

***Saraca indica* leaf powder for decontamination of Pb: removal, recovery, adsorbent characterization and equilibrium modeling**

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ABSTRACT: The present study explores the effectiveness of *Saraca indica* leaf powder, a surplus low value agricultural waste, in removing Pb ions from aqueous solution. The influence of pH, biomass dosage, contact time, particle size and metal concentration on the removal process were investigated. Batch studies indicated that maximum biosorption capacity for Pb was 95.37% at the pH 6.5. The sorption process followed the first order rate kinetics. The adsorption equilibrium data fitted best to both Langmuir and Freundlich isotherms. Morphological changes observed in scanning electron micrographs of untreated and metal treated biomass confirmed the phenomenon of biosorption. Fourier transform infrared spectroscopy of native and exhausted leaf powder confirmed lead biomass interactions responsible for sorption. Acid regeneration was tried for several cycles with a view to recover the sorbed metal ion and also to restore the sorbent to its original state. The findings showed that *Saraca indica* leaf powder can easily be envisaged as a new, vibrant, low cost biosorbent for metal clean up operations.

Key words: *Agricultural waste, lead, Saraca indica leaf powder, biosorption, regeneration, lead biomass interactions*

INTRODUCTION

Heavy metal pollution of aqueous streams is a major environmental problem for the modern world. Beyond any doubt, it has been established that dissolved substances, particularly heavy metals escaping into the environment, pose serious health hazards. To follow the fate of metallic species, after they enter the ecosystem, becomes very difficult and they start to inflict the damages as they move through, from one ecological tropic layer into another. These heavy metals accumulate in living tissues throughout the food chain. Pb is one such deleterious metal, detected in waste streams from mining operations, tanneries, electronics, electroplating and petrochemical industries (Vasuderan, *et al.*, 2003). According to US Environmental Protection Agency, the maximum permissible limit for Pb in drinking and waste water is 0.015 and 0.1 mg/L, respectively (Muralikrishna, 1997). Currently used water treatment technologies for Pb (II) involves chemical precipitation, evaporation, electro chemical treatment, ion exchange (Lo, *et al.*, 1999). These available treatment technologies are not effective enough and they are expensive and inadequate, considering the vast waste water

quantities. Search for newer treatment technologies for removal of toxic metals from waste water has directed attention to biosorption. This novel approach is competitive, effective and cheap. Natural materials that are available in large quantities are potential to be used as low cost adsorbents which are widely available and are environment friendly, as they represent unused resources (Deans and Dixon, 1992). Various plant materials viz. rice husk, maize cobs, coconut and seed hull (Gueu, *et al.*, 2007) saw dust (Abdel-Ghani, *et al.*, 2007), maize leaf (Adesola Babarinde, *et al.*, 2006) and olive pomace (Pagnenelli, *et al.*, 2006) have been studied for Pb removal from water bodies. The present investigation deals with the abatement of Pb (II) ions from aqueous system using *Saraca indica* leaf powder (SILP). The manuscript also reports the applicability of leaf biomass for Pb (II) metal ion recovery and regenerate the exhausted biosorbent thereby making the process more economical, beneficial and cost effective. *Saraca indica* Linn. (Ashok), an evergreen tree that is abundantly available throughout the Asian continent, possesses multidimensional properties and carries great significances like its use in fractures of bones, beautifying complexion, an astringent and in various

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gynecological problems since past ages (Wealth of India, 2003).

MATERIALS AND METHODS

The 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 h., crushed and finally sieved through (105 µm, 210 µm and 420 µm) mesh copper sieves. SILP was used as biosorbent. Sorption studies using standard practices were carried out in batch experiments as functions of biomass dosage (2, 4 and 6 g), contact time (10, 20, 30 and 40 min.), metal concentration (1, 5, 10, 25, 50 and 100 ppm.), particle size (105 µm, 210 µm and 420 µm) and pH (2.5, 3.5, 4.5, 5.5, 6.5, 7.5 and 8.5). The solution of Pb (II) comprised of Pb nitrate was taken in an Erlenmeyer flask. 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 taken into account for metal ion estimation using atomic absorption spectroscopy (Perkin Elmer-3100). The percentage of 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 concentration of metal ions in the solution. Pb uptake by biosorbent was calculated (Vieira and Boya, 2003) using the mass balance equation for the biosorbent:

$$q = [V(C_i - C_f)] / S$$

q = Pb uptake (mg metal / g dry weight)

V = volume of metal bearing solution contacted (batch) with the biosorbent (L)

C_i = initial concentration of metal in solution (mg/L)

C_f = final concentration of target leaf biomass in solution (mg/L)

S = dry weight of biosorbent added (g)

For the main functional groups that might be involved in metal uptake, a FTIR record in solid phase was obtained using a FTIR-8400, Shimadzu. Before and after Pb sorption process, Spectra of the sorbent were recorded. Surface morphology was studied with Scanning Electron Microscope (Steroscan 360, Cambridge Instruments, U.K). The scanning electron micrograph (SEM) of untreated (native) and Pb treated (exhausted) at bar length equivalent to 50 µm, working

voltage 20 KV with 600 x magnification were recorded. The surface area of the biosorbent was measured using a Micrometrics ASAP-2010 BET surface area analyzer. Freundlich and Langmuir isotherms were used for interpreting the Pb biosorption equilibrium onto SILP. The classical Freundlich equation is: $q = K_f C_e^{1/n}$. Here, q is the heavy metal adsorbed on the biosorbent (mg/g dry weight); C_e is the final concentration of metal (mg/L) in the solution; K_f and n are the characteristic constants.

The classical Langmuir equation is:

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

where, C_e is the equilibrium concentration; q_e is the amount adsorbed of Pb (II) 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. Lagergren plots were also obtained by conducting kinetics studies (batch) at initial Pb (II) concentration. SILP (4.0 g) was suspended in 200 mL of Pb (II) solution of known initial sorbate concentration at pH 6.5, where maximum sorption was recorded. The mixture was continuously magnetically stirred. Sample was withdrawn at predetermined time intervals in the range of 10 to 40 min., filtered and analyzed for residual Pb concentration. Desorption experiments were carried out in order to explore the feasibility of recovering the Pb (II) ion and to reuse the exhausted SILP biosorbent for further cycle of sorption. Desorption studies (batch process) were conducted to desorb target metal ions from loaded SILP biomass as a function of concentration (0.01–0.1 M) of different stripping agents (hydrochloric and nitric acid). Pb loaded biosorbent obtained from this sorption experiments was transferred to Erlenmeyer flask and shaken with 50 mL of each acid for 40 min. The filtrate was analyzed using AAS for desorbed metals. Batch experiments were conducted in replicates (N=5) and data represents the mean value. The mean values, correlation coefficients and standard deviation were calculated using SPSS PC⁺ statistical package (SPSS, 1983). For 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

Table 1 represents soluble Pb (II) ion concentration after biosorption on SILP as a function of adsorbent dosage, particle size, sorption time and metal concentration at pH 6.5. In each case, data was evaluated at significance level ($P < 0.05$). The effect of adsorbent dosage on the removal of Pb (II) from the aqueous system using SILP varied from 2.0 to 6.0 g. It is observed that the sorption percentage increased as the biomass dosage raised from 2.0 to 4.0 g. However, no significant increment in the sorption tendency was observed on further increase in the biomass dosage from 4.0 g onwards. This might be due to the attainment of equilibrium between adsorbate and adsorbent at the existing operating conditions rendering adsorbent incapable of further adsorption. Sorption behavior of Pb (II) metal ion on target leaf biomass was studied at three different range of particle size (105 μm , 210 μm and 420 μm) of SILP. The results show a decrease in particle size and exhibit a favorable effect on metal sorption. It is probably due to the fact that the smaller the size of the particle, the greater is the sorption efficiency. The reduction in particle size of the biosorbent results in an increase in the surface area.

With increased availability of the surface area, more adsorption sites are available for metal removal. The adsorption of Pb (II) metal ion on the biosorbent was studied at various time intervals within a duration of 10 to 40 min. The sorption percentage of sorption of Pb (II) ion gradually increased in a time lag from 10 to 20 min., finally reaching the optimum value in 30 min. and remains fairly uniform onwards. The rapid metal removal had significant practical importance, as this will facilitate and ensure efficiency and economy. The sorption behavior of Pb (II) was carried out over a wide range of concentration ranging from 1 to 100 mg/L. In general, the sorption of Pb (II) on SILP initially increased with increasing concentration of the metal ion as it finally reaches to the optimum level at 25 mg/L, respectively. Thereafter, no significant increase in sorption percentage was found. The pH condition of the solution is an extremely important factor in metal biosorption, since it governs a series of phenomenon such as site dissociation and chemistry of the heavy metals. The effect of pH on the binding of Pb metal ion by the SILP is shown in Fig 1. As the pH of the solution increases from 2.5 to 6.5, the metal shows an increase in binding to the biomass with an optimum sorption (i.e. 95.37%)

