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Removal of chromium from aqueous solution using polyaniline – Poly ethylene glycol composite

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ABSTRACT

The adsorption of chromium compounds from solutions by a composite of polyaniline/poly ethylene glycol (PANi/PEG) was investigated in this study. Experiments were conducted in batch mode under various operational conditions including agitation time, solution pH, PANi/PEG dose and initial concentration of chromium salts. Results showed that concentration of PEG at synthesizing stage has a significant effect on the capacity of produced composite for removal of chromium. Morphologically, PANi/PEG composite is closely dependent on the concentration of PEG. Maximum removal of hexavalent chromium was experienced when 2 g/L of PEG was used in synthesis of PANi/PEG. Removal of hexavalent chromium by PANi/PEG composite included surface adsorption and reduction reaction. The optimum pH was 5 and the equilibrium time for hexavalent chromium removal was about 30 min. Investigation of the isothermal characteristics showed that chromium adsorption by PANi/PEG composite was in high accordance with Langmuir's isotherm.

1. Introduction

Heavy metals are potentially hazardous to human health even in minute quantities. Chromium is a typical heavy metal which is used extensively in industries such as electroplating, tanning, textile and thus is widely present in the effluents of these industries. Chromium is commonly found in two oxidation states; hexavalent (Cr(VI)) and trivalent (Cr(III)). The hexavalent form is 500 times more toxic to aquatic life than trivalent one [1]. Various methods used for the removal of chromium from aqueous solutions include chemical precipitation, reverse osmosis, ion exchange and adsorption [2]. Among these methods, surface adsorption is considered more effective than others when low concentrations of Cr are present [3]. Recently, new organic adsorbents in forms of resins and polymers such as "polyaniline" and its composites have become under attention as exciting new absorbing materials. Higher removal capacities, versatility and more overall efficiency are the main reasons for development of this technology [4–6].

Polyaniline is one of the most important and widely used conducting polymers which has shown excellent environmental stability [7] and is synthesized at low cost. Polyaniline can exist in a wide range of forms, but its general molecular structure is shown in Fig. 1 [8]. It is synthesized chemically and electrochemically in the shape of powder and film and is used in many industrial applications, including charge storage devices [9], sensors [10–12], biosensors [13–15], rechargeable batteries [16], protector shield in magnetic fields [17], microwave absorption [18], catalytic application [19], image processing [20] and infrared optic applications [21]. Polyaniline has also shown good potential for adsorbing heavy metals from effluents. For example, reduction of toxic hexavalent chromium in water was successfully carried out by this compound in a powder form [22]. Another study showed that short chain polyaniline coated on jute fiber is capable of removal and recovery of chromium, as well as reduction of hexavalent to trivalent form in solution [23–25].

In this study, polyaniline was synthesized in the presence of various concentrations of PEG (1–10 g/L) as a surfactant. In the first step, dose of added PEG was optimized to achieve the PANi/PEG composite that has maximum chromium removal capacity. Next, synthesized PANi/PEG composite was tested as an adsorbent for removal and reduction of chromium from aqueous solutions under various conditions. The effect of some parameters such as solution pH, adsorption–reduction reaction time, and initial concentration of chromium were tested to find optimum conditions. Finally equilibrium concentration and isothermal behavior was investigated.

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Nomenclature

C_0	initial concentration of chromium in solution (mg/L)
C_e	concentration of chromium (mg/L) after a certain time of exposure time
C_t	concentration of chromium (mg/L) in desorbent solutions
x	amount adsorbed (mg)
m	weight of sorbent (g)
X	amount adsorbed by adsorbent (mg/g)
X_m	maximum amount adsorbed by adsorbent (mg/g)

2. Materials and methods

2.1. Materials

Sulfuric acid, potassium dichromate, potassium iodate, poly (ethylene glycol) (M_w = 35,000) and aniline used were all from “Merck Chemical Company”. Aniline monomer was purified by vacuum distillation before polymerization.

2.2. Instrumentation

A magnetic stirrer (MR 3001 K, Heidolph), analytical scale (BP 211 D, Sartorius) and pH meter (CH 9101-Herisau, Metrohm) were used in experiments. Hexavalent chromium concentration was measured by UV-Visible Spectrophotometer (Cary 300, Varian) at 540 nm wavelength by 1, 5-diphenyl carbazide method [26]. Total chromium concentration was measured by Atomic Absorption Spectrophotometer (Spectra AA, Varian) in combination with air–acetylene flame at 429 nm wavelength and slit width of 0.5 nm.

