

Relevant approach to assess the performance of sawdust as adsorbent of chromium (VI) ions from aqueous solutions

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Received 10 July 2009; revised 5 October 2009; accepted 3 November 2009; available online 1 December 2009

ABSTRACT: The biosorption of chromium (VI) ions from aqueous solutions by two adsorbents viz. mango and neem sawdust was studied under a batch mode. An initial pH of 2.0 was most favorable for chromium (VI) removal by both the adsorbents. The results obtained for the final concentration of chromium (VI) and chromium (III) at a pH range of 2-8 indicated that a combined effect of biosorption and reduction was involved in the chromium (VI) removal specially when the pH value is lower than 3. The maximum loading capacity was calculated from adsorption isotherms by applying the Langmuir model and found to be higher for neem sawdust (58.82 mg/g). Evaluation of experimental data in terms of biosorption kinetics showed that the biosorption of chromium (VI) by neem sawdust followed pseudo second-order kinetics. Therefore, the rate limiting step may be chemical sorption or chemisorption. The efficiency of this process was examined in using tannery wastewater contaminated with chromium (VI) ions in column mode.

Keywords: Biosorbent; Biosorption; Desorption; Isotherm models; Kinetics; Wastewater

INTRODUCTION

Environmental contamination by toxic metals is of great concern because of health risks on humans and animals. Among the toxic metal ions, chromium is one of the common contaminants which gains importance due to its high toxic nature even at very low concentrations (Shrestha *et al.*, 2007; Mahvi, 2008).

Chromium (VI) is a cancer-causing agent and can pose health risks such as liver damage, dermatitis and gastrointestinal ulcers (Dokken *et al.*, 1999). Wastewaters such as those generated during dyes and pigments production, film and photography, galvanometry, metal cleaning, plating and electroplating, leather and mining may contain undesirable amounts of chromium (VI) anions (Loukidou *et al.*, 2004, Venkateswaran *et al.*, 2007; Bhattacharya *et al.*, 2008).

The conventional methods used to remove Cr (VI) from aqueous effluents include chemical reduction, nanofiltration, bioaccumulation, ion-exchange, adsorption on silica composites and on activated carbon. However, these approaches have proved to be costlier and difficult to implement. Biosorption, a technically feasible and economical process, has gained increased credibility during recent years (Loukidou *et al.*, 2004).

Though a number of reports have appeared on the removal of chromium (VI) ions from aqueous solutions using inexpensive bio-waste materials (Dakiky *et al.*, 2002; Amir *et al.*, 2005; Ahalya *et al.*, 2005; Babel and Opiso, 2007; Nameni *et al.*, 2008; Wang *et al.*, 2008; Shah *et al.*, 2009; Zvinowanda *et al.*, 2009), new economical, easily available and highly effective adsorbents are still needed. Sawdust is a widely available waste by-product of the timber industry that is either used as cooking fuel or a packing material. Some reports using treated sawdust for removal of chromium (VI) ions are available (Garg *et al.*, 2004; Baral *et al.*, 2006), but information on raw sawdust is limited. The neem tree (*Azadirachta indica*) of family Meliaceae is native to the Indian sub-continent and its seeds and leaves have been used traditionally to treat a number of human ailments and also as a household pesticide. The tree itself is known as an air purifier and different parts of the tree such as leaves, bark, and seeds have been reported to possess a variety of medicinal and germicidal properties (Chopra *et al.*, 1956). The aim of the present investigation is to detect the performance of raw neem sawdust and mango sawdust on chromium (VI) removal from aqueous solutions by varying chromium (VI) concentration, pH and contact time. Adsorption

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isotherms and kinetic models were applied to fit the experimental data. The effectiveness of desorbing agent (NaOH) in stripping adsorbed metal ions from biosorbent was also investigated.

