ARCHIVES OF ENVIRONMENTAL PROTECTION

vol. 34 no. 4 pp. 35 - 45 2008

PL ISSN 0324-8461

ROLE OF BIOSORBENT *FICUS RELIGIOSA* LEAF POWDER (FRLP) IN CHEMICAL SPECIATION OF CHROMIUM: A GREEN METHOD FOR SEPARATION

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Keywords: Biosorption, *ficus religiosa*, Cr, amino acid – Cr interactions, regeneration.

Abstract: The present study explores the unexploited sorption property of the unmodified *Ficus religiosa* leaf powder (FRLP) for decontamination and a possible method of separation of environmentally important two oxidation states of chromium (Cr(III) and Cr(VI)) from aqueous media. Sorption studies using standard practices were carried out in batch experiments as functions of biomass dosage, metal concentration, contact time, particle size and pH. Sorption studies result into the standardization of optimum conditions for the removal of Cr(III) 82.47% and Cr(VI) 88.23% as follows: biomass dosage (4.0 g), initial metal concentration in the aqueous system (Cr(III) 25 mg·dm^{-3}, Cr(VI) 50 mg·dm^{-3}), particle size (105 μm) at pH (Cr(III) – 6.5 and Cr(VI) – 2.5). The adsorption data were fitted in Freundlich and Langmuir isotherms. Studies of academic interest like kinetics studies revealed that adsorption equilibrium in each case followed first order equation. Morphological changes observed in the scanning electron micrograph of native and exhausted biomass indicate the existence of biosorption phenomenon. Fourier transform infrared spectrometry of exhausted leaf biomass highlights amino acid – Cr interactions responsible for sorption phenomenon. Regeneration of exhausted biomass was attempted for several cycles for its effective reusability.

INTRODUCTION

The vitality of water is mandatory to all forms of life and fundamental for environmental health and management. According to National Water Commission, water is treated as an infinite free good yet aqueous systems are increasingly at risk from pollution, particularly of toxic metals. Extensive use of chromium in electroplating, tanning, textile, dyeing and biocide in cooling water of power plants, discharge undesirable amount of chromium containing effluents [5]. The removal of chromium from contaminated wastewater is necessary because high level of chromium in waste water is a major concern around the globe. The presence of different oxidation states of chromium in the ecosystem is the cause of many well-documented toxic effects. The maximum levels of chromium permitted in wastewater are 5 mg·dm^{-3} for Cr(III) and 0.05 mg·dm^{-3} for Cr(VI) [9]. Prolonged exposure to hexavalent chromium poses a greater health risk due to its carcinogenic activity [1]. Recently, trivalent chromium has also been associated with toxicity at higher concentration. Alkaline precipitation, ion exchange, ultra filtration and reverse osmosis are the currently available technologies for chromium removal [8]. Nevertheless, there are many constraints in the application of these technologies. The major drawbacks from these technologies include incomplete removal, high reagent, energy requirement, generation of toxic sludge and high operational cost [10]. These techniques are uneconomical and often not feasible. The search is still on for efficient yet cost effective treatment options. The increasing demand for more effective remedial and separation technologies results in a huge window of opportunity for biosorption whose competitive advantage warrants its future success.

Bioremediation is a tangible alternative to traditional methodologies that has fascinated increased scientific attention in recent years [23, 25]. Biosorption promises to fulfill the requirements, which are competitive, effective and economically viable [2, 24]. Sorption of metals depend not only on the total concentration in solution but also on its different physico-chemical forms (chemical speciation) and various interactions which metals undergo in the aquatic system resulting into the modification of metals in different forms and finally affecting the rate of biosorption. Efforts have been made to use different forms of inexpensive, non-living plant materials such as hazelnut shell [15] and *Agave lechuguilla* [11] as biosorbent for chromium removal from aqueous phase. In continuation of our work [12, 17, 19, 21] on the biosorption of toxic metals using agricultural waste from the aqueous solution, the present communication highlights the potential of *Ficus religiosa* leaf powder for removal efficiency of Cr(III) and Cr(VI) from the water bodies.

