Mechanism, Equilibrium, and Kinetics of the Adsorption of Pb(II) onto Chemically Treated Lemon Grass from Aqueous Solution

Section A -Research paper

Mechanism, Equilibrium, and Kinetics of the Adsorption of Pb(II) onto Chemically Treated Lemon Grass from Aqueous Solution

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Abstract:

In the laboratory-scale experiment, the potential of an inexpensive lignocellulosic refused lemon grass biomaterial was converted into adsorbent for the removal of lead ions from their aqueous solutions. The Lemon grass (LG) converted into adsorbent by treating with alkali solutions of NaOH (sodium hydroxide) at concentrations of 5 wt% at 80 °C for 2 h. Labelled as TLG, the treated LG departs. It was studied how the initial Pb(II) concentration (10–150 mg/l), pH (1-6), contact period (5–240 min), and adsorbent dosage (0.1–1.5 g) affected the lead adsorption. The maximum adsorption capacity was shown to be 47.61 mg/g, which was closer to the experimental capacity (45.5) when the kinetic models for the overall adsorption rate were compared. The adsorption system was best represented by the pseudo second-order kinetics. The adsorption isotherm that Langmuir's model might match. Equilibrium was reached in 240 minutes thanks to the comparatively quick adsorption process. At pH 5, Pb(II) adsorption was at its highest. According to the experimental findings, complexation is one of the main adsorption mechanisms for attracting Pb(II) ions to sorbents.

Keywords: Pb(II), adsorption, lemon grass, batch studies, mechanism

1. Introduction

The reduction of environmental contamination has always drawn a lot of interest. The challenge of cleaning up toxins from water and wastewater has gotten worse as industrialization has accelerated. Environmental contaminants like lead have been around for a while. Large amounts of wastewater that is polluted with lead are produced by process industries such those that make batteries, print and pigment, plate and finish metal, manufacture ammunition, soldering materials, ceramic and glass products, and iron and steel [1-4]. The hazardous effects of lead on the aquatic environment and the possibility of contaminating water resources intended for human use are what drive attempts to reduce lead concentration in discharge wastewaters. Lead in drinking water must not exceed 0.015 mg/L at this time [5]. Adsorption technology is currently being used widely for the removal of heavy metals from aqueous solutions because it is a cleaner, more effective, and less expensive technology. Acute lead poisoning in humans causes serious damage to the kidneys, liver, brain, and nervous system, while long-term exposure may cause sterility, abortion, and neonatal death [6, 7]. Recent years have seen a surge in interest in the biosorption of heavy metals from solutions employing various biomaterials as adsorbents. Both dead and active biomass, among the numerous resources in biological wastes, demonstrate

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particularly intriguing metal-binding abilities [8]. A lot of research has been done recently on using agricultural byproducts to remove metals from water. Numerous recent publications have described the removal of lead using a variety of low-cost and widely accessible bioadsorbents, including agricultural by products like Humulus lupulus [9], Pinus sylvestris saw dust [10], black gramme husk [11], Oriza sativa husk [12], rice husk [13], and lemon grass leaves [14] among others [15]. A significant crop that produces scent is lemon grass (Cymbopogon flexuosus), which is a member of the gramineae family. Due to the high citral content (75–85%) in its oil, lemon grass produces essential oil with a lemony fragrance. It stands two to two and a half metres tall and has dark green or yellowish green foliage. 1.5 cm diameter by 1 metre long leaf blade. De-oiled lemon grass is a fibrous substance with the cellulose, hemicellulose, and lignin components listed in Table 1.

Table 1. Physicochemical characteristics of the LG.

