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POTENTIAL OF *SARACA INDICA* LEAF POWDER (SILP) FOR CHROMIUM REMOVAL FROM AQUEOUS SOLUTION

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Summary

The paper projects the potential of agricultural waste *Saraca indica* leaf powder (SILP) in biosorbing chromium from aqueous system. The influence of pH, contact time, metal concentration, biomass dosage and particle size on the selectivity of the removal process was investigated. The maximum sorption efficiency of SILP for Cr(III): 85.23% and Cr(VI): 89.67% was found to be pH dependent giving optimum sorption at pH 6.5 and 2.5 respectively. The adsorption process fitted well to both Freundlich and Langmuir isotherms. Morphological changes observed in Scanning Electron Micrographs of metal treated biomass confirm the existence of biosorption phenomenon. Fourier Transform Infra-red Spectrometry confirms that amino acid-Cr interactions contribute a significant role in the biosorption of chromium using target leaf powder. The successful applications of easily abundant agricultural waste SILP, as a biosorbent have potential for a low technological pretreatment step, prior to economically not viable high-tech chemical treatments for the removal of Cr from water bodies.

INTRODUCTION

The increase in industrial activity during recent years is greatly contributing to the elevation of heavy metals in the environment, mainly in the aquatic systems. Water pollution with heavy metals is an issue of great environmental concern. Among variety of toxic metals, high level of Cr is a burning issue round the globe. Chromium exists in two environmentally important oxidation states as trivalent and hexavalent. Hexavalent form of Cr is carcinogenic in nature and has been placed on a list of first quartile of 53 compounds by Cancer Assessment Group. Recent reports show that trivalent form can be lethal at higher concentration [5]. Conventionally, numbers of methods are employed for the removal of chromium from effluents viz. chemical precipitation, ion exchange, adsorption etc. These methods are plagued with incomplete removal, involve the use of large volume of chemicals, high costs, are laborious and generate other waste that require further disposal [11]. An emerging field of interest is employing the use of plants or agricultural waste that possess the natural ability to uptake heavy metals for the remediation of the environment. Natural materials that are available in large quantities or certain waste from agricultural operations may have potential to be used as low cost biosorbents as they represent unused resources, widely available and are environment friendly [1]. Thus, search for newer treatment technologies for the abatement of toxic metals from aqueous system has directed attention to biosorption. A variety of low cost plant materials for Cr removal such as petiolar sheath palm [16] and cone biomass [15] have been studied. In continuation of our work on the biosorption of toxic metals in aqueous system using agricultural waste [8, 12, 14] the present communication reports the sorption potential of *Saraca indica* leaf biomass (SILP) for the removal of trivalent and hexavalent chromium from the aqueous system.

Saraca indica Linn. (Ashoka), is an evergreen tree, abundantly available throughout the Asian continent and is well documented for its various pharmacological properties [9].

EXPERIMENTAL

Biosorbent preparation

Leaves of *Saraca indica* Linn. were collected in May 2004. 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. *Saraca indica* leaf powder (SILP) was used as biosorbent.

Biosorption studies

Sorption studies using standard practices were carried out in batch experiments as functions of biomass dosage (1, 2, 4 g), contact time (10, 20, 30, 40 min), metal concentration (1, 5, 10, 25, 50,100 ppm), particle size (105–420 μm) and pH (2.5–8.5). The solutions of Cr(III) comprised of chromium chloride and Cr(VI) 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:

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

where: C_o and C_e are the initial and final concentrations of metal ions in the solution.

Fourier Transform Infrared Spectroscopy (FTIR)

In order to gain better insight into the surface functional groups that might be involved in metal sorption, FTIR analysis in solid phase was performed using a Fourier Transform Infrared Spectrometer (FTIR-8400, Shimadzu). Spectra of the sorbent before and after chromium sorption were recorded.

Scanning Electron Microscopy (SEM)

Surface morphology was studied with Scanning Electron Microscope (Steroscan 360, Cambridge Instruments, U.K). The Scanning Electron Micrograph (SEM) of untreated (native) and chromium treated (exhausted) at bar length equivalent to 50 μm, working voltage 20 KV with 600x magnification were recorded.

