

Adsorption of Chromium (VI) onto Activated Carbon Modified with KMnO_4

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Abstract: The adsorption capacity of activated carbon modified with KMnO_4 (potassium permanganate) for Cr(VI) from aqueous solution was investigated. The modified activated carbon was characterized by SEM (scanning electron microscopy), FT-IR (Fourier transform infrared spectrometer), and N_2 adsorption/desorption tests. Adsorption of Cr(VI) from aqueous solution onto the activated carbon was investigated in a batch system. In the present study, the effect of various parameters such as pH, contact time and initial concentration on the adsorption capacity were determined by ICP-AES (inductively coupled plasma atomic emission spectrometry). The Cr(VI) adsorption on the activated carbon conforms to the Langmuir and Freundlich isothermal adsorption equation. The rates of adsorption were found to conform to pseudo-second order kinetic. The modified activated carbon can be an effective adsorbent for Cr(VI) from the aqueous solution.

Key word: Modified activated carbon, chromium, adsorption isotherms, kinetics.

1. Introduction

Heavy metals are not biodegradable and tend to accumulate in living organisms. Then, they are considered to be potentially hazardous and can cause physiological and neurological disorders [1]. Heavy metals such as Mercury, Lead, Cadmium, Copper, and Chromium are toxic even in small amounts. Chromium (Cr), one of the toxic metals, is used in a variety of applications including electroplating, nuclear power plant, water cooling and chromate preparation [2, 3]. Cr consists of two stable oxidation states such as trivalent state Cr(III) and hexavalent state Cr(VI) in natural aqueous environment. It is well known that Cr(III) is essential materials for living organisms, whereas Cr(VI) is more toxic, carcinogenic and mutagenic [4-8]. The maximum levels permitted in wastewater are $5 \text{ mg}\cdot\text{dm}^{-3}$ for trivalent Cr and $0.05 \text{ mg}\cdot\text{dm}^{-3}$ for hexavalent Cr [9].

There are many processes for treatment of metal

contaminated wastewaters, including chemical precipitation, membrane filtration, reverse osmosis, ion exchange, and adsorption. However, they have a limited use due to some disadvantages [10]. Adsorption has been proved as one of the most efficient methods for the removal of heavy metals from aqueous media [11]. Activated carbon has shown great potential for the removal of various inorganic, organic pollutants and radionuclides removal due to properties such as large surface area, microporous structure, and high adsorption capacity [12-15]. It has also been used as an adsorbent to remove heavy metals from aqueous solutions and/or wastewaters. However, the low adsorption capacity of Cr(VI) on activated carbon has restricted its wide application. Recently, surface modification of activated carbon has been recognized as an attractive approach to improve and/or enhance the removal of heavy metals from wastewater [16, 17].

Considering the above-mentioned, the uptake experiments for the adsorption of Cr(VI) from aqueous solution by activated carbon modified with

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KMnO_4 (potassium permanganate) was carried out in this work. Two kinds of the concentrations of KMnO_4 (0.01 mol/L and 0.03 mol/L) were used. The objective of the present work was to investigate the efficiency of modified activated carbon for adsorbing Cr(VI) from aqueous solution. Furthermore, to evaluate the characteristics of the activated carbon modified with KMnO_4 , the surface morphology, the specific surface area and the functional group of the material were determined by SEM (scanning electron microscope), BET (Brunauer, Emmet and Teller) method and FT-IR (Fourier transform infrared spectrometer), respectively.

2. Experimental Section

2.1 Materials

Activated carbon used in the study was purchased from the Sigma-Aldrich, Inc., USA. This product is an untreated, granular carbon with a particle size of less than 75 microns (80%-90%) (100-400 mesh). The carbon is prepared from wood which has been chemically activated.

Chemical reagents including KMnO_4 were purchased from Kanto Chemical Co., Inc. (Japan). For all experiments, each stock solution was suitably diluted with deionized water for use. All reagents used were of analytical grade, and water ($> 18.2 \text{ M}\Omega$) which was treated by an ultrapure water system (Advantec aquarius: RFU 424TA) was employed throughout the work. The pH meter (HORIBA F-72) was used for measurement of pH while adjusting the pH of the solutions using 0.1 mol/L NH_4OH aqueous/0.1 mol/L HNO_3 .

