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Tailored removal of zinc and chrome ions by the adsorption onto nonwoven ACF (activated carbon fiber) prepared from PAN (polyacrylonitrile) waste

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ABSTRACT

The discharge and disposal of heavy metal ions have become urgent environmental issues. Among methods to remove and recover heavy metals from industrial or mining effluents, adsorption has been gaining more and more attention due to its satisfactory efficiency and environmental friendliness, involving numerous typical adsorbents. In the present study, this process in batch mode was designed and applied to remove heavy metals using activated carbon fiber (ACF) from waste polyacrylonitrile (PAN) fiber. The removal of Cr(VI) and Zn(II) by nonwoven ACF derived from PAN waste was the aim of this study. The effect the adsorbent pH, contact time, adsorption temperature, and initial metal ions concentrations were studied. The surface of ACF after adsorption process was investigated by Fourier-transform infrared spectroscopy and scanning electron microscopy. Both pH and temperature of adsorbent strongly affect the adsorption of Zn onto ACF, whereas adsorbent pH is the major factor in the adsorption of Cr(VI) onto ACF. The maximum removal efficiency at 30°C for Zn(II) was 75% at pH 10 and the maximum removal efficiency for Cr(VI) at pH 2 was 86%. Increasing the adsorption temperature enhance the removal efficiency as the removal efficiency of Zn(II) increased from 75% to 97%, while for Cr(VI) increased from 86% to 95%. On the other hand, the adsorption capacity of Zn(II) increased from 29 up to the maximum of 45 mg/g. Also, Cr(VI) increased from 41 up to the maximum of 46 mg/g. There was significant decrease in the value of distribution coefficient of Cr(VI) with increasing pH of adsorbent solution with 98% and the opposite situation with Zn(II) with 98.6%. Comparing the values of distribution coefficient for the sorption of Cr(VI) and Zn(II) ions in equilibrium conditions, it was clear that these metal salts are far from each other. This indicates difference in their adsorption properties, which gives a chance for selective adsorption for these two metal ions.

Keywords: Activated carbon fiber; Wastewater treatment; Adsorption; Heavy metals; Cr(IV); Zn(II)

1. Introduction

Nowadays, removal of heavy metals from industrial and domestics water is considered as vital concern to decrease water pollutions. Some industries such as chemical, textile, electroplating and metal finish, and paper manufacturing processes are among the main sources of heavy metal pollutants. These heavy metals are mainly discharged into wastewater. Increasing the concentration of such metals will increase the harmful pollutants and environmental risk [1,2]. This undesirable effect could be avoided by chemical techniques, such as reverse osmosis, precipitation, and ion-exchange. Although precipitation is the most applicable and economical technique but it produced huge amount of

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precipitate sludge that needs furthermore treatments. Both ion exchange and reverse osmoses can effectively minimize the metal ions, but their use were limited because of its high cost and limited to the pH range for the ion-exchange resin [3]. Therefore, a need for alternative technique for reduction of the heavy metals is required.

Activated carbon fiber (ACF) is considered as excellent operative adsorbents for a wide diversity of organic and inorganic pollutants which are mainly found in industrial effluents. The large adsorption capability of ACF is mainly due to the high pore volume and large surface area as well as the presence of different functional groups on the surface [4–6]. The low cost of activated carbons and easiness of their activation procedures make their application as adsorbents for toxic heavy metals very attractive. ACF was especially known for the effectiveness in removing organic chemicals from wastewater and removing inorganic heavy metal as well as the wastes of dye pollutants [7,8].

The rapidly draining of water resources and increasing of population have reduced the drinking water resource all over the world. The development of cost-operative and stable materials and methods for offering the fresh water in adequate amounts is the need of the water industries.

It should be noted that the maximum tolerable concentration of zinc in drinking water as 5.0 mg/L as suggested by World Health Organization (WHO) [3]. Chromium is present in two oxidation states (hexavalent and trivalent) in natural aqueous environment pollutants according to the Environmental Protection Agency (EPA). Cr(III) is essential for living organisms, whereas Cr(VI) is toxic, mutagenic and carcinogenic. The extreme levels licensed in wastewater are 5 mg/L for Cr(III) and 0.05 mg/L for hexavalent Cr(VI) [9,10].

Chromium existing into natural waters by a diversity of industrial wastewater including those from textile, electroplating, metal finishing, and chemical industries. Chromium, which is very toxic pollutants as defined by the EPA, present as Cr(VI) in the form of oxidized species, such as chromates $(CrO_4)^{2-}$, dichromate $(Cr_2O_7)^{2-}$ and bichromate $(HCrO)^{4-}$, depending on the pH and Cr(VI) concentration. In the pH range 2.0–6.5 Cr(VI) exist as bichromate and at pH 8.0 it exists as chromate anions [10,11]. Due to its high solubility, Cr(VI) is one of the most hazardous, since it can accumulate in the food chain and cause serious problems in the human health. The removal of Cr (VI) by activated carbon has been investigated in order to improve the adsorption efficiency. The electrostatic attraction and complexation with the functional groups on the activated carbon surface were found to be responsible for the removal of Cr(VI). Also, it was proposed that Cr(VI) is reduced to the less toxic Cr(III) by adsorption onto activated carbon under acidic conditions [9].