2.3. Chemical synthesis

PANi/PEG composite was chemically synthesized by oxidizing aniline monomer in the presence of various concentrations of PEG. In this procedure, 1.6 g of potassium iodide is dissolved in 200 mL of 1 M H_2SO_4 and fully mixed. Then 0.2 g of PEG is added to the solution and stirred for 15 min. Next, 2 mL of pure aniline monomer is added to the mixture and fully mixed about 5 h. The polymer is separated from solution by filter paper, washed with distilled water and acetone to remove impurities. The resulted polymer is left to dry and the grinded to form a powder (instead of 0.2 g of PEG, other amounts can be used to obtain PANi/PEG with different concentrations of PEG).

2.4. Experimentation

Chromium solutions were prepared by dissolving potassium dichromate in distilled water. It is expected that some of Cr(VI) is reduced to Cr(III) and some is adsorbed by PANi/PEG composite. Therefore throughout the study total chromium which is the sum of tri- and hexavalent (Cr(T), Cr(T) = Cr(VI) + Cr(III)) as well as hexavalent chromium (Cr(VI)) were measured in the reactor to establish the effectiveness of each process. Batch experiments were carried out by adding predetermined quantities of PANi/PEG composite to a 100 mL of hexavalent chromium solution. A magnetic stirrer was employed at 300 rpm for mixing to reach equilibrium concentration. Solution pH ranged from 1 to 13, adjusted by addition of 0.1 M NaOH and 0.1 M H_2SO_4. After different exposure times, chromium solutions were filtered and filtrate was used for measurement of total and hexavalent chromium concentrations. The efficiency of total and hexavalent chromium removal was calculated according to Eq. (1):

$$R = 100\frac{C_0 - C_e}{C_0} \quad (1)$$

where C_0 is the initial concentration of chromium in solution (mg/L) and C_e is the concentration of chromium (mg/L) after reaching equilibrium conditions.

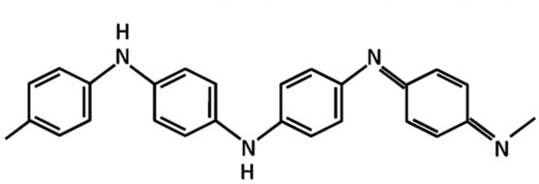

Fig. 1. General scheme of polyaniline.

2.5. Desorption and recovery of chromium

Desorption studies were also conducted in batch experiments to investigate the recovery of adsorbent. For this purpose 100 mL of a solution containing 50 ppm of Cr(VI) ion was treated with 0.1 g PANi/PEG for 30 min at pH 5. The mixture was then filtered and the filtrate was analyzed for total chromium. The solid residue remained after filtration was PANi/PEG composite saturated with chromium salts. This residue was transferred to 100 mL of two desorbents, including 0.1 M HNO_3 and 0.2 M NaOH and stirred for 60 min. The mixture was again filtered and desorbed concentration of total chromium was determined in the filtrate and desorption efficiency was calculated according to Eq. (2)

$$R = 100\frac{C_t}{C_0 - C_e} \quad (2)$$

where C_0 is the initial concentration of chromium in solution (mg/L), C_e is the remaining concentration of chromium (mg/L) and C_t is the concentration of chromium (mg/L) in desorbent solutions.

2.6. Scanning electron microscopy (SEM)

The platelets adhered on the PANi/PEG composites were studied by SEM, using a LEO 440i Scanning Electron Microscope which was operated at 15.0 kV. The prepared composites mounted on the sample studs by means of double-sided adhesive tapes and coated with platinum prior to the SEM measurement.

3. Results and discussion

3.1. Removal of Cr(VI) from solution using PANi/PEG

0.1 g sample of various PANi/PEG powders were added to 100 mL of solution containing 50 ppm Cr(VI) at pH 5. The exposure was performed in 30 min followed by filtration and separation of PANi/PEG solid particles.

Results show (Fig. 2) the PANi/PEG composites are able to remove Cr(T) and Cr(VI) from aqueous solution. Furthermore, the adsorption efficiency was closely related to PEG concentration. The maximum removal efficiency occurred when 2 g/L PEG was used. The PEG is stabilizing agent and can affect the morphology of polymer [27–29] because the additives are adsorbed physically or chemically by the growing polymer. Adsorption of PEG on the polyaniline particles is primarily due to the hydrophobic component in the PEG, probably via a hydrogen bonding mechanism with the aniline N–H groups.

