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Biosorption of Lead (Pb^{2+}) by *Luffa cylindrical* Fiber

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Abstract: The purpose of this research investigated the adsorption ability and determined the appropriate condition for adsorption of lead (Pb^{2+}) in synthetic wastewater by *Luffa cylindrical* Fiber (LCF) and 0.5 NaOH Modified *Luffa cylindrical* Fiber (MLCF). All the experiments were carried out in the batch experiment. Adsorption parameters consisted of NaOH concentration, pH, contact time, temperature, adsorbent size, agitation speed, adsorbent does and initial Pb^{2+} concentration. Moreover, the adsorption isotherm and desorption ability were examined for understanding the adsorption mechanism. The results indicated that the suitable condition for adsorption of lead (Pb^{2+}) by LCF and MLCF was pH 4.0, contact time 30 min, temperature 40°C, adsorbent size 250-600 μm, agitation speed 250 rpm, adsorbent dose 10 g L^{-1} and initial Pb^{2+} concentration 25 mg L^{-1}. In this condition, the adsorption ability of LCF and MLCF were 82.72 and 94.93%. The adsorption data was fitted well by Langmuir isotherm. The results can conclude that LCF and MLCF is an efficient adsorbent for Pb^{2+} contamination adsorption in wastewater. Finally, the LCF and MLCF utilization is an alternative waste management or environmental conservation.

Key words: Biosorption, lead, *Luffa cylindrical*, modification, adsorption isotherm, langmuir isotherm

INTRODUCTION

Rapid industrialization and population growth rate have lead to an increased disposal of heavy metals into the environment. They cause serious environmental problems in view of human health and the environment (Ramlogan, 1997). Lead is a significant heavy metal, which is used in several industrial and mining wastes, such as chemicals and allied products, lead acid storage batteries, ceramic and glass industries printing, ammunition, lead smelting and mine tailings (An *et al*., 2001). Lead poisoning in human causes severe damage to kidney, nervous system, reproductive system, liver and brain. Severe exposure to lead has been associated with sterility, abortion, stillbirths and neo-natal deaths (Wang *et al*., 2001; Ozer, 2007). Due to these effects, many researchers investigated to determine economic and environmental methods.

Adsorption is an efficient and economical method that can be used for the removal of heavy metals from wastewaters. However, the cost of adsorbents to be used is a most important restrict factors in view of applicability of adsorption process. In recent year, the methods for removing heavy metals from wastewater have resulted in the search for the development of alternatives from cheaper and readily available materials that may be useful to reduce the pollutant content to the levels established by the legislation. It has been observed that the majority of recent adsorption studies were conducted with activated carbon products from uneconomical sources, various biosorbents and mineral adsorbent with low-cost (Ayyappan *et al*., 2005). The use of low-cost biosorption such as tea leaves (Ahluwalia and Gayal, 2005), maize bran (Singh and Talat, 2006), saw dust neem bark (Noiya *et al*., 2008), cocoa shells (Meunier *et al*., 2003), maize leaf (Babarinde *et al*., 2006), lawny grass (Lu *et al*., 2009), wheat bran (Patnukao *et al*., 2008), rice husk ash (Noiya *et al*., 2009), tree fern (Ho, 2005), palm kernel fiber (Ho and Ofomaja, 2005), grape stalk (Martinez *et al*., 2006) and marine algae (Fourest and Volesky, 1997).

In this research, *Luffa cylindrical* Fiber (LCF) is shown in Fig. 1, which is an interested Thai agricultural waste is annually produced in high quantity. If its management is not enough effective, it may be cause of environmental problems. In previous researches, LCF consists of cellulose, hemi-cellulose and lignin, indicating the feasibility for removing toxic metal ions or enriching trace elements from aqueous solution due to the present of various interesting functional groups on the cellulose (Demir *et al*., 2008). Thus, the fiber and modified fiber were used to be adsorbent for Pb^{2+} adsorption in this research. NaOH was used in modification process because it can improve surface area of *Luffa cylindrical* fiber (Fig. 2)