2.2 Modification of Activated Carbon with KMnO_4

The AC was washed by deionized water (at $80 \text{ }^\circ\text{C}$) to remove fine powder and contaminants, and then dried at $110 \text{ }^\circ\text{C}$ for 2 h before use. 5 g of the activated carbon were placed in a 200 mL conical flask which containing 50 mL KMnO_4 solution (0.01 or 0.03 mol/L). Adjusting the temperature to $25 \text{ }^\circ\text{C}$ and

stirring for 12 h, the resulting solution was filtrated through $0.45 \text{ }\mu\text{m}$ membrane filter. Then, the filtrate was washed with deionized water until the pH (of the filtrate) is constant. The activated carbon was dried at $70 \text{ }^\circ\text{C}$ for 6 h. The pristine and modified (i.e., modified with 0.01 mol/L and 0.03 mol/L KMnO_4 solution) activated carbons were described as AC_0 , AC_{K1} and AC_{K3} , respectively as below.

2.3 Characterization of ACs (Activated Carbons)

Various characterization methods have been used to determine physicochemical properties of pristine and modified activated carbons. The surface morphologies of these ACs were surveyed by using a SEM (Hitachi S-4300). Surface areas and pore volumes of these ACs before and after Cr(VI) adsorption was carried out by N_2 adsorption/ desorption tests (Micromeritics TriStar 3020). Surface functional groups were identified by FT-IR spectrometer (FTIR-4200, Jasco, Japan).

2.4 Adsorption Experiments of Cr(VI)

Adsorption experiments were carried out in batch processes by agitating 5-75 mg of ACs samples with $0.1 \text{ mg}\cdot\text{L}^{-1}$ Cr(VI) solution at constant pH in a thermostated bath with a shaker. For investigating the effects of pH, contact time, sorbent dose and initial concentration on the adsorption of Cr(VI), the following adsorption experiments were performed using modified activated carbons. Activated carbons were thoroughly mixed with 50 mL of containing known amount of Cr(VI) in a 200 mL conical flask, and the suspensions were shaken in a water bath at room temperature ($25 \pm 2 \text{ }^\circ\text{C}$). Adsorption studies were carried out by varying the initial concentration ($20\text{-}200 \text{ mg}\cdot\text{L}^{-1}$) and the agitation time (1-24 h) at the pH of maximum adsorption efficiency was noted.

Following each adsorption experiment, the suspension containing carbon and the above Cr(VI) solution was filtered through a $0.45 \text{ }\mu\text{m}$ membrane filter (Advantec Mixed Cellulose Ester, 47 mm) to remove Cr(VI) that have been adsorbed into the

activated carbon, and the concentration of Cr(VI) in the filtrate was determined with an ICP-AES (inductively coupled plasma atomic emission spectrometry).

The adsorption capacities of Cr(VI) using modified activated carbon at equilibrium (q_e : $\text{mg}\cdot\text{g}^{-1}$) was calculated using the following Eq. (1):

$$q_e = \frac{(C_i - C_e) \cdot V}{m} \quad (1)$$

where, C_i and C_e are the initial and equilibrium concentrations of Cr(VI) in a batch system respectively ($\text{mg}\cdot\text{L}^{-1}$), V is the volume of the solution (L), and m is the weight of adsorbent (g).

2.5 Adsorption Isotherm Model

Adsorption isotherms are commonly used to reflect the performance of adsorbents in adsorption processes. To examine the relationship between the metal uptake (q_e) and the concentration of metal ion (C_e) at equilibrium, adsorption isotherm models are widely employed for fitting the data. To get the equilibrium data, initial concentrations of metals were varied while the adsorbent weight each sample was kept constant. Langmuir and Freundlich isotherms model was applied to evaluate the adsorption data obtained in this study.

