



Removal of chromium (VI) metal ions using Amberlite IRA-420 anions exchanger

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ABSTRACT

Anions exchanger (Amberlite IRA-420) has been used for the removal of chromium (VI) metal ions, as HCrO_4^- and $\text{Cr}_2\text{O}_7^{2-}$ ions, from dichromate contaminated water. Operational conditions such as chromium (VI) metal ions concentration, adsorption time, adsorption temperature, adsorption pH, agitation speed, and finally adsorbent dosage have been investigated, and its impact on the removal process efficiency has been presented. Moreover, FT-IR and TGA analyses have been performed for the original Amberlite and chromium (VI) metal ions adsorbed Amberlite particles. Regeneration of the chromium (VI) metal ions (mainly as HCrO_4^- ions) adsorbed on Amberlite particles using sodium hydroxide has been studied. Finally, the reuse ability has been investigated.

Keywords: Anions exchanger; Adsorption; Chromium (VI) metal ions; Dichromate; Removal

1. Introduction

Water is the most shared and widespread chemical compound in nature which is a major constituent of all living creatures. Heavy metal pollution of the environment has become a growing ecological crisis and concern and, therefore, the subject of many types of research [1]. These heavy metals are continuously released into the aquatic environment from natural processes like volcanic activity and weathering of rocks. Industrial processes like electroplating, metal finishing,

metallurgical, chemical manufacturing and mining industries have been significantly enhanced the concentration of heavy metals in the water.

Heavy metals are widely recognized and used such as Chromium (Cr), Cadmium (Cd), Copper (Cu), Mercury (Hg), Nickel (Ni), Lead (Pb) and Zinc (Zn) are also toxic in nature. Unlike organic wastes, heavy metals are non-biodegradable, and they can be accumulated in living tissues, causing various diseases and disorders. Therefore, they must be removed before discharge [2].

Chromium (Cr) compounds used in many industries such as textile dyeing, tanneries and metal electroplating. Chromium can exist mainly as Cr (VI) or Cr (III) in the natural environment. Cr (III) species are less soluble and more

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stable compared to Cr (IV) species which are highly soluble and mobile in aqueous solutions [3]. Chromium (VI) also has higher mobility than chromium (III) therefore; it has a greater potential to contaminate the groundwater. The high risk of chromium (VI) associated with its high reactivity and its potential carcinogenic properties [4].

Acute exposure to Cr (VI) causes nausea, diarrhea, liver and kidney damage, dermatitis, internal hemorrhage, and respiratory problems [5]. Inhalation may cause acute toxicity, irritation, and ulceration of the nasal septum and respiratory sensitization (asthma) [5]. Ingestion may affect kidney and liver functions. Skin contact may result in systemic poisoning damage or even severe burns, and interference with the healing of cuts or scrapes. If not treated promptly, this may lead to ulceration and severe chronic allergic contact dermatitis. Eye exposure may cause permanent damage.

Some methods had used for removing heavy metals are solvent extraction and filtration [6], chemical or electrochemical precipitation [7], cation exchange membranes [8], chemical precipitation, oxidation/reduction, filtration, ion exchange, membrane separation and adsorption [9]. Adsorption techniques are widely used to remove certain classes of pollutants from wastewater [10]. Adsorption is also considered to be a cheap and efficient method for the removal of Cr (VI) from wastewater using different adsorbents such as charcoal [11], activated carbon from various sources [12–14], polyaniline and its composites [15], and Chitosan [16].

The aim of this work is to study the removal of Cr (VI) ions from dichromates solution using strong anion exchanger of poly (styrene-co-divinylbenzene) under different operation conditions. Moreover, the regeneration process of the anion exchanger has been studied, and the reuse ability assessed.

2. Materials and methods

2.1. Materials

Amberlite (IRA 420); A commercial Amberlite IRA-420 ion exchange resin supplied by Rohm and Haas used with specifications showed in Table 1.

Potassium dichromate ($K_2Cr_2O_7$), minimum assay 99%, supplied by Sigma Aldrich, Germany. Sodium hydroxide (NaOH), minimum assay 99%, supplied by Sigma Aldrich, Germany. Sodium chloride (NaCl), minimum assay 99%, supplied by Sigma Aldrich, Germany.

