

Removal of Low Concentration Cr(VI) from Aqueous Solution by Modified Wheat Straw

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ABSTRACT: Two kinds of adsorbents were prepared by modification of wheat straw using succinic anhydride and acrylic acid. The two adsorbents were characterized by Fourier transform infrared spectroscopy, and it was found that the carboxyl groups were introduced into the straw fiber. The use of the modified wheat straws for the removal of low concentration Cr(VI) from aqueous solutions were investigated with a variety of pH value, temperature, contact time, adsorbent dosages, and initial Cr(VI) concentration. The removal percentage achieved 99.7% at an initial Cr(VI) concentration of 1 mg/L. The adsorption kinetics experiments were carried out, and the obtained data were fitted by the pseudo-first-order equation, pseudo-second-order equation, and intra-particle diffusion equation. The adsorption kinetics was found to conform to the pseudo-second-order equation with a good correlation. This study reports that the modified wheat straws are better adsorbents for the removal of low concentration Cr(VI) from aqueous solutions than crude wheat straw. © 2012 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* 129: 1555–1562, 2013

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INTRODUCTION

Chromium (Cr) exists in wastewater of industries such as nuclear power plants, electroplating, leather tanning, and so forth.¹ It has mainly two oxidation states in aqueous solutions, Cr(III) and Cr(VI). The predominant forms of Cr(VI) are $\text{Cr}_2\text{O}_7^{2-}$, HCrO_4^- , and CrO_4^{2-} in the industrial wastewater. Compared to Cr(III), Cr(VI) has the strange toxic to animal and plants even at a low concentration, because it is a strong oxidizing agent and a potential carcinogen.^{2,3} It is, therefore, very important to remove Cr(VI) from the water. Numerous methods were used to remove Cr(VI) from aqueous solutions. Amongst these methods are neutralization, precipitation, ion exchange, and carbon adsorption.^{4–6} The most widely used adsorbent is activated carbon, which is an expensive product for the removal of low concentration Cr(VI) from industrial wastewater.⁷ It is promising to find new low-cost adsorbents to remove Cr(VI) from wastewater efficiently. Considerable research work has been done for developing inexpensive adsorbents such as coir pith,⁸ Fe-modified steam wheat straw,⁹ wheat-residue derived black carbon,¹⁰ *Aeromonas caviae*,¹¹ sunflower straw-modified by phosphoric acid,¹² and barley straw.¹³

Recently, adsorbents that prepared using agriculture by-products have been concerned. Wheat, one of the most abundant agricul-

ture plants, yields 110 billion tons of straw each year in China.¹⁴ Some researchers have used wheat straw to remove metal ions from aqueous solutions,¹³ but the removal efficiency of crude wheat straw for metal ions is lower. In order to enhance its removal efficiency, some functional groups such as carboxyl and amine groups were introduced on the surface of wheat straw.^{15,16} These groups on the surface of modified wheat straw have the ability to bind metal ions.¹⁷ Because wheat straw is mainly composed of cellulose, hemicellulose, and lignin,¹⁸ which all have a large number of available hydroxyl groups, modification of wheat straw could be achieved. The preparations of adsorbents based on wheat straw have become an research interest, such as citric acid functionalized wheat straw.¹⁹ However, there are few studies on the efficient removal of low concentration Cr(VI) from aqueous solutions, such as drinking water, using modified wheat straw.

We have studied acetylation of wheat straw, rice straw, and sugarcane bagasse,^{20–22} and the products have been recommended as natural sorbent in oil spill cleanup. In present work, two kinds of adsorbents were prepared from wheat straw to remove low concentration Cr(VI) from aqueous solution. The effects of the solution pH, contact time, temperature, adsorbent dosages, and initial Cr(VI) concentration on Cr(VI) removal were investigated

to determine optimum adsorption conditions. Kinetic models were applied to study the adsorption kinetics and adsorption mechanism of Cr(VI) onto modified wheat straw.

EXPERIMENTAL

Materials and Reagents

Wheat straw was obtained from Xi'an countryside (China), and the straw was washed with distilled water to remove all the dirt materials. After being dried at 60°C in an oven for 16 h, the straw was ground to pass through a 1.0 mm screen and stored at 5°C. Succinic anhydride, dimethylacetamide (DMAc), 4-dimethylaminopyridine (DMAP), and acrylic acid were purchased from the Sigma Company (Beijing, China). All chemicals used were of analytical grade.