The Langmuir model assumes monolayer adsorption onto a surface and is given by:

$$\frac{C_e}{q_e} = \frac{C_e}{q_{\max}} + \frac{1}{K_L q_{\max}} \quad (2)$$

where, C_e is the concentration of Cr(VI) in a batch system at equilibrium ($\text{mg}\cdot\text{L}^{-1}$), q_e is the amount of adsorption of Cr(VI) at equilibrium ($\text{mg}\cdot\text{g}^{-1}$), q_{\max} is the maximum adsorption capacity on the surface of AC ($\text{mg}\cdot\text{g}^{-1}$), K_L is the Langmuir adsorption constant ($\text{L}\cdot\text{mg}^{-1}$) [18, 19]. A plot of C_e/q_e versus C_e gives a straight line with slope of $1/q_{\max}$, and intercept is $1/(K_L q_{\max})$.

On the other hand, the linearized Freundlich model isotherm is represented by the following Eq. (3):

$$\log_{10} q_e = \log_{10} K_F + (1/n) \log_{10} C_e \quad (3)$$

where, K_F and $1/n$ indicate the adsorption capacity and the adsorption intensity of the system, respectively. The plots of q_e versus C_e in log scale can be plotted to determine values of $1/n$ and K_F depicting the constants of Freundlich model.

2.6 Kinetic Studies

Kinetic models have been proposed to determine the mechanism of the adsorption process which provided useful data to improve the efficiency of the adsorption and feasibility of process scale-up [20, 21]. In the present investigation, the mechanism of the adsorption process was studied by fitting first-order and second-order reactions to the experimental data.

The pseudo first-order model is given by the following Eq. (4):

$$\ln(q_e - q_t) = \ln(q_e) - k_1 t \quad (4)$$

where, q_e and q_t are the adsorption capacities of Cr(III) or Cr(VI) using chitosan at equilibrium and time t , respectively ($\text{mol}\cdot\text{g}^{-1}$), and k_1 is the rate constant of the pseudo-first-order adsorption (h^{-1}).

The linear form of the pseudo second-order rate equation is given as follows:

$$\frac{t}{q_t} = \frac{1}{k q_e^2} + \frac{t}{q_e} \quad (5)$$

where, q_e and q_t are the adsorption capacities of Cr(VI) using modified activated carbon at equilibrium and time t , respectively ($\text{mol}\cdot\text{g}^{-1}$), and k is the rate constant of the pseudo-second-order adsorption ($\text{g}\cdot\text{mol}^{-1}\cdot\text{h}^{-1}$).

3. Results and Discussion

3.1 Characterization of ACs

3.1.1 Characterization of the Modified Activated Carbon

The FT-IR spectra of the pristine and modified activated carbon (i.e., AC_0 , AC_{K1} and AC_{K3}) are presented in Fig. 1. The broad and intense peak at 3,300 to 3,500 cm^{-1} corresponds to the O-H stretching