MATERIALS AND METHODS

All the chemicals used in this study were of analytical grade and were procured from sd-fine chem. Ltd. The adsorbents selected for the study viz. neem sawdust and mango sawdust were collected from the saw mill. These adsorbents were washed with deionized water and dried at room temperature ($28 \pm 2^\circ\text{C}$) till a constant weight of the adsorbent was achieved. The stock solution of chromium (VI) (1000 mg/L) was prepared by dissolving 2.829 g of potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$) in 1000 mL of deionised water. Experimental solutions of the desired concentrations were obtained by successive dilutions. The pH of the solution was adjusted with 0.1 N HNO_3 and NaOH solutions. Batch sorption experiments were conducted in 250 mL Erlenmeyer flasks containing 100 mL of chromium (VI) solution of known concentration and pH value. These metal solutions containing the biosorbent dosage of 2 g/L were agitated in a rotary shaker at 120 rpm for a desired time. The samples were withdrawn from the shaker at the pre-determined time intervals and adsorbent was separated by filtration. Chromium (VI) concentration in the filtrate was estimated using UV spectrophotometer at 540 nm wavelength by 1, 5- diphenyl carbazide method (APHA, 1998). The experiments were carried out by varying the chromium (VI) concentration in the solution (10-150 mg/L), pH (2.0-8.0) and contact time (5-360 min). The kinetic studies were performed by varying the biomass dosage from 1 to 3 g/L with initial metal concentration of 100 mg/L at room temperature $28 \pm 2^\circ\text{C}$ and solution pH (2.0). The samples were collected at different time intervals (5 min to 6 h) and the biosorbent was separated by filtration and supernatant analyzed for residual chromium (VI) concentrations. To study the reduction rate of chromium (VI) to chromium (III) by neem sawdust, the effect of pH (2- 8) was studied in batch mode with an adsorbent dosage of 2 g/ L at room temperature ($28 \pm 2^\circ\text{C}$) on a rotary shaker at 120 rpm. Total chromium was estimated by atomic absorption spectrophotometer (Spectra AA, Varian Model) using air-acetylene flame at 429 nm wavelength and slit width of 0.5 nm chromium (VI) concentration was estimated using UV spectrophotometer at 540 nm wavelength by 1, 5- diphenyl carbazide method (APHA 1998). chromium (III) concentration of the sample was calculated as difference

of total chromium and chromium (VI) for the same sample solution. The uptake of chromium (VI) by neem sawdust was studied in the presence of other anions (SO_4^{2-} , PO_4^{3-} , NO_3^- and Cl^-) and cations (Na^+ , K^+ , Mg^{2+} and Ca^{2+}).

The raw tannery wastewater sample collected from a common effluent treatment plant (CETP) located in Vaniyambadi, Vellore Dt., Tamil Nadu, India was analyzed promptly for determining the physicochemical characteristics using standard analytical methods (APHA, 1998). The characteristics of the wastewater were: color: black, pH: 6.1, TDS: 7794 mg/L, TSS: 4896 mg/L, sulphates: 878 mg/L, chlorides: 2927 mg/L and nitrate: 9.8 mg/L. The concentration of chromium (VI) in wastewater was detected using UV spectrophotometer: 94 mg/L and pH was adjusted to 2.0 using 0.1 N HNO_3 . The wastewater was then fed through a glass column (3 cm i.d. and 10cm long) packed with 20 g of the biosorbent neem sawdust at a flow rate of 5.0 mL/min controlled by a peristaltic pump. After treatment, the samples collected from the exit were analyzed for chromium (VI) concentrations. The wastewater was treated till the values reached to satisfy the Central Pollution Control Board (CPCB) standards (Cr (VI) - 0.1 mg/L) (CPCB, 1998).

RESULTS AND DISCUSSION

Effect of pH on sorption

Earlier studies on heavy metal biosorption have shown that solution pH is the single most important parameter affecting the biosorption process (Chen *et al.*, 2002). In order to establish the effect of pH on the biosorption of chromium (VI) ions, batch sorption studies at different pH values were conducted in the range of 2.0 to 8.0. Fig. 1 reveals that biosorption capacity of Cr (VI) ions is maximum at pH 2.0 and significantly decreases with increase in pH values upto 8.0. At pH 2 and 8 corresponding uptake yield values were found to be 80.1% and 7.6 % for neem sawdust, 60 % and 6.3 % for mango sawdust, respectively.

