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Lantana camara shoot adsorption potential for removal of Pb (II) from aqueous solutions

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Abstract: The aim of this study was to evaluate utilization of waste weed material as *Lantana camara* Shoots (LS) as adsorbent for removal of Pb(II) from aqueous solutions. The batch and column mode studies were performed for metal sequestration. The adsorbent material was low cost and found in abundant form in studied area. In batch system, effect of pH, dose, contact time, initial metal concentration and particle sizes were studied. The monolayer adsorption capacities for Pb(II) was 2.42 mg/g at pH 5. Langmuir model fitted well with Pb(II) metal uptake. Pseudo second order kinetic models suits better than Pseudo first order in all experimental data. SEM and FTIR studies describe adsorption mechanism and support adsorption phenomenon. Column studies with real textile effluent reports more than 87% metal removal within 120 min contact time. Hence, this LS adsorbent could be attributing as efficient adsorption in its natural state.

Keywords: *Lantana camara*, Metal removal, Batch, Column, Flow rate

INTRODUCTION

Industrial effluents generated from industrial process exhibits toxic heavy metals [1]. The metals like cadmium, chromium, lead, nickel, mercury and arsenic pose threat to ecosystem and human health. Heavy metals alter the physico-chemical characteristics of water environment.. Mostly, lead (Pb), nickel (Ni) are harmful elements that can cause various health disorders in human health even with small concentration accumulation. The problem of heavy metal in industrial effluent and wastewater is worldwide phenomenon [2]. Seeing its long term environmental hazards, the removal of its excess concentration from water and wastewater has paid much attention globally [3].

The numerous methods have been exploited to remove toxic metals from water, wastewater, and industrial effluents such as membrane separation, ion exchange, adsorption, chemical precipitation, and coagulation. Among these utilized methods, adsorption is compatible and feasible technique due to its high efficiency, low cost technique and ease in operations [4]. The adsorption process is dependent on adsorbent properties which are closely related to its particle size, surface area, internal structure and functional groups present on adsorbent surface [3]. This method explores adsorbents such as charcoal, activated carbon, zeolites, clays and low cost plant or forest based adsorbents [5]. However, these low cost adsorbents have several demerits like low adsorption capacities, low surface area, lacking of adequate functional groups and surface deactivators [5]. Therefore, it is required to develop low cost, ecofriendly and efficient adsorbents from plant, industrial, agriculture and forest waste products. The untreated or raw material utilized for uptake of metals like agave bagasse [6], barley straw [7], egiptial mandarin peel [8], lentil shell [9], olive stone [10], wheat shell [9] and garden grass [11].

In present study, *Lantana camara* shoots were selected for removal of Pb(II) from aqueous solution. Authors have studied literature on removal of Pb(II) concluded that adsorption of this adsorbent has not studied yet. Present work was based on batch and column mode study for abatement of Pb(II) removal from aqueous solution. Isotherm, kinetic, SEM and FTIR studies were also carried out to confirm the applicability of this work.

MATERIAL AND METHODS

Chemical and reagent

All the chemical were analytical grade, including stock solutions of lead and nickel nitrates. The desired metal concentrations were prepared by further dilutions of stock solutions. (Chemicals supplied from Naveen Traderes, Roorkee, Uttrakhand (India)).

Stock metal solution

1.598 gm and 4.479 gm of $Pb(NO_3)$ and $NiSO_4.6H_2O$ were dissolved in 1 liter double distilled water for preparation of stock solutions of 1000 mg/L concentration.

Preparation of low cost adsorbent

Lantana camara shoots were collected from road side area of Jagjeetpur, Haridwar, India. Both materials were washed with water and double distilled water and left for open sun drying for 5-7 days. The materials were cut down in small pieces and grinded by mixer grinder (Phylips 800 W). The materials were sived and drying in oven at 105°C for 24 hours.

Characterization of adsorbent

Proximate analysis was conducted to determine pH, ash content, moisture content, bulk density, particle size and surface area. Particle size and surface area was analysed by BET analyser. The surface characterization was performed by scanning electronic microscopy (JEOL Japan-6490) and EDs was analysed by EDS instrument (EDS-133) which was attached unit with SEM. The functional groups present on adsorbent surface were performed by Fourier Transform Infrared Spectroscopy (FTIR-Nicolet 6700) in a spectral range of 400-4000 cm^{-1}.

Adsorption studies

Batch mode experiment

Batch mode experiments were conducted on fixed volume of 100 mL in Erlenmeyer flasks with containing optimum dose, pH and metal concentration. The trail experiments were conducted in batch mode for optimizing pH, contact time, dose initial metal concentration and particle size. The range of optimization was for pH (1-8), doses (0.25-2.5 g/L), initial metal concentration (0.5-4.0 mg/L), contact time (5-120 min) and particle size (0-300 µm). The material was put in flasks and then on bath shaker at 120 rpm. After equilibrium, the flasks were removed from shaker and the treated solutions were filtered by Whatman No 42.

RESULTS AND DICUSSION

Characterization of LS adsorbent

Table 1 shows characteristics of LS adsorbent. The pH value was 7.2 which shows slightly alkaline in nature. The moisture content and ash contents were 4.32 and 5.12%, respectively. The low ash and moisture content exhibits good adsorbent characteristics [12]. The BET surface area was found upto 1.254 m^2/g. The pore diameter was 1.592 nm while pore volume values was 0.003 cc/g. The elemental composition shows that carbon content was more than 65 % (Table 2).

Table 1 Physico-chemical properties of LS adsorbent [13]

pH	7.2
Moisture content (%)	4.32
Ash content (%)	5.16
BET surface area (m^2/g)	1.254
Pore diameter (nm)	1.592
Pore volume (cc/g)	0.003

Table 2 Elemental and weight percent of LS adsorbents (SEM-EDX) [13]

Lantana shoot (LS)		
Element	**Weight %**	**Atomic %**
C	65.29	71.29
O	33.35	27.59
Ca	1.37	0.45

Effect of pH

pH affects the adsorption process in aqueous solution. The pH ranges from 1-8 in this study. Fig. 1 shows the behavior of pH for the uptake of Pb(II) from aqueous media. The major pH behavior was observed between 3-6. Below pH 4, lower adsorption was observed due to protonation, hydrogen ion compete with binding site on adsorption surface. Under alkaline conditions or pH >7, $Pb(OH)_2$ metal causes precipitation in aqueous solution. The maximum removal of Pb(II) was achieved on. Literature data also says that most of the metal ion removal occurred between 3.5-6.0 [14].

