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Analytical applications of newly synthesized resin derived from salicylic acid, phenylenediamine and formaldehyde

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ABSTRACT

The present paper reports the synthesis, characterization and ion exchange properties of a resin in presence of 2M HCl as catalyst. The resin has been proved to be selective chelating ion-exchange resins for certain metals. Chelation ion exchange properties have also been studied for Fe^{3+}, Cu^{2+}, Ni^{2+}, Co^{2+}, Zn^{2+}, Cd^{2+}, Pb^{2+} ions employing a batch equilibrium method. It was employed to study the selectivity of metal ion uptake involving the measurements of distribution of a given metal ion between the polymer sample and a solution containing the metal ion. The study was carried out over wide pH range and in a media of various ionic strengths. The resin showed higher selectivity for Fe^{3+}, Cu^{2+} and Ni^{2+}ions than for Co^{2+}, Zn^{2+}, Cd^{2+}, and Pb^{2+} ions. The resin SPDF was synthesized by the condensation of salicylic acid- phenylenediamine with formaldehyde in the presence of a hydrochloric acid catalyst. Resin was characterized by elemental analysis, infrared (IR) spectroscopy, nuclear magnetic resonance (NMR) spectroscopy and UV- Visible spectral studies. The number average molecular weight of the resin was determined by non-aqueous conductometric titration.

Key Words: Resin, FT-IR, Batch equilibrium,Distribution ratio .

INTRODUCTION

Synthesis, characterization and ion exchange studies of polymeric resins deserves to be investigated because their varied characteristics and beneficial properties. Ion exchangers are widely used in hydrometallurgy, antibiotic purification, analytical chemistry, separation of radioisotopes and also find large scale application in water treatment and pollution control[1-4].Resins are found to exhibits better thermal and electrical properties than those of phenol-formaldehyde type copolymer. These resins can be used as high energy materials, ion exchangers, semiconductors, bonding agent/additives, molding material, fungicides in plant and living tissues, biosensors and controlled release devices for pharmaceuticals [5-11]. Now a day's renewed interest has been evidenced in the context of obtaining materials with high temperature resistance and ion exchangers. Patel and Lad[12] reported the novel terpolymers: poly (keto-amine-ureas) and studied the glass reinforcement of such terpolymer- epoxy systems. Michael and coworkers have reported the synthesis, structural and thermal degradation of a polymer derived from salicylic acid, guanidine and formaldehyde [13].Synthesis, characterization and ion exchange properties of 4-hydroxyacetophenone, biuret and formaldehyde terpolymer resin were studied by Gurnule and coworkers [14]. Ion exchange resin derived from semicarbazone and oximes of 2-hydroxy acetophenone-substituted benzoic acid - formaldehyde studied by Nayak et.al.[15,16]. The synthesis and characterization of 2, 4- dihyroxyacetophenone with 1, 4-butanediolcopolymer and its ion exchange properties have been reported [17]. Chelation ion exchange resins derived from 2-hydroxy-4-methoxyacetophenone and thiourea with trioxane was investigated by Pancholi et. al.[18]. Parmar [19, 20] reported the ion exchange properties of resacetophenone (2, 4 dihydroxyAcetophenone) - formaldehyde and Resacetophenone oxime-formaldehyde resins. In our laboratory, extensive research work was carried out on synthesis and characterization of resins and their ion exchange properties [4, 5, 21, 22]. However, the literature studies have revealed that no resin has been synthesized using the monomers salicylic acid, phenylenediamine and formaldehyde. Therefore in the present communication we report synthesis, characterization and analytical applications of a SPDF resin.

MATERIALS AND METHODS

2.1 Chemicals

All Chemicals were AR grade. Salicylic Acid, phenylenediamine were purchased from Aldrich Chemical Co., USA while Formaldehyde from RANKEM, Ranbaxy, India, DMF and DMSO (HPLC grade) were used.