Parameter	Values
Bulk density (g/cc)	0.54
Ash (%)	11.0
Moisture (%)	6.72
pH	6.5
Specific surface area (m^2g^{-1})	11.56

The goal of this inquiry is to use oil-exhausted lemon grass as a batch mode lead ion sorbent from aqueous solution and lead removal process. Lemon grass is a desirable and reasonably priced choice for the biosorption removal of dissolved metals. There is still a demand for new, highly efficient and affordable adsorbents. In general, compared to traditional systems, biosorptive processes might lower capital costs by 20%, operational expenses by 36%, and overall treatment costs by 28% [16]. According to the literature review, lemon grass has not yet been employed as an adsorbent to remove lead (II). To clarify the equilibrium adsorption behaviour of Pb(II), values of well-known kinetics and isotherms studies have been carried out in the current work. Additionally, researchers have looked into how contact time, pH, concentration, and dosage affect adsorption.

2. Materials and Methods
2.1 Preparation of adsorbate
Dissolving 1.598 g of $Pb(NO_3)_2$ in 100 ml of demineralized water (DMW) and quantitatively diluting to 1000 ml with DMW, a standard stock solution of Pb(II) (1000.0 mg/l) solution was made from its nitrate salt (Thomas Bekar, India). By combining a stock solution with DMW to dilute it, various lead(II) concentrations were created. The glassware that was utilised was submerged in 10% (v/v) HNO_3 and repeatedly washed with DMW. Sodium acetate/hydrochloric acid and sodium acetate/acetic acid were used as buffer solutions to change the pH of working solutions from 1-3 and 4-6, respectively.

2.2 Preparation of Adsorbent

Lemon grass (LG) was obtained at a field distillation unit (F.D.U.) following the extraction of essential oils from the Fragrance and Flavour Development Centre (F.F.D.C.) in Kanpur, Uttar Pradesh, India. To get rid of any clinging dirt, the LG was washed with water. Before being ground into 60-100 mess size, they were first dried for 72 hours at 50 0C in an oven. The dried LG powder was first dewaxed by solvent extraction in a soxhlet device using hexane for six hours. The dried, dewaxed LG 8g was treated with 200 ml. of 0.1 M NaOH solution and the solution mixture was stirred for 30 min and left for 24 hours at room temperature. Alkali treated solution mixture was filtered using buchner funnel and washed again thoroughly with deionized water (DIW) to remove the presence of excess containing alkali. The residue was dried in a hot air oven at 75°C for 12 h. This alkali-treated material was stored in desiccator and abbreviated as TLG.

3. Experimental Method

Numerous variables, including pH of the solution, initial metal ion concentration, contact time, and amount of the adsorbent, influence the adsorption of metal ions by the active sites on the surface of TLG. Based on the outcomes of various early experiments, the following parameters particle size, adsorbent dosage, contact time, pH, and agitation speed were chosen and maintained constant throughout the investigation, unless otherwise noted: 130 m, 5 g/l, 240 min, and pH 5.0. In Erlenmeyer flasks set in an orbital shaker at 30 0C, tests were conducted. The LG was typically exposed to 20 ml of Pb (II) solution (100 mg/l) at a concentration of 0.1 g. The suspension was filtered after 240 minutes of contact time, and the filtrate was then tested for Pb (II) using a spectrophotometric technique with 1, 5-diphenyl thiocarbozone in aqueous micellar solutions [17]. The following equation was used to calculate the intake of Pb (II):

$$q_e = \left(\frac{C_i - C_e}{m} \right) V \qquad (1)$$

where V (l) denotes the volume of the solution, m (g) denotes the weight of the adsorbent utilised, and C_i and C_e (mg/l) denote the initial and final concentrations of Pb(II) in solution, respectively.