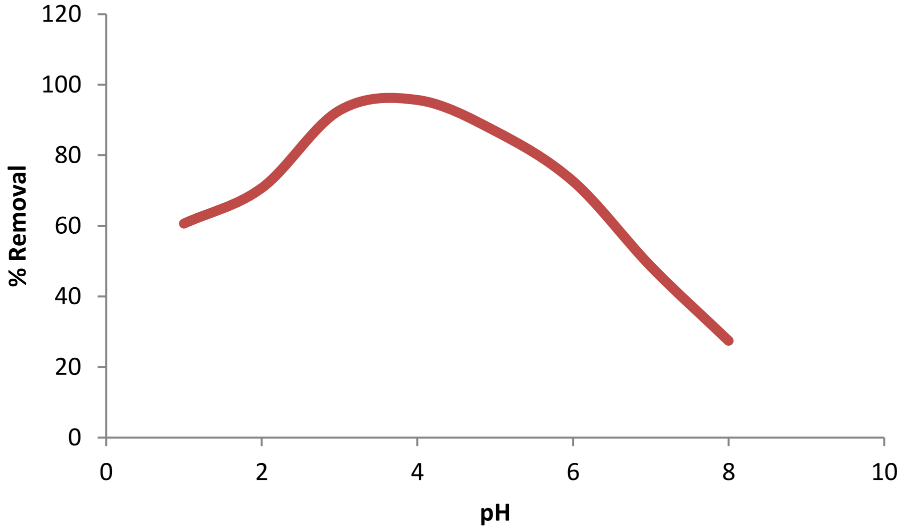

Fig. 1 Effect of pH on Pb(II) removal by LS adsorbent (dose ,2g/L; metal concentration, 2 mg/L, contact time, 120 min, 100 mL volume of solution)

Effect of contact time

In adsorption process, contact time provides reaction time needed for metal for metal binding of the adsorption system, the optimum contact time required ranged between 60-100 min [15]. In present study, the optimum contact time was 60 min from both Pb(II) uptake (Fig.2). After 60 min contact time, no further removal was occurred. This was due to unavailable of active sites on adsorbent surface. The rate of metal adsorption was high in the first 20 mins because of layer surface area of adsorbent available for metal uptake in aqueous solution [16].

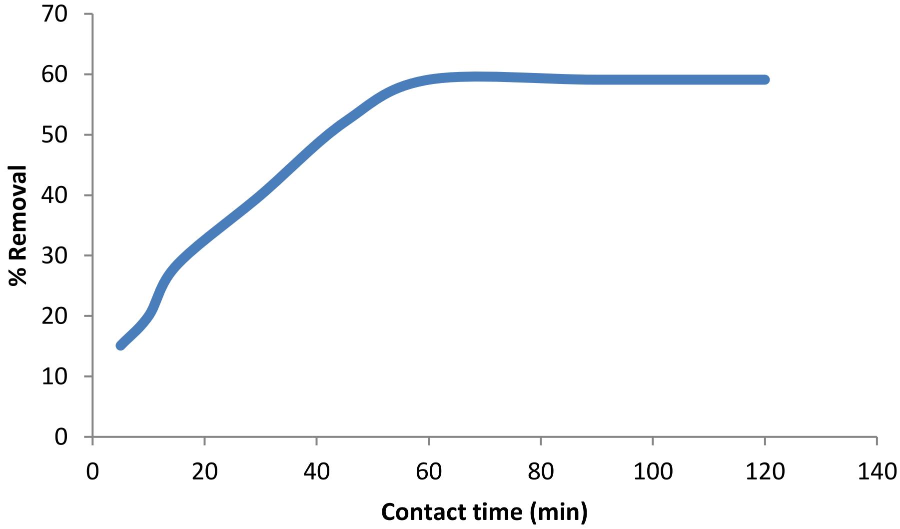

Fig. 2 Effect of contact time on Pb(II) removal by LS adsorbent (dose ,2g/L; metal concentration, 2 mg/L, pH 4, 100 mL volume of solution)

Effect of dose

Adsorbent dose provides that active binding sites for metal uptake in aqueous solution. The quantity or its concentration highly affects the reaction rate and percent removal of metal. The maximum removal for Pb(II) was 86.32% and at 2.5 g/L (Fig. 3). It was found by many researchers that increase in adsorbent dose increases the binding site available on adsorbent for metal uptake [17]. The competition for available active sites between metal ions decrease with increase in the dosages of adsorbent [15].

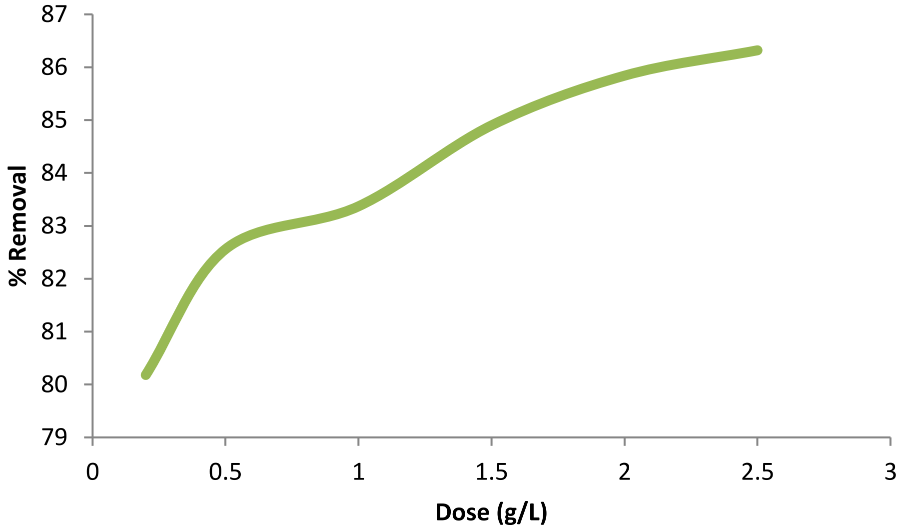

Fig. 3 Effect of adsorbent dose on Pb(II) removal by LS adsorbent pH 4 metal concentration, 2 mg/L, contact time, 120 min; 100 mL volume of solution

