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Removal of chromium (VI) from aqueous solution using treated oil palm fibre

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Abstract

This study proposed an oil palm by-product as a low-cost adsorbent for the removal of hexavalent chromium [Cr (VI)] from aqueous solution. Adsorption of Cr (VI) by sulphuric acid and heat-treated oil palm fibre was conducted using batch tests. The influence of pH, contact time, initial chromium concentration and adsorbent dosage on the removal of Cr (VI) from the solutions was investigated. The optimum initial pH for maximum uptake of Cr (VI) from aqueous solution was found to be 1.5. The removal efficiency was found to correlate with the initial Cr (VI) concentration, adsorbent dosage as well as the contact time between Cr (VI) and the adsorbent. The adsorption kinetics tested with pseudo first order and pseudo second order models yielded high R^2 values from 0.9254 to 0.9870 and from 0.9936 to 0.9998, respectively. The analysis of variance (ANOVA) showed significant difference between the R^2 values of the two models at 99% confidence level. The Freundlich isotherm ($R^2 = 0.8778$) described Cr (VI) adsorption slightly better than the Langmuir isotherm ($R^2 = 0.8715$). Difficulty in desorption of Cr (VI) suggests the suitability of treated oil palm fibre as a single-use adsorbent for Cr (VI) removal from aqueous solution. © 2007 Elsevier B.V. All rights reserved.

Keywords: Chromium (VI); Adsorption; Treated oil palm fibre; Kinetic; Isotherm

1. Introduction

Heavy metals are commonly found in various industrial wastewaters. They can cause serious water pollution and threaten the environment. It is therefore, essential to control and prevent their unsystematic discharge into the environment. For this reason, increased attention is being focussed on the development of technical know-how for their removal from metal bearing effluents before being discharged into water bodies and natural streams.

Earlier researchers have reported various methods for the removal of heavy metals from waters and wastewaters, with

0304-3894/\$ - see front matter © 2007 Elsevier B.V. All rights reserved. doi:10.1016/j.jhazmat.2007.07.033 the aim of preservation and protection of the environment from the damages due to their accumulation. Traditional technologies, such as ion exchange, chemical precipitation or reverse osmosis are often ineffective or expensive, particularly for the removal of heavy metal ions at low concentrations. Efficient and environmental friendly technologies are, therefore, needed to be developed to reduce the heavy metal contents in wastewaters to acceptable levels at inexpensive costs [1]. In recent years, the discharge of heavy metals into the environment has decreased in many countries due to more stringent legislations, improved cleaning technologies, and altered industrial activities. However, the demand for an economic and environmental friendly method for heavy metals removal still exists.

Large amounts of chromium-bearing wastewater are generated due to its widespread industrial applications; such as in steel and alloys making, electroplating, wood treatment, textile dyeing, paint production and leather tanning [2]. A

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small-sized electroplating industry (i.e. volume of wastewater <10,000 gal/day) can produce effluents with chromate concentration ranging from 10 to 500 mg/L [3]. Hexavalent chromium, Cr (VI), has been classified as a human carcinogen [4,5]. The WHO [6] allowable concentration for Cr (VI) in drinking water is 0.05 mg/L. The Malaysian limit for discharge of Cr (VI) into inland water is 0.05 mg/L [7]. Thus, it is necessary to reduce Cr (VI) concentrations from water/wastewater to acceptable levels.

Although chemical reduction has been shown to be effective in reducing chromium from waste streams, excess consumption of reducing agents, such as ferrous sulphate (FeSO₄), sodium bisulphite (Na₂S₂O₅) or sulphur dioxide (SO₂) due to the presence of dissolved oxygen in the waste streams can limit the application of this treatment process. Furthermore, the addition of sulphides and/or sulphites to the wastewater will require further treatment; while precipitation using lime will produce a large amount of sludge and increase the cost of sludge disposal [3]. The capital and/or operational costs of processes such as ion exchange, electrodialysis, reverse osmosis and evaporation are economically unfavourable for small and medium-sized industries normally established in this country.

Adsorption on activated carbon is a well-recognized method for the treatment of water and wastewater. The high material cost, however, limits its use in wastewater treatment. Therefore, in recent years, innovative adsorption using locally available low-cost adsorbents for Cr (VI) removal has been extensively investigated. Such adsorbents include fertilizer waste [8], soot [9], hazelnut shell [10,11], *Casurina equisetifolia* leaves [12], used tyres and sawdust [13], peat and coal [14], bagasse and fly ash [15], wool, olive cake, sawdust, pine needles, almond shells, cactus leaves and charcoal [16], maple sawdust [17], rosewood sawdust [18], coconut shell charcoal [19], and Moroccan stevensite [20].

