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Biosorption of lead (II) from an aqueous solution using biosorbents prepared from water plants

E.Tseveendorj[1], T.Enkhdul[1], S.Lin[2], D.Dorj[3], Sh.Oyungerel[4], T.O.Soyol-Erdene[1]*

[1]*Department of Environmental and Forest Engineering, School of Engineering and Applied Sciences, National University of Mongolia, Ulaanbaatar 14201, Mongolia*
[2]*Institute of Oceanography, National Taiwan University, Taipei 106, Taiwan (ROC)*
[3]*Department of Chemical and Biological Engineering, School of Engineering and Applied Sciences, National University of Mongolia, Ulaanbaatar 14201, Mongolia*
[4]*Department of Biology, School of Sciences and Arts, National University of Mongolia, Ulaanbaatar 14201, Mongolia*

*Corresponding author: soyolerdene@seas.num.edu.mn

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ABSTRACT

Due to its toxicity causing serious health problems, persistence in the environment and non-biodegradability, lead (Pb) is considered as one of the most harmful metals on earth. In this study, dried aquatic plants as sorbents including *Nymphoides peltata* (NP), *Typha laxmannii* (TL), and *Eichhornia crassipes* (EC) were examined and compared to discover the best biosorption for Pb. The effect of physical and chemical parameters including pH (2.0–5.5), sorbent dosage (1–5 g/l), metal concentration (20–100 mg/l), and contact time (~240 min) were investigated to determine the optimal condition for Pb(II) biosorption. As a result, the optimum pH, sorbent dosage, and contact time were 5.0, 1 g/l, and 120 minutes, respectively. Pb^{2+} biosorption data were found to follow the Langmuir isotherm model while the kinetic biosorption data followed pseudo-second-order model. The maximum biosorption capacity from Langmuir model was calculated as 63.3, 82.9, and 51.9 mg/g for EC, NP, and TL, respectively. All the results showed that biosorption efficiencies of Pb(II) by different biosorbents were in following order NP>EC>TL.

Keywords: biosorption, heavy metal, lead, *Eichhornia crassipes, Nymphoides peltata, Typha laxmannii*

INTRODUCTION

Heavy metals are considered as a priority environmental pollutant, due to the toxicity of metal ions in living organisms [1]. After release from various sources, heavy metals are non-degradable and highly persistent in the environment [2, 3]. These sources are industrial and agricultural activities, such as metal-containing pesticides, semiconductor production, mining and many other heavy machinery industries [4]. In the recent cases in the world, in Flint, Michigan, for example, thousands of people were potentially exposed to lead through drinking water due to the use of lead in the old plumbing. Between 6000 and 12000 children have been exposed to drinking water with high levels of lead and they may experience a range of serious health problems. After the change in water source, since 2014, the percentage of Flint children with elevated blood-lead levels may have risen from about 2.5% in 2013 to as much as 5% in 2015 [5]. Therefore, it is important to treat heavy metals at the source of discharge to prevent environmental toxification and specially health hazard [3].

Lead (Pb) is one of the most toxic metals that can be found in the environment [6]; it is considered an "A group carcinogenic" by the United States Environmental Protection Agency (U.S. EPA), and the International Agency for Research on Cancer (IARC) [1]. It is also one of the first metals used by man on a large scale. Although it is not a nutritionally essential element, its monitoring is important because of its toxicity to human health. Most of the lead produced in metallic form: in batteries, cable sheathing, sheets and pipes, etc., is recovered and recycled, but most lead used in compound form: like paints and petrol additives is lost to the environment, eventually ending up in the aquatic environment. Lead compounds, similar to the ones used in petrol additives, are reportedly being used in the production of mercurial fungicides [7]. In drinking water, the presence of the element should not be over the maximum allowed limit of 0.015 mg/l by the United States Environmental Protection Agency and 0.05 mg/l by World Health Organization [8]. Combustion of coal in electric power plants and industrial, commercial, and residential burners is the major source of anthropogenic lead. Combustion of leaded gasoline is also estimated to be the major source of lead to the urban atmosphere and soils. It is estimated that the largest discharges of lead are found in the soil followed by discharges found in water [7].

Currently, chemical, physical, and biological methods are implicated for the removal and stabilization of metals (including Pb) in the environment. Commonly used methods for the treatment of metals are precipitation, electrochemical treatment, membrane technologies, lime coagulation, solvent extraction, membrane filtration, reverse osmosis, ion exchange, and adsorption [9]. Each process has its own merits and limitations in application, so these traditional metal removal methods have certain disadvantages. In some cases, these methods are too expensive, inefficient, and release toxic waste [9]. Thus, it is essential to develop alternative methods for the successful treatment of metals which are low-cost, efficient, and nature-friendly.