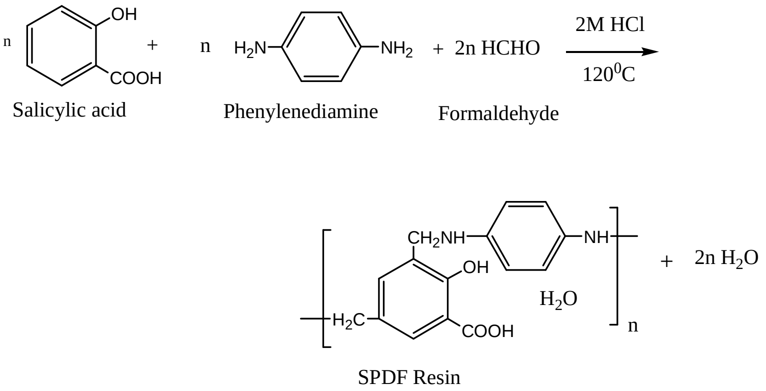
2.2 Instruments used

C, H and N were analyzed on Carlo Erber Elemental Analyzer Fourier Transform. Infrared spectrum in the region 400-4000cm^{-1} was recorded on a Perkin-Elmer with KBr pellets. The electronic spectrum of the resin was recorded in dimethylformamide (DMF) at room temperature with a UV-240 Shimadzu double beam spectrophotometer. The NMR was scanned at 300MHz with dueterated dimethylsulfoxide (DMSO) solvent at SAIF, Chandigarh.

2.3 Synthesis of SPDF Resin

2.3.1 Condensation of Salicylic acid (S) and Phenylenediamine (PD) with Formaldehyde (F)

A mixture of salicylic acid, phenylenediamine, and formaldehyde along 2M hydrochloric acid was taken in a round bottom flask fitted with water condenser and heated in an oil bath at 120^{0}C for 7 hours with occasional shaking. The temperature of electrically heated oil bath was controlled with the help of dimmerstat .The resinous solid product obtained was immediately removed from the flask as soon as the reaction period was over and then purified. The resinous product so obtained was repeatedly washed with cold distilled water dried in air and powdered with the help of agated mortar and pestle. The powder was washed many times with hot water to remove unreacted monomers. The air-dried powder was extracted with diethyl ether and then petroleum ether was used to remove salicylicacid phenylenediamine copolymer, which might be present along with SPDF resin. It was further purified by dissolving in 8% sodium hydroxide solution, filtered and reprecipeted by gradual drop wise addition of 1:1 (v/v) hydrochloric acid with constant and rapid stirring to avoid lump formation [4, 5, 21, 22]. The SPDF resin so obtained was filtered, washed several times with hot water and dried (yield=14g; mp=274^{0}C).Analytical data for C15H14N2O3•H2O as per numerical calculations and experimental evidences are mentioned below respectively. Theoretically calculated C=62.50%, N=5.32%, H=9.72%. Experimentally found C=62.54%, N=5.47%, H=9.31%.



Synthesis of SPDF resin

2.4. Ion exchange properties

The purified SPDF resin sample was finely powdered and used in all experiments carried out in the ion exchange study.

2.4.1 Determination of Metal ion uptake in the presence of Electrolytes of Different Concentration

25mg of the finely powdered resin was suspended in an electrolyte solution (25ml) of known concentration. The pH of the solution was adjusted to required value by using either 0.1M HNO$_3$ or 0.1M NaOH. The suspension was stirred for a period of 24 hours at room temperature. To this suspension 2ml of 0.1M solution of electrolyte was added and the pH was adjusted to the required value. The mixture was again stirred at 25^{0}C for 24 hour and filtered.

The polymer was washed and the filtrate and washings were combined and estimated for the metal ion content by titration against standard ethylenediaminetetraacetic acid. A blank experiment was also carried out in the same manner without adding the polymer sample to estimate the metal ion content. The amount of metal ion taken up by the resin in the presence of given electrolyte of known concentration was determined from the difference between the blank reading and the reading in the actual experiment [4, 5, 21, 22]. The experiment was repeated in the presence several electrolytes of known concentration with seven different metal ions such as Fe^{3+}, Cu^{2+}, Ni^{2+}, Co^{2+}, Zn^{2+}, Cd^{2+}, Pb^{2+}. The results with seven different metal ions are reported in table 1.

2.4.2. Evaluation of the Rate of Metal Uptake

In order to determine the time required to reach the state of to reach the state of equilibrium under given experimental conditions, series of experiments of the type described above were carried out, in which the metal ion uptake by the chelating resins was estimated from time to time at room temperature at 25^0C. It was assumed that under given conditions, the state of equilibrium is established in the 24 hrs. The rate of metal ion uptake is expressed as percentage of the amount of metal ions taken up after a certain time related to that in state of equilibrium as table 2 [4, 5, 21, 22].