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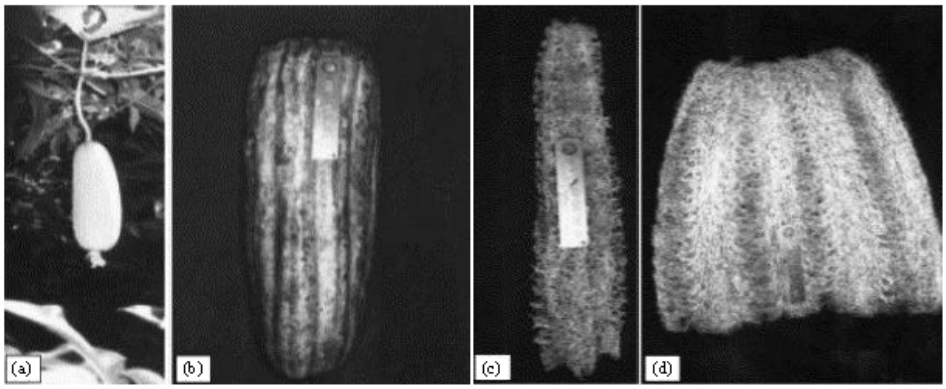

Fig. 1: The *Luffa cylindrica* plant (a) and fruit (b), the inner fiber core (c) and the outer core open as a mat (d) (Tanobe *et al*., 2005)

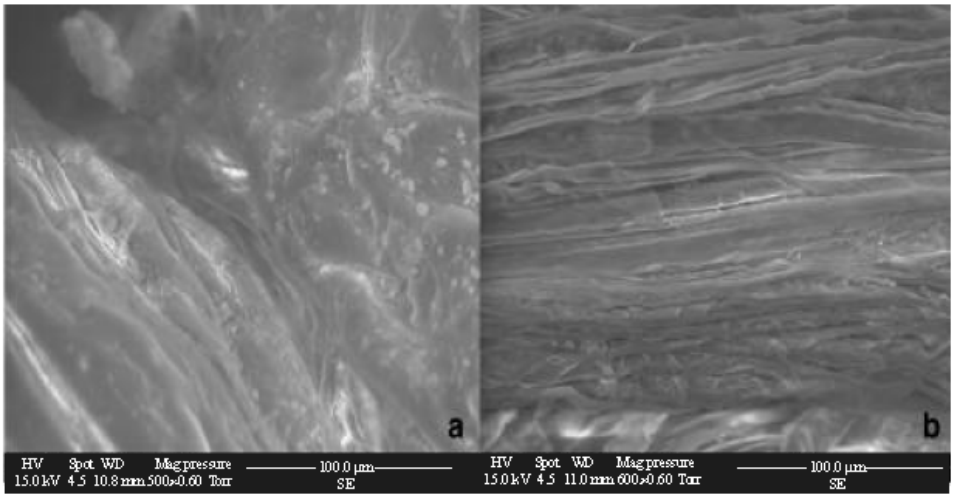

Fig. 2: SEM micrographs of a) untreated *Luffa cylindrical* fibres; b) Alkali treated *Luffa cylindrical* fibres (Ghali *et al*., 2009)

(Ghali *et al*., 2009). In the experiment, the effects of pH, contact time, adsorbent size, temperature, agitation speed, adsorbent dose, initial Pb^{2+} concentration and adsorption isotherm on Pb^{2+} adsorption was determined including adsorption isotherms. The utilization of LCF is an alternative waste management and natural conservation.

MATERIALS AND METHODS

LCF and MLCF preparation: LCF was collected from agricultural areas in Mahasarakham province, Thailand. They were washed by distilled water and dried in the oven at 105°C for 24 h. Then, they were ground to be 125-250, 250-600 and ≥ 600 µm. These samples were divided to be 2 types as unmodified and modified LCF using 0.5, 1.0 and 1.5 M NaOH as MLCF1, MLCF2 and MLCF3, respectively.

In the modification process, LCF was mixed NaOH solution (1:20) for 30 min. MLCF was washed with distilled water, until the pH was constant and then it was dried in an oven. The adsorption of Pb^{2+} was performed by batch technique.

