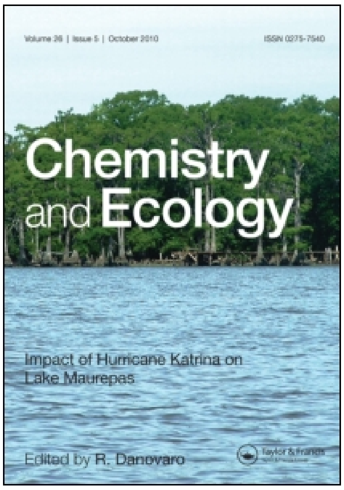

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Removal of toxic chromium(VI) from aqueous solution by activated carbon using *Casuarina equisetifolia*

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Highly activated carbon from the seed husk of Casuarina *Casuarinas equisetifolia*, a worldwide famous plant, have been prepared and tested for the removal of toxic Cr(VI) from its aqueous solution. The adsorbent was investigated for influences of initial chromium concentration (75, 100, 125, and 150 mg l^{-1}), pH, contact time, and quantity of carbon on removal of Cr(VI) from aqueous solution at room temperature (25 ± 2 °C). The adsorption kinetic of Cr(VI) was studied, and the rates of sorption were found to conform to pseudo-second-order kinetics with a good correlation ($R^2 \geq 0.99$). The Langmuir and Freundlich models fit the isotherm data well. Furthermore, the Gibbs free energy was obtained for each system and was found to be -5.29 kJ mol^{-1} for removal of Cr(IV). The negative value of $\Delta G°$ indicates the feasibility and spontaneous nature of adsorption. The results indicate that acidic pH (1.05) supported the adsorption of Cr(IV) on activated carbon. The maximum adsorption capacity of Cr(VI) on activated carbon was about 172.4 mg g^{-1} at pH 1.05.

Keywords: Casuarina; *Casuarinas equisetifolia*; Activated carbon; Chromium; Adsorption; Langmuir model; Freundlich model

1. Introduction

Water pollution due to toxic heavy metals has been a major cause of concern for chemists and environmental engineers. The development of modern industry has been increasingly responsible for serious pollution in the environment inhabited by humans, with significant health risks, including cancer [1]. Chromium occurs in wastewaters in both trivalent and hexavalent forms. The latter form is present in effluents produced during the electroplating, leather tanning, cement, dyeing, fertilizer, and photography industries, and causes severe environmental and public-health problems. It is more hazardous to living organisms than the trivalent form [1]. It is toxic to plants and animal microorganisms and causes human lung cancer, as well as kidney, liver, and gastric damage [2]. Chromium concentrations in industrial wastewaters are ranged from 0.5 to 270 mg l^{-1} [3], whereas the Environmental Protection Agency (EPA) has defined the tolerance limit for Cr discharge into inland surface waters by

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0.1 mg l^{-1} and in potable water by 0.05 mg l^{-1} [4–6]. Therefore, to comply with this limit, it is essential that industries treat their effluents to reduce the Cr(VI) to acceptable levels. The sorption of metal ions from water plays an important role in water pollution control, when using agricultural by-products as a low-cost substitute for the treatment of waters containing heavy metals [7].

Casuarina *Casuarinas equisetifolia* is a worldwide switched plant with principal photosynthesizing function in transfer stems-leaves, accumulated starch exclusively with nitrogen fixing root nodules and timber trees in [8, 9]. This paper examines the removal of Cr(VI) from its aqueous solution using active carbon prepared from seed husk of *C. equisetifolia*. The influences of pH, contact time, and amount of carbon on the removal process as well as kinetic analyses were studied to evaluate the obtained activated carbon.

2. Materials and methods

2.1 Sampling and activated carbon preparation

Seed husk of *C. equisetifolia* was collected from El-Montazah trees in Alexandria, Egypt. This material is a zero-value agricultural waste product. The collected sample was washed with tap water and then distilled water, and dried at 105 °C for 3 d. The dried seed husk (1 kg) was suspended in 1 l of 98% H_2SO_4 for 24 h, followed by refluxing in a fume hood for 10 h. After cooling, the reaction mixture was filtered, and the filtrate was washed repeatedly with distilled water and soaked in 1% $NaHCO_3$ solution to remove any remaining acid. The sample was then washed with distilled water until the pH of the activated carbon reached 6, dried in an oven at 150 °C for 48 h, sieved to a particle size of $\leq$0.63 mm, and then kept in a glass bottle.

2.2 Preparation of Cr(VI) solution

The adsorption capability of the prepared activated carbon toward Cr(VI) was investigated using its aqueous solutions. A stock solution of 1000 mg l^{-1} was prepared by dissolving the necessary amount of potassium dichromate ($K_2Cr_2O_7$) in distilled water. Analytical-grade reagents were used through out this study. Concentrations ranging between 10 and 150 mg l^{-1} were prepared from the stock solution to obtain the standard curve. All the adsorption experiments were carried out at room temperature (25 $\pm$ 2 °C).

2.3 Batch experiments

The effect of pH on the equilibrium adsorption was further investigated by employing different initial concentrations of Cr(VI) (75 and 100 mg l^{-1}) and different adsorbent dosages. The pH values were adjusted with diluted HCl and NaOH solutions. The flasks were shaken at room temperature (25 $\pm$ 2 °C) with an agitation speed of 120 rpm for the minimum contact time required to reach equilibrium. The concentration of Cr in solution was measured according to the standard method introduced by Gilcreas *et al.* [10] using a UV-vis spectrophotometer (Milton Roy, Spectronic 21D) using silica cells with a path length of 1 cm at a wavelength of 540 nm. All experiments were duplicated, and only the mean values are reported. The maximum deviation observed was less than 5%.

Adsorption of Cr(VI) was studied using different weights of activated carbon in 100 ml of solution of 75–150 mg l^{-1} of initial Cr(VI) concentration and initial pH 1.05.

2.4 *Equilibrium isotherms*

Adsorption equilibrium isotherms were studied using activated carbon dosages of 0.2, 0.3, 0.4, and 0.5 g per 100 ml of aqueous Cr(VI) solutions of initial concentrations ranging from 75 to 150 $\text{mg}\,\text{l}^{-1}$ and initial pH 1.05. For these experiments, the screw-cap conical flasks were agitated at a speed of 120 rpm and temperature of $25 \pm 2\,^{\circ}\text{C}$ for the required contact time. Then, the solution was filtered through a 0.45-μm membrane filter. The amount of Cr(VI) adsorbed ($\text{mg}\,\text{g}^{-1}$) at time t was computed using the following equation:

$$q_t = \frac{(C_0 - C_t)V}{m_\text{s}}, \tag{1}$$

where C_0 and C_t are the Cr(VI) concentrations ($\text{mg}\,\text{l}^{-1}$) initially and at a given time t, respectively. V is the volume of the Cr(VI) solutions (l), and m_s is the weight of activated carbon (g).

The percentage of removed Cr(VI) ions ($R_\text{em}\%$) in solution was calculated using equation (2):

$$R_\text{em}(\%) = \frac{(C_0 - C_t)}{C_0} \times 100. \tag{2}$$

2.5 *Theory*

The study of adsorption dynamics describes the solute-uptake rate, and evidently this rate controls the residence time of adsorbate uptake at the solid–solution interface. The kinetics of Cr(VI) adsorption on the activated carbons were analysed using pseudo-first order [11] and pseudo-second order [12]. The conformity between the experimental data and the model-predicted values was expressed by the correlation coefficients (R^2, values close or equal to 1). A relatively high R^2 value indicates that the model successfully describes the kinetics of Cr(VI) adsorption.