Succinylation of Wheat Straw with Succinic Anhydride

According to the procedure of the succinylation of wheat straw hemicelluloses (Scheme 1),²³ wheat straw (10 g) was mixed with succinic anhydride (15 g) in a beaker, and then added 200 mL DMAc as solvent and 1.5 g DMAP as catalyst. The mixture was stirred for 1 h at 100°C in an oil bath, and then the reaction solution was filtered and washed with distilled water and ethanol, and the modified wheat straw using succinic anhydride (BWS) were dried at 60°C for 16 h.

Graft Co-polymerization of Wheat Straw with Acrylic Acid

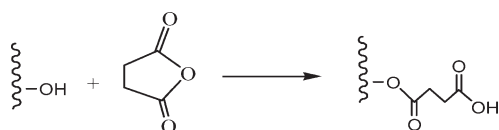
Wheat straw (10 g) was mixed with 0.1 M KMnO₄ solution (200 mL) for 30 min in a water bath at room temperature, and then added 50 mL acrylic acid. The mixture was stirred for 2 h at 69°C, and the reaction condition is based on the procedure described by Marchetti (Scheme 2).²⁴ The modified wheat straw using acrylic acid (AWS) was washed with distilled water and alcohol, filtered, and dried at 60°C for 16 h.

FT-IR Analysis

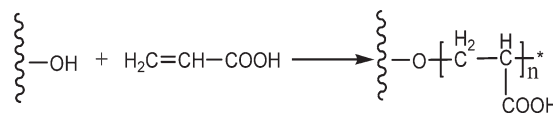
The modified wheat straw were analyzed using Fourier transform infrared spectrophotometer (Nicolet 510) within a frequency range of 4000–400 cm⁻¹ after the samples were milled and mixed with a KBr disc.

Preparation of Cr(VI) Solution and Analytical Methods

The stock solution of Cr(VI) was prepared by dissolving an accurately weighed amount of analytical grade K₂Cr₂O₇ in distilled water to the concentration of 100 mg/L. All test solutions were obtained by diluting the Cr(VI) stock solution into different initial concentrations. Diluted NaOH and HNO₃ solutions were used for the adjustment of pH value. Because 1,5-diphenylcarbazide and Cr(VI) can form the pink-colored complex, which present the maximum absorb peak at 540 nm, the concentration of Cr(VI) in the solutions was determined by UV spectrophotometer using 1,5-diphenylcarbazide at 540 nm.²⁵



Scheme 1. Succinylation of wheat straw with succinic anhydride.



Scheme 2. Graft co-polymerization of wheat straw with acrylic acid.

Adsorption Experiments

A certain amount of modified wheat straw sample and 20 mL of chromium solution were added in a conical flask, which was sealed to avoid the evaporation of the solution. The flask was placed in a water bath oscillator at a 180 rev/min rotation speed for the predetermined duration, and then the sample was filtered. The concentration of the residual Cr(VI) in the filtrate was determined by UV spectrophotometer. The adsorptive quantity q (mg Kg⁻¹) of Cr(VI) using modified wheat straw was calculated using following equation:

$$q = \frac{(C_o - C_e)V}{m}$$

where C_o and C_e represent the initial and remained Cr(VI) concentrations in mg/L, respectively; V is the volume of Cr(VI) solution in mL; m is the dry weight of the modified wheat straw in g.

The removal percentage of Cr(VI) was determined from the concentrations of Cr(VI) in the solution and particulate phase using following equation:

$$\text{Removal \% of Cr(VI)} = \frac{C_o - C_e}{C_o} \times 100\%$$

where C_o (mg/L) represents the initial concentration; C_e (mg/L) is the remained Cr(VI) concentration.

Kinetic Studies

The adsorptive kinetic experiments were carried out by adding 0.5 g BWS or AWS in several sets of 100 mL conical flasks containing 20 mL of Cr(VI) solution with an initial concentration of 1 mg/L and pH 2.0. The conical flasks were placed in a water bath oscillator at a 180 rev/min rotation speed at 30°C. These flasks were taken from the oscillator after different contact time, and the remained Cr(VI) concentration in the solution was determined by UV spectrophotometer. Adsorption kinetic models are used to explain the adsorption mechanism and adsorption process. To describe the adsorption process of Cr(VI) by modified wheat straw, three kinetic equations were used. The applicability of these equations was tested for both BWS and AWS. The data that obtained by the adsorptive kinetic experiments was fitted with the three kinetic models as follow:

Linear form of the pseudo-first-order equation is expressed as:²⁶

$$\lg(q_e - q_t) = \lg q_e - \frac{k_1 t}{2.303}$$

Linear form of the pseudo-second-order equation is expressed as:²⁷

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e}$$

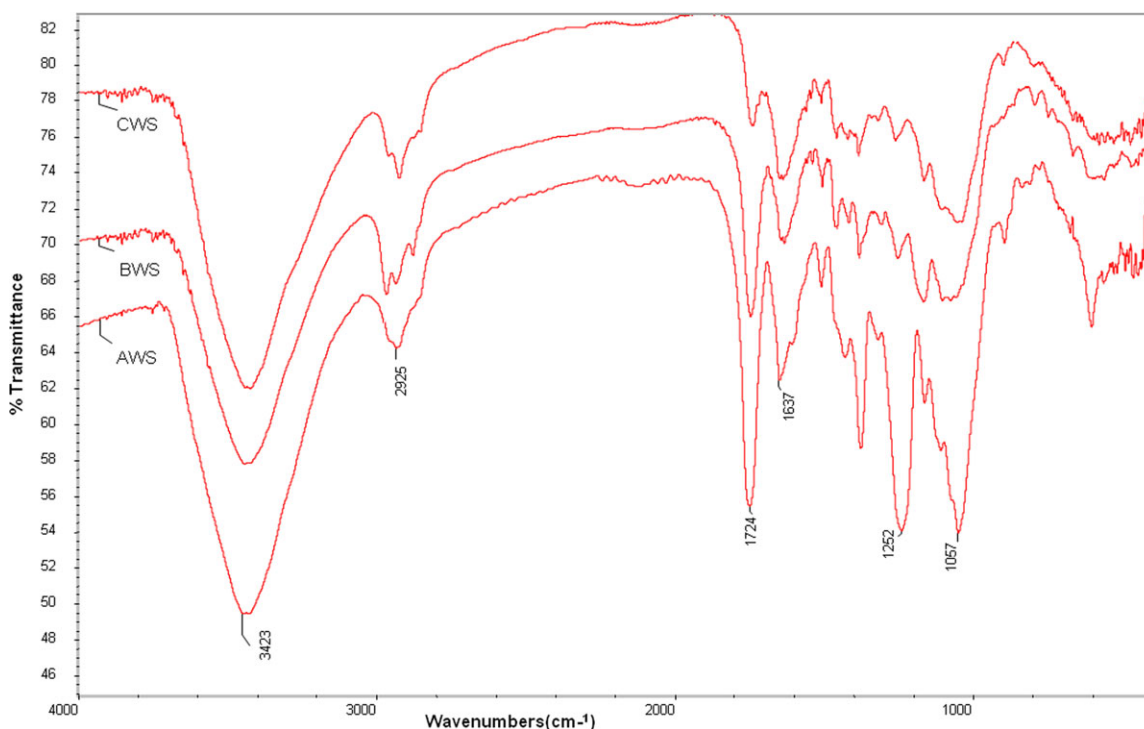


Figure 1. FT-IR spectra of CWS, BWS, and AWS. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Expression of intra-particle diffusion model:¹³

$$q_t = k_{id}t^{1/2} + C_i$$

where q_t is the amount of Cr(VI) absorbed at time t and q_e is the absorbed amount at equilibrium; k_1 and k_2 are the pseudo-first-order rate constant and the pseudo-second-order rate constant, respectively; k_{id} is the diffusion rate of intra-particle diffusion model; t represents time; C_i is a constant that provides information about the thickness of the boundary layer. When the value of C_i is high, the boundary layer effect is significant.

RESULTS AND DISCUSSION

Weight Percent Gain of Modified Wheat Straw

The weight percent gain (WPG) of the modified wheat straw was calculated by the equation: $WPG (\%) = [(weight\ gain/original\ weight)] \times 100$, and the WPG values of BWS and AWS are 36.8% and 27.3%, respectively. The WPG value represents the mean of at least triplicate reaction results. Standard errors or deviations were observed to be lower than 0.1%. The WPG of AWS is same as the result (28%) obtained by Marchetti,²⁴ and this indicated that the graft-co-polymerization of wheat straw using acrylic acid under the used condition was successful. The high WPG of BWS suggested that the succinylation of wheat straw was more easily performed than the graft co-polymerization of wheat straw using acrylic acid which may only take place on the surface of straw fiber.