ACF prepared from polyacrylonitrile (PAN) precursor has appealed much attention due to it's high adsorption performance as compared to other activated carbons precursor [4]. The present work is devoted to preparing ACF from waste PAN fiber and to evaluate their ability to remove heavy metals. The main focus will be on the removal of zinc and chromium metal salts by using different adsorption conditions with a view to investigate the adsorptive capacity of ACF by varying pH of the solution, agitation time, adsorbent temperature and initial ion concentration of metal salts.

2. Experimental

2.1. Preparation of activated carbon fiber

ACFs were prepared from the waste of PAN fiber. The fibers were pre-treated at temperature range of 200°C–300°C. It is followed by carbonization step at 700°C–900°C under inert atmosphere. The final step was the activation at 800°C–1,000°C under mixture of steam and/or CO_2 [4]. The surface area characterization of ACF was performed on ASAP 2420 accelerated surface area (Micromeritics Instrument Corporation, USA).

2.2. Materials and methods

All the reagents used were of AR grade chemicals. Stock solutions of the test reagents were prepared by dissolving zinc acetate, and potassium dichromate in distilled water. The pH of the investigated solutions was adjusted using reagent grade dilute hydrochloric acid and sodium hydroxide (0.1 mol/L).

2.3. Adsorption procedure

Adsorption experiments were carried out in batch processes by agitating 0.05 g of oven dried nonwoven ACF samples with the prepared solutions of metal ions at different pH of the solution from 2 to 10 in a thermostat bath with a shaker (Julabo AW21). The effects of pH, contact time, and initial metal ions concentrations on the adsorption of Zn(II) and Cr(VI)onto ACF were studied. The nonwoven fabric samples were thoroughly mixed with 50 ml of known amount of investigated metal salts in a 100 ml Erlenmeyer flask, and the ACF samples were shaken in a water bath (200 rpm) at room temperature (30°C ± 1°C). Adsorption studies were carried out in a range of initial concentration (30–150 ppm) of metal salts. The agitation time (5–180 min) at the pH of maximum adsorption capacity was found (first experimental run in this study). At the end of each adsorption experiment, the solution was filtered through a filter paper and the concentration of the investigated metal salts in the filtrate was determined with Flame atomic absorption spectrometer (Agilent Technologies 200 Series AA) at wavelength 428.9 nm for Cr and 213.9 nm for Zn.

The properties of adsorption process were characterized by calculating the removal efficiency (R%), adsorption capacity (SC, mg/g) and distribution coefficient (D, L/g) using the following equations:

$$R=\left[\frac{\left(C_0-C_i\right)}{C_0}\right]\times 100 \tag{1}$$

$$\text{SC}=\frac{\left[\left(C_0-C_i\right)\times V\right]}{W} \tag{2}$$

$$D=\left[\frac{R}{100-R}\right]\times\frac{V}{W} \tag{3}$$

where C_0 is the initial concentration of the investigated metal ion in the solution, mg L^{-1}; C_i is the concentration of metal ion solution after the adsorption onto ACF, mg L^{-1}; *V* is the volume of solution, (L); *W* is the weight of the sorbent material (ACF) per gram.

2.4. Fourier-transform infrared spectroscopy

The chemical structure was determined using the Fourier-transform infrared spectroscopy (FTIR) spectrometer, model NEXUS 670, NICOLET USA. The measurements were carried in the spectral range from 4,000 cm^{-1} to 500 cm^{-1}. Reflection percentage measurement technique was applied (*R*%) to all investigated samples.

2.5. Scanning electron microscopy

Fabrics morphology was characterized by scanning electron microscopy (SEM) (JEOL-Model TSM T20).

3. Result and discussion

3.1. Effect of pH on the adsorption of Cr and Zn metal ions

It is prominent that pH is considered as an important factor affecting the adsorption of heavy metals. Determining of the optimum pH condition is very important since pH affects not only the surface charge of adsorbent, but also on the degree of ionization of adsorbate through reactions. In general, adsorption of metallic species increased with increasing pH in a certain range [12]. The removal of metal ions from the solution by ACF depends on the pH of the adsorption process due to the change on the surface of ACF. The effect of hydrogen ions concentration on the removal efficiency of Zn(II) and Cr(VI) by ACF was examined and represented in Figs. 1–3. The pH was varied from 2 to 10 and the adsorption temperature at of 30°C. The other parameters such as agitation time and initial ions concentration were kept constant; the duration of adsorption is at 60 min and the agitation speed is 200 rpm.