Biosorption is one of the methods to treat heavy metals using renewable biomaterials. The growing international demand for proper adsorbents has led to a search for new, readily available, and low-cost materials [10]. In recent years, the adsorption of heavy metals on the surfaces of various natural biomasses, including dry plant leaves [11], rice husks [12, 13], barks [14], waste tea leaves [15], and many other locally abundant biomaterials have been studied [9]. Because it is known that aquatic plants, both living and dead, are heavy metal accumulators, several recent studies have investigated the removal of heavy metals by using biomaterials prepared using dried plant species [16, 17]. Subsequently, the application of biosorbent materials prepared from aquatic plants for the removal of heavy metals from wastewater has gained widespread interest in environmental remediation [18]. The selection of an optimal plant species for metal removal includes a variety of factors, such as the yield of biomass, the ease of growth of the plant, and the ease of harvest under the conditions of application [9].

It has been well-established that *EC* and its dried biosorbents, possesses a strong ability to remove heavy metals from aqueous solution, with the sorption performances vary depending on the metal species, concentration, and solution chemistry [16, 19, 20]. A high sorption of heavy metals, such as cadmium (Cd), zinc (Zn), chromium (Cr), lead (Pb), and copper (Cu), by *EC* or its dried materials has been reported in previous studies [19, 21-25]. In relatively colder regions, such as Mongolia, the application of dried biomasses from naturally abundant plants which can be used as effective sorbent for metal removal is important to minimize the environmental problems related to mining activities. However, studies in this respect to NP and TL are scattered and the underlying sorption availability of locally abundant aquatic plants for metal removal is still very scarce, so far. The aim of the present work is to investigate the performance of dried *NP* and *TL* in removing Pb^{2+} ions from aqueous solution, which, compared with commercial *EC*, are in great supply, inexpensive, and easily found around Central Mongolia. The performance of these two species for any of the heavy metal removal is not documented in the available literature. The influence of initial concentration and contact time, related isotherm and kinetic models were investigated in batch mode for a better understanding of the biosorption process. Fourier transform infrared spectroscopy (FT-IR) study was employed to analyze the possible mechanism of biosorption. The research findings are accommodating to apply new and effective biosorbents for the removal of Pb and other heavy metals, is regarded as priority pollutant due to its non-biodegradability, toxicity and persistence in the environment.

EXPERIMENTAL

The optimum condition with maximum efficiency for Pb(II) sorption were determined at several parameters including solution pH, sorbent dosage, and initial concentrations of Pb were examined. The kinetic study for biosorption processes was also performed to explain possible sorption mechanism. The experimental conditions for each set of experiments are illustrated in Table 1.

Table 1. General conditions to influence study

Influence study	**V_s, ml**	**pH**	**Bio sorbent dosage, g/l**	**C_{Pb}, mg/l**	**Contact time, min**
pH	50	2-5.5	1	50	120
Biosorbent dosage	50	5	1-5	50	120
Initial metal ion concentration	50	5	1	20-100	120
Kinetics or contact time	100	5	1	50	240

Preparation of biosorbents: In this study, three different types of water plants' biomass, *EC, NP,* and *TL,* were used as biosorbent materials. *NP* and *TL* were collected from Tuul river, in an inactive Zaamar gold mining area in Mongolia, whereas the reference water plant *EC* was purchased from the market. All plants were washed thoroughly three times with tap water followed by rinsing in distilled water to separate dirt from the samples and then dried at 105°C for 10 hours until they obtained a constant weight. The dried plants were then powdered using agate mortar and pestle. The powdered biosorbents were passed through a sieve of mesh size 1 mm and stored in a desiccator until further use. Infrared Spectra were recorded using Fourier Transform Infrared Spectrometer (Bruker, FT-IR - Alpha T, USA) to determine key functional groups of three biosorbents for biosorption of Pb^{2+} ion.

Preparation of lead (II) solutions: A stock solution of Pb^{2+} (1000 mg/l) was prepared by dissolving the required quantity of analytical grade lead nitrate ($Pb(NO_3)_2$) using double distilled water and stored in an acid washed high density polyethylene (HDPE) bottle after acidification by concentrated HNO_3. Then the desired concentrations of diluted working solutions were prepared using volumetric flasks and adjustable pipettes.

Batch sorption experiments: Adsorption experiments were carried out to study the influence of contact times between Pb^{2+} solution and biosorbents, pH values of solutions, sorbent dosages, and metal concentrations, on the removal efficiency of biosorbents at room temperature. All batch sorption experiments were carried out in a 250 mg/l Erlenmeyer flask containing 0.05 g of biosorbents. Desired concentrations of 50 mg/l Pb^{2+} ion solutions were used for batch experiments and each experiment was performed for 120 minutes at 150 rpm on an orbital shaker (Dragon Lab, Orbital shaker- SK -O180-E, PRC). After agitation, the solutions containing residual of Pb^{2+} ion were filtered slowly through paper filter. The residual Pb^{2+} ion concentration was analyzed using a Flame atomic absorption spectrometry (Thermo Elemental, Flame-AAS -Solar M5, USA) at the Laboratory of Atomic Spectroscopy, National University of Mongolia. All analyses were repeated, and the results were presented as an average. The biosorption efficiency was calculated using following equation:

$$BE,\% = \left(\frac{C_0 - C_t}{C_0}\right) \times 100\% \qquad \textbf{(1)}$$

where:

BE - biosorption efficiency (%)
C_0 - initial concentration (mg/l),
C_e - equilibrium concentration (mg/l).