Synthetic waste water preparation: Aqueous solutions of Pb^{2+} was prepared by dissolving $PbNO_3.4H_2O(s)$ in distilled water and diluted to get the desired concentration. Pb^{2+}concentrations were measured by Atomic Adsorption Spectrophotometer (AAS) (Shimadzu AA-670; 283.3 nm) using respective standard solutions to calibrate the instrument.

Batch mode adsorption studies: Batch experiments were carried out using a series of Erlenmeyer flasks of capacity 250 mL covered with paraffin sheet to prevent any foreign particle contamination. The effects of pH (2.0-7.0), contact time (0, 10, 30, 60, 120 and 300 min), adsorbent size (≤ 125, 125-250 and 250-600 µm), temperature (30, 40 and

60°C), agitation speed (50, 150 and 250 rpm), adsorbent dose (5, 10, 15, 20 and 25 g L^{-1}) and initial Pb^{2+} concentration (10, 25, 35 and 75 mg L^{-1}) were investigated.

The initial pH adjustments were carried out either by 0.1 M H_2SO_4 and 0.1 M NaOH. Samples were taken at certain time intervals, filtered through Whatman GF/A for removing the suspended biomass and analyzed for residual Pb^{2+} concentration. The Pb^{2+} concentration in the supernatant solution was determined using AAS. The removal efficiency of metal ion was calculated from:

$$\text{Removal } (\%) = \frac{C_0 - C_e}{C_0} \times 100\% \quad (1)$$

where, C_0 and C_e are the initial and equilibrium concentration (mg L^{-1}).

The adsorption capacity of metal ion is the concentration of metal ion on the adsorbent and it can be calculated based on the mass balance principle where;

$$q_e = \frac{(C_0 - C_e) \times V}{m} \quad (2)$$

In these two equations, q_e represents the amount of metal uptake per unit mass of the adsorbent (mg L^{-1}), V is the volume of the solution (l), m is the dry mass of the adsorbent (g), respectively (Ghali *et al*., 2009).

Adsorption isotherm: Adsorption isotherm is a functional expression that correlates the amount of adsorbed solute per unit weight of the adsorbent and the concentration of an adsorbate in bulk solution at a given temperature under equilibrium conditions. It is important to establish the most appropriate correlations for the batch equilibrium data using empirical or theoretical equations as it plays a functional role in predictive modeling procedures for analysis and design of adsorption systems. The adsorption isotherms are one of the most useful data to understand the mechanism of the adsorption and the characteristics of isotherms are needed before the interpretation the kinetics of the adsorption process (Guo *et al*., 2009). In this research, two isotherm models were used to describe the equilibrium adsorption as Langmuir and Freundlich models.

The Langmuir model assumes that energies of adsorption onto a surface and no transmigration of adsorbate in the plane of the surface (Guo *et al*., 2009). The Langmuir model for equilibrium ion removed is given by:

$$q_e = \frac{q_{max} b C_e}{(1 + bC_e)} \quad (3)$$

Where:

q_{max} = The maximum metal uptake per unit mass of adsorbent (mg g^{-1})

b = Langmuir constant (L mg^{-1}) related to energy of adsorption which reflects quantitatively the affinity between the adsorbent and adsorbed ions

The values of q_{max} and b are the characteristics of the Langmuir model. They can be determined by linearizing Eq. 3 as shown in Eq. 4:

$$\frac{C_e}{q_e} = \frac{1}{q_{max} b} + \frac{C_e}{q_{max}} \quad (4)$$

or

$$\frac{1}{q_e} = \frac{1}{q_{max} b} \frac{1}{C_e} + \frac{1}{q_{max}} \quad (5)$$

Therefore, a plot of $1/C_e$ versus $1/q_e$, gives a straight line of slope $1/q_{max}$ b and intercept $1/q_{max}$.