Ficus religiosa Linn. (Pipal) is a large evergreen tree with spreading branches abundantly available through the Central India. Various parts of the plant possess pharmacological properties. Pipal is well renowned for its diverse applications such as it cures blood diseases and burning sensation. Ripe fruit acts as cardiac tonic and is cold in potency. It cures bleeding diathesis, poisoning, pain, burning sensation, vomiting, edema and anorexia [18].

EXPERIMENTAL

Biosorbent preparation

Leaves of *Ficus religiosa* Linn. were collected in June 2007. They were washed repeatedly with water to remove dust and soluble impurities, dried at 65°C for 24 hours, crushed and finally sieved through (105 μm, 210 μm and 420 μm) mesh copper sieves. No other chemical or physical treatments were used prior to the adsorption experiments.

Sorption studies

Sorption studies consisted of a series of batch experiments as a function of biomass dosage (2.0, 4.0 and 6.0 g), contact time (10, 20, 30, and 40 minutes), metal concentration (1, 5, 10, 25, 50 and 100 $mg \cdot dm^{-3}$), particle size (420 μm, 210 μm and 105 μm) and pH (2.5, 3.5, 4.5, 5.5, 6.5, 7.5 and 8.5). The solutions containing Cr(III) as chromium chloride and Cr(VI) as potassium dichromate were taken into separate Erlenmeyer flasks. After pH adjustment, required quantity of biosorbent was added and finally metal bearing suspension was allowed to settle. The residual biomass sorbed with metal ion was filtered using Whatman 42 filter paper. Filtrate was collected and subjected for metal ion estimation using Atomic Absorption Spectroscopy (Perkin Elmer-3100). Percent metal sorption by the sorbent was computed using the equation:

$$\% \text{ Sorption} = (C_o - C_e)/C_o \cdot 100$$

where:

C_o and C_e – the initial and final concentration of metal ions in the solution.

Fourier transform infrared spectroscopy (FTIR)

For the analysis of the functional groups that might be involved in metal sorption, FTIR analysis in solid phase was performed using a Shimadzu 8400 FTIR. Spectra of the sorbent before and after chromium (Cr(III) and Cr(VI)) sorption were recorded.

BET surface area analysis

The surface area of the biosorbent was measured using a Micromeritics ASAP-2010 BET surface area analyzer.

Scanning electron microscopy (SEM)

The comparison of the surface morphology of untreated (native) and chromium treated FRLP (exhausted) was observed with a scanning electron micrograph at bar length equivalent to 200 μm, working voltage 20 KV with 200x magnification.

Adsorption capacity

Soluble metal fractions of Cr(III) and Cr(VI) ions after FRLP biosorption under different experimental conditions were carried out to determine sorption capacity. Sorption isotherms of chromium were obtained in the concentration range (1.0–100 $mg \cdot dm^{-3}$) on the optimum biosorbent (4.0 g) at pH 6.5 and 2.5 for Cr(III) and Cr(VI) respectively and plotted between metal adsorbed [mg] per unit mass [g] of sorbent against concentration. The concentration of metals in the sorbent phase was calculated as:

$$q = (C_0 - C) \cdot V/W$$

where:

C_0 and C – denote the initial and equilibrium concentration of the metals in the aqueous phase,

V – volume of the aqueous phase,

W – dry weight of the sorbent.

The adsorption capacity and affinity of FRLP for Cr(III) and Cr(VI) was determined with two well-known isotherm models (Freundlich and Langmuir). Freundlich sorption isotherm is given in the form of linearized equation as:

$$\log q = \log K_f + 1/n \log C_e$$

where:

q – metal uptake per unit weight of biosorbent,

C_e – equilibrium (residual) concentration of metal ion in solution,

K_f and n – characteristic constants.