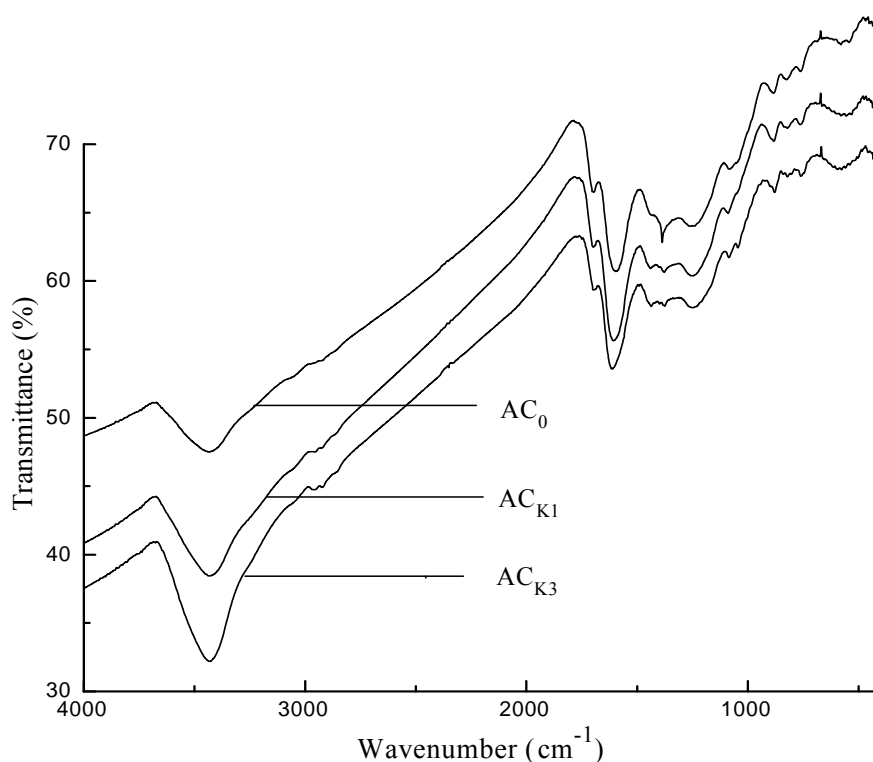


Fig. 1 FT-IR spectra of AC_0 , AC_{K1} and AC_{K3} .

Table 1 Textural characteristics of the activated carbon.

Adsorbent	BET surface area ($\text{m}^2 \cdot \text{g}^{-1}$)	Pore volume ($\text{cm}^3 \cdot \text{g}^{-1}$)	Pore size (nm)
AC_0	381	0.402	4.23
AC_{K1}	373	0.390	4.18
AC_{K3}	346	0.348	4.03

vibration of hydroxyl groups of activated carbons. The peak at $3,433 \text{ cm}^{-1}$ is related to hydroxyl groups (-OH) stretch from deprotonation process of the pristine and modified activated carbon. The wide peak at ($1,550$ to $1,750 \text{ cm}^{-1}$) shows the asymmetric stretch of the carboxylate (-COO-) group [1, 11, 22].

The surface properties of the activated carbon were investigated by N_2 adsorption (TriStar II 3020 Micromeritics); and the analytical results for the adsorption/desorption isotherms are shown in Table 1.

The pore volume was calculated from the amount of N_2 adsorbed at relative pressure of 0.99. The pore size was calculated from adsorption average pore width ($4V/A$ by BET) in this work. From Table 1, it is found that the pore volume and pore size as well as the

specific surface area decreased significantly after modification with KMnO_4 . The isotherm showed a type H1-type isotherm pattern with a clear hysteric loop, which is a typical characteristic of disordered micro porous materials.

The SEM micrographs of the ACs are shown in Fig. 2. The modified ACs (Fig. 2a and 2c) seem to exhibit more slightly compact stacking morphology than the pristine activated carbon (Fig. 2a) due to the cohesive force, which may be generated from the introduction of oxygen containing functional groups. These results are consistent with those of N_2 adsorption-desorption experiment. The decrease of the pore volume and pore size may be related to the increase of the amount of acidic groups on the surface of AC by treating with KMnO_4 .

3.2 Adsorption of Cr(VI) on Modified Activated Carbon

3.2.1 Effect of pH

The removal of metallic species from water by an adsorbent is highly dependent on the pH of the

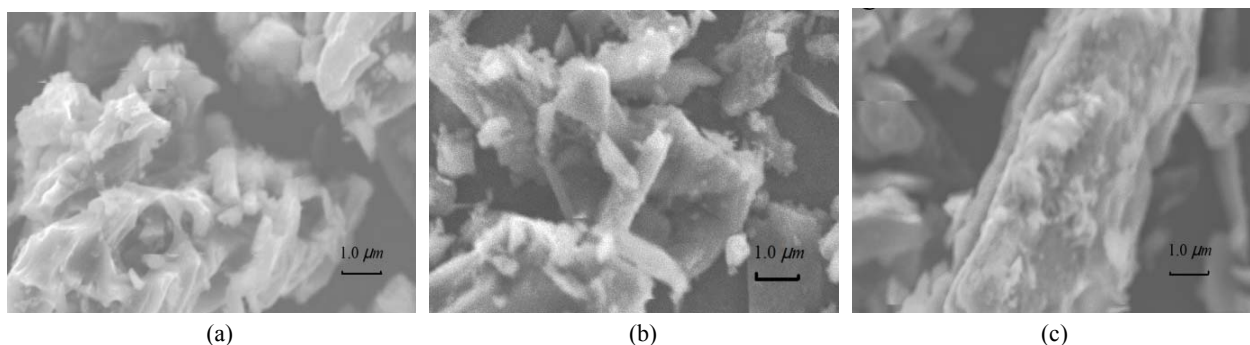
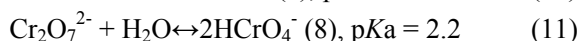
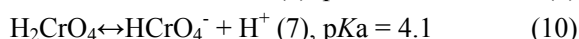
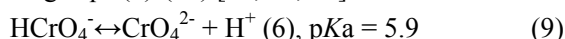
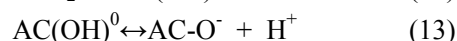
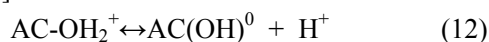


