**Original Research Paper**

# Biosorption of Lead Ions From Aqueous Solution by *Terminalia catappa*: Equilibrium and Kinetic Studies

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## ABSTRACT

In the present investigation, the sorption is carried out to test the suitability of abundantly available plant based material *Terminalia catappa* L. leaf powder as an adsorbent for removal of lead ions from aqueous solution. The equilibrium studies are systematically carried out in a batch process, covering various process parameters that include contact time, adsorbent size and dosage, initial lead ion concentration and pH of the aqueous solution. It is observed that there is a significant increase in percentage removal of lead ions as pH increases from 2 to 6 and attain maximum at pH 6. The contact time is to be 20 minutes. The Langmuir isotherm is more suitable for sorption followed by Freundlich and Temkin isotherms with a maximum adsorption capacity of 28.63 mg/g of lead ion on *T. catappa* leaf biomass. The sorption of lead ions follows second order kinetic model having a correlation coefficient of 0.9996.

## INTRODUCTION

Heavy metals such as lead, mercury, arsenic, copper, zinc and cadmium are highly toxic to the life. They can cause accumulative poisoning, cancer, brain damage, etc. Lead is a general metabolic poison and enzyme inhibitor. It can cause mental retardation and semipermanent brain damage in young children. Lead has the ability to replace calcium in bones to form sites for long-term release. Toxic metals are released into the environment in a number of ways. Coal combustion, sewage wastewaters, automobile emissions, battery industry, mining activities, and the utilization of fossil fuels are just a few examples. Among the various water-treatment techniques described, adsorption is generally preferred for the removal of heavy metal ions due to its high efficiency, easy handling, availability of different adsorbents and cost effectiveness .

Some types of biomass can serve as biosorbents to remove heavy metals. Biosorption, a term that describes the removal of heavy metals by the passive binding to non-living biomass from an aqueous solution (Davis et al. 2003), is considered as an alternative to conventional methods of metal recovery from solutions (Singh et al. 1998). Although freely suspended biomass may have better contact with adsorbates during biosorption process, the suspended biomass is normally not the practical form for the direct use in the removal of heavy metals. Since cell immobilization can enhance its stability, mechanical strength, reusability and the ease of treatment, the technique has been well used to remove toxic heavy metals. Usually a fixed bed reactor, in which a certain type of immobilized biomass is packed, is designed for effective and continuous removal of heavy metals.

The effectiveness of biosorption for the removal of heavy metals has been shown in a number of studies. However, only when the cost of the biosorption process can complete with the existing technologies, it will be accepted commercially. The cost of biomass production plays an important

role in determining the overall cost of a biosorption process. Therefore, low cost biomass becomes a crucial factor when considering practical application of biosorption. The present work investigates the potential use of untreated *Terminalia catappa* biomass as metal sorbent for removal of lead from aqueous solution. *T. catappa* was chosen as a biosorbent because of the relative lack of information about its sorption ability. Parameters affecting the biosorption process such as pH, contact time, metal ion concentration, adsorbent concentration and adsorbent size were evaluated. The equilibrium adsorption data were evaluated by Langmuir, Freundlich and Temkin isotherm models. The kinetic experimental data were correlated by first and second order kinetic models.

## MATERIALS AND METHODS

**Preparation of biosorbent:** The *T. catappa* leaves were collected, washed with deionized water several times to remove dirt, dried, powdered using domestic grinder to the powder size of 75-212 μm and used as biosorbent without any pretreatment for lead adsorption.

**Chemicals:** Analytical grade $Pb(NO_3)_2$, HCl and NaOH were purchased from Merck (Mumbai). Lead ions were prepared by dissolving its corresponding salt in distilled water. The pH of solutions was adjusted with 0.1 N HCl and NaOH.

All the experiments were repeated five times and the average values were recorded. Also, blank experiments were conducted to ensure that no adsorption was taking place on the walls of the apparatus used.