Fig. 1 shows that the increase in the pH of the solution from 2 to 10 caused a linear increase in Zn(II) removal efficiency, where the hydrogen ion concentration plays an important role in the solubility of the metal ions as well as adsorbent function groups during the reaction processes. The maximum removal efficiency of ACF for Zn(II) was 75% removal occurred at pH 10 and the total Zn(II) removal decreased to 4% at acidic pH of the bath at pH 2. This is due to the surface of ACF became deprotonated at pH 10, so increase the adsorption of zinc metal ion as result of increase the electrostatic interaction between the Zn positive ions and the negative hydroxyl groups on the surface of ACF [13] Also, ion exchange between the metal cations and H^+ ions at the surface of the ACF affects the adsorption features of ACF [14].

On the other hand, the maximum removal efficiency of ACF for Cr(VI) was 87% occurred at pH 2 and the total Cr(VI) removal decreased to 8.4% with increasing the pH of the bath at 10. Hexavalent chromium $(Cr_2O_7)^{2-}$ is related to the concentration of H^+ to a great extent, the higher concentration present at the higher the concentration of H^+. Therefore, increase the hydrogen ion concentration (pH < 7) will increase the protonation of ACF, so the adsorption efficiency for the chromate anions from the solution increases to great extent due to electrostatic attraction forces. With decreasing the H^+ concentration (pH > 7), the surface of ACF became deprotonated with increase in the hydroxyl ion on the surface of ACF, so repulsion between the hydrogen chromate anions and hydroxyl groups occurred [15]. Therefore, the Cr adsorption on ACF decreases.

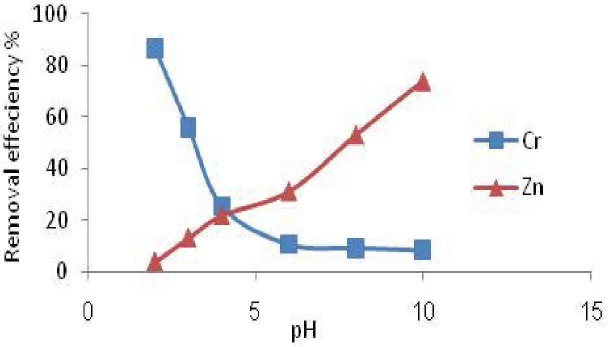
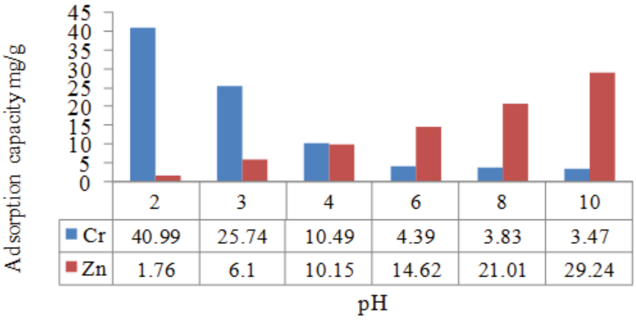


Fig. 1. Effect of pH on the removal efficiency of Zn(II) and Cr(VI) on ACF.



	2	3	4	6	8	10
🟦 Cr	40.99	25.74	10.49	4.39	3.83	3.47
🟥 Zn	1.76	6.1	10.15	14.62	21.01	29.24

Fig. 2. Effect of pH on the adsorption capacity of Zn(II) and Cr(VI) on ACF.

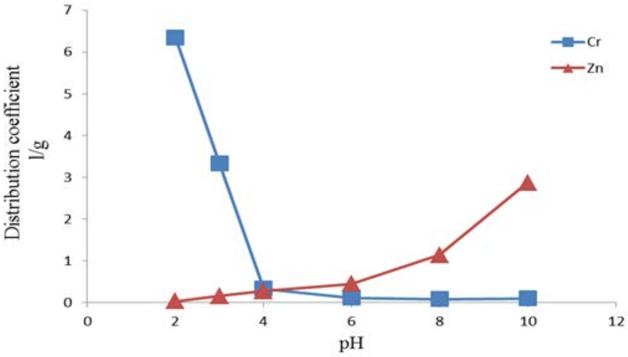


Fig. 3. Effect of pH on the distribution coefficient of Zn(II) and Cr(VI) on ACF.

The effect of pH on the adsorption capacity and distribution coefficient is shown in Figs. 2 and 3, respectively. The data presented in Fig. 2 shows that the maximum adsorption capacity for Cr(VI) occurred at pH 2 (41 mg/g), while the maximum adsorption capacity for Zn(II) occurred at pH 10 (29 mg/g).

