Synthesis of crosslinked methacrylic acid-co-ethylene glycol dimethacrylate polymers for the removal of copper and nickel from water

H Hari Prasad, Kirit M Popat & Pritpal Singh Anand

Separation Technology Discipline, Central Salt & Marine Chemicals Research Institute, Bhavnagar 364 002, India

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Crosslinked copolymers based on methacrylic acid and ethylene glycol dimethacrylate were prepared by suspension polymerization and investigated for their ability to remove copper and nickel at trace level from heavy metal polluted water. The copolymers were characterized for physico-chemical properties like surface area, porosity and scientific weight capacity. The Langmuir and Freundlich adsorption isotherms were used to represent the experimental data. Saphn-Schlunder and Morris-Webber models applied to determine the external and intraparticle diffusion coefficients. The absorption process follows first order kinetics and the specific rate constant K was obtained by the application of Lagergan equation. Concentration ratios of 1:60 and 1:30 have been achieved for nickel and copper respectively under dynamic conditions.

In recent years interest has been focused on the removal of heavy metal ions from contaminated or waste water effluents, due to their well-known toxicity. When they are discharged in small quantities by various industrial activities into the environment, there is significant contamination of fresh water sources resulting in accumulation of toxic metals in the human food chain. For these reasons WHO (World Health Organization) has prescribed the maximum concentration level of heavy metals like copper and nickel in drinking water as 1.3 mg/L and 0.1 mg/L for copper and nickel respectively. Therefore, special attention has been given by various investigators to develop methods for water purification based on low cost materials like chitosan, fly-ash, peat, biomass, coirpith, bituminous coal, seed and seed shell of magisterandica, congo red attached to poly (ethylene glycol dimethacrylate-hydroxymethacrylate), supported liquid membranes, iminodiacetic acid and thio urea based resins. Recently the removal of various metal ions such as copper and nickel by using poly methacrylate micro beads carrying ethylenediamine, chelate polymers and ion exchange resins, polymer immobilized rhizpone oryzae, low grade phosphate mineral surface and saw dust, etc., has been reported.

All materials barring a few mentioned above are either inorganic materials or carbon compounds which are mostly used in granular form and are difficult to reuse. Liquid membranes need very cumbersome separation techniques, whereas weakly acidic sodium form of carboxylic acid type cation exchangers are quite appropriate for the removal of metal ions from dilute solutions. It is well established that the metal ions like M(II) form complex with carboxylic groups at pH 4 to 5.

$$2\text{RCOO}^+ + \text{M}^{++} \rightarrow [\text{RCOO}]_2\text{M}^{++} + 2\text{Na}^+$$

The exhausted ion exchanger bed can be regenerated by dilute mineral acids resulting into eluate which can be directly utilized for various applications after minor concentration make up. The material can be regenerated several times and gainfully reused. There is no pressure drop during the column operation as bead shape of resin allows easy passage of process liquors. The operation is eco-friendly as compared to similar sorption processes using inorganic materials.

This paper describes the synthesis and characterization of methacrylic acid-co-ethylene glycol dimethacrylate sorbents and their application for the removal of nickel and copper ions from waste water.

Experimental Procedure

Materials

Methacrylic acid and ethylene glycol dimethacrylate were used after distillation under vacuum, n-heptane, starch, sodium chloride, toluene,
cyclohexane, methanol, nickel ammonium sulfate and cupric chloride were used as received. Benzoyl peroxide was recrystallised from methanol-chloroform mixture.

**Synthesis of porous copolymer**

Crosslinked copolymers were synthesized by suspension polymerization method\(^{16}\). A suspension medium composed of saturated sodium chloride solution containing 2% (w/v) starch as a suspension stabilizer was taken in a 3 neck round bottom flask equipped with an anchor type stirrer, thermometer and reflux condenser. Suspension medium was heated up to 90°C and brought down to 80°C, a mixture of methacrylic acid, ethylene glycol dimethacrylate, n-heptane and benzoyl peroxide (1% of monomer mixture) was added in required proportions. The ratio of the reaction mixture (monomer plus diluent) to suspension medium was kept at 1:4 (w/v). Copolymerization was initially carried out at 80°C for 2h and then at 100°C for 2h. The copolymer beads thus obtained were washed with hot water several times to remove any adhering stabilizer and then air dried for 16h. They were extracted in a soxhelet apparatus with acetone to remove any unreacted monomers and other trapped porogenic agents etc., and air-dried. A sieved fraction between -18+52 BSS mesh size (0.3 -1 mm) was selected for further studies. Following this procedure polymeric sorbents having varying degrees of crosslinking and \(F_m\) value \([F_m\) value is the ratio of the total quantity of the monomer mixture to the total quantity of the monomers plus diluent (w/v)] were synthesized. The polymeric sorbents in bead shape thus synthesized were designated as follows:

(i) APR\(_r\)-5-0.7-nH  
(ii) APR\(_r\)-5-0.8-nH  
(iii) APR\(_r\)-5-0.9-nH  
(iv) APR\(_r\)-5-1.0 (GEL)  
(v) APR\(_r\)-10-0.7-nH  
(vi) APR\(_r\)-10-0.8-nH

In this nomenclature APR\(_r\) stands for methacrylic acid based polymer, the first numerical indicates the percentage of crosslinking monomer, the second numerical indicates the \(F_m\) value and nH stands for the diluent n-heptane. The letters GEL represent conventional gel type matrix synthesized in the absence of diluent. The copolymers were evaluated for porosity, pore volume, solvent uptake, apparent density, ion exchange capacity and surface area by \(p\)-nitrophenol adsorption method\(^{17}\) and the results were tabulated in Tables 1 and 2. The effect of various parameters such as \(pH\) of equilibrating solution, dissolved salt effects in equilibrating solution, period of equilibration and column operations on the sorption of metal ions was studied for APR\(_r\)-10-0.8-nH because, it has shown good ion exchange capacity, high metal uptake and physical stability compared to other resins synthesized.

**Spectral Characterization**

FT-IR spectra were obtained on IR spectrophotometer as KBr pellet on Perkin-Elmer FT-IR spectrophotometer.

**Procedure for Cu(II) and Ni(II) sorption**

Cu (II) and Ni (II) solutions were prepared in de-mineralized water by cupric chloride and nickel ammonium sulphate. Static equilibrium studies were carried out by equilibrating separately 50 mL of metal ion solutions of pre-decided concentrations (0.01 to 0.1 M) with about 0.2 g of sorbent in Na\(^+\) form with occasional stirring at room temperature (30°C) for 16h. After equilibration, solutions were filtered and metal ion concentrations were estimated by EDTA (ethylene diamine tetra-acetic acid)\(^{18}\). The data on the uptake of metal ions by the sorbents are presented in Tables 3 and 4. The same procedure was followed for further estimations in determining the effect of different parameters such as \(pH\) of equilibrating solution.

**Table 1—Preparation conditions of methacrylic acid (MAA)-ethylene glycol dimethacrylate (EGDMA) copolymer**

<table>
<thead>
<tr>
<th>Polymer matrix</th>
<th>MAA (g)</th>
<th>EGDMA (g)</th>
<th>n-Heptane (g)</th>
<th>% of Crosslinking</th>
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Crosslinked methacrylic acid-co-ethylene glycol dimethacrylate polymers for Cu and Ni removal

Table 2—Characterization of methacrylic acid-ethylene glycol dimethacrylate copolymers

| Polymer Mat. | IEC Meq/g | MR % | AD g/ml | Po m/l | Pm m/l | SU (m l/g) Toluene Cy. hex (m²/g) SA |
|--------------|-----------|------|----------|--------|--------|----------------|-------------------|
| 1            | 10.86     | 79.73| 0.4546   | 0.3113 | 0.6848 | 1.3358        | 1.925             | 97.70             |
| 2            | 10.89     | 71.59| 0.4546   | 0.3113 | 0.6848 | 1.1752        | 1.1434            | —                 |
| 3            | 10.68     | 61.37| 0.5405   | 0.1812 | 0.3352 | 0.3136        | 0.1820            | 75.80             |
| 4            | 9.44      | 53.82| 0.6601   | —      | —      | —             | —                 | 47.40             |
| 5            | 9.19      | 68.43| 0.3352   | 0.4441 | 1.2832 | 1.8878        | 2.4690            | 142.20            |
| 6            | 8.90      | 62.76| 0.3871   | 0.4301 | 0.8832 | 1.3820        | 1.4162            | 116.90            |
| 7            | 8.47      | 63.66| 0.3268   | 0.3419 | 1.3334 | 1.1328        | 1.2649            | 113.80            |
| 8            | 7.90      | 46.47| 0.5882   | —      | —      | 0.0178        | 0.2214            | 50.60             |
| 9            | 8.34      | 72.13| 0.3196   | 0.4896 | 1.5289 | 1.7934        | 1.8442            | 284.20            |
| 10           | 8.55      | 70.96| 0.3615   | 0.4546 | 1.1663 | 1.0642        | 1.1243            | 255.90            |
| 11           | 8.29      | 58.85| 0.3409   | 0.4216 | 1.3599 | 0.7712        | 0.7910            | 211.70            |
| 12           | 2.37      | 18.59| 0.6250   | —      | —      | —             | —                 | 75.80             |