**Biosorption experiments:** Biosorption experiments were performed at room temperature $(30 \pm 1°C)$ in a rotary shaker at 180 rpm containing 30 mL of different lead concentrations using 250 mL Erlenmeyer flasks. After one hour of contact (according to the preliminary sorption dynamics tests) with 0.1 g *T. catappa* leaves biomass, equilibrium was reached and the reaction mixture was centrifuged for 5 min. The metal content in the supernatant was determined using Atomic Absorption Spectrophotometer (GBC Avanta Ver 1.32, Australia) after filtering the adsorbent with 0.45 μm filter paper. The amount of metal adsorbed by the leaves was calculated from the difference between metal quantity added to the biomass and metal content of the supernatant using the following equation.

$$q = \left(C_0 - C_f\right)\frac{V}{M} \qquad ..(1)$$

Where $q$ is the metal uptake (mg/g); $C_o$ and $C_f$ the initial and final metal concentrations in the solution (mg/L), respectively; $V$ the solution volume (mL) and $M$ the mass of biosorbent (g).

The Langmuir sorption model was chosen for the estimation of maximum lead sorption by the biosorbent, which can be expressed as:

$$q = \frac{Q_{max}bC_{eq}}{1+bC_{eq}} \qquad ...(2)$$

Where $Q_{max}$ indicates the monolayer adsorption capacity of adsorbent (mg/g) and the Langmuir constant $b$ (L/mg) is related to the energy of adsorption. For fitting the experimental data, the Langmuir model was linearized as:

$$\frac{1}{q} = \frac{1}{Q_{max}} + \frac{1}{bQ_{max}C_{eq}} \qquad ...(3)$$

The freundlich model is represented by the equation:

$$q = KC_{eq}^{\frac{1}{n}}$$                ...(4)

Where $K$ (mg/g) is the Freundlich constant related to adsorption capacity of adsorbent and $1/n$ is the Freundlich exponent related to adsorption intensity (dimensionless). For fitting the experimental data, the Freundlich model was linearized as follows:

$$\ln q = \ln K + \frac{1}{n} \ln C_{eq}$$                ...(5)

The Temkin isotherm has generally been applied in the following form.

$$q = \frac{RT}{b_T} \ln\left(A_T C_{eq}\right)$$                ...(6)

Where $A_T$ (L/mg) and $b_T$ are Temkin isotherm constants.

**Biosorption kinetics:** The kinetic studies were carried out by conducting batch biosorption experiments with different initial lead concentrations. Samples were taken at different time periods and analysed for their lead concentration.

## RESULTS AND DISCUSSION

**The effect of contact time:** The data obtained from the biosorption of lead ions on the *T. catappa* showed that a contact time of 20 min was sufficient to achieve equilibrium and the adsorption did not change significantly with further increase in contact time. Therefore, the uptake and unadsorbed lead concentrations at the end of 20 min are given as the equilibrium values ($q_e$, mg/g; $C_{eq}$, mg/L) respectively (Fig. 1) and the other adsorption experiments were conducted at this contact time of 20 min.

**Effect of pH:** The pH of the medium affects the solubility of metal ions and the concentration of the counter ions on the functional groups of the biomass cell walls, so pH is an important parameter for biosorption of metal ions from aqueous solutions (Gong et al. 2005).

*T. catappa* presents a high content of ionizable groups (carboxyl groups from mannuronic and guluronic acids) on the cell wall polysaccharides, which makes it very liable to the influence of the pH. As shown in Fig. 2, the uptake of lead increased with the increase in pH from 2.0 to 6.0. Similar results were also reported earlier for different biomass systems (Matheickal & Yu 1999). At pH values lower than 2.0, lead removal was inhibited possibly as a result of the competition between hydrogen and lead ions on the sorption sites, with an apparent preponderance of hydrogen ions, which restricts the approach of metal cations as in consequence of the repulsive force. As the pH increased, the ligands such as carboxylate groups in *T. catappa* would be exposed increasing the negative charge density on the biomass surface, which in turn increases the attraction of metallic ions with positive charge and allowing the biosorption onto the cell surface.

In this study, the lead cations at around pH 6 were expected to interact more strongly with the negatively charged binding sites in the adsorbent. As a result, the optimum pH for lead adsorption was found as 6 and the other adsorption experiments were performed at this pH value.