Langmuir equation has the general form as:

$$C_e/q_e = (1/Q_o b) + (C_e/Q_b)$$

where:

C_e – equilibrium concentration,

q_e – amount adsorbed of Cr(III) and Cr(VI) at equilibrium,
Q_o and b – Langmuir constants related to adsorption capacity and energy of adsorption respectively.

The biosorption capacity (K_f and Q_o) and biosorption intensity/energy (1/n and b) were estimated from the slope and intercept of the Freundlich and Langmuir isotherms. Lagergren plots were also obtained by conducting kinetic studies (batch) at initial Cr(III) and Cr(VI) ions concentration. FRLP (4.0 g) was suspended in 200 cm^3 of Cr(III) and Cr(VI) solution of known initial sorbate concentration at pH 2.5 for Cr(VI) and 6.5 for Cr(III) where maximum sorption was recorded. The mixture was continuously stirred using a magnetic stirrer. Samples were withdrawn at pre-determined time intervals in the range of 10–40 minutes, filtered and analyzed for residual Cr(III) and Cr(VI) ions concentration.

Desorption of chromium from loaded biomass

Desorption studies (batch process) were conducted to restore the biomass as a function of concentration (0.01–0.1 M) of hydrochloric and nitric acids. Chromium loaded biosorbent (Cr(III) and Cr(VI)) obtained from our sorption experiments were transferred to Erlenmeyer flask and shaken with 50 cm^3 of each acid for 40 minutes. The filtrate was analyzed for desorbed chromium.

Statistical analysis

Batch experiments were conducted in triplicate (N = 3) and data represent the mean value. Mean values, correlation coefficients, and standard deviations were calculated using SPSS PC^{+}_{TM} statistical package [20]. For the determination of intergroup mean values differences, each parameter was subjected to the student-t test for significance level ($p < 0.05$).

RESULTS AND DISCUSSION

Sorption studies result into the standardization of the optimum conditions as biomass dosage (4.0 g), metal concentration (25 $mg \cdot dm^{-3}$ for Cr(III), 50 $mg \cdot dm^{-3}$ for Cr(VI)), and contact time (30 min) at pH 6.5 for Cr(III) and 2.5 for Cr(VI) (Tab. 1). Maximum sorption for Cr(III) and Cr(VI) species is 82.47 and 88.23% respectively.

The findings indicate that FRLP has considerable potential to be used as biosorbent for chromium removal from the aqueous system and require the understanding of the mechanistic aspects of interactions of leaf biomass with metal ions. Basing on our experimental findings, we synthesize a possible mechanism for chromium binding to the target biomass. The aqueous solution of Pipal leaves is a heterogeneous complex mixture possessing prominently low molecular weight amino acids [3, 14]. The proteinaceous amino acids have variety of structurally related pH – dependent properties of generating appropriate atmosphere (positively and/or negatively charged sites) for attracting the cationic or anionic species of metal ions [6].

Mechanistic aspects of sorption

Solution chemistry of Cr(III) and Cr(VI) shows their existence in cationic (Cr^{3+}, $Cr(OH)^{2+}$) and anionic ($Cr_2O_7^{2-}$, $HCr_2O_7^{-}$, $HCrO_4^{-}$ and CrO_4^{2-}) forms in the pH range 4.0 to 6.0 [16]

Table 1. Soluble Cr (Cr(III) and Cr(VI)) ion concentration in test volume (200 cm^3) at optimum time (40 min) after sorption on unmodified FRLP as functions of metal concentration and biomass dosage at pH 6.5 for Cr(III) and pH 2.5 for Cr(VI) respectively