BET Surface Area Analysis

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

Sorption Isotherms

The experimental data for the sorption of chromium ions by the SILP over the studied concentration range processed in accordance with the two of the most widely used adsorption isotherms: Freundlich and Langmuir isotherm describing the adsorption phenomenon [6].

The classical Freundlich equation is given below:

$$q = K_f C_e^{1/n}$$

where: q is the heavy metal adsorbed on the biosorbent [mg/g dry weight]; C_e is the final concentration of metal [mg/dm^3] in the solution; K_f and n are the characteristic constants.

The classical Langmuir equation is given below:

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

where: C_e is the equilibrium concentration, q_e is the amount adsorbed of Cr(III) and Cr(VI) at equilibrium, Q_o and b are the 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.

Kinetics experiment

To determine the kinetics of sorption of chromium onto SILP, Lagregan plots were obtained at initial Cr(III) and Cr(VI) concentration. SILP (4.0 g) was suspended in 200 cm^3 of the metal solutions of known initial sorbate concentration at pH 6.5 for Cr(III) and 2.5 for Cr(VI), where maximum sorption was recorded. The mixture was continuously stirred using a magnetic stirrer. Samples were withdrawn at predetermined time intervals in the range of 10 to 40 minutes, filtered and analyzed for residual chromium concentration.

Desorption of chromium from loaded biomass

Desorption studies help in elucidating the mechanism of metal ion removal and recovery from metal loaded biosorbent and also for the regeneration and recycling of spent adsorbents, which in turn may reduce operational cost and protect the environment. In order to assess the reusability of SILP, desorption studies (batch process) were conducted to restore the biomass as a function of concentration (0.01–0.1 M) of different stripping agent (hydrochloric and nitric acid). Cr(III) and Cr(VI) loaded biosorbents obtained from our sorption experiments were transferred to Erlenmeyer flask and shaken with 50 cm^3 of each acid for 40 min. The filtrate was analyzed for desorbed chromium.

Statistical analysis

Batch experiments were conducted in replicates (N = 5) and data represent the mean value. Mean values, correlation coefficients and standard deviation were calculated using SPSS PC$^+_{\text{TM}}$ statistical package [13]. For the determination of inter group mean value differences, each parameter was subjected to the student's t – test for significance level ($p < 0.05$).

RESULTS AND DISCUSSION

The main parameters influencing metal sorption were investigated: contact time, metal concentration, amount of adsorbent and pH value of solution. Table 1 and Table 2 represent soluble chromium ion concentration after biosorption on unmodified *Saraca indica* leaf powder. Sorption studies result into the standardization of the optimum conditions such as biomass dosage (4.0 g), metal concentration (25 mg/dm^3), contact time (30 min) at pH 6.5 for Cr(III) and 2.5 for Cr(VI) respectively. The results indicate that SILP has considerable potential to be used as biosorbent for chromium removal from the water bodies. Studies would therefore have to be carried out to provide insight into the mechanism of interactions between chromium and SILP.

Table 1. Soluble Cr(III) ion concentration [μM] after adsorption on unmodified *Saraca indica* leaf powder (SILP) as functions of contact time and biomass dosage at: volume 200 cm^3, particle size 105 μm and pH 6.5

