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Removal of Cr(VI) from aqueous solution using brick kiln chimney waste as adsorbent

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ABSTRACT

Heavy metals are toxic to aquatic flora and fauna even in relatively low concentrations. Chromium is a commonly occurring toxic metal in ecosystems. This study aims the adsorption of Cr(VI) on a novel low-cost carbonaceous material under different experimental conditions such as contact time, initial concentration of metal ions, pH, and temperature. The adsorbent was characterized by using scanning electron microscopy and energy-dispersive X-ray spectroscopy. The equilibrium data were fitted well with Langmuir and Freundlich isotherms. Adsorption kinetics of Cr(VI) ions onto chimney waste adsorbent were analyzed by pseudo-first-order and pseudo-second-order models. The adsorption process is favored by acidic pH and followed the second-order kinetics. Various thermodynamic parameters like activation energy (E_a), Gibbs free energy change (ΔG^o), enthalpy change (ΔH^o), and entropy change (ΔS^o) were calculated. The results showed that the carbonaceous material obtains from bricks kiln chimneys can be efficiently used for Cr(VI) removal from wastewater.

Keywords: Adsorption; Chromium; Heavy metals; Langmuir; Kinetics

1. Introduction

The discharge of industrial effluents containing heavy metals creates a major problem for the aquatic environment. Mostly, heavy metals are present in industrial effluents, and these metal ions do not degrade into harmless end product. Most of the heavy metals are toxic even in minute quantities [1]. Chromium is among the toxic metals, found in the aquatic system in two oxidation state Cr(III) and Cr(VI). The Cr(VI) is extremely toxic and shows carcinogenic effect [2]. Major sources of hexavalent chromium are effluents released from electroplating [3], metal finishing, aluminum [4], dyes, chrome tanning [5], textile, and pigment industries [6].

The United States Environmental Protection Agency has regulated the permissible limit of $0.1\,\mathrm{mg\,L^{-1}}$ of total chromium in drinking water [7]. The chromium content of the effluents should be

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reduced to permissible limits before their discharge into surface water. Different methods are available for chromium removal from wastewaters. Some of the well-established methods are chemical precipitation, electrodialysis [8], membrane process [9], ion exchange [10], reverse osmosis, nanofiltration, ultrafiltration [11], and emulsion pertraction technology [12]. Adsorption process using activated carbon proved to be an effective technique for the removal of Cr(VI) from wastewaters. However, due to the high cost of the good-quality granular carbon and availabilities makes the process less attractive for many industries. A number of low-cost and abundantly available adsorbent have been tried for the removal of chromium Cr(VI) from wastewater [13]. Some of them are rice husk [14], bagasse [15], wheat straw [16], sawdust [17] and used tyre carbon [18], surface-modified coconut shell charcoal and commercially activated carbon [19]. This study introduces a novel carbonaceous material, which is obtained from chimneys of brick kilns as an adsorbent for Cr(VI) removal. All brick kilns uses coal and wood as fuel for firing the bricks. A huge chimney makes the emission of smoke from the brick kiln. The massive ashes are accumulated in the chimneys, which are removed after some time and discarded. This carbonaceous material mainly consists of amorphous carbon and has large surface area, pore volume, and good adsorptive properties. and thus predominantly used as adsorbent for the removal of Cr(VI) from wastewater. Hence, an attempt has been made to use this material as adsorbent for Cr(VI) removal from waste water. This adsorbent designated as a chimneys carbonaceous material (CCM). This adsorbent is cost-effective, require less reagent consumption, simple practical steps and easy availability; moreover, it may reduce the economy burden and will be useful in the wastewater treatment before discharge. The study includes an observation of various influential parameters such as the initial concentration of hexavalent chromium, agitation time, temperature, and pH of the solution on the adsorption kinetics. Equilibrium data are fitted with various adsorption isotherms. A simple pseudo-first- and second-order model is used to fit the kinetic data. The rate constants from these models are evaluated at four different temperatures.

2. Materials and methods

2.1. Material development and solutions

All the reagents and solvents used were of analytical grade. Potassium chromate, hydrochloric acid, sodium hydroxide, 1, 5-diphenylcarbazide, etc. were purchased from Merck (Pvt.) Ltd., Pakistan.

2.2. Adsorbate solution

A stock solution of Cr(VI) ($1\,\mathrm{g\,L^{-1}}$) was prepared from potassium chromate in double-distilled water. Working solutions were prepared by successive dilution of stock solution.