Initial concentration [mg·dm^{-3}] [μM]	Soluble Cr(III) concentration in FRLP		
	Biomass 2.0 g	Biomass 4.0 g	Biomass 6.0 g
01 (03.84)	$03.12 \pm 0.16^{+\Phi}$	$02.17 \pm 0.10^{+\Phi}$	$02.17 \pm 0.10^{+\Phi\Phi}$
05 (19.23)	$13.13 \pm 0.67^{+\Phi}$	$06.73 \pm 0.36^{+\Phi}$	$06.71 \pm 0.35^{+\Phi\Phi}$
10 (38.46)	$16.12 \pm 0.86^{+\Phi}$	$08.42 \pm 0.47^{+\Phi}$	$08.38 \pm 0.46^{+\Phi\Phi}$
25 (96.15)	$36.54 \pm 1.97^{+\Phi}$	$18.48 \pm 1.00^{+\Phi}$	$18.42 \pm 1.01^{+\Phi\Phi}$
50 (192.30)	$72.92 \pm 3.79^{++\Phi}$	$36.56 \pm 2.04^{++\Phi}$	$36.45 \pm 1.99^{++\Phi\Phi}$
100 (384.61)	$145.91 \pm 7.58^{++\Phi}$	$72.82 \pm 3.93^{++\Phi}$	$72.71 \pm 4.12^{++\Phi\Phi}$
Correlation coefficient (r)	r = 0.97	r = 0.99	r = 0.99
Initial concentration [mg·dm^{-3}] [μM]	Soluble Cr(VI) concentration in FRLP		
	Biomass 2.0 g	Biomass 4.0 g	Biomass 6.0 g
01 (3.84)	$03.38 \pm 0.17^{+\Phi}$	$01.91 \pm 0.10^{+\Phi}$	$01.91 \pm 0.10^{+\Phi\Phi}$
05 (19.23)	$13.26 \pm 0.75^{+\Phi}$	$05.92 \pm 0.33^{+\Phi}$	$05.89 \pm 0.33^{+\Phi\Phi}$
10 (38.46)	$15.79 \pm 0.89^{+\Phi}$	$07.59 \pm 0.43^{+\Phi}$	$07.52 \pm 0.40^{+\Phi\Phi}$
25 (96.15)	$37.47 \pm 2.16^{+\Phi}$	$17.19 \pm 0.98^{+\Phi}$	$17.13 \pm 0.88^{+\Phi\Phi}$
50 (192.30)	$66.91 \pm 3.87^{+\Phi}$	$22.73 \pm 1.31^{+\Phi}$	$22.68 \pm 1.28^{+\Phi\Phi}$
100 (384.61)	$133.45 \pm 7.74^{++\Phi}$	$45.35 \pm 2.57^{++\Phi}$	$44.89 \pm 2.42^{++\Phi\Phi}$
Correlation coefficient (r)	r = 0.97	r = 0.99	r = 0.99

Numbers in parenthesis represent soluble metal concentrations in [μM],
Standard deviations ±.
Mean value difference (initial Cr(III) and Cr(VI) loaded versus soluble Cr(VI) [μM]) as functions of metal concentration: significant ($p < 0.05$), insignificant ($p > 0.05$).
Biomass dosage: $^{\Phi}$ – significant ($p < 0.05$), $^{\Phi\Phi}$ – insignificant ($p > 0.05$).

and 1.0 to 4.0 [4] respectively. The majority of amino acids present in target leaf powder have isoelectric points in the pH range 4.0 to 8.0 [7]. In this range of pH, over 90% of the amino acid molecules are in ionized state, i.e. they have both positively charged amino groups as well as negatively charged carboxylate ion. As shown in Figure 1, sorption tendency of Cr(III) is very less at lower pH values. This is because at lower pH, binding sites (amino acid moieties) in the biomass are generally protonated, thus repulsion occurs between the metal cations (Cr(III)) and the positively charged sites of the leaf biomass.

Our experimental findings showed that sorption of Cr(III) increases with rise in pH attaining a plateau around 6.5 to 7.5. At pH above 4.5, the carboxylic groups are deprotonated and as such are negatively charged. These negatively charged carboxylate ligands are likely to attract the cationic metallic species. The higher binding of Cr(VI) at low pH values (2.5 to 3.5) could be explained through binding with positively charged ligands (amino groups). Optimum sorption of Cr(VI) occurs in the acidic range 2.5 to 3.5. At acidic pH (2.5), the sorbent is positively charged due to protonation of amino groups, while the sorbate, dichromate ion, exists mostly as an anion leading to electrostatic attraction between sorbent and sorbate. This results in increased sorption efficiency of FRLP for anionic metallic species at low pH. However, investigation on low pH variation beyond 7.5 yielded a false increase in sorption up to pH 8.5, which might be due to the precipitation carry over of Cr(III) ion which starts at pH 7.5.

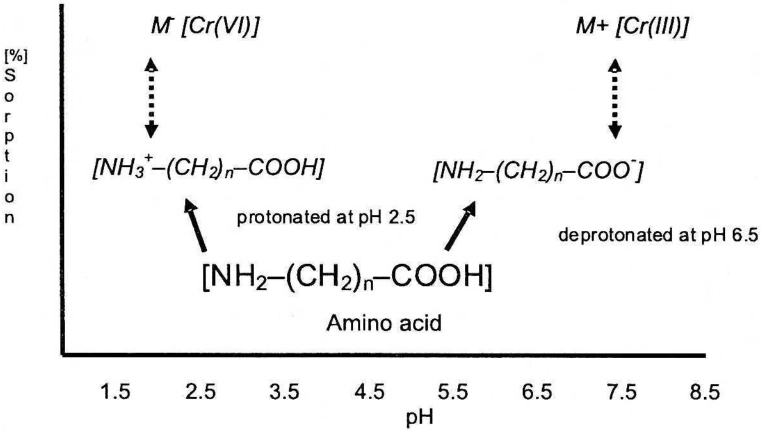

Fig. 1. pH profile and amino acid – chromium interactions on FRLP

Characterization of the biosorbent

IR spectrum of the solid phase of untreated (native) and Cr treated FRLP (exhausted) samples reflect the complex nature of the biomass. In the case of Cr(III) treated FRLP, NH_3^+ stretching band shifts from 2689.67 to 2712.23 cm^{-1}. The characteristic peak shifting of carboxylate ion from 1432.76 to 1673.17 cm^{-1} confirms ionic interaction between carboxylic groups of amino acid with the cationic metal species. In the case of IR spectrum of Cr(VI) treated FRLP, a weak COO^- symmetric stretching from 1432.76 to 1463.83 cm^{-1} and a well pronounced shift of NH_3^+ from 2689.67 to 2845.18 cm^{-1} were observed, which corroborates the electrostatic attraction between NH_3^+ and anionic metal species.

The powdered FRLP sieved at 105 μm had a surface area of 4.17 $m^2 \cdot g^{-1}$. The records of scanning electron micrographs of untreated (native) and metal treated FRLP (exhausted) are depicted in Figures 2 to 3.The comparison of scanning electron micrographs of untreated and metal treated FRLP represents large spherical clusters having a pore area of 7.67 μm^2 in the case of untreated FRLP while dense, agglomerated, etched dendrite type morphology having pore area of 0.72 μm^2 in Cr(III) and 0.64 μm^2 in Cr(VI) treated FRLP. Observed aggregation and reduction in pore area of Cr(III) and Cr(VI) treated FRLP may be ascribed to the liquid phase concentration of metal ions at experimental pH, confirming the biosorption phenomenon.

Sorption isotherms

Study of isotherms in Figures 4 and 5 indicates the adsorption capacity of leaf powder for the removal of target metal ions from the solution under standardized conditions. Adsorption isotherms had accurately described the biosorption experimental data of Cr(III) and Cr(VI) ions.

