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A New Cationic Poly[1-vinyl-3-ethyl imidazolium iodide], P(VEII) Hydrogel for the Effective Removal of Chromium (VI) from Aqueous Solution

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A New Cationic Poly[1-vinyl-3-ethyl imidazolium iodide], P(VEII) Hydrogel for the Effective Removal of Chromium (VI) from Aqueous Solution

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Cationic porous and non-porous hydrogels have been prepared via free radical solution polymerization using newly synthesized 1-vinyl-3-ethyl imidazolium iodide (VEII) monomer and N,N′-methylenebisacrylamide as crosslinker. The hydrogel has been characterized by FT-IR, mercury porosimetery and TGA analysis. Their surface morphology was observed using scanning electron microscope (SEM). The swelling kinetics of porous and non-porous P(VEII) hydrogels in solutions of chromium(VI) was determined using a gravimetric method. A batch system was applied to study the adsorption of chromium(VI) from aqueous solutions by cationic hydrogels. The effect of treatment time, pH of the medium, amount of adsorbent dose and initial feed concentration of metal ion on adsorption of chromium(VI) from their solution were also investigated. Adsorption of chromium(VI) increases with the increase in treatment time, adsorbent dose and initial feed concentration and decreases with the increase in pH of the medium. Desorption of chromium(VI) was carried out using various eluents and almost 100% desorption was observed, so the P[VEII] hydrogel can be used repeatedly for the adsorption of chromium(VI).

Keywords: Cationic hydrogels, metal removal, adsorption, batch equilibrium, chromium(VI)

1 Introduction

Hydrogels are water-swollen networks (crosslinked structure) of hydrophilic homo or copolymers. They acquire great interest due to the facility of the incorporation of different chelating groups into the polymeric networks. Recently, it has been reported that chelating hydrogels having specific functional groups can be applied to the recovery of precious metal, removal of toxic or radioactive elements from various effluents and to metal preconcentration for environmental sample analysis (1–6). Metals are released into the environment by a large number of processes such as electroplating, leather tanning, wood preservation, pulp processing, steel manufacturing, etc., and the concentration levels of metals in the environment widely varies. Among these, the electroplating industry is one of the most hazardous chemical-intensive industries (7).The removal of toxic and polluting metal ions from industrial effluents, water supplies, as well as mine waters, is an important challenge to avoid one of the major causes of water and soil pollution and it has received much attention in recent years (8–12).

Inorganic effluents from the industries contains toxic metals such as Cd, Cr, Cu, Ni and Zn (13), which tend to accumulate in the food chain. Because of their high solubility in the aquatic environments, heavy metals can be absorbed by living organisms. Once they enter the food chain, large concentrations of heavy metals may accumulate in the human body. If the metals are ingested beyond the permitted concentration, they can cause serious health disorders (14).

The removal of Cu, Ni and Cr are of major concern because of their larger usage in developing countries and their non-degradability nature. Hexavalent chromium is highly soluble in water and carcinogenic to humans. Different treatment techniques for wastewater laden with heavy metals have been developed in recent years both to decrease the amount of wastewater produced and to improve the quality of the treated effluent. Although various treatments, such as chemical precipitation (15–16), coagulation–flocculation (17), flotation (18), ion exchange (19–21) and membrane filtration can be employed to remove heavy metals from contaminated wastewater, they have their inherent advantages and limitations in application.

Recently, hydrogels based on N-vinylimidazole has been used for the adsorption of some toxic metals (22–23). In

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the present study, we introduce a new cationic poly[1-vinyl-3-ethyl imidazolium iodide], porous P(VEII) hydrogel for the effective removal of chromium (VI) from aqueous solutions.

2 Experimental

2.1 Chemicals

1-Vinylimidazole monomer and initiator 2,2′-azobis(2-methylpropionamidine)dihydrochloride (V-50) were supplied by Aldrich (Steinheim, Germany). The crosslinker N,N′-methylenebisacrylamide (MBAm) and ethyl iodide were from Merck (Darmstad,Germany). Acetone was from S. D. Fine Chemicals (Mumbai, India). Double distilled water was used in hydrogel synthesis and distilled water was used for the metal removal studies.

2.2 Instrument

Bruker Avance II 400MHz NMR spectrophotometer was used to record the ^{1}H and ^{13}C-NMR spectra using DMSO-$d_6$as solvent. IR spectra were recorded on a Perkin-Elmer spectrum GX FT-IR spectrophotometer in the 400-4000 cm^{-1} range via KBr pellet. A Philips XL 30 ESEM (Environmental Scanning Electron Microscope) was used to observe the surface morphology. A Themoquest Italia S.P.A Pascal 440 and 140 Mercury Porosimeter was used to characterize pores. A Perkin-Elmer's Thermo Gravimetric Analyzer (TGA) was used to determine the thermal stability of the hydrogel. Elemental analysis was carried out using a Perkin-Elmer 2400 Series II elemental analyzer.