Fig. 2 SEM micrographs of the surface of the activated carbon. (a): unmodified, (b): modified with 0.01 mol/L KMnO_4 , (c): modified with 0.03 mol/L KMnO_4 .

solution, which affects the surface charge of the adsorbent, the degree of ionization and the chemical speciation of the adsorbate species [21]. To investigate the effect of solution pH on Cr(VI) adsorption efficiency, the pH of the solution was varied from 3 to 7, while the Cr(VI) concentration was kept constant at $100 \mu\text{g}\cdot\text{L}^{-1}$. The experimental results are presented in Fig. 3. From Fig. 3, it is obviously found that Cr(VI) adsorption was strongly pH dependent, and that Cr(VI) adsorption was superior at low pH [23]. The distribution of Cr(VI) at different pH is shown in Fig. 4. Cr(VI) exists as hydrogen chromate anions (HCrO_4^-) between pH 2.0 and pH 6.5 and it exists as chromate ions (CrO_4^{2-}) at pH 8 according to the following Eqs. (9)-(11) [21, 23, 24]:



On the other hand, the possible reactions on carbon surface with increasing pH can be expressed as follows [24]:



The protonated carbon surface shows great affinity to the anions because of the electrostatic attraction. Therefore, it promotes the adsorption of the negatively charged hydrogen chromate ions from the solution. Proton dissociation occurs with increasing pH, and consequently, deprotonated surface repulses the hydrogen chromate ions [24]. Cr(VI) exists as chromate ion (CrO_4^{2-}) at pH above 6.5. At pH 8, the

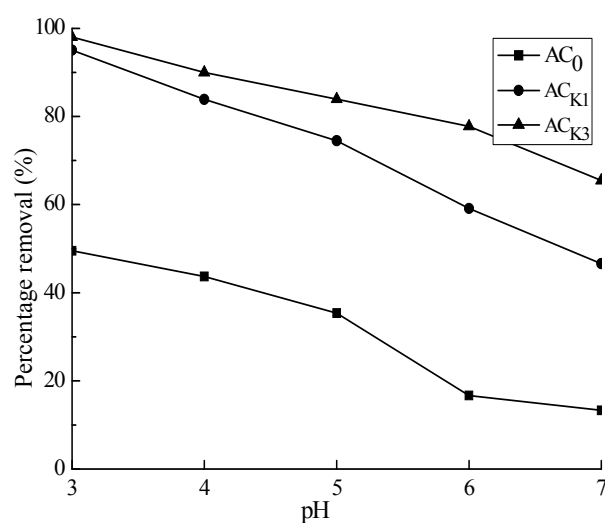


Fig. 3 Effect of pH on percent removal of Cr(VI) using modified activated carbon.