At lower pH, the biosorbent is positively charged due to protonation and dichromate ion exists as anion leading to an electrostatic attraction between them (Boddu *et al.*, 2003). Thus the uptake of chromium (VI) increased markedly with decreasing pH. A sharp decrease in adsorption above pH 4 may be due to occupation of the adsorption sites by anionic species like HCrO_4^- ; $\text{Cr}_2\text{O}_7^{2-}$; CrO_4^{2-} ; etc. which retards the approach of such ions further towards the sorbent surface (Das *et al.*, 2000; Donmez and Aksu, 2002). Popuri *et al.* (2007) has reported that the decrease in adsorption at high pH values may be due to the competitiveness



of the oxyanion of chromium and OH ions in the bulk. These results suggest that pH affects the solubility of metals and the ionization state of the functional groups like carboxylate, phosphate and amino groups of the cell walls of the biosorbent.

Adsorption isotherm

Adsorption isotherms are basic requirements for designing any adsorption system. Isotherm expresses the relation between the amounts of adsorbate (mg) removed from the liquid-phase by unit of mass of biosorbent (g) at fixed temperature (Freundlich, 1906; Langmuir, 1918; Redlich and Peterson, 1959; Sips, 1959). The Langmuir and Freundlich isotherm model were used to describe the biosorption equilibrium of biomass. The Freundlich isotherm is a nonlinear sorption model. This model proposes a monolayer sorption with a heterogeneous energetic distribution of active sites, accompanied by interactions between adsorbed molecules. The general form of this model is:

$$q_e = K_f C_e^{1/n} \tag{1}$$

Where, K_f (mg/g) stands for adsorption capacity and n for adsorption intensity.

The logarithmic form of Eq. 1 is:

$$\log q_e = \log K_f + 1/n \log C_e \tag{2}$$

The Langmuir model represents one of the first theoretical treatments of nonlinear sorption and suggests that uptake occurs on a homogeneous surface by monolayer sorption without interaction between adsorbed molecules. In addition, the model assumes

uniform energies of adsorption onto the surface and no transmigration of the adsorbate.

The Langmuir isotherm is represented in the following equation:

$$q_e = \frac{q_{max} b C_e}{1 + b C_e} \tag{3}$$

Where, q_{max} (mg/g) and b are Langmuir constants related to adsorption capacity and the energy of biosorption, respectively.

Experimental adsorption isotherms of chromium ions, obtained with different initial metal ion concentrations are presented in Figs. 2 and 3 which indicate the linear relationship between the amounts (mg) of chromium (VI) ions sorbed per unit mass (g) of the biosorbent against the concentration of chromium (VI) ions remaining in solution (mg/L). The values of regression coefficients obtained from these models were used as the fitting criteria to find out these isotherms. It was observed that the experimental data fits well to Langmuir adsorption isotherm indicating both monolayer biosorption and heterogenous surface conditions. The adsorption capacities and values of regression coefficients are shown in Table 1. Based on the q_{max} values, it can be noteworthy that neem sawdust has maximum potential for the removal of chromium (VI) ions from aqueous solution.

The experimental conditions i.e adsorption capacity, pH and initial metal ion concentration (C_0) values for the biosorption of chromium (VI) ions on different sawdust have been shown in Table 2. Compared to the reported ones, the biosorption capacity of neem sawdust for

Table 1: Linear regression data for Langmuir and Freundlich isotherms for chromium (VI) biosorption

Biosorbents	Langmuir parameters			Freundlich parameters		
	q_{max} (mg/g)	b (L/mg)	R^2	N	K_f	R^2
Neem sawdust	58.82	13.68	0.9809	2.1659	8.3926	0.739
Mango sawdust	37.73	7.84	0.9784	2.5967	7.1779	0.8782

Table 2: Comparison of adsorption capacities of different types of sawdust reported in literature

Adsorbents	Adsorption capacity mg/g	pH	C_0 mg/L	References
Coconut tree sawdust	3.60	3	20	Selvi <i>et al.</i> , 2001
Beech sawdust	16.10	1	200	Acar and Malkoc, 2004
Treated sawdust of Indian Rosewood	10.00	3	100	Garg <i>et al.</i> 2004
<i>Hevea brasiliensis</i> sawdust activated carbon	44.05	2	200	Karthekeyan <i>et al.</i> , 2005
Treated sawdust of Sal tree	9.55	3.5	40	Baral <i>et al.</i> , 2006
Neem sawdust	58.82	2	150	Present study
Mango sawdust	37.73	2	150	Present study



chromium (VI) is higher than the majority of the sawdust (treated or untreated). Since mango sawdust showed less potentiality compared to neem sawdust, further studies were carried out using neem sawdust as adsorbent.