Table 1
Properties of the resin Amberlite IRA-420

Producer	Rohm and Haas
Functionality	$-N^+-(CH_3)_3$
Matrix type	Polystyrene-DVB
Standard ionic form	Cl^-
Total exchange capacity (meq/g)	3.80
Bed Porosity	0.32
Wet resin density (g/cm^3)	1.15
Bed density (g/cm^3)	0.68
pH operation range	0–14
Maximum operating temperature	77°C
Mean wet particle radius (mm)	0.30–0.70

2.2. Preparation of primary dichromate solution

Potassium dichromate, the stock solution was prepared by dissolving 1 g of dichromate in 1,000 mL distilled water using magnetic stirrer. The dichromate concentration in the supernatant and residual solutions determined by measuring their absorbance using 1 cm light-path cell at Max wavelength 380 nm using UV- Visible spectrophotometer.

2.3. Standard curve of dichromate concentration

Varied Dichromate solution concentrations from 10 to 300 mg/L have been prepared. The absorbance (A_{abs}) of the samples has been measured using a UV-Visible spectrophotometer and plotted against their concentrations. From the slope, we can be derivative the following relation between absorbance and concentration:

$$\text{Concentration (mg / L)} = A_{abs} \times \text{Constant (1 / Slope)} \quad (1)$$

2.4. Adsorption experiment

The adsorption experiments were carried out in a batch process by using aqueous dichromate solution. The variable parameters namely; the initial Cr (VI) ions concentration, the adsorbent amount, the contact time, and the adsorption temperature were studied. The Cr (VI) ions adsorption studies performed by mixing 0.2 g of wet Amberlite IRA 420 with 50 mL of 300 mg/L Cr (VI) ions. The mixture was agitated at R.T using magnetic stirrer for 30 min then left to settle for 1 min to separate the adsorbent out of the liquid phase. The Cr (VI) ions concentration at mg/L, before and after the adsorption, for each solution, was determined by measuring the absorbance at the maximum wavelength ($\lambda_{max} = 380 \text{ nm}$) using UV-Visible spectrophotometer and multiply by 156.25 constant extracted from the slope of the standard curve. Cr (VI) ions removal percentage calculated according to the following formula:

$$\text{Cr (VI) ions removal (\%)} = \left[\frac{(C_o - C_t)}{C_o} \right] \times 100 \quad (2)$$

C_o and C_t are the initial and the final concentrations (mg/L) of Cr (VI) ions at zero and particular time.

To elucidate the uptake capacity of the metal ion, the adsorption amounts per gram of AMB was evaluated from the change in solution concentration using Eq. (3):

$$Q(\text{mg/g}) = V(C_o - C_t) / M \quad (3)$$

where Q is the uptake capacity (mg/g), V is the volume of the Cr (VI) ions solution (ml), and M is the mass of the AMB (g).

2.5. Desorption experiment

Desorption experiments have been carried out in a batch process by using desorbant solution. The variable parameters namely; the desorbants type (NaCl and NaOH), initial desorbant concentration, and desorption time have been studied. The Cr (VI) ions desorption studies have been performed by mixing 0.2 g of wet Cr-Amberlite IRA 420 with 50 mL

of NaOH. The mixture has been agitated at R.T using magnetic stirrer for 60 min then left to settle for 1 min to separate the adsorbent out of the liquid phase. The Cr (VI) ions concentration at mg/L has been determined during the desorption process as mentioned above.

2.6. Reuse ability

The capacity to reuse the Amberlite for adsorption chromium (IV) ions has been examined by conducting consecutive adsorption-desorption cycles and following the changes in the removal efficiency of chromium (IV) ions. Chromium (IV) ions solution (150 mg/L) has been used in the adsorption experiments. NaOH solution (0.5 M) has been utilized for the desorption experiments.

2.7. Fourier transform infrared spectroscopic analysis

The Fourier transforms infrared spectroscopic (FTIR) spectra of the Amberlite, and Amberlite-Cr particles have been recorded with an FTIR spectrometer in the spectral range 4,000–500 cm^{-1} .

2.8. Thermogravimetric analysis

The thermal analysis of the Amberlite and Amberlite-Cr particles has been carried out using a thermogravimetric analyzer (TGA). About 6 mg of particles have been placed in ceramic pans of TGA instrument and analyzed over the temperature range 0°C–600°C with the heating rate of 10°C/min under dry N_2 flow at the rate of 10 mL/min.