**Effect of metal ion concentration:** Fig. 3 shows the effect of metal ion concentration on the adsorption of lead by *T. catappa.* The data show that the metal uptake increases and the percentage

adsorption of lead decreases with increase in metal ion concentration. This increase (5.53-22.07 mg/g) is a result of increase in the driving force, i.e. concentration gradient. However, the percentage adsorption of lead ions on *T. catappa* was decreased from 92.18 to 73.57%. Though an increase in metal uptake was observed, the decrease in percentage adsorption may be attributed to lack of sufficient surface area to accommodate much more metal available in the solution. The percentage adsorption at higher concentration levels shows a decreasing trend whereas the equilibrium uptake of lead shows an opposite trend. At lower concentrations, all lead ions present in solution could interact with the binding sites and, thus, the percentage adsorption was higher than that at higher lead ion concentrations. At higher concentrations, lower adsorption yield is due to the saturation of adsorption sites. As a result, the purification yield can be increased by diluting the wastewaters containing high metal ion concentrations.

**Effect of adsorbent size:** The effect of different adsorbent particle sizes on percentage removal of lead was investigated and shown in Fig. 4. It reveals that the adsorption of lead on *T. catappa* decrease from 92.18 to 76.42 % with the increased particle size from 75 to 212 μm at an initial concentration of 20 mg/L. The smallest size obtained was 75μm due to the limitation of available grinder configuration. It is known that decreasing the average particle size of the adsorbent increases the surface area, which in turn increases the adsorption capacity.

**Effect of adsorbent dosage:** The effect of adsorbent dosage on the % removal at equilibrium conditions is shown in Fig. 5. It was observed that the amount of lead adsorbed varied with varying adsorbent dosage. The amount of lead adsorbed increases with an increase in adsorbent dosage from 0.1 to 0.5 g. The percentage lead removal was increased from 92.18 to 98.03% for an increase in adsorbent dosage from 0.1 to 0.5 g at initial concentration of 20 mg/L. The increase in the adsorption of the amount of solute is obvious due to increasing biomass surface area. Similar trend was also observed for lead removal using *Azadirachta indica* as adsorbent (Bhattacharyya & Sharma 2004).

## BIOSORPTION EQUILIBRIUM

The equilibrium biosorption of lead on the *T. catappa* as a function of the initial concentration of lead is shown in Figs. 6-9. There was a gradual increase of adsorption for lead ions until equilibrium was attained. The Langmuir, Freundlich and Temkin models are often used to describe equilibrium sorption isotherms. The calculated results of the Langmuir, Freundlich and Temkin isotherm constants are given in Table 1.

It was found that the adsorption of lead on the *T. catappa* was correlated well with the Langmuir equation and Freundlich as compared to Temkin equation under the concentration range studied. Examination of the Temkin data shows that this isotherm was not modelled as well across the concentration range studied.

## KINETICS OF ADSORPTION

The prediction of adsorption rate gives important information for designing batch adsorption systems. Information on the kinetics of solute uptake is required for selecting optimum operating conditions for full-scale batch process. Fig. 10 shows the plottings between amount adsorbed, $q_e$ (mg/g) versus time, $t$ (min) for an initial concentration of 20 mg/L. The adsorption rate within the first 5 min was observed to be very high and thereafter the reaction proceeds at a slower rate till equilibrium and finally a steady state was obtained after equilibrium. The saturation time was found to be 20 min based on the initial metal concentration. The kinetics of the adsorption data was analysed using two

kinetic models, pseudo-first- and pseudo-second-order kinetic models. These models correlate solute uptake, which is important in predicting the reactor volume. These models are explained below.

**The pseudo-first-order equation:** The pseudo-first-order equation of Lagergren is generally expressed as follows:

$$\frac{dq_t}{dt} = k_1(q_e - q_t) \qquad \qquad ...(7)$$

Where $q_e$ and $q_t$ are the sorption capacities at equilibrium and at time $t$, respectively (mg/g) and $k_1$ is the rate constant of pseudo first-order sorption (min$^{-1}$). After integration and applying boundary conditions, $q_t = 0$ to $q_t = q_t$ at $t = 0$ to $t = t$, the integrated form of eq. (7) becomes:

$$\log(q_e - q_t) = \log(q_e) - \frac{k_1}{2.303}t \qquad \qquad ...(8)$$

The pseudo-first-order rate constant $k_1$ can be obtained from the slope of the graph between log (qe – q) versus time, $t$ (Fig. 11). The calculated $k_1$ values and their corresponding linear regression correlation coefficient values are given in Table 2. The linear regression correlation coefficient value $R^2_1$ was found to be 0.9695, which shows that this model cannot be applied to predict the adsorption kinetic model.