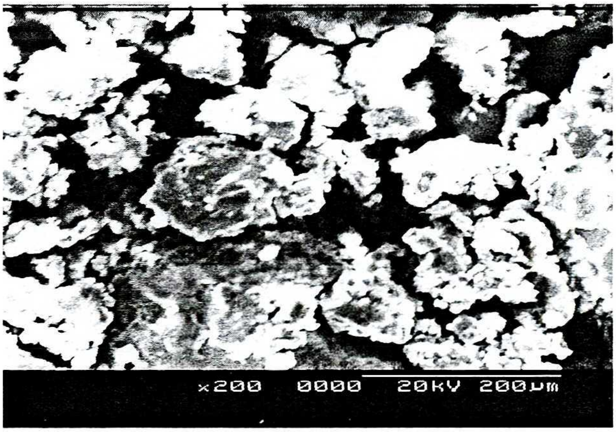

Fig. 2. Scanning electron micrograph of untreated FRLP showing large spherical clusters type morphology

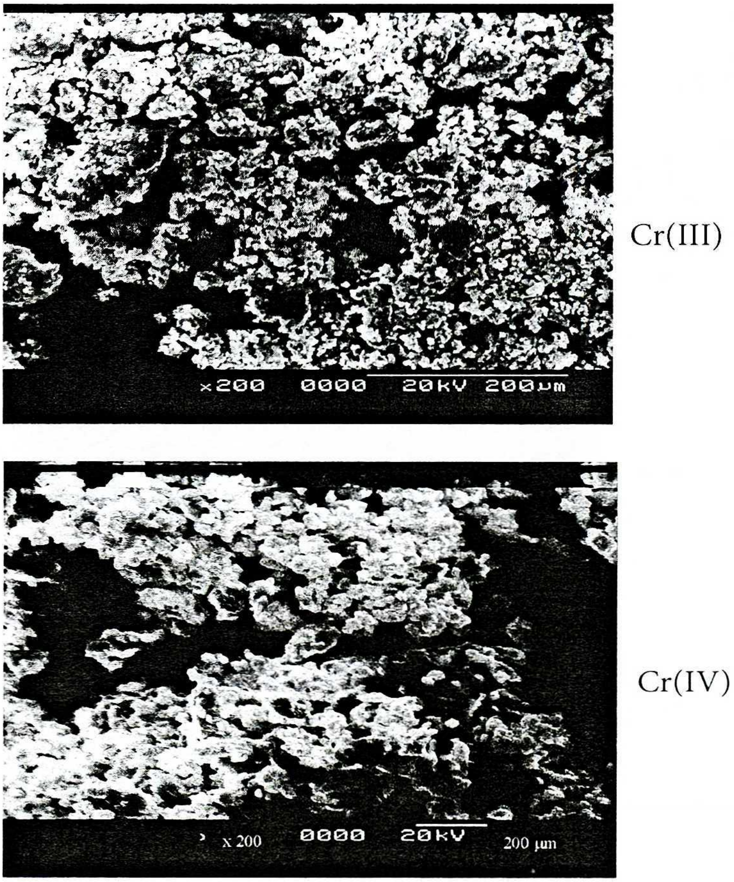

Fig. 3. Scanning electron micrographs of Cr(III) and Cr(VI) treated FRLP showing dense agglomerated, etched dendrite type morphology

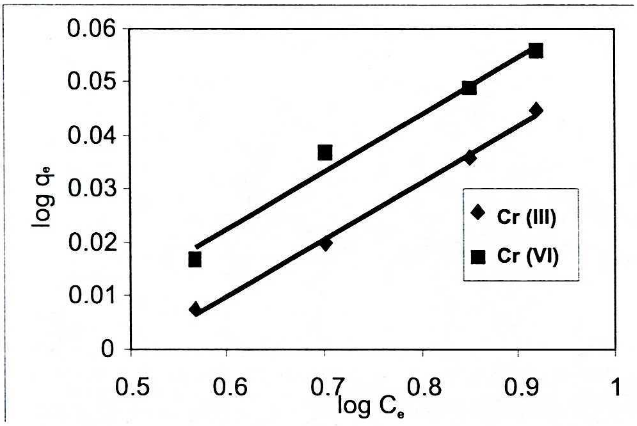

Fig. 4. Freundlich isotherm plot for the adsorption of Cr(III) and Cr(VI) on FRLP