The SEM micrographs of PANi/PEG composites using various concentrations of PEG are shown in Figs. 3–7. Synthesized polyani-

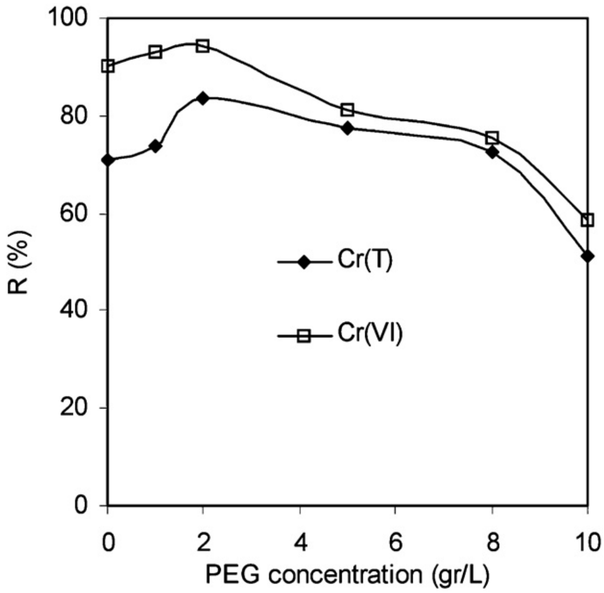

Fig. 2. Effect of PEG concentration on Cr(T) and Cr(VI) removal using PANi/PEG composite (PANi/PEG dose 1 g/L; initial Cr(VI) 50 ppm; pH 5; agitation time 30 min).

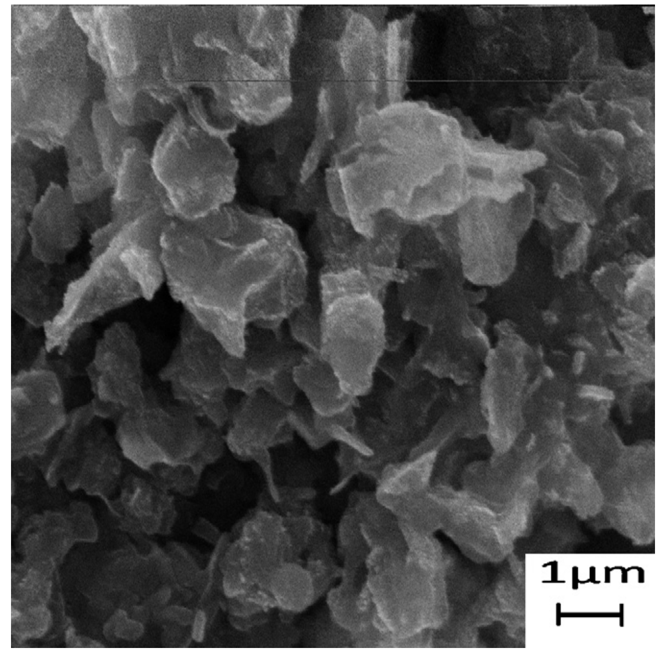

Fig. 4. SEM micrograph (10,000×) of PANi/PEG composite (PEG dosage 1 g/L).

line alone and synthesized polyaniline in the presence of 2 and 5 g/L PEG have a steady granular form surface, but the granules sizes are different. The synthesized polyanilines in the presence of 1 and 10 g/L PEG have sharp scale-like surfaces. The variation of morphology for these composites is as a result of the amount of chemically adsorbed surfactant on polyaniline particles. The variation of PANi/PEG morphology affects the capacity of the composite by changing the interface between the composite and the solution. Specific surface and adsorption sites on the surface of composites are results of morphological changes.

The removal mechanism of chromium for PANi/PEG composite seems to be the combination of surface adsorption and reduction of chromium(VI). When PANi/PEG was used, total and hexavalent chromium removal rate were not equal. It means that after contact time between PANi/PEG and Cr(VI) solutions, Cr(III) appears in solutions, which indicates that PANi/PEG are responsible for reduction of Cr(VI) to Cr(III). Another mechanism is the surface adsorption. The nitrogen atoms in PANi/PEG composite can make co-ordinate bond with positive charge of metals due to the presence of lone pair electron of nitrogen. This co-ordinate bond is the plausible mechanism for adsorption of Cr(VI) from solution. Moreover, under acidic conditions the nitrogen atoms of PANi/PEG are protonated due to polyaniline nature [30]. The protonated form of nitrogen atoms in PANi/PEG can form bonds with solution anions (chromate and dichromate) by electrostatic attraction.

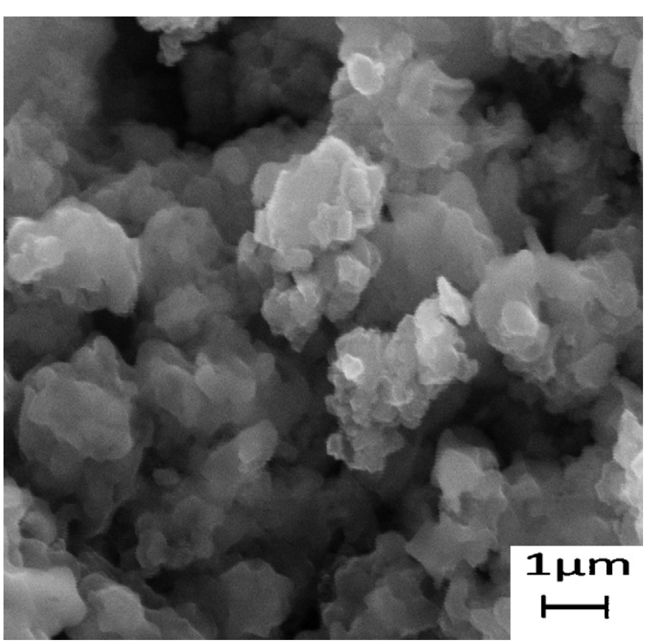

Fig. 3. SEM micrograph (10,000×) of polyaniline.