The pseudo-first-order equation [11] is generally expressed as follows:

$$\frac{dq_t}{dt} = k_1(q_\text{e} - q_t), \tag{3}$$

where q_e and q_t are the adsorption capacity at equilibrium and at time t, respectively ($\text{mg}\,\text{g}^{-1}$), and k_1 is the rate constant of pseudo-first-order adsorption ($\text{l}\,\text{min}^{-1}$). The linear form of equation (3) can be expressed as follows:

$$\log(q_\text{e} - q_t) = \log(q_\text{e}) - \frac{k_1}{2.303}t. \tag{4}$$

The values of $\log(q_\text{e} - q_t)$ were linearly correlated with t. The plot of $\log(q_\text{e} - q_t)$ vs. t should give a linear relationship from which k_1 and q_e can be determined from the slope and intercept of the plot, respectively.

The pseudo-second-order adsorption kinetic rate equation is expressed as follows [10]:

$$\frac{dq_t}{dt} = k_2(q_\text{e} - q_t)^2, \tag{5}$$

where, k_2 is the rate constant of pseudo-second-order adsorption ($\text{g}\,\text{mg}^{-1}\,\text{min}^{-1}$). The integrated form of equation (5) can be expressed as follows:

$$\frac{1}{(q_\text{e} - q_t)} = \frac{1}{q_\text{e}} + kt, \tag{6}$$

which is the integrated rate law for a pseudo-second-order reaction. The linear form of equation (6) can be obtained as follows:

$$\left(\frac{t}{q_t}\right) = \frac{1}{k_2 q_e^2} + \frac{1}{q_e}(t). \tag{7}$$

The plot of (t/q_t) vs. t from equation (7) gives a linear relationship in which q_e and k_2 can be determined from the slope and intercept of the plot, respectively.

The chromium sorption data are correlated with the theoretical models of Langmuir and Freundlich [13]. The Langmuir equation may be written as:

$$q_e = \frac{Q_0 b C_e}{1 + b C_e}, \tag{8}$$

which can be represented in the following linear form:

$$\frac{1}{q_e} = \frac{1}{Q_0} + \frac{1}{Q_0 b} \times \frac{1}{C_e}. \tag{9}$$

The Freundlich equation may be written as:

$$q_e = K_F C_e^{1/n}, \tag{10}$$

which can be represented in the following form:

$$\log q_e = \frac{1}{n} \log C_e + \log K_F, \tag{11}$$

where q_e is the amount of Cr ions adsorbed per unit mass of activated carbon (mg g^{-1}), C_e the equilibrium concentration of Cr in the bulk solution (mg l^{-1}), Q_0 the monolayer adsorption capacity (mg g^{-1}), and b the constant related to the free energy of adsorption. b is the reciprocal value of concentration at which half the saturation of the adsorbent is reached. The constants K_F and $1/n$ of the Freundlich model are the constants indicative of the relative adsorption capacity of the adsorbent (mg g^{-1}) and the constant indicative of the intensity of the adsorption, respectively.

3. Results and discussion

The performance of this new active carbon was evaluated by studying its maximum ability to remove Cr(VI) from aqueous solution. The sorption capacity could be related to the porous nature of carbon and its interaction with the metal ions.

3.1 *Effect of pH*

The effect of pH (1.05–9.0) on the removal of Cr(VI) was studied using different weights of carbon and different concentrations (100 and 75 mg l^{-1}) of Cr(VI). It was observed that the maximum percentage of Cr(VI) removal was at pH $= 1.05$ by using 4 and 5 g l^{-1} of activated carbon at room temperature ($25 \pm 2\,°$C). Increasing the pH from 1.05 to 9 led to a dramatic decrease in adsorption from 100% to 38% (figure 1). This behaviour can be explained, considering the nature of the adsorbent at different pHs in metal adsorption. The cell wall of activated carbon contains a large number of surface-functional groups. The pH

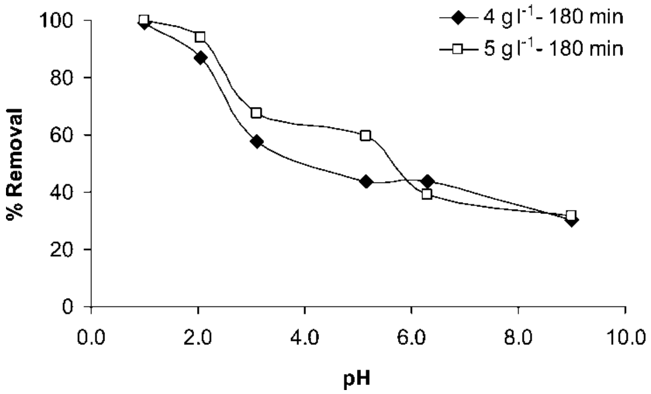

Figure 1. Effect of initial pH value on percentage removal of Cr using 4 and 5 g l^{-1} of activated carbon and 100 mg l^{-1} of Cr(VI).

dependence of metal adsorption can largely be related to the type and ionic state of these functional groups, as well as the metal chemistry in solution [1].

It is well known that the predominant form of Cr(VI) at pH 2 is $HCrO_4^-$. Increasing the pH will shift the concentration of $HCrO_4^-$ to other forms, CrO_4^- and $Cr_2O_7^{2-}$. The maximum adsorption was at pH 1.05, which indicates that Cr(VI) is in the $HCrO_4^-$ form. This is the predominant form between pH 1 and 4 that is adsorbed preferentially on the activated carbon, which is in agreement with Namasivayam and Yamuna [14]. Adsorption of Cr(VI) at a pH value below 3.0 suggests that its negatively charged forms bind through electrostatic attraction to positively charged sites on the surface of activated carbon because at this pH, more sites carrying positive charges would be exposed. At a pH above 3, it seems that activated carbon possesses more sites carrying a net negative charge, which tends to repulse the chromium anions. However, there is also a removal above pH 3.0, as shown in figure 1, but the rate of removal is considerably reduced. Hence, it could be said that above pH 3.0, other mechanism like physical adsorption on the surface of adsorbent could play an important role in the adsorption of chromium, and the exchange mechanism might have reduced.

3.2 *Effect of adsorbent dose and contact time*

The effect of adsorbent dosage on the percentage removal of Cr(VI) is shown in figure 2. It can be seen from the figure that initially the percentage removal increases sharply with increasing adsorbent dosage, but beyond 5 g l^{-1}, the percentage removal reached almost 100% removal. This trend is expected because as the adsorbent dose increases, the number adsorbent particles increases, and so more Cr(VI) is attached to their surfaces. A maximum removal of 97% was observed at an adsorbent dosage of 2 g l^{-1} at pH 1.05 for an initial Cr(VI) concentration of 75 mg l^{-1}. Therefore, the use of an adsorbent dose of 2 g l^{-1} is justified for economical reasons.

Plot of the percentage removal of Cr(VI) vs. contact time of 150 mg l^{-1} initial Cr(VI) concentration on 1, 2, 3, 4, and 5 g l^{-1} activated carbon at pH value of 1.05 was presented in figure 3. The results show that the equilibrium time required for the adsorption of Cr(VI) on activated carbon is almost 3.5 h. These results also indicate that the removal process can be considered to be very rapid, since the largest amount of Cr(VI) attached to the sorbent within the first 60 min of adsorption. It was found that the Cr(VI) removal increased with increasing contact time. More than 50% of Cr(VI) adsorption occurred in the first 10–15 min, and thereafter the rate of removal was found to be slow. In addition, the increase in carbon concentrations from

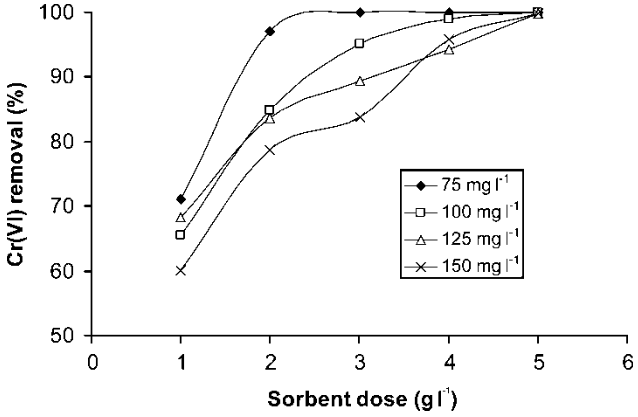

Figure 2. Effect of sorbent dose on Cr(VI) removal at solution pH 1.05.

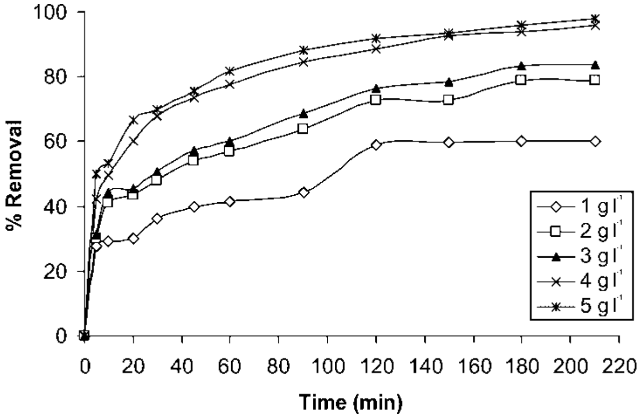

Figure 3. Effect of different concentrations of active carbon and contact time on the percentage removal of $150\,\mathrm{mg\,l^{-1}}$ of Cr(VI).

1 to $5\,\mathrm{g\,l^{-1}}$ gave an increase in Cr(VI) removal from 50 to 97%, due to the increased absorbent surface. The results show that for removal of $150\,\mathrm{mg\,l^{-1}}$ of Cr(VI) in 100 ml of solution, a minimum dosage of 0.4 g of activated carbon is required for 95% removal of Cr(VI).

3.3 *Adsorption-rate constant*

Numerous kinetic models have been proposed to elucidate the mechanism by which pollutants may be adsorbed. The mechanism of adsorption depends on the physical and/or chemical characteristics of the adsorbent as well as the mass-transport process. The mechanism of Cr(VI) adsorption was studied using the pseudo-first-order kinetic model given by Lagergen [11]. To calculate the rate constant, K_1, of adsorption at room temperature ($25 \pm 2\,^{\circ}\mathrm{C}$), Lagergren equation is plotted for three different sorbent concentrations (2, 3, and $4\,\mathrm{g\,l^{-1}}$) with a fixed Cr(VI) concentration (figure 4). The values of K_1 for three different carbon concentrations and calculated q_e value along with the correlation coefficients are given in table 1. The experimental q_e values slightly agree with the calculated values, obtained from the linear plots, which represent less applicability of the pseudo-first-order kinetics. However, the pseudo-second-order kinetic model is also expressed using equation (7) [12], when a plot of t/q_t vs. t showed

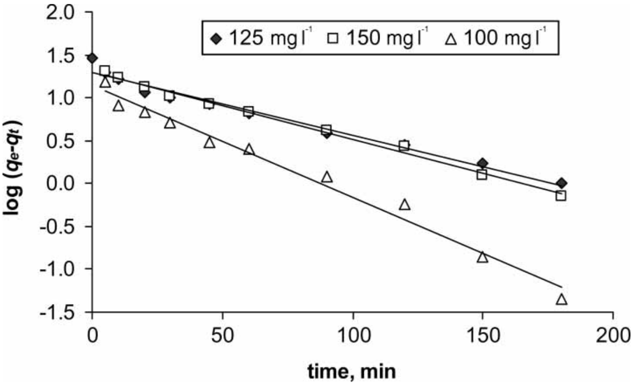

Figure 4. Cr(VI) uptake by active carbon according to the Lagergren pseudo-first-order model.

a linear relationship (figure 5). Values of q_e obtained from a pseudo-second-order model showed a good agreement with experimental q_e values (table 1). The correlation coefficients for the pseudo-second-order kinetic model are greater than 0.99. The results show that the adsorption of Cr(VI) on activated carbon follows the pseudo-second-order kinetics rather than the pseudo-first-order kinetics.

3.4 *Adsorption isotherm*

Figure 6 shows the experimental sorption isotherms for the Cr(VI)-activated carbon system using the Langmuir isotherm model. The Langmuir constants Q_0 and b related to the maximum adsorption capacity, i.e. the complete coverage of available adsorption sites (mg g^{-1}) and energy/intensity of adsorption, respectively, were obtained from the intercept and slope of the linear plot of $1/q_e$ vs. $1/C_e$ from equation (9) (table 2). The good fit of the experimental data and the high correlation coefficients (r^2) indicate the applicability of the Langmuir isotherm

Table 1. Comparison of the pseudo-first- and second-order adsorption rate constants for calculated and experimental q_e values at different initial Cr(VI) concentrations and active carbon concentrations.

	Experimental parameter		First-order kinetic model			Second-order kinetic model		
Carbon (g l^{-1})	Initial Cr(VI) conc. (mg l^{-1})	q_e (exp.) (mg g^{-1})	K_1 (min^{-1})	q_e (calc.) (mg g^{-1})	r^2	K_2 (g mg^{-1} min^{-1})	q_e (calc.) (mg g^{-1})	r^2
1	75	54.33	0.01	43.27	0.9769	0.0007	55.47	0.9905
2		36.95	0.02	25.96	0.975	0.0014	38.76	0.9959
4		18.9	0.1	16.17	0.9963	0.0184	19.16	0.9997
1	100	59.27	0.02	46.49	0.9729	0.0006	61.11	0.9977
2		41.88	0.02	46.48	0.9742	0.0009	42.17	0.9941
3		31.38	0.02	31.61	0.9953	0.0016	34.25	0.9961
4		23.98	0.02	17.07	0.9836	0.005	25.97	0.9997
1	125	66.58	0.01	41.01	0.8833	0.0005	67.11	0.9906
2		46.93	0.02	39.09	0.732	0.0009	43.55	0.9891
3		36.08	0.02	27.64	0.961	0.0011	37.46	0.9937
4		28.48	0.02	19.82	0.9753	0.0022	30.15	0.9978
5		24.77	0.03	15.75	0.9398	0.0031	25.91	0.9981
1	150	100.22	0.02	98.62	0.9813	0.0005	100	0.9906
2		62.7	0.02	51.94	0.9511	0.0009	64.94	0.9891
3		41.56	0.02	28.93	0.9778	0.0011	42.45	0.9937
4		32.74	0.02	20.32	0.9941	0.0022	33.59	0.9978
5		29.16	0.02	14.55	0.9932	0.0031	30.39	0.9981

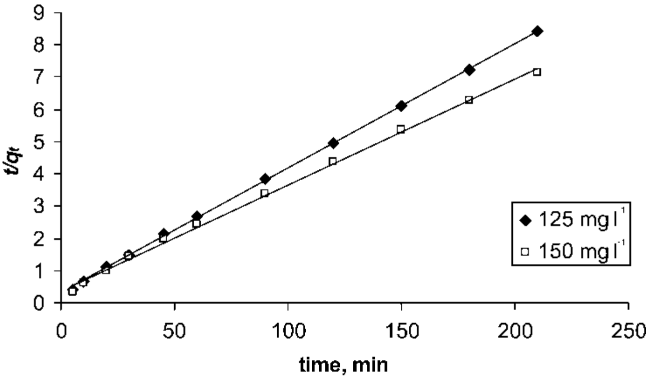

Figure 5. Cr(VI) uptake by active carbon according to the pseudo-second-order model.

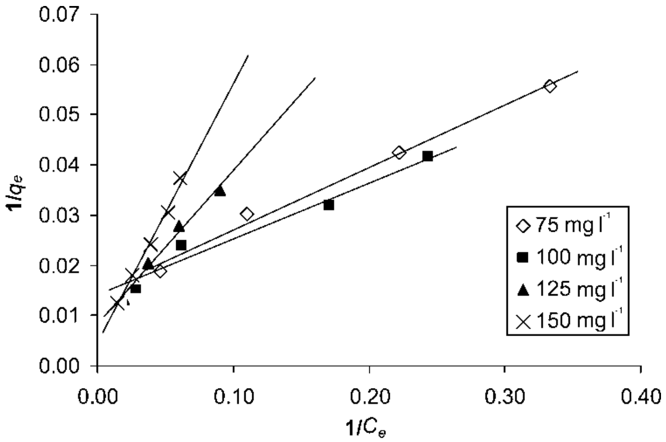

Figure 6. Linearized Langmuir adsorption isotherm for Cr(VI) with activated carbon.

model. The monolayer maximum adsorption capacity (Q_0) for Cr(VI) is 172.4 mg g^{-1}. This adsorption capacity for chromium is considerably higher than the values obtained with other adsorbents (50–120 mg g^{-1}) [15]. Since the value of n is more than 1, this indicates a favourable adsorption.

The essential characteristics of the Langmuir isotherm can be expressed in terms of a dimensionless constant called the separation factor or equilibrium parameter (R_L), which is

Table 2. Langmuir and Freundlich constants for the adsorption of Cr(VI) onto activated carbon.

	Langmuir				Freundlich		
Cr(VI) conc.	Q_0 (mg g^{-1})	b (l g^{-1})	R_L	r^2	K_F (mg g^{-1})	n	r^2
75	68.03	0.118	0.102	0.990	17.20	2.91	0.974
100	70.42	0.128	0.072	0.964	15.23	2.55	0.983
125	120.48	0.027	0.229	0.987	9.02	1.992	0.991
150	172.41	0.012	0.357	0.992	36.09	8.10	0.991

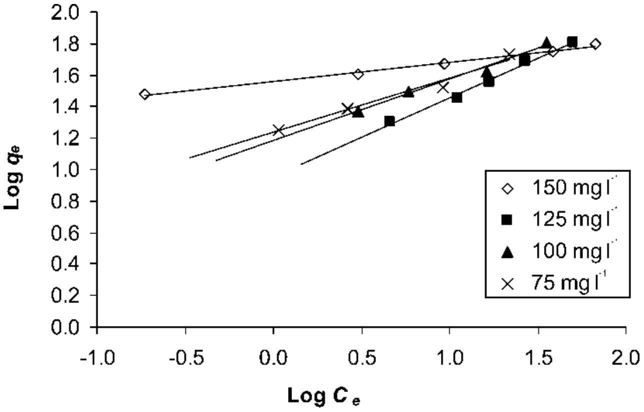

Figure 7. Linearized Freundlich adsorption isotherm for Cr(VI) with activated carbon.

defined by the following equation [16]:

$$R_L = \frac{1}{1 + bC_0}, \tag{12}$$

where b is the Langmuir constant, and C_0 is the initial concentration of the Cr(VI) ion. The value of R_L indicated that the type of Langmuir isotherm was irreversible ($R_L = 0$), favourable ($0 < R_L < 1$), linear ($R_L = 1$), or unfavourable ($R_L > 1$). Table 2 shows that Cr(VI) sorption on activated carbon developed from seed husk of *C. equisetifolia* is favourable under the conditions used in this study.