Malaysia is the world's largest producer and exporter of crude palm oil (CPO); accounting for half of the world's output [21]. However, large quantities of wastes are generated in the production process. An average-sized CPO factory of 6 tonnes CPO production per hour (equivalent to 30 tonnes fresh fruit bunches (FFB) per hour) produces approximately 300 tonnes per day of solid wastes consisting of empty fruit bunches (EFB), fibre and shell [22]. Thus, in this study, this abundantly available oil palm by-product, i.e. fibre, was used as adsorbent in the removal of Cr (VI) from aqueous solution simulating chromium-containing wastewater.

2. Materials and methods

2.1. Materials

All primary chemicals used were of analytical reagent (AR) grade. The chemicals, potassium dichromate ($K_2Cr_2O_7$), sulphuric acid (H_2SO_4), sodium hydroxide (NaOH), nitric acid (HNO₃), hydrochloric acid (HCl), sodium carbonate (Na₂CO₃), sodium bicarbonate (NaHCO₃) and sodium chloride (NaCl) were used without further purification. Aqueous solutions were prepared with distilled water. A 1000 mg/L Cr (VI) stock solution was prepared using $K_2Cr_2O_7$. This solution was subse-

quently diluted to obtain the desired working solutions for batch experiments.

Palm fibre supplied by the Palm Oil Research Institute of Malaysia (PORIM) was activated with sulphuric acid and heat, for further use in the adsorption experiments, according to the simple method used by Garg et al. [18]. One part of air-dried adsorbent was mixed with one part of concentrated sulphuric acid in a ceramic crucible and heated in a muffle furnace for 24 h at 150 °C. The furnace was equipped with an extractor fan to catch acid vapours. The heated material was cooled and washed five to six times with distilled water followed by soaking in 1% sodium bicarbonate solution overnight to remove residual acid. The liquid portion was decanted and the solid was dried in an oven for 24 h at 105 °C. The resulting adsorbent was then used in the experiment without further grinding and sieving. The scanning electron micrograph (Fig. 1) of the untreated and treated oil palm fibre shows a change from fibre to grains with cavities. The Fourier transform infrared spectroscopy (FTIR) results show adsorption bands of hydroxyl, carbonyl and aliphatic groups.



(a) - Untreated oil palm fibre



(b) — Treated oil palm fibre



2.2. Procedures

Batch tests were conducted at room temperature $(28 \pm 1 \,^{\circ}\text{C})$ to obtain the adsorption rates and equilibrium data. Unless otherwise stated, 250 mL conical flasks, each filled with 100 mL of Cr (VI) solutions and 500 mg treated fibre were used in all experiments. The desired initial pH and Cr (VI) concentrations of the test solutions were adjusted according to the purpose of the test. The flask contents were agitated using an orbital shaker for the desired period at a speed of 350 rpm. Whatman No. 1 filter paper was used to filter the contents of the flask, followed by the analysis of Cr (VI) in the filtrate using a Hach DR 2010 Spectrophotometer.

To study the effect of pH, Cr (VI) samples of 20 mg/L concentration were adjusted to pH 1.5–5.0 as previous studies [17,23] have shown Cr (VI) removal to be favoured at low pH. The initial pH adjustment was made by the addition of 1N H₂SO₄ or NaOH. The flasks were agitated on the orbital shaker for 2 h. Solutions with 20–200 mg/L Cr (VI) were agitated for up to 6 h to determine the effect of initial concentration and contact time on Cr (VI) removal. Kinetic study of Cr (VI) adsorption on the adsorbent was also conducted based on these observations. The data were fitted to the following pseudo-first order and pseudo-second order kinetic equations [13]

$$\frac{\mathrm{d}q}{\mathrm{d}t} = k_1(q_\mathrm{e} - q) \tag{1}$$

$$\frac{\mathrm{d}q}{\mathrm{d}t} = k_2 (q_\mathrm{e} - q)^2 \tag{2}$$

where q_e is the amount of solute adsorbed at equilibrium per unit weight of adsorbent (mg/g), q the amount of solute adsorbed at time t per unit weight of adsorbent (mg/g) and k_1 and k_2 are constants. The following linearised time dependent functions can be obtained by integrating and rearranging Eqs. (1) and (2) for the boundary conditions t=0 to >0 and q=0 to >0 [13]