2.4.3. Evaluation of distribution of metal ions at different pH values

The distribution of each of the metal ions Fe^{3+}, Cu^{2+}, Ni^{2+}, Co^{2+}, Zn^{2+}, Cd^{2+}, Pb^{2+} between the resin phase and aqueous phase was estimated at 25^0C using 1M $NaNO_3$ solution. The experiments were carried out as described above at different pH values.

The distribution ratio "D" was determined by the following equation [4, 5, 21, 22]:

$$D = \frac{\text{Weight of metal ions taken up by 1g of resin}}{\text{Weight of metal ions present in 1ml of solution}}$$

The results are presented in table 3.

RESULTS AND DISCUSSION

The resin was soluble in DMF and DMSO; however it is insoluble in common organic solvents. The composition of the polymeric unit was assigned on the basis of a detailed study of the elemental analysis of the polymer, ultraviolet-visible (UV-Visible), IR and NMR spectral studies.

3.1. Characterization of resin

3.1.1. Determination of molecular weight by conductometric titration

Molecular weight (Mn) of the resin was determined by non-aqueous conductometric titration in DMF against ethanolic KOH by using 50mg of sample. A plot (Fig.1) of the specific conductance against the milieqvivalents of potassium hydroxiderequired for neutralization of 100g of resin was made. Inspection of such a plot revealed the breaks in the plot. The first break at 180 miliequivalent of base and the last break at 2520 miliequivalent of base were noted. The calculations of (Mn) by this method is based on the following considerations [4, 5, 21, 22]: (1) the first break corresponds to neutralization by the more acidic phenolic hydroxy group of all the repeating units; (2) The break in the plot beyond which a continuous increase is observed represents the stage at which phenolic hydroxy group of all the repeating units are neutralized. On the basis of the average degree of polymerization (DP) is given by the following relation.

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

The average degree of polymerization (DP), which is given by the following relation, is found to be 14 and the number average molecular weight (M_n) is 4032 as obtained by multiplying the DP by the formula weight of the repeating unit.[4, 5, 21, 22]

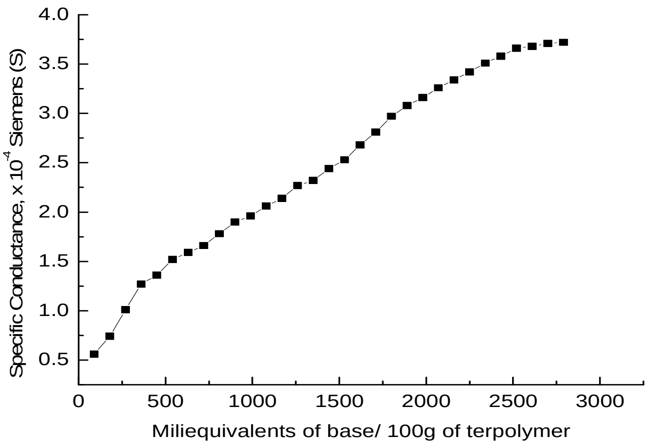


Figure 1: Conductometric titration curve of SPDF Resin

3.1.2. UV-Visible studies

The electronic spectrum of the SPDF resin is shown in fig.2. The spectra depicted two characteristic bands in the region of 280 nm and 340nm. The band at 280 nm indicate the presence of a carbonyl (>C=O) group containing a carbon oxygen double bond in conjugation with an aromatic nucleus and can be accounted for $\pi \rightarrow \pi^*$ transition while the latter band (less intense) may be due to $n \rightarrow \pi^*$electronic transition. The additional shift of absorption to the longer wavelength region i.e. bathochromic shift from the basic value (237nm and 320nm, respectively) may be due to conjugation effect and presence of phenolic hydroxy group (auxochrome) is responsible for hyperchromic effect i.e. higher ε_{max} value [4, 5, 21, 22]