3. Results and discussion

The sorption process of Cr (VI) ions is dependent on pH of the equilibrium solution. The hexavalent chromium exists primarily as chromic acid (H_2CrO_4) and its salts, hydrogen chromate (HCrO_4^-) and chromate (CrO_4^{2-}) ions depending on the sample pH [17]. The dichromate ion ($\text{Cr}_2\text{O}_7^{2-}$) is formed when the concentration of chromium exceeds approximately 1 g/L. In the solution within the whole range of concentrations and when $\text{pH} > 6.5$, only CrO_4^{2-} ions exist. In the pH range from 0 to 6.5, HCrO_4^- and $\text{Cr}_2\text{O}_7^{2-}$ ions are predominant [18]. Accordingly, the Cr (VI) ions in our case where the concentration used is less than 1 g/L, and the solution pH is below 6.5 will be present as HCrO_4^- and $\text{Cr}_2\text{O}_7^{2-}$ ions.

3.1. Adsorption process

A standard curve of Cr (VI) ions concentration has been shown in Fig. 1. From the curve's slope, the constant has been calculated and has been found equal to 156.25.

Factors affecting the Cr (VI) ions removal process using Amberlite have been investigated. These factors included the Cr (VI) ions concentration, the adsorption time, temperature, and pH, the agitation speed and finally the adsorbent dose. The effect of variation of the factors as mentioned above on the removal percentage of Cr (VI) ions and the adsorbent capacity of the Amberlite has been studied, and the obtained results have been presented in the following.

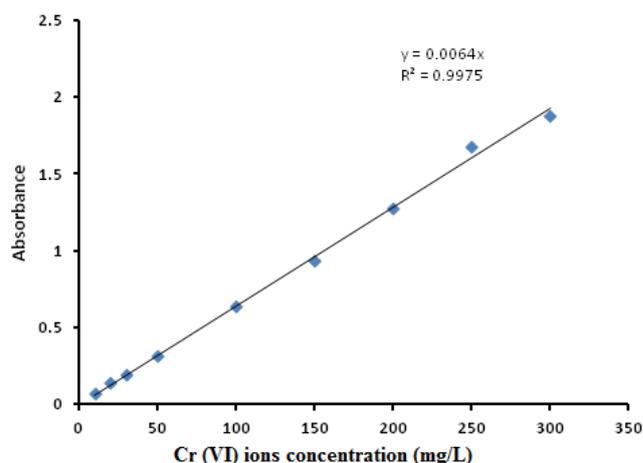


Fig. 1. Standard curve for Dichromate concentration.

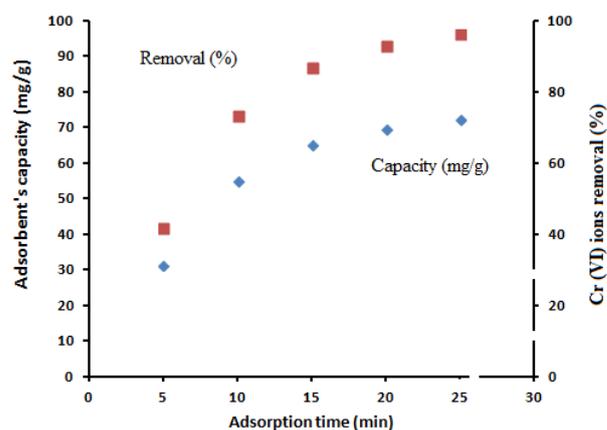


Fig. 2. Effect of adsorption time on the removal percentage and adsorbent capacity (50 mL 300 mg/L Dichromate solution, 0.2 g AMB, R.T., pH 5.6, 200 rpm).

3.1.1. Effect of the adsorption time

Fast adsorption is one of the key features of the efficient adsorbent. Accordingly, the effect of variation of the adsorption time on the removal percentage and adsorbent capacity has been presented in Fig. 2.

From the illustrated data in Fig. 2, it is clear that fast adsorption process has been recognized through the removal of 96% of Cr (VI) ions within 25 min. The Cr (VI) ions concentration after adsorption has been dropped sharply to 40 mg/L only after 15 min of the adsorption time. The rate of the adsorption process starts to decrease after. That behavior has been expected since the remaining Cr (VI) ions in the solution phase became very low and so the ions exchange site over the Amberlite beads (solid phase) which represent the driving forces of the adsorption process.

On the other hand, the adsorbent capacity shows a similar behavior to the Cr (VI) ions removal (%) where has been increased rapidly in the early time of the adsorption process then started to level off.