**The pseudo-second-order equation:** If the rate of sorption is a second-order mechanism, the pseudo-second-order chemisorption kinetic rate equation is expressed as (Ho & McKay 1998):

$$\frac{dq_t}{dt} = k(q_e - q_t)^2 \qquad \qquad ...(9)$$

Where $q_e$ and $q_t$ are the sorption capacity at equilibrium at time $t$, respectively (mg/g) and $k$ is the rate constant of pseudo-second-order sorption (g/mg. min). For the boundary conditions $q_t = 0$ to $q_t = q_t$ at $t = 0$ to $t = t$; the integrated form of eq. (9) becomes:

Table 1: Langmuir, Freundlich and Temkin isotherm constants and correlation coefficients.

| Langmuir | | Freundlich | | Temkin | |
|---|---|---|---|---|---|
| Q (mg/g) | 28.636 | $K_f$ (mg/g) | 4.5552 | $A_T$ (L/mg) | 1.4091 |
| b (L/mg) | 0.1213 | n(g/L) | 2.0125 | $b_T$ | 392.37 |
| $R^2$ | 0.9979 | $R^2$ | 0.9956 | $R^2$ | 0.9811 |

Table 2: Kinetic constants for lead onto *Terminalia catappa* L.

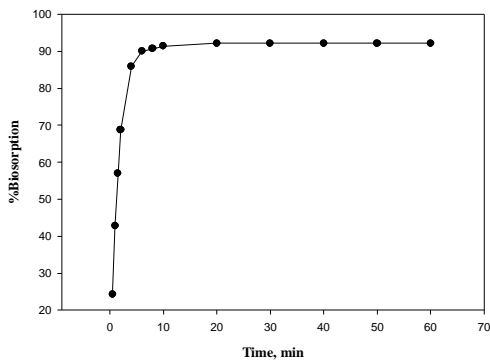| Initial Concentration (mg/L) | Pseudo-first-order | | | Pseudo-second-order | | |
|---|---|---|---|---|---|---|
| | Rate constant $K_1$ (min$^{-1}$) | Amount of lead absorbed on adsorbent, $q_e$ (mg/g) | Correlation coefficient $R^2_1$ | Rate constant $K_2$ (min$^{-1}$) | Amount of lead absorbed on adsorbent, $q_e$ (mg/g) | Correlation coefficient $R^2_2$ |
| 20 | 0.6032 | 9.532 | 0.9695 | 0.1165 | 5.535 | 0.9996 |

Fig. 1: Effect of contact time on biosorption of lead by
*Terminalia catappa* L. for 20 mg/L of metal and
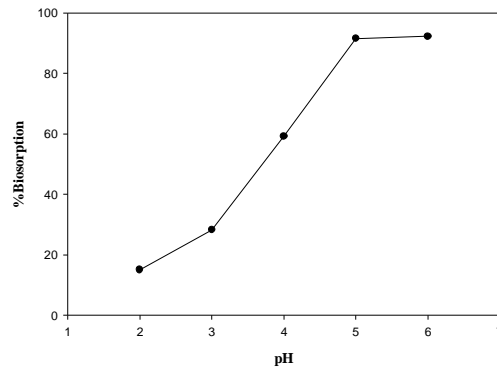0.1 g/30 mL of biosorbent concentration.



Fig. 2: Effect of pH on biosorption by *Terminalia catappa*
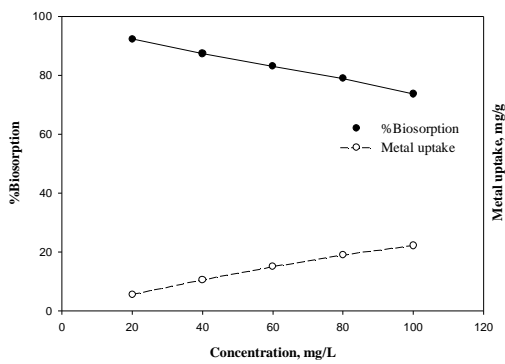L. for 20 mg/L of metal and 0.1 g/30 mL of biosorbent
concentration.