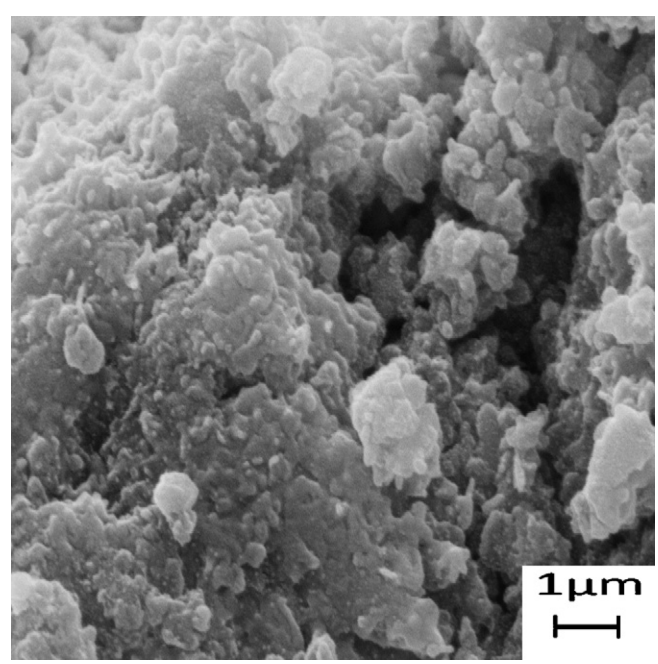

Fig. 5. SEM micrograph (10,000×) of PANi/PEG composite (PEG dosage 2 g/L).

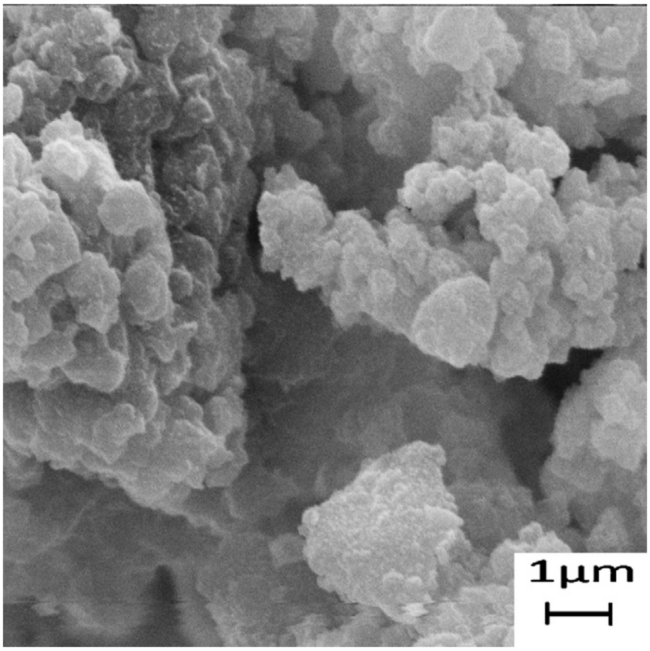

Fig. 6. SEM micrograph (10,000×) of PANi/PEG composite (PEG dosage 5 g/L).

3.2. Effect of pH

Solution pH is one of the most important parameter in adsorption processes. The effect of pH on adsorption of Cr(T) and Cr(VI) by PANi/PEG was investigated to find the optimum pH for maximum removal efficiency. For this purpose, 0.1 g of PANi/PEG was added to 100 mL of Cr(VI) solution with concentration of 50 ppm at various pHs (1–13) and mixed for 30 min.

