

Synthesis, Charecterization and Chelation Ion-Exchange Studies of a Resin Copolymer**Derived from 8-Hydroxyquinoline-Formaldehyde-pyrogallol****ABSTRACT**

A chelating ion exchange resin was synthesized from 8-hydroxyquinoline and pyrogallol using formaldehyde as a cross linking agent in DMF solution. The resin was characterized by FTIR and elemental analysis. The morphology of the synthesized resin was studied by optical photograph and scanning electron microscopy (SEM).

The physico-chemical properties of the resin have been studied. The total cation exchange capacity was measured and effect of pH and metal ion concentration on ion exchange capacity were studied. The rate of cation exchange reactions and distribution coefficient values in tartaric acid media at different pH were also studied using batch equilibration method.

Keywords: Chelating resin, Batch equilibration, Physico-chemical properties, Distribution coefficient

1. INTRODUCTION

In recent years, there has been a growing concern for the immobilization of metal ions introduced into water and wastewater bodies by increasing human technological activities. It has been established beyond doubt that heavy metal ions in the environment (water, soil and air) pose a very serious threat to human health. With the exponentially increasing population, there is a need for controlling heavy metal discharge before the toxic metal ions enter the complex ecosystem.

25 Separation, removal, and enrichment of trace metals in aqueous solutions play an important
 26 role in the analysis of wastewaters, industrial and geological samples. Solid phase extraction
 27 of metal ion has gained rapid acceptance because of its various advantages over other
 28 methods. The extraction of metal ions using chelating ion exchange resin is a green
 29 analytical method since it does not involve the use of toxic chlorinated organic solvents,
 30 which are very frequently used in conventional liquid–liquid extraction techniques. The main
 31 objective of most of the research on chelating resins was the preparation of insoluble
 32 functionalized polymers that can provide more flexible working conditions together with
 33 good stability and high capacity for metal ions.

34 The use of modified clay minerals for the adsorption of metal ions from aqueous solutions
 35 for water purification or industrial wastewater treatment has been widely studied. These clay
 36 minerals, when used as colloids or powders, have been found to be equally efficient as ion
 37 exchange resin but it is difficult to recover such adsorbents from filters after use. This further
 38 renders the regeneration and possible reuse of clay adsorbents very difficult.

39 Chelating ion exchange resins were also prepared by copolycondensation of 8-
 40 hydroxyquinoline or phenol derivatives like *o*-aminophenol, resorcylic acid or resorcinol
 41 with formaldehyde [1]. Antico *et al.* synthesized a gel type chelating ion exchange resin from
 42 glycol methacrylate with 8-hydroxyquinoline and used it to study the separation of Pb(II) and
 43 Cu(II) in their chloride solution [2]. The chelating ion exchange resins were also synthesized
 44 by Friedel-Craft's condensation of 8-hydroxyquinoline [3] and substituted 8-
 45 hydroxyquinoline [4] with 1,2-dichloroethylene. The synthesized resins were proved to be
 46 selective for certain metal ions over a wide pH range. The chelating behavior of poly (8-

hydroxyquinoline-5,7-dimethylene) [5] and its crosslinked polymer [6] derived from reaction with various amounts of bisphenol-A towards some trivalent lanthanide ions such as La(III) and Gd(III) has been investigated by a static batch equilibration method. Vernon *et al.* prepared oxine containing resins and studied their chelation properties towards transition metal ions [7,8]. From stability tests, they have suggested that the gel type polymers must never be allowed to dry; otherwise their advantageous properties are destroyed. Shah *et al.* [9,10] and Warshwsky *et al.* [11] reported some resins based on 8-hydroxyquinoline and substituted 8-hydroxyquinoline, respectively, along with their chelation properties towards transition and post transition metal ions.

So far no resin based on 8-hydroxyquinoline formaldehyde-pyrogallol in DMF medium has been reported for the quantitative removal and separation of transition and post transition metal ions. As industrial effluents are often rich in transition and post transition metal ions, removal of these metals is an important industrial task. The work described in the present communication deals with the synthesis, characterization and thermal studies of the aboveresin along with the systematic studies of various ion-exchange properties of

2. EXPERIMENTALS

2.1 Materials

8-Hydroxyquinoline (Prolabo, IGT Paris) and formaldehyde were purified by recrystallization method.

Pyrogallol (Glaxo extra pure) was purified by rectified spirit.

Metal ion solutions (KANTO CHEMICAL CO, INC.) were used as received.

2.2 Instruments

Infra-Red spectra of the synthesized resin sample have been carried out without solvent on a Thermo Scientific Nicolet spectrometer IR-200 FT-IR, which is the measurement accuracy

71 of 4 cm^{-1} in the area $400\text{-}4000\text{cm}^{-1}$.

