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Removal of chromium(VI) from aqueous solution by carbon waste from thermal power plant

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ABSTRACT

Carbon waste was used as an adsorbent for the removal of Cr(VI) from aqueous solution of $K_2Cr_2O_7$. The experiments were carried out at different doses of adsorbent (0.1–0.8 g), pH (1–7), and initial metal ion concentration (1–30 mg/L). Different adsorption isotherms such as Langmuir, Freundlich, Temkin, and Dubinin–Radushkevich were tested. Langmuir adsorption isotherm was found to have highest regression value and hence the best-fit. Pseudo-second-order fitted best the kinetic data. Further, it was found that 92% chromium (VI) removal is possible at pH 2.0, adsorbent dose 0.6 g/50 ml, metal ion concentration 20 mg/L, and time 120 min. Thermodynamic parameters showed that the sorption process is endothermic and spontaneous in nature.

Keywords: Chromium(VI); Carbon waste; FTIR; SEM; Isotherms; Kinetics

1. Introduction

Rapid industrialization is mainly responsible for the inclusion of heavy metals in the environment. These metals are found well above the tolerance limit in aquatic environment [1]. Chromium compounds are widely used by different industries such as metal plating, paints and pigments, leather tanning, textile dyeing, printing inks, and wood preservation, so huge quantity of wastewater containing chromium is discharged into the environment [2]. Chromium exists in the aquatic environment mainly in two states: trivalent Cr(III) and hexavalent Cr(VI). Hexavalent chromium is primarily present in the form of chromate (CrO_4^{2-}) and dichromate (CrO_7^{2-}) ions. The latter form is the most toxic and is recognized as a carcinogenic and mutagenic agent [3]. Chromium(VI) can cause severe health problems from simple skin irritation to lung carcinoma. Therefore, removal of Cr(VI) from industrial wastewater is essential before disposal. Several technologies such as chemical precipitation, coagulation/flocculation processes, membrane filtration, oxidation processes, reverse osmosis, ion exchange, and solvent extraction have been developed to remove heavy metals, particularly chromium, from industrial wastewater [4]. However, these methods are costly and not feasible for small-scale industries. In contrast, an adsorption technique is economical and used widely. The most common adsorbent materials are: alumina silica, metal hydroxides, and activated carbon [5]. Removal of heavy metals by a large number of adsorbents has been reported [6–9], however, activated

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carbon is economically favorable and technically easier to operate [10–14]. Activated carbons with their large surface area, microporous character, and chemical nature of their surface have made them potential adsorbents for the removal of heavy metals from industrial wastewater. In this paper, a carbon waste (CW) generated from thermal power plant was obtained from a coal refining industry and was used as an adsorbent for the removal of Cr(VI) from aqueous solution under different conditions. This material has been used for the first time as an adsorbent. The use of CW as a water purifier may be economical and reduces the pollution generated by thermal power plant. Adsorption studies have been made and optimum conditions for Cr(VI) removal have been established.

2. Materials and methods

2.1. Materials

CW was taken from a coal refining industry, B.R. Private Limited, Ghaziabad, UP, India, which was obtained from thermal power plant. In the industry, this is commonly known as Khangar. This was washed twice with water to remove contaminations and impurities. It was dried at room temperature in shade and then in an oven for 2 h at 110˚C. The dried CW was crushed in a mechanical grinder and sieved through 350 μm mesh sieve to obtain fine powder. $K_2Cr_2O_7$ was taken for Cr(VI).

2.2. Methods

2.2.1. Study of adsorption of Cr(VI) on CW

A stock solution of potassium dichromate ($K_2Cr_2O_7$) of 100 ppm concentration was prepared from which solutions of different concentration (5–30 ppm) were obtained by dilution. The pH of $K_2Cr_2O_7$ solutions was adjusted by adding 1.0 M H_2SO_4. In order to determine the concentration of $K_2Cr_2O_7$, UV–visible spectra of $K_2Cr_2O_7$ solution were recorded and λ_{maxima} (540 nm) was found out. At λ_{maxima}, optical densities of $K_2Cr_2O_7$ solutions of different concentrations were recorded and a calibration curve was plotted. A known amount of CW in 50 ml of $K_2Cr_2O_7$ solution was added and stirred magnetically for different intervals of time and filtered. Optical densities of the filtered solutions were determined at 540 nm and the concentrations of $K_2Cr_2O_7$ adsorbed on the surface of CW were determined from the calibration curve. The experiments were performed at different pH (1–7), temperature (300, 310, and 320 K), time (30–240 min), and in the presence of different amounts of CW (0.1–0.8 g). The percentage of Cr(VI) removed from the solution was calculated by Eq. (1):

$$\text{Cr(VI) removal (\%)} = ((C_o - C_e)/C_o) \times 100 \qquad (1)$$

where C_o and C_e are initial and equilibrium concentrations (mg/L) of Cr(VI) in the solution.