2.3. Equipments

The pH was measured using pH Meter model Hanna Instruments® HAN-HI8314. The pH meter was calibrated using standard buffer solutions. A spectrophotometer model SP-300 Microprocessor-Controlled Spectrophotometer OPTIMA INC, Japan was used for the absorbance measurements. Scanning electron microscope (SEM) model JSM 5910, JEOL-JAPAN was used for the analysis of surface micrograph. EDS was done using Energy Dispersive X-ray Analyzer INCA-200, Oxford instruments, U.K. The surface area and pore size of the adsorbent were determined by BET-N_2 adsorption method using Surface Area Analyzer NOVA 2200e Quanta Chrome, USA. The samples were degassed at 100°C for one hour for the surface area analysis.

The adsorbent used in this study was obtained from chimneys of brick kilns located in Peshawar region (Pakistan). The adsorbent material was washed thoroughly with deionized water to remove dust and other foreign matter and then dried at 60°C for "5–6 h". Dried adsorbent material was then soaked in 0.1 M H_2SO_4 over night and after that it was washed again with doubly distilled water. The adsorbent was soaked in 0.1 M NaOH for a period of 4–5 h to remove traces of acid and then dried in the oven at 80°C for one hour. The sample was milled into powder and passed through 200 and 150-μm sieves. The sample was transferred into a large size refractory container and was activated by placing in a vacuum oven at 200°C for 2 h. After activation, the sample was stored in a vacuum desiccator for further study.

3. Batch adsorption experiments

Batch mode adsorption studies were carried out by taking 30 mL of the Cr(VI) solution of the desired concentration ($25–400\,\mathrm{mg\,L^{-1}}$) and desired weight of CCM in 100-mL conical flasks. Then, the conical flasks were agitated at 120 rpm using a mechanical shaker. pH was adjusted using 0.1 N hydrochloric acid or 0.1 N sodium hydroxide. After different interval of time, the adsorbent and adsorbate were separated by filtration using Whatman No 41 filter paper and solution was analyzed spectrophotometrically with 1,5 diphenylcarbazide by SP-300 Microprocessor-Controlled spectrophotometer at maximum peak is at about 540 nm to determine the quantity.

The amount adsorbed "q" (mg g^{-1}) was calculated using the Eq. (1).

$$q = \frac{(C_i - C_e) \times V}{W} \tag{1}$$

where C_i is the initial concentration (mg L^{-1}), C_e is the equilibrium concentration (mg L^{-1}), V is the volume of solution in mL^{-1} and W is the amount of adsorbent in g L^{-1}. The experimental parameters varied were the Cr(VI) initial concentration (5–400 mg L^{-1}) and the temperatures (10, 20, 30, and 40°C). All the experiments were performed in duplicate, and the mean values are reported in this paper.

4. Results and discussion

4.1. Physicochemical characteristics of the CCM adsorbent

Physicochemical characteristics and proximate analysis, such as ash content, moisture content, volatile matter, were determined by ASTM D3173 method [20], and results are presented in Table 1. It is noted that the surface area of the adsorbent is quite greater than some reported adsorbents. The low moisture content, ash content, volatile matter contents, and high percentage of carbon are the characteristics of an effective adsorbent. The EDS and scanning electron microscopy studies were carried out for the adsorbent material and the spectra are given in Figs. 1–5. Fig. 1 indicates that the carbonaceous material composed of carbon, oxygen, sulfur and magnesium with percentage of 87.55, 11.57, 0.41, and 0.47, respectively. It is obvious that adsorbent contain large amount of fixed carbon, which is mainly responsible for adsorption. SEM images at 2,000× and 4,000× magnification obtained before and after adsorption to determine the surface morphology, as shown in Figs. 2–5. Figs. 2 and 3 show irregular structure having small pores, which facilitate the process of adsorption. But the SEM micrograph in Figs. 3 and 4 are indicating that these pores become filled with the adsorbate after adsorption.

Table 1
Physicochemical parameters of adsorbent material

S.no.	Parameters	Values
1	pH (slurry)	3.45
2	Bulk density (g cm^{-3})	1.06
3	Moisture (%)	2.95
4	Ash content (%)	4.02
5	Volatile matter (%)	4.79
6	Surface area (BET), m^2 g^{-1}	253.56
7	Surface area (DR), m^2	1,144.30
8	Surface area (BJH), m^2 g^{-1}	77.68
9	Surface area (Langmuir), m^2 g^{-1}	1,287.88

4.2. Effect of pH on Cr(VI) removal

The effect of pH on the process is presented in Fig. 6. The percentage adsorption of Cr(VI) is decreasing with increasing pH. The maximum adsorption took place in the pH range 2–4.