The biosorption capacity (BC, mg/g) of each biosorbent was identified using the following equation:

$$BC = \frac{C_0 - C_t}{m_{BS}} \qquad \textbf{(2)}$$

where:

m_{BS} - biosorbent mass (g).

Effect of pH: Studies on the effect of solution pH (pH of the solutions adjusted by - Lida instrument, PHS-3C Precision pH/mV meter, PRC) on the biosorption efficiency were performed within the pH range of 2.0–5.5. The pH of the solutions was adjusted by the addition of HNO_3 and NaOH solutions. Lead (Pb^{2+}) ion precipitated as forming lead hydroxide at pH 6.0 and 7.0, therefore, the experiments were not performed for these pH range.

Effect of sorbent dosage: The effect of sorbent dosage on the biosorption efficiency varied from 1 to 5 g/l and this study was conducted at 50 mg/l of Pb^{2+} initial concentration, pH 5.0, under 150 rpm shaking for 120 min.

Effect of initial metal ion concentration: The metal uptake onto biosorbents as a function of concentration was studied at fixed pH value (pH 5.0) and biosorbent dosage (1 g/l) by the concentration of Pb^{2+} ranged from 20–100 mg/l and shook for 120 minutes of equilibrium time. Equilibrium data were then tested by Langmuir and Freundlich isotherm models to determine biosorption mechanism. The linear equations are shown in Table 2.

Biosorption kinetics: The kinetic studies were conducted in 250 ml conical flasks containing 1 g/l of biosorbents and 100 ml of 50 mg/l Pb^{2+} ion solution at an optimum pH value (pH 5.0) for a duration of 240 minutes. Three ml of the solutions were sampled out at appropriate time intervals (5, 10, 15, 20, 25, 30, 60, 120, and 240 minutes) to 15 ml centrifuge tubes. Then the samples were centrifuged (SUM YAU instruments, 80-2 Centrifuge, PRC) at 4000 rpm for 5 minutes to separate solutions from the residual sorbent materials. After the separation, 2.5 ml of solution was taken out and diluted by 10 times for further analysis. The most common kinetic models, pseudo-first order and pseudo-second order, were employed to describe sorption kinetics. Linear pseudo-first order and pseudo-second order equations are presented in Table 3.

Table 2. Linear equations of Isotherm models

Isotherm model	Linear equation	Parameters
Langmuir isotherm	$\frac{C_e}{q_e} = \frac{C_e}{q_m} + \frac{1}{q_m K_L}$	q_e (mg/g) and q_m (mg/g) are the biosorption capacity at equilibrium and maximum biosorption capacity calculated from the equation, respectively; C_e (mg/l) is equilibrium concentration; K_L is Langmuir constant; K_F and n are the Freundlich constants
Freundlich isotherm	$log q_e = log K_F + \frac{1}{n} log C_e$	

Table 3. Linear equations of Kinetic models

Kinetic model	Linear equation	Parameters
Pseudo-first order	$ln(q_e - q_t) = ln q_e - k_1 t$	q_e (mg/g) and q_t (mg/g) are biosorption capacity at equilibrium and time t (min); k_1 (1/min) and k_2 (g/mg min) are the pseudo-first and pseudo-second order rate constants
Pseudo-second order	$\frac{t}{q_t} = \frac{t}{q_e} + \frac{1}{k_2 q_e^2}$	

RESULTS AND DISCUSSION

FTIR spectrum of prepared biosorbents: Fourier transform infrared spectroscopy (FT-IR) is widely used to study the surface groups of adsorbents and to determine those groups accountable for metal ion removal [26]. The FT-IR spectra of three biosorbents before Pb(II) biosorption are shown in Figure 1.

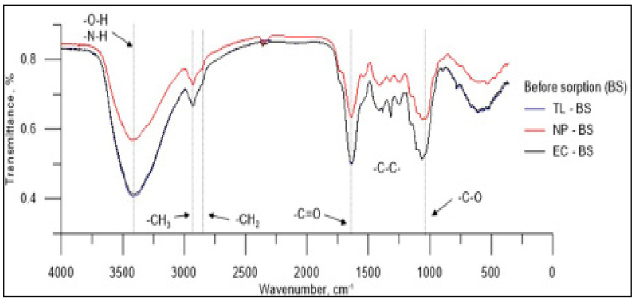


Fig. 1. IR spectra of biosorbents before Pb(II) biosorption

As can be observed, all three biosorbents had similar trends of intensity, and displaying number of absorption peaks, which indicates the presence of different types of functional groups in the biosorbent. The broad peak at 3416 cm^{-1} can be attributed to the stretching vibration absorption band of the O–H or N–H groups present in alcohols, phenols, primary and secondary amides [27, 28]. The peaks at 2925 cm^{-1} can be assigned to asymmetric CH_2 group vibration of lipid moiety. The peak located at 1750 cm^{-1} is a characteristic of carbonyl group of aldehydes, ketones, carboxylic acids, esters or amides [29]. The peaks at 1635 cm^{-1} indicate the presence of a –C=C bond. The peaks at 1244 cm^{-1} is related to –C–O bond or P- and S-containing compounds [28], and 1065 cm^{-1} reveal the –C–O bond of carboxylic acid or C–O–P stretching of phosphodiesters [28, 30].