Sorption kinetics

The kinetics of metal ion sorption is an important parameter for designing sorption systems. In addition to the uptake capacity, the rate of uptake of the adsorbate by the biosorbent is also critical as far as the reactor configuration is concerned. A rapid kinetics will facilitate smaller reactors (lower retention time for effective metal uptake) whereas a slow rate of uptake will necessitate long column or series of columns to utilize maximum potential of the biosorbent. The rate of sorption onto a sorbent surface depends upon a number of parameters such as structural properties of the sorbent, initial concentration of the solute and the interaction between the solute and the active sites of the sorbent (Oliveira *et al.*, 2005). The effect of external film diffusion on biosorption rate is assumed to be insignificant and ignored in many kinetic analyses, particularly when the biosorbent is employed as a free suspension in a well agitated batch system. Rate of adsorption is usually measured by determining the change in concentration of the adsorbate in contact with the adsorbent as a function of time. The kinetics of chromium (VI) sorption on the biosorbent neem sawdust was analyzed using two simple kinetic models viz. the pseudo-first order (Lagergren, 1898) and pseudo-second order (Ho and Mckay, 1999).

Pseudo first-order model

The adsorption kinetic data of chromium (VI) adsorption were analyzed using Lagergren rate equation (Lagergren, 1898) which considers that the rate of occupation of biosorption sites is proportional to the number of unoccupied sites (Cruz *et al.*, 2004).

$$\frac{dq_t}{dt} = k_1 (q_e - q_t) \quad (4)$$

Integrating Eq. 2 between limits, $q_t = 0$ at $t = 0$ and $q_t = q_t$ at $t = t$, Eq. 4 is obtained.

$$\log \frac{q_e}{q_e - q_t} = \frac{k}{2.303} t \quad (5)$$

Eq. 5 can be rearranged to obtain a linear form:

$$\log(q_e - q_t) = \log q_e - \frac{k}{2.303} t \quad (6)$$

Where, q_e and q_t are the amounts of adsorbed chromium (VI) ions on the biosorbent at equilibrium and at time t (mg/g), respectively and k_1 is the equilibrium rate constant of pseudo-first-order adsorption. The slope and intercept of the plot, $\log(q_e - q_t)$ versus t were used to obtain the first-order rate constant k_1 and equilibrium adsorption density q_e (Fig. 4). Lagergren first-order rate constant k_1 and q_e determined from the model indicated that this model had failed to estimate q_e since the experimental values of q_e differs from estimated ones (Table 3). This shows that the adsorption of chromium (VI) onto neem sawdust was not appropriate to describe the entire process and not a first-order reaction.

Pseudo second-order model

The sorption data were analyzed according to the pseudo second-order kinetic model (Ho and Mckay, 1998). This kinetic model is based on the assumption that the biosorption process follows a second order mechanism, with chemisorption as the rate limiting step. Therefore, the occupation rate of adsorption sites is proportional to the square of the number of unoccupied sites (Antunes *et al.*, 2003) and can be expressed as follows:

$$\frac{t}{q_t} = \frac{1}{k_2(q_e)^2} + \frac{t}{q_e} \quad (7)$$

Table 3: Adsorption first-order and second-order kinetic model rate constants of neem sawdust for biosorption of chromium (VI) for various biosorbent dosages

Biosorbent dosage (mg/L)	$q_{e,exp}$ (mg/g)	First-order kinetic model			Second-order kinetic model		
		K_1 (L/min)	$q_{e,cal}$ (mg/g)	R^2	K_2 (L/min)	$q_{e,cal}$ (mg/g)	R^2
1	74.7	0.023	32.56	0.9682	0.0884	75.1	0.9877
2	40.1	0.060	23.47	0.9709	0.1379	40.0	0.9805
3	28.4	0.076	22.70	0.9002	0.1074	29.9	0.9701

(Initial metal concentration- 100 mg/L, pH- 2, temp. 28 ±1°C, agitating rate-120 rpm)