$$\ln(q_e - q) = \ln q_e - k_1 t \tag{1a}$$

$$\frac{t}{q} = \frac{1}{k_2 q_\mathrm{e}^2} + \frac{t}{q_\mathrm{e}} \tag{2a}$$

Cr (VI) solutions of concentration 100 mg/L were used to determine the effect of adsorbent dosage. The amount of treated fibre in the flasks was varied from 200 to 1000 mg and the flasks were agitated for 3 h. The data obtained was used for plotting the adsorption isotherms. The most commonly used isotherms in water and wastewater treatment are the empirical relationship of Freundlich (Eq. (3)) and the theoretically derived Langmuir relationship (Eq. (4)) [24]

$$q_{\rm e} = K_{\rm f} C_{\rm e}^{1/n} \tag{3}$$

$$q_{\rm e} = \frac{bQ_0C_{\rm e}}{1+bC_{\rm e}}\tag{4}$$

where q_e is the amount of solute adsorbed per unit weight of adsorbent (mg/g), C_e the equilibrium concentration of adsorbate in solution after adsorption (mg/L) and K_f , n, Q_0 and b are constants. The straight-line forms of the Freundlich and Langmuir

isotherms are as follows:

$$\ln q_{\rm e} = \ln K_{\rm f} + \frac{1}{n} \ln C_{\rm e} \tag{3a}$$

$$\frac{C_{\rm e}}{q_{\rm e}} = \frac{1}{Q_0 b} + \frac{1}{Q_0} C_{\rm e} \tag{4a}$$

For the desorption test, a set of seven conical flasks containing 1000 mg treated oil palm fibre was filled with 100 mL of 100 mg/L Cr (VI) solution and adjusted to the optimum pH for adsorption. The flasks were agitated using an orbital shaker maintained at 350 rpm for a contact time of 2 h. The liquid portions were then decanted and analysed for Cr (VI) concentration, whereas the solids were washed five to six times with distilled water to remove traces of Cr (VI) not adsorbed by the adsorbent. The adsorbent was then dried in an oven at 105 °C for 24 h and the actual mass of adsorbent left from each adsorption test was measured using an analytical balance. The adsorbents were then mixed with 100 mL of 1N H₂SO₄, 1N HNO₃, 1N HCl, 1N NaOH, 1N Na₂CO₃, 1N NaHCO₃ and 1N NaCl solutions and agitated as described before in the adsorption test. The concentrations of Cr (VI) released into the solutions were analysed and the desorption efficiency was calculated based on mass balance.

3. Results and discussion

3.1. Effect of pH

Cr (VI) was completely removed ($\approx 100\%$) from solution at initial pH of 1.5–2.0 (Fig. 2). Similar observations were made on the uptake of Cr (VI) from aqueous solutions by *Casurina equisetifolia* leaves (pH 2.5–3.0) [12], sawdust (pH 3) [18], natural and copper coated moss (pH 2) [25], immobilized fungal biomass (pH 2) [26], peat (pH 1.5–3.0) [27], etc. A sharp reduction in removal efficiency was noted when the initial pH of the solution was increased to higher values. Cr (VI) removal was only 5% at pH 5.

The optimum adsorption of Cr (VI) by treated oil palm fibre at low pH may be attributed to the presence of large amount of hydrogen ions (H^+) at such pH levels, which neutralize the negatively charged adsorbent surface and thus reduce hindrance



Fig. 2. Effect of pH on the removal of Cr (VI) by oil palm fibre: [Cr (VI)] = 20 mg/L; volume, 100 mL; contact time, 2 h; agitation speed, 350 rpm; adsorbent dosage, 5 g/L; and temperature, $28 \pm 1 \degree$ C.



Fig. 3. Effect of contact time and initial Cr (VI) concentration on the removal efficiency of Cr from aqueous solutions: pH, 1.5; volume, 100 mL; agitation speed, 350 rpm; adsorbent dosage, 5 g/L; and temperature, 28 ± 1 °C.

to the diffusion of Cr (VI) anions. At high pH values, the abundance of hydroxide ions (OH^-) increases hindrance to diffusion of Cr (VI) anions, thereby reducing the removal efficiency [15].

In aqueous solution, $Cr_2O_7^{2-}$ hydrolyzes as follows [28]:

$$Cr_2O_7^{2-} + H_2O \rightleftharpoons 2HCrO_4^-, \quad K_a = 0.0302 \text{ at } 25\,^{\circ}C \quad (7)$$

Although dichromate anion $(Cr_2O_7^{2-})$ of $K_2Cr_2O_7$ was the source of Cr (VI) in the synthetic wastewater used in this study, at pH < 6.0, the predominant form of Cr (VI) in aqueous solution is HCrO₄⁻ [19]. Therefore, the HCrO₄⁻ form is inferred as the predominant species of Cr (VI) preferentially adsorbed by treated oil palm fibre in the pH range of 1.5–2.0.