Results shown in Fig. 8, under acidic medium (pH 3–5) indicates high rate of Cr(VI) and Cr(T) removal is attainable. At these pHs, the nitrogen atoms of PANi/PEG are protonated. The protonated form of nitrogen atoms of PANi/PEG can form bonds with chromate and dichromate anions by electrostatic attraction for high adsorption

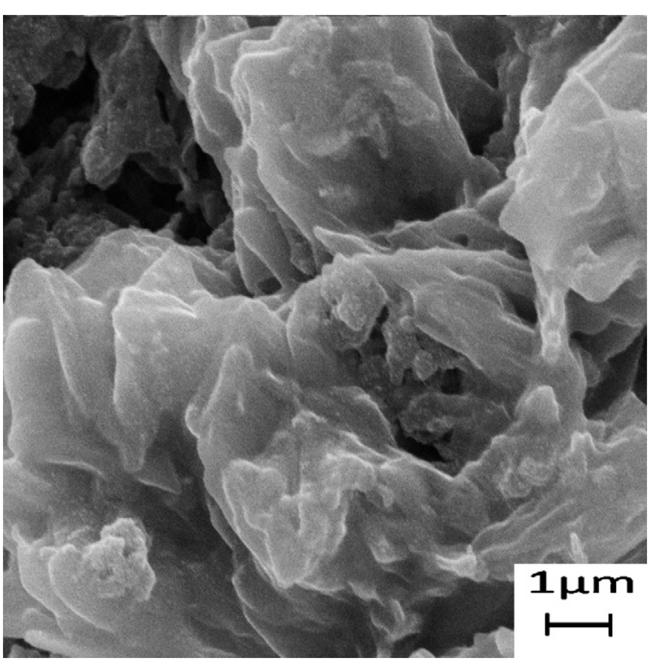

Fig. 7. SEM micrograph (10,000×) of PANi/PEG composite (PEG dosage 10 g/L).

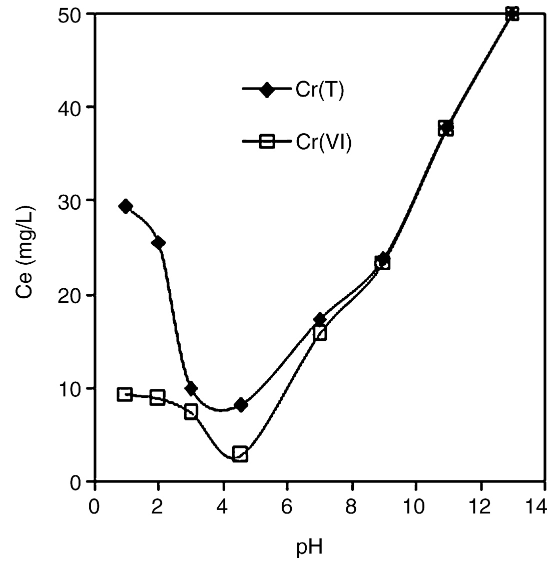

Fig. 8. Effect of pH on total and hexavalent chromium removal (PANi/PEG dose 1 g/L; initial Cr(VI) 50 ppm; agitation time 30 min).

of Cr(VI) to occur. As pH increases, the degree of protonation of the PANi/PEG surfaces reduces gradually resulting in a gradual decrease in adsorption of Cr(VI) ions.

Under acidic condition, in addition to adsorption, the strong reduction reactions were also occurred. In this pHs, a part of Cr(VI) is reduced to Cr(III). Therefore, under acidic pHs, Cr(VI) removal is more than Cr(T) removal, where some of Cr(VI) is reduced to Cr(III). With increase of pH to neutral and alkaline condition, the reduction mechanism was decreased and the major mechanism was surface adsorption of Cr(VI). Under neutral and alkaline condition, Cr(T) and Cr(VI) removal are nearly equal.

3.3. Effect of agitation time

Contact and equilibrium time is another important parameter in adsorption processes. It is expected that removal efficiency increases gradually with contact time until equilibrium is reached. When 0.1 g of PANi/PEG was added to 100 mL of solution containing 50 ppm Cr(VI) at pH 5 (optimum pH), most of chromium ions were removed rapidly, within the first 2 min (Fig. 9). However total and

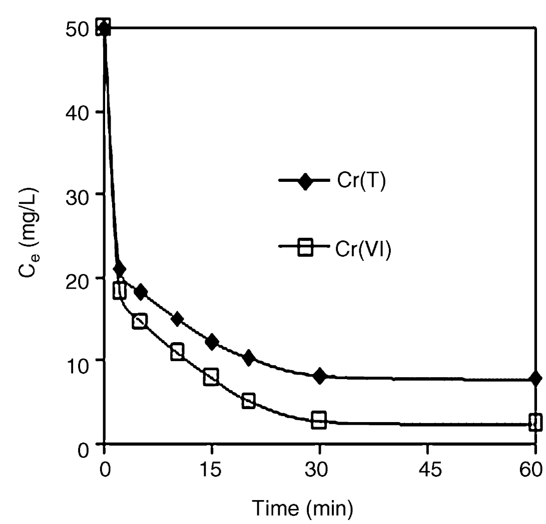

Fig. 9. Effect of agitation time on total and hexavalent chromium removal (PANI/PEG dose 1 g/L; initial Cr(VI) 50 ppm; pH 5).