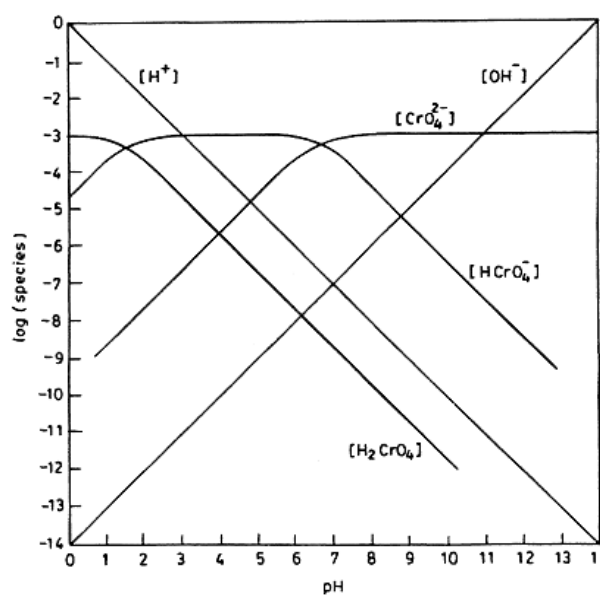


Fig. 4 Logarithmic concentration diagram for 10^{-3} M $\text{H}_2\text{Cr}_2\text{O}_4$ solution.

uptake capacities are very low. This could be explained as competitiveness between chromate and hydroxyl ions.

Hence, pH 3 was taken for further experimental work.

3.2.2 Effect of Contact Time

The effect of contact time on Cr(VI) adsorption efficiency using 1.0 g·L⁻¹ AC_{K3} (100 μg·L⁻¹ of solution) is investigated at pH 3.

The removal of Cr(VI) more than 85% was observed within 1h, and it gradually increased up to the contact time of 2 h. Approximately 95% of Cr(VI) removal from the solution was observed at the contact time of 4 h, and after that there was no appreciable change. Hence, 4 h was chosen as the optimized contact time for the rest of the experimental work.

3.2.3 Adsorption Isotherms

Adsorption isotherms are commonly used to reflect the performance of adsorbents in adsorption processes. In this paper, Langmuir and Freundlich isotherms were applied to the data obtained in this work. The linear plots of $C_e/q_e - C_e$ and $\lg q_e - \lg C_e$ were presented for Langmuir (Fig. 5) and Freundlich (Fig. 6) models, and the coefficient of both isotherms are shown in Table 2.

From Table 2, it is also found that R^2 value for each adsorbent is comparatively large. That is to say, favorable adsorption for Cr(VI) by the activated carbon is presented. This result suggests that the both physical and chemical adsorptions are existing in these ACs.

3.2.4 Kinetic Studies

The linear plot of t/q_t versus t for metal adsorption

system under the optimized experimental conditions is shown in Fig. 7. The pseudo-second-order rate constant (k) and the amount of adsorbed lead (q_e), obtained from the intercept and slope of the plot of t/q_t

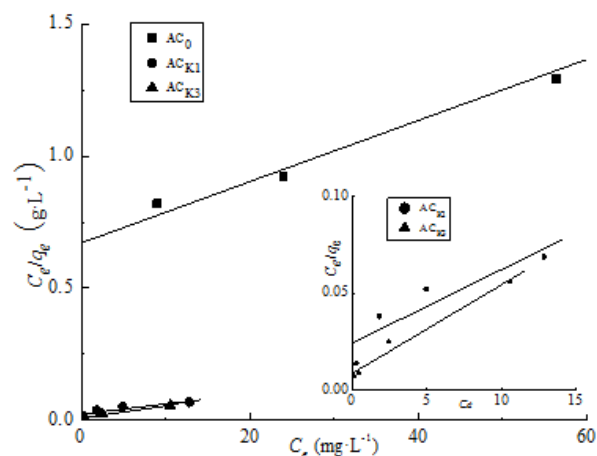


Fig. 5 Langmuir isotherm of Cr(VI) adsorption onto activated carbon.

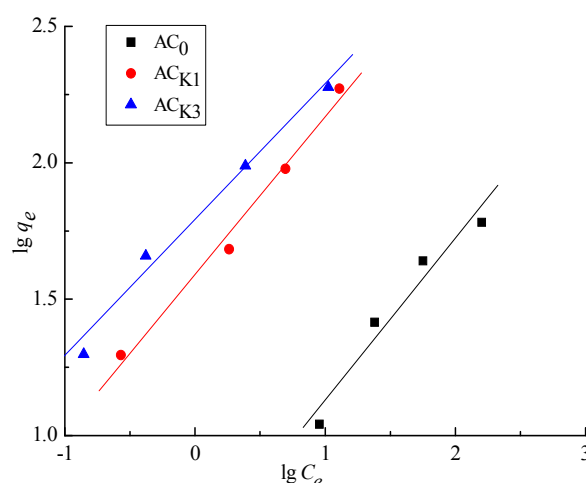


Fig. 6 Freundlich isotherm of Cr(VI) adsorption onto activated carbon.