FT-IR Characterization of Modified Wheat Straw

The FT-IR analysis is usually used to identify some characteristic functional groups in the molecules. Figure 1 shows the FT-IR spectra of crude wheat straw (CWS), BWS and AWS. As shown in Figure 1, the three spectra displayed a large number of absorption

peaks, and this indicated that their structures are relatively complex and there are many characteristic functional groups. The broad absorption peaks around 3423/cm was attributed to the existence of hydroxyl groups. Wheat straw is mainly composed of cellulose, hemicellulose, and lignin, which all contain a large number of hydroxyl groups. The peaks observed at 2925/cm and 1374/cm were due to the stretch vibration and bending vibration of C—H bond in methyl or methylene group, respectively. The peak around 1724/cm was resulted from stretch vibration of carbonyl groups in hemicellulosic and lignin polymers.¹⁸

The major observation in the FT-IR spectrum of BWS is an obvious enhance in the stretching vibration absorbance of carbonyl groups at 1724/cm. The increased intensity was resulted from the changes in electric dipole moment because of the introduction of carbonyl group. In general, the absorption of carbonyl groups in esters gives peaks at 1740/cm, and one in carboxylic acids exhibits a band at 1700/cm.²⁸ The two bands were strongly overlapped and therefore gave rise to a peak centered at 1724/cm.

Through the comparison of the FT-IR spectra of AWS and CWS, it was found that the FT-IR spectrum of AWS had an obvious peak at 1252/cm, which was due to the stretch vibration and bending vibration of C—O—C bond, and the strong characteristic absorptions of carboxylic acid at 1724/cm and 3423/cm indicated that the graft co-polymerization of wheat straw with acrylic acid was successful.

It was well shown that carboxyl and hydroxyl groups were present in abundance in BWS and AWS. Since these groups may function as proton donors, deprotonated hydroxyl and carboxyl groups would involve in coordination with metal ions. The rough surface and functional groups of BWS and AWS are beneficial to remove metal ions from aqueous solutions.

Table I. Removal Percentage of Cr(VI) by Crude Wheat Straw and Modified Wheat Straw

Adsorbent	AWS	BWS	CWS
Removal percentage	99.7	99.4	58.2

Removal Percentages of Crude Wheat Straw and Modified Wheat Straw

Table I presents the removal percentages of Cr(VI) by crude wheat straw (CWS), AWS, and BWS, and the experiments were performed under the conditions of adsorbent dosage 0.5 g, contact time 2 h, pH 2.0, 30°C, and initial Cr(VI) concentration of 1 mg/L. It can be seen that the removal percentage by modified wheat straw was very high, and it was up to above 99.7% at an initial concentration of 1 mg/L. This indicated that it was beneficial to introduce carboxyl groups into wheat straw for chromium adsorption, attributing to offer more adsorption sites. During the treatments of industrial wastewater and surface water in North China, the low concentration Cr(VI) ions are difficult to remove, but these remaining Cr(VI) ions have strong toxicity to animal and plants. The prepared wheat straw adsorbents can remove Cr(VI) ions efficiently from these water areas.

Effect of pH on Cr(VI) Removal

The pH value of aqueous solution is an important factor for Cr(VI) removal. The effect of pH value on adsorptive capacity is mainly due to the chemical form of Cr(VI) in the solution at a specific pH, and pH controls the surface charge and surface properties of adsorbent as well as degree of ionization.²⁹ Because of the functional groups on the modified wheat straw, which offer adsorptive sites for metal ion binding at a specific pH, the removal efficiencies had direct relation with pH value. The effect of pH on Cr(VI) removal was investigated over a range from pH 2 to 10 with a contact time of 4 h at 30°C. The obtained results are shown in Figure 2, and it is evident that the removal percentage of Cr(VI) was higher at lower pH (<2.5), and the removal percentage arrived above 97.0%. When the pH value increased to 5, the removal percentage of Cr(VI) markedly decreased to 70.0%. The removal efficiency using AWS was hardly affected at pH 5–7 and decreased again at pH above 7. The removal efficiency using BWS was continuously decreased over a range from pH 5 to 10, and the removal percentage was only 46.1% at pH 10. As shown in the Figure 2, pH 2 was the best adsorption condition. The higher removal percentage at low pH was resulted from the direct reduction of Cr(VI) to Cr(III) in the solution and the electrostatic attraction between the Cr(VI) species and adsorbent surface.⁸ According to Chen's study,¹² Cr(VI) in the solution mainly existed with the forms of HCrO_4^- , CrO_4^{2-} , and $\text{Cr}_2\text{O}_7^{2-}$ at low pH, and these ions are easily adsorbed to the proton of the adsorption point by electrostatic adsorption. In addition, Cr(VI) has strong oxidation in the acidic environment, and it may occur that the oxidation and reduction reactions with $[\text{Oxygen bond}]\text{OH}$ of oxygen-containing acid functional groups such as carboxylic acid, thus the removal percentage of Cr(VI) was high at acidic condition. On the other hand, the entire surface of the adsorb-