2.3 Synthesis of 1-Vinyl-3-ethyl imidazolium iodide (VEII)

1-Vinylimidazole 30 g (0.33 mols) and 50 mL dry acetone was charged in a 250 mL three-necked round bottom flask equipped with addition funnel, condenser and guard tube. The flask was cooled in ice bath at 5°C. The acetone solution containing 54.6 g (0.35 mols) ethyl iodide was added dropwise over a period of 1 h with constant stirring. The reaction mixture was then allowed to equilibrate to room temperature and then heated at 50°C for 1 h. After cooling the reaction mass, the separated white crystalline solid was filtered, washed with acetone, and dried in a vacuum oven at 60°C; yield: 94%, melting point: 106°C.

The synthesized monomer has been characterized by elemental analysis, FT-IR, ^{1}H and ^{13}C-NMR spectroscopy.

IR: 3132 cm^{-1}= C-H stretching, 2990 cm^{-1} C-H stretching, 1375 cm^{-1} C-H bending of $-CH_2$, 1582 cm^{-1} C=C stretching of imidazole ring, 1658 cm^{-1} C=C stretching of vinyl group, 1167 cm^{-1} C-N stretching, 1546 cm^{-1} C=N stretching, 771 cm^{-1}, 845 cm^{-1} and 923 cm^{-1} for C-H bending of imidazole ring.

^{1}H-NMR (δ ppm)**:** 1.64–1.67 (3H, t), 4.50–4.55 (2H, q), 5.44–5.47 (1H, dd, J = 2.96,8.69), 6.10–6.15 (1H, dd, J = 2.96,8.69), 7.42–7.48 (1H, dd, J = 8.72, 16.88), 7.94–7.95 (1H, m), 8.14–8.15 (1H, m), 10.23 (1H, s).

^{13}C-NMR (δ ppm)**:** 14.85, 44.83, 109.32, 119.15, 122.26, 127.31 and133.49.

2.4 Elemental Analysis

Calculated: C 33.62%, H 4.43%, N 11.20%; Found: C 33.44%, H 4.75%, N 11.48%

2.5 Hydrogel Synthesis

In a three-necked 250 mL round bottom flask equipped with a nitrogen inlet and condenser, VEII monomer (40 g) was charged with water (60 mL). To it, 0.28 g (N,N-methylene bisacrylamide) crosslinker and 0.30 g 2,2′-azobis(2-methylpropionamidine)dihydrochloride (V-50) initiator were added with continuous stirring. The nitrogen was purged into a reaction mass for half an hour and then heated at 60°C in an oil bath. After 2 h, gelation starts, but for complete gelation, it has been kept for 8 h under the same conditions. The formed gel was purified by immersing it into double distilled water for two days by changing the water at 3 h intervals. Half of the swollen gel was dried by an air forced oven, drying in order to prepare a non-porous hydrogel and the other half was dried using a solvent drying method to prepare a porous hydrogel. The detailed procedure of solvent drying is given in our previous paper (24). In a similar way, a non-cationic imidazole hydrogel was prepared from 1-vinylimidazole following the same reaction conditions.

2.6 Swelling Measurement

The swelling kinetics of porous and non-porous hydrogels were also studied. Figure 1 represents the swelling kinetics of porous and non-porous hydrogels. A swelling kinetics study indicates that for porous hydrogels, the time required to reach equilibrium was 2 min, whereas for the non-porous hydrogel it was 4.5 h. Another observation is, a porous hydrogel has higher % Seq value as compared to a non-porous hydrogel. The reason for the higher swelling rate and % Seq is the presence of interconnected pores, which provide more space to accommodate the water and fast adsorption of water by capillary action.

The % swelling was calculated using the following Equation 1:

$$\%\text{swelling} = \frac{W_o - W_t}{W_o} \times 100 \qquad (1)$$

Where W_t is the weight of swollen gel at time t and Wo is the weight of dry gel. The maximum % swelling was considered as the equilibrium percentage swelling (%Seq).

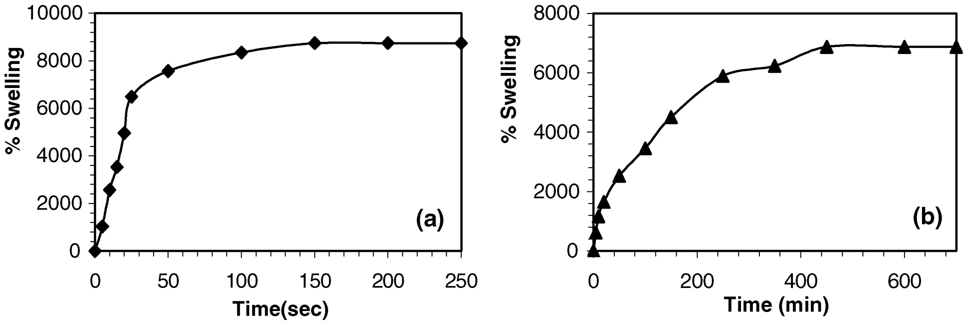


Fig. 1. Swelling kinetic curves of (a) porous and (b) non-porous cationic P(VEII) hydrogel.