1: MAA-EGDMA-5-0.7-nH; 2: MAA-EGDMA-5-0.8-nH; 3: MAA-EGDMA-5-0.9-nH; 4: MAA-EGDMA-5-1.0 (GEL); 5: MAA-EGDMA-10-0.7-nH; 6: MAA-EGDMA-10-0.8-nH; 7: MAA-EGDMA-10-0.9-nH; 8: MAA-EGDMA-10-1.0 (GEL); 9: MAA-EGDMA-15-0.7-nH; 10: MAA-EGDMA-15-0.8-nH; 11: MAA-EGDMA-15-0.9-nH; 12: MAA-EGDMA-15-1.0 (GEL)

Polymer Matrix: Polymer Matrix; IEC: ion-exchange capacity; MR: moisture retention; AD: apparent density; Po: porosity; Pm: pore volume, SU: solvent uptake; SA: surface area; Cy. hex: cyclohexane.

Table 3—Effect of metal ion concentration of nickel in solution on its uptake (mg/g)

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<th>Polymer Matrix</th>
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Solution, concentration of metal ion in solution, period of equilibration and dissolved salts on the uptake of metal ion. The uptake of Ni(II) and Cu(II) was also studied under dynamic conditions by percolating 100 ppm of spiked metal ion solutions through the resin bed at different service flow rate, till effluent showed leakage of metal ion of 1 mg/L. Passing 1 N hydrochloric acid for nickel and a mixture of 2 N sulphuric acid and 2% sodium chloride for copper eluted the adsorbed metal ions from the sorbent.

Results and Discussion

Polymer synthesis

The crosslinked copolymers of methacrylic acid and ethylene glycol dimethacrylate were synthesized by suspension polymerization in presence of benzoyl peroxide as an initiator under different experimental conditions. The chemical structure of the copolymer is shown in Fig. 1.

Characterization

The experimental data presented in Table 1 and 2 show the characteristics of sorbent such as porosity, surface area, and toluene and cyclohexane uptake. The uptake of toluene and cyclohexane decreases with increase in Fₘ value at particular crosslinking density which indicates decrease in porosity. With increase in degree of crosslinking porosity increases at constant Fₘ value. This is experimentally supported by the uptake of toluene and cyclohexane. At constant Fₘ
value, uptake of toluene and cyclohexane increases with degree of crosslinking up to a certain value and then decreases at higher crosslinking because of tight micro porous structure. For example, the uptake of toluene were 1.1752, 1.382 and 1.0642 mL/g at 5, 10 and 15% degree of crosslinking, at \( F_{m} \) value 0.8. The same trend was observed in earlier studies with porous polymers.\(^{19,20}\) Surface area measurements by \( p \)-nitro phenol adsorption shows that there is considerable increase in surface area in the case of 15% cross-linked polymers than those of 5 and 10% cross-linked sorbents. At higher crosslinking, surface area is contributed by the intermolecular surfaces as well.

### Spectral characterization

IR spectra of copolymer and metal complexes are shown in Fig. 2. In Fig. 2(A) the broad band around 3450 cm\(^{-1}\) is attributed to COOH stretching. A strong peak at 1718 cm\(^{-1}\) is due to carbonyl stretching of carboxylic acid and C=O present on the ethylene glycol dimethacrylate. The bands at 1481 and 1394 cm\(^{-1}\) can be attributed to the stretching of C-O and O-H of the carboxylic group respectively.