Fig. 3 shows the distribution coefficient values of metal ions between adsorbent and liquid phase, which give a prediction about the possibility of selective adsorption from mixed solutions containing various metals. This selectivity is considered a significant characteristic of the adsorption process of ACF. It was clear that the data were in good agreement with the sorption capacity. There was significant decrease in the value of *D* in adsorption of Cr(VI) with increasing pH of the medium and the opposite situation with Zn(II), in other words the maximum adsorption for Cr(VI) occurred in acidic medium, while the maximum adsorption for Zn(II) occurred in basic medium. Comparing the values of distribution coefficient for the sorption of Cr(VI) and Zn(II) ions in equilibrium conditions, it was clear that they are far from each other which indicate that they are differ in their adsorption, give chance for selective adsorption for these two metal ions [16].

Activated carbon is amphoteric material, which can have positive or negative charges depending on the solution pH. Attraction between ACF and anionic or cationic ions is principally related to the surface characteristics. So, the pH is considered as an important factor to design the adsorption behavior of ACF according to what kind of metal salts needed to be removed. For example, more negatively charged ions are created and accumulated on the porous surface of ACF at higher pH values. As a result, it favors the uptake of more cationic groups due the decrease in the electrostatic repulsion between cations and the surface of activated carbon and vice versa [17]. It has been also postulated that, in the presence surface active groups on the surface of ACF, Cr(VI) removal from aqueous phase occurred through reduction from Cr(VI) to Cr(III) and Cr(VI) adsorption, resulting in predominant attachment of Cr(VI) species [1,18]. The oxidation of Cr(III)to Cr(VI) is not possible with the presence of ACF [9]. Therefore, Cr(VI) removal by ACF was based on the reduction of Cr(VI) to Cr(III) followed by Cr(III) adsorption. Cr(VI) adsorption was highly dependent on the pH value, and the best result was obtained under acidic conditions ($pH < 5$) because of the easier oxidation of C–H and C–OH to C–OH and C=O, respectively accompanied by the reduction of Cr(VI) to Cr(III). Under acidic conditions, the carbon surface was positively charged due to protonation, leading to electrostatic attraction with negatively charged Cr(VI) anions ($HCrO_4^-$ and $Cr_2O_7^{2-}$) [19,20].

3.2. Effect of initial metal ions concentrations on the adsorption properties

Fig. 4 presents the removal efficiency % for Cr(VI) and Zn(II) ions as function of initial metal ions concentrations. The metal concentrations were varied between 30 and 150 ppm and the weight of ACF used was 0.05 g. The pH parameters were kept at the optimum conditions for Cr(VI) and Zn(II) as shown before (i.e. pH 2 for Cr adsorption and pH 10 for Zn adsorption) for the other operational parameters, the temperature was kept at 30°C, the duration of adsorption is at 60 min and the agitation speed is 200 rpm. It is clear that, by increasing the concentration of initial metals solutions led to increase removal efficiency of Cr(VI) and Zn(II) until it reaches a constant removal efficiency (above 50 ppm) regardless increasing of the metals ions concentrations. This is predicted because more available sites for metals ions at those concentrations. This is credited to that the available active groups of the ACF become saturated when the metal ion concentration reached 70 ppm. Also, the entrance to the porous structure of ACF became unavailable due to the increased accumulation of metal ions on the surface of ACF [21,22]. The highest removal efficiency of Cr(VI), Zn(II) was 87%, 74%, respectively.

Figs. 5 and 6 show the consequence of initial concentration of metal ions on the amount of metal ions retained in the surface of ACF. It was noticed that the amount of metal ions retained in the porous structure of ACF increased with increasing the concentration up to 70 ppm. After this concentration, there is a slight increase occurs with increasing the concentrations of metal ions, which is expected due to the increase in charge accumulation on the surface of ACF as well as increase in the repulsion of ions carrying the same charge. This effect is more clearly appeared with chromate anions than in (Zn^{++}) as shown in Figs. 5 and 6.

3.3. Effect of temperature on the adsorption of metal ions

Temperature has an important role on the adsorption of Zn(II) and Cr(VI) onto ACF. The effect of the temperature on the removal efficiency was studied within the range of 25°C–80°C. It can be seen from Figs. 7–9 that, increase the temperature from 25°C up to 80°C has a significant effect on the removal efficiency and adsorption capacity of Zn(II) and Cr(VI). The removal efficiency of Zn metal ions increased from 74% to 97%, while for Cr ions increased from 86% to 95%. On the other hand, the adsorption capacity of Zn(II) increased from 29 to 45 mg/g and for Cr(VI) increased from 41 to 46 mg/g. The increase in adsorption capacity of ACF with temperature may be caused by the enhancement of the swilling ability of the porous structure of ACF as well as increase the movement of metal ions. As a result, the metal ions can easily penetrate into the porous network structure of ACF and increase the adsorption of metal ions into the pores of ACF. The distribution coefficient between the adsorbed Zn and Cr on ACF and un-adsorbed Zn and Cr (still in the solution) is shown in Fig. 9. It shows that