In the case of Freundlich, the energetic distribution of sites is heterogeneous, due to the diversity of sorption sites or the diverse nature of the metal ions adsorbed, free or hydrolyzed species (Kadirvelu and Namasivayam, 2003). This model is chosen to evaluate parameters associated to the sorption behavior. The equation is commonly represented by:

$$q_e = K_f C_e^{1/n} \quad (6)$$

where, K_f and n are Freundlich constants, indicating the adsorption capacity and the adsorption intensity, respectively.

The above equation is rearranged in linear form to give:

$$\ln q_e = \ln K_f + \frac{1}{n} \ln C_e \quad (7)$$

Thus, a plot of ln C_e versus ln q_e, gives a straight line of slope 1/n and intercept ln K_f.

RESULTS AND DISCUSSION

Effects of pH on adsorption: The pH of the solutions has been identified as the most important variable governing metal adsorption. This is partly due to the fact that hydrogen ions themselves are strong competing ions and partly that the solution pH influences the chemical speciation of the metal ions as well as the ionization of the functional groups onto the adsorbent surfaces (Kadirvelu and Namasivayam, 2003). Effects of pH on the uptake capacities of Pb^{2+} on LCF, MLCF1, MLCF2 and MLCF3 were investigated. Experimental studies were

carried out at temperature 30°C with initial Pb^{2+} concentration 25 mg L^{-1} using adsorbent dosage (adsorbent size 250-600 μm) 10 g L^{-1} at pH 2.0-7.0 and agitation speed 120 rpm for 30 min. This result is shown in Fig. 3.

From Fig. 3, the lead uptake is found to increase with increasing pH and it increases gradually in pH from 2.0-4.0. The increase in metal removal as pH increases can be explained on the basis of a decrease in competition between hydronium ions and metal species for the surface sites and also by the decrease in positive surface charge on the adsorbent, which resulted in a lower electrostatic repulsion between the surface and the metal ions and hence uptake of metal ions increase (Patnukao *et al.*, 2008). The suitable pH for Pb^{2+} adsorption on LCF and MLCF is 4.0. At higher pH, lead tends to hydrolyze and precipitate instead of adsorption and adsorbent was deteriorated with accumulation accumulation of matel ions, making true adsorption studies impossible (Guo *et al.*, 2009).

Furthermore, adsorption of Pb^{2+} by MLCF has been found to be higher than LCF. In the modification of LCF, NaOH could rapidly react with water and give Na^{2+} and OH^-. The OH^- was adsorbed on surface area of absorbent. It increased the attractive charge between Pb^{2+} ions and OH^- adsorbent.

Gardea-Torresdey *et al.* (1998) attempted to promote increased metal binding by the creosote biomass, the biomass was reacted with 0.5 M NaOH so that ester groups might be converted into carboxylate groups. The following chemical reaction that produces carboxylate groups from methyl esters is depicted below:

$$R\text{-}COO\text{-}CH_3 + NaOH \rightarrow R\text{-}COO^- + CH_3OH + Na^+ \quad (8)$$

The possible formed carboxylate groups tended to have a much higher metal binding ability than methyl ester groups. These results confirmed that the hydroxyl and matoxy groups (in carboxylic groups and aromatic rings) were involved in Pb^{2+}adsorption by LCF.

Effects of contact time on adsorption: The rate of adsorption take place is one of the parameters for economical wastewater treatment plant application. Consequently, it is important to establish the time dependence of such systems under various process conditions (Kadirvelu and Namasivayam, 2003). Effects of contact time on the removal of Pb^{2+} are presented in Fig. 4. Experiment studies were carried out at temperature 30°C with initial Pb^{2+} concentration 25 mg L^{-1} using adsorbent dosage (adsorbent size 250-600 μm) 10 g L^{-1} at pH 4.0 and agitation speed 120 rpm for contact time 0, 10, 30, 60, 120 and 300 min.