Initial conc. ppm/[μM]	Time interval [minutes]			
	10	20	30	40
Biomass dosage (2.0 g)				
01 (03.84)^{a}	01.24±0.12$^{bx+\Phi}$	01.01±0.11^{x+}	0.45±0.09^{x+}	0.45±0.09^{xx+}
05 (19.23)	14.11±0.56^{x+}	12.38±0.49^{x+}	10.76±0.45^{x+}	10.74±0.44^{xx+}
10 (38.46)	24.69±1.02^{x+}	21.61±0.94^{x+}	19.57±0.83^{x+}	19.54±0.82^{xx+}
25 (96.15)	55.46±2.33^{x+}	46.50±1.96^{x+}	40.96±1.76^{x+}	40.80±1.74^{xx+}
50 (192.3)	110.92 ±4.92^{x+}	93.01±4.84^{x+}	81.92±3.83^{x+}	81.60±3.82^{xx+}
100 (384.61)	221.82±11.3^{x++}	118.4±9.6^{x++}	163.8±8.76^{x++}	162.7±8.74^{xx++}
Correlation coefficient (r)	0.95	0.94	0.93	0.96
Biomass dosage (4.0 g)				
01 (03.84)^{a}	02.57±0.07$^{x+\Phi}$	01.23±0.05^{x+}	01.03±0.04^{x+}	01.03±0.04^{xx+}
05 (19.23)	07.76±0.30^{x+}	06.03±0.22^{x+}	04.88±0.18^{x+}	04.88±0.18^{xx+}
10 (38.46)	12.80±0.53^{x+}	09.65±0.39^{x+}	07.53±.27^{x+}	07.51±0.27^{xx+}
25 (96.15)	27.26±1.53^{x+}	19.42±1.39^{x+}	14.20±1.23^{x+}	14.11±1.21^{xx+}
50 (192.3)	54.38±3.31^{x+}	38.84±2.90^{x+}	28.30±2.64^{x+}	28.15±2.64^{xx+}
100 (384.61)	108.9±7.31^{x++}	77.68±6.8^{x++}	56.60±5.64^{x++}	56.30±5.64^{xx++}
Correlation coefficient (r)	0.96	0.94	0.97	0.98
Biomass dosage (6.0 g)				
01 (03.84)^{a}	2.57±0.07$^{x+\Phi\Phi}$	01.23±0.05^{x+}	01.03±0.04^{x+}	01.03±0.04^{xx+}
05 (19.23)	07.75±0.30^{x+}	06.02±0.22^{x+}	04.86±0.18^{x+}	04.86±0.18^{xx+}
10 (38.46)	12.78±0.53^{x+}	09.61±0.39^{x+}	07.52±.27^{x+}	07.50±0.27^{xx+}
25 (96.15)	27.23±1.47^{x+}	19.39±1.35^{x+}	14.16±1.21^{x+}	14.09±1.21^{xx+}
50 (192.3)	54.32±3.27^{x+}	38.83±2.82^{x+}	28.23±2.58^{x+}	28.11±2.57^{xx+}
100 (384.61)	108.1±7.21^{x++}	77.57±6.5^{x++}	56.51±5.61^{x++}	56.21±5.62^{xx++}
Correlation coefficient (r)	0.95	0.95	0.97	0.97

[a] number in parenthesis represent soluble metal concentrations [μM],

[b] standard deviation values of replicate (N = 5) determinations,

mean difference (initial Cr(III) loaded [μM] versus soluble Cr(III) [μM]) as functions of time: ^{x} significance ($p < 0.10$), ^{xx} insignificance ($p > 0.10$),

metal concentration: $^{+}$ significance ($p < 0.01$), $^{++}$ insignificance ($p > 0.01$),

biomass dosage: $^{\Phi}$ significance ($p < 0.01$), $^{\Phi\Phi}$ insignificance ($p > 0.01$).

Table 2. Soluble Cr(VI) ion concentration [μM] after adsorption on unmodified *Saraca indica* leaf powder (SILP) as functions of contact time and biomass dosage at: volume 200 cm^3, particle size 105 μm and pH 2.5