2.7 Metal Removal Study

The stock solutions were diluted to the required concentration. The adsorption experiments were performed by agitating a specified (20 mg) amount of P[VEII] hydrogel in 50 mL of metal solutions of the desired concentrations at varying pH in a 100 mL stoppered conical flask. The pH of the solutions in the experiments was adjusted with 0.1 N HCl and 0.1 N NaOH. The reaction mixture was agitated at 200 rpm for a known period of time at 30°C in a Scigenics Orbitek mechanical shaker. After equilibrium, the concentration of metal ions remaining in the solution were determined by a Shimanzu-18A UV-Visible spectrophotometer using precalibrated curves. The amount of metal ions adsorbed on the hydrogel was calculated based on the difference of the metal ions concentration in the aqueous solution before and after adsorption, according to the following Equation 2:

$$Q = \frac{(C_0 - C_e)V}{W} \tag{2}$$

Where Q(mg/g) is the amount adsorbed per unit mass of adsorbent at equilibrium, C_0 and Ce denote the initial and equilibrium metal concentrations respectively (mg/L), V is the volume of the solution (L), and W is the weight of the adsorbent used (g). The metal adsorption equilibrium studies include: the effect of contact time, pH of the medium, effect of adsorbent doses, and effect of initial feed concentration of metal ions.

3 Results and Discussion

In this study, the porous P[VEII] hydrogel has been investigated for its chromium (VI) removal efficiency from the aqueous solution. The metal ions adsorbed hydrogels have been characterized by FT-IR spectroscopy, Scanning Electron Microscope (SEM) and Thermo Gravimatric Analysis (TGA). The removal of chromium (VI) from aqueous solutions by the hydrogel was examined by a batch equilibrium method. The influence of contact time, pH, initial concentration of the metal ions and adsorbent dose on the amount of adsorbed metal ions was studied. Swelling of the hydrogel was also carried out in metal ion solutions. The desorption study of metal ion adsorbed hydrogel was also carried out.

The FT-IR spectrum of porous P[VEII] hydrogel is presented in Figure 2. In IR spectra the strong and intense peaks appeared at 1160 cm^{-1} and 1548 cm^{-1} are of C-N and C=N stretching, respectively. The peaks at 1383 cm^{-1} and 1445 cm^{-1} corresponds to C-H bending of $-CH_2$ and $-CH_3$ of the ethyl group. The C=C and C-H stretching band were observed at 1623 cm^{-1} and 3066 cm^{-1}, respectively. Other peaks appeared at 647 cm^{-1}, 753 cm^{-1} and 831 cm^{-1} are of C-H bending vibration of imidazole ring.

As a result of the binding of metal ions with hydrogel, some bands appear and a certain shift took place in the IR spectrum of P[VEII] hydrogel. In the IR spectrum of chromium(VI) adsorbed hydrogel one additional peak appeared at 939 cm^{-1}. The appearance of this peak may be due to the M-O (metal-oxygen) bond. The peak appeared at 1623 cm^{-1} and 3066 cm^{-1} in IR spectrum of porous P[VEII] hydrogel shifted to higher frequency at 1629 cm^{-1} and 3075 cm^{-1}, respectively in the IR spectrum of chromium(VI) adsorbed hydrogel in Figure 3.

The difference in the morphology of porous P[VEII] hydrogel with and without adsorbed metal ions was observed by SEM analysis. The SEM microphotographs are presented in Figure 4 (a and b). The adsorbed metal ions on the surface of hydrogels are clearly seen in Figure 4b and we can't see any pores i.e., there may be a trapping of pore by the metal ions.

Figures 4 (a and b) represent the SEM microphotographs of P(VEII) hydrogel.

The TGA thermograms were taken for hydrogels with and without metal ion absorption. The relative thermal stabilities of both the different hydrogels were assessed by comparing the weight loss in the temperature range 35–600°C. The TGA data furnished in Figures 5 and 6 shows the nature of the thermo grams of the hydrogel and the hydrogel with adsorbed metal ions. From the TGA thermograms it is observed that the chromium(VI) adsorbed P[VEII] hydrogel in Figure 6 has very higher thermal stability as compared to a virgin porous P[VEII] hydrogel in Figure 5. The % weight loss of Virgin P[VEII] at 300°C and

