**Research Article**

# REMOVAL OF LEAD (II) IONS FROM SYNTHETIC WASTEWATER USING LANTANA CAMARA LEAVES BIOCARBON

## Starlin Shyla Sathianesan[1]., Mohan Kumar Rajayyan[2] and Singanan Malairajan[3]*

[1]Department of Physical Science, Our Lady College of Education, Maduravoyal, Chennai-600 095, Tamil Nadu, India
[2]Department of Chemistry, Rajeswari Vedachalam Government Arts College, Chengalpattu-603 001, Tamil Nadu, India
[3]Department of Chemistry, Presidency College (Autonomous), Chennai-600 005, Tamil Nadu, India

## ARTICLE INFO

## ABSTRACT

Lead is a very toxic element known to cause detrimental effects to human health even at very low concentrations. So it is a necessity to remove these heavy metals from wastewater in order to prevent contamination of natural water bodies by effluents containing toxic metals. Removal of lead from aqueous solution was studied using a low-cost adsorbent material prepared from the leaves of Lantana camara plant. It is known as Lantana camara leaves biocarbon (LCLBC) Batch adsorption experiments were conducted to examine the effect of contact time, pH, initial metal ion concentration and adsorbent dosage at room temperature. The influence of pH of the metal ion solutions on the uptake levels of the metal ions by the adsorbent was carried out between pH 3 - 8. The optimum pH required for maximum adsorption of lead was found to be 6. An equilibrium time of 150 min. was required for the adsorption of Pb (II) ions onto the adsorbent. The percentage removal of lead onto the biocarbon was 95.50 %. Adsorption parameters were determined using both Langmuir and Freundlich isotherms, but the experimental data were well fitted to the Langmuir equation than to Freundlich equation, indicating monolayer coverage by the Pb (II) ions. From biosorption kinetics, it was observed that the pseudo-second-order model is well fitted for Pb (II) ions. The correlation coefficient $(r^2)$ for the second-order model is 0.9805. This study also showed that LCLBC can be efficiently used as low-cost alternative adsorbent for the removal of metal ions.

## INTRODUCTION

Heavy metal pollution has been one of the most challenging environmental problems due to its toxicity, persistence and bioaccumulation tendencies (Mwangi *et al*. 2012; Mihajlovic *et al*. 2015). Most of the industries produce and discharge metal-containing wastes mostly into water bodies, which affect the aesthetic quality of the water and also increase the concentrations of metals present in it (Yu *et al*. 2014; Hernandez-Montoya *et al*. 2013). As a result of rapid industrialization, toxic metals and metalloids such as cadmium, lead, chromium, mercury, arsenic and copper are released into the environment resulting to damage in ecosystem and human health. Unlike organic pollutants, heavy metals are non-biodegradable in the environment and can accumulate in living tissues particularly in human bodies causing significant physiological disorders such as damage of central nervous system, blood composition and irreversible damage of vital organs of body.

*Corresponding author:* **Singanan Malairajan**
Department of Chemistry, Presidency College (Autonomous), Chennai-600 005, Tamil Nadu, India

Hence the presence of heavy metals in natural or industrial wastewater is a subject of great interest in environmental science which is one of the most serious worldwide environmental problems (Axtell *et al*. 2003; Amarasinghe and Williams 2007).

Various sources of heavy metals in water are the effluents from battery manufacturing, basic steel, paper, pulp, metal plating, leather tanning, agrochemicals, petrochemicals, chemical manufacturing, mining and fertilizer industries (Momcilovic *et al*. 2011). Among the heavy metals, lead is one of the most toxic elements even at low concentrations. Lead is a heavy, soft, malleable and bluish grey metal. Most of the lead in the air comes as aerosols, fumes and sprays. It is widely used in storage batteries. Motor vehicle exhaust is the major source of lead pollution in the urban area. Other anthropogenic sources of lead include the combustion of coal, processing and manufacturing of lead products and lead additives. Lead particles are also introduced into the atmosphere during incineration of residues of lead containing pesticides. Lead is a systemic poison causing anemia, kidney malfunction, brain tissue damage and even death in extreme poisoning (Acharya *et al*. 2009; Ho and McKay 2000). The removal of heavy metal contaminants from aqueous solution is one of the most

important environmental concerns because metals are bio refractory and are toxic to many life forms.