Initial conc. ppm/(μM)	Time interval (minutes)			
	10	20	30	40
Biomass dosage (2.0 g)				
01 (03.84)[a]	02.38±0.17$^{bx+\Phi}$	02.23±0.15^{x+}	2.21±0.14^{x+}	2.21±0.14^{xx+}
05 (19.23)	11.51±0.52^{x+}	10.38±0.46^{x+}	10.23±0.42^{x+}	10.21±0.39^{xx+}
10 (38.46)	20.69±0.88^{x+}	18.61±0.83^{x+}	17.57±0.79^{x+}	16.54±0.76^{xx+}
25 (96.15)	46.02±2.23^{x+}	41.50±1.96^{x+}	40.96±1.76^{x+}	40.80±1.74^{xx+}
50 (192.3)	91.73 ±4.49^{x+}	82.01±4.01^{x+}	80.92±3.96^{x+}	79.60±3.78^{xx+}
100 (384.61)	183.82±09.3^{x++}	164.4±8.6^{x++}	160.8±8.52^{x++}	159.7±8.46^{xx++}
Correlation coefficient (r)	0.95	0.94	0.93	0.96
Biomass dosage (4.0 g)				
01 (03.84)[a]	02.13±0.13$^{x+\Phi}$	01.97±0.11^{x+}	01.88±0.09^{x+}	01.88±0.09^{xx+}
05 (19.23)	05.76±0.47^{x+}	04.76±0.39^{x+}	04.42±0.35^{x+}	04.38±0.35^{xx+}
10 (38.46)	08.80±0.73^{x+}	06.65±0.56^{x+}	06.07±.47^{x+}	06.03±0.39^{xx+}
25 (96.15)	15.76±0.72^{x+}	15.42±0.70^{x+}	09.96±0.46^{x+}	09.81±0.46^{xx+}
50 (192.3)	31.38±1.47^{x+}	24.84±1.14^{x+}	19.88±0.92^{x+}	19.65±0.90^{xx+}
100 (384.61)	62.5±3.01^{x++}	48.68±2.3^{x++}	39.60±1.88^{x++}	38.30±1.88^{xx++}
Correlation coefficient (r)	0.96	0.94	0.97	0.98
Biomass dosage (6.0 g)				
01 (03.84)[a]	2.13±0.13$^{x+\Phi\Phi}$	01.97±0.11^{x+}	01.88±0.09^{x+}	01.88±0.09^{xx+}
05 (19.23)	05.72±0.46^{x+}	04.69±0.37^{x+}	04.35±0.34^{x+}	04.32±0.34^{xx+}
10 (38.46)	08.69±0.71^{x+}	06.57±0.54^{x+}	05.97±.45^{x+}	05.92±0.37^{xx+}
25 (96.15)	15.61±0.67^{x+}	15.31±0.59^{x+}	09.89±0.38^{x+}	09.73±0.32^{xx+}
50 (192.3)	31.19±1.34^{x+}	24.67±1.03^{x+}	19.83±0.83^{x+}	19.59±0.78^{xx+}
100 (384.61)	62.3±2.89^{x++}	48.53±2.7^{x++}	39.47±1.61^{x++}	37.03±1.57^{xx++}
Correlation coefficient (r)	0.95	0.95	0.97	0.97

[a] number in parenthesis represent soluble metal concentrations [μM],

[b] standard deviation values of replicate (N = 5) determinations,

mean difference (initial Cr(VI) loaded [μM] versus soluble Cr(VI) [μM]) as functions of time: ^{x} significance ($p < 0.10$), ^{xx} insignificance ($p > 0.10$),

metal concentration: $^{+}$ significance ($p < 0.01$), $^{++}$ insignificance ($p > 0.01$),

biomass dosage: $^{\Phi}$ significance ($p < 0.01$), $^{\Phi\Phi}$ insignificance ($p > 0.01$).

pH dependency for chromium ion binding

The variation of sorption efficiency with pH (2.5–8.5) in the case of Cr(III) and Cr(VI) has been presented in Fig. 1. The biosorption efficiency of Cr(III) increased gradually with rise in pH from 2.5 to 6.5 attaining optimum sorption (85.23%) at 6.5. However, percent sorption remained almost constant with further rise in pH up to 7.5. Increase in pH from 7.5 to 8.5, resulted into Cr(III) hydroxide precipitation. Inverse trend was obtained for anionic metallic species Cr(VI), where optimum sorption (89.67%) was observed in acidic pH range from 2.5 to 3.5. Further increase in pH from 3.5 to 8.5 resulted into a sharp decreasing trend of sorption.