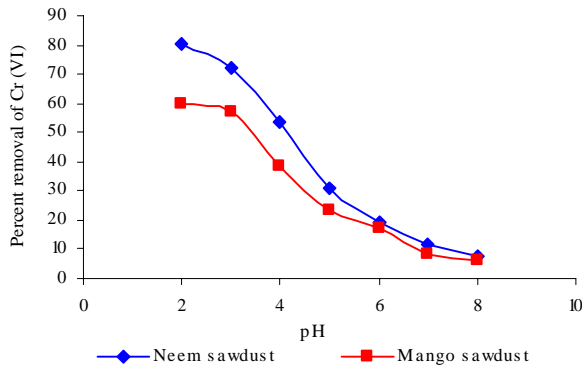


Fig. 1: Effect of pH on biosorption of Cr (VI) biosorbent dosage: 2 g/L, Initial metal concentration: 100 mg/L, contact time: 2 h

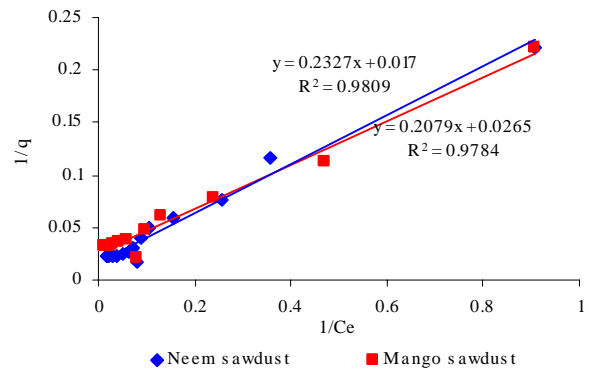


Fig. 2: Langmuir adsorption isotherms for Cr (VI)

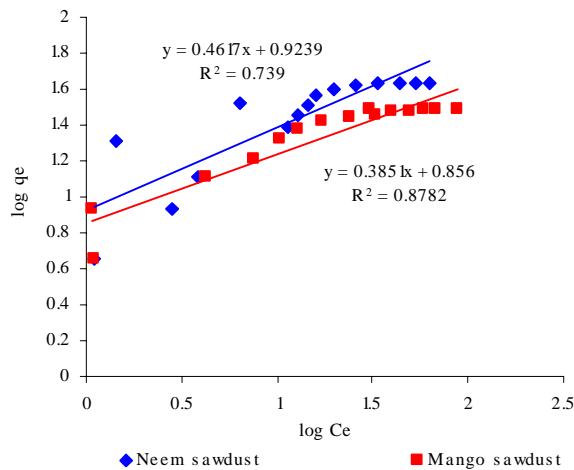


Fig. 3: Freundlich adsorption isotherms for Cr (VI)

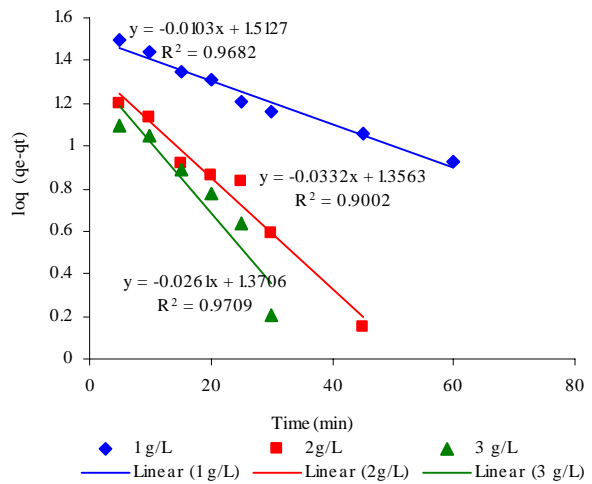


Fig. 4: Plot of the pseudo-first order kinetics of Cr (VI) for various biosorbent dosages

Where, k_2 is pseudo-second order rate constant. After integration and applying the same boundary conditions $t=0$ and $q_t=0$ to $t=t$ and $q_t=q_e$ at equilibrium, Eq. 7 takes the following form:

$$\frac{dq}{dt} = k_2(q_e - q_t)^2 \quad (8)$$

The second order rate constant k_2 and q_e values were determined from the slopes and intercepts of the plots and are presented in Fig. 5. The correlation coefficients (R^2) also shown in Table 3, are indicative of the strength of the linear relationship. The theoretical q_e values (i.e. $q_{e,cal}$) agree well with the experimental q_e values (i.e. $q_{e,exp}$), suggesting the sorption data tend to follow second order kinetics for chromium (VI) sorption onto neem sawdust. Therefore, the rate limiting step may be

chemical sorption or chemisorption. Similar results have been observed in the adsorption of chromium (VI) onto used tyres and sawdust (Hamadi *et al.*, 2001) and activated carbons derived from agricultural waste materials (Mohan *et al.*, 2005).