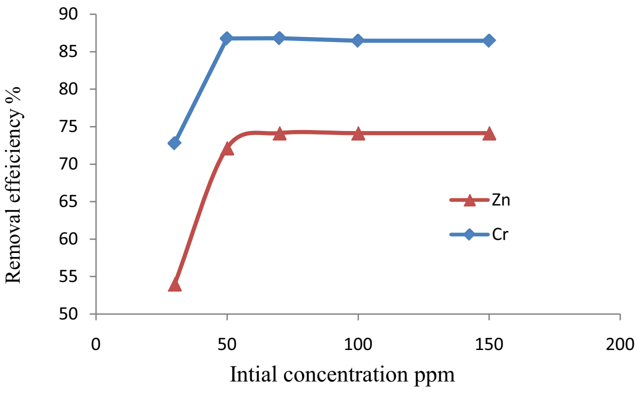


Fig. 4. Effect of initial metal ion concentration on the removal efficiency% of Zn(II) and Cr(VI).

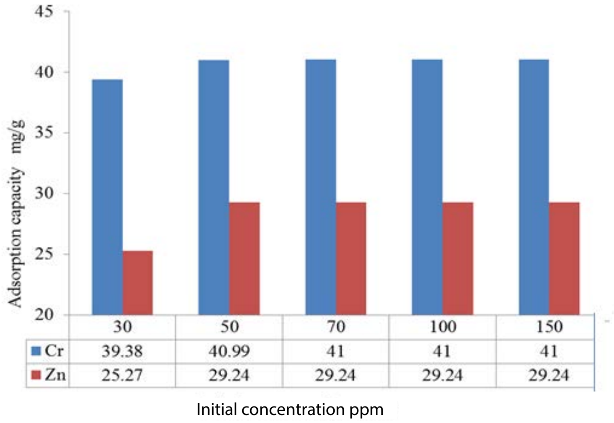


Fig. 5. Effect of initial metal ion concentration on the adsorption capacity of Zn(II) and Cr(VI) on ACF.

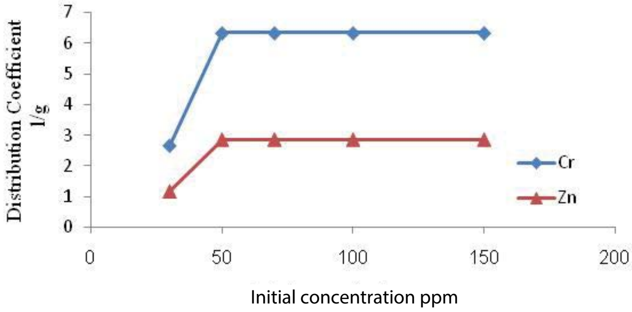


Fig. 6. Effect of initial metal ion concentration on the distribution coefficient of Zn(II) and Cr(VI) on ACF.

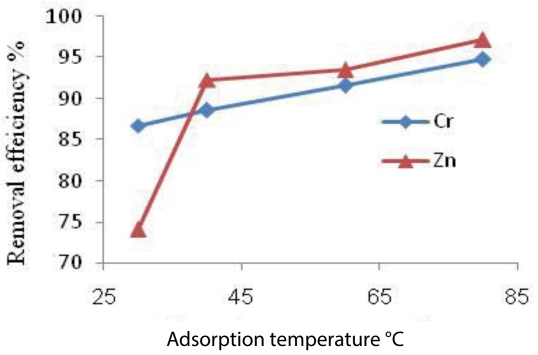


Fig. 7. Effect of adsorption temperature on the removal efficiency of Zn(II) and Cr(IV) on ACF.

how drastically the increase in the temperature favors the presence of the metal ions in the adsorbed form onto ACF rather than the soluble forms (i.e. increase the adsorption onto ACF).

It was noticed that there is significant enhancement of the removal efficiency and adsorption capacity in case of Zn(II) comparing with Cr(VI). This can be explained and concluded that the type of adsorption of two metal cations differ from each other. So, the interaction between the ACF and Zn(II) differ from the interaction between Cr(VI) and ACF. Both pH and temperature can strongly affect the adsorption process of Zn onto ACF, whereas the role of pH is the major factor in the adsorption of Cr(VI) onto ACF.