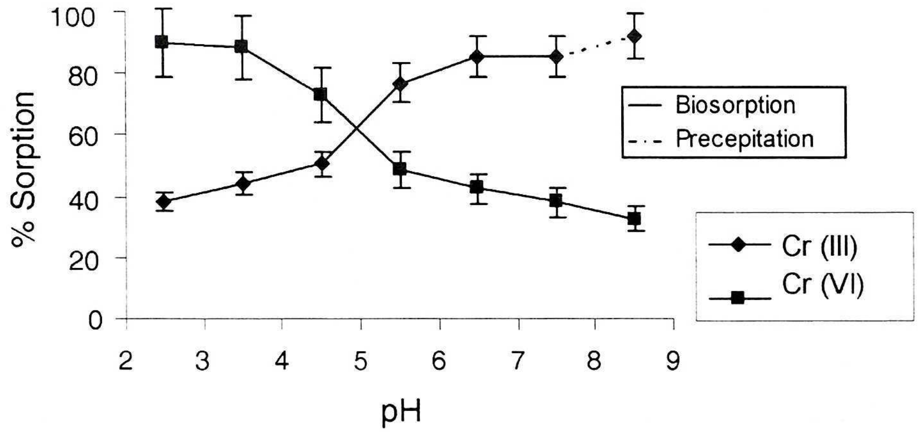

Fig. 1. Effect of pH on sorption behavior of [Cr(III) and Cr(VI)] on *Saraca indica* leaf powder (SILP); error bars represent standard deviation of five replicates determination (C_o – 25 mg/dm^3; m – 4.0 g; t – 30 min)

Basing on our experimental findings and relevant literature available on *Saraca indica* leaf powder (SILP), we synthesize a possible mechanism for chromium binding to the leaf biomass. The aerial part of *Saraca indica* possesses various organic chemical moieties prominently large proportions of low molecular weight amino acids [10]. Amino acids have been found to constitute a physiologically active group of binding agents working even at low concentration, which due to ability to interact with metal ions are likely to be active sites for the sorption of metal ions [3]. pH profile and sorption behavior of Cr(III) and Cr(VI) provide an insight into the mechanistic aspect of sorption.

Solution chemistry of Cr shows its existence as Cr(III) in cationic forms, such as Cr^{3+}, $Cr(OH)_2^+$ in the pH range from 4.0 to 6.0 [7], whereas Cr(VI) exist in anionic forms, such as $Cr_2O_7^{2-}$, $HCr_2O_7^-$, $HCrO_4^-$ and CrO_4^{2-} at low pH values from 1.0 to 4.0 [2]. The majority of amino acid present in SILP have isoelectric points in the pH range from 4.0 to 8.0 [4]. 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. Biosorption of Cr(III) at pH 6.5 may be ascribed to the interaction of negatively charged carboxylic group with positively charged metallic species. Optimum sorption for Cr(VI) was obtained at pH 2.5. At lower 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. Cr – SILP interactions can further be highlighted on the basis of the records of IR spectrum of native and exhausted SILP after metal sorption. In the case of Cr(III), the characteristic peak shifting of carboxylate ion from 1350.86 and 1552.14 cm^{-1} to 1427.62 and 1678.23 cm^{-1} confirms an ionic interaction between carboxylic groups of amino acid with cationic metallic species. A well pronounced shift of NH_3^+ in native sample from 2428.52 and 2722.5 cm^{-1} to 2634.23 and 2752.34 cm^{-1} in exhausted sample is resulted. This shifting in peak shows the electrostatic attraction between NH_3 of amino acids present in SILP with anionic metallic species.

The powdered SILP sieved at 105 μm had a surface area of 4.23 m^2/g. The records of scanning electron microscopy of native and exhausted SILP are presented in Figs 2 and 3 respectively. The comparison of scanning electron micrograph of untreated and Cr(III) and Cr(VI) treated SILP represents large spherical clusters having pore area 6.32 μm^2 in the case of native sample while dense agglomerated, irregular type morphology having pore area of 0.63 μm^2 and 0.58 μm^2 in Cr(III) and Cr(VI) treated SILP respectively. Observed aggregation and reduction in pore area of chromium treated SILP may be ascribed to liquid phase concentration of metal ions at experimental pH, thus confirming the biosorption phenomenon.

Sorption capacity using isotherms

The extent of sorption of Cr on SILP 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): 0.73, 0.15 and Cr(VI): 0.57, 0.05 depict the higher sorption efficiency of SILP 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): 2.79, 1.29; Cr(VI): 2.87, 0.46 which represent their favorable sorption. Moreover, high values of correlation coefficient (R^2) [Cr(III): 0.98 and Cr(VI): 0.96] 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.