The amount of Cr(VI) adsorbed (q) in (mg/g) was calculated using Eq. (2).

$$q = V(C_o - C_e)/M \qquad (2)$$

where M is the mass of CW used (g) and V is the volume of the solution (ml).

Langmuir, Freundlich, Temkin, and Dubinin–Radushkevich were tested.

2.2.2. SEM studies of CW

Scanning electron microscopic pictures of CW before and after Cr(VI) adsorption were recorded. Using sputtering technique, the surfaces were gold coated.

2.2.3. FTIR studies

The FTIR spectra of Khangar before and after adsorption of Cr(VI) were recorded in KBr phase in the wave length range 400–4,000 cm^{-1}.

3. Results and discussion

Scanning microscopic pictures of CW before and after adsorption of Cr(VI) are shown in Figs. 1 and 2, respectively. It appears that the surface morphology is slightly changed after Cr(VI) adsorption. The FTIR spectrum of CW (Fig. 3) shows a broad vibrational band at 3,420.18 cm^{-1} indicative of the existence of bound OH group due to adsorption of water molecules. This band is shifted to lower frequency and a new absorption band at 3,129.59 cm^{-1} is obtained after the adsorption of Cr(VI) on the surface of CW. Band at about 2,900 cm^{-1} is due to CH stretching modes. A strong band at 1,091.09 cm^{-1} may be due to =C=O stretching vibrations which is not affected after adsorption of Cr(VI) on the surface of CW. In the Cr(VI) adsorbed CW, two new bands (Fig. 4) appearing at around 1,600 and 1,400 cm^{-1} suggest that Cr is adsorbed on CW. Appearance of a new band at 3,129.59 cm^{-1} suggests association of oxygen with chromium. This oxygen may be of OH group.

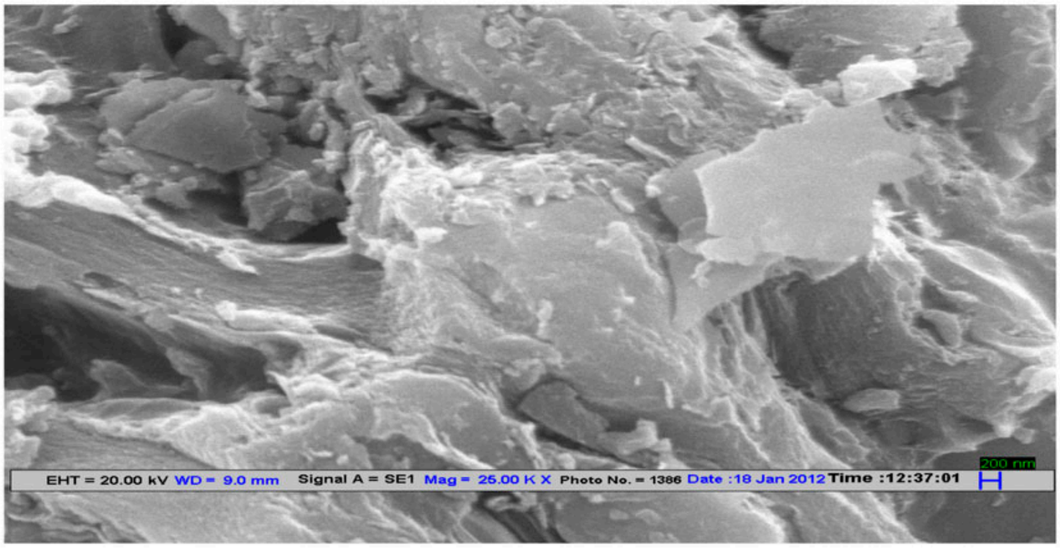

Fig. 1. SEM images of CW before adsorption.