3.1.2. Effect of the Cr (VI) ions concentration

The effect of variation the Cr (VI) ions concentration on the removal percentage and the adsorbent capacity has been presented in Fig. 3. From the illustrated results, it is clear that the removal percentage has not been affected significantly by variation of the Cr (VI) ions concentration. The removal percentages were ranged between 90% and 92%.

On the other hand, an apparent increase of the adsorbent capacity has been observed. That increment could be explained by increase the concentration gradient between the Cr (VI) ions phase (solution phase) and the Adsorbent phase (solid phase). This gradient acting as the driving force of the Cr (VI) ions toward the Amberlite surface and the interior pores in addition to the hydrophobic and the ions exchange interactions. The individual or the synergetic effect of all the three factors leads finally to the obtained results.

3.1.3. Effect of the adsorption temperature

Energy consumption is one of the main concerns of the efficiency and the practical points of view while performing any adsorption process. The effect of variation the adsorption's temperature on the removal percentage and the adsorbent capacity has been presented in Table 2.

From the tabulated results, it is clear that the adsorption temperature sets back the adsorption process which reflects its exothermic nature. Both the removal percentage and the adsorbent

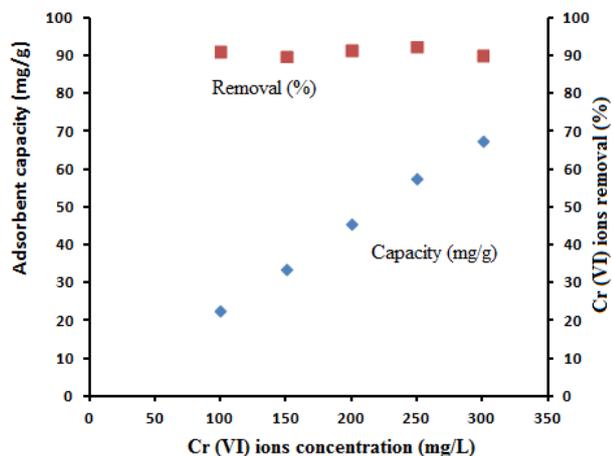


Fig. 3. Effect of Dichromate concentration on the removal percentage and adsorbent capacity (50 mL Dichromate solution, 0.2 g AMB, 30 min, R.T., pH 5.6, 200 rpm).

Table 2

The effect of variation the adsorption temperature on the removal percentage, and the capacity of adsorbent (Dichromate concentration; 1,000 ppm)

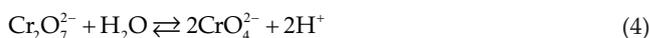
Temperature (°C)	Removal percentage (%)	Adsorbent capacity (mg/g)
35	36.3	90.75
45	29	72.5
52	23.3	58.25

capacity have been decreased linearly with the increase of the adsorption's temperature. That decrement could be explained by the early stage fast adsorption at high temperature which reduces the concentration gradient between the Cr (VI) ions liquid phase and the Amberlite solid phase. Accordingly, the principal driving force of the adsorption process has been reduced and the adsorbed Cr (VI) ions on the beads surface create a "repulsion layer" which retards the diffusion of the soluble Cr (VI) ions from the liquid phase to the free ions exchange sites in the Amberlite beads interior. From the cost point of view, the highest adsorption at the lowest temperature considered as an advantage for the adsorption process.

3.1.4. Effect of the adsorption pH

Aqueous solution pH is a critical parameter as it strongly affects metal sorption, the surface charge of the adsorbent, the degree of ionization and the speciation of adsorbate species [19].

Chromium (VI) ions exist in the anionic forms ($\text{Cr}_2\text{O}_7^{2-}$, HCrO_4^- or CrO_4^{2-}) in water depending on the pH and concentration. In highly acidic media (pH < 1), the Cr (VI) ions exist mostly as H_2CrO_4 ($\text{pK}_1 = 6.51$ and $\text{pK}_2 = 5.65$). At pH between 2 and 6 there is equilibrium between $\text{Cr}_2\text{O}_7^{2-}$ and HCrO_4^- ions. Under alkaline conditions (pH > 8), it exists predominantly as chromate anions. The equilibrium between chromate and dichromate ions in water can be shown as the following equation [15]:



The Cr (VI) ions adsorption has been carried out at different pH of the solution varied from 2 to 7, constant initial concentration, contact time, and temperature to optimize the pH for maximum removal efficiency. The maximum removal percentage has been recognized at pH range 3.0–4.0; Fig. 4. Further increase in the solution pH up to 7.0, the removal percentage has been decreased, because OH^- ions increased the hindrance of diffusion, as well as some of the trivalent cations, may react with OH^- ions and precipitated and thereby decreased the free metal ions available in the solution [20].