72 The elemental analysis was carried out on a Carlo EDAX ZAF Quantification (Standard
73 less).

74 A pH meter (Elico, model CL-44) was used to measure the pH.

75 **2.3 Synthesis Method**

76 8-hydroxyquinoline (14.5 g, 0.1 mol) was ground to a fine powder and taken in a (250 ml)
77 round bottom flask and dissolved in DMF (25 ml) to give a clear yellow solution.
78 Formaldehyde (7.5 ml, 0.25 mol as 37%) was added and stirred until a red colored solution
79 was obtained. A solution of pyrogallol (11 g, 0.1 mol) in (10 ml) of DMF was added to
80 above solution and stirred for 3 h. Then the mixture was refluxed on a water bath at 90 ± 5
81 $^{\circ}\text{C}$ with constant stirring for 3-5 h and then on sand bath at $120 \pm 5\text{ }^{\circ}\text{C}$ for 7-8 h until a
82 viscous solution with the formation of a hard mass of brown color resin was obtained. The
83 synthesized resin was withdrawn from the reaction vessel and cured in an oven at $70\text{-}80\text{ }^{\circ}\text{C}$
84 for 12 h. The resulting resin was washed with DMF and with deionized water in order to
85 remove unreacted monomers as well as impurities. After the complete washing cycle, the
86 production yield of the resin synthesized reaction was found to be 50% (15 g).

87 The purified and dried resin sample was finely ground and sieved to obtained uniform
88 particle of 50-70 mesh size and stored in polyethylene bottle. This sieved resin was then
89 characterized using different instrumental analysis, techniques and was used for all of the
90 experiments during the research period. The solubility tests of resin in different solvent were
91 performed at room temperature and pressure with intermittent shaking. The resin was found
92 to be insoluble in all common organic solvents like acetone, ethanol, benzene, chloroform
93 *etc.* and all acids and alkalies of higher strengths.

94 The FTIR spectra with assignment of peaks are shown in Fig. 1.

95 The results of elemental analysis are in good agreement with calculated values of %C,
96 %H and %N, which is presented in Table 1.

97 To convert the resin sample into its H⁺ form, it was equilibrated with 1 M HCl solution for
98 24 h and washed with deionized water till it was free from chloride by testing with silver
99 nitrate solution. This H⁺ form of resin was used for further studies. The batch equilibrium
100 method was adopted to study the ion-exchange properties.

101 The physico-chemical properties like moisture and solid contents, apparent density, true
102 density and void volume fraction were studied according to the literature methods [12] and
103 the results are presented in Table 2.

104 The sorption properties such as rate of exchange, effect of pH on exchange capacity, effect of
105 the cation concentration on exchange capacity and distribution coefficient (K_d) values for
106 different metal ions as a function of pH and concentration of electrolyte were studied
107 according to the literature methods [13,14]. The value of void volume fraction was found to
108 be 0.36, while other reported resins. 8-hydroxyquinoline formaldehyde [8] and 8-
109 hydroxyquinoline formaldehyde resorcinol [9] exhibited void volume fraction values of 0.54
110 and 0.59, respectively.

111 **2.4 Rate of Exchange of Metal Ions**

112 For this experiment, an accurately weighed (0.250 ± 0.001 g) dry resin in the H⁺ form was
113 taken in different glass stoppered bottles and equilibrated with buffer solutions of desired pH
114 values for 24 h. After decanting the buffer solution, 50 ml (0.2 M) metal ion solution of the
115 same pH was added. The amount of unchelated metal ions was determined by Atomic
116 absorption spectroscopy (AAS) at fixed time intervals. The results are shown in Fig. 2.

117 **2.5 Effect of pH on Metal Ion Exchange Capacity**

118 To study the effect of pH on the metal ion uptake, it is necessary to buffer the resin and the
119 solutions used. To do this, buffer solutions of pH range 3-6 were prepared from 0.2 M acetic
120 acid and 0.2 M sodium acetate solutions [15].

121 Different sets of weighed (0.250 ± 0.001 g) dry resin were equilibrated with
122 buffer in different stoppered bottles for 24 h, so that resin attained desired pH value.

123 After 24 h, the buffer solutions were decanted and (50 ml) of 0.2 M metal ion solutions of
124 varying pH from 3-6 were added. Metal ion solutions were equilibrated at room temperature
125 for 24 h with intermittent shaking. After 24 h, the solutions were decanted and metal ion
126 concentration in the supernatant was measured by a method Atomic absorption spectroscopy
127 (AAS) which is a spectral procedure for the quantitative determination of chemical elements
128 employing the absorption of optical radiation (light) by free atoms in the gaseous state.