Because of the above factors, there is a necessity to remove these metals from wastewater in order to prevent the contamination of natural water bodies by effluents containing toxic metals. The common methods for removing metal ions from water and wastewater include chemical precipitation, oxidation, reduction, reverse osmosis, membrane filtration and ion exchange. The major disadvantages of these methods lie in the cost involved, the efficiency of the processes and disposal of wastes generated (Sreejalekshmi *et al.* 2009).

Among the above methods, the promising process for the removal of metal ions from water and wastewater is adsorption, because the employed adsorbent can be regenerated by suitable desorption process and it is highly effective and economical. Adsorption using naturally available materials has been reported to be efficient in the removal of hazardous metals from industrial effluents. The materials employed in this technique often range from the use of microbes and naturally abundant plant materials to dead waste biomass (Wang 2009; Das *et al.* 2008).

Several adsorbents from plant origin have been used and modified for heavy metal removal from wastewater and aqueous solution which include: maize tassels (Gavrilescu 2004) , watermelon shell (Banerjee *et al.*2012), coffee beans (Katsuya *et al.* 2007), coconut shell (Amuda *et al.* 2007), peanut shell (Witek-Krowiak *et al.* 2011), Annona squamosa shell (Isaac and Sivakumar 2013), rice husks (Ghorbani *et al.* 2012), rice bran (Fatima *et al.* 2013), orange peels (Feng *et al.* 2011), sunflower stem(Das *et al.* 2008), groundnut shells (Malik *et al.* 2006) and avocado seed (Elizalde-Gonzalez *et al.* 2007). The use of mucilage extracted from plant materials and parts of mucilaginous plants for heavy metals remediation has been recently reported (Fox *et al.* 2012; Gupte *et al.* 2012; Maji *et al.*2014).

The sorption capacity of some biosorbent is high due to the presence of adequate functional groups that sequester metals from aqueous solutions (Moyo and Chikazaza 2013). The use of this technique is cheap, eco-friendly and naturally available. These bio-based materials have shown the tendency to remove metals at trace levels, thus overcoming some of the major shortcomings of the conventional methods (Olorundare *et al.* 2012). In this article, "*Lantana camara* leaves," which is a medicinal plant material was selected for the production of biocarbon as adsorbent for the removal of heavy metals from aqueous solutions.

## MATERIALS AND METHODS

### Preparation of Metal ion Solution

The aqueous solution of metal ion used in this present investigation was prepared by using analytical grade chemicals. The stock solution of Pb (II) ions of 1000 mg/L concentration was prepared by dissolving 1.60 g $Pb(NO_3)_2$ in 1L deionized water. The stock solution was used to prepare a series of dilute solutions. The pH of the solutions in the range of 3 - 8 was adjusted by adding 0.1 N HCl and 0.1N NaOH. The pH value of resulting solutions was measured by a pH meter. The Pb (II) ions concentration in aqueous solutions was determined by Atomic Absorption Spectrophotometer using a calibration curve prepared with standard metal ion solutions.

### Preparation of Biocarbon

Lantana camara leaves were collected and washed several times with distilled water to remove adhered impurities from its surface. The leaves were air dried for 48 hours. The dried leaves were grounded in ball mills and the screened homogeneous powder was used for the preparation of biocarbon. Activated biocarbon was prepared by treating the powdered leaves with concentrated sulphuric acid (SG 1.84) in a weight ratio of 1:1.8 (biomaterial: acid). The resulting black product was kept in an oven maintained at 150 ± 2°C for 6 hours. The obtained carbon was washed with distilled water and then dried in oven at 120 ± 2°C for 4 - 6 hours. The particle size of activated carbon between 90 and 125 μm was used. The resulting black mass is known as lantana camara leaves biocarbon (LCLBC) and preserved for further use.

### Biosorption Process

Batch adsorption experiments were carried out to assess the effect of various adsorption parameters such as pH, contact time, adsorbent dosage and initial metal ion concentration of $Pb^{2+}$ ion by using LCLBCas an adsorbent. The experiments were conducted in Erlenmeyer flask containing adsorbent (activated carbon from lantana camara). In each experimental flask 50 ml of 100 mg/L lead ion solution was taken and the flasks were agitated in an orbital shaker at 250 rpm and 28 ± 2°C until the equilibrium is reached. After decantation and filtration through Whatman 42 filter paper, the equilibrium concentrations of metal ion in the solution were measured using Atomic Absorption Spectrometer. The effects of concentration of metal ions (10 - 100 mg/L), contact time (30 - 210 min), solution pH (3 - 8) and adsorption dose (1 - 3.5 g) were studied. Blank solutions were treated similarly (without adsorbent) and the recorded concentration at the end of each operation was taken as the initial one. Experiments were conducted in duplicate and mean values were used.