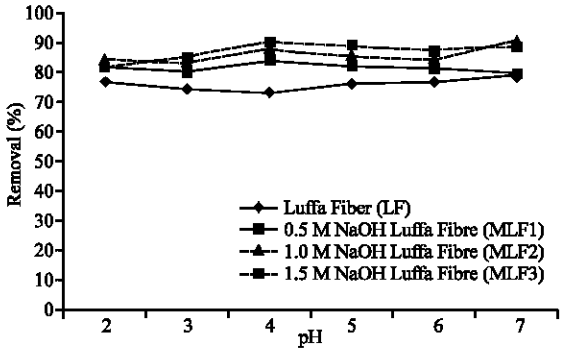

Fig. 3: Effects of pH on adsorption of Pb^{2+} by LCF and MLCF

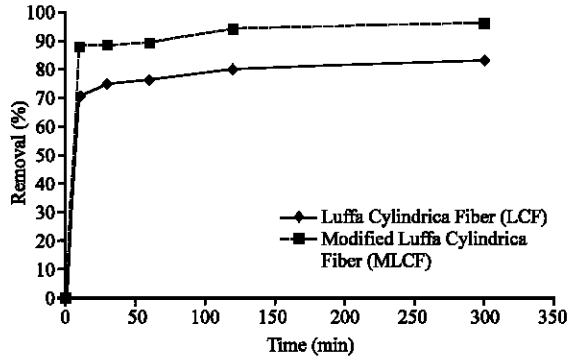

Fig. 4: Effects of contact time on adsorption of Pb^{2+} by LCF and MLCF

In Fig. 4, it can be seen that the rate of Pb^{2+} adsorption is found to be very rapid during the initial 10 min and thereafter, the rate of Pb^{2+} ions removal decreases considerably. No significant change in Pb^{2+} ions removal is observed about 30 min later. During the initial stage of adsorption, a large number of vacant surface sites are available for adsorption. After a lapse of some time, the remaining vacant surface sites are difficult to be occupied due to repulsive forces between the adsorbate molecules on the solid surface and in the bulk phase. Besides, the metal ions are adsorbed into the mesopores that get almost saturated with Pb^{2+} ions during the initial stage of adsorption (Lu *et al.*, 2009). In this study, the appropriate contact time for Pb^{2+} adsorption on LCF and MLCF is 30 min.

Effects of adsorbent size on adsorption: Effects of adsorbent size on the removal of Pb^{2+} are presented in Fig. 5. Experiment studies were carried out at temperature 30°C with Pb^{2+} initial concentration 25 mg L^{-1} using adsorbent dosage (adsorbent size ≤125, 125-250 and 250-600 μm) 10 g L^{-1} at pH 4.0 and agitation speed 120 rpm for 30 min.