Effect of pH on biosorption: As shown in Figure 2, the biosorption efficiency of Pb(II) was low at acidic condition (pH 2.0-3.0) for each EC, NP, and TL biosorbents, 61.9%, 80.0%, and 40.5%, respectively. At low pH, compared with Pb^{2+} ion concentration the amount of proton (H^+) ions was high, which suppresses the biosorption process on the surface of biosorbents [30]. While from pH 4.0 the biosorption efficiency increased to 81.6%, 85.1%, and 71.4% for EC, NP, and TL, respectively. This means that the competing H^+ ion concentration decline resulting in better biosorption efficiency [27, 31]. There was no significant difference on biosorption efficiency at pH 4.5 to 5.5. At higher pH, above 5.5, lead precipitated as a hydroxide. The highest sorption efficiency of Pb^{2+} ion for all three biosorbents was obtained around the pH value of 4.5 – 5.0, which is similar with the results of previous studies used other biosorbent materials [32, 33]. Consequently, pH 5.0 was selected as an optimal value for further experiments. Amongst the three chosen biosorbents, NP showed the highest biosorption efficiency at this stage and the removal efficiency of biosorbents was decreased in following order: NP>EC>TL at pH 5.0.

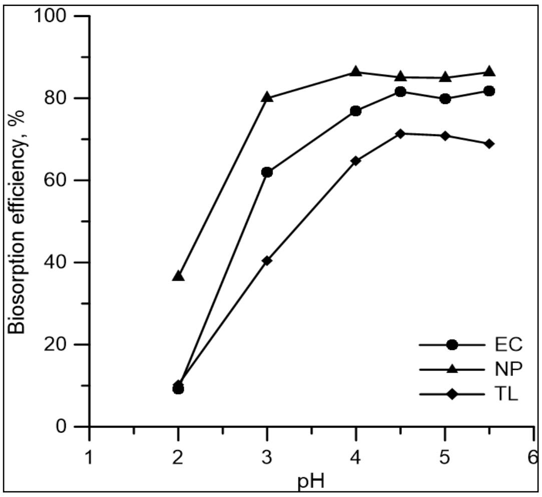


Fig. 2. Influence of pH on biosorption efficiency

Effect of sorbent dosage on biosorption process: The experimental results of sorbent dosage effect on Pb(II) biosorption efficiency and adsorbed amount are described in Figure 3 (a, b).

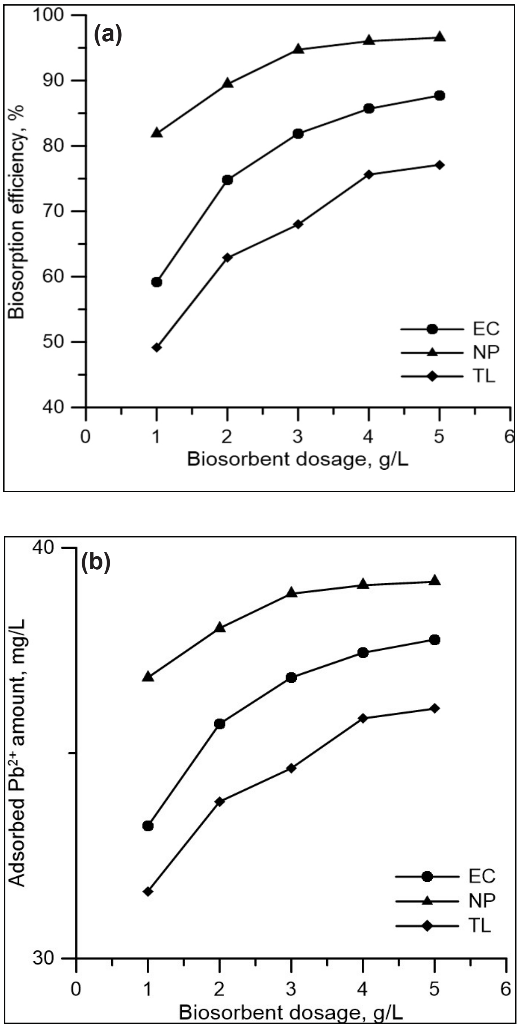


Fig. 3. Influence of sorbent dosage (a) on biosorption efficiency and (b) on adsorbed amount

As seen from figures, the biosorption efficiency and adsorbed amount were increased with the increase of biosorbent dosage from 1 g/l to 5 g/l. The accessibility of more active sites and functional groups for biosorption of Pb^{2+} ions resulted in high biosorption efficiency [26, 27]. Afterwards, when biosorbent dosage increased from 3 g/l to 5 g/l the biosorption efficiency was decreased due to the overlapping or aggregation of active sites at higher dosages [27, 34]. The biosorption efficiency of biosorbents were declined in the following order NP>EC>TL. For NP, biosorption efficiency of Pb^{2+} at dosages 3, 4, and 5 g/l was an almost stable 97.9–98.6%. While the other two biosorbents showed a bit of increasing removal stabilizing at 4 and 5 g/l dosages. Similar trend was observed in other studies that with increase of biosorbent dosage the biosorption efficiency decreased [35]. Increasing amount of biosorbent in solution caused biosorbents to combine with each other and larger particles were formed, which resulted in the decrease of biosorption potential as well as surface area.