The percentage removal of heavy metal was calculated using the following equation (1).

$$\% \, Removal = \frac{C_o - C_f}{C_o} \, x \, 100 \qquad 1$$

Where, $C_o$ and $C_f$ were the initial and final concentration of lead (II) ions in the solution in mg/L. The amount of metal ion adsorbed in milligram per gram was determined using the mass balance equation (2):

$$q_e = \frac{C_i - C_e}{M} V \qquad 2$$

Where $q_e$ is the metal uptake (mg metal adsorbed per g adsorbent), $C_i$ and $C_e$ is the initial and equilibrium metal ion concentration in solution (mg/L), V is the volume of the solution (mL) and M is the weight of activated carbon (g).

## RESULTS AND DISCUSSION

### SEM Micrographs

Scanning Electron Microscope (SEM) was used to observe the pore structure of the activated carbon. Pores present in activated carbon act as the active sites, where adsorption take place. The surface morphology of the biocarbon was studied before and after the removal of Pb (II) ions by SEM analysis. Figure 1is the SEM of pure LCLBC and Figure 2 is the SEM picture of LCLBC after the adsorption of Pb (II) ions. It

confirmed the presence of chemical interactions on the surface of the adsorbent. The micrographs clearly show a number of macro-pores and well defined crystalline structure in biocarbon samples.
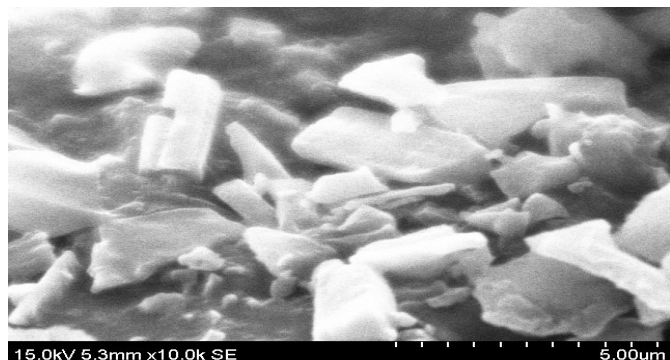