129 The same method was followed throughout the study to calculate the ion exchange capacity
130 of the resin as:

Exchange capacity

131

$$= \frac{[\text{Initial molarity of the metal ion}] - [\text{Remaining molarity of the metal ion}]}{\text{Atomic molar mass of the metal} * \text{Weight of the resin sample}}$$

132 **2.6 Effect of Metal Ion Concentration on Exchange Capacity**

133 To study the effect of metal ion concentration on uptake of different metal ions by the resin,
134 the resin was equilibrated with acetate buffer at desired pH values (pH value of highest
135 exchange) for 24 h and then buffer solutions were decanted. An accurately weighed ($0.250 \pm$
136 0.001 g) dry resin was equilibrated with metal ion solutions (50 ml) of varying mass
137 concentration (i.e., 1 ppm, 2 ppm, 3 ppm, 4 ppm and 5 ppm) at the same pH value at room

138 temperature for 24 h with intermittent shaking. After 24 h, the metal ion solutions were
139 decanted and unchelated metal ions were estimated by Atomic absorption spectroscopy
140 (AAS).

141 **2.7 K_d Values for Metal Ions in Presence of Electrolyte (Tartaric Acid) Solution**

142 Measurement of distribution coefficient of metal ions over a wide range of condition is a
143 good way to avoid choosing eluting conditions for column separations by a strictly trial and
144 error method. The batch distribution coefficient K_d is defined as:

$$145 \quad K_d = \frac{\text{mmole of metal ion on resin} * \text{volume of metal ion solution}}{\text{mmol of metal in solution} * \text{weight of dry resin}}$$

146 Although this distribution coefficient is measured on a batch basis, it can be used to predict
147 elution behavior of metal ions eluted from an ion exchange column. To separate two
148 substances, conditions should be selected such that the distribution coefficient of one of them
149 is low (preferably 1 or less) so that it elution from the column will be rapid, while the
150 distribution coefficient of the other substance under the same conditions should be as large as
151 possible (more than 10 times) so that this substance will be tightly held by the resin [16].

152 Effect of different concentrations and pH of electrolyte (tartaric acid) on metal ion uptake by
153 the synthesized resin was studied. An accurately weighed (0.250 ± 0.001 g) dry resin sample
154 was suspended in the 50 ml electrolyte solution of tartaric acid of different known
155 concentrations(i.e., 1 ppm, 2 ppm, 3 ppm, 4 ppm and 5 ppm). The pH of the suspension was
156 adjusted to the desired values using acetate buffer and the resin was equilibrated for 24 h. To
157 the suspension, 2.0 ml of solutions of different metal ions under study (5 mg.ml^{-1}) was
158 added and equilibrated for 24 h with intermittent shaking. After 24 h, the solutions were
159 decanted and unadsorbed metal ions were estimated. The results of K_d values are given in

160 Table 3.

161 3. RESULTS AND DISCUSSION

162 3.1 Spectral Characterization of Resin

163 The FTIR spectrum of the resin is shown in Fig. 1. A strong band at 3300 cm^{-1} is due to the
 164 $\nu(\text{O-H})$ stretching of phenolic group, a medium band at 3066 cm^{-1} is due to the $\nu(\text{C-H})$
 165 stretching of aromatic ring and the presence of a mediumstrong band at 2900 cm^{-1} is due to
 166 the $\nu(\text{C-H})$ stretching of methylene group. The bands at 1666 , 1585 and 1447 cm^{-1} can be
 167 assigned to $\nu(\text{C=N})$ heterocyclic ring and $\nu(\text{C=C})$ aromatic ring stretching, respectively. A
 168 band at 1442 cm^{-1} is also due to $\delta(\text{C-H})$ deformation of methylene group [9]. The presence of
 169 a medium-strong band at 1384 cm^{-1} can be assigned to in plane $\delta(\text{O-H})$ bending of aromatic
 170 and a band at 1285 cm^{-1} is due to aromatic $\nu(\text{C-O})$ stretching. A sharp single band at 781 cm^{-1}
 171 ¹, which can be assigned to the presence of 1,2,3,4,5-penta substituted benzene ring [10],
 172 confirms the polymerization of monomers.

173 3.2 Elemental Analysis

174 Theoretical percentage of carbon, nitrogen and hydrogen content of the resin were calculated
 175 from the general formula ($\text{C}_{17}\text{H}_{13}\text{NO}_4$) of the repeating unit of the probable structure
 176 (Scheme 1). Table 1 shows that the results of the elemental analysis are in good agreement
 177 with calculated values. The elemental analysis results are in support of the proposed structure
 178 of the resin presented in Scheme 1.

179 3.4 Optical and SEM Photographs

180 The morphology of the reported resin sample was investigated by scanning electron
 181 micrographs, which are shown in Fig. 2 and Fig.3, respectively. The morphology of the resin
 182 shows a fringed model of the, crystalline-amorphous structure. The fringes represent
 183 transition state between the crystalline and amorphous phase [17]. The resin exhibits more

184 amorphous character with closed packed surface having deep pits (Figs. 3a and 3b) as it
185 compared with anthranilic acid-formaldehyde-resorcinol resin reported earlier [17].