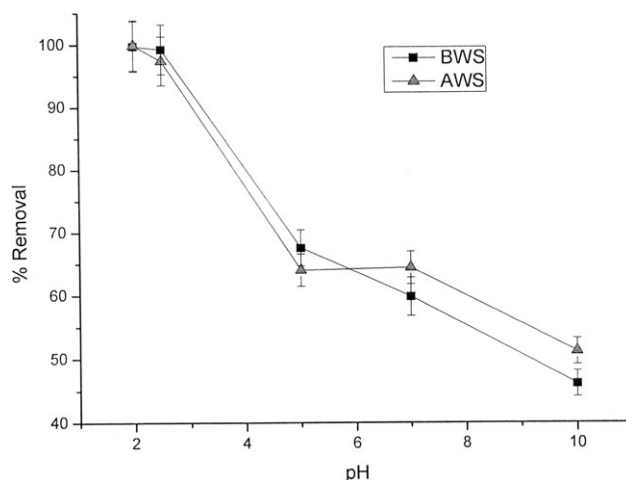
ent become increasingly negative charge with increasing pH value, which lead to the decrease of absorption capacity because of mutual exclusion; and the $[\text{Oxygen bond}]\text{OH}$ is oxidized at higher pH, and this also would reduce their adsorption capacity.

Effect of Temperature on Cr(VI) Removal

A study of the temperature dependence of adsorption process gave valuable knowledge about the enthalpy and entropy changes during adsorption. The experiments were performed under the conditions of adsorbent dosage 0.5 g, contact time 2 h, pH 2.0, and initial Cr(VI) concentration of 1 mg/L. The removal of Cr(VI) by BWS or AWS was studied at various temperatures ranging from 3°C to 45°C. The results are shown in Figure 3(a), and it is very clear that the removal percentage of Cr(VI) increased when the temperature was high, especially for BWS, indicating that the adsorption of Cr(VI) onto the modified wheat straw was endothermic in nature. This may be attributed to the chemisorptions reaction or activated adsorption between Cr(VI) and functional groups on the surface of the modified wheat straw, which involved valences forces through sharing or exchanges of electrons between the modified wheat straw and Cr(VI).³⁰ When the temperature increased to 25°C, the removal percentage inclined to stabilization. Adsorption process had achieved its equilibrium at 30°C, and the removal percentage of Cr(VI) by BWS and AWS was 99.9%, 99.7%, respectively. The result indicated that BWS or AWS, as an adsorbent, can give an excellent removal percentage at the temperature (25–30°C). Therefore, the modified wheat straw is very advantage for dealing with wastewater containing Cr(VI) at room temperature.

Effect of Contact Time on Cr(VI) Removal

The time that was taken to attain equilibrium is an important parameter to predict the efficiency and feasibility of an adsorbent when this adsorbent is used for removing metal ion from water solution. Effect of contact time on Cr(VI) removal by the modified wheat straw was studied using batch contact time ranging from 0.5 h to 6 h. In these experiments, other parameters were, excepting contact time, kept constant. As shown in