4. Results and Discussion

4.1 FTIR Charactrization

The interaction between an adsorbate and the active groups on the surface of the adsorbent can be studied using the FTIR spectroscopy technique. The chemical makeup of the TLG is used to interpret the FTIR in this adsorption behaviour. At 3396 cm^{-1}, the TLG exhibits a robust inter/intra-hydrogen bonded (O-H) stretching absorption. In cellulose, there are hydrogen bonds that are both intra- and intermolecular. Figure 1 depicts the precise structure of this hydrogen bond network. In addition, the fingerprint region between 1500 and 600 cm^{-1} contains a large number of clearly defined peaks: Unconjugated C=O in hemicellulose has a length of 1737.71

cm^{-1}, lignin and carbohydrates have a length of 1463 cm^{-1}, cellulose and hemicellulose have a length of 1426 cm^{-1}, cellulose has a length of 1425 cm^{-1}, syringyl rings have a length of 1246 cm^{-1}, C-O stretch has a length of 1049 cm^{-1}, and C-H deformation has a length of 607 cm^{-1}. Figure1 displays the FTIR of TLG and TLG-Pb(II). There were obvious band shifts and intensities in comparison to the FTIR spectra before and after adsorption. These bands, which are functional groups of TLG, take part in the adsorption of lead as illustrated in Table 2.
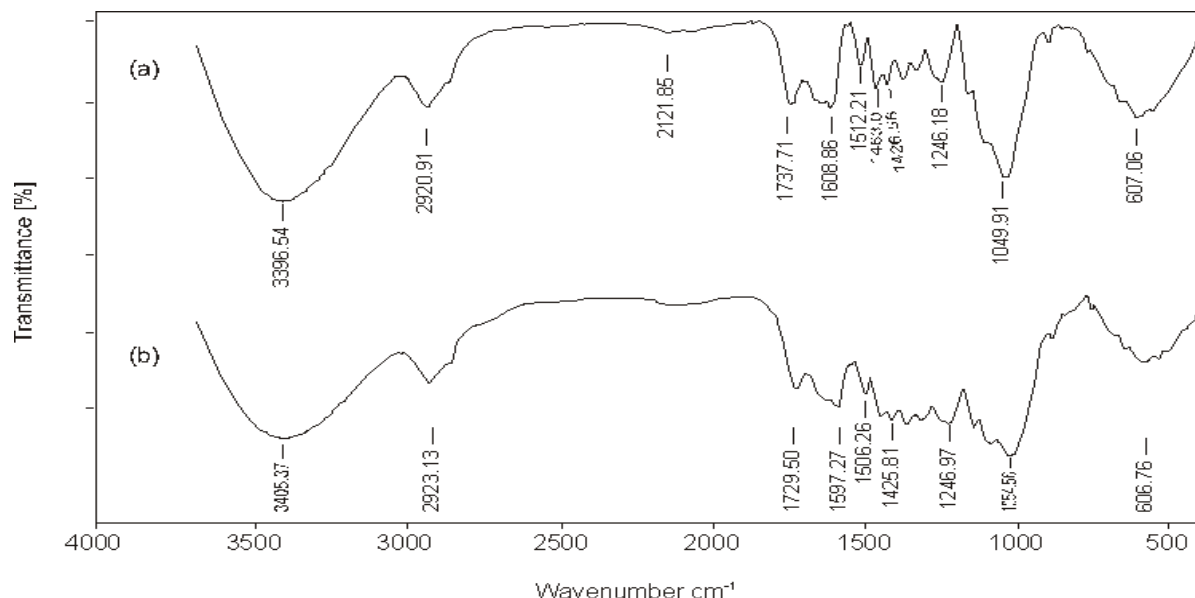

Figure 1. FT-IR spectra of TLG (a) before and (b) Pb(II) adsorbed.

Table 2. FT-IR spectral characteristics of LG before and after adsorption of Pb(II).

IR peaks	Adsorption bands (cm^{-1})		Differences	Assignment
	Before adsorption	**After adsorption**		
1	3396	3404	+7	O–H stretching vibration
2	2920	2923	+3	C-H vibration of CH_3
3	1737	1729	-8	C=O carbonyl vibration
4	1463	1506	+43	Phenyl propane skeleton vibration
5	1426	1425	-1	C–H aromatic –CH_3vibration
6	1049	1054	-5	C–O stretching (alcohalic)vibration
7	607	606	+1	C-H deformation in cellulose