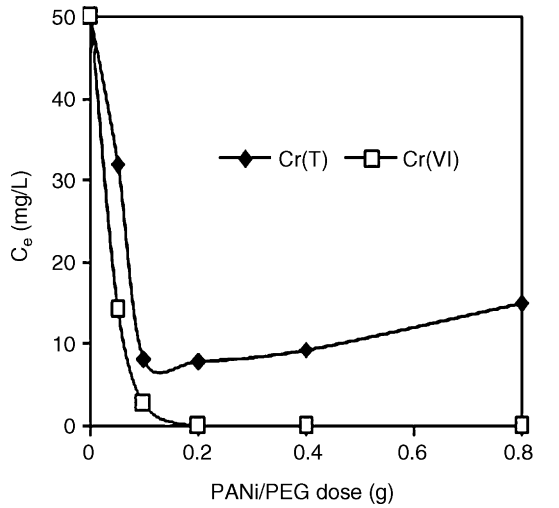

Fig. 10. Effect of PANi/PEG dose on total and hexavalent chromium removal (initial Cr(VI) 50 ppm; pH 5, agitation time 30 min).

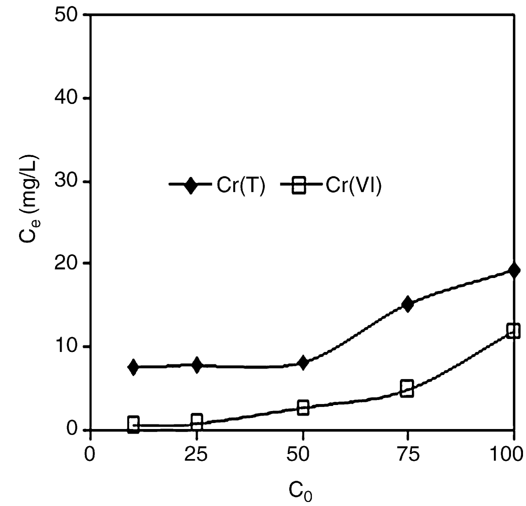

Fig. 11. Effect of initial concentration on total and hexavalent chromium removal (PANI/PEG dose 1 g/L; agitation time 30 min; pH 5).

hexavalent chromium concentration in solution decreased with time but was not significant after almost 30 min. It is therefore concluded that the equilibrium time for Cr(T) and Cr(VI) removal using PANi/PEG was approximately 30 min.

3.4. *Effect of PANi/PEG dose*

The effect of PANi/PEG dosage on the removal of Cr(T) and Cr(VI) was studied. Different amounts of PANi/PEG, within the range of 0.05–0.8 g were added to 100 mL of 50 ppm Cr(VI) solution while the solution pH was kept at 5 (optimum pH) and contact time was 30 min (already established as optimum conditions).

The results (Fig. 10) show that the Cr(T) and Cr(VI) concentration in solution decrease's sharply when the adsorbent dose reached 0.1 g. Further addition of PANi/PEG did not have any significant effect on total chromium removal but increased Cr(VI) reduction to almost 100%. It appears that higher doses of PANi/PEG increase the reduction reaction of hexavalent chromium to trivalent state. The positive charge of protonated surface of PANi/PEG at this pH (pH 5) repulse's the Cr(III) ions (Cr(III) is in cationic form) and so very slight adsorption of Cr(III) is observed under these conditions. Therefore, there is an optimum dose of PANi/PEG for Cr(T) removal (0.1 g per 100 L of solution in this case) while Cr(VI) reduction requires higher doses.

3.5. *Effect of initial concentration of chromium*

The effect of solution concentration on removal efficiency was tested using solutions containing 10–100 ppm of Cr(VI). At optimum pH and contact time, 0.1 g of PANi/PEG was added to 100 mL of Cr(VI) solution with various concentrations. The results (Table 1 and Fig. 11) show that with increasing the initial concentration of Cr(VI) from 10 to 100, the Cr(T) and Cr(VI) concentration in solution after exposure time were increased, although higher amounts of these ions were adsorbed. This behavior is typical for most adsorption processes.

Table 1
Total concentration of Cr(T) and Cr(VI) using polyanilines/polyethylene(glycol) composite in various initial concentration of Cr(VI) solution(polyaniline/polyethylene glycol) dose 1 g/L; pH 5; agitation time 30 min).