**Fig 1** SEM photograph of LCLBC before Pb(II) ions removal
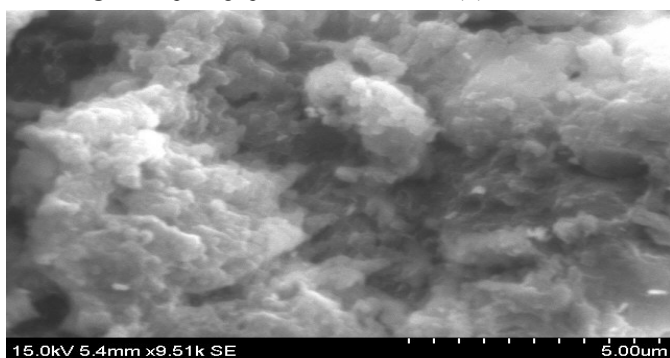


**Fig 2** SEM photograph of LCLBC after Pb(II)ions removal

### FTIR Analysis of Pb (II) ions

By Fourier transform infrared (FTIR) spectroscopy, functional groups present on the adsorbent surface were identified. The FTIR spectrum of biocarbon before and after the removal of Pb (II) ions are shown in the Fig 3. It reveals that the biocarbon has different functional groups which may act as selective active sites for metal ions coordination. The vibrating signals in both cases of adsorption of Pb (II) ions were just similar. The adsorption band at 3416 - 3417 cm$^{-1}$ is due to-OH groups. The strong band at 2859 - 2925 cm$^{-1}$ could be assigned to the aliphatic C - H group (Shriner*et al.* 1998).The adsorption band at 1630 - 1711 cm$^{-1}$ are attributed to C=O and C=C strong stretching vibrations. The peak observed at 1114 - 1161 cm$^{-1}$ corresponds to C-O group present in biocarbon matrix. When compared the two spectra which shows that there was no shift in wavelength. This indicates that -OH, -CH and C=O groups are likely to participate in Pb (II) ions adsorption. The FTIR-spectra indicates the presence of ionizable functional groups and their ionization leaves vacant sites which can be replaced by metal ions. The ion exchange process may be the mechanism involved in the biocarbon and Pb (II) ions interaction. This indicates that LCLBC can be used as an adsorbent for heavy metal removal. It also suggests that, the biocarbon matrix is not damaged or altered and it can be reused with simple washings.
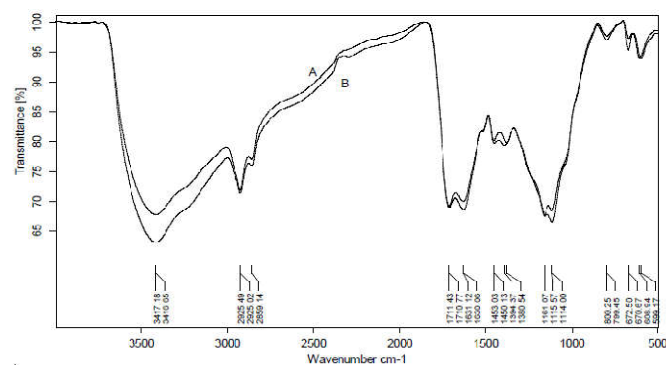


**Fig 3** FTIR spectrum for pure LCLBC(A) and Pb (II) ions adsorbed LCLBC(B)

### Effect of pH

The effect of pH on the removal of Pb (II) ions from synthetic wastewater was investigated by varying the pH of metal ion solutions in the range of 3 - 8 (Fig 4). The adsorption of Pb (II) ions depends on the pH value of the aqueous solution. It is noticed that, there is a gradual enhancement of the Pb (II) ions adsorption with the increase of pH from 3 to 6. The Pb (II) ions removal efficiency increased from 55 to 97.5 %. At low pH values, metal removal was inhibited possibly due to a competition between protons and lead ions on the sorption sites. As the pH value was increased, the negative charge density on LCLBC surface increased due to deprotonation of the metal binding sites resulting in an increase of the adsorption of metal ions. The high dependence on the pH for Pb (II) ions adsorption could be explained on the basis of surface charge density of biocarbon and O - H groups.
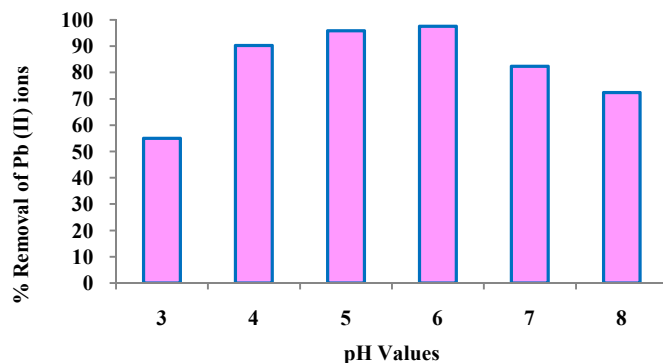


**Fig 4** Effect of pH on the removal of Pb (II) ions

### Effect of contact time

The effect of contact time on the removal of Pb (II) ions was studied for a period of 30 - 210 min. The effect of contact time for the sorption of Pb (II) ions onto the surface of LCLBC is shown in the following Fig 5. The kinetics of lead removal by biocarbon is characterized by a strong increase of the amount of lead sorbed during the first minutes of contact solution. A rapid sorption rate was observed up to 150 min of the sorption process and thereafter, it remains nearly constant. As an approximation, the removal of Pb (II) ions can be said to take place in two distinct steps: a relatively fast followed by a slower rate. This rapid initial uptake is similar to previous reports on the biosorption of heavy metals by different biosorbents (Yin *et al*. 1999).
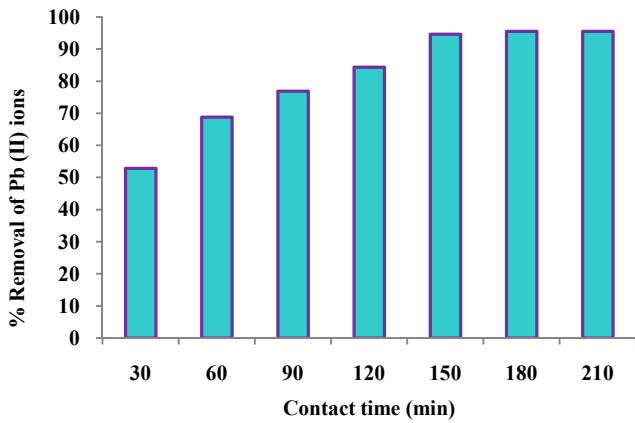
**Fig 5** Effect of contact time on the removal of Pb (II) ions