**Figure 2.** Effect of pH on Cr(VI) removal by BWS and AWS.

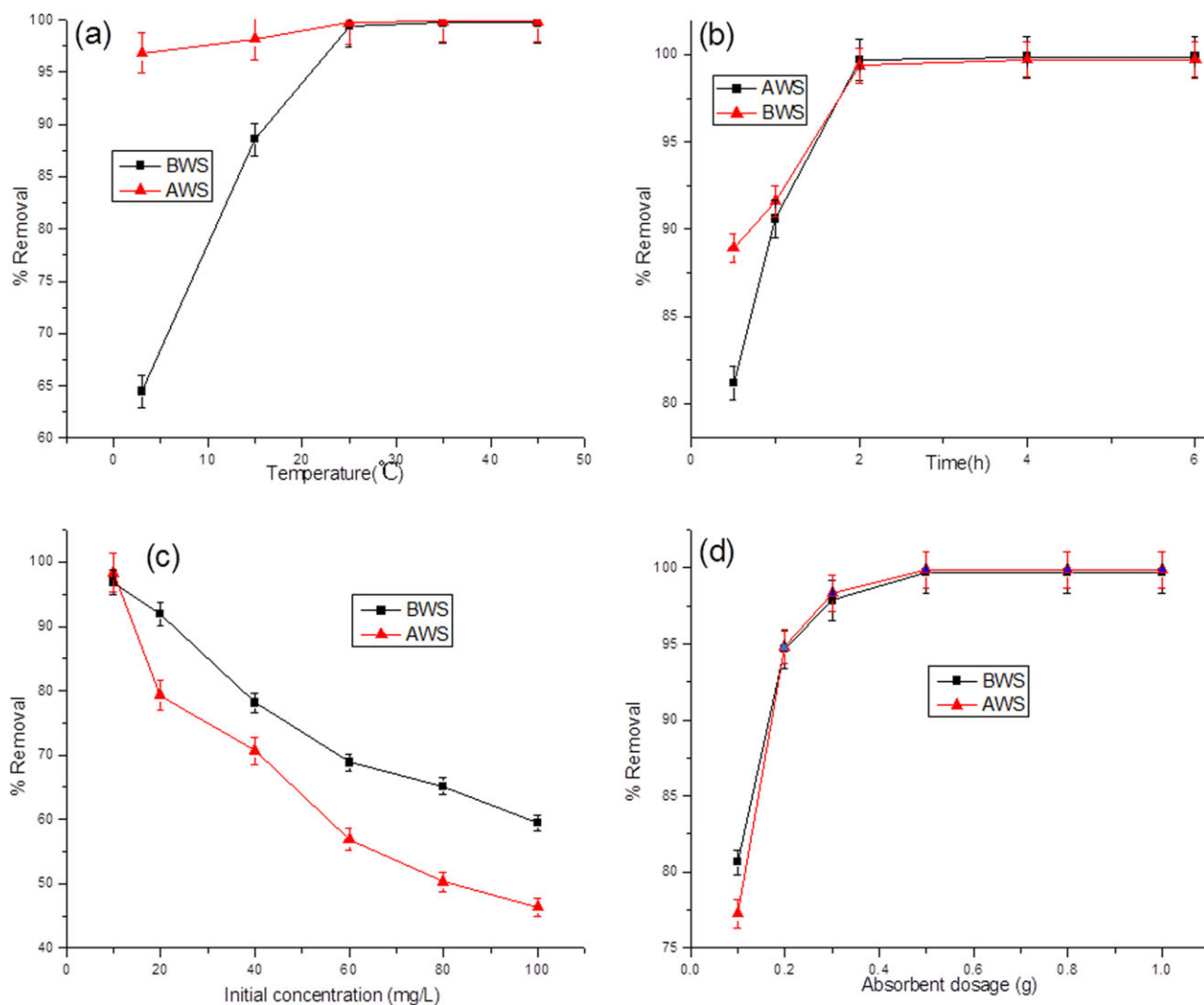


Figure 3. Effects of temperature (a), contact time (b), initial concentration (c), and absorbent dosage (d) on Cr(VI) removal by BWS and AWS. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Figure 3(b), the removal percentage by BWS and AWS enhanced with an increase of contact time. During the initial 2 h, it can be observed that the removal percentage of Cr(VI) was rapidly increased. Adsorption process reached to the equilibrium in 4 h, and the removal percentages were above 99.0% for both BWS and AWS. The adsorption sites were open and Cr(VI) interacted easily with the sites during the initial absorption period, hence a higher percentage of removal was obtained.

Effect of Initial Cr(VI) Concentration on Cr(VI) Removal

The effect of initial Cr(VI) concentration on Cr(VI) removal is shown in Figure 3(c). When the initial concentration increased from 10 mg/L to 100 mg/L, the removal percentage of Cr(VI) decreased. The decrease in the removal percentage at this range of the initial concentration may be attributed to a competition of the Cr(VI) ions for the adsorption sites available. It was found that the decreased extents were slightly different for the two adsorbents, BWS and AWS. The removal percentage of Cr(VI) by BWS decreased from 96.8% to 59.4%, while the removal percentage of Cr(VI) by AWS decreased from 98.4% to

46.4%. This indicated that initial Cr(VI) concentration more markedly affected the adsorptive Cr(VI) amount using AWS than that using BWS. However, the amount of Cr(VI) that adsorbed by two adsorbents enhanced with an increase of initial Cr(VI) concentration. It may be because higher initial concentration led to faster and stronger binding sites in comparison with lower initial concentration.¹²

Effect of Absorbent Dosage on Cr(VI) Removal

The effect of absorbent dosage on Cr(VI) removal by BWS or AWS is shown in Figure 3(d). When absorbent dosage increased from 0.1 g to 1.0 g, the removal percentage of Cr(VI) increased significantly. This may be resulted from the increase of effective adsorption sites which remained unsaturated when absorbent dosage was increased.³¹ With more than 0.5 g of absorbent dosage in 20 mL solution, the equilibrium adsorption of Cr(VI) was achieved in 2 h and the removal percentage of Cr(VI) become stable, and this indicated that it was ideal when absorbent dosage was 25 g/L at the initial concentration. Otherwise, with increasing absorbent dosage, it is better to increase initial