Reduction of Cr (VI) to Cr (III) by neem sawdust

Experiments were carried out to verify the mechanism of chromium (VI) biosorption on neem sawdust by varying the pH from 2-8. The final concentrations of the total chromium, chromium (VI) and chromium (III) in a pH range of 2-8 were analyzed. The percent removal of chromium (VI) and reduction to chromium (III) as a function of pH is shown in Table. 4 The results indicated that in acidic pH a combined effect of Cr (VI) biosorption and reduction to Cr (III) was found. The chromium (VI)



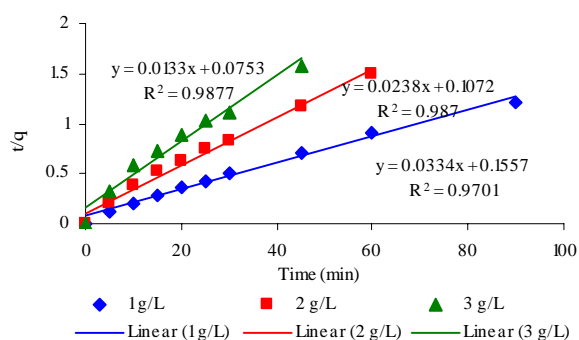


Fig. 5: Plot of the pseudo-Second order Kinetics of Cr (VI) for various biosorbent dosages

Table 4: Removal and reduction efficiency of chromium (VI) at different pH

pH	% Removal of chromium (VI)	% Reduction to chromium (III)
2	80.11	15.3
3	71.9	11.9
4	53.8	7.4
5	30.8	0.6
6	19.2	0
7	11.4	0
8	7.6	0

was reduced to chromium (III) when contacted with neem sawdust in acidic pH. As the pH increases the percent removal of chromium (VI) and reduction to chromium (III) reduced gradually, thus, the mechanism involved in biosorption of chromium (VI) may be adsorption coupled reduction. However, further studies need to be carried out to understand the actual mechanism involved in the adsorption of chromium (VI) onto neem sawdust.

Effect of co-ions

Actual industrial wastewaters contain different kinds of co-ions i.e cations (Na^+ , K^+ , Mg^{2+} and Ca^{2+}) and anions (SO_4^{2-} , PO_4^{3-} , NO_3^- and Cl^-) which may interfere with the uptake of chromium (VI) ions during biosorption. Hence, in the present investigation, the effect of different cations and anions on chromium (VI) biosorption by neem sawdust was studied. It was found that, under the conditions used, the influence of cations such as Na^+ , K^+ , Mg^{2+} and Ca^{2+} did not interfere much with adsorption of chromium (VI) whereas anions like SO_4^{2-} , PO_4^{3-} , NO_3^- and Cl^- influenced the removal of chromium (VI) significantly (Table 5). This shows that the above anions competed for the adsorption sites with chromium (VI)

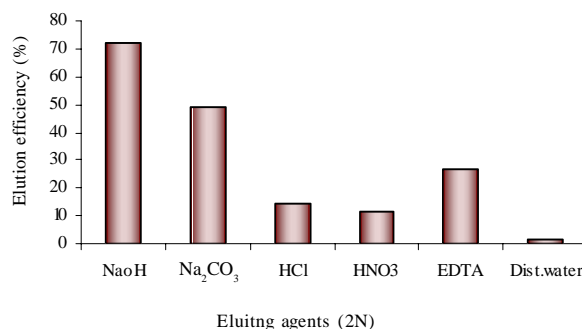


Fig. 6: Desorption of Cr (VI) by different desorbing agent

Table 5: Effect of co-ions on biosorption of chromium (VI) onto neem sawdust

Co-ions added	Concentration of co-ions (mg/L)	Percent uptake of Cr (VI)
Cr (VI) alone	-	81.70
Cations		
Cr (VI) + Na^+	1250	78.80
Cr (VI) + K^+	1250	79.65
Cr (VI) + Mg^{2+}	1250	79.50
Cr (VI) + Ca^{2+}	1250	78.70
Anions		
Cr (VI) + SO_4^{2-}	1250	65.10
Cr (VI) + PO_4^{3-}	1250	70.60
Cr (VI) + NO_3^-	1250	68.10
Cr (VI) + Cl^-	1250	62.90

species and hence, there were no specific or exclusive sites for chromium (VI) adsorption on the surface of the sorbent.