Fig. 3: Effect of metal concentration on the biosorption of
lead by *Terminalia catappa* L. for 0.1 g/30 mL of
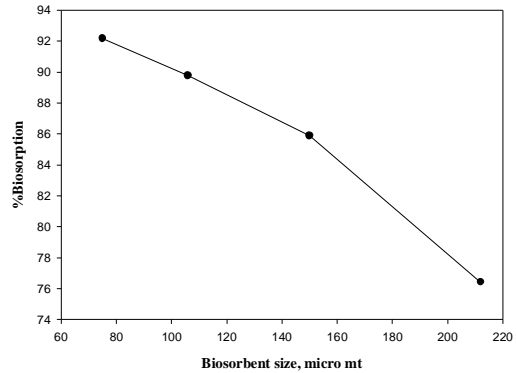biosorbent concentration.



Fig. 4: Effect of *Terminalia catappa* L. particle size on
biosorption of lead for 20 mg/L of metal and
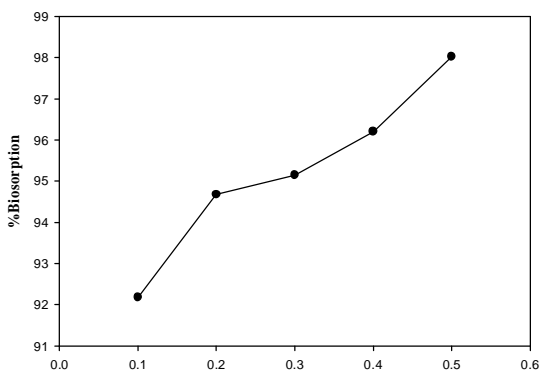0.1 g/30 mL of biosorbent concentration.



Fig. 5: Effect of *Terminalia catappa* L. dosage on
biosorption of lead for 20 mg/L of metal concentration.
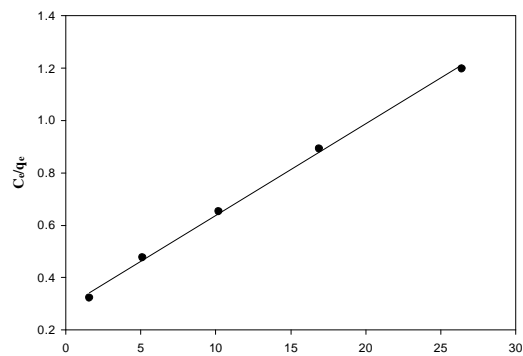


Fig. 6: Langmuir biosorption isotherm for lead at 0.1
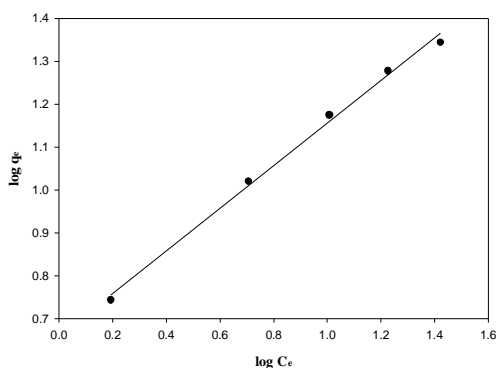g/30 mL of biosorbent concentration.

Fig. 7: Freundlich biosorption isotherm for lead at 0.1
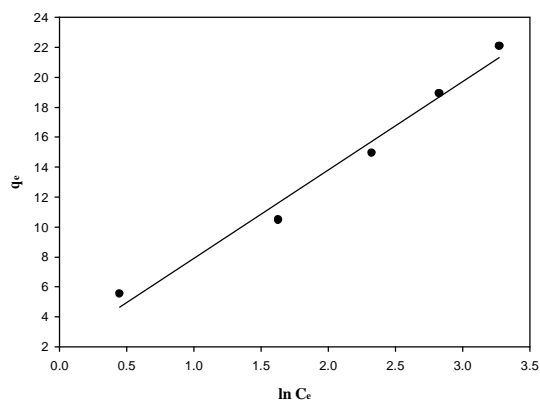g/30 mL of biosorbent concentration.



Fig. 8: Temkin biosorption isotherm for lead at 0.1 g/30 mL
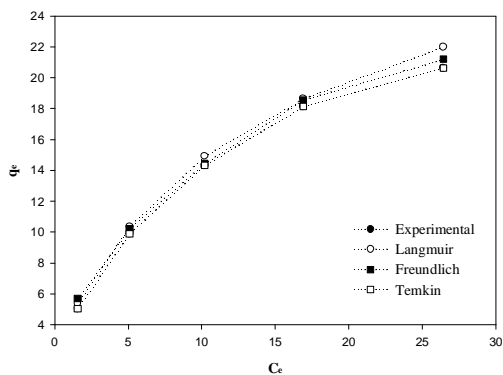of biosorbent concentration.