Figure 2 (B) & (C) show the complexed spectra of the metal ions with sorbent after equilibration. In the spectra of metal complexed sorbents the band due to OH group of carboxylic acid is observed at around 3426 cm\(^{-1}\) and the band due to carbonyl group of carboxylic acid and ethylene glycol dimethacrylate have been shifted towards lower frequency range by about 9 cm\(^{-1}\) due to complexation of metal ions.

### Effect of metal ion concentration on uptake

The experimental data presented in Tables 3 and 4 show the effect of nickel and copper ion concentrations in solution on its uptake by the sorbent. The data reveal that the amount of adsorbed metal ion increased with increase in concentration of metal ion up to a particular concentration and then become constant. At lower concentration of metal ions the number of metal ions available in solution are
less as compared to the available sites on the sorbent. However, at higher concentrations the available sites for sorption are remaining the same, whereas more metal ions are available for sorption and subsequently the sorption becomes almost constant.

**Sorption isotherms**

An equilibrium is described by usual isotherm equations such as Langmuir and Freundlich isotherms. A preliminary screening of the corresponding equations has shown that the Langmuir and Freundlich models best fit the experimental data and the following equations are used for the modeling of results.

Langmuir model: \( \frac{C_e}{Q_e} = \left( \frac{1}{Q_m b} \right) + \left( \frac{C_e}{Q_m} \right) \)

where \( C_e \) is the equilibrium concentration (mg/L), \( Q_e \) is the amount adsorbed at equilibrium (mg/g), and \( Q_m \) and \( b \) are Langmuir constants related to adsorption capacity and energy of adsorption respectively. The linear plot (Fig. 3) of \( C_e/Q_e \) versus \( C_e \) shows that adsorption obeys Langmuir model. These constants were calculated as 416.67 and 588.24 mg/g as maximum sorption capacities for copper and nickel respectively. 0.00018 L/mg for nickel, and 0.00012 L/mg for copper as sorption energy constants.

This adsorption was also confirmed by Freundlich model

\[ \log q_e = \log k_f + \left( \frac{1}{n} \right) \log C_e \]

where \( C_e \) is the equilibrium concentration (mg/L) and \( q_e \) is the amount adsorbed (mg/g). A linear Plot (Fig. 4) of \( \log q_e \) versus \( \log C_e \) shows the applicability of Freundlich isotherm. Freundlich constants \( K_f \) and \( 1/n \) were calculated as 0.3441 and 0.7483 for Ni(II) and 0.3066 and 0.7729 for Cu(II) ions. Values of \( 1/n<10 \) show the positive sorption of metal ions on the sorbent.

The specific rate constant, \( K_s \), for the sorbent was determined by Lagergran equation

\[ \log(q_e-q) = \log q_e - (K_s \times t)/2.303 \]

where \( q_e \) and \( q \) (mg/g) are the amounts of metal ion adsorbed at equilibrium and at time \( t \), respectively. The straight-line plot (Fig. 5) of \( \log(q_e-q) \) versus
time, at 30°C indicates the validity of the Lagergran equation for the system and explains that the process follows first order kinetics. The values of $K_i$ were calculated from the slope of the plot and found to be $11.3 \times 10^{-3}$/min and $9.9 \times 10^{-3}$/min for Ni(II) and Cu(II) respectively.

**Diffusion models**

Sorption kinetics are mainly controlled by various factors including (i) solute transfer from the solution to the boundary film surrounding the particle (bulk diffusion), (ii) diffusion from the film to the surface of the sorbent (external diffusion), (iii) diffusion from the surface to the intraparticle sites (intraparticle diffusion), (iv) solute adsorption by complexation or physico-chemical sorption or ion exchange. By providing sufficient agitation to avoid particle and solute gradients in the batch reactor makes it possible to ignore bulk diffusion. The process of external diffusion and intraparticle diffusion are the rate controlling steps and several models have been described. In the present work Spahn and Schlunder model$^{23}$ and Morris and Weber model$^{24}$ were chosen to describe the external diffusion and intraparticle diffusion on the sorbent.

**Spahn and Schlunder model**

\[
\ln \left( \frac{C_t}{C_0} \right) = -k_e (A/V) \times t
\]

where $C_t$ and $C_0$ are the solution concentrations (mg/L) at time $t = 0$ and at different time intervals respectively; $t$ is the time (s); $k_e$ represents the external mass transfer coefficient (m/s) and $V$ is the volume of equilibrating solution (mL) and $A$ indicates surface area. The external diffusion coefficients were calculated for Cu(II) and Ni(II) as 0.00781 (m/s) and 0.009985 (m/s) respectively.