186 The physico-chemical properties of the synthesized resin are presented in table 2.

187 **3.5 Rate of Exchange for Metal Ions**

188 The exchange of metal ions on resin exhibits a time dependent phenomenon. The rate of
189 exchange for different metal ions was to ascertain the shortest time period for which
190 equilibrium could be carried out [18]. The graph shown in Fig.4 indicates that the time
191 required for 50% exchange ($t_{1/2}$) for plomb(II) is 30 min and for copper(II) and zinc(II) are 50
192 and 56 min, respectively. The fast rate of exchange in the beginning can be explained on the
193 basis of law mass action. The faster rate of exchange facilitates column chromatographic
194 separation. Kinetics of metal ion exchange mainly depends on various physical properties
195 including particle size distribution, pore size, physical core structure and diffusion of counter
196 ion [12,13].

197 **3.6 Effect of pH on Exchange Capacity**

198 The removal of metal ions from aqueous solution by sorption is highly dependent on pH of
199 the solution which affects the surface charge of the sorbent [18]. Chelating ligands form
200 complexes with various metal ions at specific pH conditions [19]. Therefore, the synthesized
201 resin was used to study the effect of variation in pH on its chelating ability towards various
202 metal ions. The results of the exchange capacity vs. pH for different metal ions are presented
203 in Fig. 5. The results show that sorption of metal ions are increased with increasing in pH up
204 to a maximum value and thereafter decreased. The maximum sorption of Zn(II) took place at
205 pH 4, Cu(II) at pH 6 and Pb(II) at pH 4. The selectivity order for metal ions is Pb(II) > Zn(II)
206 > Cu(II).

207 An increase in pH increases the negatively charged nature of the sorbent surface. This leads

208 to an increase in the electrostatic attraction between positively charged metal ions and
 209 negatively charged sorbent and results in increased sorption of metal ions. At lower pH, the
 210 removal of metal ions is decreased due to the higher concentration of H^+ ions present in the
 211 reaction mixture which compete with the metal ions for the sorption sites at the surface.
 212 Meanwhile, the observed decrease in sorption capacity is due to the formation of insoluble
 213 metal ion hydroxides [20].

214 **3.7 Effect of Metal Ion Concentration on Exchange Capacity**

215 The examination of data presented in Fig. 6 reveals that the amount of adsorbed metal ion
 216 increases with increasing concentration of metal ions in solution until a maximum value, and
 217 will remain constant upon further increase in metal concentration. At lower concentration of
 218 metal ions, the number of metal ions available in solution is low as compared to the available
 219 sites on the sorbent [20]. However, at higher concentrations the available sites of sorption
 220 remain same whereas more metal ions are available for sorption and subsequently the
 221 sorption becomes almost constant then after [9,10].

222 **3.8 Effect of Electrolyte Concentration and pH on Distribution Coefficient (K_d) Values**

223 The distribution coefficient values (K_d) for different metal ions were determined by the batch
 224 equilibration method. The K_d values of the metal ions were studied as a function of pH
 225 and concentration of electrolyte solution and the results are presented in Table 3. The present
 226 investigation limits the distribution studies up to a certain pH for each metal ion to prevent
 227 the hydrolysis of metal ions at higher pH [9]. As is obvious from Table 3, at all electrolyte
 228 concentrations and entire pH range, the K_d values decrease in the order $Pb(II) > Zn(II) >$
 229 $Cu(II)$. Meanwhile, in all cases, the K_d values decrease with increasing electrolyte
 230 concentration.

231 Obviously, to achieve more clear separation of metal ions, large ΔK_d values should be

232 selected on the same experimental conditions [21].

233 4. CONCLUSIONS

234 The present work has been focused on the preparation, characterization and chelating
 235 properties of a Resin Copolymer Derived from 8-Hydroxyquinoline-Formaldehyde-
 236 Pyrogallol. The chelating properties of this resin are influenced by the medium, particularly
 237 the pH of the solution and the nature and concentration of counter ion used. In the present
 238 study, it can be concluded that the resin is an effective material for the preconcentration and
 239 separation of trace metal ions.

240 The crystals are smaller in surface area with more closely packed structures with no visible
 241 pits. This could be the reason of low ion-exchange capacity of higher hydrated size of metal
 242 ions (lead ion).

243 The synthesized resin shows lower moisture content compared to other reported resins
 244 [9,10], indicating the high degree of cross-linking in this resin. Void volume fraction of
 245 resin is relatively lower than other reported resins [8,9] causing a lower exchange rate.
 246 It is similar to that reported resins derived from salicylic acid-furfural-hexamine [22] and
 247 salicylic acidacetaldehyde- thiourea [23].