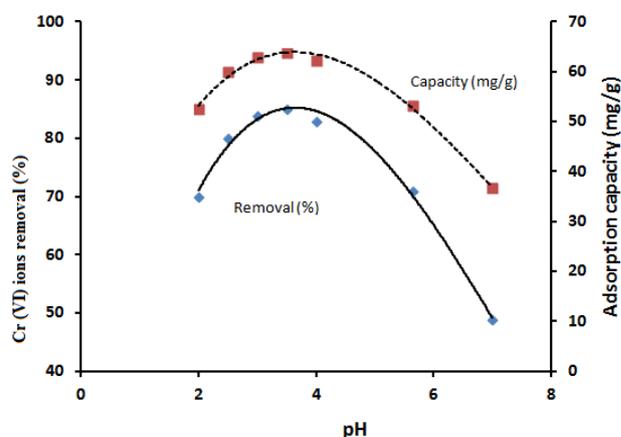


Fig. 4. Effect of adsorption pH on the removal percentage and adsorbent capacity (50 mL 300 mg/L Dichromate solution, 0.2 g AMB, R.T., 10 min, 200 rpm).

3.1.5. Effect of the agitation speed

The effect of variation the agitation speed from 200 to 300 rpm on the removal percentage and adsorption capacity of Cr (VI) ions has been studied (Table 3). From the table, it is clear that a slight enhancing effect of increasing the agitation speed from 200 to 300 rpm on the removal percentage of Cr (VI) ions has been observed. That enhancement is because Cr (VI) ions, through their transportation to the solid phase, meet resistance in the liquid phase, through the boundary layer. The motion induced through the agitation of suspensions during experiments leads to a decrease in the boundary layer thickness and a consequent decline in the transportation resistance of Cr (VI) ions [21]. That increases the transfer rate of the ions, and thus, the Cr (VI) ions removal percentage.

The adsorption capacity of the resin has been expressed in Table 3. From the table, it is clear that the resin capacity was found equal to 58.58 (mg/g) after 10 min contact time at 300 rpm compared with 55.5 and 53.6 (mg/g) at 250 and 200 rpm, respectively. The high affinity of the ion exchange sites on the ions exchanger beads for the Cr (VI) ions in the solution eliminates the effect of variation the agitation speed to great extent. That considered as an advance from the economic point of view based on saving energy.

3.1.6. Effect of the adsorbent dose

The effect of variation the adsorbent dose on the removal percentage and the adsorbent capacity have been studied and presented in Fig. 5.

The illustrated results in Fig. 5 reveal that the Cr (VI) ions removal (%) has been increased with increase the adsorbent dose while the remaining Cr (VI) ions concentration decreased. Within the studied adsorption's time, 4% and 90% of the Cr (VI) ions have been removed using 0.5 g and 0.2 g of the adsorbent. On the other hand, it is clear that the adsorbent's capacity increases with increase the adsorbent dose. It is logic enough since increase the adsorbent's dose increase the available active sites for adsorption the Cr (VI) ions. The adsorption capacity increase tends to level off due to the limitation of the available Cr (VI) ions.

3.2. Desorption process

Desorption experiments have been performed with the purpose of reusing the adsorbents and also to understand the mechanism of adsorption. If water can desorb the adsorbant, it can be concluded that the attachment of the adsorbant onto the adsorbent is by weak bonds. If the strong electrolyte, such as NaCl, HCl or NaOH can desorb the adsorbant, it can

Table 3
Effect of the agitation speed on the removal percentage and adsorbent capacity

Agitation speed (rpm)	Removal Percentage (%)	Adsorption capacity (mg/g)
200	71.4	53.55
250	74.0	55.50
300	78.1	58.58

be concluded that the attachment of the adsorbant onto the adsorbent is by ion exchange or electrostatic attraction [22].

3.2.1. Effect of the desorbant type

Desorption of the adsorbed Cr (VI) ions using water, NaCl, and NaOH solutions has been tried, and the data have been presented in Fig. 6. No Cr (VI) ions desorption has been noticed using water. From the Figure, it is clear that both NaCl and NaOH are capable of Cr (VI) ions desorption sufficiently within 60 min where 97% and 99% of adsorbed Cr (VI) ions have been detected in the desorbing solution. Fast desorption rate has been noticed using NaOH where 98% desorption achieved after only 15 min compared with 60 min using NaCl. NaOH has been used for further investigation of the desorption process.