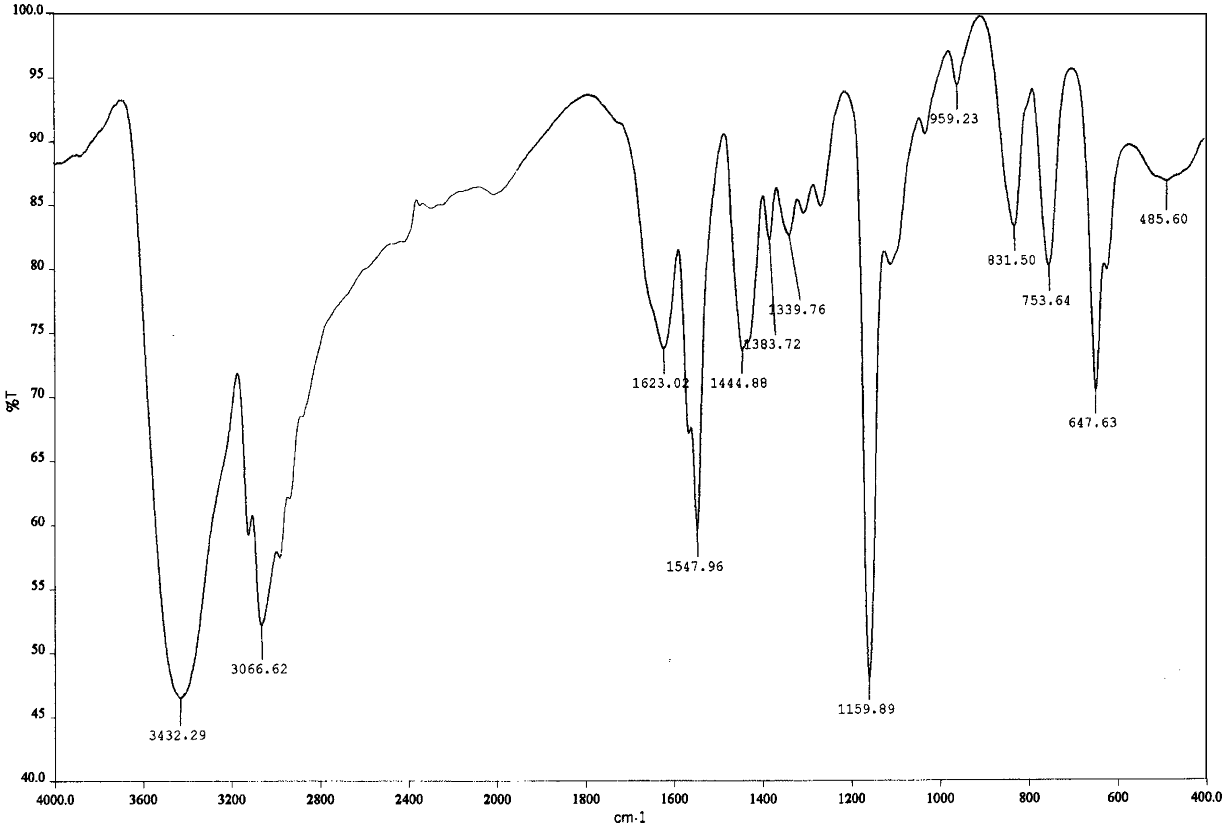


Fig. 2. FT-IR spectrum of porous P[VEII] hydrogel.

