



## Biosorption of lead contaminated wastewater using cattails (*Typha angustifolia*) leaves: kinetic studies

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**Abstract:** In this work, dried leaves of *Typha angustifolia* (TA), also known as the common cattail, were used as an adsorbent in kinetic studies of Pb(II) adsorption from synthetic aqueous solutions. Batch adsorption studies with dried TA leaves were conducted and they were able to adsorb Pb(II) from 100 mL of a 25 mg L<sup>-1</sup> Pb(II) solution effectively with the optimized dosage of 0.6 g. Adsorption equilibrium was achieved within 8 h with an effective removal of 86.04 %. Adsorption kinetics was further evaluated using four kinetic models, i.e., the pseudo-first order, pseudo-second order, intraparticle diffusion and Elovich model. Fitting of the data was performed based on linear regression analysis. The sorption kinetic data fitted best to the pseudo-second order model with an *R*<sup>2</sup> of 0.9979, followed closely by the Elovich model with an *R*<sup>2</sup> of 0.9952. The obtained results showed the adsorption of Pb(II) by TA leaves, which is an abundant biological material, is feasible, cheap and environmentally friendly.

**Keywords:** adsorption; lead; kinetics; *Typha angustifolia*.

### INTRODUCTION

The presence of heavy metals, such as cadmium, chromium and lead, at toxic levels in rivers and streams near human habitats is a cause for concern. According to EPA National Primary Drinking Water Regulations, the maximum contaminant level (*MCL*) for cadmium is 0.005 mg L<sup>-1</sup>, chromium 0.1 mg L<sup>-1</sup> and lead 0.015 mg L<sup>-1</sup>.<sup>1</sup> An overdose of chromium in the body causes infertility while lead results in delayed physical and mental development in children and infants. Mining, tannery, chemical, metallurgical and the electronic and electrical manufacturing industries, which are usually located near rivers and streams, are the main contributors to heavy metal poisoning.<sup>2</sup> Various water treatment methods have been applied to reduce the presence of these metals such as chemical

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precipitation, flocculation, membrane separation, ion exchange and adsorption.<sup>3–5</sup> However, these methods have limitations and are costly to operate. Precipitation for example produces a large amount of sludge that requires treatment. Membrane filtration and ion exchange, though being effective, require high maintenance. Therefore, an alternative solution needs to be employed. Agricultural and biological waste materials, such as saw dust,<sup>6,7</sup> acorn waste,<sup>8</sup> rice husk ash,<sup>9</sup> tree leaves,<sup>10</sup> cassava<sup>11</sup> and spent grain,<sup>12</sup> have been proven to be cheap and biodegradable adsorbents that provide good loading capacities towards a wide range of heavy metals, such as zinc, lead, cadmium, copper, chromium and nickel. In this work, dried leaves of the narrow leaf cattail plant, *Typha angustifolia* (TA), a common aquatic plant with no economic value in the tropics, was used as a biosorbent for lead removal from synthetic wastewater.

This aquatic perennial plant offers a large amount of biomass that can be used for such purposes. It has been commonly used in constructed wetlands as an effective alternative for the treatment of septic effluents in small villages due its biomass.<sup>13,14</sup> Furthermore, the plants play an important role in metal removal via filtration, adsorption and cation exchange through plant-induced chemical changes within the rhizome.<sup>15</sup> This species used in constructed wetlands was reported to achieve a removal of 100 % for copper and zinc and 96 % for nickel.<sup>16</sup> Lead concentrations were found to be 13 mg kg<sup>-1</sup> in the root and 8 mg kg<sup>-1</sup> in the leaves of cattail.<sup>17</sup> This proves that the root of aquatic plants generally has a higher uptake capacity of these heavy metals as compared to the leaf. The same trend was also reported by for chromium, nickel, copper and cadmium.<sup>18</sup> Dried cattail leaves demonstrated effectiveness in adsorbing reactive dyes in synthetic wastewater due to the intracellular space within the leaf.<sup>19</sup> Pre-treating cattail leaves with formaldehyde, sulfuric acid and sodium hydroxide was found to improve the removal of Astrazon red 6B and Basilen red M5B by approximately 98 %.<sup>20</sup> In this study, the biosorption kinetics involved in the removal of lead in synthetic wastewater using the dried leaves of TA were investigated.