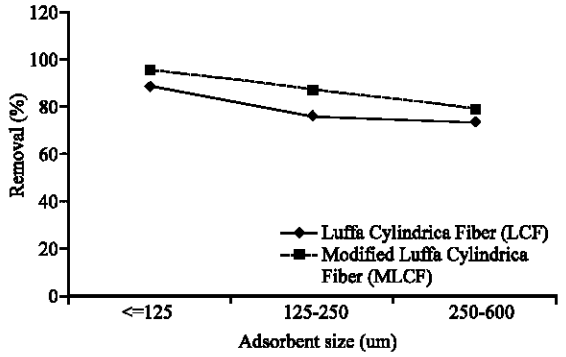

Fig. 5: Effects of absorbent size on adsorption of Pb^{2+} by LCF and MLCF

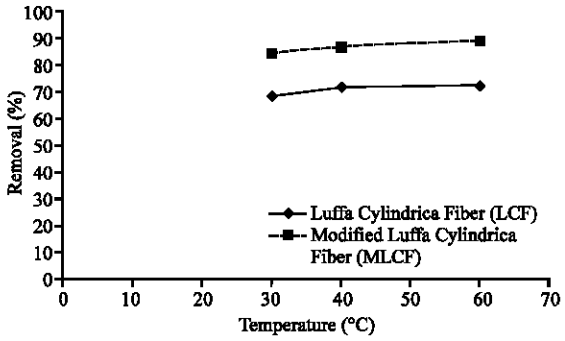

Fig. 6: Effects of temperature on adsorption of Pb^{2+} by LCF and MLCF

In Fig. 5, decreasing of adsorbent size leads to increase in surface area and the increase in the adsorption opportunity at the outer surface of the adsorbent. Besides, adsorption at the outer surface of the adsorbent there is also the possibility of intraparicle diffusion from the outer surface into mass transfer is greater for large particles. Because of various factors, such as diffusional path length or mass transfer resistance, contact time and blockage sections of the particle may not be utilized for adsorption, therefore, the adsorption capacity of large particles may be low (Ngeontae *et al*., 2007; Romero-Gonzalez *et al*., 2001; Shukla *et al*., 2002). The suitable adsorbent size for Pb^{2+} adsorption by LCF and MLCF is 250-600 μm.

Effects of temperature on adsorption: The temperature is an important in the context of adsorption on solid phase. Increasing the temperature is known to increase the rate of diffusion of the adsorbate molecules across the external boundary layer and in the internal pores of the solution. In addition, changing temperature will change the equilibrium capacity of the adsorbent for a particular adsorbate (Babarinde *et al*., 2006). Effects of temperature on the removal of Pb^{2+} are shown in Fig. 6. Experiment studies were carried out at temperature 30, 40 and 60°C with initial Pb^{2+} concentration 25 mg L^{-1} using adsorbent dosage (adsorbent size 250-600 μm) 10 g L^{-1} at pH 4.0 and agitation speed 120 rpm for 30 min.

From Fig. 6, the adsorption increases with an increase in temperatures indicates the increase in the mobility of large metal molecules with increasing temperatures and the ongoing adsorption process is endothermic. Increasing the temperature may produce a swelling effect within the internal structure of adsorbent enabling metal ions to penetrate further. That is the rise in sorption capacity with temperature is caused by the rise in the kinetic energy of adsorbent particles. Firstly, the collision frequency between adsorbent and adsorbate increases, this results in the enhanced sorption on to the surface of the adsorbent. Secondly, at high temperature due to bond rupture of functional groups on adsorbent surface there may be an increase in number of active sorption sites, which may also lead to enhance sorption with the rise in temperature (Guo *et al*., 2009). The optimal temperature for Pb^{2+} adsorption on LCF and MLCF is 40°C.

Effects of agitation speed on adsorption: Kinetic investigation is necessary for determining the rate of reaction and mechanism. Effects of agitation speed on the removal of Pb^{2+} are shown Fig. 7. Experimental studies carried out at temperature 40°C with initial Pb^{2+} concentration 25 mg L^{-1} using adsorbent dosage (adsorption size 250-600 μm) 10 g L^{-1} at pH 4.0 and agitation speed 50, 150 and 250 rpm for 30 min.

In Fig. 7, it can be seen that the rate of Pb^{2+} adsorption increases with increasing of agitation speed between 50-250 rpm due to the fact that better and more uniform mixing or there may possibly be some boundary layer resistance to reaction, which is decreased by increasing the agitation. The optimal agitation speed of this study was 250 rpm (Patnukao *et al*., 2008).

Effects of absorbent dose on adsorption: Effects of adsorbent dose on the removal of Pb^{2+} are shown in Fig. 8. Experimental studies were carried out at temperature 40°C with Pb^{2+} initial concentration 25 mg L^{-1} using adsorbent dosage (adsorption size 250-600 μm) 5, 10, 15 and 20 g L^{-1} at pH 3.0 and agitation speed 250 rpm for 30 min.

In Fig. 7, it shows the adsorption of Pb^{2+} as a function of LCF and MLCF dosages. It is apparent that increasing of the adsorbent dose causes to increase the amount of Pb^{2+} but decrease the amount adsorbed per unit mass, adsorption density. It is readily understood that the number of available adsorption sites increases by increasing the adsorbent dose and it therefore, results in