Table 1: Soluble Pb (II) ion concentration (μM) after adsorption on unmodified SILP as functions of contact time and biomass dosage at volume (200 mL), particle size (105 μm) and pH (6.5)

Initial concentration (μM)	Time interval (min.)			
	10	20	30	40
	Biomass dosage (2.0 g)			
01 (0.96) ^a	0.51 \pm 0.03 ^{b*}	0.44 \pm 0.02 ⁺	0.37 \pm 0.02 ⁺	0.37 \pm 0.02 ^{***}
05 (4.82)	2.12 \pm 0.11 ⁺	1.75 \pm 0.09 ⁺	1.46 \pm 0.08 ⁺	1.46 \pm 0.08 ^{***}
10 (9.65)	3.56 \pm 0.23 ⁺	2.92 \pm 0.21 ⁺	2.41 \pm 0.18 ⁺	2.36 \pm 0.14 ^{***}
25 (24.13)	7.79 \pm 0.53 ⁺	6.13 \pm 0.48 ⁺	4.89 \pm 0.39 ⁺	4.88 \pm 0.23 ^{***}
50 (48.26)	15.54 \pm 1.19 ^{***}	12.25 \pm 1.08 ^{***}	9.74 \pm 0.92 ^{***}	9.60 \pm 0.78 ^{***}
100 (96.52)	31.02 \pm 2.84 ^{***}	24.38 \pm 2.6 ^{***}	19.4 \pm 2.43 ^{***}	19.3 \pm 2.17 ^{***}
Correlation coefficient (r)	0.97	0.97	0.98	0.99
	Biomass dosage (4.0 g)			
01 (0.96) ^a	0.39 \pm 0.02 ^{b*}	0.30 \pm 0.02 ⁺	0.25 \pm 0.01 ⁺	0.24 \pm 0.01 ^{***}
05 (4.82)	1.47 \pm 0.09 ⁺	1.08 \pm 0.07 ⁺	0.88 \pm 0.06 ⁺	0.83 \pm 0.05 ^{***}
10 (9.65)	2.27 \pm 0.18 ⁺	1.48 \pm 0.15 ⁺	0.93 \pm 0.09 ⁺	0.87 \pm 0.04 ^{***}
25 (24.13)	4.52 \pm 0.37 ⁺	2.47 \pm 0.29 ⁺	1.16 \pm 0.15 ⁺	1.11 \pm 0.04 ^{***}
50 (48.26)	8.97 \pm 1.07 ^{***}	4.88 \pm 0.93 ^{***}	2.16 \pm 0.82 ^{***}	2.12 \pm 0.69 ^{***}
100 (96.52)	17.9 \pm 2.59 ^{***}	9.75 \pm 2.40 ^{***}	4.29 \pm 2.23 ^{***}	4.22 \pm 2.04 ^{***}
Correlation coefficient (r)	0.96	0.97	0.97	0.98
	Biomass dosage (6.0 g)			
01 (0.96) ^a	0.39 \pm 0.02 ^{b*}	0.30 \pm 0.02 ⁺	0.25 \pm 0.04 ⁺	0.24 \pm 0.04 ^{***}
05 (4.82)	1.46 \pm 0.08 ⁺	1.08 \pm 0.06 ⁺	0.84 \pm 0.05 ⁺	0.81 \pm 0.05 ^{***}
10 (9.65)	2.25 \pm 0.16 ⁺	1.43 \pm 0.12 ⁺	0.88 \pm 0.07 ⁺	0.87 \pm 0.03 ^{***}
25 (24.13)	4.50 \pm 0.35 ⁺	2.45 \pm 0.24 ⁺	1.13 \pm 0.12 ⁺	1.09 \pm 0.09 ^{***}
50 (48.26)	8.96 \pm 1.01 ^{***}	4.86 \pm 0.89 ^{***}	2.12 \pm 0.75 ^{***}	2.09 \pm 0.64 ^{***}
100 (96.52)	17.8 \pm 2.51 ^{***}	9.68 \pm 2.3 ^{***}	4.21 \pm 2.18 ^{***}	4.09 \pm 1.89 ^{***}
Correlation coefficient (r)	0.96	0.96	0.98	0.97