## EXPERIMENTAL

### *Biosorbent preparation*

Mature TA leaves were harvested from a pond in the University Malaysia Sabah campus, Sabah, Malaysia. The leaves are thick, ribbon-like structures which have a D-shaped cross-section showing air channels. These leaves were washed with pipe water to remove dirt and sand before rinsing with distilled water. Then the leaves were placed flat on a clean table to dry before drying in a hot air convection oven (Protech) at 103 °C for 72 h. The dried leaves were then sliced to a thickness of 2 cm and kept in an airtight container.

### *Pb(II) ion solution*

The stock solutions of Pb(II) were prepared by dissolving 1.599 g of Pb(NO<sub>3</sub>)<sub>2</sub> in 1 L of deionized water with 5 % v/v of HNO<sub>3</sub>. Both the Pb(NO<sub>3</sub>)<sub>2</sub> and HNO<sub>3</sub> were of AR grade obtained from Systerm, Malaysia. The solution was then diluted with deionized water for the



adsorption studies. The natural pH of the solution, pH 5.5, was maintained throughout the experiment. This pH was chosen to simulate the pH of water bodies contaminated with industrial effluents. Fresh dilutions were made prior to each experiment.

#### *Pb(II) metal analysis*

The residual concentration of Pb(II) in the solution was determined using a flame atomic absorption spectrophotometer (Perkin Elmer 4100) in triplicate. Each adsorption experiment meanwhile was conducted in duplicate. Single factor analysis of variance (ANOVA,  $\alpha=0.05$ ) was used to analyze the data in order to ascertain the statistical differences between the expected values and the observed values.

#### *Batch adsorption*

Batch adsorption experiments were conducted in 250 mL Erlenmeyer flasks with 100 mL of a 25 mg L<sup>-1</sup> Pb(II) solution and 0.6 g of dried TA leaves. The mixtures were shaken at room temperature on an orbital shaker (PROTECH) at 170 rpm. The biosorption kinetics involved was examined by varying the contact time of the sorbent (15–540 min). The solution pH was maintained at the natural pH of 5.5 throughout the experiments. The mixtures were then filtered using Advantech No. 9 filter paper and kept in stoppered plastic bottles. The final concentration of Pb(II) was then determined using FAAS. The percentage of removal and the amount of Pb(II) adsorbed onto a unit mass of TA was calculated according to Eqs. (1) and (2), respectively:

$$q = \frac{(c_0 - c_e)V}{m} \quad (1)$$

$$\% \text{ Removal} = \left( \frac{c_0 - c_e}{c_0} \right) \times 100 \quad (2)$$

where  $q$  is the amount of Pb(II) adsorbed per gram adsorbent, mg g<sup>-1</sup>,  $c_0$  is the initial Pb(II) concentration, mg L<sup>-1</sup>,  $c_e$  is the concentration of Pb(II) at equilibrium with the solid phase, mg L<sup>-1</sup>,  $V$  is the volume of the working solution used, L, and  $m$  is the mass of sorbent used, g.

#### *Kinetic modeling*

Kinetic models were used to determine the sorption mechanism of lead onto TA leaves and the rate controlling steps involved in the process. The kinetic models considered were the pseudo-first order equation, the pseudo-second order equation, the intraparticle diffusion equation and the Elovich equation. The fit of the data was evaluated based on the linear coefficient of determination,  $R^2$  and the Chi-square test. The Chi-square test Equation is given by:

$$\chi_t^2 = \sum \frac{(q_{e,\text{exp}} - q_{e,\text{calcd}})^2}{q_{e,\text{calcd}}} \quad (3)$$

where  $q_{e,\text{exp}}$  is the experimental sorption capacity at equilibrium, mg g<sup>-1</sup>, and  $q_{e,\text{calcd}}$  is the calculated sorption capacity at equilibrium according to the respective kinetic model, mg g<sup>-1</sup>.