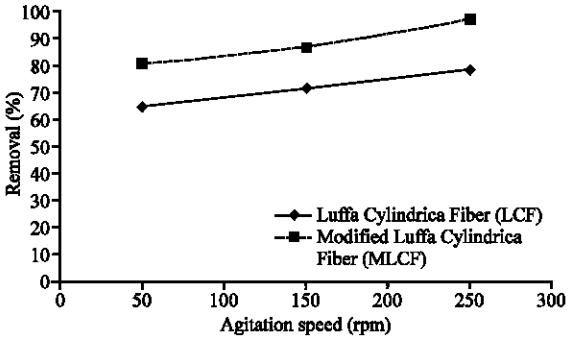

Fig. 7: Effects of agitation speed on adsorption of Pb^{2+} by LCF and MLCF

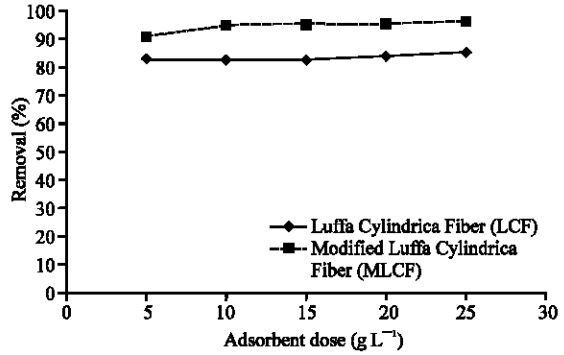

Fig. 8: Effects of adsorbent dose on adsorption of Pb^{2+} by LCF and MLCF

the increase of the amount of adsorbed Pb^{2+} The decrease in adsorption density with increase in the adsorbent dose is mainly because of unsaturation of adsorption sites through the adsorption process (Yu *et al*., 2003; Pehlivan *et al*., 2008) Another reasons may be due to the particle interaction, such as aggregation, resulting from high adsorbent dose. Such aggregation would lead to decrease in total surface area of the adsorbent and an increase in diffusion path length. The results can be concluded that the appropriate adsorbent dose is 10 g L^{-1}. After this dose, the increasing rate of Pb^{2+} removal decreases. Figure 8, the adsorption density decreases with increase in the adsorbent dose is mainly because of unsaturation of adsorption sites through the adsorption process. Another reason may be due to the particle intraction, such as aggregation, resulting from high adsorbent dose. Such aggregation would lead to decrease in total surface area of the adsorbent and an increase in diffusional path length (Shukla *et al*., 2002).

From Fig. 3-9, the results can give the appropriate condition for removing Pb^{2+} from 25 mg L^{-1} initial Pb^{2+}concentration by LCF and MLCF as pH 4.0, contact time 30 min, adsorbent size 250-600 μm, temperature 40°C,