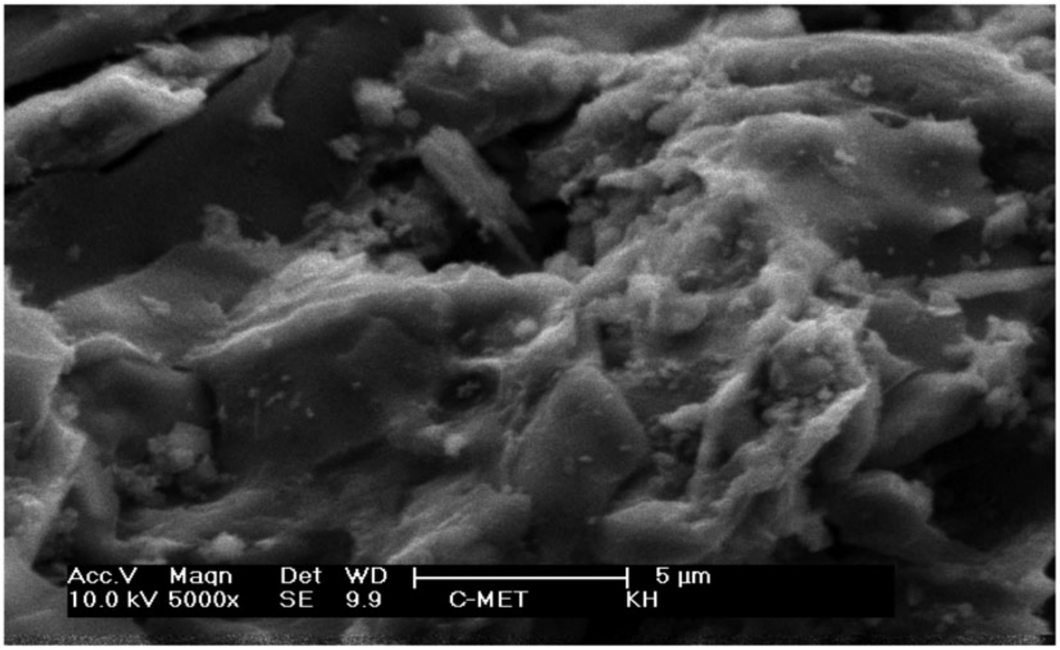

Fig. 2. SEM images of CW after Cr(VI) adsorption.

The effect of pH on the adsorption behavior of Cr(VI) on CW is shown in Fig. 5. Results show that Cr(VI) adsorption on CW is dependent upon the pH of the solution and decreased with increase in the pH of the solution. The maximum Cr(VI) adsorption by CW was observed at pH 2. The pH dependence of chromium adsorption can largely be related to the type and ionic state of the functional groups present on the adsorbent. In the acidic, chromium ions coexist in different forms such as $Cr_2O_7^{2-}$, $HCrO_4^-$, $Cr_3O_{10}^{2-}$ and $Cr_4O_{13}^{2-}$ of which $HCrO_4^-$ predominates. As the pH of the solution increases, the predominant species are then CrO_4^{2-} and $Cr_2O_7^{2-}$. More adsorption in acidic medium (low pH) may be due to increase in H^+ ions on the adsorbent surface that results in significantly strong electrostatic attraction between positively charged adsorbent surface and chromate ions. On the other hand, at high pH

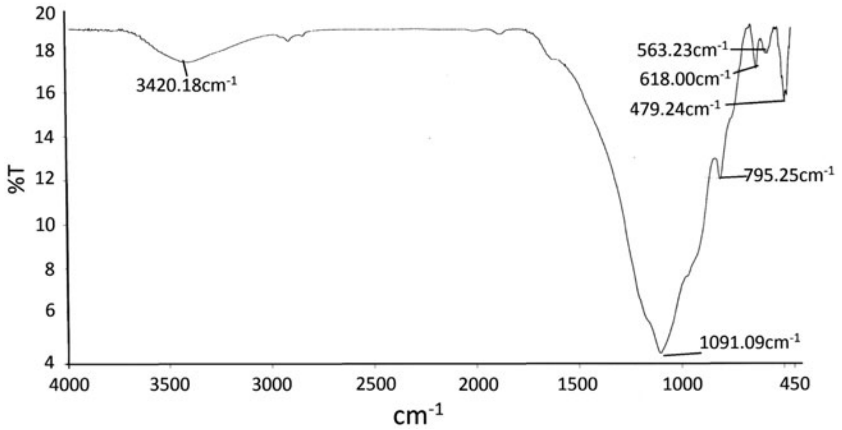

Fig. 3. FTIR spectra of CW before Cr(VI) adsorption.

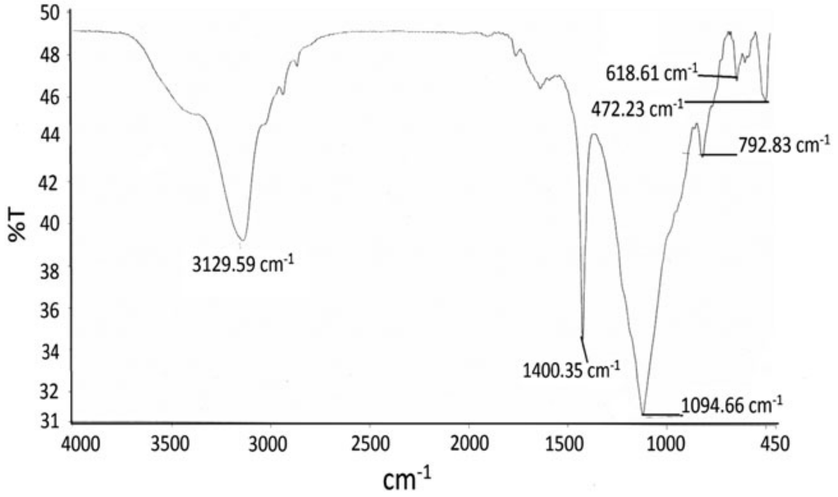

Fig. 4. FTIR spectra of CW after Cr(VI) adsorption.