The chromium sorption isotherm followed the linearized Freundlich model, as shown in figure 7. The relation between the metal uptake capacity (q_e) of adsorbent and the residual metal ion concentration (C_e) at equilibrium is given by equation (11). The isotherm data fit the Freundlich model well ($r^2 > 0.974$). The values of the Freundlich constants K_F and n were calculated and are presented in table 2. Since the value of n is higher than 1, it indicates a favourable adsorption and signifies that the surface of the activated carbon developed from seed husk of *C. equisetifolia* is heterogeneous in nature and corresponds to the L-type of normal Langmuir isotherm [17, 18].

Thermodynamic equilibrium constant (K°) for various systems using carbon developed from seed husk of C. *equisetifolia* as adsorbent was obtained at room temperature (25 ± 2°C) as follows:

$$K^\circ = \frac{C_a}{C_e}, \tag{13}$$

where C_a is the concentration of Cr(VI) on the adsorbent at equilibrium in $\mathrm{mg\,l^{-1}}$, and C_e is the equilibrium concentration of Cr(VI) in solution in $\mathrm{mg\,l^{-1}}$. The Gibbs free energy (ΔG°) for the adsorption process for initial Cr(VI) concentration was obtained using the following formula [19]:

$$(\Delta G^\circ) = -RT \ln K^\circ, \tag{14}$$

where R is the ideal gas constant ($8.314\,\mathrm{J\,mol^{-1}\,K^{-1}}$), and T (K) is the absolute temperature.

The values of ΔG° and the thermodynamic constant K° for various systems are shown in table 3. The Gibbs free energy indicates the spontaneity of the adsorption process, where higher negative values reflect a more energetically favourable adsorption process. The negative ΔG° values obtained for various systems in this study confirm the feasibility of the process and spontaneous nature of sorption with a high preference for Cr(VI) to sorb onto peat.

Table 3. Thermodynamic parameters for the adsorption of Cr(VI) by activated carbon developed from *C. equisetifolia* seed husk.

pH	C_e	C_a	C_a/C_0	ln (K°)	ΔG° (kJ mol^{-1})
75 mg l^{-1} of Cr(VI) and 4 g l^{-1} of carbon					
1.0	10.28	64.72	6.30	1.84	−4.54
2.1	11.21	63.79	5.69	1.74	−4.29
3.1	19.31	55.69	2.88	1.06	−2.61
5.2	33.63	41.37	1.23	0.21	−0.51
6.3	40.79	34.21	0.84	−0.18	0.43
100 mg l^{-1} of Cr(VI) and 4 g l^{-1} of carbon					
1.0	10.46	89.54	8.56	2.15	−5.29
2.1	13.08	86.92	6.65	1.89	−4.67
3.1	42.35	57.65	1.36	0.31	−0.76
5.2	56.05	43.95	0.78	−0.24	0.60
6.3	65.39	34.61	0.53	−0.64	1.57

Higher negative values of ΔG° indicate a more favourable adsorption and higher capacity. The positive ΔG° values obtained for pH > 6.3 (table 3) indicate a decrease in adsorption process at pH > 6.3, which can be attributed to the surface nature of activated carbon and chemical form of chromium. The studies further confirm that as the pH of the system decreases, the adsorption of Cr increases. This indicates that the activated carbon developed from Casuarinas has a higher affinity towards Cr(VI) adsorption.

4. Conclusion

Casuarina *C. equisetifolia* is found to be a good raw material for developing activated carbon. This study demonstrated that H_2SO_4 is a suitable activating agent for the preparation of high-porosity carbons from seed husk of *C. equisetifolia*. The aqueous adsorption tests indicate that the *C. equisetifolia*-seed-husk-derived activated carbon has a notable adsorption capacity for Cr(VI). The kinetics of Cr(VI) adsorption followed the pseudo-second-order rate expression very well. The uptake of the Cr(VI) was greatly affected by the pH of the solution. The data thus obtained may be helpful for designing and establishing a continuous treatment plant for water and waste waters enriched in Cr(VI). The cost of removal is expected to be quite low, as the adsorbent is cheap and easily available in large quantities.

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