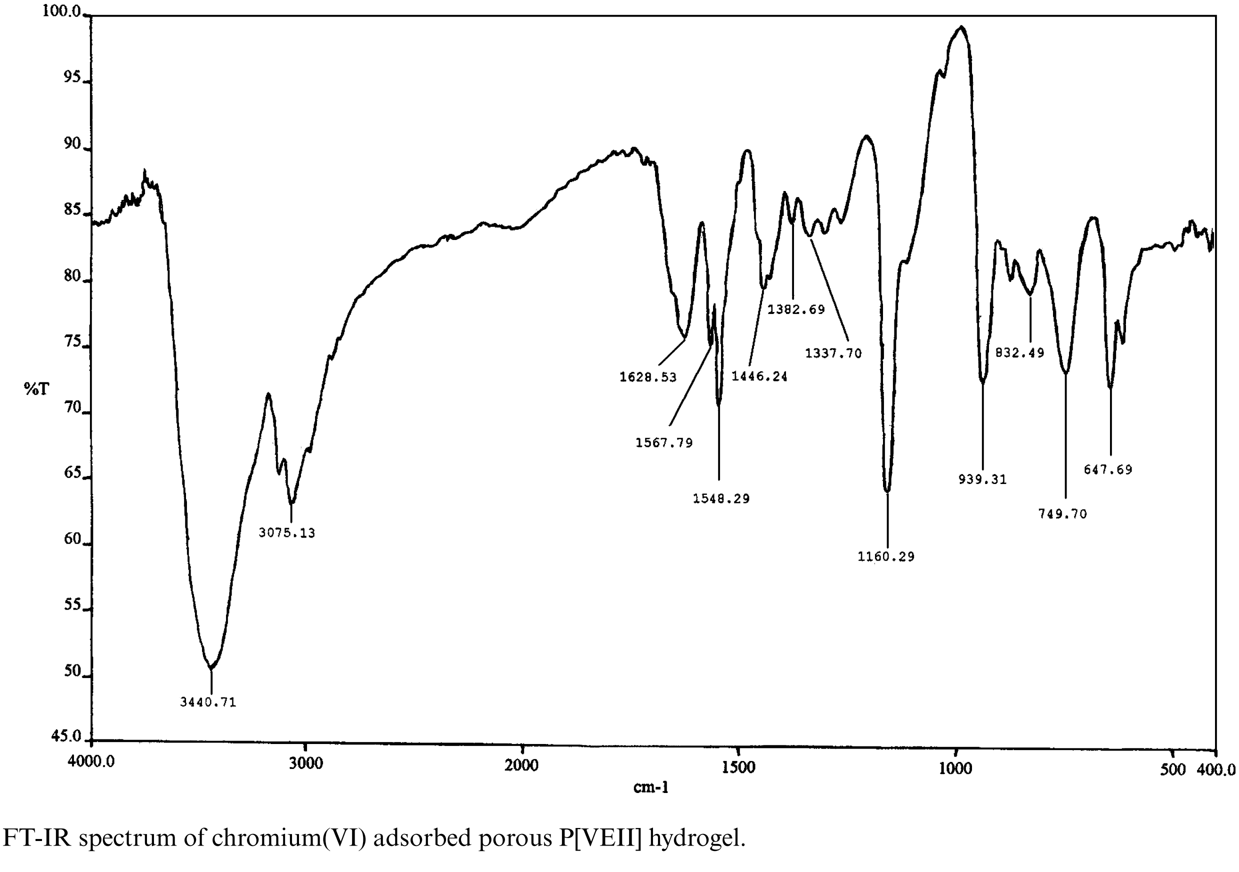
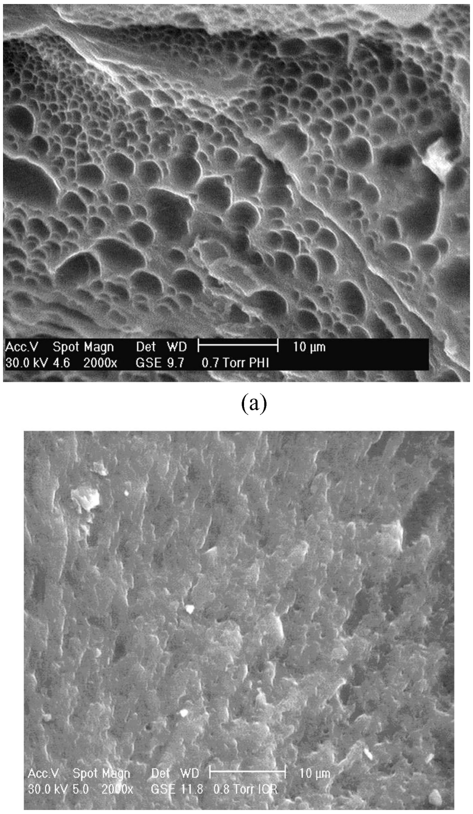


Fig. 3. FT-IR spectrum of chromium(VI) adsorbed porous P[VEII] hydrogel.



(a)

(b)

Fig. 4. (a) Virgin porous P[VEII] hydrogel, (b) Cr(VI) adsorbed P[VEII] hydrogel.

600°C are 83.9 and 100.0, respectively while that of metal adsorbed hydrogel is 38.72 and 62.56, respectively.

3.1 Swelling Studies

The swelling kinetics of porous and non-porous P[VEII] hydrogels in 100 mg/L solutions of chromium(VI) were determined at 30°C using a gravimetric method (25). The percentage degree of hydration (%H) was calculated using the earlier Equation (1). Figure 7 and 8 respectively shows the %H of porous and non porous P[VEII] hydrogel in Chromium(VI) metal solutions with respect to time.

Figure 7 shows that porous hydrogels swell rapidly and achieve equilibrium within 150 sec. This porous hydrogel behavior is due to the presence of interconnected pores within the hydrogel which absorb water rapidly via capillary action.

Figure 8 shows that in non-porous hydrogels, such types of pores are not available, so the rate of water absorption is very slow as compared to porous hydrogels.

3.2 Effect of Contact Time

Time course metal ions chelation by hydrogel was investigated to determine the time at which equilibrium adsorption has occurred. In this experiment 20 mg of hydrogel was agitated with 50 mL solution (100 mg/L) of chromium(VI) and the binding ratio was calculated at different time intervals until the equilibrium was reached.