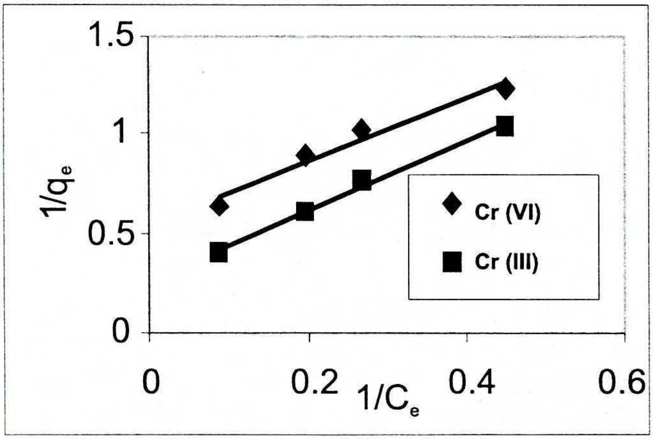

Fig. 5. Langmuir isotherm plot for the adsorption of Cr(III) and Cr(VI) on FRLP

The extent of sorption of Cr(III) and Cr(VI) on the FRLP has been described by Freundlich and Langmuir isotherms. According to Freundlich model, the maximum biosorption capacity (K_f) and intensity (1/n) obtained for Cr(III) – 0.72, 0.18 and Cr(VI) – 0.53, 0.09 depict the higher sorption efficiency of FRLP for chromium. Similarly, the values of biosorption capacity (Q_0) and energy (b) obtained from Langmuir model for the target metals were found to be Cr(III) – 2.59, 1.24; Cr(VI) – 2.74, 0.35 which represent their favorable sorption. Moreover, high values of correlation coefficient (R^2) (Cr(III) – 0.99 and Cr(VI) – 0.97) indicate that the adsorption pattern for both the species of chromium followed Freundlich and Langmuir isotherms. The linearity of Freundlich and Langmuir plots suggested the formation of homogenous monolayer of Cr(III) and Cr(VI) on the outer surface of the biosorbent.

Maximum uptake of Cr(III) and Cr(VI) over FRLP was 1.01 $mg \cdot g^{-1}$ and 2.20 $mg \cdot g^{-1}$ respectively. Kinetics experiments showed that the adsorption of Cr(III) and Cr(VI) increases with the lapse of time. However, the adsorption of Cr(III) and Cr(VI) was found to be rapid during 10 to 30 minutes of contact time after which the rate slowed down as the equilibrium approached at 30 minutes of contact time (Fig. 6). No further increase in sorption was observed with further increase of contact time up to 40 minutes. Adsorption rate constant (K_{ad}) was determined from the following first order rate expression.

$$\log (q_e - q_t) = \log (q_e) - (K_{ad}/2.303)t$$

where:

q_e and q_t [$mg \cdot g^{-1}$] are the amount of target metals adsorbed per unit mass of FRLP at equilibrium and at time t respectively,

K_{ad} [min^{-1}] was calculated from the slope of the linear plot of log (q_e - q_t) versus time.

The adsorption rate constants were Cr(III) – 0.38 min^{-1} for 25 $mg \cdot dm^{-3}$ and 0.42 min^{-1} for 50 $mg \cdot dm^{-3}$, Cr(VI) – 0.46 min^{-1} for 25 $mg \cdot dm^{-3}$ and 0.48 min^{-1} for 50 $mg \cdot dm^{-3}$ found to be independent of initial Cr concentration indicating that the adsorption of Cr(III) and Cr(VI) on FRLP follows first order rate kinetics.