Initial concentration	10	25	50	75	100
Total concentration of Cr(T)	1.5	3.89	8.2	22.74	38.5
Total concentration of Cr(VI)	0.1	0.32	2.77	7.28	23.91

3.6. *Adsorption isotherms*

In order to investigate the sorption behavior, adsorption isotherms were studied at room temperature. Both Langmuir and Freundlich equations were tested to find the most suitable isotherm model. Langmuir and Fruendlich equations are defined as Eqs. (3) and (4), respectively. The linear form of Fruendlich equation can be shown as Eq. (5).

$$\frac{1}{X} = \frac{1}{X_m} + \frac{1}{bX_m C_e} \quad (3)$$

$$\frac{x}{m} = kC_e^{1/n} \quad (4)$$

$$\log\left(\frac{x}{m}\right) = \log k + \frac{1}{n}\log(C_e) \quad (5)$$

where C_e is the equilibrium concentration of chromium in solution (mg/L), x is the amount adsorbed (mg), m is the weight of sorbent (g), X is the amount adsorbed by adsorbent (mg/g) and X_m is the maximum amount adsorbed by adsorbent (mg/g). Langmuir isotherm assumes that the number of adsorption sites is fixed and that adsorption is reversible. Therefore, b is Langmuir's constant signifying energy of sorption. The parameters, k and n are Fruendlich's constants indicating sorption capacity and intensity, respectively. The plots of $1/X$ versus $1/C_e$ and $\log(x/m)$ versus $\log(C_e)$ enable the constants of Freundlich and Langmuir adsorption isotherms to be determined.

Constant weights of sorbents (0.1 g) were used with 100 mL of Cr(VI) solution at pH 5 (considered as optimum pH) at different initial Cr(VI) concentrations (10–100 ppm) for 30 min exposure time (optimum agitation time). Table 1 shows the concentration of Cr(T) and Cr(VI) after agitation when the solution had reached equilibrium.

According to results (Figs. 12–15), adsorption of Cr(T) and Cr(VI) by PANi/PEG can be well fitted in Langmuir equation. Table 2 shows the calculated k, n, b and X_m constants from plots. For a suitable sorbents, constant n in Freundlich equation is normally between 2 and 10. These results indicate that PANi/PEG can be used as good adsorbents for removal of hexavalent chromium in solutions. The maximum adsorption of total and hexavalent chromium was calculated as 109.9 and 68.97 (mg/g) for PANi/PEG composite respectively. It should be noted that the important characteristic

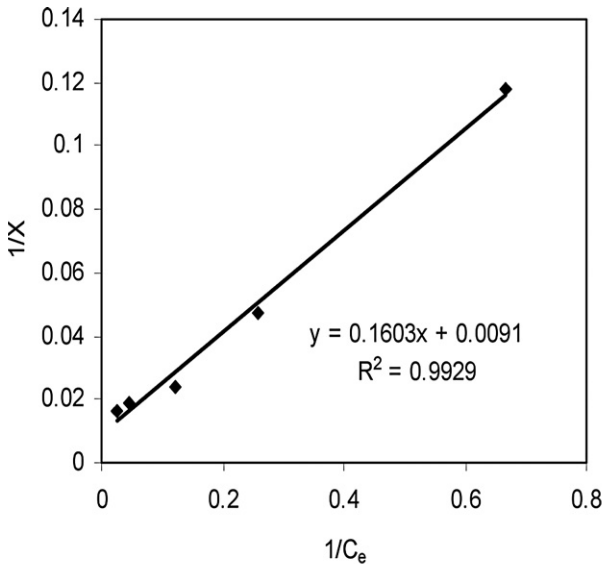

Fig. 12. Adsorption isotherm of total chromium using Langmuir equation obtained for PANi/PEG.

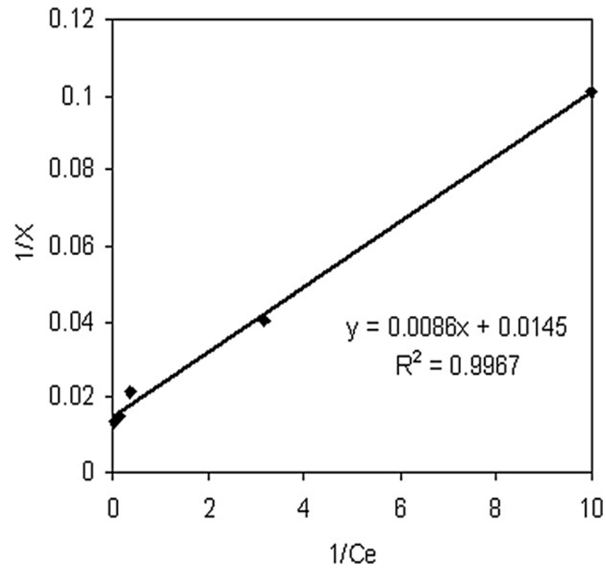

Fig. 14. Adsorption isotherm of hexavalent chromium using Langmuir equation obtained for PANi/PEG.