3.2. Effect of initial concentration and contact time

An adequate contact time between adsorbate and adsorbent is essential to allow the adsorbent to approach equilibrium with the adsorbate. Fig. 3 shows the removal efficiencies for 20–200 mg/L Cr (VI) with contact times up to 6 h. It is evident that an increase in the contact time resulted in an increase in the removal efficiency of Cr (VI) from the solutions.

Fig. 4 provides a better understanding of Cr (VI) adsorbed per unit weight of fibre (q). For the highest concentration tested, i.e. 200 mg/L, a rapid uptake of Cr (VI) occurred in the first 1.5 h of contact and was followed by a more gradual uptake rate until equilibrium was attained. A similar trend was observed for the 150 mg/L Cr (VI) solution. However, distinctly less time was required to achieve equilibrium in the treatment of lower concentrations of Cr (VI).

It can be observed from Fig. 3 that the equilibrium contact time for complete removal of 20, 50 and 100 mg/L Cr (VI) obtained from the study are 2, 2.5 and 3.5 h respectively. A contact time of 5 h was needed to remove over 99% of Cr (VI) from both 150 and 200 mg/L of Cr (VI) solutions. However, the actual uptake of Cr (VI) was higher for higher initial concentrations; the uptake being 4, 10, 20, 29.98 and 39.98 mg/g adsorbent for initial Cr (VI) concentrations of 20, 50, 100, 150 and 200 mg/L, respectively (Fig. 4).



Fig. 4. Effect of contact time and initial Cr (VI) concentration on the uptake of Cr per unit weight of adsorbent: pH, 1.5; volume, 100 mL; agitation speed, 350 rpm; adsorbent dosage, 5 g/L; and temperature, $28 \pm 1 \,^{\circ}$ C.

3.3. Kinetic study

Adsorption kinetic models correlate the solute uptake rate; thereby these models are important in water treatment process design [13]. In this study, two commonly used kinetic models, i.e. pseudo first order and pseudo second order, were employed to identify the nature of Cr (VI) adsorption on treated palm fibre.

Figs. 5 and 6 show the pseudo first order and pseudo second order kinetic plots for adsorption of Cr (VI) on treated oil palm fibre. The reaction rate constants of both models, calculated from Eqs. (1a) and (2a), are shown in Table 1. The high R^2 values obtained from the first and second order kinetic plots (i.e. from 0.9254 to 0.9998) suggest that adsorption of Cr (VI) on treated oil palm fibre can be described by both of these models. Nevertheless, the R^2 values as well as Figs. 5 and 6 also indicate that the sorption kinetics of Cr (VI) is better expressed by the pseudo second order reaction rate. The analysis of variance (ANOVA) performed on R^2 values obtained from the two models showed significant difference between the two at 99% confidence level. Compliance to the pseudo second order kinetic



Fig. 5. Pseudo first order kinetic plot for Cr (VI) adsorption at different initial concentrations: pH, 1.5; volume, 100 mL; agitation speed, 350 rpm; adsorbent dosage, 5 g/L; and temperature, 28 ± 1 °C.



Fig. 6. Pseudo second order kinetic plot for Cr (VI) adsorption at different initial concentrations: pH, 1.5; volume, 100 mL; agitation speed, 350 rpm; adsorbent dosage, 5 g/L; and temperature, 28 ± 1 °C.

Table 1

Pseudo first order and pseudo second first order model constants for Cr (VI) adsorption

Initial Cr (VI) concentration (mg/L)	Pseudo first-order model		Pseudo second-order model	
	$k_1 (h^{-1})$	R^2	$k_2 (g/mg h)$	R^2
20	1.974	0.9631	1.734	0.9998
50	2.502	0.9254	0.354	0.9991
100	1.440	0.9350	0.168	0.9991
150	0.648	0.9772	0.066	0.9955
200	0.648	0.9870	0.042	0.9936

model strongly suggests chemical adsorption or chemisorption between the adsorbent and adsorbate.

3.4. Effect of adsorbent dosage

The test to observe the effects of different mass of adsorbent on the adsorption of Cr(VI) from a fixed adsorbate concentration was carried out using 100 mg/L of Cr (VI) solutions. Fig. 7 shows the removal efficiencies and the uptake of Cr (VI) per







Fig. 8. Freundlich isotherm for Cr (VI) adsorption: pH, 1.5; [Cr (VI)] = 100 mg/L; volume, 100 mL; agitation speed, 350 rpm; and temperature, 28 ± 1 °C.

unit weight of adsorbent. While the amount of Cr (VI) adsorbed (mg/g adsorbent) by fibre decreased with an increasing amount of adsorbent (g/L), the removal efficiency increased from 52% to 97%.