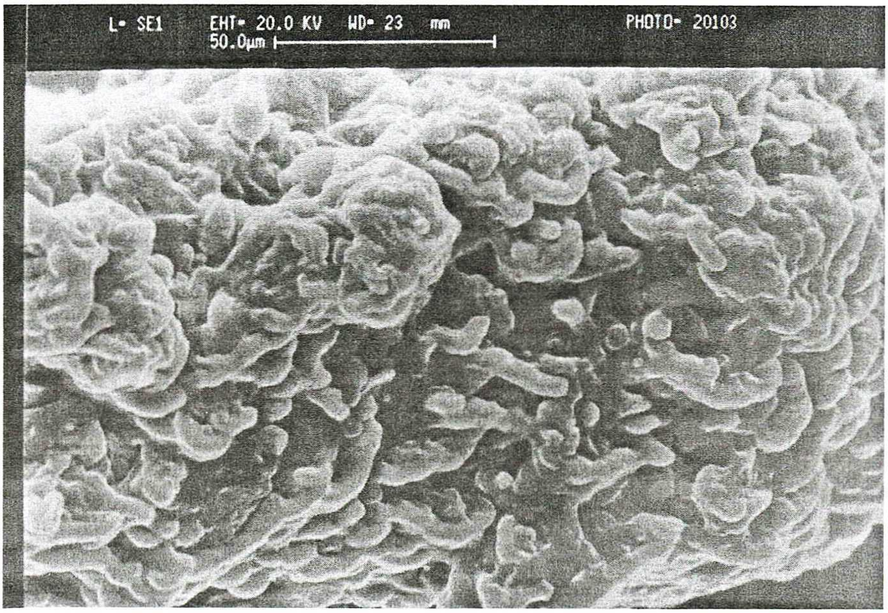

Fig. 2. Scanning electron micrograph of untreated *Saraca indica* leaf powder (SILP) showing large spherical clusters type morphology