Three biosorbents (EC, NP, and TL) had high biosorption capacity at 1 g/l biosorbent dosage, 33.2 mg/g, 36.8 mg/g, and 31.6 mg/g, respectively. Since the biosorbent increase is causing just a little difference on efficiency, economically, it is best to use slight amounts of biosorbents to remove more pollutants. Therefore, the optimum dosage for each biosorbent of 1 g/l were selected for further experiments.

Effect of initial Pb^{2+} ion concentration: In Figure 4 (a) and (b), the impacts of initial Pb^{2+} ion concentration on biosorption efficiency and on adsorbed lead amount are illustrated. From the Figure 3 (a), increasing initial metal concentration resulted in decrease of efficiency, which is due to the fewer binding sites at a given biosorbent dosage. Biosorption efficiency was high when metal concentration was low. On the other graph, as the initial metal concentration increases the adsorbed amount of lead (Pb^{2+}) has begun saturating from 50 mg/l due to its decrease in binding sites responsible to the adsorption of lead. Their adsorbed amount was decreased by following order NP>EC>TL.

At the maximum concentration of 100 mg/l of Pb^{2+}, the biosorption efficiency was decreased by the following order NP>EC>TL (75.1> 62.0> 50.7%, respectively). Also, from the following figure, it can be easily seen that the NP remained, even in the higher metal concentration, with high removal efficiency. The results show that for high pollution level, NP is the most suitable biosorbent for Pb(II) removal which has almost 10–25% greater removal efficiency than the other two at this concentration. Besides that, two different isotherm models, which are Langmuir and Freundlich isotherm, were applied, and the results are described in Table 4 and Figure 5. As for Langmuir isotherm model, it assumes that the sorption goes on a homogenous surface without any sorbate trans-migration, while the Freundlich isotherm describes monolayer adsorption with lateral interaction between adsorbed species on the surface.

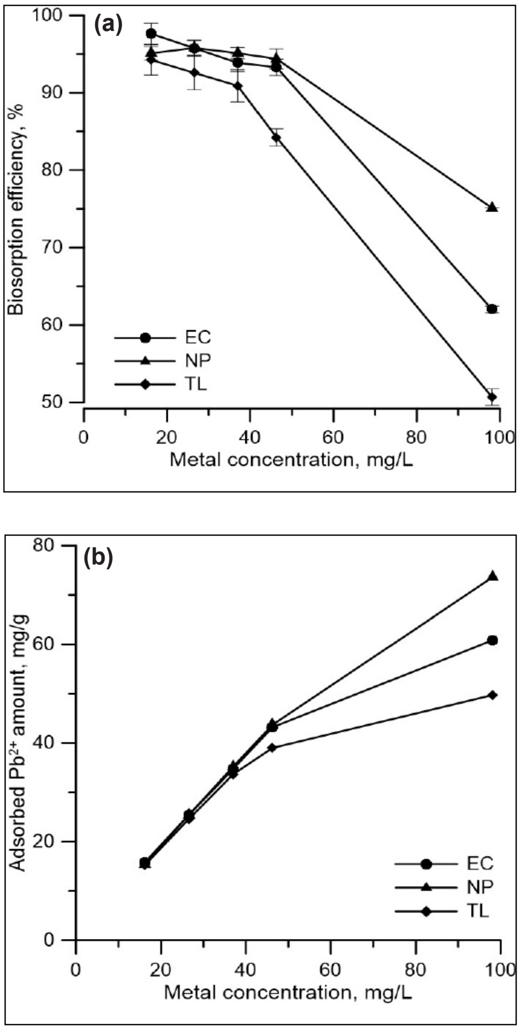
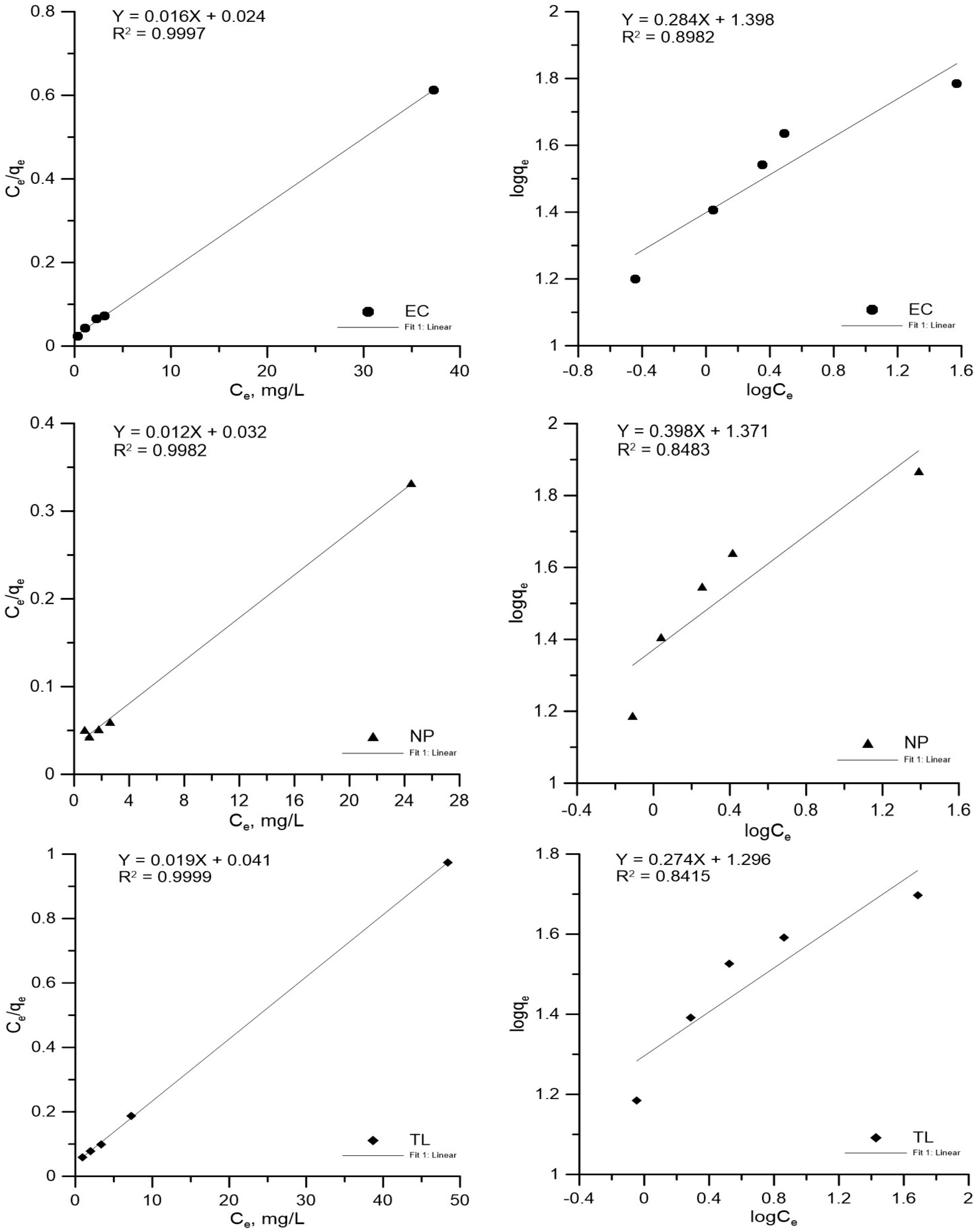