4.3. Effect of agitation time

The effect of contact time on adsorption of Cr(VI) is presented in Fig. 7. It is apparent from Fig. 7 that the percent removal increases with increasing contact time, and the equilibrium was established after 90 min and that time was considered for whole batch experiments.

4.4. Adsorption study of Cr(VI) on CCM

The effect of shaking time and temperature on the adsorption of Cr(VI) on CCM from aqueous solution was also studied.

4.5. Kinetics study

The adsorption of Cr(VI) on CCM from aqueous solution at different temperatures and shaking time was studied. pH, adsorbent dosage, initial Cr(VI) concentration, and agitation time were kept constant. The results are given in Table 1 and shown in Fig. 7. It is evident from the figure that the adsorption of Cr(VI) on CCM increases with passage of time. It may be observed from the Figure that Cr(VI) up take by CCM was rapid in the initial 60 min and then slowed down up to 100 min. The equilibrium reached after 90 min. The initial rapid uptake of Cr(VI) ions from solution was due to large number of active sites are available on the surface of material. As time passes the sited become saturated and adsorption rate decreases and become constant because the free space was not available to the adsorbate molecule.

In order to investigate the kinetics of adsorption of Cr(VI) on CCM, the following Lagergren Eq. (2) was applied to the adsorption data.

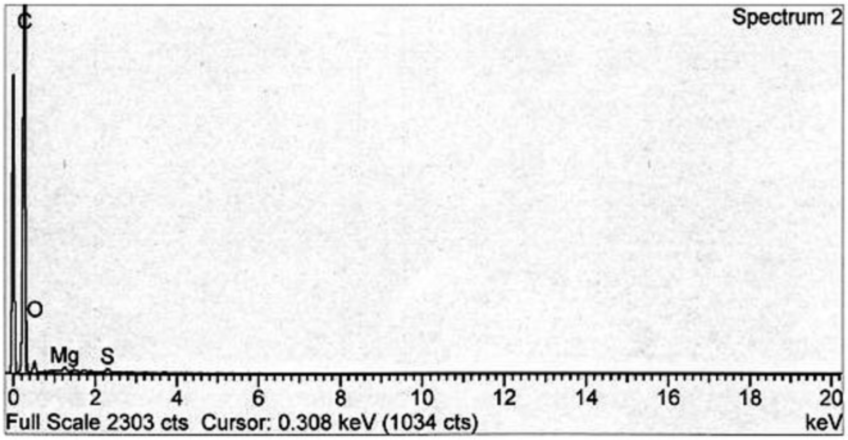

Fig. 1. EDS spectra of CCM.

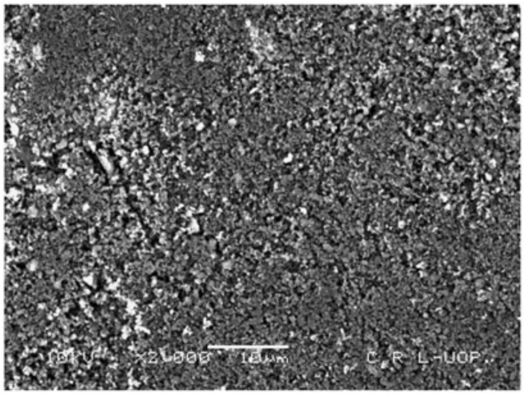

Fig. 2. SEM spectra of the adsorbent before adsorption of Cr(VI) at 2,000× magnification.

$$\log(q_e - q_t) = \log q_e - \left(\frac{k1}{2.303}\right)t \tag{2}$$

where q_t is the amount adsorbed at time t (mg g^{-1}), k_1 is the rate constant of the equation (min^{-1}); q_e is the amount adsorbed at equilibrium (mg g^{-1}).

Straight lines were obtained by plotting log ($q_e - q_t$) against t, as shown in Fig. 8, which indicates that the adsorption process follows first-order kinetics. The values of rate constant k were calculated from the slopes of straight lines of Fig. 8 and are given in Table 2.

4.6. Pseude-second-order model

A pseudo-second-order model also describes the kinetics of adsorption the linear form of the model shown by Eq. (3).