*Pseudo-first order equation.* The pseudo-first order equation is generally expressed as:<sup>7</sup>

$$\frac{dq_t}{dt} = k_1(q_e - q_t) \quad (4)$$

where  $q_e$  and  $q_t$  are the sorption capacity at equilibrium and at time  $t$ , mg g<sup>-1</sup>, respectively, and  $k_1$  is the rate constant for pseudo-first order sorption, min<sup>-1</sup>. This model considers the rate of



occupation of adsorption sites to be proportional to the number of unoccupied sites.<sup>10</sup> The integrated form of this equation is:

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad (5)$$

*Pseudo-second order equation.* This model is based on the assumption that the rate of sorption is proportional to the square of the number of unoccupied sites.<sup>21</sup> The kinetic rate Equation is expressed as:<sup>7</sup>

$$\frac{dq_t}{dt} = k_2(q_e - q_t)^2 \quad (6)$$

Integrating Eq. (6) gives:

$$\frac{1}{q_e - q_t} = \frac{1}{q_e} + k_2 t \quad (7)$$

where  $q_e$  is the amount adsorbed at equilibrium, mg g<sup>-1</sup>, while  $k_2$  is the equilibrium rate constant for a pseudo-second order adsorption, g mg<sup>-1</sup> min<sup>-1</sup>. A linear graph can be plotted by linearizing Eq. (7) to give Eq. (8) as given below:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (8)$$

with  $k_2 q_e^2 = h$ , where  $h$  is the initial sorption rate, mg g<sup>-1</sup> min<sup>-1</sup>. This model is more likely to predict the behavior over the whole range of adsorption and is in agreement with chemisorptions being the rate controlling step.<sup>12</sup>

*Intraparticle diffusion (IPD) equation.* In this equation, a fractional approach to equilibrium is evaluated based on the function  $(Dt/r^2)^{1/2}$ , where  $r$  is the radius of an adsorbent particle and  $D$  is the effective diffusivity of the adsorbate within the particle. The initial rate is given by the linearized equation of the curve  $q_t = f(t^{1/2})$ :<sup>7</sup>

$$q_t = k_p t^{1/2} + C \quad (9)$$

where  $k_p$  is the IPD rate constant, mg g<sup>-1</sup> min<sup>-1/2</sup>, while  $C$  is the boundary layer thickness, mg g<sup>-1</sup>. A plot of  $q_t$  versus  $t^{1/2}$  will be linear if IPD is involved in the overall adsorption mechanism. If the line passes through the origin, i.e.,  $q_t = k_p t^{1/2}$ , IPD is the rate controlling step of the process.<sup>22</sup>

*The Elovich Equation.* The Elovich Equation is given below and is suitable for systems with heterogeneous adsorbing surfaces:<sup>23</sup>

$$\frac{dq_t}{dt} = \alpha \exp(-\beta q_t) \quad (10)$$

The linearized equation is given as:

$$q_t = \frac{1}{\beta} \ln(\alpha\beta) + \frac{1}{\beta} \ln t \quad (11)$$

where  $\alpha$  is the initial sorption rate, mg g<sup>-1</sup> min<sup>-1</sup> and  $\beta$  is related to the extent of surface coverage and the activation energy of chemisorption, g mg<sup>-1</sup>.

## RESULTS AND DISCUSSION

### Effect of adsorbent–adsorbate contact time

The duration of the batch experiments was varied from 15 to 540 min. The concentration of lead solution was 25 mg L<sup>-1</sup>. Volume of the solution was 100 mL,



while the quantity sorbent was 0.6 g. The pH of the solution was the natural pH of 5.5. As shown in Fig. 1 (1a and 1b), lead exhibited a rapid adsorption during the first 15 min. The percentage removal during that period was 41.96 %. This was then followed by an ongoing slow adsorption until equilibrium was achieved at 480 min. By then, the percentage of removal achieved was 86 %. Therefore, 480 min was fixed as the optimum contact time to attain equilibrium. Thus, the