4.2 Impact of contact time

The impact of contact time on lead adsorption is seen in Figure 2. Pb(II) is absorbed in greater quantities when contact time is prolonged. The initial rapid rate of adsorption that occurred during the first few minutes of contact was typically followed by a slower rate until equilibrium was attained. This was caused by the presence of a large number of unoccupied active sites,

however as adsorption continued, these active sites gradually became saturated over time. The pace of movement of the adsorbent particles from the exterior to the interior site controls the rate of absorption as the surface adsorption sites are depleted [18]. Additionally, as a 24 hour increase in contact time had no appreciable impacts, the time needed to establish equilibrium was closer to 240–300 min. As a result, the remaining tests were kept at 240 minutes of contact time.
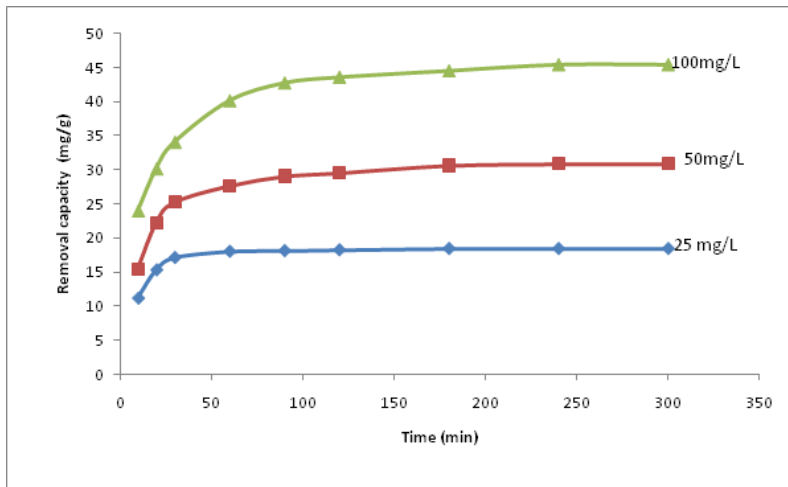

Figure 2. Effect of contact time on the adsorption of Pb(II)onto TLG.

4.3 Impact of pH

In the adsorption process, the pH of the aqueous solution is a crucial regulating factor. The presence of negatively charged groups at the sorbent surface is required for the metal to bind. Since H^+ and H_3O^+ compete with metal ions for the system's high net positive charge at pH 2, active sites get protonated to the virtual exclusion of metal binding on the sorbent surface. Accordingly, the attraction between the sorbent and the metal cations is reduced at increasing H^+ concentrations because the sorbent surface becomes more positively charged [19]. The availability of additional negatively charged surfaces expands as the pH rises, allowing for more metal uptake. However, if the pH rises more, the solubility of the metals drops, causing them to precipitate as hydroxides and lowering the sorbent's ability for sorption. According to research done at various pH levels, Figure 3 shows how the pH affects the elimination of lead. As can be observed, the pH has a significant impact on the ability of TLG to bind lead(II). The most lead(II) is successfully eliminated at pH 5. The removal of lead by other vegetable materials, like P. sylvestris [10], and crop milling waste [11], has also been noted to follow the similar pattern. The LG sorbent has a sorption capacity comparable to other chemically treated agricultural and industrial materials for the removal of lead ions from aqueous solutions. Minimal physicochemical conditioning is TLG's advantage in this situation.
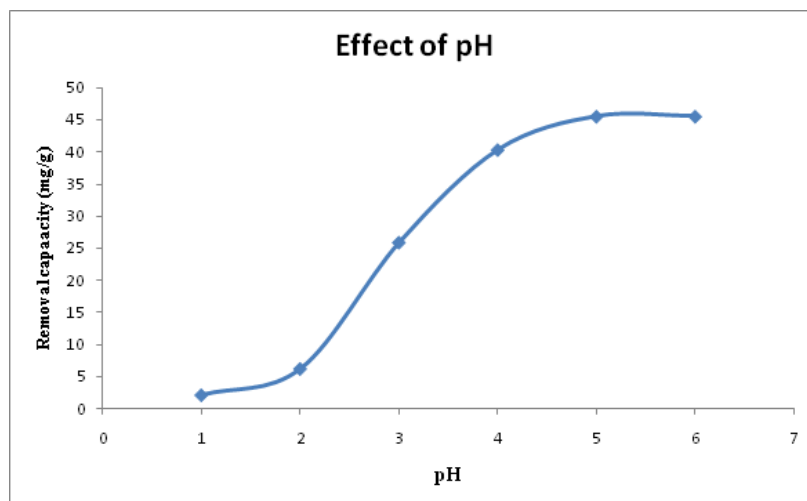
Figure 3. Effect of pH of solution on the adsorption of Pb(II)onto TLG.