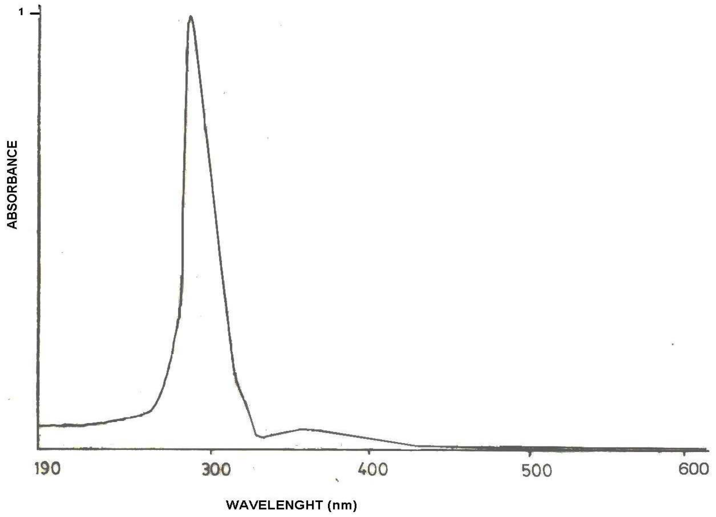


Figure2: Electronic spectra of SPDF Resin

3.1.3Infra Red Studies

The IR spectrum of the newly synthesized SPDF resin is presented in fig.3. The assignment of vibrational frequencies is mainly based on the data available in the literature. A broad band appeared at 3275cm^{-1} might be due to the stretching vibrations of phenolic hydroxyl group exhibiting intermolecular hydrogen bonding which exists between -OH group of different polymer chains [4, 5, 21, 22]. The band observed at 3064 cm^{-1} may be due to the stretching vibrations of –NH (imide) [4, 5, 21-26]. The inflections around 1454cm^{-1}, 1290cm^{-1} and 763cm^{-1} suggest the presence of bending, wagging, rocking vibrations of methylene ($-CH_2-$) bridges in polymeric chains [21-26,]. The sharp band at 1621cm^{-1} may be due to the stretching vibrations of >C=O [21-26]. The bands obtained at 1490cm^{-1}, 763 and 691cm^{-1} may be assigned to –NH- bending, wagging and deformation out of plane vibrations in terpolymer resin respectively[4, 5, 21, 22]. The band at 1332 cm^{-1} may be ascribed to phenolic >C-O stretching modes of vibration [21-29]. The band at 904,1015and1171 cm^{-1}suggests that the aromatic ring is 1, 2, 3, 5- tetra substituted [21-29]. This fact is further supported by the presence of band at 849 cm^{-1} for tetra-substituted benzene ring [21-26].

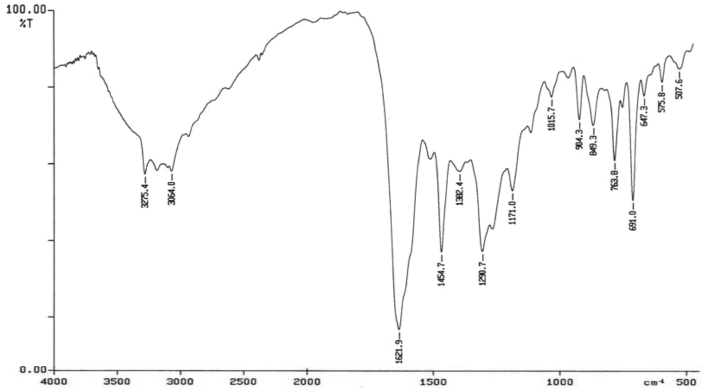


Figure3: Infrared spectra of SPDF Resin

3.1.4. NMR studies

The NMR spectrum of the SPDF resin is shown in fig. 4 and exhibited signal in the region of 7.72 δ (ppm), which may be due to the protons of the aromatic ring (Ar-H), and the signal in the region 7.83 δ (ppm) can be assigned to the phenolic-OH proton in hydrogen bonding (Ar-OH) [4, 5, 21,22,29,31,35]. The signal displayed at 7.43 δ (ppm) may be due to the carboxylic proton of Ar-COOH [29]. The presence of a broad signal around 6.91 δ (ppm) is attributed to the presence of –NH bridge [4, 5, 21, 22, 29]. A methylene proton Ar-CH_2-N moiety was inferred by the appearance of a weak singlet signal at 3.89 δ (ppm) [29, 31].

