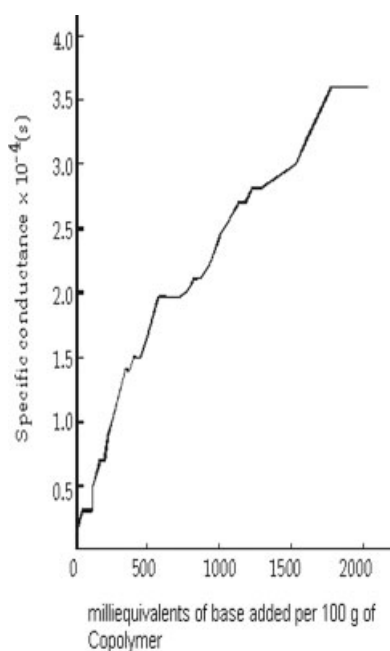
sponds to neutralization of the more acidic phenolic hydroxy group of all the repeating units, and second, the break in the plot beyond which a continuous increase is observed represents the stage at which the phenolic hydroxy groups of all repeating units are neutralized on the basis of the average degree of polymerization (D_p), which is given by the following relation:

$$D_p = \frac{\text{Total meq. of base required for complete neutralization}}{\text{Meq. of base required for smallest interval}}$$

M_n was obtained by the multiplication of D_p by the formula weight of the repeating unit and found to be 10,260.

Viscometric measurements were carried out in a DMF solution at 33°C with a Tuan-Fuoss viscometer.[15] The reduced viscosity (η_{red}) versus the concentration (3 to 0.5%) was plotted for the set of data. [η] was determined from corresponding linear plots (Fig. 3). Equations (1) and (2) were used to determine the Huggin[16] and Krammer[17] constants, respectively. As η_{red} is dependent on the concentration of the polymer sample, it is necessary to extrapolate a plot of η_{red} against the polymer concentration (C) to the zero concentration to get [η], which is a characteristic parameter of a polymer. Hence, [η] is given in equations (3) and (4) as follows:

$$[\eta] = (\eta_{sp}/C)_{C \to 0} \tag{3}$$

or

$$[\eta] = (\ln \eta_{rel}/C)_{C \to 0} \tag{4}$$

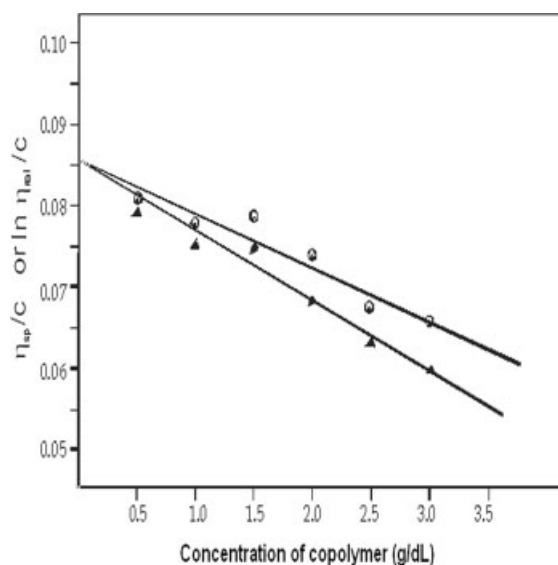**Figure 2** Conductometric titration curve of the BPhF copolymer.

Figure 3 Viscometric plot of the BPhF copolymer.

According to this relation, the plots of η_{sp}/C and ln η_{red}/C against C were linear with slopes of k_1 and k_2, respectively. The intercept on the viscosity function axis gave the $[\eta]$ value in both plots. The calculated values of the constants k_1 (0.6522) and k_2 (−0.0885) satisfy the relation $k_1 + k_2 = 0.5$ favorably,[13] and this indicates that the solvent used for the viscosity measurement was of good quality; $[\eta]$ for the polymer was found to be 0.086 dL/g.

The ultraviolet–visible spectrum (Fig. 4) of the BPhF copolymer in pure DMF was recorded in the region of 280–400 nm at a scanning rate of 100 nm/min and at a chart speed of 5 cm/min. The copolymer sample displayed two characteristic bands at 295 and 330 nm. The intense band at 295 nm is due to the $\pi \rightarrow \pi^*$ allowed transition of the biphenyl moiety, which readily attains coplanarity, and the shoulder merging (loss of fine structure) band at 330 nm may be due to n–π^* transitions.[19] The bathochromic shift from the basic values of 252 and 270 nm, respectively, may be due to a combined effect of the conjugation and phenolic hydroxy group (auxochrome),[19] or it may be due to the conversion of phenol to the corresponding anion.