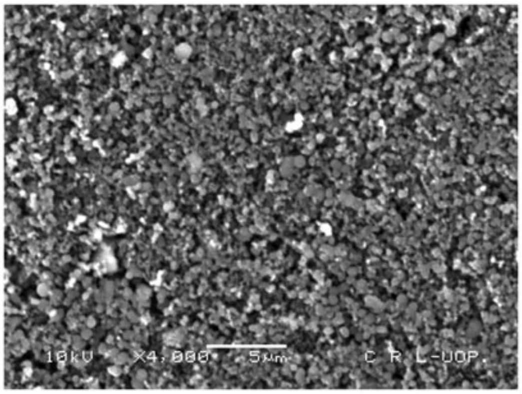

Fig. 3. SEM spectra of the adsorbent before adsorption of Cr(VI) at 4,000× magnification.

$$\frac{t}{q_1} = \frac{1}{K_2 q_e^2} + \left(\frac{1}{q_e}\right)t \tag{3}$$

where k_2 is the rate constant of the second-order equation (g mg^{-1} min^{-1}); q_t the adsorption time t (min) (mg g^{-1}); q_e is the amount of adsorption equilibrium (mg g^{-1}).

Fig. 9 shows the applicability of second order for the adsorption process. The amount of adsorption at equilibrium "q_e" rate constant "k" and correlation coefficient "R^2" for second order are given in Table 2. The validity of the kinetic models is tested by the magnitude of the correlation coefficient R^2 that is given in Table 2. It is important to note that the correlation coefficient for first order is less than second-order correlation coefficient at every experimental temperature. Therefore, first-order model is not suit-

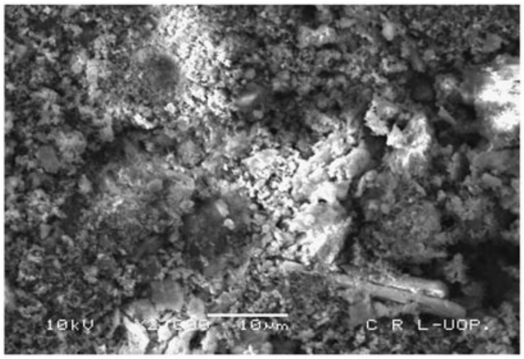

Fig. 4. SEM spectra of the adsorbent after adsorption of Cr(VI) at 2,000× magnification.

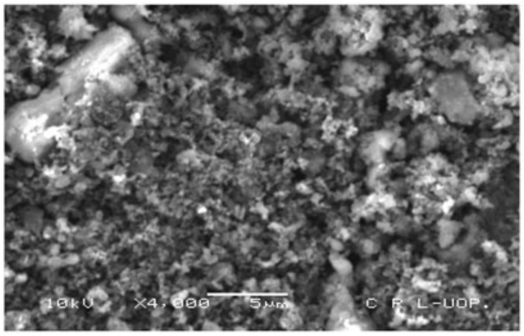

Fig. 5. SEM spectra of the adsorbent after adsorption of Cr(VI) at 4,000× magnification.

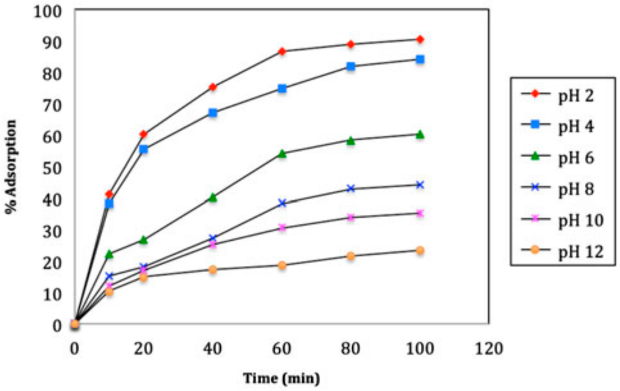

Fig. 6. Effect of initial pH on adsorption of Cr(VI) on CCM at constant temperature, 150 mg L^{-1} initial Cr(VI) concentration, 120 rpm agitation speed, 10 g L^{-1} adsorbent dosage.