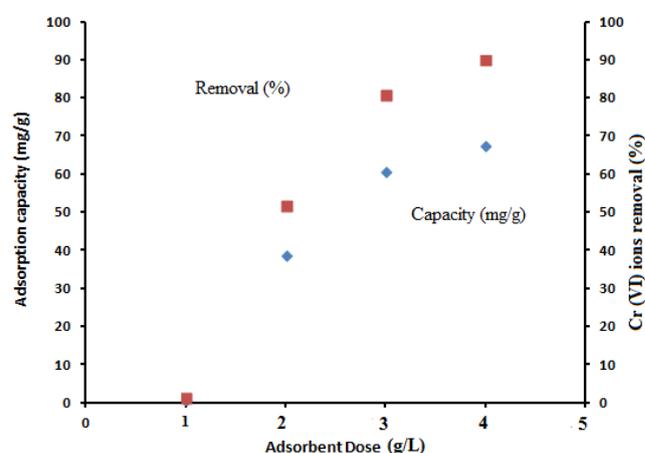


Fig. 5. Effect of adsorbent dose on the removal percentage and adsorbent capacity (50 mL 300 mg/L Dichromate solution, R.T., pH 5.6, 10 min, 200 rpm).

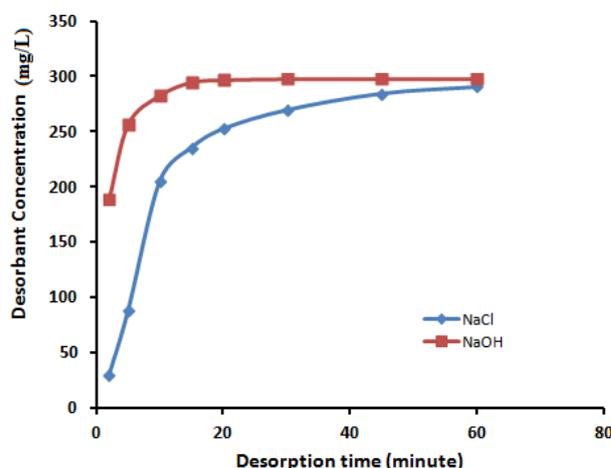


Fig. 6. Effect of desorbant agent type on the desorption percentage (0.2 g wet AMB-Cr, 50 mL 1 M NaCl and 1 M NaOH solutions, R.T., 60 min, 300 rpm).

3.2.2. Effect of the desorbant concentration

The effect of variation the NaOH concentration on the Cr (VI) ions desorption has been presented in Fig. 7. From the Figure, it is clear that 92% of the adsorbed Cr (VI) ions has been desorbed using 0.25N NaOH after 30 min. Further increase of the NaOH concentration to 1N, increased the desorption percentage to reach about 98%. From the results, we can be observed that the low NaOH concentration could be used efficiently but with prolongation of desorption time.

3.2.3. Effect of the desorption time

Fig. 8 shows the effect of variation of desorption time on the desorption process of Cr (VI) ions. From the Figure, it can be seen that fast linear desorption has been noticed with 12 min where around 50% of the adsorbed Cr (VI) ions have been detected in the elution. Longer desorption time

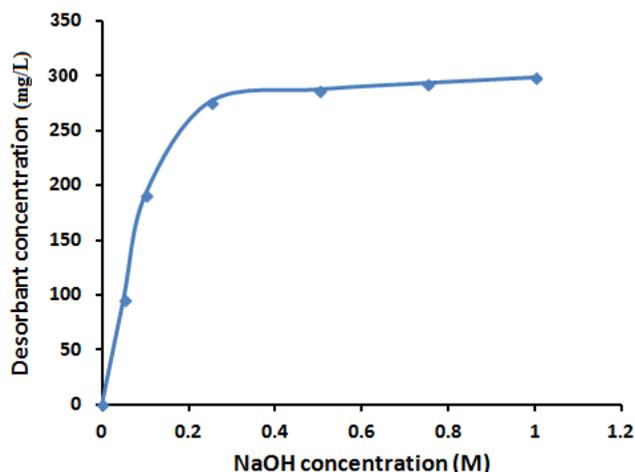


Fig. 7. Effect of desorbant concentration on the desorption percentage (0.2 g wet AMB-Cr, 50 mL NaOH solution, R.T., 60 min, 300 rpm).