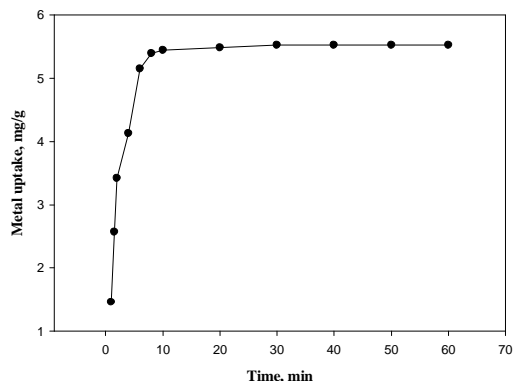Fig. 9: Equilibrium curves for lead onto *Terminalia
catappa* L.



Fig. 10: Effect of contac time on lead uptake by *Terminalia
catappa* L. for 20 mg/L of metal and 0.1 g/30 mL of
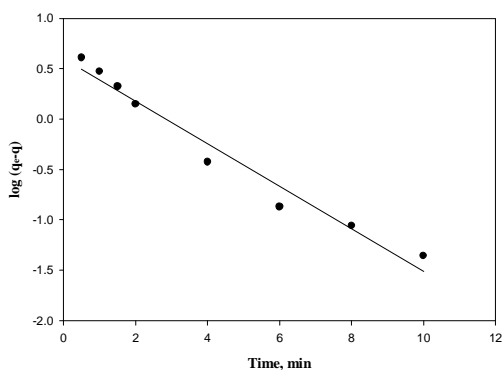biosorbent concentration.



Fig. 11: Pseudo-first order biosorption of lead uptake by
*Terminalia catappa* L. for 20 mg/L of metal and
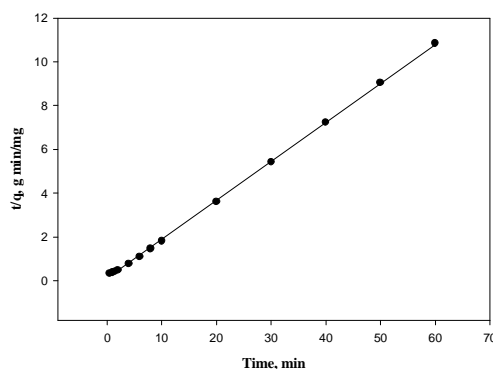0.1 g/30 mL of biosorbent concentration.



Fig. 12: Pseudo-second order biosorption of lead uptake by
*Terminalia catappa* L. for 20 mg/L of metal and
0.1 g/30 mL of biosorbent concentration.

Table 3: Maximum adsorption capacities for lead adsorption to different adsorbents.

| Adsorbent material | Adsorption capacity (mg/g) | pH | Reference |
|---|---|---|---|
| Na-mont morillonite | 3.61 | 5 | (Abollino et al. 2003) |
| Crushed concrete fines | 33 | 5.5 | (Coleman et al. 2005) |
| Coir | 8.6 | 5.5 | (Kathrine & Hansen 2006) |
| Barley straw | 5.3 | 5.5 | (Kathrine & Hansen 2006) |
| Peat | 11.71 | 5.5 | (Kathrine & Hansen 2006) |
| Coniferous bark | 7.4 | 5.5 | (Kathrine & Hansen 2006) |
| Sil/PE1/GA$_{0.5}$ | 32.79 | 5-6 | (Ghoul et al. 2003) |
| *Fontinalis antipyretica* | 14.7 | 5.0 | (Martins et al. 2004) |
| *Mucor rouxii* (live) | 4.89 | 5.0 | (Yan & Viraraghavan 2000) |
| *Mucor rouxii* (NaOH pretreated) | 5.63 | 5.0 | (Yan & Viraraghavan 2000) |
| *Mucor rouxii* (Na$_2$CO$_3$ pretreated) | 3.26 | 5.0 | (Yan & Viraraghavan 2000) |
| *Mucor rouxii* (NaHCO$_3$ pretreated) | 6.28 | 5.0 | (Yan & Viraraghavan 2000) |
| *Sargassum* sp. | 24.35 | 4.5 | (Esteves et al. 2000) |
| Animal bones | 11.55 | 5.0 | (Banat et al. 2000) |
| *Syzygium cumuni* L. | 32.47 | 6 | (King et al. 2007) |
| *T. catappa* L. | 28.63 | 6 | Present study |