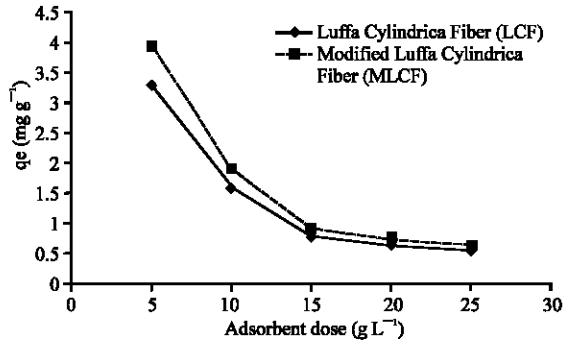

Fig. 9: Effects of adsorbent dose on Pb^{2+} adsorption capacity of LCF and MLCF

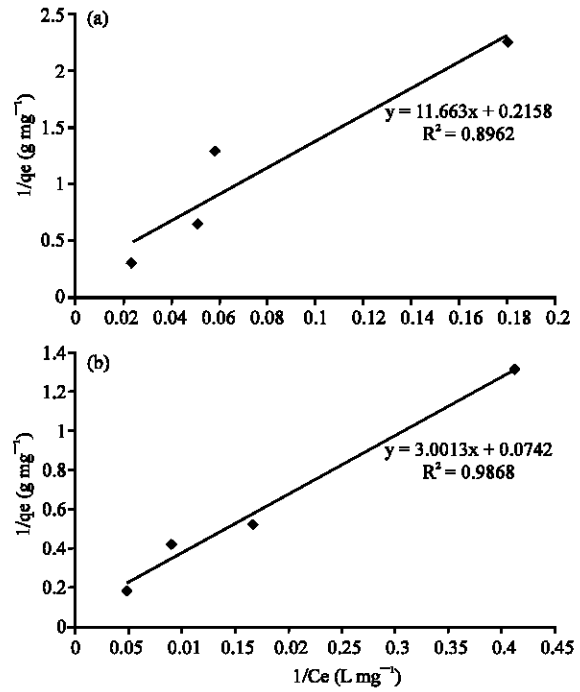

Fig. 10: Adsorption isotherms of Pb^{2+} adsorption by LCF and MLCF (a) Langmuir isotherm of Pb^{2+} adsorption by LCF (b) Langmuir isotherm of Pb^{2+} adsorption by MLCF

agitation 250 rpm and adsorbent dose 10 g L^{-1}. In this condition, the Pb^{2+} removal efficiency of LCF and MLCF are 82.72 and 94.93%, respectively.

Adsorption isotherm: Adsorption isotherm for adsorption of Pb^{2+} on LCF and MLCF, the results present that the experiment data were better fitted to the Fruendlich equation (R^2_{LCF} = 0.8962 and R^2_{MLCF} = 0.9868) than to the Langmuir equation (R^2_{LCF} = 0.8937 and R^2_{MLCF} = 0.9683). Thus, assumption of Fruendlich equation explain that

Table 1: Langmuir isotherm constants for adsorption of Pb^{2+} on LCF and MLCF

	Langmuir isotherm		
Adsorbent	q_{max}	b	R^2
LCF	4.63	0.025	0.8962
MLCF	13.48	0.019	0.9868

Table 2: Comparison of adsorption capacity of the adsorbent for the removal of Pb^{2+} with those other adsorbents

Adsorpbent	Adsorption capacity	References
Barley straw	23.2	Pehlivan *et al.* (2008)
Almond shells	2	Mehrasbi *et al.* (2009)
Alkali-modified almond shells	7	Mehrasbi *et al.* (2009)
Sawdust	3.19	Yu *et al.* (2001)
Palm kernel fiber	20.0	Ho and Ofomaja (2005)
Waste tea leaves	2.096	Ahluwalia and Gayal (2005)
Grape stalk	9.15	Martinez *et al.* (2006)
Baggase fly ash	2.50	Gupa and Ali (2004)
Luffa cylindrica Fiber (LCF)	4.63	This study
Modified *Luffa cylindrica* Fiber (MLCF)	13.68	This study

energies of adsorption onto a surface and no transmigration of adsorbate in the plane of the surface (Guo *et al.*, 2009) (Fig. 10a and b).

From Table 1, the adsorption capacity of LCF and MLCF are 4.63 and 13.48 mg g^{-1}.

Comparison of adsorption capacity with different adsorbents reported in literature: The adsorption capacities of the adsorbents for the removal of Pb^{2+} have been compared with those of other adsorbents reported in the literature and the values of adsorption capacities have been presented in Table 2. The values reported in the form of monolayer adsorption capacity. The experimental data of the present investigations are comparable with the reported values.

CONCLUSION

Luffa cylindrica fiber is an interested alternative adsorbent for heavy metals because of its useful to be alternative waste management and friendly with ecosystems. Moreover, it can be increased the effective of Pb^{2+} adsorption from LCF by NaOH modification. All adsorption parameters as pH, contact time, temperature, adsorbent size, agitation speed, adsorbent does and initial Pb^{2+} concentration have affect to the Pb^{2+} adsorption by LCF and MLCF. that the optimal condition for adsorption of lead (Pb^{2+}) by LCF and MLCF was pH 4.0, contact time 30 min, temperature 40°C, adsorbent size 250-600 μm, agitation speed 250 rpm, adsorbent dose 10 g L^{-1} and initial Pb^{2+} concentration 25 mg L^{-1}. In this condition, the adsorption ability of LCF and MLCF were 82.72 and 94.93%.

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