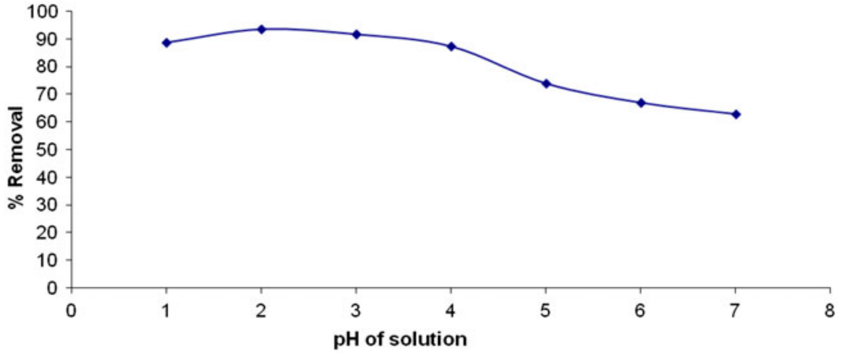

Fig. 5. Effect of pH on percentage Cr(VI) removal (adsorbent dose: 0.5 g/50 ml; metal ion concentration: 30 mg/L; time: 120 min).

there will be abundance of negatively charged hydroxyl ions in the aqueous solution, causing hindrance in adsorption [15].

In acidic solution, when there is an intimate contact between the adsorbent and adsorbate, Cr(VI) may have a high positive redox potential lying between 1.33–1.38 V and may be unstable. The surface of adsorbent contains –COOH, –OH, –CHO, –C–O–C– groups, and acts as electron donor in solution so Cr (VI) is easily reduced to Cr(III) ions via the following equation.

$$3HCrO_4^- + 21H^+ + 17e^- \rightarrow Cr^{3+} + 12H_2O \tag{3}$$

The above equation indicates that the reduction of the Cr(VI) oxyanion is accompanied by a considerable consumption of protons from the acidic medium. Thus, pH plays the decisive role in the removal of Cr (VI) ions [16,17].

Percent removal of Cr(VI) from aqueous solution in the presence of fixed amount of CW (0.5 mg/50 ml) at pH 2 was studied. Fig. 6 shows percent removal of Cr(VI) from aqueous solution at different time intervals and at different Cr(VI) concentrations. It is found that the percent removal increased with time and concentration. However, the removal was found to be the maximum at 120 min. The time required to attain this value is termed as the equilibrium time. The variation of Cr(VI) removal from aqueous solution as a function of chromium concentration at pH 2 and adsorbent 0.5 g/50 ml solution is shown in Fig. 7. From the figure, it is evident that the percentage removal shows an increasing trend up to 20 ppm and then decreases continuously. It appears that at lower concentrations, all Cr(VI) ions present in the sorption could interact with the binding sites of CW, hence higher removal. However, at higher concentrations, because of the saturation of the sorption sites, the percentage uptake of the chromium by CW shows a decreasing trend. A similar trend was reported by Gupta and Rastogi [18].

The effect of adsorbent dose on the percent removal of Cr(VI) is shown in Fig. 8. A maximum removal of Cr(VI) (92.93%) is achieved by CW at an optimum adsorbent dose of 0.6 g/50 ml for Cr(VI) concentration of 20 mg/L at pH 2. It could be seen from the figure that initially the percentage removal increased very sharply with the increase in adsorbent dose but after a certain value (0.6 g/50 ml) the percentage removal was almost constant. Increase in adsorption with adsorbent dosage can be attributed to the availability of more adsorption sites [19,20].

Adsorption studies were made at different temperatures and Langmuir, Freundlich, Temkin, and Dubinin–Radushkevich isotherms were tested.

The Langmuir isotherm assumes monolayer coverage of adsorbate over a homogeneous adsorbent surface and is dependent on the assumption that the adsorbent surface consists of active sites having a uniform energy and, therefore, the adsorption energy is constant [21]. The Langmuir adsorption isotherm is expressed as:

$$Q_e = Q_0 b C_e / 1 + b C_e \tag{4}$$

Eq. (4) can be rearranged to the following form:

$$1/Q_e = 1/Q_0 + 1/Q_0 \cdot b \cdot C_e \tag{5}$$

where C_e is the equilibrium concentration (mg/L), Q_e is the amount of Cr(VI) ions adsorbed at equilibrium (mg/g), Q_0 is the maximum adsorption capacity (mg/g), b is the adsorption equilibrium constant (L mg^{-1}). A plot of $1/Q_e$ vs. $1/C_e$ gave straight lines (Fig. 9). The values of different quantities of Eq. (5)