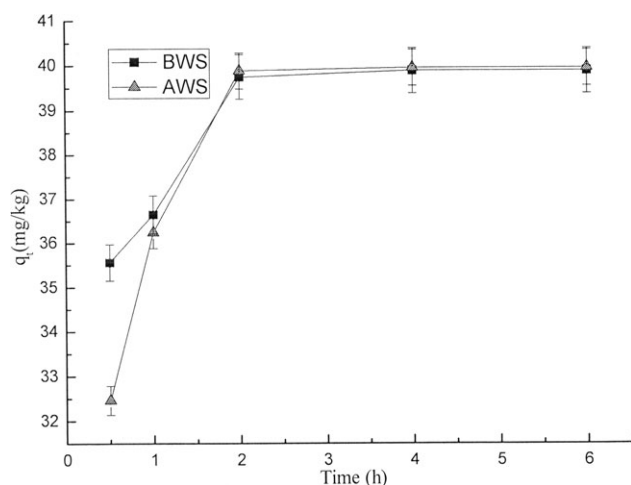


Figure 4. The adsorption kinetic plot for Cr(VI) removal by AWS and BWS ($t = 30^\circ\text{C}$, $C_o = 1 \text{ mg/L}$, adsorbent dosage = 0.5 g, $V_o = 20 \text{ mL}$).

concentration, that is, when initial concentration is high, the adsorbent dosage should be increased to gain a high removal percentage.

Adsorption Kinetics Studies

Figure 4 shows a plot of the absorbed Cr(VI) amount (mg/kg) vs. contact time when keeping other parameters constant. It was observed that the uptake amount of Cr(VI) increased with an extension of contact time from 0.5 h to 3 h, and the adsorption process was rapid for the initial 2 h, and then it proceeded at a slower rate and sustained stable.

The fitting plots using the pseudo-first-order equation, pseudo-second-order equation, and intra particle diffusion equation are described in Figures 5–7. The slopes and intercepts of these curves were used to determine the values of k_1 , k_2 , k_{id} , q_e , and C_i . The kinetic parameters that were acquired from the fitting results are summarized in Table II.

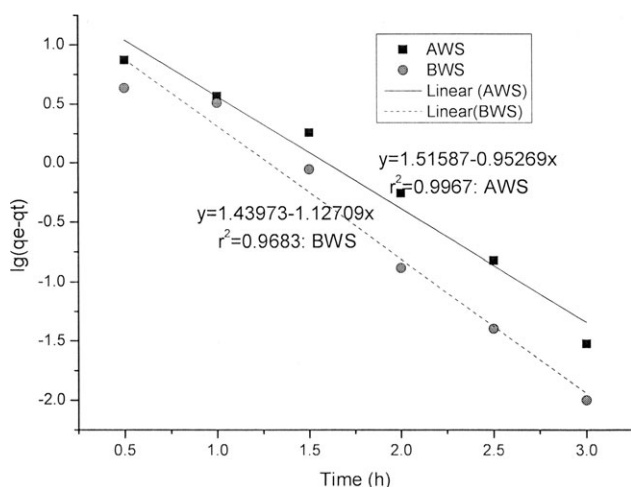


Figure 5. Pseudo-first-order adsorption kinetics of Cr(VI) onto BWS and AWS.

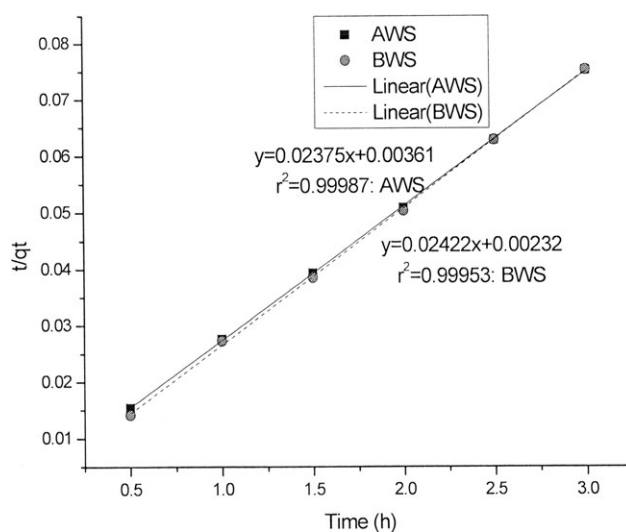


Figure 6. Pseudo-second-order adsorption kinetics of Cr(VI) onto BWS and AWS.