**Morris and Weber model**

\[
C_t - C_i = K_w \times t^{1/2}
\]

where $K_w$ represents the initial rate of intraparticle diffusion coefficient (mg/L s$^{-1/2}$) and were calculated for Cu(II) and Ni(II) as 1.38 and 5.18 (mg/L s$^{-1/2}$) respectively. From these external and intraparticle diffusion coefficients, it can be concluded that the diffusion process is mainly controlled by intraparticle diffusion under agitation conditions.
**Effect of pH on sorption of metal ions**

The aqueous chemistry of metal ions is highly dependent on the pH of the system, which affects the surface charge on the sorbent. The experiments with solution pH as variable were run to determine the optimum pH range at which maximum sorption could take place and the results are shown graphically in Fig. 6. Metal ion sorption increased with increase in pH of the system up to a certain range and then decreased. The maximum sorption took place around pH 7.89 for nickel and copper ions respectively. The presence of C=O group also plays a role in the sorption mechanism due to the polar attraction between the positively charged metal ions and polarized carbonyl group. An increase in pH increases the negatively charged nature of the sorbent surface and that leads to an increase in the electrostatic attraction between positive substrate and negatively charged sorbent. The decrease in sorption of metal ions at lower pH is mainly due to the competition between the high concentration of H⁺ and metal ions for the sorption sites on the sorbent. Decrease in sorption at alkaline pH range is apparently due to the formation of insoluble metal hydroxide complexes.

**Effect of dissolved salt concentration**

Figure 7 (A) & (B) shows the effect of dissolved salts such as NaCl, Na₂SO₄, NaHCO₃, on the sorption of metal ions. At lower concentration of these salts there is no considerable decrease in sorption, but at higher concentration, uptake decreased. This indicates that chemisorption and ion exchange takes place simultaneously. In the presence of Ca²⁺ and Mg²⁺, uptake is lower because of the competition of these ions for the sites present on the sorbent. In the case of HCO₃⁻ ions uptake of nickel increased slightly up to a certain concentration of bicarbonate ions and then decreased due to the change in pH from 4.43 to 6.89. In the presence of HCO₃⁻ ions copper is precipitated as hydroxide complex.

**Column operations**

The dynamic performance of the sorbent was tested by running the column operations by taking 26 cm³ resin and an influent with 100 ppm concentration of metal ions. The sharp movement of boundary was observed with progressive exhaustion of bed. The capacity of sorbent for 1-ppm leakage of metal ions at different influent flow rates such as 5, 10 and 15 B.V/h were determined. As the flow rate increases from 5 to 10 B.V/h the capacity of sorbent bed to treat the metal ion solution was decreased from 237.81 to 140.39 B.V for nickel and 219.78 to 176.47 B.V for copper. This can be attributed that, as the flow rate increases the sorption of metal ion decrease due to the lesser contact time with the sorbent.

**Desorption**

The exhausted beds were stripped off by passing 1N HCl and a mixture of 2N H₂SO₄ + 2% NaCl for Ni(II) and Cu(II) respectively. In the case of nickel, metal ions were eluted with 75 mL of eluting agent.
and 100 mL of eluting agent was used for the copper ions at 2 B.V/h eluting flow rate.

Conclusions
A simple treatment of wastewater contaminated with heavy metal ions like copper and nickel was carried out with methacrylic acid and ethylene glycol dimethacrylate copolymer as a sorbent. The influence of the pH and dissolved salt concentrations on sorption performance was discussed. The maximum sorption capacities for copper and nickel ions were obtained as 588.24 and 416.67 mg/g respectively and rate constant, sorption energy, external diffusion coefficient and intraparticle diffusion coefficients were obtained by the application of different models. Under column run experimental conditions it was observed that at lower flow rates the sorbent could treat more bed volumes of metal ion solutions than at higher flow rates. The loaded metal ions could be eluted with mineral acids such as 1N HCl and a mixture of 2N H$_2$SO$_4$ + 2% NaCl for nickel and copper ions respectively, and the sorbent can be reused for further cycles.

References: