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Biosorption of Cr(VI) on Japanese Cedar Bark

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ABSTRACT

The ability of Japanese cedar (*Cryptomeria japonica*) bark to remove Cr(VI) from aqueous solutions was investigated. The research parameters included the solution pH, temperature, and initial concentration of Cr(VI) in solution. The removal of Cr(VI) was highly solution pH dependent and adsorbate concentration dependent, and mainly governed by physicochemical adsorption under the weak acidic conditions studied (initial solution pH ≥ 3). However, the reduction of Cr(VI) to Cr(III) occurred extensively at low solution pH (initial solution pH ≤ 2). The equilibrium data at different temperatures fit well in the Langmuir isotherm model. The endothermic nature of the adsorption was confirmed by the positive value of enthalpy change ($18.9\,\mathrm{kJ\,mol^{-1}}$). The positive value of entropy change ($65.2\,\mathrm{J\,mol^{-1}\,K^{-1}}$) suggested the increased randomness at the

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solid–solution interface during the adsorption. The studies showed that Japanese cedar bark can be used as a cost-effective adsorbent for the removal of Cr(VI) from wastewater.

Key Words: Removal of Cr(VI); *Cryptomeria japonica* bark; Adsorption; Langmuir isotherm; Adsorption thermodynamics; Wastewater treatment.

INTRODUCTION

Because chromate compounds are widely used in various industries such as electroplating, leather tanning, corrosion inhibition, dye and pigment preparation, and wood preservation, hexavalent chromium, Cr(VI), is often found in industrial waste streams. It is a strong oxidant and known to be both acutely and chronically toxic to human beings and animals.

Conventional methods for eliminating Cr(VI) from industrial wastewaters include chemical reduction and precipitation, ion exchange, and carbon adsorption. Recent attention has been focused on cost-effective adsorbents for removing heavy metal ions from contaminated waste effluents.[1] Kratochvil et al.[2] studied the removal of both Cr(III) and Cr(VI) by *Sargassum* seaweed biomass and concluded that the biosorption of these chromium species was governed by ion-exchange reaction. It also has been reported that certain wood wastes, such as tree leaves,[3–7] barks,[8–12] sawdust,[13,14] and pine cones,[15] effectively adsorb chromium species from aquatic systems. Among these, barks are especially promising because of their availability in large quantities from local sawmills.

Japanese cedar (*Cryptomeria japonica* D. Don), an economically important tree plantation species, is widely distributed in Japan. The wood is used for lumber and veneer, while the bark generally is of little economic value. Usually, the bark is either discarded in sawmills or burned. In this paper, the ability of *C. japonica* bark to remove Cr(VI) from aqueous solution was investigated. The parameters studied include initial solution pH, temperature, and Cr(VI) concentration.

MATERIALS AND METHODS

Preparation of the Adsorbent Materials

Fresh bark from an 80-year-old *C. japonica* was separated into inner and outer bark. These were air-dried and ground in a vibrating sample mill to pass through a 100 mesh (0.149 mm) screen. To remove alkaline metal salts and/or alkali earth metal salts originally present in the barks, the ground barks were soaked in 0.1 N HNO_3 overnight, washed thoroughly with distilled water, and air-dried in the dark.

Equilibrium Experiments

The ability of the bark to remove Cr(VI) from aqueous solutions was determined under batch-mode conditions. Cr(VI) was given as $K_2Cr_2O_7$. The pH of each solution was adjusted to the desired value with 0.1 M H_2SO_4 or 0.1 M NH_4OH. The test solutions (50 cm^3) were added to the adsorbent (0.1 g), and the suspensions were shaken for 72 hr. Preliminary runs showed that the adsorption equilibrium could be attained within 40 hr. The shaking speed used throughout the study was 100 strokes min^{-1}. The suspensions were then filtered off, and residual Cr ions in the filtrates were determined colorimetrically by using the standard procedures.[16] Cr(VI) was analyzed by the purple-violet colored complex formed by the reaction between Cr(VI) and 1,5-diphenylcarbazide in an acidified solution. The total Cr present in the solution was determined by oxidizing any Cr(III) formed with $KMnO_4$, followed by the same procedures described for the determination of Cr(VI). The adsorption density was calculated by the difference between the initial concentration of Cr(VI) and the final concentration of total Cr, while the difference between the final total Cr and Cr(VI) concentrations represented the Cr(III) concentration. All determinations were replicated five times, and the means and standard deviations of the analytical data were obtained.

Desorption Experiments

The bark (0.1 g) was shaken with 50 cm^3 of a 5 mM $K_2Cr_2O_7$ solution (pH 3) at 30°C for 72 hr. The suspension was filtered off, and the resulting solid residue was washed thoroughly with distilled water and air-dried in the dark. The Cr-laden adsorbent (0.1 g) was heated with a mixture of 30% H_2O_2 (10 cm^3) and concentrated H_2SO_4 (2 cm^3) to afford a pale blue solution. The amount of Cr(III) in the solution was determined by the standard method.[16] Cr content of the Cr-laden adsorbent was 46.6 ± 0.3 mg Cr g^{-1} (mean and standard deviation for five determinations).

The Cr-laden adsorbent (0.1 g) was soaked in 50 cm^3 of distilled water, 0.1 M HNO_3, 1.0 M HNO_3, or 0.1 M NaOH at 30°C for 72 hr. The amounts of

Cr(VI) and total Cr in the supernatant were determined by the standard method.[16]

Fourier Transform Infrared Analysis

The adsorbent, Cr-laden adsorbent containing 46.6 mg Cr g^{-1}, $K_2Cr_2O_7$, and a mixture of the adsorbent and $K_2Cr_2O_7$ were subjected to infrared (IR) spectroscopy by using a Fourier transform infrared (FT-IR) 8900 spectrophotometer (Japan Spectroscopic Co. Ltd., Tokyo). Pressed KBr pellets (sample and KBr ratio of 1 : 160 w/w) were scanned 100 times.

RESULTS AND DISCUSSION

Preliminary Results

Although the tissues of tree bark are complex, they are easily separated into two distinct parts, i.e., inner bark (yield: 40% by weight based on total bark) and outer bark (yield: 60% by weight based on total bark). Because of their morphological and chemical differences, the bark adsorbents were prepared, from both the inner and outer bark of *C. japonica*, separately. Their abilities to remove Cr(VI) from aqueous solution were examined by using an acidic $K_2Cr_2O_7$ solution (pH 3) containing 1.5 mM Cr at 30°C. As expected, there were distinct differences between the abilities of inner and outer bark to remove Cr(VI) from the test solution (Table 1). About 70% Cr(VI) could be removed by the outer bark, while more than 40% Cr species [42% as Cr(VI) and 43% as total Cr] were still remained in the solution, after contacting with the inner bark. Because the adsorption capacity of the outer bark was greater than that of the inner bark, an adsorbent prepared from the outer bark was used for succeeding experiments.

Table 1. Removal of Cr(VI) from a $K_2Cr_2O_7$ solution by *C. japonica* bark.[a]

			As Cr(VI)		As total Cr	
Adsorbent	pH_i[b]	pH_f[b]	mg g^{-1}	%	mg g^{-1}	%
Inner bark	3.0	6.0	23.0 ± 0.1	59.4 ± 0.1	22.2 ± 0.1	57.5 ± 0.2
Outer bark	3.0	6.9	28.4 ± 0.1	72.1 ± 0.1	28.3 ± 0.1	71.9 ± 0.2

[a] Each figure is the mean ± standard deviation for five determinations.
[b] pH_i, initial pH of dichromate solution and pH_f, equilibrium pH.

Effect of pH

Blank tests (without dichromate) showed that no significant changes were recognized in the initial solution pH, ranging 1–4. However, the solution pH was lowered from 5.0 to 4.3 after contact with the adsorbent at 30°C for 72 hr. The pH change observed may be due to only a small amount of acid in the adsorbent, because the adsorbent was pre-washed with dilute nitric acid.

Dependence of the removal of both Cr(VI) and total Cr on the initial pH of dichromate solution at initial concentrations of 1.5 mM Cr and at 30°C is shown in Fig. 1. The initial pH of the solution varied from 1 to 5, because Cr(III) forms hydroxides to yield insoluble precipitates above pH 5.5. As shown in Fig. 1, the amount of Cr(VI) removed decreased steadily at the pH range of dichromate solution from 2 to 5, while the Cr adsorption density peaked at pH 3. The originally added Cr(VI) could be completely removed by the outer bark, when the initial pH of the solution was 2 or below. However, 39% of Cr still remained in the supernatant as Cr(III) at the initial solution pH of 2.

The reduction of Cr(VI) to Cr(III) occurred extensively below pH 2 (Fig. 1 and Table 2). It is well known that chromic ions exhibit a typical cationic adsorption behavior.[2,17] Low uptake of Cr(III), formed by the chemical reduction at low pH, may be caused by an increased ability of protons to compete with Cr(III) for the binding sites of the adsorbent.[2] Further chromic ion species exist in acidic solution as bulky hydrated species, e.g., $[Cr(H_2O)_6]^{3+}$.[18] The hexaaqua ion is too large to enter parts of the microporous system of the adsorbent.[19] Jayson et al.[20] have reported that chromium adsorption onto

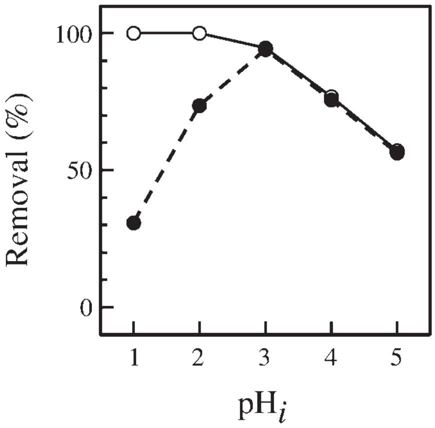

Figure 1. Effects of initial pH (pH$_i$) of dichromate solution on removal of Cr(VI) by *C. japonica* bark: ○, as Cr(VI) and ●, as total Cr.

Table 2. Effect of initial pH of dichromate solution on the uptake of Cr from the solutions.

Initial conditions			Equilibrium conditions (after 72 hr)		
C_i[a] [Cr(VI)] (mM)	C_i[a] [Cr(III)] (mM)	pH_i[b]	C_f[a] [Cr(VI)] (mM)	C_f[a] [Cr(III)] (mM)	pH_f[b]
1.51	0	1.0	0	1.12	1.1
1.51	0	2.0	0	0.59	2.4
1.52	0	3.0	0.42	0.00[c]	6.9
1.49	0	4.0	0.61	0.01	6.7
1.51	0	5.0	0.71	0.01	6.7
Blank	0	3.0	—	—	3.0

[a] C_i, initial content of Cr and C_f, equilibrium content of Cr.
[b] pH_i, initial pH of dichromate solution and pH_f, equilibrium pH.
[c] Cr(III) content in the equilibrium solution was less than 0.01 mM.

carbon cloth from aqueous chromate solutions was always greater by a factor of 10 than that from chromic solutions at pHs 3 and 11.

As shown in Table 2, only small amounts of Cr(III) were detected in the supernatants (0.2–0.4%) at higher initial solution pH ranging 3–5. In addition, the adsorption of Cr(VI) increased with rising equilibrium pH of the adsorption system. This implies that the uptake of Cr(VI) by the bark may be responsible for the depletion of protons in the system.[2] The Cr(VI) speciation diagram shows that Cr(VI) species exist predominantly in acidic solution (pH 2–5) as $HCrO_4^-$, indicating the anionic species to be taking part in the adsorption process.[21–23] The consumption of protons in the system is possibly explained by an exchange reaction with hydroxyl ions during adsorption, which can be shown in Eq. (1)[24,25]

$$C_xO + H_2O + HCrO_4^- = C_xOHO_3Cr^+ + 2OH^- \tag{1}$$

Hence, the removal of Cr(VI) by the bark may be mainly governed by physicochemical adsorption at a moderate pH (initial pH of dichromate solution ≥ 3). Similar results have been reported on the removal of Cr(VI) by tree leaves.[3,5–7]

Adsorption Isotherm

Table 3 shows the effect of dichromate concentration on the removal of Cr(VI) at different temperatures. The amounts of total Cr adsorbed onto the

Table 3. Effect of initial concentration of Cr(VI) on the removal of Cr(VI).

Initial conditions		Removal (mg g^{-1})								
		30°C			40°C			50°C		
C_i^a (mM)	pH_i^b	As Cr(VI)	As total Cr	pH_f^b	As Cr(VI)	As total Cr	pH_f^b	As Cr(VI)	As total Cr	pH_f^b
1.52	3.0	28.4 ± 0.1	28.3 ± 0.1	6.9	32.0 ± 0.1	31.7 ± 0.2	7.5	33.9 ± 0.1	32.2 ± 0.1	6.9
2.01	3.0	34.4 ± 0.3	34.2 ± 0.3	6.7	40.1 ± 0.2	39.9 ± 0.2	7.0	42.4 ± 0.2	41.9 ± 0.3	7.1
3.00	3.0	44.7 ± 0.3	44.6 ± 0.2	6.7	52.2 ± 0.3	52.0 ± 0.1	7.1	56.8 ± 0.4	56.2 ± 0.5	7.1
3.96	3.0	52.0 ± 0.3	51.5 ± 0.4	6.4	61.2 ± 0.4	60.6 ± 0.3	7.0	66.6 ± 0.5	66.2 ± 0.5	6.9
4.98	3.0	58.3 ± 0.5	57.9 ± 0.4	6.4	68.2 ± 0.4	67.6 ± 0.3	6.7	76.8 ± 0.6	76.2 ± 0.6	6.9

[a] C_i, initial content of Cr(VI).