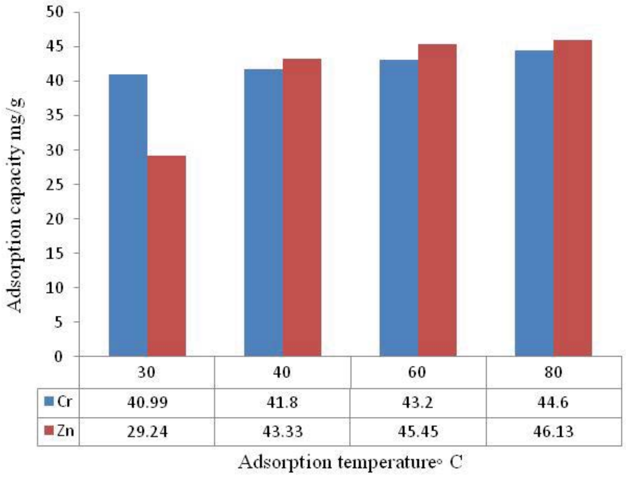


Fig. 8. Effect of adsorption temperature on the adsorption capacity of Zn(II) and Cr(VI) on ACF.

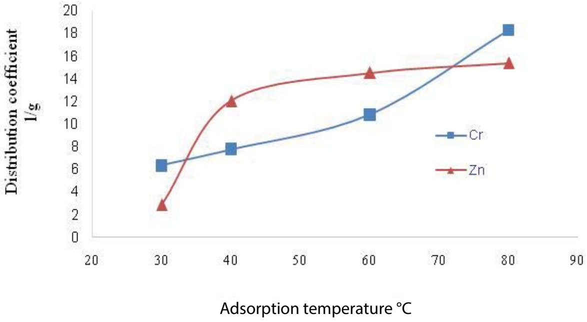


Fig. 9. Effect of adsorption temperature on the distribution coefficient of Zn(II) and Cr(VI) on ACF.

3.4. Effect of contact time on the adsorption of metal ions

The removal of both Cr(VI) and Zn(II) were also studied as a function of contact time as shown in Fig. 10. It is studied at the optimum condition of initial metal concentration and pH. It was noticed that the removal efficiency of Cr(VI) and Zn(II) increased with increasing the contact time and then become almost constant regardless of the duration. It was noticed that at 5 min duration, the removal efficiency of Cr and Zn were 54% and 40%, respectively. At 60 min, it reached to highest values of 86.5% for Cr and 74% for Zn.

Fig. 11 shows the influence of contact time on the adsorption capacity of ACF, which determined the amount of metal ions retained in the ACF. It was noticed that the amount of metal ions retained in ACF increased with increasing the contact time. The maximum value was achieved within 60 min to reach 50 and 30 mg/g for Cr(VI) and Zn(II), respectively. The adsorption capacity ACF increased rapidly up to 60 min. After that, the adsorption efficiency is almost constant for both Cr(VI) and Zn(II). The trend also clearly appears in Fig. 12 which explains the effect of contact time

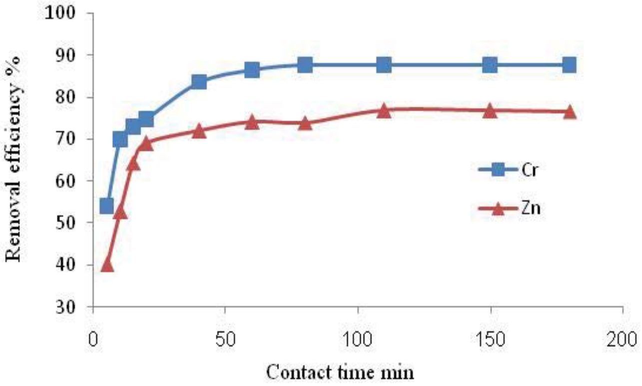
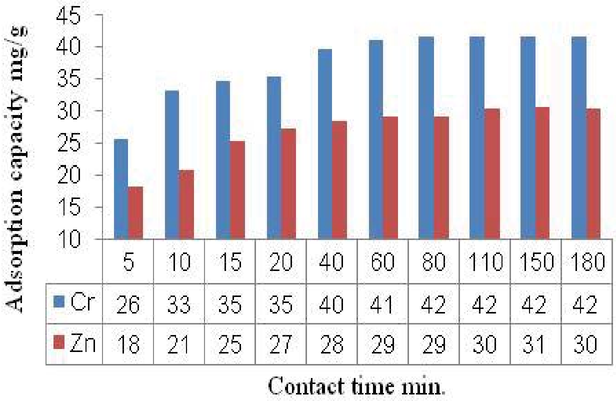


Fig. 10. Effect of contact time on the removal efficiency % of Zn(II) and Cr(IV) on ACF.



	5	10	15	20	40	60	80	110	150	180
■ Cr	26	33	35	35	40	41	42	42	42	42
■ Zn	18	21	25	27	28	29	29	30	31	30

Fig. 11. Effect of contact time on the adsorption capacity of Zn(II) and Cr(VI) on ACF.