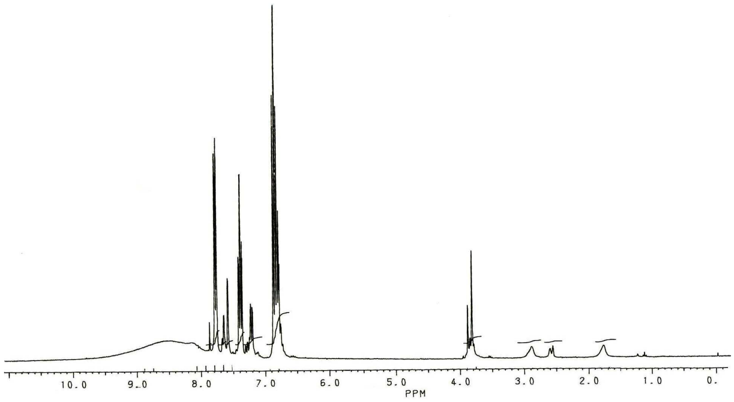


Figure4: NMR spectrum of SPDF Resin

3.2. Ion-exchanging properties

To ascertain the selectivity of SPDF, we have studied the influence of various electrolytes, the rate of metal uptake and the distribution of metal ions between the resin and solution. The results of the batch equilibrium study carried out with resin sample SPDF are presented in tables 1,2 and 3.

3.2.1. Effect of Electrolyte on Metal Uptake

The data reveals that the amount of metal ions taken up for a given amount of resin sample depends on the nature and concentration of electrolyte present in the solution. In presence of chloride and nitrate ions, the amount of Cu^{2+}, Fe^{3+} and Ni^{2+} ions taken up by the resin sample increases while in presence of sulphate ions the amount of above mentioned ions taken up by the resin samples decreases with increasing concentration of electrolyte. Moreover, The amount of Co^{2+}, Zn^{2+}, Cd^{2+} and Pb^{2+} ions taken up by the resin samples decreases with increasing concentration of the chloride, nitrate and sulphate ions. This may be explained on the basis of stability constant of the complexes with Fe^{3+}, Cu^{2+}, Ni^{2+} Co^{2+}, Zn^{2+}, Cd^{2+}, and Pb^{2+} metal cations [4,5,21,22,32-35].

Table 1: Evaluation of the effect of different electrolytes on the uptake of several metal ions of SPDF Resin

Metal ion	Electrolyte Conc. (mole/lit)	Weight of metal ion (mg) taken up in presence of		
		$NaNO_3$	NaCl	Na_2SO_4
Fe^{3+}	0.01	1.88	1.82	2.58
	0.05	2.04	1.99	2.45
	0.1	2.15	2.13	2.11
	0.5	2.46	2.44	1.94
	1	2.64	2.61	1.81
Cu^{2+}	0.01	0.59	0.57	1.44
	0.05	0.81	0.76	1.34
	0.1	1.09	1.01	1.04
	0.5	1.35	1.28	0.75
	1	1.63	1.48	0.55
Ni^{2+}	0.01	0.9	0.84	1.74
	0.05	0.97	0.92	1.39
	0.1	1.17	1.08	0.95
	0.5	1.53	1.44	0.79
	1	1.95	1.92	0.71
Co^{2+}	0.01	1.77	1.69	1.73
	0.05	1.49	1.45	1.41
	0.1	1.16	1.07	1.06
	0.5	1.02	0.95	0.95
	1	0.87	0.85	0.83
Zn^{2+}	0.01	1.36	1.33	1.31
	0.05	1.21	1.28	1.23
	0.1	0.97	0.87	0.92
	0.5	0.78	0.71	0.68
	1	0.69	0.64	0.59
Cd^{2+}	0.01	1.29	1.19	1.05
	0.05	1.17	1.04	0.96
	0.1	0.96	0.86	0.69
	0.5	0.85	0.67	0.61
	1	0.66	0.61	0.45
Pb^{2+}	0.01	1.27	1.05	0.89
	0.05	1.05	0.79	0.63
	0.1	0.87	0.64	0.54
	0.5	0.71	0.55	0.37
	1	0.63	0.37	0.13

3.2.2. Rates of metal uptake

The rates of metal adsorption by the SPDF resin was measured for Fe^{3+}, Cu^{2+}, Ni^{2+} Co^{2+}, Zn^{2+}, Cd^{2+} and Pb^{2+} ions, in order to know the equilibrium time required. The term "rates" refers merely change in the concentration of metal ion in the aqueous solution, which is in contact with given resin. The experimental results which are given in table 4, shows that the time taken for the uptake of the metal ions at a given stage depends on the nature of the metal ion under the given conditions. As the size of the metal ion increases time taken for the uptake metal ion also increases. The experimental results indicate that Fe^{3+} ions required less time of 4 hours for the establishment of equilibrium. Cu^{2+}, Ni^{2+}, Co^{2+}, Zn^{2+}, ion required 5 hours while Cd^{2+} and Pb^{2+} ions required about 6 hour reaching for the establishment of equilibrium. It is further revealed that the rates of metal ion uptake follow the order: $Fe^{3+} > Cu^{2+} \sim Ni^{2+} \sim Co^{2+} \sim Zn^{2+} > Cd^{2+} \sim Pb^{2+}$ for the resin [4, 5, 21, 22, 32-35].