[b] pH_i, initial pH of dichromate solution and pH_f, equilibrium pH.

bark increased with increasing the concentration of Cr(VI) in solution. The adsorption data followed the rearranged Langmuir model [Eq. (2)], where C_e is the equilibrium concentration (mg dm^{-3}) and q_e, the amount adsorbed at equilibrium (mg g^{-1}). The Langmuir constants, Q^0 (mg g^{-1}) and b (dm^3 mg^{-1}) represent the adsorption capacity and energy of adsorption, respectively. The values of Q^0 and b were obtained from the slopes and intercepts of the linear plots of C_e/q_e vs. C_e (Fig. 2).

$$\frac{C_e}{q_e} = \frac{1}{Q^0 b} + \frac{C_e}{Q^0} \tag{2}$$

As shown in Table 4, the applicability of the Langmuir isotherm for the present system suggests the formation of a monolayer of $HCrO_4^-$ covering the surface of the adsorbent.[23,26]

Effect of Temperature

The effect of solution temperature on the Cr(VI) adsorption also was studied. The Langmuir constant Q^0 increased from 71.9 to 90.9 mg g^{-1} as the solution temperature was increased from 30°C to 50°C (Table 4), indicating the process to be endothermic. The increase in adsorption with the rise in temperature may be explained by the thermodynamic parameters.

For adsorption of an adsorbate from solution to the solid phase, an equilibrium is established between the liquid and solid phases. The

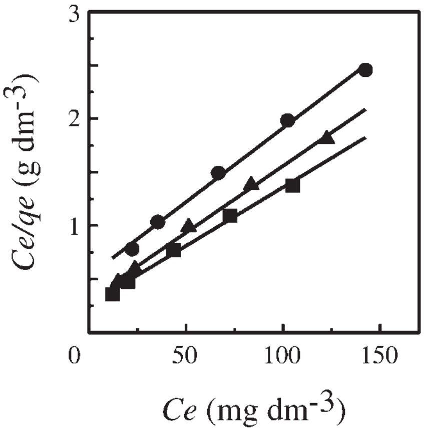

Figure 2. Langmuir plots for adsorption of Cr(VI) on *C. japonica* bark: ●, 30°C; ▲, 40°C; and ■, 50°C.

Table 4. Langmuir constants and thermodynamic parameters.

	Langmuir constant			Thermodynamic parameter	
Temperature (°C)	Q^0 (mg g^{-1})	b (dm^3 mg^{-1})	r^2	K_0	ΔG^0 (kJ mol^{-1})
30	71.94	0.026	0.9956	1.308	−0.68
40	80.00	0.040	0.9971	1.995	−1.80
50	90.91	0.042	0.9930	2.074	−1.96

thermodynamic parameters are calculated from the variation of the equilibrium constant K_0, and the values are given in Table 3. K_0 for the adsorption was obtained by plotting $\ln(q_e/C_e)$ vs. q_e and extrapolating to zero q_e (Fig. 3).[27] The standard free energy (ΔG^0) is calculated from Eq. (3), where T is the temperature in Kelvin, and R is the universal gas constant (8.314 J mol^{-1} K^{-1}). The enthalpy change (ΔH^0) and entropy change (ΔS^0) were obtained from the slope and intercept of van't Hoff plots of $\ln K_0$ vs. T^{-1} [Eq. (4)].

$$\Delta G^0 = -RT \ln K_0 \tag{3}$$

$$\ln\ K_0 = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{RT} \tag{4}$$

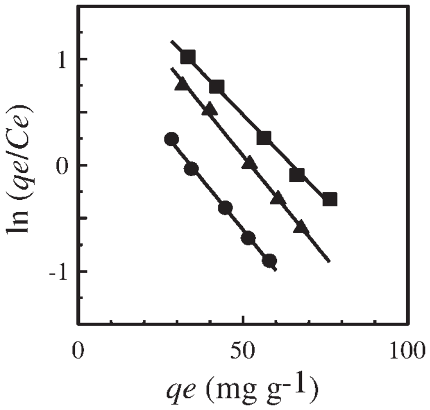

Figure 3. Plots of $\ln(q_e/C_e)$ vs. q_e for adsorption of Cr(VI) on *C. japonica* bark: ●, 30°C ($r^2 = 0.9990$); ▲, 40°C ($r^2 = 0.9986$); and ■, 50°C ($r^2 = 0.9981$).