a: Number in parenthesis represent soluble metal concentrations in μM .; b: Standard deviation values of replicate (N=5) determinations
Mean difference [initial Pb (II) loaded (μM) versus soluble Pb (II) (μM)]; as functions of Time *: Significance ($p < 0.05$), **: Insignificance ($p > 0.05$)
Metal concentration +: Significance ($p < 0.05$), ++: Insignificance ($p > 0.05$); Biomass dosage x: Significance ($p < 0.05$), xx: Insignificance ($p > 0.05$)

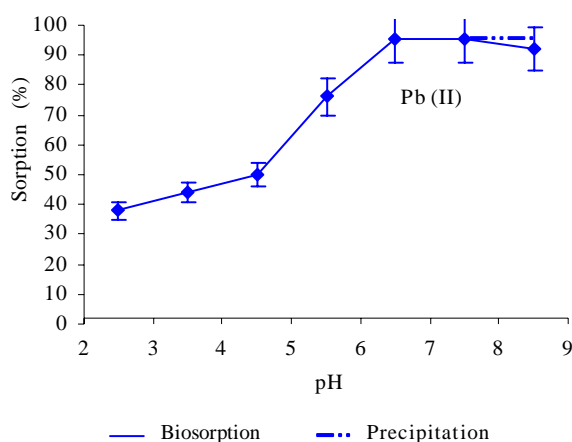


Fig. 1: Effect of pH on sorption behavior of Pb (II) on SILP. Error bars represent standard deviation of five replicates determination (Co: 25 mg/L; m: 4 g; T: 30 min.)

occurring at 6.5. There was no significant difference in sorption behavior with further increase in pH up to 7.5.

Investigation on pH variation beyond 7.5 yielded an increase in sorption up to pH 8.5, which might be due to the precipitation carry-over of Pb (II) which starts at pH 7.5 (Iqbal, *et al.*, 2002). Pb (II) precipitation interferes and is undistinguishable from sorption phenomenon at pH 7.5. Based on this experimental findings and pertinent information available on the relevant topic, an appropriate mechanism is synthesized for metal binding to the leaf powder. The aerial part of the plant *Saraca indica* possesses various organic chemical moieties and prominently large proportions of low molecular weight amino acids (Ray and Chatterjee, 1995). Amino acids have been found to constitute a physiologically active group of binding agents working even at low concentration which, because of the ability to interact with metal ions, is likely to be active sites for the sorption of metal ions (Brostlap and Schuurmans, 1998). The experimental findings show that as the pH of the solution increases from 4.5 to 7.5, lead reflect increased binding behavior to the biomass with an optimum binding at pH 6.5. This provides an insight into the mechanism of the metal binding involved with the SILP. The majority of naturally occurring amino acids have isoelectric point between pH 4.0 and 8.0 (Delvin, 2002). In this range of pH, over 90% of the amino acid molecules are in an ionized state. Biosorption of target metals may be ascribed to the availability of carboxyl ligands of amino acids for interaction with metal ions at pH 4.5 to 6.5. Pb-SILP binding thus appears to be an ion exchange