Effect of initial metal concentration

The effect of initial metal concentration was studied between metal range from 0.5-4.0 mg/L. Fig. 4, shows the Pb(II) metal ion effects on different contact time. Experimental results show that LS adsorbent upto 45% and 70% at 2 mg/L and 1 mg/L, respectively. As concentration increased, the adsorption removal increased (Fig. 4). At, increase in Pb(II) concentrations results in a decrease in the initial rate of external diffuse and increase in the intraparticle diffuse rate [18].

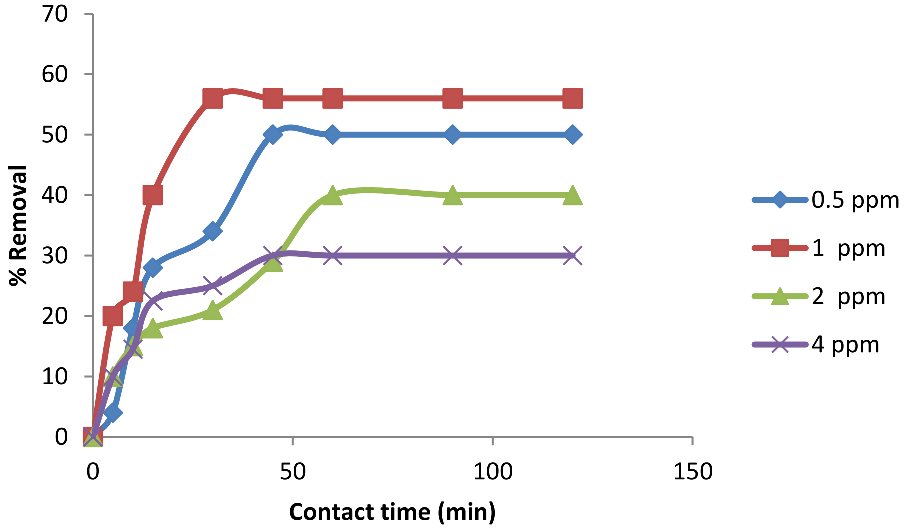

Fig. 4 Effect of Pb(II) metal ion concentration adsorption by LS adsorbent, pH 4, contact time, 120 min, 100 mL volume of solution

Effect of particle size

To investigate the particle size on the adsorption of Pb(II) and Ni(II), 0-75,75-150,150-200,200-250 and 250-300 µm mesh sizes were selected at optimum adsorption conditions. As shown in Fig 1, the adsorption process was increase as decreased in particle size. Decrease in particle size with adsorbent dose can be attributed to higher surface area and active sites on adsorption surface [19]. Adsorption percent was increase from 51.04-71.07% for Pb(II) as the particle size decreased (Fig. 5).

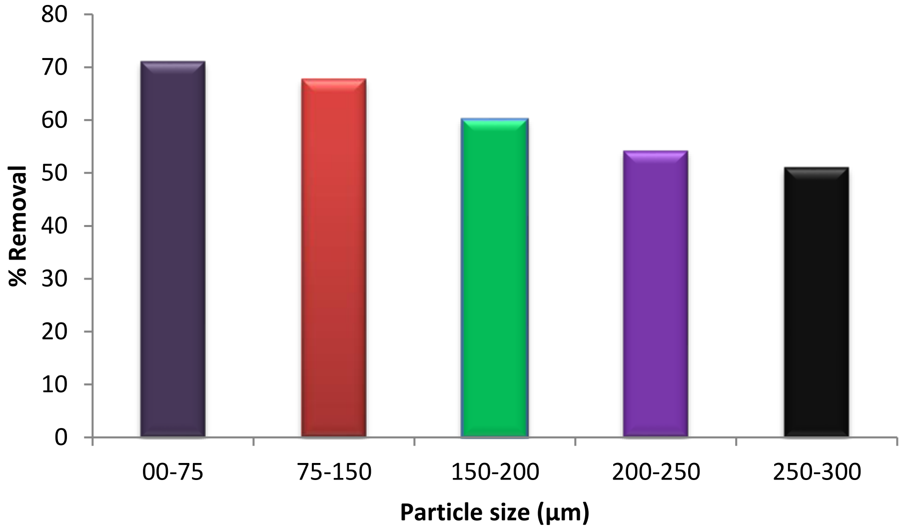

Fig. 5 Effect of particle size of adsorbent on Pb(II) removal by LS adsorbent pH 4, metal concentration, 2 mg/L, contact time, 120 min, 100 mL volume of solution

Adsorption isotherm

The adsorption isotherm reveals the equilibrium conditions occurred in aqueous solution between adsorbent and adsorbate [20]. Mostly, Langmuir and Freundlich isotherm models were used due to these models variables suited with adsorption equilibrium data. Linear regression (R^2) model was used to find out the suitability with experimental data.

3.7.1 Langmuir isotherm

The Langmuir isotherm is applied on the basis of formation of saturated monolayer on the adsorbent surface [20]. The adsorption process takes place with same binding energy between adsorbent and adsorbate. The adsorbed metal ions on the surface of adsorbent are not moved or no migration.

The linear form of Langmuir isotherm equation is represented by following equation [21] :

$$\frac{1}{q_e} = \frac{1}{Q^o} + \frac{1}{bQ^oC_e}$$

Where q_e is the amount adsorbed at equilibrium time (mg/g), C_e is the equilibrium concentration of the adsorbate ions (mg/l), and Q^o and b are Langmuir constants related to maximum adsorption capacity (monolayer capacity) and energy of adsorption, respectively.

The equilibrium data were fitted with metal uptakes by adsorbents (Table 3). A plot of 1/Qe vs 1/Ce produced straight line (Fig. 6). The separation factor or dimensionless parameter (R_L) was assessed to interpret adsorption condition from the Langmuir isotherm. The separation factor (R_L) was defined by the following expression:

$$R_L = \frac{1}{(1 + bC_i)}$$

Where, C_i is the initial metal concentration of metal ion (mg/L) and b is the Langmuir adsorption isotherm constant (L/mg). For a favourable adsorption process the R_L values lies between 0 and 1 (Table 4) [22].

3.7.2 Freundlich isotherm

The Freundlich isotherm model fitness represents the heterogeneous surface adsorbents. The model has significant even at low metal concentrations and also showed the ratio of metal adsorbed to the metal concentration as a function of metal solution concentration [23].

The Freundlich equation has the general form [24]:

$$q_e = K_F C^{1/n}$$

The Freundlich equation is basically empirical but is often useful as means for data description. Data are usually fitted to the logarithmic form of the equation:

$$\log q_e = \log K_F + \frac{1}{n} log C_e$$

Where q_e is the amount adsorbed (mg/g), C_e is the equilibrium concentrations of the adsorbate ions (mg/l), and K_F and n are Freundlich constants related to adsorption capacity and adsorption intensity respectively. The values of R^2 (>0.84) show that the experimental data fit to the Freundlich isotherm (Fig.7).

It was depicted from correlation coefficients, Langmuir isotherm fitted well than Freundlich isotherm (Table 3)

Table 3 Adsorption isotherm data for adsorption of metals by LS

Metal	**Langmuir constants**			**Freundlich constants**		
	Qe (mg/g)	b	R^2	K_F	n	R^2
Pb(II)	2.42	0.49	0.94	3.93	1.68	0.91

Table 4 R_L values for adsorption of Pb(II) and Ni(II) by LS adsorbent

Metal	**C_i (mg/L)**	**R_L**
Pb(II)	0.5	0.8013
	1.0	0.6685
	2.0	0.5021
	4.0	0.3352

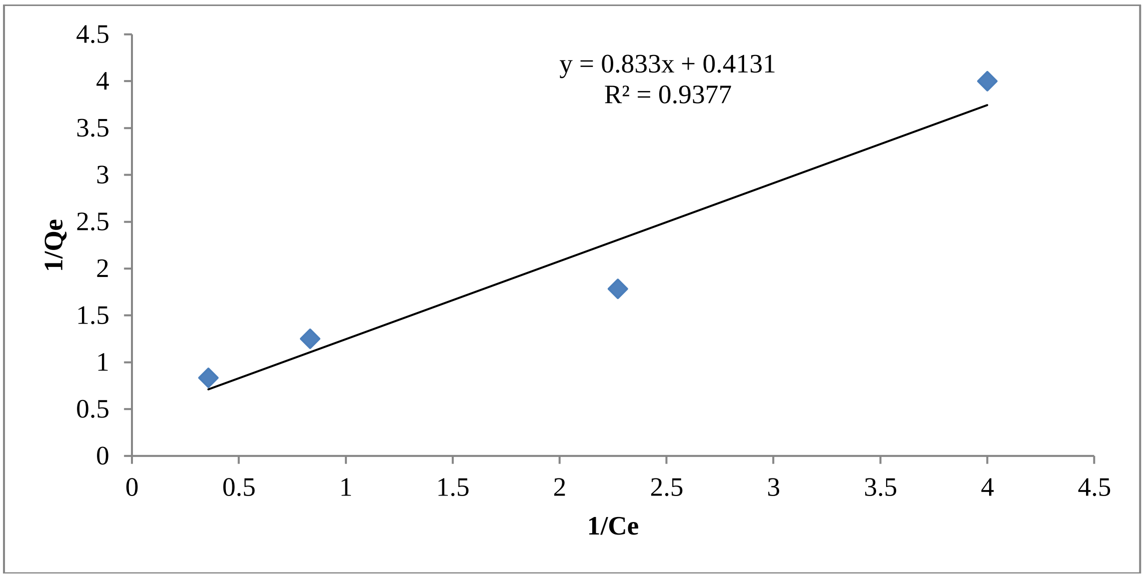

Fig. 6 Langmuir isotherm plot for lead ion adsorption onto LS

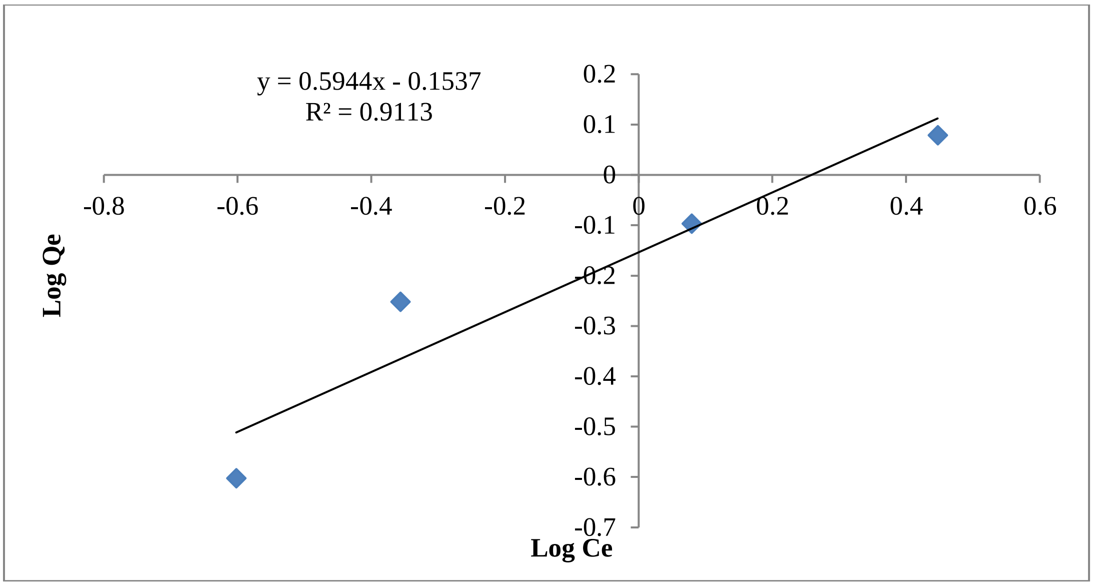

Fig. 7 Freundlich isotherm plot for lead ion adsorption onto LS

Table 5 Kinetic parameter for adsorption of Pb(II) onto LS at different concentration

		Pseudo first order constants				Pseudo second order constants				
Metal	**Conc.**	**Equation**	**K_1(/min)**	**Qe cal (mg/g)**	**R^2**	**Equation**	**K_2**	**qe (cal)**	**qe (exp)**	**R^2**
Pb(II)	0.5 mg/L	Y=0.0121-0.7272	0.02786	0.183	0.89	Y=0.0121-0.7272	0.0278	0.183	0.89	0.91
Pb(II)	1.0mg/L	Y=0.0197-0.3681	-0.04537	0.428	0.85	Y=0.0197-0.3681	-0.0453	0.428	0.85	0.97
Pb(II)	2.0 mg/L	Y=0.0178-0.2766	0.040993	1.890	0.73	Y=0.0178-0.2766	0.0409	1.890	0.73	0.83
Pb(II)	4.0 mg/L	Y=0.0344-0.0189	0.079223	0.957	0.92	Y=0.0344-0.0189	0.0792	0.957	0.92	0.99

Adsorption kinetics

Adsorption kinetics is usually proceeded out to predict rate of reaction and dynamics in adsorption process [25]. The linearised kinetic models like Pseudo first order and pseudo second order model were mainly used. The plots of Pseudo first order and Pseudo second order kinetic model is given in Fig. 8 & 9 and Table 5.

Pseudo first order

Pseudo first order kinetic model implies on the adsorption of a solute from an aqueous solution. Pseudo first order depends on the concentration of the one reaction [26].

A simple kinetic model that applicable in the process of adsorption is the pseudo-first order. The Pseudo first order kinetic equation expressed as:

$\mathrm{Log}(q_e\text{-}q_t)=\log q_e\text{-}\frac{k1}{2.303}t$ (linear form)

$q_t=q_e(1\text{-}e_1^{-kt})$ (non-linear form)

where $k_1(h^{-1})$ is the first order rate constant of adsorption, q_e is the amount adsorbed at equilibrium and q_t is the amount of metal adsorbed at time t.

Pseudo second order

The Pseudo second order kinetic model implies on the concentration of more than one reactant [27].

The pseudo-second order equation is expressed as

$$\frac{t}{q_t}=\frac{1}{k_2 q_e^2}+\frac{t}{q_e}$$

Where q_e and q_t are amount of metal ion adsorbed (mg/g) at equilibrium and time at time‘t’ respectively. The product $k_2q_e^2$,the initial sorption rate, represented as $h=k_2q_e^2$.

The plot of R^2 values as given in Table 5, indicates the better applicability of Pseudo second order model as compare to Pseudo first order kinetic model.

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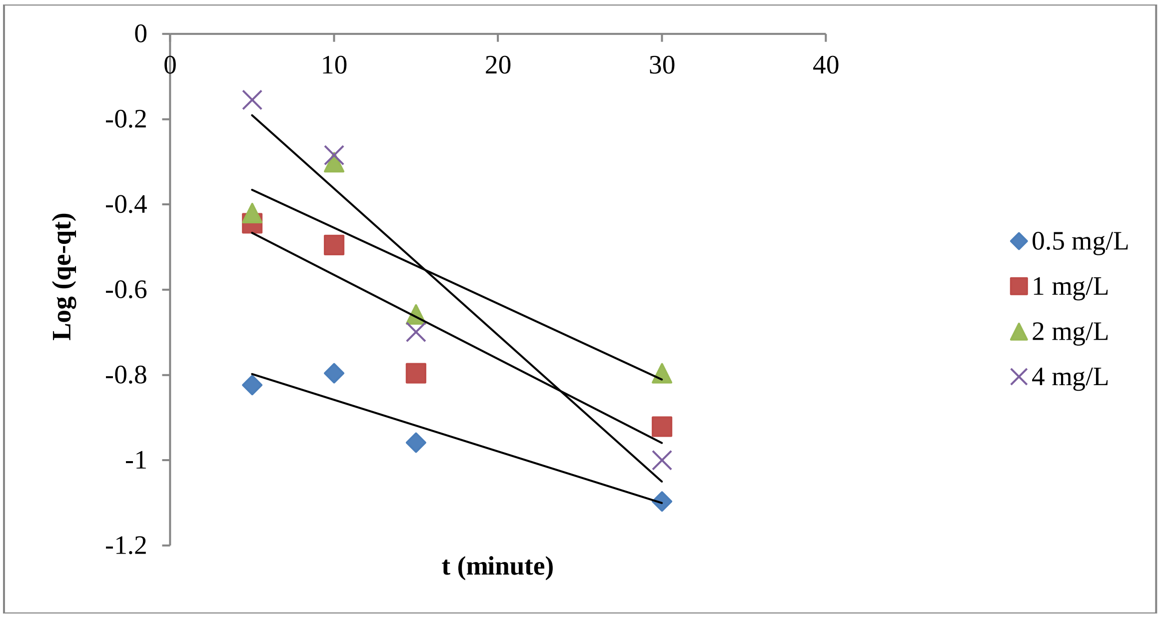

Fig. 8 Pseudo first order (PFO) kinetic model for lead adsorption onto LS

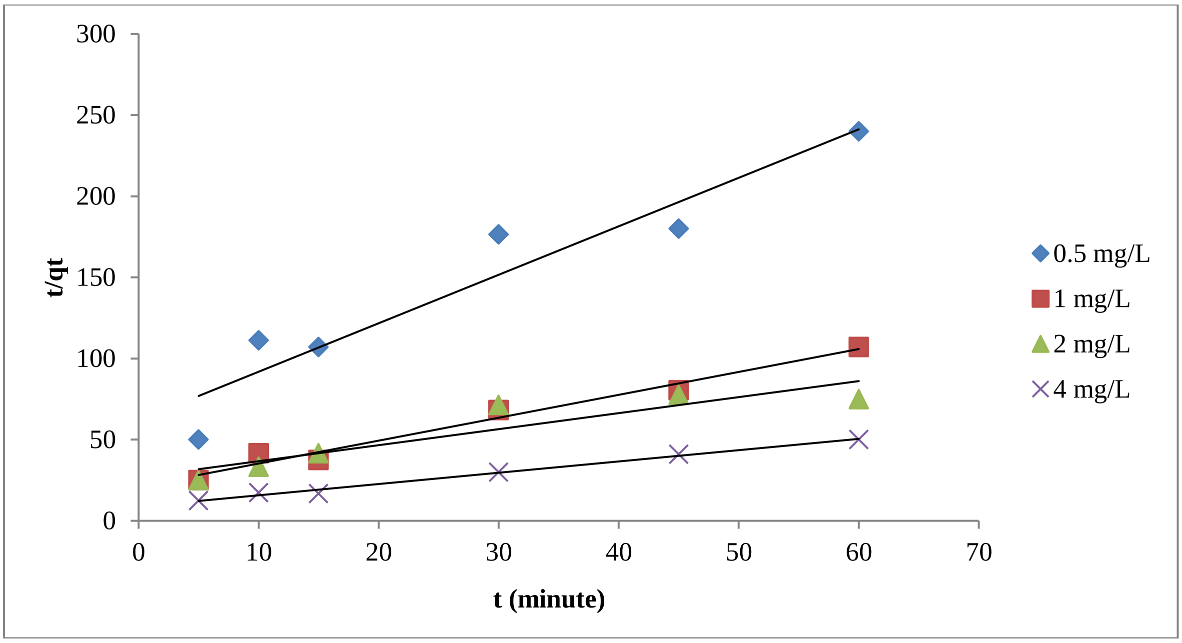

Fig. 9 Pseudo second order (PSO) kinetic model for lead adsorption onto LS

Column studies

Fixed bed column study was conducted using glass column of 55 cm height and 2.5 cm internal diameter. The schematic diagram of fixed bed column is illustrated in Fig. 10. The flow rates of 2.5 and 5.0 mL/min were used in this study which was controlled by manually through separating funnel knob. The real textile industrial effluent collected from industrial area, Sec-25 Panipat, Haryana (India) was passes through downward mode in glass column with flow rates of 2.5 and 5.0 mL/min at different time intervals (0-300 min). The percent removal of Pb(II) and was given in Fig 15. The graph of Pb(II) removal percent was higher at lower flow rate. The metal removal percent of Pb(II) was 87.5 and 75% at 2.5 and 5.0 mL/min flow rates, respectively (Fig. 11). It was due to increase in residence time of metal in fixed bed column. Therefore, there is adequate time for adsorption equilibrium to be achieved [28].

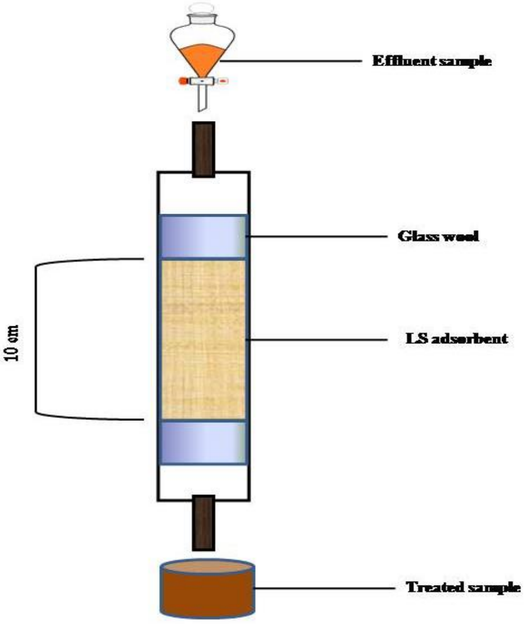

Fig. 10 Experimental setup for column study

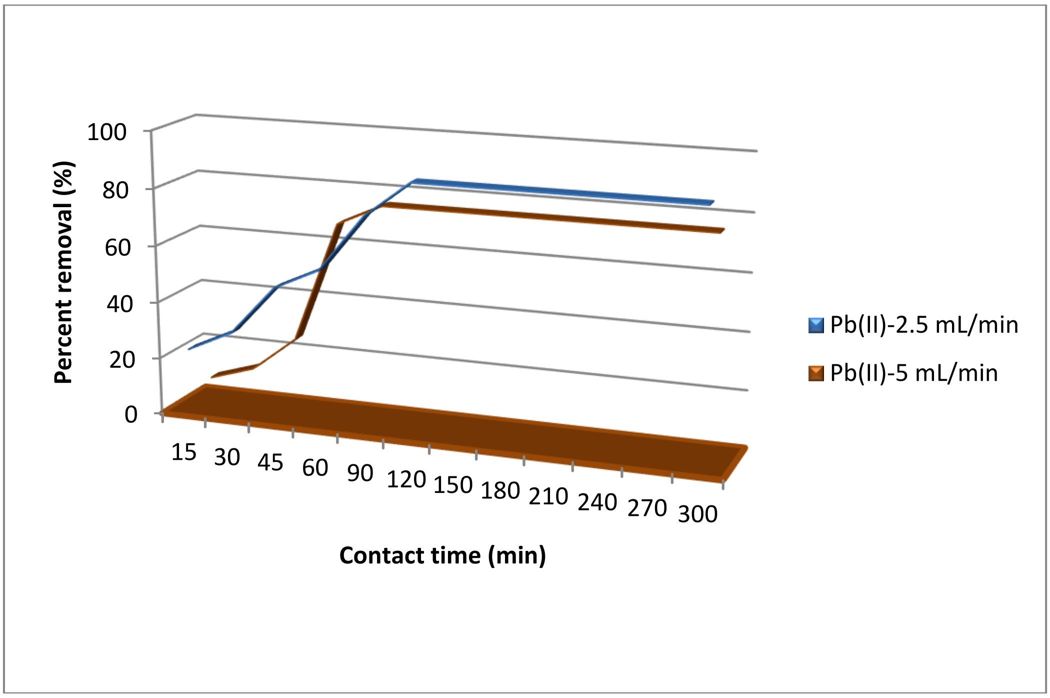

Fig.11 Effect of effluent fixed flow rates on Pb(II) metal ions removal onto LS at different flow rate

Adsorption mechanism

The adsorption mechanism includes external diffusion and intraparticle diffusion. The surface functional groups like carbonyl, hydroxyl, amine, amide, phenolic and carboxylic acids are present of surface of adsorbent and pores (mainly mesopore and micropore). Adsopriton mechanism of Pb(II) was schematic shown in Fig. 16. The adsorption of metal ion onto LS adsorbent occurred due to electrostatic interaction between negative charged functional groups ($-COO^-$, $C=O^-$ and $-OH^-$ etc.) and positively charged metal ions (Pb^{+2}). Co-precipitation of metal ions and inner and outer structure of adsorbent also affect binding affinity of metal ions [29].

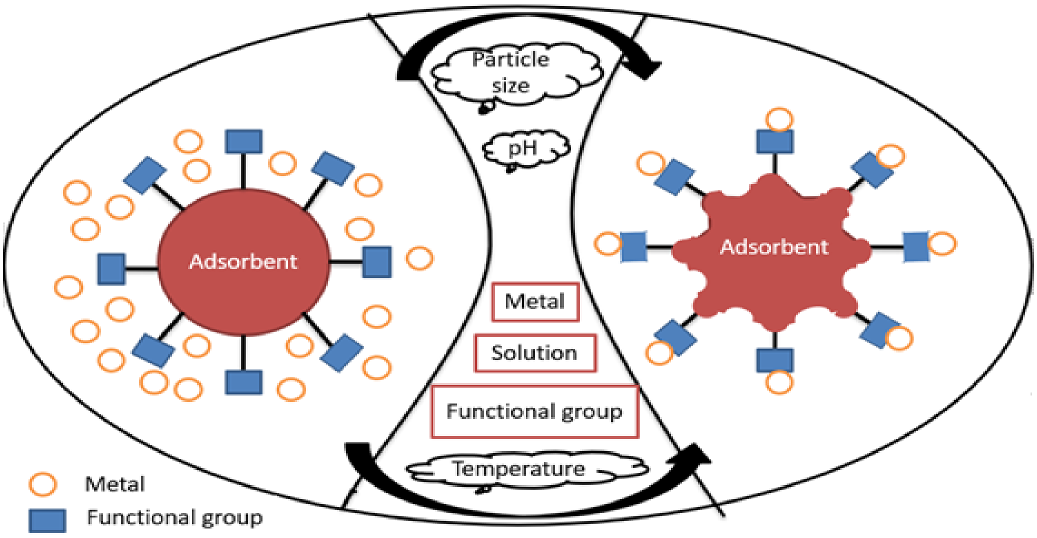

Fig 12. Adsorption mechanism of metal uptake in aqueous environment

External diffusion

External diffusion takes place from boundary film to adsorbent surface in aqueous phase [30].The metal species are bounded with adsorbent surface through mass transfer phenomenon. External diffusion occurred until vacant sites for metal uptake. The process of external diffusion is slow and takes time to achieved equilibrium conditions.

Intraparticle diffusion

Intraparcticle diffusion is phenomenon of movement of metal species from solution to the intraparticle space and pores [31]. Intraparticle diffusion model was represented by Weber-Morris model.

SEM studies

The SEM micrographs of LS adsorbent were taken before and after adsorption at magnification of x5000. The Fig.17 shows the raw, Pb(II) and Ni(II) loaded micrographs of LS adsorbent. The uneven and irregular surfaces are found on LS adsorbent surface and these structures are might be support metal binding. EDS study shows the carbon content more than 65 percent as elemental composition (Table 2).

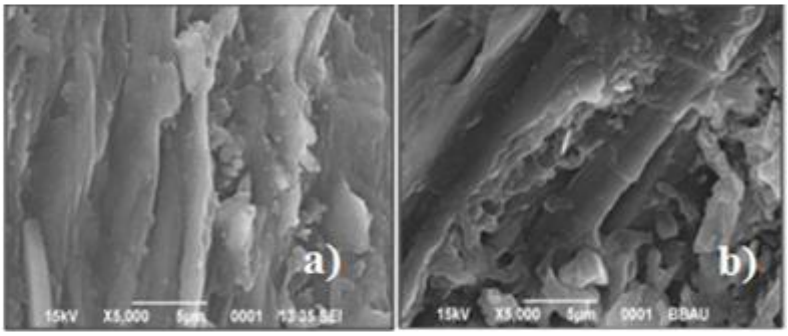

Fig. 13 SEM images of a) raw LS, b) Pb(II) treated

FTIR studies

The FTIR spectra of LS are shown at 400-4000 cm^{-1} before and after metal adsorption (Fig.14 and Table 6). The bands at 3571.4 cm^{-1} shifted towards 3580.2 cm^{-1} which are attributed –OH groups. The wavenumber shifted from 3571.4-3580.2 cm^{-1} after loading of metal ions. This finding suggests that the –OH group played major role in Pb(II) adsorption. The wavenumber of 2941.9 cm^{-1} decreased after Pb(II) adsorption upto 2833.7 cm^{-1} due to –CH stretching. The wave number of 1734.4 cm^{-1} slightly increase after Pb(II) uptake which corresponds to C=O group Table 7). The wavenumber was shifted from 1428.7-1370.0 cm^{-1} after metal adsorption (-CO and carboxylic acid stretch). The wavenumber of 1161.0 cm^{-1}(CN, amine) was shifted towards 1061.0 & 1161.1 cm^{-1} which confirms –CN (amine stretching).

Table 6 The FTIR spectral characteristics of raw LS before and after biosorption of Pb(II)

Before loading of metal (wavenumber, cm^{-1})	After loading of metal Pb(II) (wavenumber, cm^{-1})	Assignments
3571.4	3580.2	-OH, hydroxyl
3289.6	3276.5	-OH, hydroxyl
2941.9	2942.8	-CH stretching
2884.8	2883.0	-CH stretching
2838.8	2833.7	-CH stretching
1734.4	1736.7	C=O, carbonyl group
1654.4	1658.1	-COO, carboxylic group
1507.4	1506.8	-COO, carboxylic group
1428.7	1428.9	-CO stretch
1371.5	1370.8	-CO stretch
1326.2	1325.7	-CO stretch
1161.0	1161.1	-CN (Amine)
1110.3	1111.7	-CN (Amine)
1057.3	1057.5	-CN (Amine)

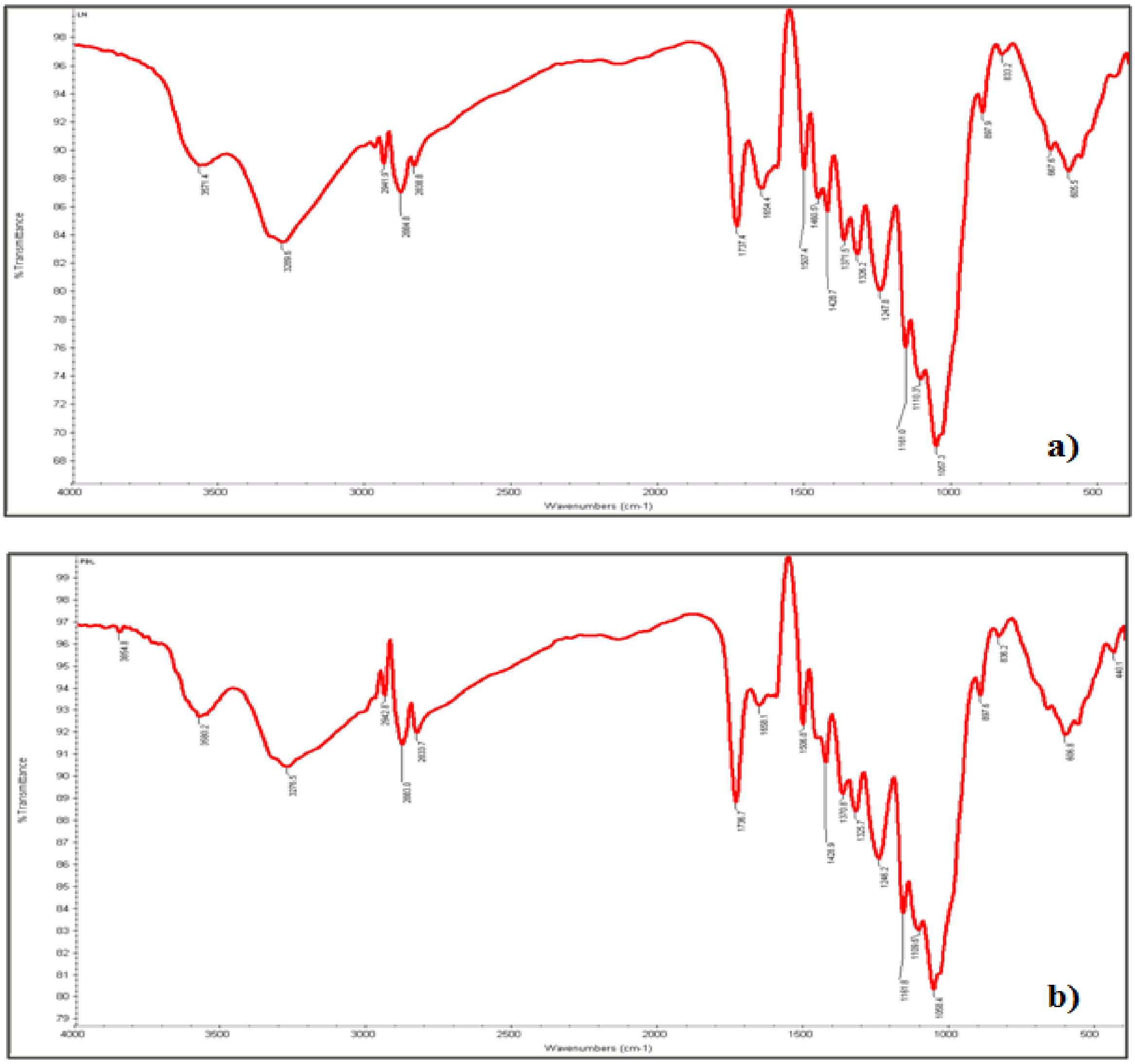

Fig. 14 FTIR spectra of a) raw LS b) Pb(II) loaded

Comparison of metal removal efficiency

A comparison of metal removal efficiencies by low cost adsorbents for Pb(II) and Ni(II) removal were shown in Table 7. The metal removal efficiency of Lantana shoots were found comparable with other adsorbents.

Table 7 Comparison of metal removal efficiencies by unmodified low cost adsorbents for Pb(II) Uptake

Adsorbent	Metal	pH	Adsorption capacity (mg/g)	Reference
Acacia nilotica	Pb(II)	4	2.51	[32]
Bagasse	Pb(II)	6	2.50	[33]
Coir	Pb(II)	4.9	0.12	[34]
Seed hull of palm tree	Pb(II)	**4**	3.77	[35]
Seeweed brown	Pb(II)	4	1.35	[36]
Lantana camara shoots	Pb(II)	4	2.42	Present study

CONCLUSIONS

Lantana camara shoots were used to abate Pb(II) metal ions from aqueous solutions. LS adsorbent showed an efficient adsorption efficiencies 2.42 mg/g at pH 4 for Pb(II) uptake. Adsorption isotherm followed Langmuir for Pb(II) adsorption. Pseudo second order model fitted better than Pseudo first order kinetic model in whole experimental data. SEM images showed the formation of pores and grooves due to increase in effective diffusion area and hydrophobic nature of metal ion surface. Surface functional groups like hydroxyl, carboxylic and amine play important role in adsorption mechanism.

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