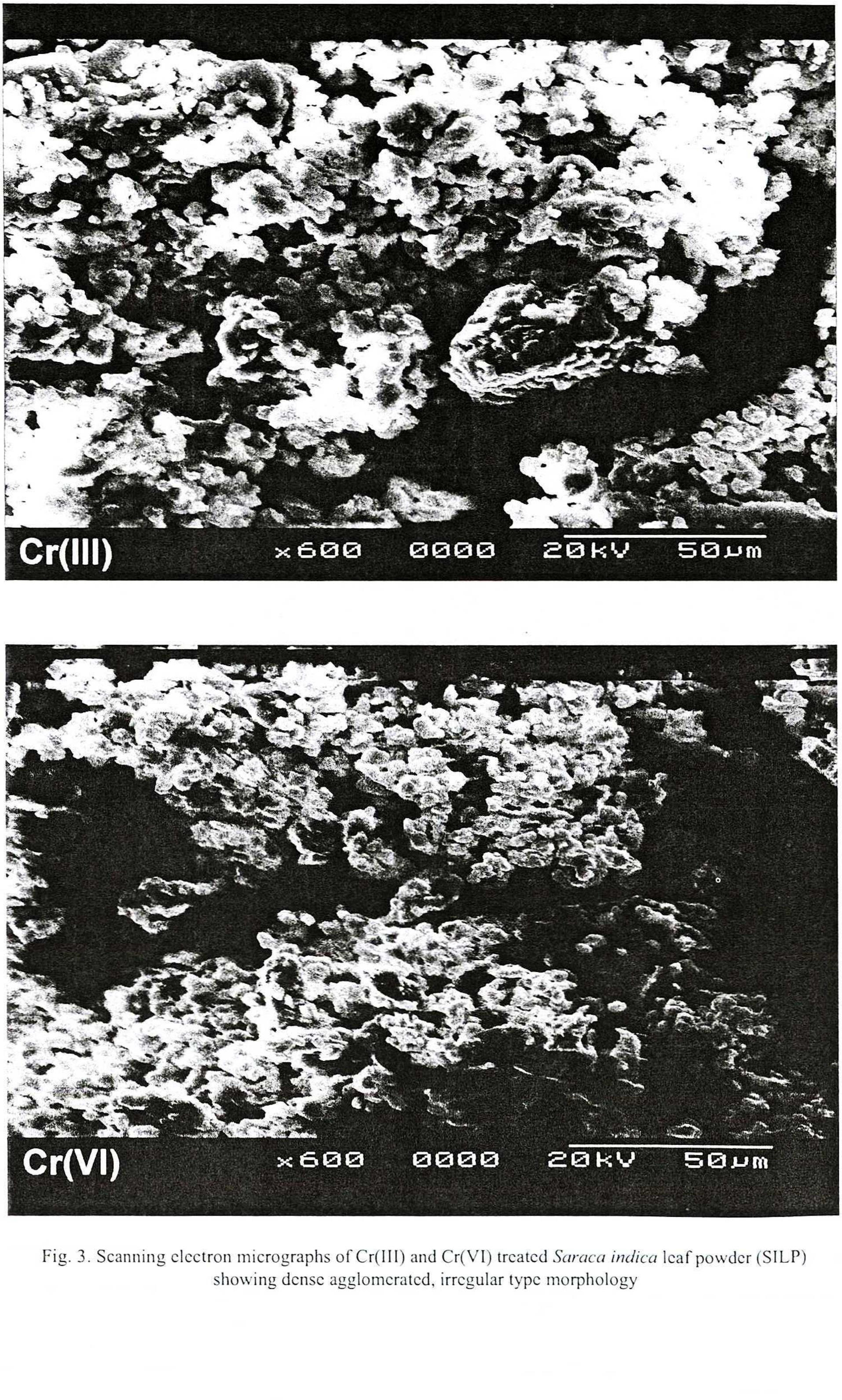

Fig. 3. Scanning electron micrographs of Cr(III) and Cr(VI) treated *Saraca indica* leaf powder (SILP) showing dense agglomerated, irregular type morphology

Kinetics experiment showed that the adsorption of Cr(III) and Cr(VI) increased with the lapse of time. The adsorption of Cr(III) and Cr(VI) was rapid during 10–30 min. of contact time after which the rate slowed down as the equilibrium reached 30 min. of contact time. 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/g] are the amount of Cr adsorbed per unit mass of SILP at equilibrium and at time 't', respectively. K_{ad} [min^{-1}] was calculated from the slope of linear plot of log (q_e - q_t) versus time. The value of adsorption rate constant for Cr(III) 0.47 and Cr(VI) 0.52 min^{-1} indicate that adsorption of Cr on SILP follows first order rate kinetics.

Regeneration Studies

Regeneration and recovery of spent adsorbent is very important as it will reduce the cost of remediation of heavy metals and other contaminants in the environment. With this aim in view, desorption behavior of Cr(III) and Cr(VI) from metal loaded biomass was observed after eluting with two mineral acids (hydrochloric acid, nitric acid). The maximum desorption [Cr(III) 94.37%; Cr(VI) 96.53%] was observed at hydrochloric acid strength (0.05 M) however, better desorption [Cr(III) 98.46%, Cr(VI) 99.67%] could be achieved with the same strength of nitric acid as eluant. Sorption of Cr(III) and Cr(VI) was highest in regenerated cycles and then started decreasing [Cr(III) 78.27%; Cr(VI) 84.57%] in the 4th cycle.

CONCLUSIONS

Experimental results have demonstrated that *Saraca indica* leaf powder can be utilized as cost effective and environmentally friendly technique for the abatement of Cr(III) (85.23%) and Cr(VI) (89.67%) from aqueous system. Freundlich and Langmuir isotherms supported the suitability of the proposed method thus explaining the easy separation of chromium ions from the aqueous system. The present biosorbent can be used successfully for resorption up to three cycles. Amino acid-Cr interactions play a significant role in biosorption. These laboratory based findings open up new avenues in the abatement of chromium by *Saraca indica* leaf powder which is, in fact an agricultural waste owing to heavy leaf fall in autumn and spring seasons. Thus, it introduces a less expensive, domestic and environment friendly green method for the removal of chromium from water bodies.

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