248 From the results of exchange rate studies, it was found that copper(II) takes 18 h to attain
 249 equilibrium, while it takes 21 h for other metal ions. General trend of chelating ability of
 250 resin is $\text{Pb(II)} > \text{Cu(II)} > \text{Zn(II)}$. The K_d determinations revealed that there is a considerable
 251 difference between the distribution coefficients of metal ions at optimum condition, which
 252 can be used in the successful separation of heavy metal ions.

253 The presence of heavy metal ions viz Pb(II) and Cu(II) in aquatic systems pose heavy risks to
 254 human health. Therefore, removal of such metal ions from water bodies may be considered

255 as an interesting and important research activity.

256

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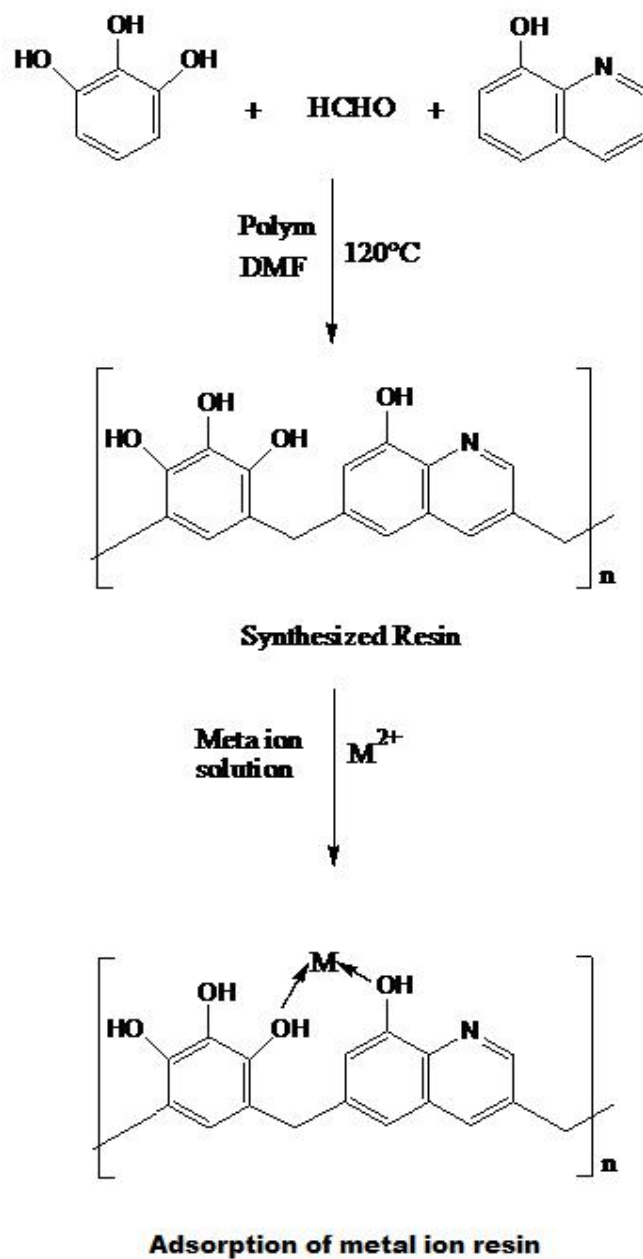
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Scheme 1