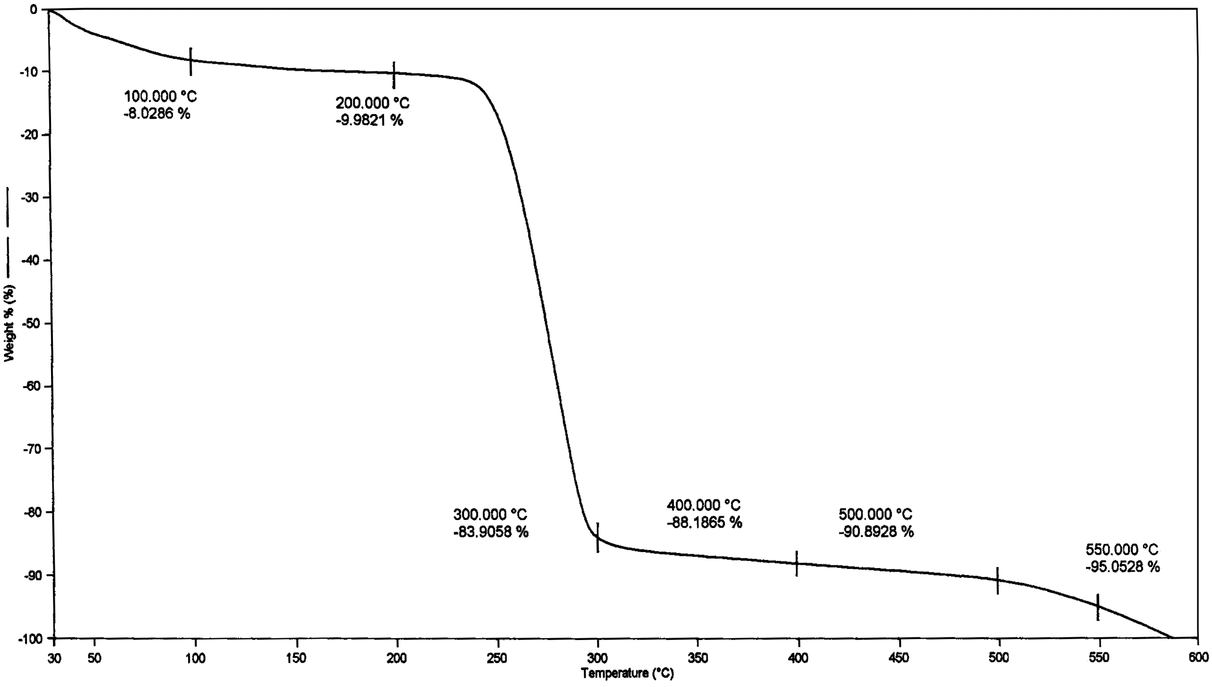


Fig. 5. TGA thermogram of porous P[VEII] hydrogel

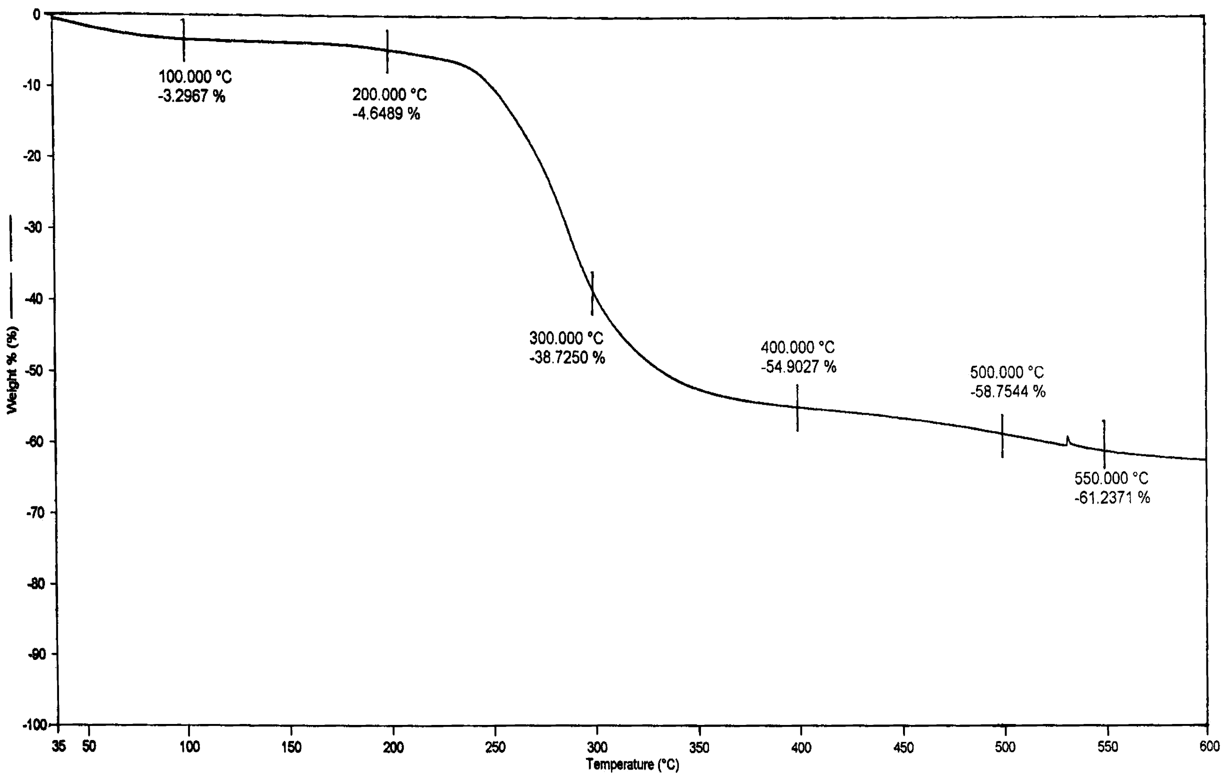


Fig. 6. TGA thermogram of chromium(VI) adsorb porous P[VEII] hydrogel

Figure 9 shows the effect of contact time for the adsorption of chromium(VI) on porous P[VEII] hydrogels. It is evident that the time has significant influence on the adsorption of chromium(VI). It is apparent from Figure 8 that up to 15 min, the binding ratio (Q) increases rapidly and reaches up to 36 mg/g. After that, the Q values increase slowly till 30 min and reaches up to 45 mg/g. A further increase in contact time has no significant effect on the Q values. Therefore, the contact time of 30 min could be considered for the adsorption of chromium(VI) on the porous P[VEII] hydrogel for the entire batch studies.