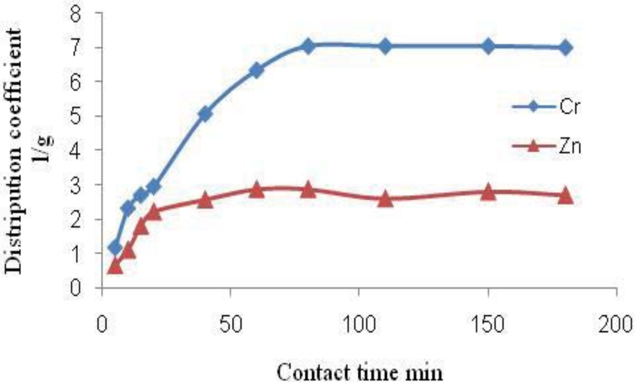


Fig. 12. Effect of contact time on the distribution coefficient Zn(II) and Cr(VI) on ACF.

on the distribution coefficient. This can be referred to the competition between the adsorbate molecules for the adsorption sites available onto the porous ACF. The Competition between the metal ions increases when the surface coverage of the ACF increases as the contact time increases. This will slow down the adsorption capacity of ACF and give constant adsorption properties directly above 60 min. Therefore, the optimum contact time was affected by the ratio of the number of adsorption sites to the number of metal species.

3.5. Scanning electron microscopy

SEM was used to examine the surface topography of ACF before and after metal adsorption of metal ions as shown in Fig. 13. Blank sample appear as filamentous smooth surfaces. The surface of ACF after adsorptions is characterized by the formation of some precipitations on the surface of ACF. These precipitations and granulation are noticeably

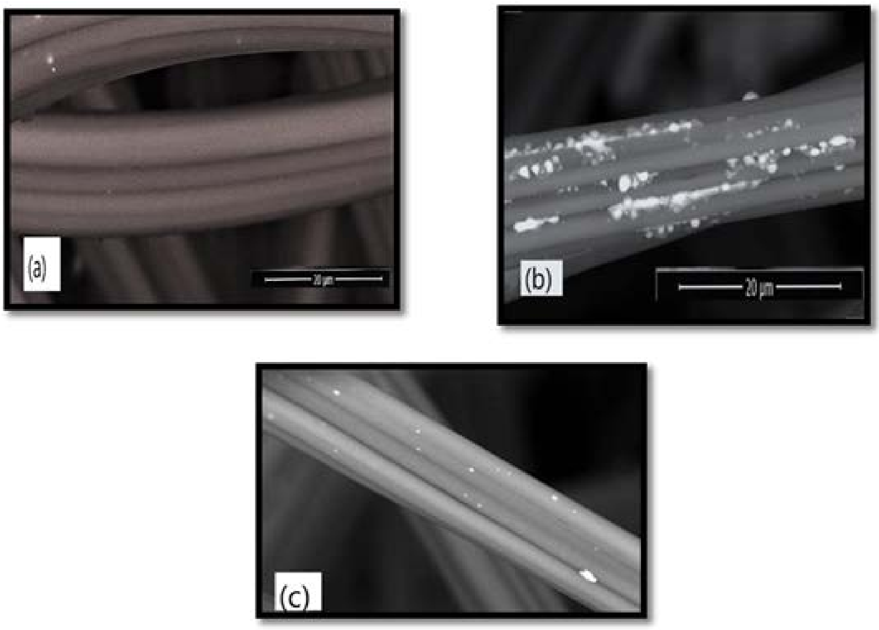


Fig. 13. SEM Micrographs of ACF. (a) Blank sample of ACF, (b) ACF after Zn adsorption, and (c) ACF after Cr adsorption.