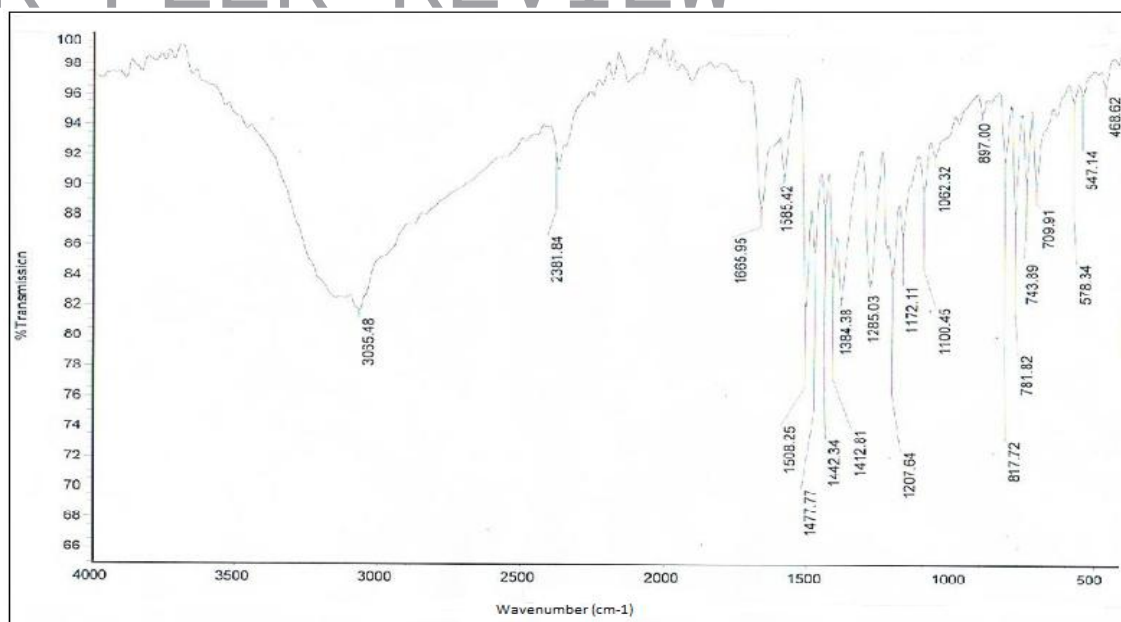


Fig. 1. FTIR spectrum of resin

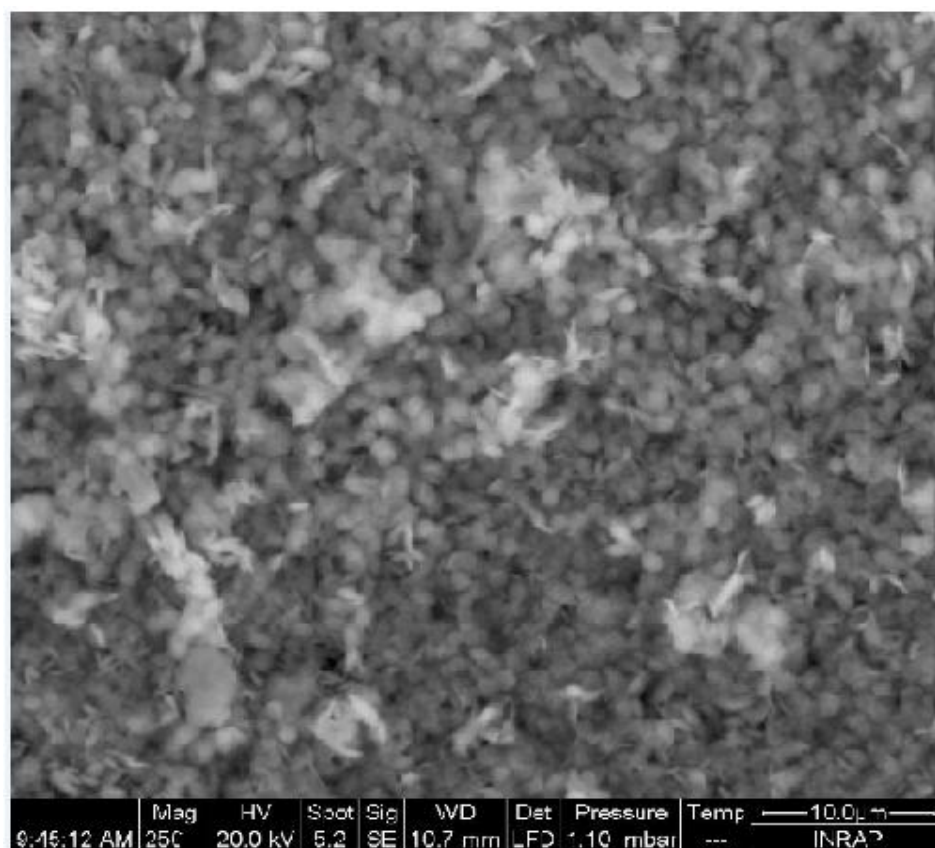


Fig. 2. Optical photograph of resin 250X Magnifications

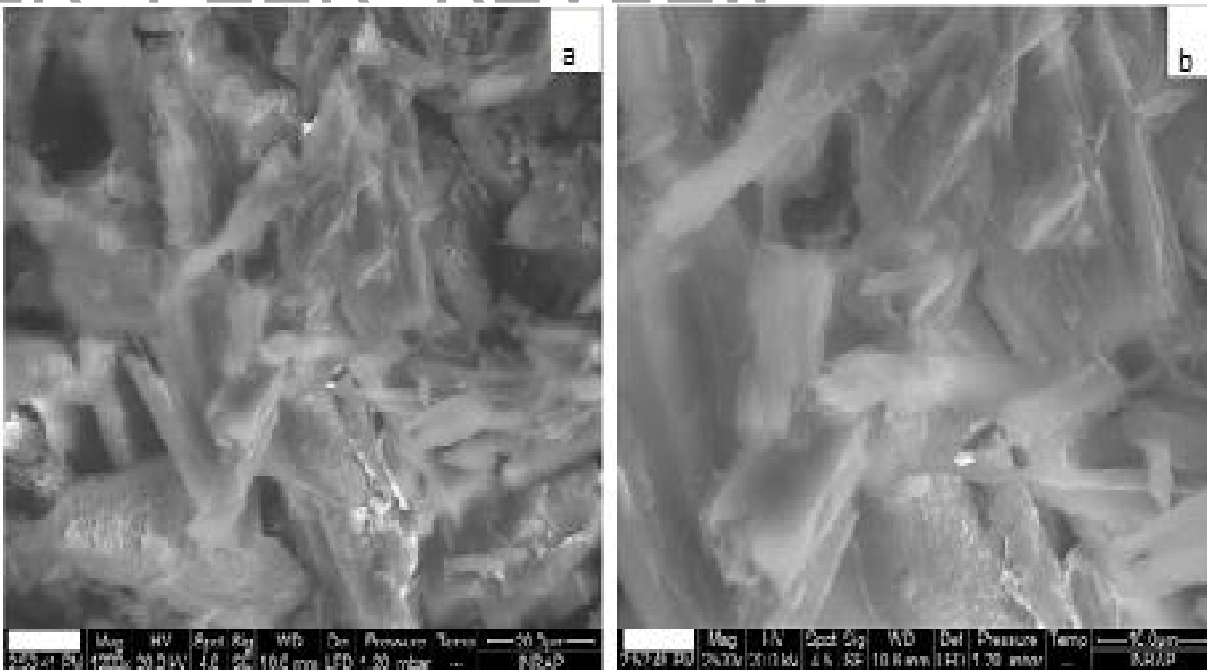


Fig. 3. SEM photograph of resin at (a) 1200X and (b) 2500X Magnifications

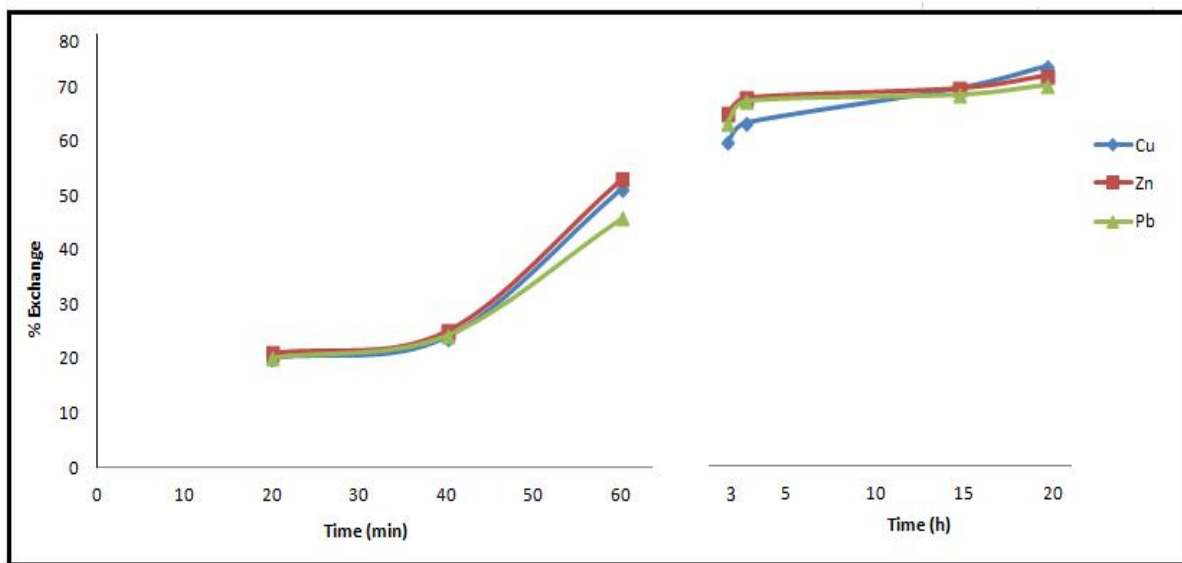


Fig. 4. Rate of exchange of cations on resin

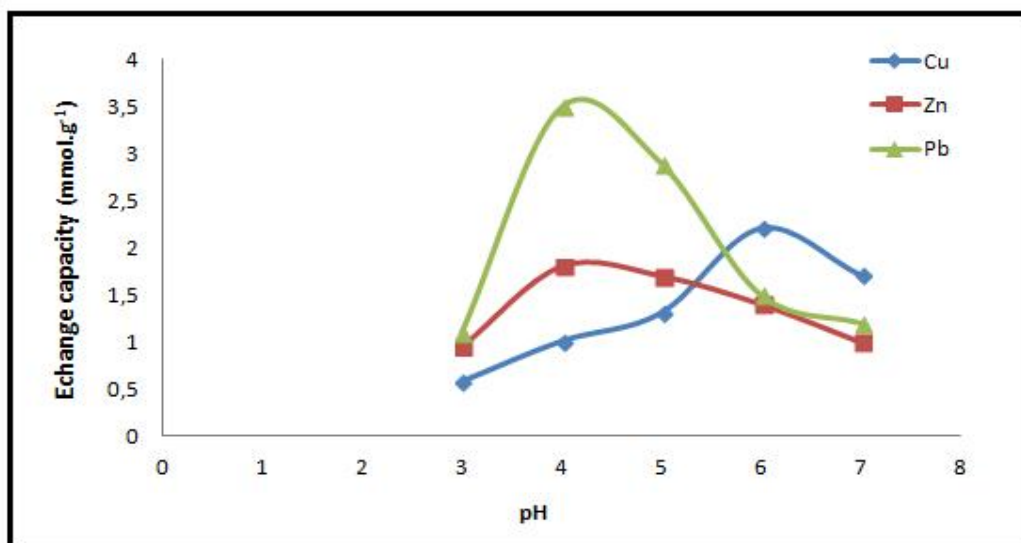


Fig. 5. Effect of pH on cation exchange capacity

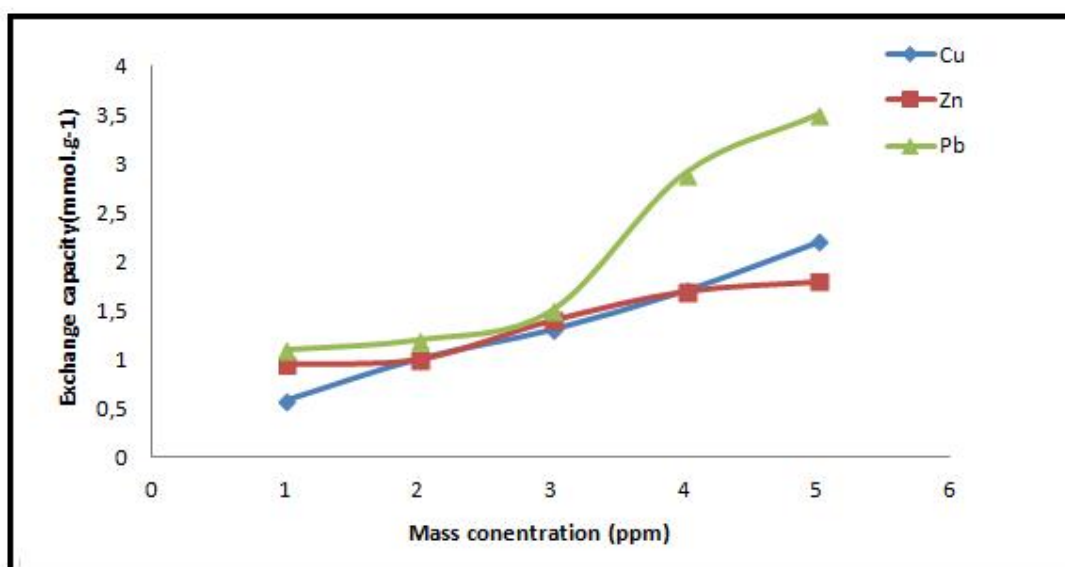