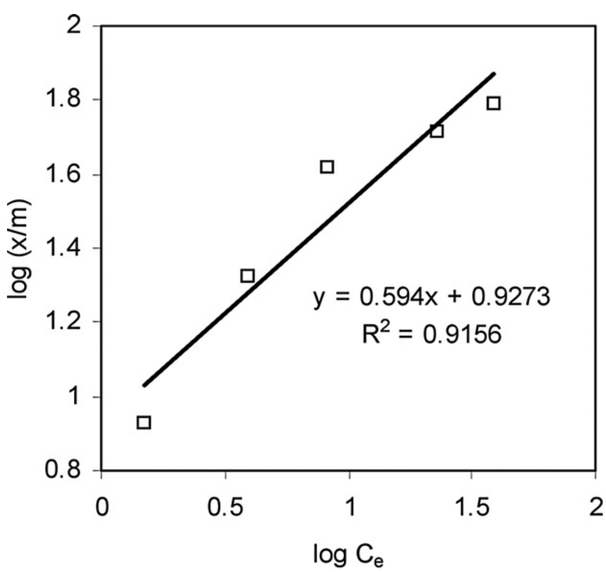

Fig. 13. Adsorption isotherm of total chromium using Freundlich equation (linear form) obtained for PANi/PEG.

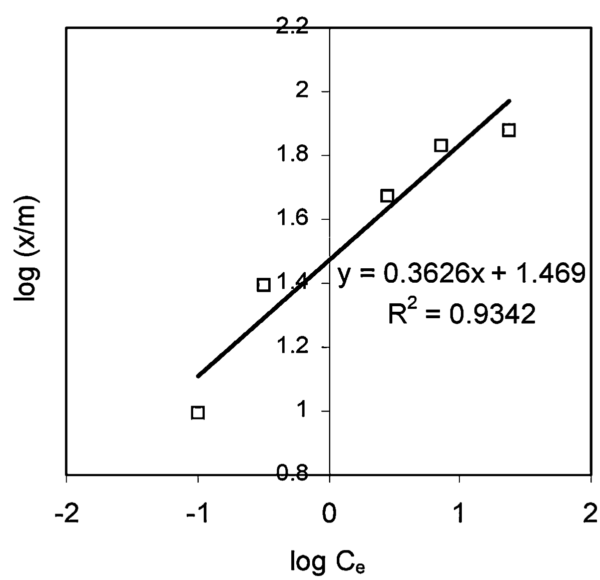

Fig. 15. Adsorption isotherm of hexavalent chromium using Freundlich equation (linear form) obtained for PANi/PEG

of PANi/PEG composite is its capability in reducing Cr(VI) to Cr(III) which is not observed in other adsorbents. Fixation and immobilization of PANi composites on surfaces has been reported by some researchers [31–33].

3.7. Desorption and recovery of adsorbed chromium

A sample of PANi/PEG already saturated with Cr(VI) was tested for desorption. The sample had been applied to a 100 mL solution containing 50 ppm Cr(VI) and mixed for 30 min at pH 5. Chromium containing PANi/PEG was transferred to 100 mL of two desorbents including 0.2 M NaOH and 0.1 M HNO_3 solutions and stirred for 60 min. During desorption, 38.33 and 83.02% of total chromium were released in solution by 0.2 M NaOH and 0.1 M HNO_3, respectively.

Table 2
Comparison of Freundlich and Langmuir constant of values Cr(T) and Cr(VI) for polyaniline/poly(ethylene glycol) composite.

Isotherm constants	*n*	*k*	*b*	X_m
Cr(T)	1.68	8.46	0.057	109.9
Cr(VI)	2.76	0.68	1.69	68.97

4. Conclusion

The effect of polyaniline/poly (ethylene glycol) (PANi/PEG) composite on the hexavalent chromium removal from aqueous solution was investigated. The results showed that the PEG and its concentration play a major role on the adsorptive properties of PANi/PEG composite. PEG is a stabilizing agent which affects the morphology of prepared composite. Maximum removal of chromium was occurred for synthesized composite in the presence of 2 g/L PEG. Sorption experiments for PANi/PEG composite at the optimum state (PEG concentration 2 g/L) showed that the removal mechanism of chromium by PANi/PEG is the combination of surface adsorption and reduction reaction. The optimum pH occurred at pH 5 and the equilibrium time for total and hexavalent chromium removal was about 30 min using PANi/PEG composite. Investigating the isothermal characteristics show that PANi/PEG can be used as good adsorbents for removal of total and hexavalent chromium in aqueous solutions. The maximum adsorption of total and hexavalent chromium was calculated as 109.9 and 68.97 (mg/g) for PANi/PEG. The desorption experiments indicated that 38.33 and 83.02% of total chromium can be recovered using 0.2 M NaOH and 0.1 M HNO_3, respectively.

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