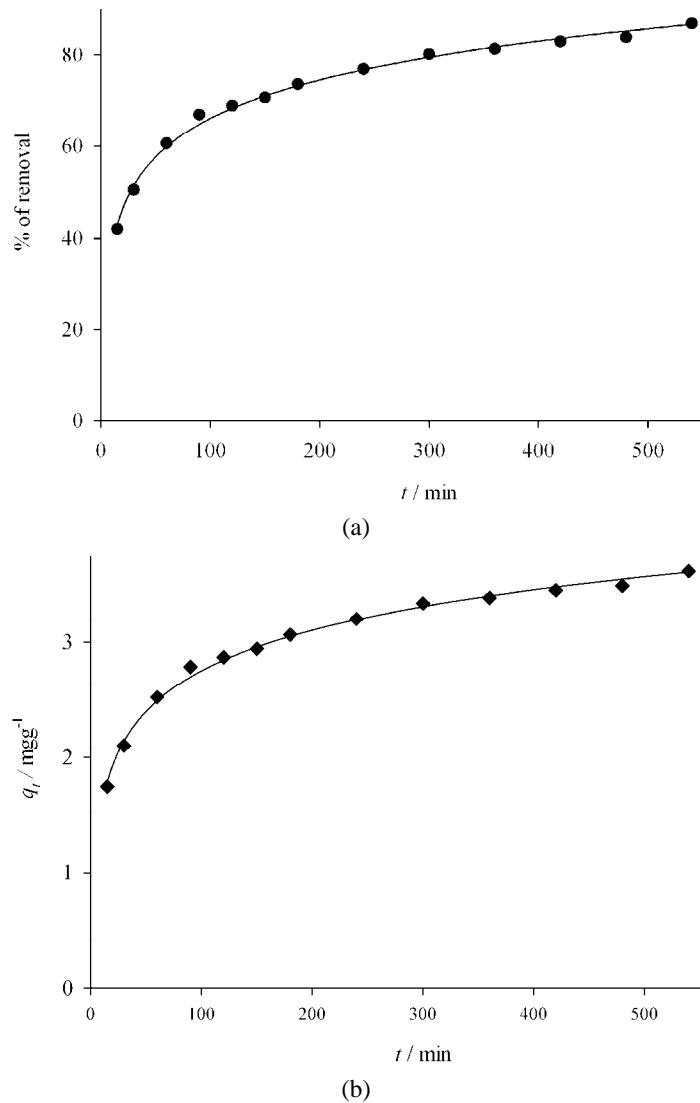


Fig. 1. a) Percentage removal of lead ions with respect to contact time. b) Amount of metal removed  $q_t$ ,  $\text{mg g}^{-1}$  with respect to contact time. The lead concentration remained at  $25\text{mg L}^{-1}$ , pH 5.5 with 0.6 g of TA leaves shaken at 170 rpm at room temperature.

experimental sorption capacity at equilibrium  $q_e$ , mg g<sup>-1</sup> was determined to be 3.620 mg g<sup>-1</sup>.

The adsorption of lead unto TA leaves occurred in two stages; an initial rapid uptake due to surface adsorption onto the TA leaves and a subsequent slow uptake due to diffusion of the lead ions onto the inner surface of the TA leaves. This was similar to the studies reported in the literature using different sorbents.<sup>24–26</sup>

The initial rapid uptake was due to the presence of vacant adsorption sites on the biomass. Sorption thus became less efficient as more adsorption sites were occupied by the metal ions, as shown in the slower stage.<sup>10</sup> This slow uptake is due to the diffusion of metal ions into the inner surfaces of the TA leaves due to repulsive forces between the ions on the leaf surfaces and the bulk phase.<sup>6,9,27</sup> The fast metal uptake by the sorbents is attributed to its highly porous and mesh-like structure that provides a large surface area for the sorption of lead onto the binding sites.<sup>28</sup>

#### *Adsorption kinetics models*

The study of the kinetics is important for evaluating the efficiency and mechanism of adsorption. In this study, four models were used, that is, the pseudo-first order, the pseudo-second order, the intraparticle diffusion and the Elovich Equation.

*Pseudo-first order.* The pseudo-first order kinetic model is commonly used for the adsorption of a solute onto an adsorbent. According to Eq. (5), a linear plot of  $\ln (q_e - q_t)$  versus  $t$  would confirm the fit of the first order kinetic model. The linear plot is shown in Fig. 2a. Ideally,  $\ln q_e$  should be equal to the intercept of the plot if the first order model were to be obeyed. In this work, the calculated  $q_e$  for the first order model deviated greatly from the experimental  $q_e$  value of 1.876 mg g<sup>-1</sup>. Furthermore, this model gave the lowest correlation coefficient,  $R^2$  using linear regression analysis as compared to the other models (Table I). According to Ho *et al.*,<sup>29</sup> the first-order model does not fit well for the whole range of contact times because the equilibrium sorption capacity,  $q_e$ , needs to be known, which is not the case as chemisorptions tends to become slow which causes the amount sorbed to remain significantly smaller than the equilibrium value. Thus,  $q_e$ , is usually obtained *via* trial and error. Therefore, the pseudo-first order model did not accurately represent of the adsorption kinetics in this study.