Fig. 6. Effect of cation concentration on exchange capacity

Table1. Element Analysis of the Resin

% Calculated (% Found)		
C	H	N
69.15	4.04	4.74

68.15 3.98 4.65

336

337

338 **Table 2.** Physicochemical Properties of the synthesized Resin

Property	Values (S.D)
% Moisture content	5.25 (±0.05)
% Solid	93 (±0.05)
True density	0.95 (±0.01) g.cm ⁻³
Apparent density	0.51 (±0.01) g.cm ⁻³
Void volume fraction	0.38 (±0.025)

339

340 **Table 3.** K_d Values of Metal Ions at Various Tartaric Acid Concentrations and pH Values

Metal	[Tarttric acid]	K _d values at different pH				
ion	(M)					
		3	3.5	4	5	6
	0.1	1101	670.8	527.1	201.7	155.2
	0.2	698.3	320.2	129.9	110	79.7
Cu(II)	0.3	250.1	105.5	80.5	50.8	38.9

	0.5	55.6	55.6	39.7	52.3	55
	1	14	19.3	11.8	8.4	6.5
	0.1	1800	1320	432.4	401.9	200
	0.2	640.7	617.8	220.1	110.9	130.5
Zn(II)	0.3	588.2	300.4	95	71.8	59
	0.5	134	115.1	74	28.1	36.8
	1	81	35.5	25.2	11.8	5.3
	0.1	2221.3	980.4	610.3	420.9	261.2
	0.2	668.4	640.2	230.7	116	142.3
Pb(II)	0.3	435.9	340.7	95.3	82.2	30.4
	0.5	130.2	82.5	80	40.3	4.7
	1	10.8	45.4	34.9	17	2.8

341