$$\frac{t}{q_t} = \frac{1}{kq_e^2} + \frac{1}{q_e}t \qquad \qquad ...(10)$$

Where $t$ is the contact time (min), $q_e$ (mg/g) and $q_1$ (mg/g) are the amount of the solute adsorbed at equilibrium. Eq. (10) does not have the problem of assigning an effective $q_e$. If pseudo-second-order kinetics is applicable, the graph $t/q_t$ against $t$ of eq. (10) should give a linear relationship, from which $q_e$ and $k$ can be determined from the slope and intercept of the plot (Fig. 12) and there is no need to know any parameter beforehand.

The pseudo-second-order rate constant $k_2$, the calculated $q_e$ value and the corresponding linear regression correlation coefficient value $R_2^2$ are given in Table 2. At an initial lead concentration of 20 mg/L, the linear regression correlation coefficient $R_2^2$ value was higher. The higher $R_2^2$ value confirms that the adsorption data were well represented by pseudo-second order kinetic model.

A comparison of the maximum capacity $Q_{max}$ of *T. catappa* with those of some other adsorbents reported in literature is given in Table 3. Differences of metal uptake are due to the properties of each adsorbent such as structure, functional groups and surface area.

## CONCLUSIONS

The present study shows that the *T. catappa* is an effective biosorbent for the adsorption of lead ions from aqueous solution. The Percentage biosorption of lead ions by *T. catappa* was increased by increasing the pH up to 6. The uptake of lead ions by *T. catappa* was increased by increasing the metal ion concentration. The percentage biosorption of lead ions by *T. catappa* was increased with increasing the adsorbent dosage. The Percentage biosorption of lead ions by *T. catappa* was decreased with increase in the adsorbent size. The adsorption isotherms could be well fitted by the Langmuir equation followed by Freundlich equation. The biosorption process could be best described by the second-order equation.

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# ENVIRONMENTAL NEWS

**Innovation Centres for Clean Technologies**

The approval of India's technology transfer proposal to establish a global network of climate innovation centres for developing and deploying clean technologies at the just concluded climate change talks in Copenhagen should cheer businesses. 'In its submission to the UNFCCC, India had suggested that climate innovation centres be located in various parts of the world for undertaking development and deployment of clean technologies suited to their respective regions. The centres would do so by forging private and public sector partnerships. The centres would undertake R&D after identifying the local needs and appropriate technologies, and develop and deploy them after undertaking capacity building to enhance their faster absorption. The Indian Institute of Technology, Delhi and UK-based Carbon Trust have estimated that five regional centres would entail an initial investment of $2.5 billion, and potentially trigger an investment of $2.5 billion by the private sector.

While the centres may not deliver breakthrough technologies in geo-engineering or carbon capture and storage, they are expected to deliver utilitarian technologies like development of cleaner cooking ranges and deployment of energy efficient lighting solutions, catering to the existing market and beyond. Today, the global low carbon and environmental goods and services sector is estimated to be worth more than $4 trillion. It comprises technological solutions for tackling pollution of air, water and land; renewable technologies for small hydro, wind, solar etc and low-carbon technologies for construction, transport and energy sectors.

*The Financial Express, December 14, 2009*

**Organic Food**

The study done by the London School of Hygiene and Tropical Medicine (LSHTM) and funded by the Food Standards Agency (FSA), claims that organic food is no healthier than normally grown food. A team of about 50 researchers reviewed all papers published over the past 50 years that related to the nutrient content and health difference between organic and conventional food. The team, led by Dr Alan Dangour, says it is the most comprehensive study in the area to-date. "Observations, as above, are based on systemic review of literature. A small number of differences in nutrient content were found to exist between organically and conventionally produced crop and livestock, but these are unlikely to be of any public health relevance," said Dr Dangour.

Further, researchers did not find any difference in the level of most nutrients, including vitamin C, calcium and iron in crops. The same result held for meat, dairy products and eggs.

*Business Standard, August 21, 2009*