The infrared spectrum of the BPhF copolymer resin is shown in Figure 5. The IR spectral study revealed that a broad band appeared in the region of 3200–3400 cm^{-1}, which may be assigned to the stretching vibration of phenolic hydroxyl groups exhibiting intermolecular hydrogen bonding.[19,20] The bands observed at 2925, 1499, 1444, 1342, and 767 cm^{-1} suggest the presence of methylene (—CH$_2$—) bridges in the copolymer.[19,20] A sharp doublet in the region of 1617–1622 cm^{-1} may be ascribed to substituted aromatic skeletal ring breathing modes.[19,20] The band at 1296–1230 cm^{-1} may be due to >C—O stretching of the polymeric phenol.[19,20] The band at
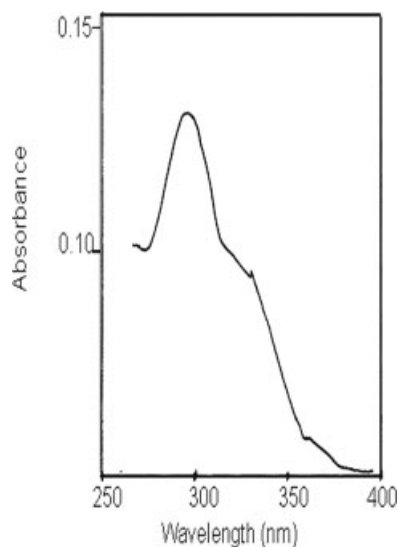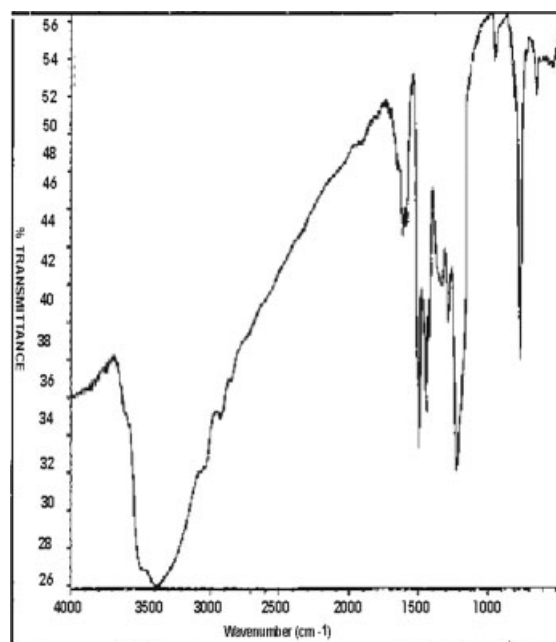

Figure 5 Infrared spectrum of the BPhF copolymer.

900 cm^{-1} may be due to one isolated H atom situated between two substituents,[19,20] and the band at 644 cm^{-1} is due to O—H bending out of plane.[19]

The NMR spectrum of the copolymer was scanned in DMSO-d_6. The spectral data are given later in Table III, and the spectrum is depicted in Figure 6. From the spectrum, it is revealed that the signal at $\delta = 3.5$–3.9 ppm may be due to methylenic protons of the Ar—CH$_2$—Ar bridge.[19] The triplet in the region of $\delta = 6.8$–7.2 ppm is due to aromatic protons[19]; this is because the resin possesses benzenoid hydrogen, which exhibits three types of coupling. First, the
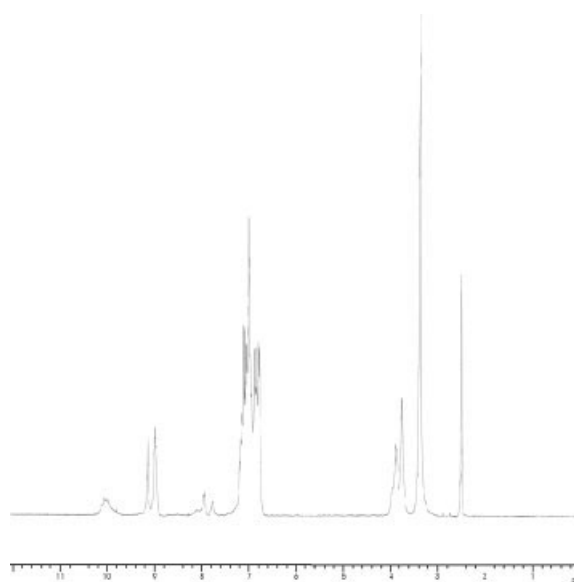

Figure 4 Electronic spectrum of the BPhF copolymer.