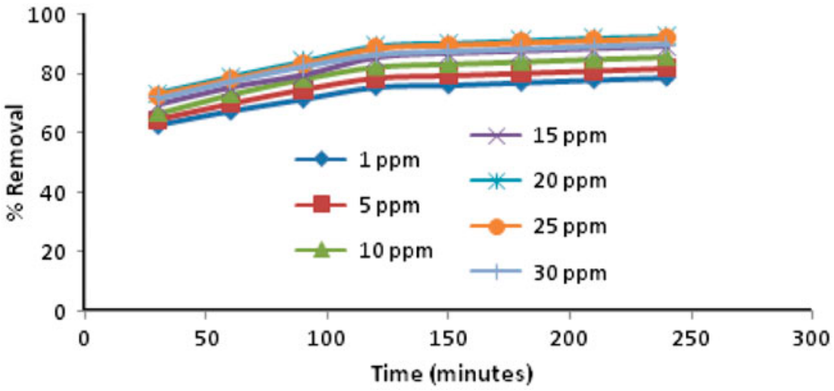

Fig. 6. Effect of contact time on percentage Cr(VI) removal (Temp. = 320 K; adsorbent dose: 0.5 g/50 ml; pH: 2).

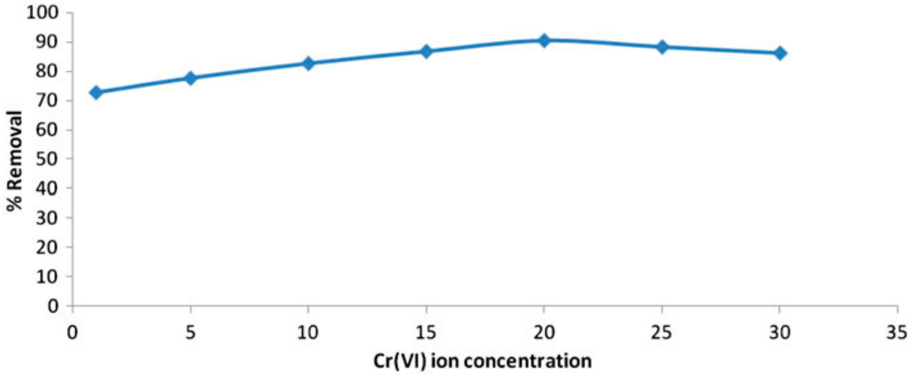

Fig. 7. Effect of initial metal ion concentration on percentage Cr(VI) removal (adsorbent dose: 0.5 g/50 ml; pH: 2; time: 120 min).

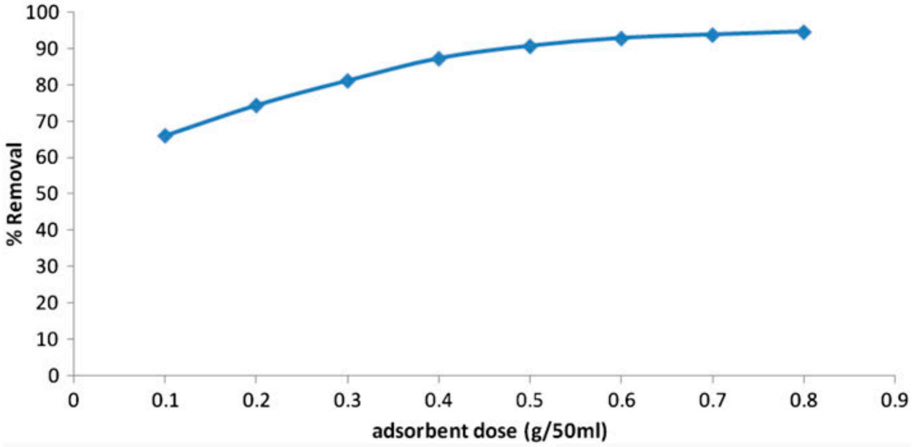

Fig. 8. Effect of adsorbent dosage on percentage Cr(VI) removal (pH: 2; metal ion concentration: 20 mg/L; time: 120 min).

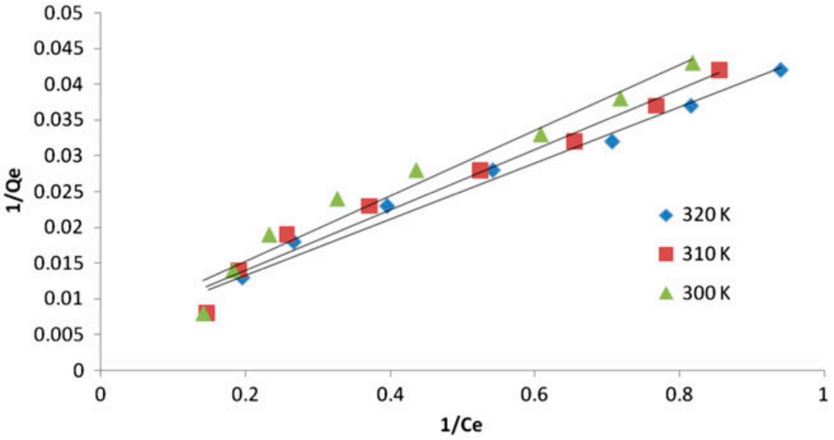

Fig. 9. Langmuir isotherm for Cr(VI) removal at various solution temperatures using CW.