Fig. 4. Influence of metal ion concentration on biosorption efficiency (a) and on adsorbed lead amount (b)

The information deduced from Langmuir isotherm plots showed that q_{max} values for NP was higher than that for EC and TL. The best fit to the experimental data from Langmuir isotherm model were deduced from the higher correlation coefficients that all three biosorbents active sites were homogenously distributed. From the Freundich model, lower correlation coefficients were observed suggesting that the model was not appropriate. The comparison of biosorption capacity of three biosorbents for Pb(II) with various biomasses is presented in Table 4. It was found to be comparable and higher than those of many corresponding sorbents in literature [27, 28, 34, 36, 37]. The maximum biosorption capacity range of biosorbents were 51.9–82.9 mg/g, which is 6-10 times higher when comparing with the previous similar research that used a Lipton tea leaves as a biosorbent [32]. Consequently, it can be revealed that EC, NP, and TL have a considerable potential for the removal of Pb(II) from aqueous solution.

Table 4. Langmuir and Freundlich isotherm constants compared to reference studies

Reference	**Biosorbent**	**Metal**	**Langmuir isotherm**			**Freundlich isotherm**		
			q_{max}, mg/g	**K_L, l/mg**	**R^2**	**K_F, mg/g**	**1/n**	**R^2**
Altangerel A	Lipton tea	Pb	7.81	0.519	0.989	10.78	0.018	0.990
Lodeiro P	*Cystoseria baccata*	Pb	182.34±4.14	0.053±0.014	0.980	-	-	-
Morosanu L	*Rapeseed biomass*	Pb	21.29	0.400	0.997	3.02	1.06	0.989
Moyo M	*Marula seed husk*	Pb	20	0.52	0.999	1.65	0.568	0.974
	EC		63.3	0.667	0.999	25.04	0.284	0.898
This study	NP	Pb	82.9	0.381	0.998	23.51	0.398	0.848
	TL		51.9	0.471	0.999	19.78	0.275	0.841



Langmuir isotherm

Freundlich isotherm

Fig. 5. Langmuir and Freundlich isotherm curves of each biosorption process

Biosorption kinetics: Removal efficiencies of EC and TL have stabilized from initial 30 minutes of contact time, while NP has reached to the equilibrium from around 120 minutes, which is being far slower than others (Figure 6). This can be elucidated by the large availability of the active sites that can be easily accessed [28]. Compared with NP, the other two biosorbents EC and TL have low active sites resulting in a rapid saturation of biosorbents and indicating as stable biosorption efficiency.