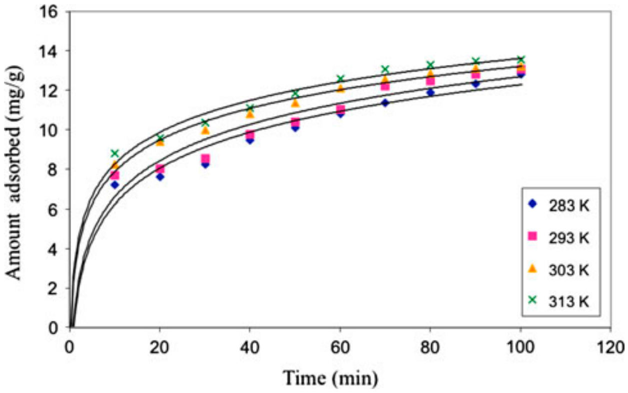

Fig. 7. Effect of contact time on adsorption of Cr(VI) on CCM at different temperatures, 150 mg L^{-1} initial Cr(VI) concentration, 120 rpm agitation speed, 10 g L^{-1} adsorbent dosage.

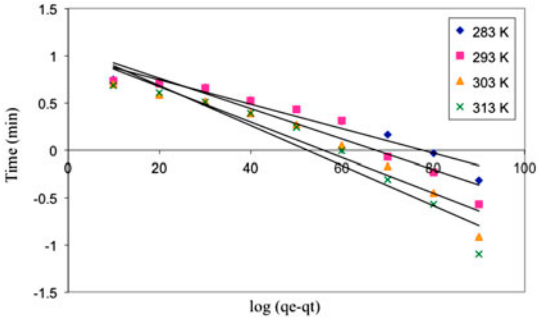

Fig. 8. Lagergren plot for the adsorption Cr(VI) by CCM at different temperatures.

able for modeling the adsorption of Cr(VI) on this carbonaceous material.

The activation energy E_a, for adsorption was found by using Arrhenius Eq. (4).

$$\ln k = \frac{E_a}{RT} + \text{constant} \tag{4}$$

where E_a is the activation energy, R is gas constant, and T is the absolute temperature. The value of activation energy was determined from the slope of the plot of $\ln k$ vs. $1/T$ and shown in Fig. 10. It was found to be 7.291 kj mol^{-1}. The activation energy gives us idea about the adsorption process that weather it is physical or chemical the results obtained suggest that it was physical adsorption.

Table 2
Rate adsorption constants for two kinetic models at different temperatures

T (K)	First-order (Lagergren equation)				Second-order		
	q_e (experimental) (mg g^{-1})	q_e (calculated) (mg g^{-1})	k_1 (10^{-2} min^{-1})	R^2	q_e (calculated)	k_1(10^{-3} g mg^{-1}min^{-1})	R^2
283	12.800	9.999	2.97	0.9429	14.864	3.476	0.9821
293	13.056	11.989	3.70	0.9114	15.128	3.587	0.9806
303	13.200	11.043	4.30	0.9267	14.641	5.724	0.9954
313	13.551	12.656	4.85	0.9221	15.037	5.783	0.9953

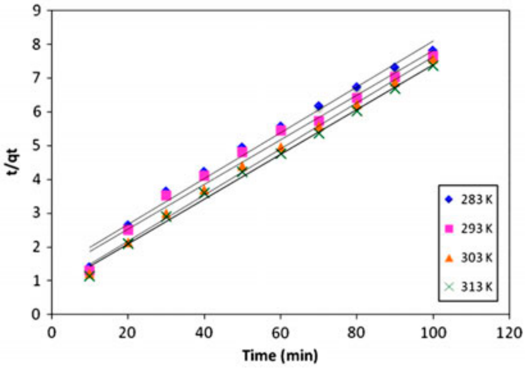

Fig. 9. Second-order plot for the adsorption of Cr(VI) at different temperatures, 150 mg L^{-1} initial Cr(VI) concentration, 120 rpm agitation speed, 10 g L^{-1} adsorbent dosage.

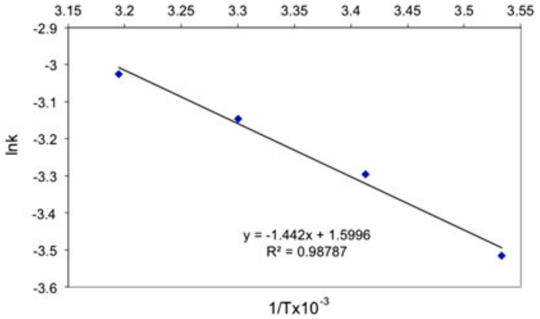

Fig. 10. Arrhenius plot for the adsorption of Cr(VI) on CCM.