Table II shows that the correlation coefficient (R^2) values (>0.96) were slightly different between the pseudo-first-order equation and pseudo-second-order equation, but the q_e^b value that was calculated from the pseudo-second-order equation is much closer to the experimental q_e^a value than the value that calculated from the pseudo-first-order equation. It is concluded that the adsorption of Cr(VI) by AWS or BWS can be considered conforming to the pseudo-second-order equation. This suggests that the adsorption process is a chemisorptions process involving exchange or sharing of electrons between Cr(VI) and functional groups of the modified wheat straw.

Figure 7 presents the plot of q_t vs. $t^{1/2}$ for Cr(VI) adsorption onto AWS or BWS, respectively. The values of k_{id} and C_i are also listed in Table II. The experimental data that obtained by

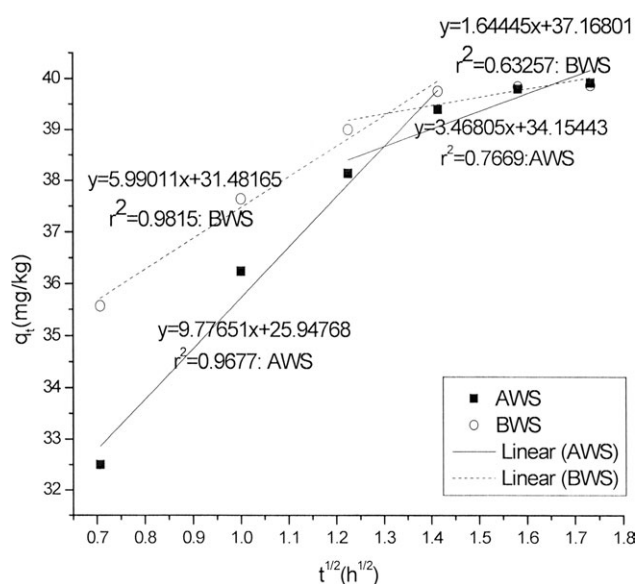


Figure 7. Intraparticle diffusion kinetics of Cr(VI) onto BWS and AWS.

Table II. Kinetic Parameters for Cr(VI) Removal

	AWS	BWS
q_e^a , mg/kg	39.95	39.88
Pseudo-first-order		
K_1 , per hour	2.19	2.60
q_e^b , mg/kg	32.80	27.53
R^2	0.9667	0.9683
Pseudo-second-order		
K_2 , kg/mg min	0.15625	0.26664
q_e^b , mg/kg	42.11	41.29
R^2	0.99987	0.99953
Intraparticle diffusion		
$+K_{1d}$, mg/kg h ^{1/2}	9.78	5.99
C_1	25.95	31.48
R^2	0.9677	0.9815
K_{2d} , mg/kg h ^{1/2}	3.47	1.64
C_2	34.15	37.17
R^2	0.7969	0.63257

^aExperimental; ^bCalculated.

using AWS or BWS gave two straight lines, indicating two different stages in the adsorption process. It showed that the external resistance to mass transfers surrounding the particles is significant only in the early stage of the adsorption, and this is represented by the first sharper linear portion. The second linear portion indicated the gradual adsorption stage with intraparticle diffusion dominating.³² C_1 and C_2 were not zero, and the lines did not pass through the origin point, and this indicated that pore diffusion was not the limiting step. Therefore the adsorption process may be of a complex nature consisting of both surface adsorption and intraparticle diffusion.^{15,33}

CONCLUSIONS

This study reports that AWS and BWS can be used as adsorbents for the removal of low concentration Cr(VI) from aqueous solutions. The modified wheat straw can be filled into a column, and the separation column can be used for the treatment of industrial wastewater, and the column also can be reused and the adsorbed metals can be recovered after washed with an alkali solution. In comparison with common adsorbents, the modified wheat straws are better adsorbents for the removal of low concentration Cr(VI) from aqueous solutions because of the high removal percentage (up to 99.7%).

Removal percentage improved with increasing temperature, contact time, and adsorbent dosage, and declined with increasing initial Cr(VI) concentration and pH value. The adsorption kinetic data were fitted by the pseudo-first-order equation, the pseudo-second-order equation, and intraparticle diffusion equation. The adsorption kinetic was found to conform to pseudo-second-order equation with a good correlation. The adsorption process may be of a complex nature consisting of both surface adsorption and intraparticle diffusion.

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