However, by comparing the sorption efficiency for each biosorbent, the NP has performed the best results. And, once again, the end results of this experiment from the highest to the least efficient was in following order NP>EC>TL, which verifies the previous results in terms of sorption efficiency.

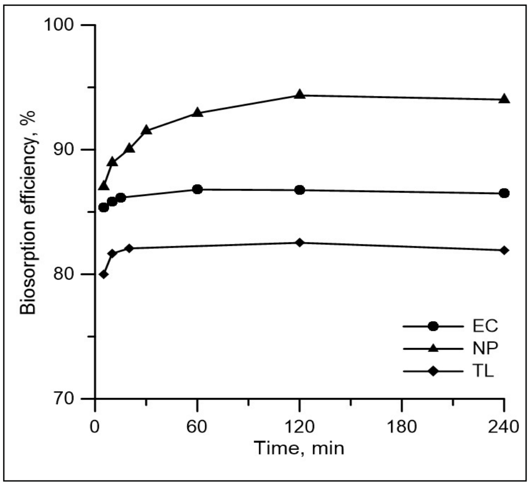


Fig. 6. Biosorption kinetic curve

The rate controlling steps which include mass transport, chemical reaction processes and biosorption mechanism can be effectively investigated by different kinetic models. In this study pseudo-first-order and pseudo-second-order order models were used for determining the mechanism of the biosorption process as shown in Table 5, and in Figure 7.

From the data of sorption kinetic study, some of the values were calculated using the pseudo-I and pseudo-II order equations. Pseudo-I order equation suggests that there is a weak physical sorption between sorbent-sorbate system, while the pseudo-II order equation suggests that there is a strong chemical sorption. The coefficients of determination for the pseudo-I order were low for all three biosorbents, while correlation coefficients (*R^2*) values of pseudo-II order kinetic model were 1.000 for all three biosorbents. As seen from these graphs and the table of results, the rate of biosorption is clearly determined by the pseudo-II order equation, and this indicates that the sorbents used in this study are going under strong chemisorption process with the Pb^{2+} ion. For the removal of lead by other biosorbents, a similar reaction rate was determined as a pseudo-II order process [30, 38]. According to the pseudo-II order kinetic model the biosorption of lead ions onto EC, NP, and TL are based on chemical reaction, involving sharing or exchange of electrons between sorbent and sorbate [28, 37]. The pseudo-II order kinetic model also can be confirmed by the initial results of FT-IR spectroscopy (Figure 1), which demonstrates binding sites of biosorbents. The complexity of the biosorption process can be demonstrated as in Figure 8.

The high affinity of biosorbents for lead ions most likely due to various mechanisms operating simultaneously: electrostatic interactions, and chemical interactions, comprising ion exchange reactions (with H^+ of the carboxyl and hydrohyl groups or K^+, Ca^{2+}, Mg^{2+}). Binding sites of biosorbent for lead ion must be either and or both hydroxyl or carboxyl groups forming …–O–Pb… like combination or with amide groups in amino acids [28, 37].

Table 5. Pseudo first and second order kinetic constants compared to reference studies

			Pseudo-I order			Pseudo-II order		
Reference	**Biosorbent**	C_0, mg/l	q_e, mg/g	k_1, min^{-1}	R^2	q_e, mg/g	k_2, g/(mg*min)	R^2
Altangerel A	Lipton tea	50	15.9	0.006	0.973	16.05	0.006	0.999
Sharain-Liew	*Typha angustifolia*	-	1.88	0.005	0.932	3.72	0.009	0.998
Hao Chen	*Cinnamomum camphora*	100	7.83	0.113	0.920	43.75	0.074	0.999
Morosanu L	*Rapeseed biomass*	50	2.43	11.28	0.973	6.79	8.81	0.999
This study	EC	50	0.35	-0.008	0.315	39.38	- 1.472	1.000
	NP	50	2.38	-0.012	0.908	43.01	0.033	1.000
	TL	50	0.49	-0.003	0.148	37.29	- 0.405	1.000