4.7. Adsorption isotherms

Adsorption isotherm is important in designing the nature of adsorption system. The data of this study are evaluated by applying the Langmuir and Freundlich isotherms at various temperatures. The linear form of Langmuir adsorption isotherm:

$$\frac{C_e}{q_t} = \frac{1}{K_1 X_m} + \frac{C_e}{X_m} \tag{5}$$

where C_e is the equilibrium concentration (mg L^{-1}), q_t is the amount (mg g^{-1}) of Cr(VI) adsorbed, X_m (mg g^{-1}) and K_1 (L mg^{-1}) are Langmuir constants, representing the adsorption capacity (mg g^{-1}) and energy of adsorption (mg g^{-1}), respectively.

Fig. 11 shows plot of C_e/q_t against C_e indicate the applicability of Langmuir adsorption isotherm, consequently the formation of monolayer surface of the adsorbate on the surface of the adsorbent. Langmuir constant X_m (adsorption capacity) and K_1 (binding energy constant of adsorption or energy of adsorption) were calculated from the slopes and intercepts of plots of C_e/q_t against C_e, respectively, and are given in Table 3.

RL is one of the essential characteristics of Langmuir isotherm, which is a dimensionless separation factor as described [21].

$$RL = \frac{1}{1 + K C_o} \tag{6}$$

The magnitude of RL shows the feasibility of adsorption process, K is Langmuir constant, and C_o is the initial concentration of Cr(VI). The value lies

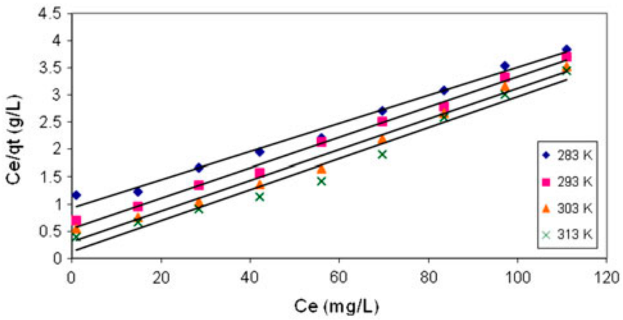

Fig. 11. Langmuir isotherm for the adsorption of Cr(VI) on CCM at different temperatures.

Table 3
Langmuir and Freundlich constants for the adsorption of Cr(VI) at different temperatures

T (K)	Langmuir constant				Freundlich constant		
	X_{m1}(mg/g)	K_1(L/mg)	R_L	R^2	$1/n$(gL^{-1})	K	R^2
283	38.759	0.0276	0.645	0.9869	0.310	6.194	0.9545
293	35.842	0.0503	0.493	0.9938	0.307	6.382	0.9572
303	35.460	0.0932	0.341	0.9832	0.302	6.622	0.9506
313	35.211	0.2108	0.088	0.9698	0.302	6.776	0.9461

between 0 and 1 which indicates favorable adsorption as shown in Table 3, while Eq. (7) expressed the linear form of Freundlich isotherm which was also applied to the adsorption data of Cr(VI) as given in Table 3.

$$\log \frac{x}{m} = \log k + \frac{1}{n \log C_e} \tag{7}$$

where K (μmol g^{-1}) and $1/n$ (g L^{-1}) are Freundlich constants, indicating the adsorption capacity and adsorption intensity, respectively. Straight lines were obtained by plotting log x/m against log C_e as shown in Fig. 12 which show that the adsorption of Cr(VI) obeys Freundlich isotherm very well. Values of Freundlich constants are given in Table 3. Since the values of $1/n$ are less than 1, it indicates favorable adsorption. Smaller values of $1/n$ indicate the formation of relatively stronger bond between adsorbate and adsorbent. Data of Table 3 also shows that as temperature increases the value of $1/n$ decreases and hence maximum adsorption take place at higher temperature.

The value of correlation coefficient R^2 for Langmuir isotherm is greater than that of the R^2 of Freundlich isotherm, and it means that Langmuir isotherm is more suitable for the adsorption of Cr(VI) on carbonaceous material.