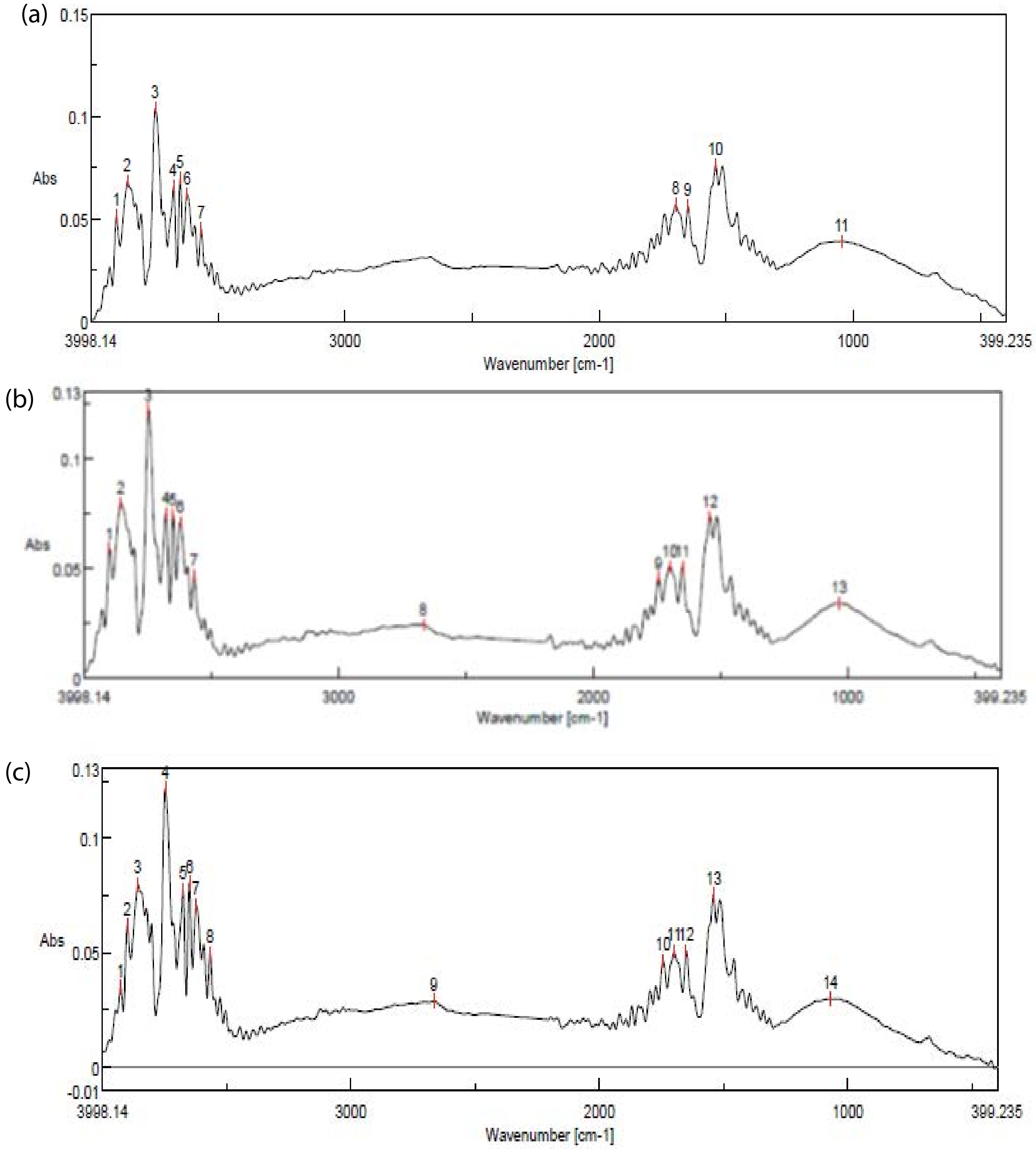


Fig. 14. FTIR spectrum of ACF before and after adsorption of Zn (II) and Cr (VI) on ACF (a) Blank sample of ACF, (b) ACF after Zn adsorption, and (c) ACF after Cr adsorption.

observed in case of Zn(II) which may be due to filling the pores on the surface of ACF. These observations agree with the result discussed previously and confirm the difference between the adsorption between Zn(II) and Cr(VI).

3.6. Structure characterizations by FTIR

The FTIR spectra of the ACF before and after adsorption process were investigated (Fig. 14). It was noticed that new absorption peaks appeared at 1,742 cm^{-1} corresponding to CO stretching band and new peak at 2,666 cm^{-1} could be assign to CH stretching of aldehyde. The peak appeared at 1,046 cm^{-1} in the ACF before adsorption and shifted to 1,033 cm^{-1} after Zn(II) adsorption (ACF surface was deprotonated). While in the Cr(VI) adsorption the peak shifted to 1,070 cm^{-1} (ACF surfaces was protonated) which may be assign to C–O stretching of ester group. These shifts in the peak could be attributed to change in the pH of the adsorption bath which affects the active groups on the surface of ACF [23].

4. Conclusion

The heavy metal adsorption using the ACF is considered the most effective and environmentally promising methods used for waste water treatments. The maximum removal efficiency of ACF for Zn(II) occurred at pH 10 and the minimum removal efficiency occurred at pH 2. This is due to the surface of ACF became deprotonated at pH = 10, so increased the adsorption of Zinc^{++} as a result of increasing of the electrostatic interaction between the Zn positive ions and the negative hydroxyl groups on the surface of ACF. The maximum removal efficiency of ACF for Cr(VI) occurred at pH 2 and the minimum removal efficiency occurred at pH 10. With decreasing the H^{+} concentration, the surface of ACF became deprotonated with increasing in the hydroxyl anion. Therefore, repulsion between the hydrogen chromate anions and hydroxyl groups decreases the adsorption of Cr(IV) on ACF surface.

Tailoring the adsorption properties of ACF can essentially contribute to increased specificity and selectivity of heavy metal adsorption, which will enhance performance required for the rapid reduction of pollutants. This can be accomplished by different treatments, which can be done by changing the pH of the solution as well as the treatment temperature.

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