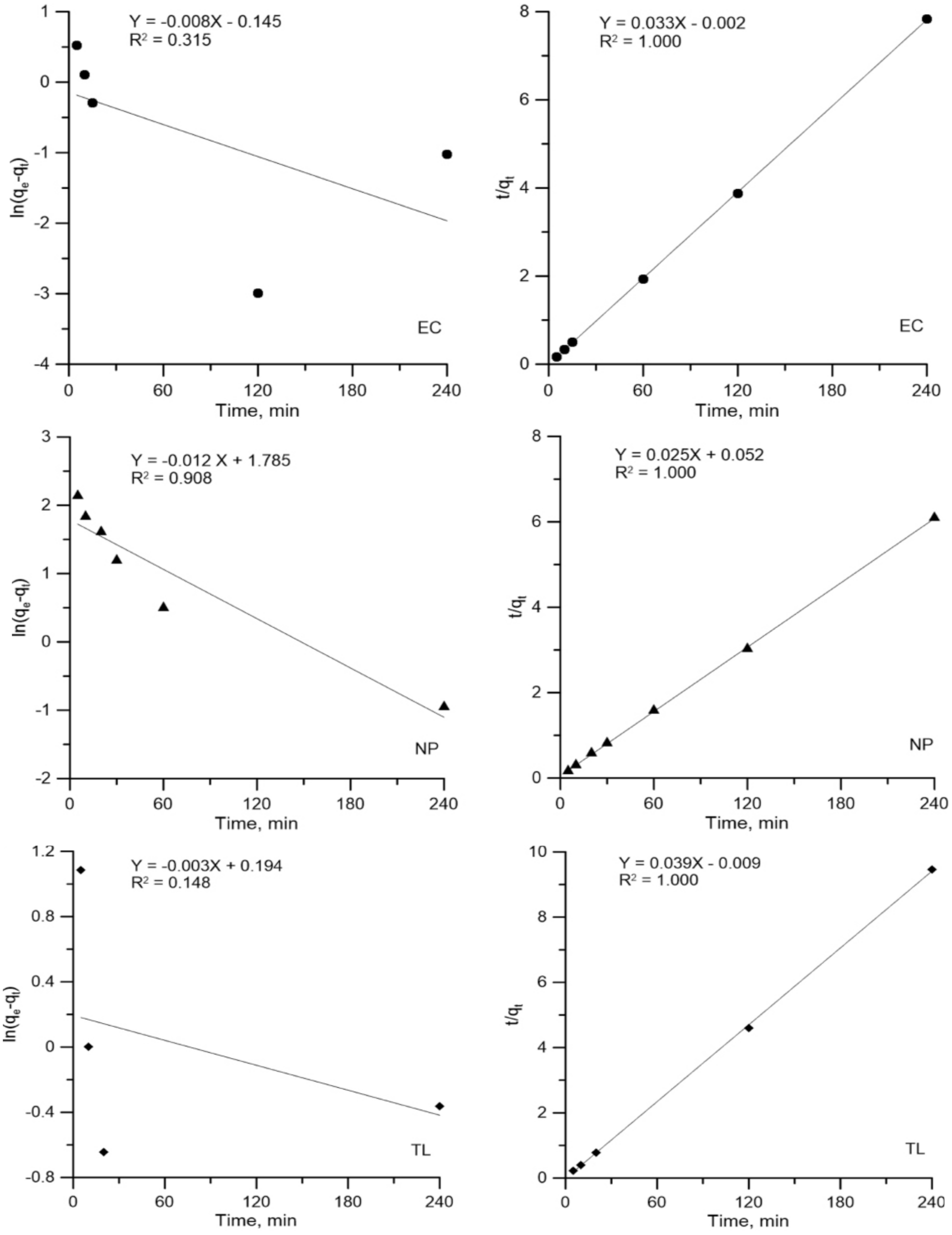


Fig. 7. Pseudo first and second order kinetic curves

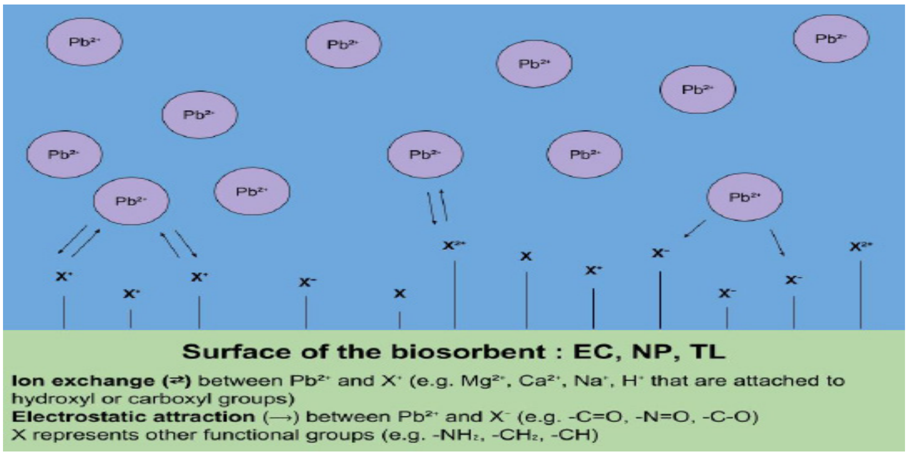


Fig. 8. Schematic illustration of possible interactions involved in the Pb^{2+}/biosorbent sorption process

CONCLUSION

The optimum conditions for biosorption of Pb^{2+} ion using the three different biosorbents were determined to be a solution of pH of 5.0, biosorbent dose of 1 g/l. Langmuir isotherm shows best fit for the adsorption data on the three biosorbents, which indicates that sorption processes involve monolayer formation on the surface at equilibrium. The maximum biosorption capacity for each biosorbent was 63.3 mg/g, 82.9 mg/g, and 51.9 mg/g for EC, NP, and TL respectively.

Kinetic analysis revealed that biosorption processes best fit to pseudo-second-order kinetics, which means that Pb^{2+} biosorption undergoes the chemisorption process.

Finally, from the three biosorbents, NP was the best sorbent material for the removal of Pb^{2+} from the solution. Therefore, this plant should be studied for the removal of other heavy metals which are discharged from mining and other industrial activities. The further research will be conducted to characteristics of adsorption-desorption cycles to metals recovery and to reutilize of sorbent biomasses.

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