process involving electrostatic attraction between negatively charged groups of amino acids and metallic cations. Pb interaction with target leaf biomass (SILP) can be further highlighted on the basis of IR spectrum of native and exhausted SILP after metal sorption shown in Figs. 2 and 3. In this case, NH_3^+ stretching band shifts from 2428.52 and 2722.50/cm to 2652.39 and 2781.23/cm. The characteristic peak shifting of carboxylate ion from 1350.86 and 1552.14/cm to 1489.51 and 1709.32/cm confirms an ionic interaction between carboxylic groups of amino acids with the cationic metal species. The powdered SILP sieved at 105 μm had a surface area of 4.23 m^2/g . Fig. 4 represents the scanning electron micrograph of untreated SILP morphology seems to be of large spherical clusters having pore area: 6.32 μm^2 . Fig. 5 represents dense agglomerated, etched clusters type morphology having pore area: 0.42 μm^2 which is in sharp contrast to spherical type clusters appeared in untreated SILP. Observed aggregation and reduction of pore area of lead treated biomass of SILP may be ascribed due to the liquid phase concentration of metal ions at experimental pH, thus confirming the biosorption phenomenon. The sorption isotherm modeling is important in the design of biosorption systems. This indicates the capacity of the sorbent. Fig. 4 shows the Freundlich isotherm. According to the Freundlich model, the maximum biosorption capacity (K_f) and intensity (L/n) obtained for Pb (II): 0.42 and 0.03 depict the higher sorption efficiency of SILP for Pb. Similarly, the values of biosorption capacity (Q_0) and energy (b) obtained from Langmuir model for the target metal was found to be Pb (II): 3.23, 0.35 which represents their favorable sorption (Fig. 7). Moreover, high values of correlation coefficient (R^2): 0.99 indicate that the adsorption pattern for both kinds of lead followed the Freundlich and Langmuir isotherms. The linearity of Freundlich and Langmuir plots suggested the formation of homogenous monolayer of Pb (II) on the outer surface of the biosorbent. Maximum lead uptake was found to be 1.19 mg/g (Fig. 8). The sorption kinetics study has been carried out at room temperature for SILP. The adsorption of Pb (II) increased with the lapse of time. The adsorption of Pb (II) was rapid during 10-30 min. of contact time after which the rate slowed down as the equilibrium reached the 30 min. of contact time. No further increase in sorption was observed with further increase in contact time up to 40 min. The kinetic data for the adsorption of the Pb (II) was tested for the first order reaction.