*Pseudo-second order.* The pseudo-second order model is given by Eq. (8), whereby a plot of  $t/q_t$  versus  $t$  should give a linear curve. This model is more likely to predict the behavior over the whole range of adsorption and it is in agreement with chemisorption being the rate limiting step.<sup>30</sup> The graphical interpretation of the data is shown in Fig. 2b, while the calculated  $q_e$  value, rate constant  $k_2$  (g mg<sup>-1</sup> min<sup>-1</sup>), the initial sorption rate,  $h$  (mg g<sup>-1</sup> min<sup>-1</sup>), the correlation



coefficient,  $R^2$  and Chi-square,  $\chi^2$ , are given in Table I. The correlation coefficient for this model was the best among all the considered models with  $R^2 = 0.9979$ , which shows that the second-order model fits well the data. The calculated  $\chi^2$  was also the lowest, further validating that this model best fits the experimental data. The calculated  $q_e \text{ calc}$  value of  $3.719 \text{ mg g}^{-1}$  agrees closely with the experimental  $q_{e,\text{exp}}$  value. This shows that the adsorption of lead onto TA leaves is second-order in nature, *i.e.*, chemisorption is the rate-controlling step involving valence forces *via* the sharing and exchange of electrons between

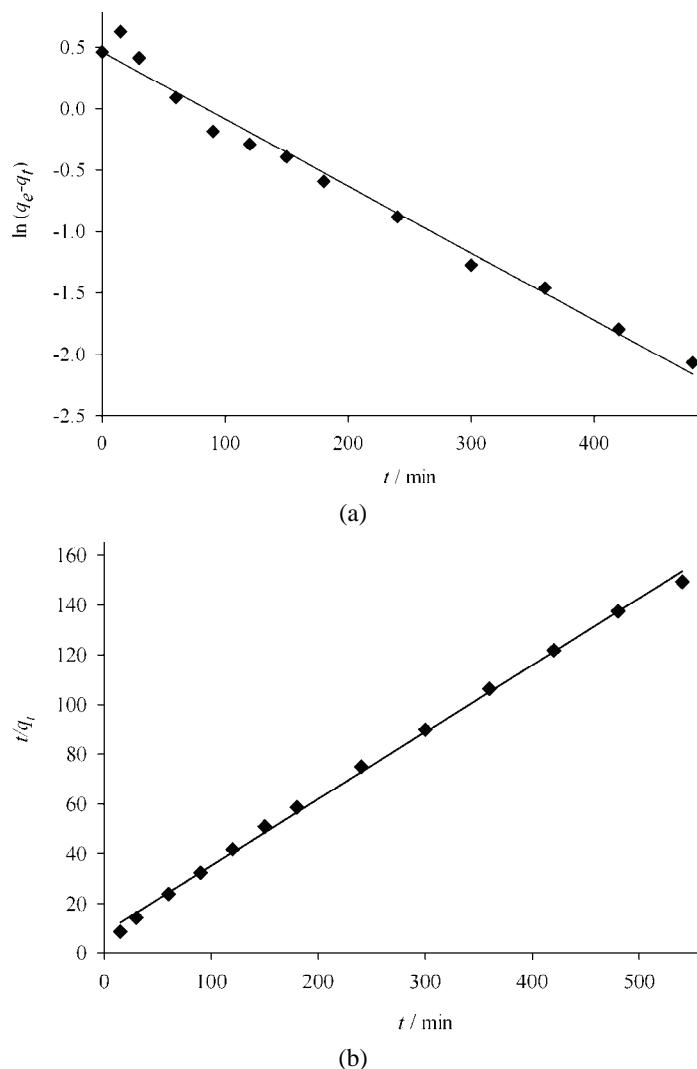


Fig. 2. a) Pseudo-first order kinetic plot for the adsorption of lead ions at room temperature.  
b) Pseudo-second order kinetic plot for the adsorption of lead ions at room temperature.

the lead and the TA leaves.<sup>31,32</sup> The obtained rate constant  $k_2$  was  $0.0087 \text{ g mg}^{-1} \text{ min}^{-1}$ .