Figure 6 ^{1}H-NMR spectrum of the BPhF copolymer.

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TABLE I
Evaluation of the Influence of Different Electrolytes on the Uptake of Several Metal Ions of the BPhF Copolymer

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Metal ion	Electrolyte (mol/L)	Weight of the metal ion uptake (mg)		
		In the presence of NaCl	In the presence of NaNO$_3$	In the presence of Na$_2$SO$_4$
Fe^{3+}	0.01	0.21	0.67	1.90
	0.05	0.49	1.13	1.80
	0.1	1.39	1.54	0.87
	0.5	1.70	1.59	0.41
	1.0	2.36	2.11	0.05
Cu^{2+}	0.01	0.85	1.02	2.92
	0.05	1.58	1.46	2.69
	0.1	2.72	2.51	1.64
	0.5	2.75	2.51	0.94
	1.0	3.68	2.60	0.06
Ni^{2+}	0.01	1.03	0.59	2.75
	0.05	1.35	1.30	2.21
	0.1	2.24	2.48	1.67
	0.5	2.59	2.86	1.03
	1.0	3.29	3.02	0.03
Co^{2+}	0.01	1.68	1.52	1.46
	0.05	1.03	0.87	1.03
	0.1	0.87	0.76	0.81
	0.5	0.65	0.65	0.54
	1.0	0.16	0.32	0.11
Zn^{2+}	0.01	2.47	1.80	1.38
	0.05	2.05	1.20	1.02
	0.1	1.26	0.90	0.90
	0.5	0.54	0.66	0.30
	1.0	0.12	0.24	0.18
Cd^{2+}	0.01	1.55	1.45	1.65
	0.05	1.03	1.14	1.03
	0.1	0.72	0.83	0.83
	0.5	0.31	0.72	0.72
	1.0	0.10	0.21	0.21
Pb^{2+}	0.01	0.67	0.95	—
	0.05	0.65	0.76	—
	0.1	0.61	0.57	—
	0.5	0.57	0.68	—
	1.0	0.34	0.40	—

[M$_t$(NO$_3$)$_2$] = 0.1 mol/L; volume = 2 mL; volume of the electrolyte solution = 25 mL; weight of the resin = 25 mg; time = 24 h; temperature = room temperature; pH of Fe^{3+} = 2.5; pH of Cu^{2+}, Ni^{2+}, and Pb^{2+} = 4.5; pH of Co^{2+} and Zn^{2+} = 5; pH of Cd^{2+} = 6.

hydrogen adjacent to the OH group gives a doublet due to o-coupling. The p-coupling is very small in this case and thus manifests only a small broadening of the doublet. Second, the hydrogen flanked between two substituents is at the lowest field and gives a doublet due to small m-coupling. Third, the hydrogen atom adjacent to the —CH$_2$— substituent on the ring displays a double doublet due to simultaneous coupling to both H atoms (o-coupling and m-coupling).

The doublet at δ = 7.7–8.0 ppm is due to Ar—OH polymeric association.[19,21] A doublet at δ = 9–9.2 ppm and a broad signal at δ = 10 ppm show Ar—OH protons undergoing a slow exchange of protons (a signal for both Ar—OH exchanging protons) and an intermediate proton exchange reaction of Ar—OH.[19,21] Solvent peaks appear in the region of δ = 2.5 ppm and δ = 3.25 ppm.

However, on the basis of the spectral data, the nature and reactive sites of the monomers, and the linear structure of other substituted phenol–formaldehyde polymers, the most probable structure proposed for the BPhF copolymer is shown in Figure 1.

Ion-exchange properties

The results of the batch equilibrium study carried out with the copolymer sample of BPhF are presented in Table I–III. From this study of seven metal ions with limited variations of the experimental conditions, a certain generalization may be made about the behavior of the copolymer sample.

Effect of the electrolytes on the metal uptake

We examined the influence of NO$_3^-$, Cl$^-$, and SO$_4^{2-}$ at various concentrations on the equilibrium of the metal–resin interaction. Table I shows that the amount of metal ions taken up by a given amount of the copolymer depends on the nature and concentration of the electrolyte present in the solution. In the presence of chloride and nitrate ions, the uptake of

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TABLE II
Comparison of the Rate of Metal Ion Uptake of the BPhF Copolymer

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Metal ion	Metal (M$_t$) ion uptake (%)[a]						
	1 h	2 h	3 h	4 h	5 h	6 h	7 h
Fe^{3+}	59	72	84	95	—	—	—
Cu^{2+}	43	52	56	79	90	—	—
Ni^{2+}	27	45	58	78	89	—	—
Co^{2+}	27	46	58	77	89	—	—
Zn^{2+}	28	45	58	78	89	—	—
Cd^{2+}	—	14	18	41	55	66	75
Pb^{2+}	—	—	17	24	29	64	72