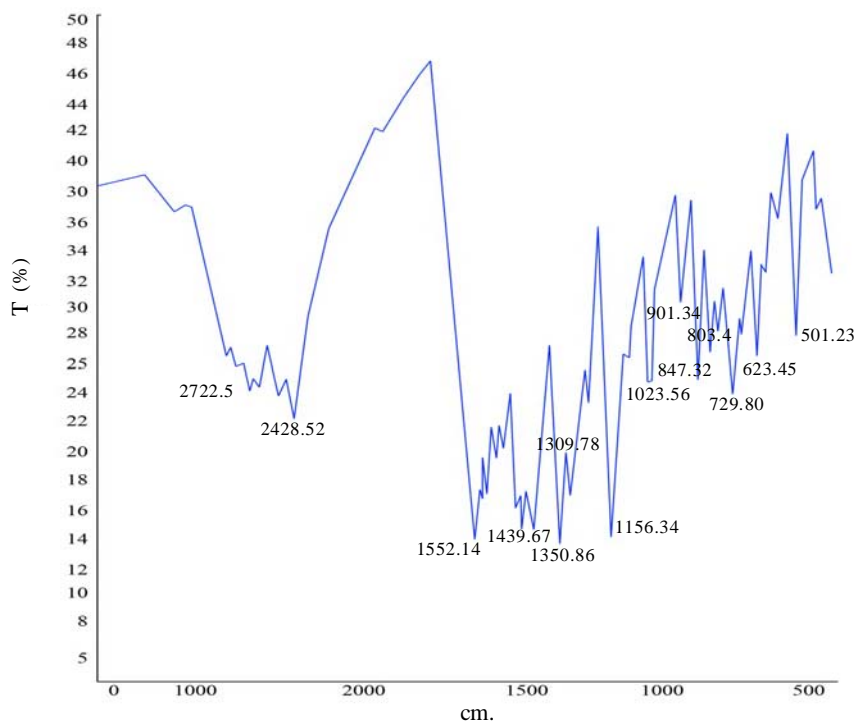


Fig. 2: Fourier transform infrared spectra of untreated SILP

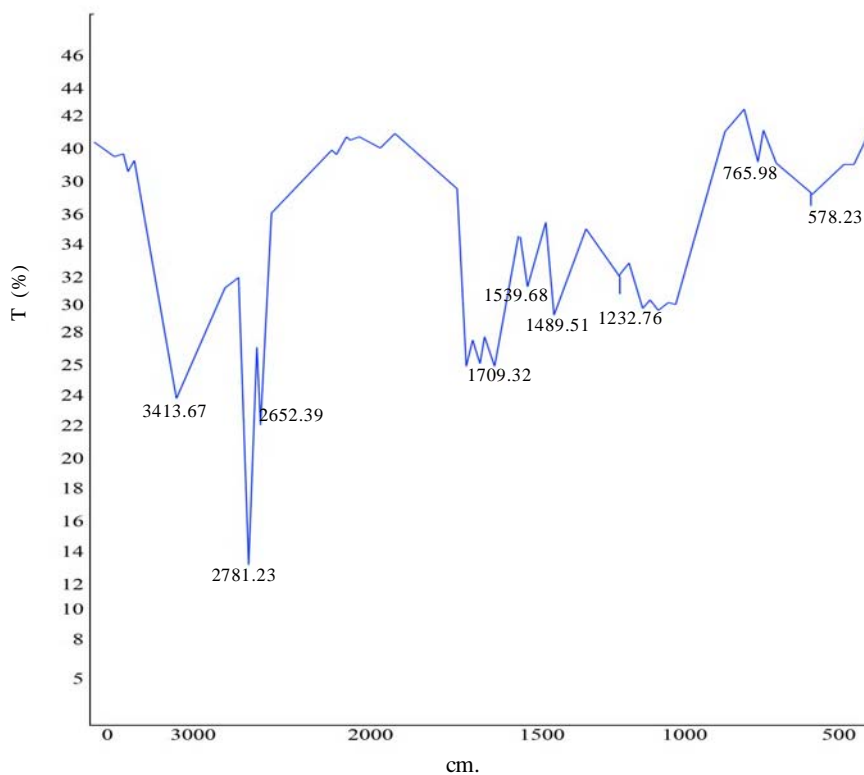


Fig. 3: Fourier transform infrared spectra of Pb (II) treated SILP

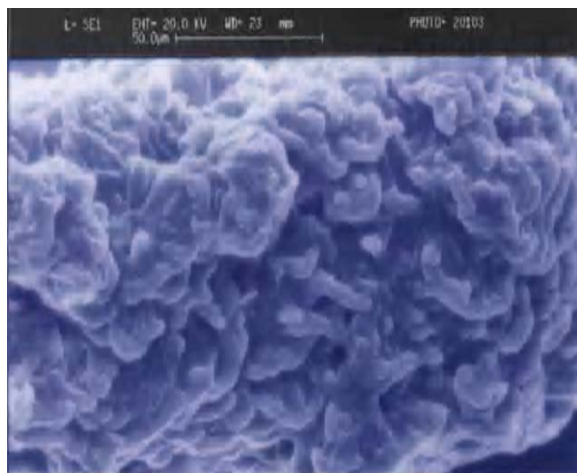


Fig. 4: Scanning electron micrograph of untreated SILP showing large spherical clusters type morphology



Fig. 5: Scanning electron micrograph of Pb (II) treated SILP showing dense agglomerated, etched cluster type morphology

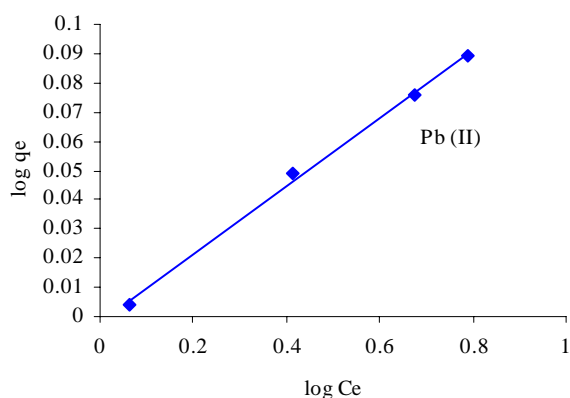


Fig. 6: Freundlich isotherm plot for the adsorption of Pb (II) on SILP

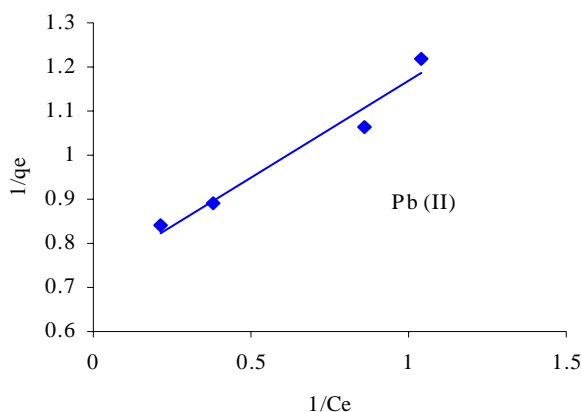


Fig. 7: Langmuir isotherm plot for the adsorption of Pb (II) on SILP

Kinetic constant (K_{ad}) was determined from the 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 Pb (II) adsorbed per unit mass of SILP at equilibrium and at time 't', respectively. $K_{ad}/\text{min.}$ was calculated from the slope of linear plot of $\log (q_e - q_t)$ versus time (Fig. 7). The value of kinetic constant for Pb(II) 0.42/min. was found to be independent of initial Pb concentration, indicating that adsorption of Pb (II) on SILP follows first order rate kinetics. Desorption studies help with elucidating the mechanism of metal ion removal and recovery from metal loaded adsorbent and also for the regeneration and recycling of spent adsorbents, which in turn may reduce operational cost. With this aim in