Table 2: Comparison of the rates of metal ion uptake of SPDF Resin

Metal ions	Percentage of the metal ion uptake at different times (h)						
	0.5	1	2	3	4	5	6
Fe^{3+}	57	65	76	83	94	-	-
Cu^{2+}	31	42	63	70	84	91	-
Ni^{2+}	17	30	45	63	73	92	-
Co^{2+}	11	26	41	56	70	90	-
Zn^{2+}	10	16	28	54	69	90	-
Cd^{2+}	10	22	52	64	70	83	92
Pb^{2+}	8	17	30	62	72	83	90

$[M^+(NO_3)_2]$=0.1 mol/l, Vol.=2ml,$NaNO_3$=0.1mol/l, Vol.of electrolyte solution=25ml, time=24hr (equilibrium state), room temperature.

3.2.3. Evaluation of distribution coefficient of the metal ions over wide pH range

Perusal of data given in table 3 the selectivity of resin samples for $Fe^{3+}$is higher than that of other metal ions .The lower distribution ratio of Fe^{3+} due to sterric hindrance imposed by polymer matrix [4,5,21,22,32-35]. Among the other metal ions Cu^{2+} and Ni^{2+} ions are taken up more selectively by resin. The other metal ions Co^{2+}, Zn^{2+}, Cd^{2+} and Pb^{2+}have distribution ratio, 'D' over the pH range of 4 to 6.5. This could be attributed to the low stability constant i.e. the weak ligand stabilization energy of the metal complexes. The possible order of selectivity of a cation-exchange resin for divalent metal ions is: Pd>Cu>Ni>Co>Zn>Cd>Fe>Mn>Mg. Comparison of the values of distribution coefficients of a given metal ion for all the four molar ratio of resin reveals that there is no much difference in the values. Thus in the present study the observed order of distribution ratio of metal ions measured in the range of 1.5to 6.5pH was found to be Fe^{3+} > Cu $^{2+}$> Ni^{2+} > Co^{2+} > Zn^{2+} > Cd^{2+} > Pb^{2+}. Earlier workers have also suggested this order of selectivity for a salicylic acid containing polymer.

Table 3 : Distribution ratio of the metal ion as a function of the pH of SPDF Resin

Metal ion	Distribution ratio of the metal ion at different pH								
	1.5	2	2.5	3	4	4.5	5	6	6.5
Fe^{3+}	168.6	243.3	527.2	-	-	-	-	-	-
Cu^{2+}	-	-	-	81.4	147.7	323	533.4	904.6	1061.3
Ni^{2+}	-	-	-	68.3	96.4	126.6	533.4	904.6	1061.3
Co^{2+}		-	-	41	75.5	89.4	132.7	194.5	338.9
Zn^{2+}	-	-	-	23.4	42.4	62.6	123.3	193.1	294.8
Cd^{2+}	-	-	-	19.1	41.1	58.3	113.5	152.3	236.3
Pb^{2+}	-	-	-	30.5	47.1	63.2	97.6	161.2	254.8

[$M^+(NO_3)_2$]=0.1 mol/l, Volume = 2ml , $NaNO_3$ = 0.1mol/l,Volume of electrolyte solution=25ml, time=24hr (equilibrium state), room temperature.

CONCLUSION

The following conclusion can be drawn from the study-
1. Resin is selective chelating ion-exchange resin for Fe^{3+}, Cu^{2+}, Ni^{2+}, Co^{2+}, Zn^{2+}, Cd^{2+} and Pb^{2+} metal ions.
2. This resin showed a higher selectivity for Fe^{3+}, Cu^{2+} and Ni^{2+} ions as compared to Zn^{2+}, Cd^{2+} and Pb^{2+} ions.
3. The resin showed higher selectivity for Fe^{3+} at pH 2.5 as compared to other metal ions.
4. The results of ion exchange study are helpful in selecting the optimum pH for a selective uptake of a particular metal ion from a mixture of a different metal ion.
5. Thus resin can be useful in water treatment and pollution control.

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