Desorption and reuse

Regeneration of biosorbent for repeated reuse is of crucial importance in industrial practice for metal removal from wastewater. Experiments were conducted for regenerating sawdust using various desorbing agents (HCl, HNO₃, NaOH, Na₂CO₃ and EDTA). The elution efficiency of these desorbing agents is shown in Fig. 6. The results obtained indicated that desorption of chromium (VI) ions with acids was marginal. Maximum desorption was achieved using NaOH as desorbing agent. Experiments conducted with different concentrations of NaOH (0.5N, 1N, 2N) showed that the percent desorption of chromium (VI) increased with



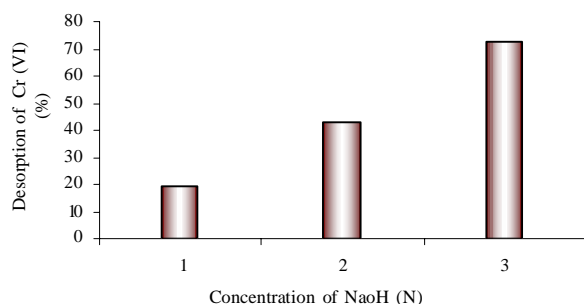


Fig. 7: Desorption of Cr (VI) using different concentrations of NaOH

increase in concentration of the desorbing agent (Fig. 7). In order to show the reusability of the biosorbent, experiment on cyclic sorption of chromium was conducted. For the cyclic loading of chromium (VI), 0.2g of neem sawdust was contacted with 100 mL of chromium (VI) solution having concentration of 100 mg/L for 1 h. The cycle was repeated for 4 times at pH-2 and temperature $28 \pm 2^\circ\text{C}$ followed by successive desorption after each biosorption cycle. At the end of every sorption cycle, metal solution was filtered and the metal concentration determined. The total amount of chromium (VI) sorbed on neem sawdust was 81.7 %, 76.4 %, 71.1 % and 65.6 % after 1st, 2nd, 3rd and 4th cycle, respectively. This observation indicates that neem sawdust was able to adsorb chromium (VI) ions even up to the 4th cycle, though the extent of metal sorption has gradually decreased. The results of the above experiments indicate the possibility of reusing the spent sorbent.

Column experiments using tannery wastewater

In order to demonstrate the practical application of the biosorption process, column experiment using neem sawdust was conducted with wastewater and the efficiency of the process was determined. The results showed that about 20 g of biosorbent was sufficient to treat a wastewater volume of 1.5 L containing chromium (VI) ions. The wastewater was treated till the chromium (VI) concentration reached the values established by (CPCB, 1998) (chromium (VI)-0.1 mg/L). Therefore, the present study confirmed that neem sawdust could serve as an economically viable potential biosorbent for the treatment of wastewater containing Cr (VI) ions.

CONCLUSION

This study identified neem sawdust as a suitable biosorbent for chromium (VI) removal in batch and column experiments. The adsorption process was pH

dependent and the optimum pH was 2.0 for biosorption and reduction of chromium (VI). The probable mechanism of chromium (VI) adsorption may be adsorption coupled reduction. Adsorption isotherm data at various initial metal concentrations were in good agreement with the Langmuir isotherm. Second-order kinetics was found to explain the kinetics of chromium (VI) adsorption most effectively. Present research also confirmed the potentiality of neem sawdust for the removal of chromium (VI) ions from real wastewater using column mode.

ACKNOWLEDGEMENTS

The authors would like to express their gratitude and appreciation to VIT University, Vellore for providing technical support to carry out the research work.

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How to cite this article: (Harvard style)

Vinodhini, V.; Das, N., (2010). Relevant approach to assess the performance of sawdust as adsorbent of chromium (VI) ions from aqueous solutions. *Int. J. Environ. Sci. Tech.*, 7 (1), 85-92.