mind, desorption studies were carried out. Biosorption was found to be completely reversible, with two mineral acids (hydrochloric acid and nitric acid). The maximum desorption (97.23%) was observed at hydrochloric acid strength (0.05 M). However, better desorption (99.86%) could be achieved with the same strength of nitric acid as eluant. The sorption of Pb (II) on regenerated biosorbent remained constant up to three cycles (95.35%) and then started decreasing (to 87.23%) in the 4th cycle.

Experimental results have demonstrated that SILP can be utilized as cost-effective and environment-friendly techniques in the removal of Pb (II) (95.37%) from aqueous system.

The characterization of Pb uptake showed that the Pb binding is dependent on pH, initial Pb ion concentration, particle size, contact time and biomass dosage. These sets of experiments standardize the optimum conditions for the percentage removal of Pb (II) ion using SILP: biomass dosage (4.0 g), metal concentration (25 mg/L), contact time (30 min.) and particle size (105 μm) at pH 6.5. Amino acid lead interactions plays a significant role in the biosorption of Pb (II) using the target SILP. The data fitted well into both the Freundlich and Langmuir isotherms. The magnitude of K_f (0.42), $1/n$ (0.03), Q_0 (3.23) and b (0.35) shows ease of separation of heavy metal ions from metal contaminated water with high adsorption capacity. Recyclability results have shown that SILP

can be recycled and reused for a minimum of three times for Pb (II). The laboratory-based findings open up new avenues in the abatement of Pb by SILP which is infact an agricultural waste owing to heavy leaf fall in autumn and spring season. Thus, it introduces a less expensive, domestic and environment-friendly green method for the removal of Pb from water bodies. In addition, *Saraca indica* leaf biomass is more non-toxic, biodegradable and environmentally acceptable than chemical coagulants. It could be a potential challenge as primary and domestic coagulant and can be used as a pre-treatment step for the large scale removal of toxic metals from aqueous media, leading a step towards sustainable development of the environment.

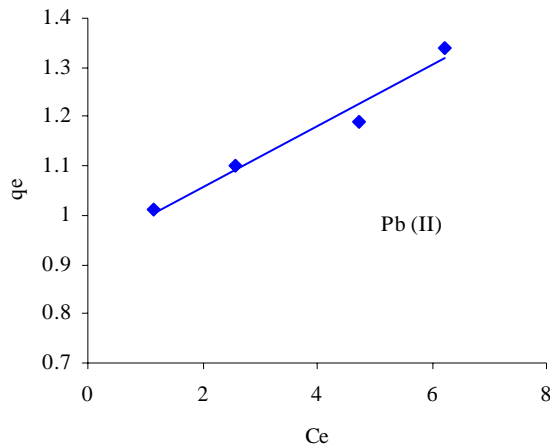


Fig. 8: Effect of lead concentration on biosorption by SILP

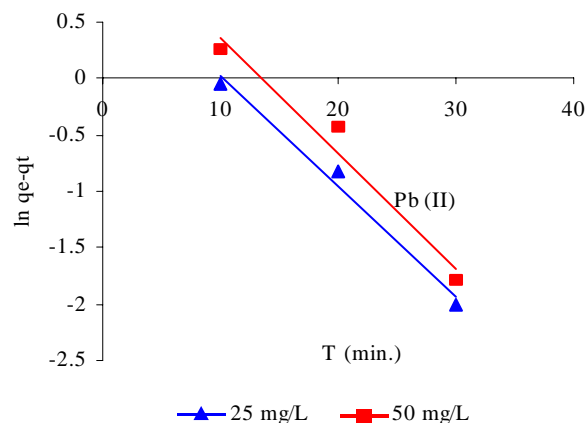


Fig. 9: Lagergren plots for adsorption of Pb (II) on SILP (Co: 25 mg/L and 50 mg/L; m: 4 g; T: 30 min.)

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