4.8. Thermodynamics study

Temperature has an important effect on the process of adsorption. The Cr(VI) adsorption on carbonaceous material was studied as a function of temperature range 273–303 K. Fig. 7 shows the effect of temperature on the adsorption of Cr(VI) on CCM from aqueous solution. It is clear from the figure that the amount of Cr(VI) adsorbed increases with rise in temperature, which means that Cr(VI) adsorption from aqueous solution endothermic in nature. The increase in the adsorption capacity may be due to the chemical interaction between adsorbate and adsorbent creation of some new adsorption sites or the increase rate of intra particle diffusion of Cr(VI) ions into the pores of the adsorbate at higher temperature.

Various thermodynamics parameters, such as free energy change, entropy change and enthalpy change of Cr(VI) adsorption, were calculated by using the following equation.

$$\ln K_1 = \frac{-\Delta H}{RT} + \frac{\Delta S}{R} \tag{8}$$

$$\Delta G = \Delta H - T\Delta S \tag{9}$$

The value of ΔH° was calculated from the slope and ΔS° from the intercept of the linear plot of lnK_1 vs.

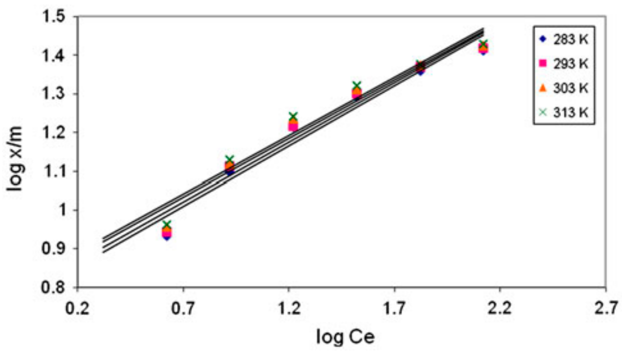

Fig. 12. Freundlich isotherm for the adsorption of Cr(VI) on CCM at different temperatures.

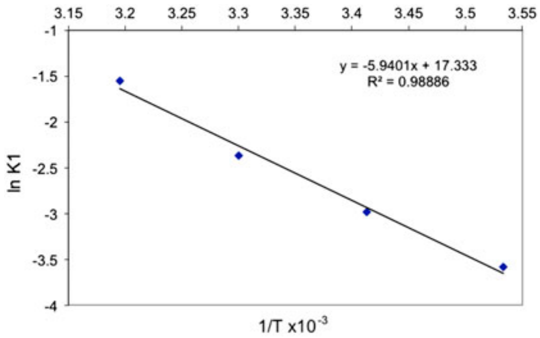

Fig. 13. ΔH° value was calculated from the slope and ΔS° from the intercept of the linear plot of lnK_1 vs. $1/T$.

Table 4
Thermodynamic parameters for the adsorption of Cr(VI) at various temperatures

T (K)	$1/T \times 10^{-3}$ (Kelvin^{-1})	ln K_1	ΔH (kJ mol^{-1})	ΔS (kJ mol^{-1} K^{-1})	ΔG (kJ mol^{-1})	R^2
283	3.533	−3.589	49.385	144.106	−40.732	0.98886
293	3.413	−2.989			−42.176	
303	3.300	−2.373			−43.614	
313	3.195	−1.556			−45.055	

$1/T$ as shown in Fig 13. The values of ΔH°, ΔS°, and ΔG° are given in Table 4. The negative value of ΔG° obtained at various temperatures show the spontaneous nature of adsorption process. The values for ΔG° decrease with the increase in temperatures. The positive value of ΔH° shows that process of adsorption is endothermic in nature. The value of entropy is positive which shows that upon adsorption on to a surface, most of the translational, and often the rotational degree of freedom are formed, and so the order of the system is decrease.

5. Conclusions

Adsorption of Cr(VI) on low-cost carbonaceous material was studied under different experimental conditions such as contact time, initial concentration of metal ions, pH, and temperature. The equilibrium time for the adsorption of Cr(VI) on activated carbonaceous material was found 90 min. The adsorption process of Cr(VI) was described by Langmuir and Freundlich isotherm models and Langmuir model show best fit than Freundlich. The kinetic was explained by Lagergren first-order and pseudo-second-order model. A good correlation coefficient was observed using pseudo-second-order kinetic model which indicate the process followed pseudo-second-order rate equation. It was observed that removal of Cr(VI) increases with increase of adsorbent dosage. The acidic pH favor adsorption of Cr(VI) on CCM and maximum adsorption occurred at pH 2–4. It was observed that CCM has good adsorption capacity for Cr(VI), and it is effectively used for the removal of Cr (VI) from the wastewater.

Acknowledgement

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