Table 1
Isotherm model coefficient for the adsorption of Cr(VI) on CW at various temperatures

	Freundlich isotherm			Langmuir isotherm			D–R isotherm			Temkin isotherm		
Temp. (K)	$1/n$	K	R^2	Q_0	b	R^2	X_m	β	R^2	A	B	R^2
300	0.84	1.27	0.93	0.006	0.046	0.96	4.34	0.0062	0.738	3.988	52.94	0.757
310	0.844	1.31	0.95	0.006	0.042	0.972	4.41	0.0059	0.791	7.438	53.2	0.798
320	0.819	1.33	0.95	0.005	0.039	0.977	4.4	0.0052	0.785	11.516	56.46	0.803

were calculated and are given in Table 1. To investigate the favorability of a process, the dimensionless separation factor R_L was also calculated from Eq. (6) and found to be less than 1.

$$R_L = 1/(1 + bC_0) \tag{6}$$

In Eq. (6) b is Langmuir constant and C_0 is optimum initial concentration of Cr(VI). The values of R_L were 0.5213, 0.5417, and 0.5599, respectively, at 300, 310, and 320 K. If the value of R_L is less than 1.0, the adsorption is favorable. Thus, Cr(VI) adsorption on CW is favorable [22].

The Freundlich adsorption isotherm model assumes that the adsorbent surface energy is heterogeneous and the stronger binding sites are occupied first. Also the binding strength decreases with the increasing degree of site occupation. The logarithmic linear form of Freundlich isotherm is given by Eq. (7).

$$\log Q_e = \log k + 1/n \log C_e \tag{7}$$

where k (L/mg) and n are the Freundlich constants [23]. A plot of log Q_e versus log C_e gave straight lines (Fig. 10). Isotherm constants and the regression coefficients (R^2) are given in Table 1.

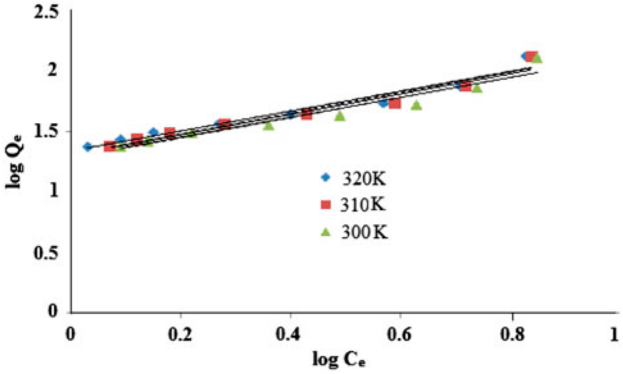

Fig. 10. Freundlich isotherm for Cr(VI) removal at various solution temperatures using CW.

The Temkin isotherm equation assumes that the heat of adsorption of all the molecules in the layer decreases linearly with coverage due to adsorbent–adsorbate interactions. Tempkin isotherm is expressed by Eq. (8):

$$Q_e = B \ \ln A + B \ln C_e \tag{8}$$

where Q_e is the adsorbed amount (mg/g), A (L/g) is Temkin isotherm equilibrium binding constant, B (J/mol) is a constant related to heat of sorption which is given by Eq. (9).

$$B = RT/b \tag{9}$$

where R is the gas constant, T is the absolute temperature (K), and b is Tempkin isotherm constant [24]. A plot of Q_e versus ln C_e enables the determination of the isotherm constants A and B from slope and intercepts (Fig. 11). Values of constants are shown in Table 1 along with the value of correlation coefficient.

The Dubinin–Radushkevich isotherm is generally used to describe the adsorption mechanism with a Gaussian energy distribution onto a heterogeneous surface [25] and expressed as:

$$\ln Q_e = \ln X_m - \beta\varepsilon^2 \tag{10}$$

where Q_e is the adsorbed amount (mg/g), X_m (mol/g) is Dubinin–Radushkevich constant representing saturation capacity, β (mol^2/J^2) is the activity coefficient related to mean free energy of adsorption per mol of the adsorbate, and ε is Polanyi potential which is equal to:

$$\varepsilon = RT \ \ln\,(1 + 1/C_e) \tag{11}$$

where R is the ideal gas constant and T (K) is the temperature of adsorption. The mean energy of sorption, E (kJ/mol), is calculated by the following equation:

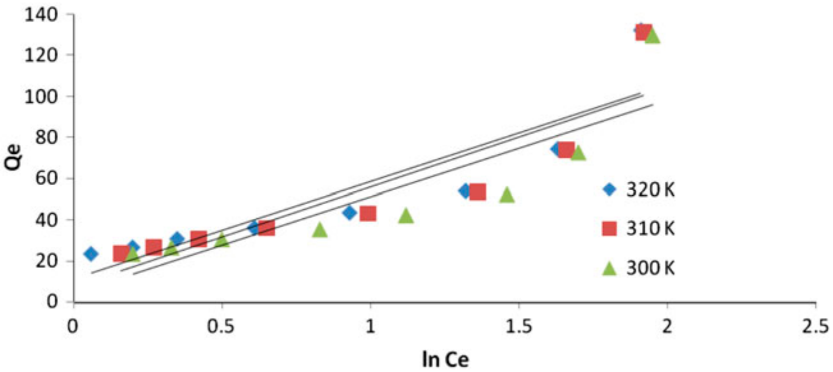

Fig. 11. Temkin isotherm for Cr(VI) removal at various solution temperatures using CW.

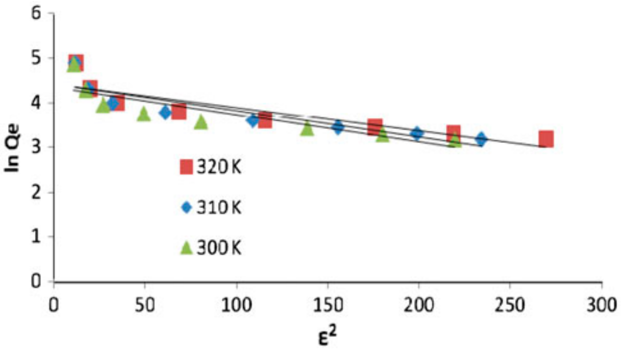

Fig. 12. Dubinin–Radushkevich isotherm for Cr(VI) removal at various solution temperatures using CW.

$$E = 1/\sqrt{2}\beta \tag{12}$$

A plot of ln Q_e versus ε^2 enables the determination of the isotherm constants X_m and β from intercepts and slope (Fig. 12). Values of constants are given in Table 1 along with the value of correlation coefficients.

Based on the average values of the statistical parameters for each isotherm, it can be concluded that Langmuir isotherm provides the best representation of the experimental equilibrium data and the next is Freundlich model.

In order to investigate the sorption process of Cr (VI) on CW, the data obtained from adsorption kinetic experiment were simulated using pseudo-first-order and pseudo-second-order reaction rate equation.

The pseudo-first-order model: the Lagergren rate constant values at different temperatures are determined from the first-order rate expression given by [26].

$$\ln\,(Q_{e(exp)} - Q_{t(exp)}) = \ln\ Q_{e(theo)} - k_1 t \tag{13}$$

where q_e and q are the amounts of Cr(VI) adsorbed (mg/g) at equilibrium and at time t (min), respectively, and k_1 is the rate constant of adsorption (L/min). The values of k_1, q_e calculated from the equation, and the correlation coefficient (R^2) values are presented in Table 2. A plot of ln ($Q_e - Q_t$) versus t for pseudo-first-order rate equation is shown in Fig. 13.

The pseudo-second-order rate expression is expressed by Eq. (14) [27,28]:

$$t/Q_{t(exp)} = 1/k_2Q^2_{e(teho)} + t/Q_{e(thoe)} \tag{14}$$

where k_2 is the equilibrium rate constant of pseudo-second-order (g/mg min). A plot t/Q_t versus t is shown in Fig. 14. The values of k_2, q_e calculated from the equation, and the correlation coefficient (R^2) values are given in Table 2.

From the graphs and Table 2, it can be seen that the first-order model did not fit well ($R^2 < 0.84$). This suggests that the adsorption of Cr(VI) does not follow first-order kinetics. However, the experimental equilibrium data indicate a good compliance with the second-order kinetic model with a higher correlation coefficient ($R^2 > 0.99$). Thus, the adsorption process is pseudo-second-order. This suggests that the overall rate of the adsorption process was controlled by chemisorptions which involved valency forces through the sharing or exchange of electrons between the sorbent and sorbate [29,30].