[M$_t$(NO$_3$)$_2$] = 0.1 mol/L; volume = 2 mL; NaNO$_3$ = 1.0 mol/L; volume of NaNO$_3$ = 25 mL; temperature = room temperature.
[a] Metal ion uptake = $\dfrac{\text{Amount of metal ion absorbed} \times 100}{\text{Amount of metal ion absorbed at equilibrium}}$

TABLE III
Distribution Ratio of Different Metal Ions as a Function of the pH of the BPhF Copolymer

Metal ion	Distribution ratio of metal ions at different pHs[a]									
	1.5	1.75	2.0	2.5	3.0	3.5	4	5	6	6.5
Fe^{3+}	14.15	106.1	268.7	403.3	—	—	—	—	—	—
Cu^{2+}					84.8	155.4	341.9	536.9	1243.0	1511.8
Ni^{2+}					73.0	204.3	277.3	496.3	951.6	1036.3
Co^{2+}					33.8	187.8	162.2	189.2	216.2	222.9
Zn^{2+}					19.6	39.1	80.9	130.4	195.6	221.7
Cd^{2+}					14.5	21.7	58.0	159.5	181.3	188.5
Pb^{2+}					18.6	33.7	75.9	109.7	185.6	198.3

$[M_t(NO_3)_2]$ = 0.1 mol/L; volume = 2 mL; $NaNO_3$ = 1.0 mol/L; volume = 25 mL; time = 24 h (equilibrium state); temperature = room temperature.

[a] Distribution ratio = $\frac{\text{Weight (mg) of the metal ions taken up by 1 g of the copolymer}}{\text{Weight (mg) of the metal ions present in 1 mL of solution}}$.

Fe(III), Cu(II), and Ni(II) ions increases with an increasing concentration of the electrolytes, whereas in the presence of sulfate ions, the amount of the aforementioned ions taken up by the copolymer decreases with an increasing concentration of the electrolyte. Moreover, the amount of Co(II), Zn(II), Cd(II), and Pb(II) ions decreases with increasing concentrations of chloride, nitrate, and sulfate ions.[15,18] This may be explained on the basis of stability constants of complexes with those metal ions.[22]

Rate of the metal ion uptake

The rate of metal adsorption was determined to find out the shortest period of time for which equilibrium could be carried out as close to equilibrium conditions as possible. Table II shows the dependence of the rate of metal ion uptake on the nature of the metal. The rate refers to the change in the concentration of the metal ions in the aqueous solution that is in contact with the given polymer. The results show that the time taken for the uptake of the different metal ions at a given stage depends on the nature of the metal ion under the given conditions. It has been found that Fe(III) requires about 4 h for the establishment of the equilibrium, whereas Cu(II), Ni(II), Co(II), and Zn(II) require about 5 h. Thus, the rate of metal ion uptake follows the order of Fe(III) > Cu(II) > Ni(II) = Co(II) = Zn(II) > Cd(II) > Pb(II) for the BPhF copolymer.

Distribution ratio of the metal ion at different pHs

The effect of pH on the amount of metal ions distributed between two phases can be explained by the results given in Table III. The data on the distribution ratio as a function of pH indicate that the relative amount of metal ions taken up by the BPhF copolymer increases with the increasing pH of the medium.[16,18,22] The magnitude of the increase, however, is different for different metal ions. The BPhF copolymer resin takes up Fe(III) more selectively than any other metal ions under study. The order of the distribution ratio of the metal ions measured in the pH range of 2.5–6.5 is found to be Fe(III) > Cu(II) > Ni(II) > Co(II) > Zn(II) > Cd(II) > Pb(II). Thus, the results of such a type of study are helpful in selecting the optimum pH for a selective uptake of a particular metal cation from a mixture of different metal ions.[23,24] For example, the results suggest an optimum pH of 3 for the separation of Cu(II) and Cd(II) with the copolymer resin with the distribution ratios of 84.8 and 14.5, respectively.

CONCLUSIONS

1. A BPhF resin based on the condensation reaction of 2,2′-dihydroxybiphenyl with formaldehyde in the presence of hydrochloric acid as a catalyst was prepared.
2. The resin was found to be highly selective for Fe(III).
3. The results of an ion-exchange study are helpful in selecting the optimum pH for a selective uptake of a particular metal cation from a mixture of different metal ions.

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