Similar results were obtained by Lee et al. [25] for adsorption of Cr (VI) and Cr (III) on copper-coated moss, which suggested that the relationship between amounts of adsorbent and Cr adsorbed was close to a hyperbolic curve. Therefore, an optimum adsorbent dosage, which limits the amount of Cr adsorbed, is an important parameter to be considered in the batch treatment process.

3.5. Adsorption isotherms

Adsorption isotherm plays a crucial role in the predictive modelling procedures for the analysis and design of an adsorption system. Therefore, in this study, the adsorption data of Cr (VI) were tested with Freundlich and Langmuir isotherm models. The plots of linearized Freundlich and Langmuir equations (Eqs. (3a) and (4a)) for Cr (VI) are shown in Figs. 8 and 9, respectively. The Freundlich and Langmuir isotherm constants were obtained as $K_f = 7.1285$, n = 3.4542, $Q_0 = 22.73$ and b = 0.1807. The corresponding R^2 values were 0.8778 and 0.8715.



Fig. 9. Langmuir isotherm for Cr (VI) adsorption: pH, 1.5; [Cr (VI)] = 100 mg/L; volume, 100 mL; agitation speed, 350 rpm; and temperature, 28 ± 1 °C.

It can be seen that both the Freundlich and Langmuir models are comparable (similar R^2 values) in the description of Cr (VI) adsorption on treated palm fibre; with the Freundlich model being somewhat better (Figs. 8 and 9). The Freundlich model encompasses the heterogeneity of the adsorbent surface, the exponential distribution sites and their energies [29]. The 1/n value (0.2895) is favourably smaller than 1; indicating good adsorption for Cr (VI). On the other hand, the Langmuir model makes several assumptions, such as the adsorption energies are uniform and independent of surface coverage and complete coverage of the surface by a monolayer of adsorbate indicates maximum adsorption.

3.6. Desorption study

The possibility of adsorbent regeneration (desorption) and metal recovery was primarily studied based on the general assumption that regeneration of adsorbent promotes economic adsorption treatment. The results showed 9% and 0.4% desorption with 1N solutions of NaOH and Na₂CO₃, respectively. No measurable desorption was noted with the other chemicals tested. Desorption of Cr (VI) from treated palm fibre seems to be very difficult. In a previous study, Bai and Abraham [28] have also noticed that solutions of alkali and highly alkaline salts (NaOH, Na₂CO₃ and NaHCO₃) were more potent than acids or mineral salts for Cr (VI) desorption from immobilized fungal biomass. Continuous desorption of Cr (VI) from *Sphagnum* moss peat studied by Sharma and Forster [30] using 1 M NaOH is another example that favoured alkaline desorption.

From this study, it appears that higher concentration of NaOH may be required to increase the Cr (VI) desorption efficiency. The study with more concentrated NaOH solutions was not pursued further as the resulting treatment cost would question the application of the method under discussion as a low-cost technology for industries.

4. Conclusions

The Cr (VI) adsorption capacity of treated oil palm fibre was favoured at low pH; being attributed to the presence of excess positive charge on its surface. Complete removal of Cr (VI) was achieved at pH 1.5. The amount of Cr (VI) adsorbed increased with the increase in contact time and initial Cr (VI) concentration. High R^2 values of 0.9254–0.9870 and 0.9936–0.9998 were obtained with the pseudo first order and pseudo second order models respectively. ANOVA showed significant difference between the R^2 values of the two models at 99% confidence level. Adsorption of chromium could be adequately described by both Freundlich ($R^2 = 0.8775$) and Langmuir ($R^2 = 0.8715$) isotherms. The Freundlich model presented a slightly better fit. Desorption of Cr (VI) was difficult and suggests that treated oil palm fibre may be efficiently employed as a single-use adsorbent and may not be suitable for economic regeneration. Nevertheless, as the adsorbent is cheap and locally available in abundance, the requirement of adsorbent regeneration is not very crucial.

Oil palm fibre, widely available in Malaysia, can be utilized as a low-cost adsorbent for the removal of Cr (VI) from aqueous solutions. Simple chemical modifications of the fibre using sulphuric acid and heat treatment will be required to enhance the adsorption capacity, thereby preserving the economic aspects of the treatment.

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