Thermodynamic parameters such as Gibbs free energy change (ΔG°), enthalpy change (ΔH°), and entropy change (ΔS°), for adsorption can be expressed by Eqs. (15–17) [31,32]:

$$\Delta G^\circ = -RT\ \ln\ b \tag{15}$$

Table 2
Pseudo-first-order and pseudo-second-order model parameters and correlation coefficients for the adsorption of Cr(VI) on CW at various temperatures

	Pseudo-first-order kinetic model			Pseudo-second-order kinetic model		
Temp. (K)	q_e (mg/g)	K_1 (1/h)	R^2	q_e (mg/g)	K_2 (g/mg h)	R^2
300	34.0136	4.91	0.8485	32.4675	0.85	0.9996
310	33.6700	4.4	0.8173	33.0033	0.82	0.9996
320	34.0136	3.67	0.8239	33.6700	0.82	0.9997

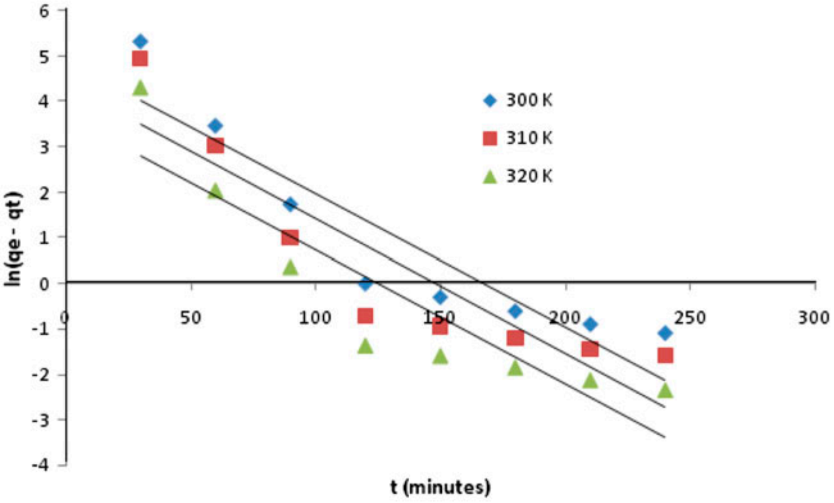

Fig. 13. Pseudo-first-order kinetics of Cr(VI) adsorption on CW at various temperature.

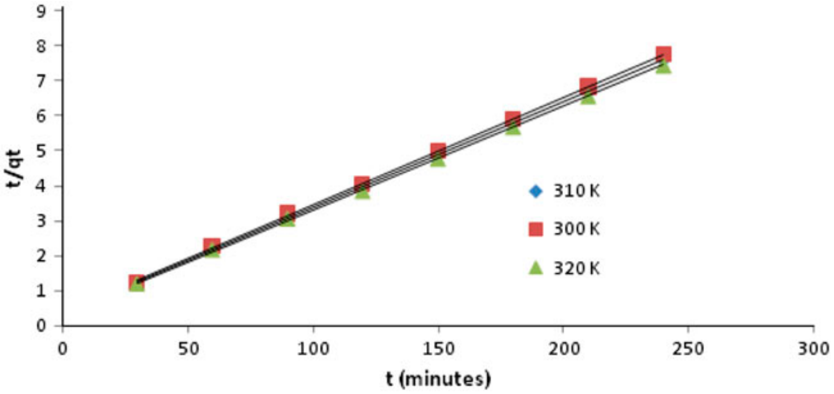

Fig. 14. Pseudo-second-order kinetics of Cr(VI) adsorption on CW at various temperature.

$$\Delta H^\circ = -R(T_2T_1/T_2 - T_1)\ \ln\,(b_2/b_1) \qquad (16)$$

$$\Delta S^\circ = \Delta H^\circ - \Delta G^\circ/T \qquad (17)$$

where R is the universal gas constant, T, T_1, T_2 are temperatures, and b, b_1, b_2 are Langmuir constants at respective temperatures. The Gibbs free energy was found to be negative at all temperatures, indicating spontaneous process of adsorption with a high preference of Cr(VI) by CW. The decrease in the negative value of ΔG° with an increase in temperature indicates that the adsorption process of Cr(VI) on CW becomes

Table 3
Thermodynamic parameters for the adsorption of Cr(VI) on CW

		ΔG° (kJ/mol)		
ΔH° (kJ/mol)	ΔS° (kJ/mol K)	300 K	310 K	320 K
53.68	53.93	−79.79	−82.56	−84.19

more favorable at higher temperatures. The positive value of ΔH° indicates that the adsorption reaction is endothermic (Table 3). The positive value of entropy suggests that some structural changes occur on the adsorbent and the randomness at the solid/liquid interface in the adsorption system increases during adsorption process [33].

4. Conclusions

From the results it is concluded that

(1) Khangar (CW), a waste material of Thermal power plant from coal refining industry, can be used as a potential and effective adsorbent for the removal of Cr(VI) from aqueous solution over a wide range of concentrations.
(2) Adsorption of Cr(VI) was found to increase with the increase in time and adsorbent dose.
(3) Acidic solution (low pH) proved to be more favorable for the adsorption of Cr(VI) on the CW.
(4) The adsorption of Cr(VI) using CW followed Langmuir adsorption isotherm.
(5) The kinetics of the adsorption process was found to follow the pseudo-second-order kinetic model. The evaluated changes in free energy (ΔG°) and enthalpy (ΔH°) indicated that the adsorption process was spontaneous and endothermic in nature.

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