TABLE I. Pseudo-first order, pseudo-second order, intraparticle diffusion and Elovich models for the adsorption of lead ions onto dried TA leaves at room temperature (experimental sorption capacity at equilibrium,  $q_{e,\text{exp}} = 3.620 \text{ mg g}^{-1}$ )

Model	Parameter	Value for Pb(II)
Pseudo-first order	$k_1 / \text{min}^{-1}$	0.0048
	$q_{e,\text{calcd}} / \text{mg g}^{-1}$	1.876
	$R^2$	0.9324
	$\chi^2$	74.849
Pseudo-second order	$k_2 / \text{g mg}^{-1} \text{ min}^{-1}$	0.0087
	$h / \text{mg g}^{-1} \text{ min}^{-1}$	0.032
	$q_{e,\text{calc}} / \text{mg g}^{-1}$	3.719
	$R^2$	0.9979
Elovich	$\alpha / \text{mg g}^{-1} \text{ min}^{-1}$	0.292
	$B / \text{g mg}^{-1}$	1.313
	$R^2$	0.51
	$\chi^2$	0.9952

*Intraparticle diffusion (IPD).* Adsorption is a multiple step process which is controlled by either one or more steps, e.g., film or external diffusion, pore diffusion, surface diffusion or a combination of more than one step. The IPD model investigates the possibility of diffusion contributing towards the adsorption of lead.

The intraparticle diffusion model is given by Eq. (9). A plot of  $q_t$  versus  $t^{1/2}$  should be linear if IPD is involved in the overall adsorption mechanism and the intraparticle diffusion rate constant  $k_p, \text{ mg g}^{-1} \text{ min}^{-1/2}$  can be calculated. If the plot of the graph were to pass through the origin, it is concluded that intraparticle diffusion is the rate controlling step.<sup>26</sup> For this work, the plot shown in Fig. 3 exhibits multi-linearity, indicating the adsorption process consisted of three steps as shown by the region k1, k2 and k3.<sup>8,9,26,33</sup> Table II shows the corresponding IPD rate constants,  $k_p$ , and the correlation coefficients,  $R^2$ , for the individual regions. A trend can be seen whereby  $k_{p1} > k_{p2} > k_{p3}$ , where  $k_{p1}$  is the rate constant for the first region depicting macropore diffusion, while  $k_{p2}$  depicts mesopore diffusion for region k2.<sup>9</sup> The rate constant  $k_{p3}$  for region k3 has the lowest rate constant as it is the final equilibrium stage where intraparticle diffusion starts to slow down due to the extremely low lead concentration left in the solution.<sup>34</sup> However, as the line does not pass through the origin, IPD is not the rate controlling step.<sup>35</sup> The intercept,  $C$ , shown for k1, indicating the boundary layer thickness, shows that rapid adsorption occurs within a very short time.<sup>36</sup>



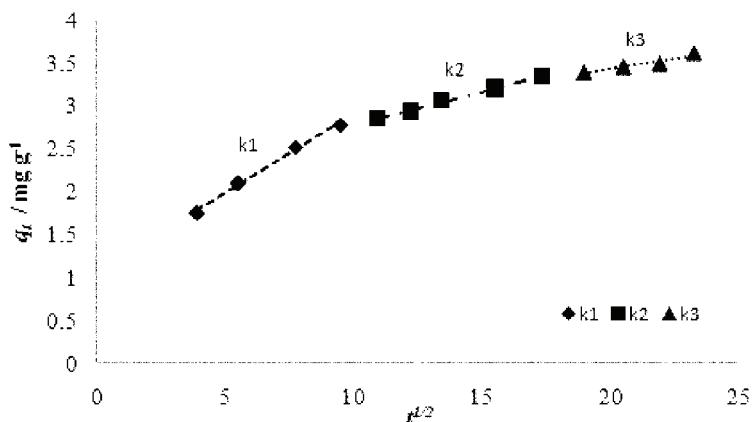


Fig. 3. Intraparticle diffusion kinetic plot for the adsorption of lead ions at room temperature exhibiting multi linearity in the regions k1, k2 and k3.