The ΔG^0 of the process decreased with the rise in temperature, indicating the spontaneous nature of adsorption. The positive value of ΔH^0 (18.9 kJ mol^{-1}) suggests the process to be endothermic. The positive value of ΔS^0 (65.2 J mol^{-1} K^{-1}) shows the increased randomness at the solid–solution interface during the adsorption of $HCrO_4^-$ onto *C. japonica* bark.[28,29]

Infrared Studies

Figure 4 shows the IR spectra of the adsorbent, the Cr-laden adsorbent containing 46.6 mg Cr g^{-1}, $K_2Cr_2O_7$, and a mixture of the adsorbent and $K_2Cr_2O_7$ (Cr-adsorbent ratio, 1 : 44 w/w). The two characteristic bands in the spectrum of potassium dichromate near 950 and 760 cm^{-1} were absent in the

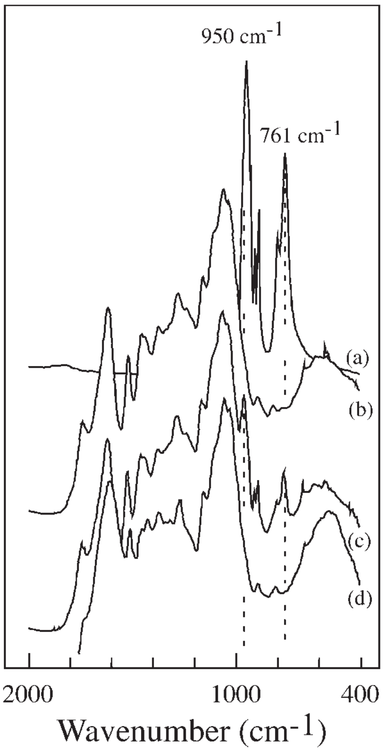

Figure 4. Infrared spectra of $K_2Cr_2O_7$ (a), adsorbent (b), a mixture of adsorbent and $K_2Cr_2O_7$ (c), and Cr-laden adsorbent (d).

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spectrum of the Cr-laden adsorbent, suggesting the occurrence of chemical reaction between dichromate ions and the surface active sites of the adsorbent. The reaction may involve the chemical reduction of Cr(VI) species captured by the adsorbent to Cr(III).

Desorption Studies

Recovery of the adsorbed metal and regeneration of the adsorbent are important aspects of wastewater treatment. Studies also have been carried out for the desorption of Cr(VI) using distilled water, HNO_3, and a dilute NaOH solution at 30°C for 72 hr. The total Cr eluted by distilled water was insignificant (recovery $< 0.3\%$). Approximately 19% and 9% total Cr was eluted by 0.1 M HNO_3 and 0.1 M NaOH, respectively, while 1 M HNO_3 could release 82% of adsorbed Cr as the trivalent state.

To evaluate the adsorption capacity to Cr(VI), the Langmuir constant Q^0 of the bark was compared with those of other low-cost adsorbents reported in the literature (Table 5). The magnitude of Q^0 shows that *C. japonica* bark is an effective adsorbent for the removal of Cr(VI) under suitable operation conditions. Recovery of Cr and regeneration of the exhausted bark by dilute mineral acid or dilute alkaline solution were incomplete. However, the bark is inexpensive compared with conventional

Table 5. Langmuir constants (Q^0) of various low-cost adsorbents.

Adsorbent	Langmuir constant ($mg\,g^{-1}$)	Optimum pH_i[a]	Adsorbate concentration ($mg\,dm^{-3}$)	References
Japanese cedar bark	71.9	3.0	79–259	Present study
Larch bark	31.3	3.0	10–300	[12]
London plane leaves	75.8	3.0	53–316	[7]
Saw dust	39.7	2.0	10–1000	[14]
Sugar beet pulp	17.2	2.0	10–500	[14]
Maize cob	13.8	1.5	10–300	[14]
Sugar cane bagasse	13.4	2.0	10–500	[14]
Sphagnum moss peat	65.8	2.0	130–440	[25]
Carbon slurry	24.1	2.5	52–130	[22]
Biogas residual slurry	5.9	2.0	10–40	[30]
Distillery sludge	5.7	3.0	10–40	[31]
Fly ash-Wollastonite	2.9	2.0	3–8	[23]

[a] pH_i, initial pH of dichromate solution.

adsorbents such as activated carbons, and, hence, the recovery of Cr can be carried out by burning the exhausted adsorbent followed by extraction of the metal with mineral acids.

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