### Effect of adsorbent dose

The effect of adsorbent dose on the equilibrium adsorption of Pb (II) ions from synthetic wastewater onto LCLBC was studied and the results are shown in Fig 6. From the results, it is observed that, the adsorption of Pb (II) ions increased from 46.2 to 95.5 % with an increase of the adsorbent dose from 1 to 2.5g. This can be described by the more accessibility of surface sites with the increase of the adsorbent dose (Jung *et al.* 2001). This attributed the similar behaviour to the fact that the surface complexation is the major mechanism in the sorption process. It is also noted that, further increase of the LCLBC dose does not lead to any significant change of the Pb (II) ions adsorption. However, the adsorption density decreased is mainly due to the unsaturation of adsorption sites. It could be also endorsed to the accumulation of adsorbent particle resulted from high sorbent concentration which would lead to a decrease in the total surface area of the adsorbent and an increase in diffusional path (Shukla *et al.* 2002).
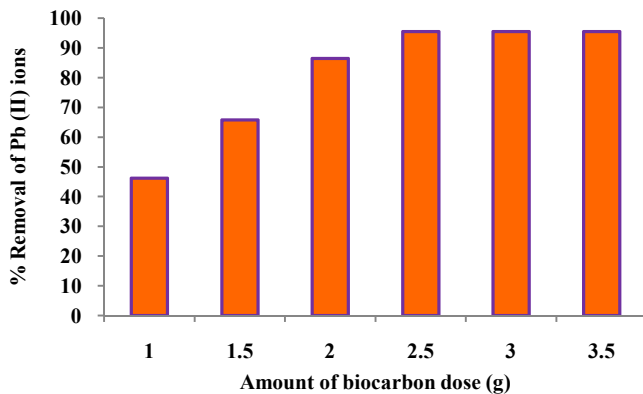


**Fig 6** Effect of biocarbon dose on the removal of Pb (II) ions

### Effect of initial metal ion concentration

The influence of initial metal ion cocentrtaion on the removal of Pb (II) ions is reported in the following Fig 7. With the increasing concentration from 10 to 100 mg/L, removal rate of Pb (II) ions was 95.8 %. The removal rate was progressively increased with increasing of metal ion concentration. The rate of removal was higher at the beginning due to larger surface area of biocarbon being available for the adsorption. Once saturation point is reached the capacity of the adsorbent gets exhausted and removal rate is controlled by the sorbate.
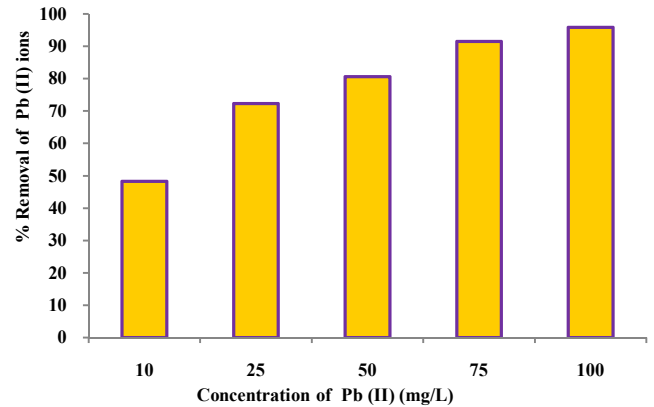


**Fig 7** Effect of initial metal ion concentration on the removal of Pb (II) ions

### Adsorption Isotherms

The study of application of adsorption isotherm models to the equilibrium data is important to optimize the design of the biosorption system for the removal of the metal ions. The adsorption of a substance from one phase to another leads to a thermodynamically defined distribution of that substance between the phases as the system reaches equilibrium state (Naiya *et al.* 2009). The most common adsorption isotherm models of Langmuir and Freundlich have been tested in the present study. The linear form of the Langmuir isotherm is given by the following equation (3).