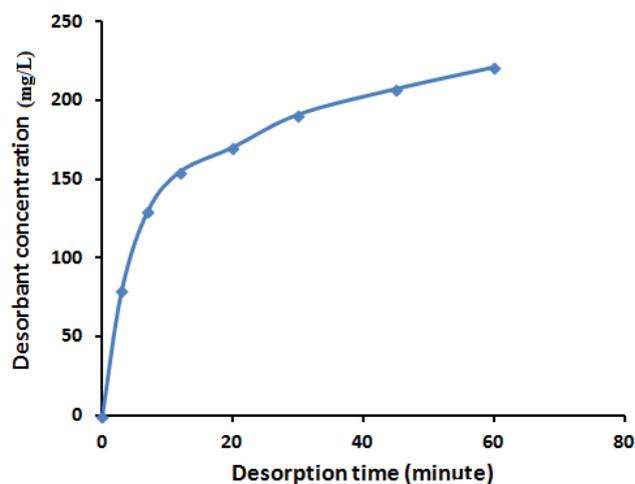


Fig. 8. Effect of desorption time on the desorption percentage (0.2 g wet AMB-Cr, 50 mL 0.1 M NaOH solution, R.T., 300 rpm).

increases the desorption percentage but with a lower rate. After 60 min, around 75% of the adsorbed Cr (VI) ions have been detected in the elution. The fast desorption step could be referred to desorption of the surface adsorbed Cr (VI) ions. The adsorbed Cr (VI) ions in the beads interior take more time to leach out due to the time needed for NaOH diffusion. It is worthy to mention here that the Cr (VI) ions leached as CrO_4^{2-} ions due to the alkalinity of the desorption medium.

3.2.4. Reuse ability

The main goal of the regeneration process is to reuse the adsorbent materials as much as possible. Desorption cycles of the adsorbed Cr (VI) ions have been tried under the same conditions using 0.5N NaOH eluting solution (Fig. 9). During 11 successive cycles, the removal percentage ranged between 84% and 99% of 150 mg/L. The adsorption percentage almost has not been affected during the first three cycles. After that, a gradual decrease has been observed where the minimum adsorption percentage has been obtained after the fifth cycle and remains constant for the following three cycles. At the eighth cycle, a gradual recovery of the adsorption efficiency has been recognized to reach 95% at the eleventh one. Longer desorption time at a higher temperature than 25°C may help in maintaining the adsorption efficiency unaffected.

3.3. Matrix characterization

3.3.1. FT-IR analysis

The characteristic bands for the AMB polymeric backbone, poly (styrene-co-divinylbenzene), have been shown in the spectrum at 700, 800, 900, 1,000, 1,400–1,600, and 2,900–3,000 cm^{-1} [23]. The characteristic band for Cr (VI) recognized at Cr (VI) adsorbed onto AMB beads (AMB-Cr) at 755 cm^{-1} ; (Fig. 10).

3.3.2. TGA analysis

The thermal behavior of the amberlite (AMB) and Amberlite-Cr beads (AMB-Cr) has been studied by TGA

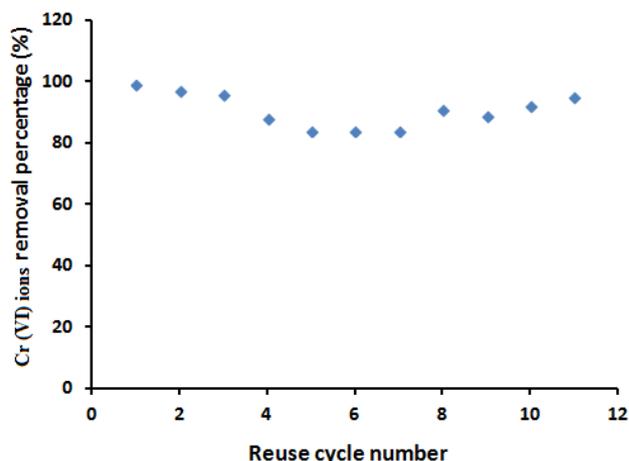


Fig. 9. Effect reuse cycles on the adsorption percentage (0.2 g wet AMB-Cr, 50 mL 0.5 M NaOH solution, R.T., 60 min, 300 rpm).