3.3 Effect of pH

20 mg of the porous P[VEII] hydrogel was agitated with different pH solutions (50 mL) of chromium(VI) ions in the pH range of 2–12, and the results are presented in Figure 10. Figure 10 illustrates the effect of pH on chromium(VI) ion adsorption by the porous P[VEII] hydrogel. The adsorption of chromium(VI) ions by the porous P[VEII] hydrogel decreased with an increase in pH of the solution until pH 8.0 and remained constant at basic pH. The maximum absorption capacity of the porous P[VEII] hydrogel occurred at a pH value of around 2.0–4.0.

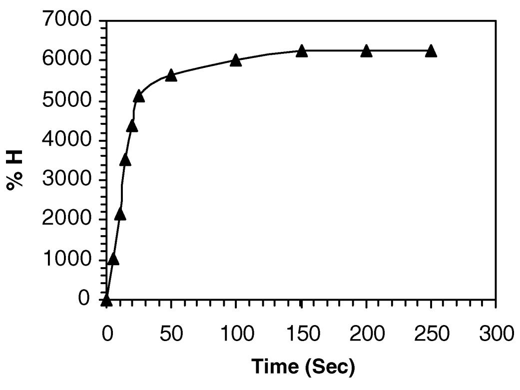


Fig. 7. The swelling curves of porous P[VEII] hydrogel in chromium(VI) solution

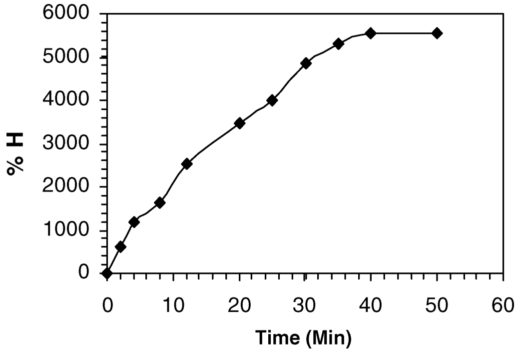


Fig. 8. The swelling curves of non porous P[VEII] hydrogel in chromium(VI) solution

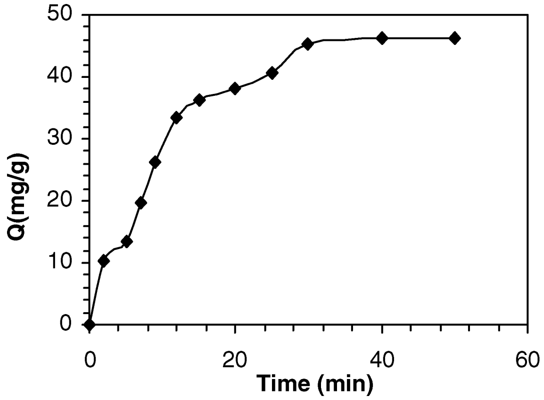


Fig. 9. The effect of contact time on the chromium(VI) ion adsorption by P[VEII] hydrogel.

As we know, the P[VEII] hydrogel is independent over the entire pH range even though there is a variation of binding ratio Q with respect to pH. It is due to a pH dependent protic equilibrium in which at acidic pH, the predominant species is $HCrO_4^-$ and at basic pH, the deprotonated species CrO_4^{-2} is predominant (26). As the cationic hydrogel is not influenced by pH, the decrease in absorption capacity at basic pH is attributed to the fact that, to neutralize the CrO_4^{-2}species, two quaternary nitrogens are necessary, while only one is needed to neutralize an $HCrO_4^-$species.

3.4 Effect of Adsorbent Doses

The dependence of absorption of chromium(VI) on the amount of the porous P[VEII] hydrogel is studied by varying the adsorbent doses from 25 mg to 75 mg, while keeping the volume (80 ml) and concentration (100 mg/L) of the chromium(VI) metal solution constant. The results are shown graphically in Figure 11.

It is apparent that the binding ratio (Q) increases rapidly with an increase in the dose of adsorbent to reach a maximum at certain value and then, it tends to level off at a higher adsorbent dose. The maximum binding ratio observed for chromium(VI) metal is 95.23 at 65 mg dose of adsorbent. At this adsorbent dose, the 100% removal is achieved and after this with the increase of adsorbent dose, a decrease in Q value is observed.