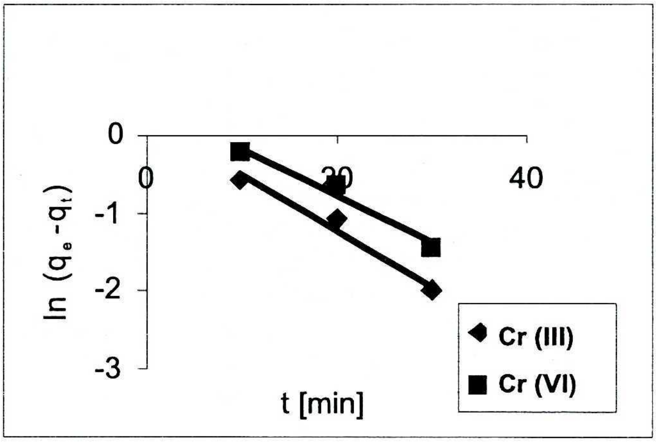

Fig. 6. Lagergren plots for adsorption of Cr(III) and Cr(VI) on FRLP (Cr – 25 $mg \cdot dm^{-3}$ and 50 $mg \cdot dm^{-3}$, m – 4 g, t – 30 min)

Desorption and regeneration studies

To make the proposed process of sorption more economical, attempts were made to regenerate the exhausted biomass for its effective reuse. Desorption behavior of Cr(III) and Cr(VI) from metal loaded biomass was observed after eluting with two mineral acids (hydrochloric and nitric acid). The maximum desorption (Cr(III) – 92.06% and Cr(VI) – 94.05%) was observed at hydrochloric acid strength (0.05 M). However, better desorption (Cr(III) – 98.63% and Cr(VI) – 99.12%) could be achieved with the same strength of nitric acid as eluant. Sorption of Cr on regenerated FRLP remained constant to the value (Cr(III) – 82.47% and Cr(VI) – 88.23%) up to three regeneration cycles and then started decreasing (Cr(III) – 78.14% and Cr(VI) – 81.90%) in the 4th cycle.

CONCLUSIONS

Sorption studies result into the standardization of the optimum conditions as biomass dosage (4.0 g), metal concentration (25 mg·dm^{-3} for Cr(III); 50 mg·dm^{-3} for Cr(VI)), and contact time (30 min) at pH 6.5 for Cr(III) and 2.5 for Cr(VI). Maximum sorption for Cr(III) and Cr(VI) species is 82.47 and 88.23% respectively. The study highlights the efficacy of FRLP in a new vision as environmental counterbalance to industrial pollution, at least, as a pretreatment step for the removal of chromium from aqueous media. Experimental data adequately fit into the Freundlich and Langmuir isotherm models. Adsorption process followed a first order kinetics. Amino acid – Cr interactions contribute the significant role in the biosorption of chromium using target leaf biomass. The spent biosorbent was regenerated and effectively reused, making the adsorption process more economical. Considering various aspects like metal concentration, volume of test solution and biomass dosages, the sorption efficiency of the present process is matching with earlier reported sorption of Cr on other biosorbents like cone biomass [22] and petiolar sheath of palm [13]. The findings open up new avenues in the decontamination and separation of metals using FRLP and have enough potential in the modern technology of water treatment as biosorbent.

Acknowledgment

The authors gratefully acknowledge Professor V.G Dass and Professor L.D. Khemani, Director and Head of the Department of Chemistry, Faculty of Science, Dayalbagh Educational Institute, Dayalbagh, Agra, for providing the necessary research facilities. Dr. T. K. Roy, Director, Central Pulp & Paper Research Institute, Saharanpur and Prof. K.K. Pant, Department of Chemical Engineering and Biotechnology, Indian Institute of Technology, New Delhi, are gratefully acknowledged for recording scanning electron micrographs and determination of BET surface area of the biosorbent. One of the authors, Pritee Goyal, is thankful to University Grants Commission, New Delhi for providing financial assistance.

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Received: September 21, 2007; accepted: May 14, 2008.