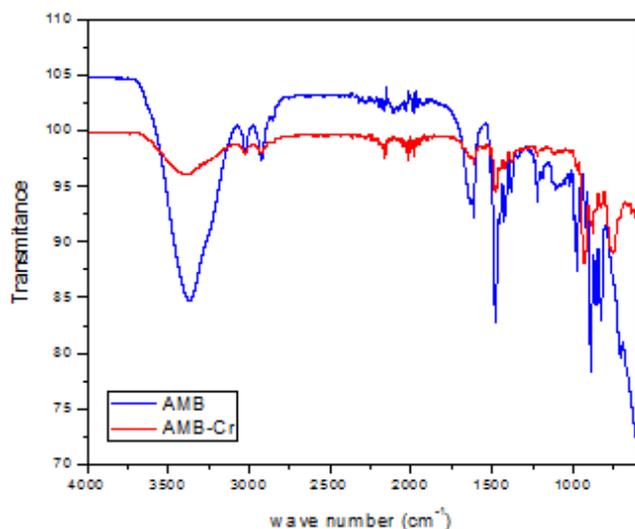


Fig. 10. FTIR spectra of AMB and AMB-Cr particles.

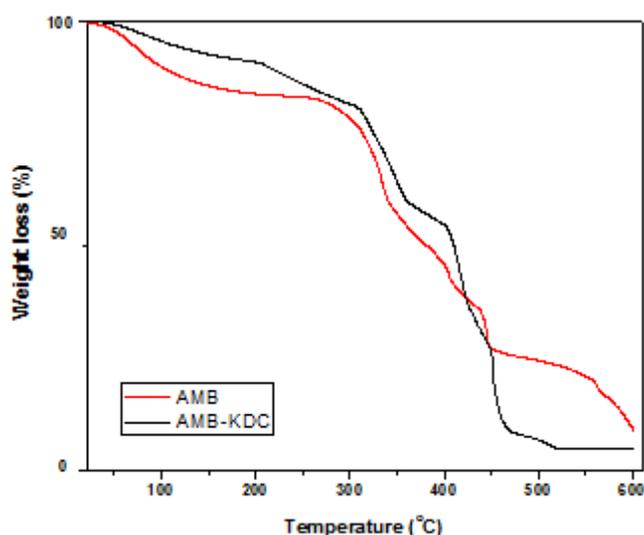


Fig. 11. TGA thermo-grams of AMB and AMB-Cr particles.

shows a particular “step weight” loss (Fig. 11). In the first step (25°C–250°C), the constant weight loss (15%) has been noticed for AMB, which attributed to the evaporation of physically adsorbed general volatiles in the polymer. This step was shorter in the case of AMB-Cr (25°C–200°C) where only 10% weight loss has been detected. In the second step (250°C–440°C) for AMB, the weight loss (55%) has been attributed to the depolymerization of the polystyrene chains and the degradation of styrene oligomers. The second step for AMB-Cr (200°C–440°C), the weight loss is 65%. In the third step (440°C–600°C), the weight loss (20%), is mostly attributable to divinyl benzene degradation [23,24]. The weight loss reached 15% at the third step for AMB-Cr. Such kind of the observed thermal stability improvement has resulted from the interaction of the Cr (VI) ions with the exchange sites over the Amberlite beads which considered as a crosslinking.

4. Conclusion

Poly (styrene-co-divinyl benzene) anions exchanger (Amberlite IRA-420) has been used for the removal of chromium (VI) metal ions from dichromate contaminated water. Operational conditions such as the chromium (VI) metal ions concentration, adsorption time, adsorption temperature, adsorption pH, agitation speed, and finally the adsorbent dosage have been investigated, and its impact on the removal process efficiency has been presented. Chromium (VI) metal ions removal percentage ranged from 90% to 92% for concentrations ranged from 0 to 300 mg/L. Adsorption temperature has an adverse impact on the adsorption process. The acidic medium of the pH range from 3.0 to 4.0 has been found as the optimum for the adsorption process. The agitation speed slightly affected the adsorption process in a positive manner where increased the adsorption percentage from 71% to 78% with increase the agitation speed from 200 to 300 rpm. Moreover, FT-IR and TGA analyses have been presented for the original Amberlite and chromium (VI) metal ions adsorbed Amberlite particles. Regeneration of the chromium (VI) metal ions adsorbed Amberlite particles has been studied. Over 97% of the adsorbed chromium (VI) metal ions have been desorbed using sodium chloride or hydroxide solutions within 60 min. Higher desorption rate has been noticed using sodium hydroxide. Finally, the reuse ability has been investigated.

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