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

Where, $q_e$ is the equilibrium metal ion concentration on the biosorbent (mg/g), $q_{max}$ is the maximum biosorption capacity of biosorbent (mg/g) and $C_e$ is the equilibrium concentration of the metal ion in solution (mg/L). The data obtained from biosorption of heavy metals were analyzed using Langmuir isotherm. The plot of $C_e$ versus $C_e/q_e$ should give a straight line, its slope equals to $1/q_{max}$ and the intercept has the value of $1/q_{max}b$, where b is the biosorption coefficient (L/mg). From the plot (Fig 8), $q_{max}$ and $r^2$ values of Pb (II) ions were calculated and given in the Table 1.
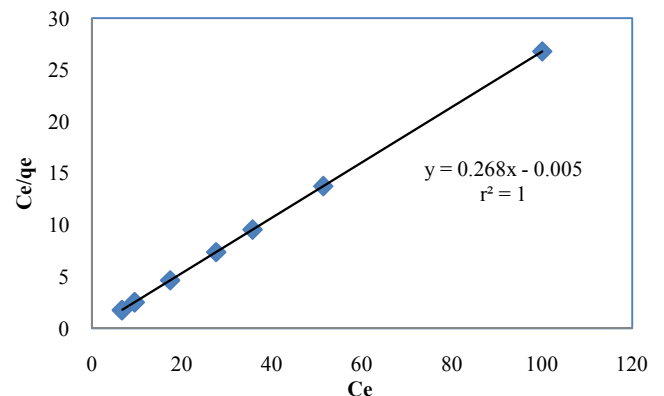


**Fig 8** Langmuir isotherm for the removal of Pb (II) ions

The Freundlich biosorption isotherm model is an empirical equation based on the biosorption on a heterogeneous surface. The linear form of Freundlich biosorption isotherm can be defined by the following equation (4):

$$Log \, q_e = Log \, K_f + \frac{1}{n} \log C_e \qquad (4)$$

The constant is an empirical parameter which reflects the intensity of adsorption that varies with the degree of heterogeneity and $K_f$ is a constant related to adsorption capacity. The constants n and $K_f$ were calculated by plotting log $C_e$ against log $q_e$ (the slope = 1/n and the intercept = log $K_f$). The results were illustrated in Fig 9. The values of n are between 1 and 10 represent a favorable adsorption. The obtained value of n for Pb (II) ions was shown in the Table 1 and it indicates that the biosorption is more favorable.
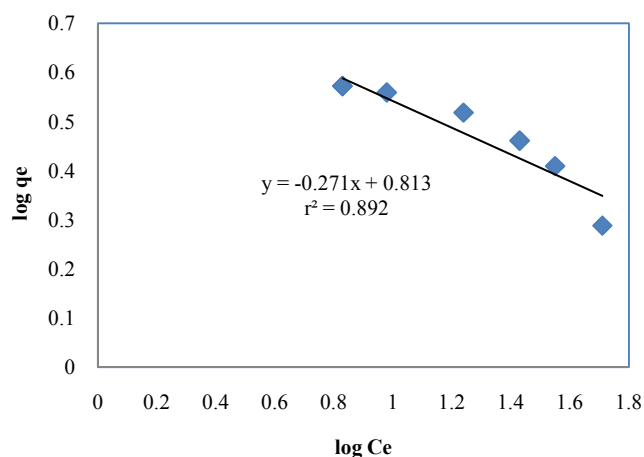


**Fig 9** Freundlich isotherm for the removal of Pb(II) ions

Comparing the $r^2$ values of the two isotherms (Table 1), the adsorption of LCLBC fitted well with the Langmuir model suggesting that the adsorption of Pb (II) ions onto this adsorbent is a mono-layer process and the adsorption behaviour is homogeneous rather than heterogeneous.