Table 2 Coefficient of Langmuir and Freundlich isotherms for Cr(VI) using activated carbon.

	Langmuir isotherm			Freundlich isotherm		
	q_{max} (μg·g ⁻¹)	K_L (dm ⁻³ ·μg ⁻¹)	R^2	K_F (μg·g ⁻¹)· (dm ⁻³ ·μg ⁻¹) ^{1/n}	n	R^2
AC ₀	4.30E+01	1.72E-02	0.998	3.44	1.68	0.975
AC _{K1}	1.09E+02	1.88E-01	0.910	38.9	1.73	0.994
AC _{K3}	1.34E+02	4.42E-01	0.988	61.7	2.01	0.987

q_{max} : the maximum adsorption capacity on the surface of activated carbon (mg·g⁻¹);

K_L : the Langmuir adsorption constant (L·mg⁻¹);

R : the correlation coefficient.

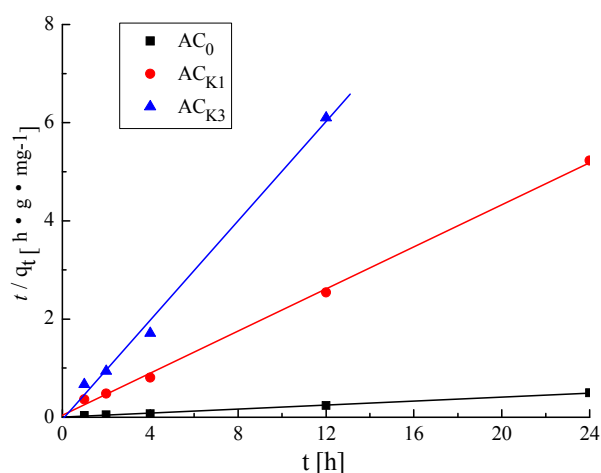


Fig. 7 The pseudo second-order kinetic model for activated carbons.

Table 3 Kinetic coefficient for Cr(VI) adsorption on activated carbon.

Activated carbon	q_e ($\mu\text{g}\cdot\text{g}^{-1}$)	k ($\text{g}\cdot\text{mol}^{-1}\cdot\text{h}^{-1}$)	R
AC_0	39.5	1.29×10^{-4}	0.998
AC_{K1}	93.2	1.15×10^{-3}	0.999
AC_{K3}	99.5	5.14×10^{-3}	0.997

q_e : the amount of adsorbed Cr(VI) on the surface of activated carbon at equilibrium ($\text{mg}\cdot\text{g}^{-1}$);

k : the rate constant of the pseudo-second-order adsorption ($\text{g}\cdot\text{mol}^{-1}\cdot\text{h}^{-1}$);

R : the correlation coefficient.

vs. t are listed in Table 4 along with the regression coefficients (R^2).

It implies that the adsorption kinetics based on the experimental values are in good agreement with the pseudo second-order kinetic model. The intraparticle diffusion model indicated that the relationship between the concentration of Cr(VI) and the square root of time are linear. This suggests that the adsorption process could be controlled by intraparticle diffusion. Moreover, the adsorption at higher temperatures became more dependent on intraparticle diffusion, which would be the rate-determining step.

4. Conclusions

The modified AC was prepared with KMnO_4 (0.01 mol/L and 0.03 mol/L). The activated carbon modified with KMnO_4 exhibits higher adsorption capacity and

stronger chemical affinity than pristine activated carbon. The removal of Cr(VI) is more than 95% under this experimental conditions. The isotherm models such as Langmuir and Freundlich were used to analyze the adsorption data. The adsorption of Cr(VI) by the activated carbon was well fitted to Langmuir and Freundlich isotherms. The kinetic data was found to be applicable to pseudo-second order kinetic model.

From these results, the chemically modified activated carbon can be an effective adsorbent for Cr(VI) from aqueous solution, which provide very significant information from the viewpoint of environmental protection.

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