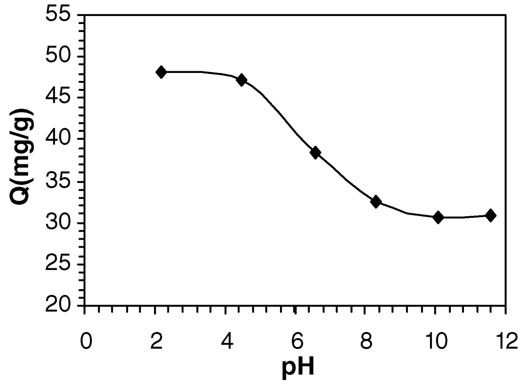


Fig. 10. The effect of pH on the chromium(VI) ion adsorption by the P[VEII] hydrogel

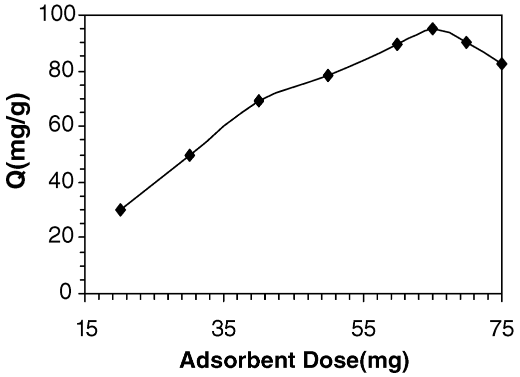


Fig. 11. Effect of adsorbent dosage on adsorption of chromium(VI)

3.5 Effect of Initial Feed Concentration of Metal Ions

This study was carried out by equilibrating a fixed amount of hydrogel (20 mg) with a series of metal ion solutions of a gradually increasing concentration (20-200 mg/L). The sorption capacity is the maximum amount of metal ions removed from the solution when the adsorbents are saturated.

Figure 12 shows the uptake profile of the porous P[VEII] hydrogel towards chromium(VI) ions with a different initial feed solution concentration varying from 20 to 200 mg/L. Figure 12 clearly shows a dependence of the uptake profile on the initial metal ion concentration, i.e., increasing the concentration of metal ions results in increasing the amount of metal ions uptake to reach a maximum at a certain value, then it tends to level off and became constant at higher feed solution concentration.

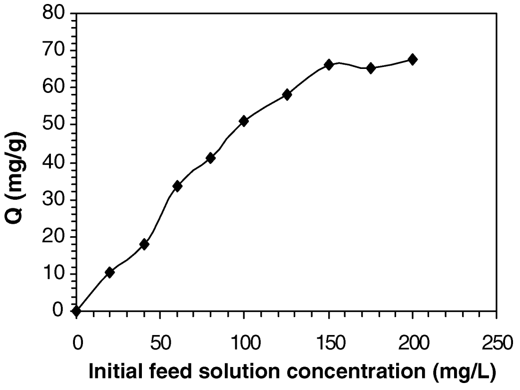


Fig. 12. The uptake profile of the porous P[VEII] hydrogel with different initial feed solution concentrations of chromium(VI) ions.

Table 1. Desorption percentage of chromium(VI) using various eluents.

Eluent	*Concentration (mol/L)*	*Desorption (%)*
NaOH/NaCl	1.0/1.5	99.91
HCl	1.0	62.35
HCl	2.0	75.67
HNO_3	1.0	81.34
NH_4Cl	2.0	52.36
NH_4Cl	3.0	64.81
NaOH	2.0	88.79
KCl	2.0	66.37
KCl	3.0	80.26

3.6 Desorption Study

In this study, the chromium(VI) adsorbed hydrogel was immersed in different eluents and agitated for 2 h and the concentration of desorbed chromium(VI) in solution was measured and the results are presented in Table 1. The desorption studies were conducted in aqueous solution using NaOH, HCl, HNO_3, KCl, NH_4Cl and NaOH/NaCl as eluents in the concentrations range of 1.0 to 3.0 mol/L.

Table 1 shows desorption percentages for each eluent, as well as their concentration. One can observe that the best desorption results were obtained with NaOH/NaCl. These results suggest the possibility of using the recycled porous P[VEII] hydrogel.

4 Conclusions

The metal removal study of the porous P[VEII] hydrogel has been carried out using chromium(VI) ions in the form of $Cr_2O_7^{-2}$. The porous P[VEII] hydrogel has a higher % degree of hydration in chromium(VI) solution as compared to the non-porous hydrogel. The thermal stability of the metal adsorbed porous P[VEII] hydrogel is too high as compared to the parent hydrogel. The maximum binding ratio observed for chromium(VI) metal is 95.23 mg/g at a 65 mg dose of hydrogel. The adsorption of chromium(VI) ions was dependent on contact time, pH of medium, dose of hydrogel and initial concentration of metal solution. At acidic pH chromium(VI) adsorption is higher as compared to basic pH. Desorption of chromium(VI) ions from the adsorbed hydrogel can be done using various eluents, but the best results were obtained with NaOH/NaCl.

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