**Table 1** Langmuir and Freundlich parameters for the removal of Pb (II) ions on biocarbon

| Metal | Langmuir parameters | | Freundlich parameters | | |
|---|---|---|---|---|---|
| | $q_{max}$ (m/g) | $r^2$ | $k_f$ ((mg/g)(mg/L)$^n$) | n | $r^2$ |
| Pb(II) | 3.731 | 1 | 6.50 | 3.690 | 0.8929 |

### Biosorption Kinetics

The kinetics of biosorption of Pb (II) ions was studied by using the two important kinetic models of Lagergren pseudo-first-order and pseudo-second-order equations. The general pseudo-first-order equation is expressed as (5)

$$\log(q_e - q_t) = \log(q_e) - \left(\frac{k_1}{2.303}\right) \qquad (5)$$

Where $q_e$ and $q_t$ are the amounts of metal ions sorbed onto the biocarbon (mg/g) at equilibrium and at time t (min) respectively. By plotting the log ($q_e$ - $q_t$) versus time t, the first-order rate constant $k_1$(1/min) and the equilibrium capacity $q_e$ can be obtained from the slope and intercept respectively. The linear form of the pseudo-second-order chemisorption kinetics rate equation is expressed as (6)

$$\left(\frac{t}{q_t}\right) = \left(\frac{1}{k_2 q_e^2}\right) + \left(\frac{t}{q_e}\right) t \qquad (6)$$

The constants can be determined by plotting $t/q_t$ versus time t. The second-order rate constant $k_2$ (g/ mg/min) and $q_e$ (mg/g) values can be calculated from the intercept and slope of the plot.

All the kinetic data of the respective metal ions were tested using the pseudo first order and pseudo second-order rate equation. In case of the first-order rate equation, data were poorly fitted (data and figure not given) and the Lagergren correlation coefficient ($r^2$) value was very low. Therefore, the observed result suggest that, lead ion removal follows only Lagergren pseudo-second order kinetics than pseudo-first order. The results of the pseudo second-order kinetics are shown in the Fig10.

The Lagergren parameters of the biosorption kinetics are presented in the Table 2. It was observed that, the pseudo-second-order model was well fitted for Pb (II) ions removal. The correlation coefficient ($r^2$) for the second-order model is 0.9805.
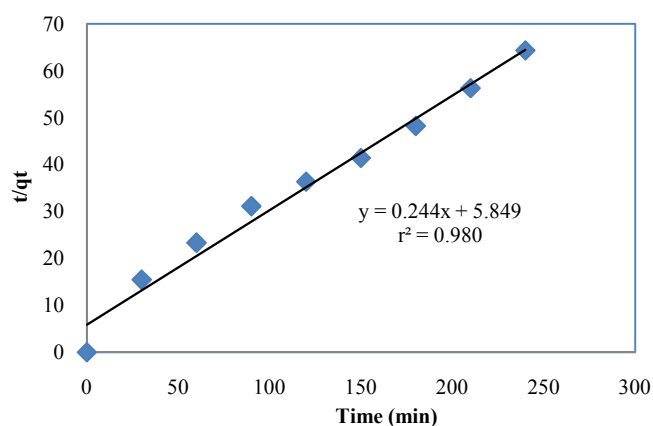


**Fig 10** Lagergren second order plot for the removal of Pb (II) ions

**Table 2** Biosorption kinetics for the removal of Pb(II) ions on biocarbon

| Metal | Second order | |
|---|---|---|
| | $k_2$ (g mg$^{-1}$ min$^{-1}$) | $r^2$ |
| Pb(II) | 1.228 x10$^{-2}$ | 0.9805 |

## CONCLUSION

The present study has shown that activated carbon prepared from the *Lantana camara* leaves biocarbon (LCLBC) can be used as an eco-friendly and low cost adsorbent for the removal of Pb (II) ions from synthetic aqueous solution. The adsorption characteristics of Pb (II) ions onto LCLBC was found to be influenced by initial pH of the solution, amount of biocarbon dose, concentration of metal ions and contact time. The empirical values are evaluated according to the Langmuir and Freundlich isotherms that are generally used to describe the adsorption process. Langmuir model suggests that the adsorption of Pb (II) ions onto LCLBC is a mono-layer process and the adsorption behaviour is homogeneous rather than heterogeneous. The pseudo-second-order model is well fitted for the removal of Pb (II) ions and the correlation coefficient ($r^2$) for the second-order model is 0.9805. The biosorption results indicate that, the maximum removal of Pb (II) ions by LCLBC was 95.5% under optimum conditions such as pH of 6, biocarbon dosage of 2.5g/100mL and equilibrium contact time 150 minutes. These results show that the LCLBC which have a very low economic value may be used effectively for

the removal of Pb (II) ions from aqueous systems for environmental protection purpose.

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