Table II. Kinetic parameters based on the intraparticle diffusion model exhibiting multi-linearity. Three regions of multi linearity were shown, i.e., k1, k2 and k3

Region	$k_p / \text{mg g}^{-1} \text{ min}^{-1/2}$	$R^2$
k1	0.1847	0.9946
k2	0.0754	0.9908
k3	0.0452	0.936

*The Elovich Equation.* The linearized Elovich Equation as shown by Eq. (10) was used to plot a graph of  $q_t$  versus  $\ln t$  obtained by integrating Eq. (11) and applying the boundary conditions  $q_t = 0$  and  $t = 0$ .<sup>37</sup> The Elovich Equation gave a high linear correlation,  $R^2 = 0.9952$  as shown in Fig. 4. The values of  $\alpha$ , the ini-

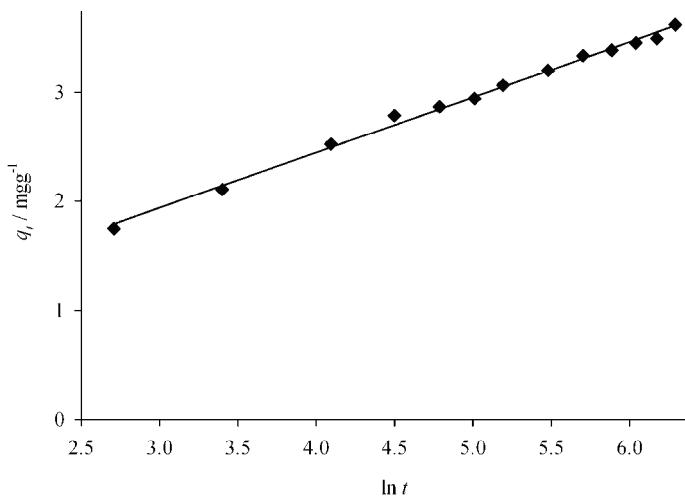


Fig. 4. Elovich kinetic plot for the adsorption of lead ions at room temperature.

tial sorption rate,  $\text{mg g}^{-1}\text{min}^{-1}$  and  $\beta$ , the extent of surface coverage and activation energy of chemisorptions,  $\text{g mg}^{-1}$ , are given in Table I. This equation is commonly used to determine the kinetics of chemisorption of gases onto heterogeneous solids; thus, it is quite restricted as it only describes a limiting property ultimately reached by the kinetic curve.<sup>38</sup> This is obviously shown by the huge  $\chi^2$  value as compared to the other kinetic models (Table I). This proves that the Elovich Equation is not a suitable representation of the adsorption process, despite giving excellent linearity, as was also found by Chang *et al.*<sup>39</sup>

#### CONCLUSIONS

In this study, the biosorption of lead from contaminated synthetic wastewater using dried TA leaves was successfully performed. The adsorption kinetics, evaluated using pseudo-first order, pseudo-second order, intraparticle diffusion and Elovich models, proved that the pseudo-second order model describe the experimental data the best. This was further validated by the excellent linearity and lowest  $\chi^2$  value. Thus, chemisorption was determined as the rate controlling step for the adsorption process. Regarding the Elovich model, the high  $\chi^2$  value shows that the model does not fit well with the experimental data despite the excellent linearity. Therefore, the Elovich model was not suitable for describing the adsorption kinetics in this work.

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И З В О Д

БИОСОРПЦИЈА ИЗ ОТПАДНЕ ВОДЕ ЗАГАЂЕНЕ ОЛОВОМ ПОМОЋУ ЛИСТОВА РОГОЗА (*Typha angustifolia*): КИНЕТИЧКА СТУДИЈА

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У овом раду су коришћени осушени листови биљке *Typha angustifolia* (ТА), познате и као рогоз, као адсорбент у кинетичкој студији адсорпције Pb(II) у синтетичким воденим растворима. Спроведена су испитивања шаржном адсорпцијом која су показала да је ТА у стању да делотворно адсорбује Pb(II) оптимизованом дозом од 0,6 г. Адсорпциона равнотежа је постигнута у року од 8 часова са ефективним процентом уклањања од 86,04 %. Затим је одређена кинетика адсорпције помоћу кинетичких модела као што су псеудо-први ред, псеудо-други ред, интрачестична дифузија и Elovich модел. Апроксимација тачака је урађена на основу линеарне регресије. Подаци о кинетици сорпције најбоље су одговарали моделу псеудо-другог реда са  $R^2$  од 0,9979 и само мало лошије Elovich моделу са  $R^2$  од 0,9952. Из резултата се може закључити да је адсорпција Pb(II) помоћу ТА, који је обилан биолошки материјал, изводљива и повољна за животну средину.

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