4.4 Impact of concentration

The initial metal content in the solution has a big impact on how metals are absorbed from aqueous solutions. Figure. 4's findings indicate that as the starting Pb(II) concentration was raised, more Pb(II) was adsorbed per adsorbent mass unit. This resulted from a rise in the concentration gradient, which is the mass transfer's driving factor. Additionally, as the initial Pb(II) concentration in the solution increased, it was possible to see a decrease in Pb(II) removal because the active sites in the solution were becoming saturated. Having a larger initial concentration of Pb(II) ions was anticipated to result in more metal ions being adsorbed. The percent adsorption rose as the initial Pb(II) ions concentration (10-150 mg/l) at pH 5.0 increased, while the dosage (5 g/l) remained constant. The reason for the decline in % adsorption is because the biosorbent's metal-binding sites quickly become saturated with a rise in initial metal concentration [20].
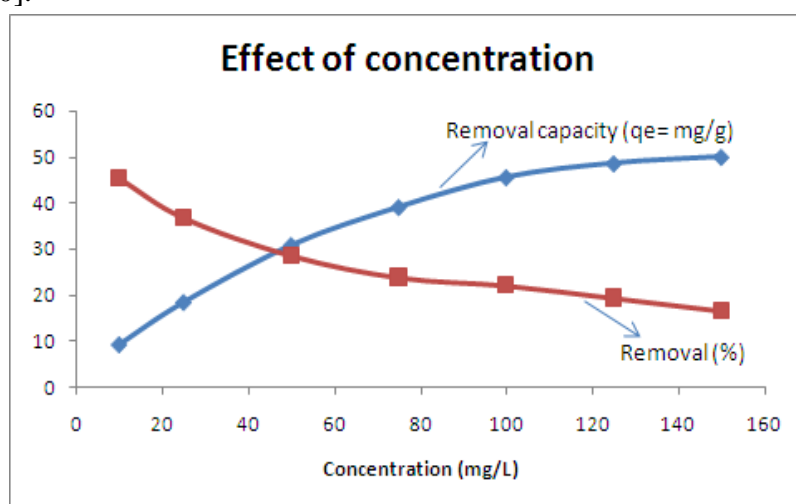

Figure 4. Effect of concentration on the adsorption of Pb(II) onto TLG.

4.5 Impact of adsorbent dose

Because it establishes an adsorbent's capacity for a specific initial concentration of the adsorbate, this value is crucial. Figure.5 displays the findings of the studies with various adsorbent doses. An increase in Pb(II)% removal was seen when the adsorbent dosage was increased. The rise rate of this parameter was especially high for larger adsorbent doses due to the greater availability of active sites on the surface of the materials and low for lower adsorbent doses due to the progressive saturation of these active sites [21]. Though as the adsorbent dose increased, the amount of Pb(II) absorbed per adsorbent mass unit significantly reduced. This is a result of the adsorption reaction's adsorption sites continuing to be unsaturated. Furthermore, it was observed that for the majority of the investigated materials, steady state was attained with an adsorbent dosage value between 5 and 75 g/l. In all of the remaining trials, the ideal adsorbent dose of 5 g/l was chosen.
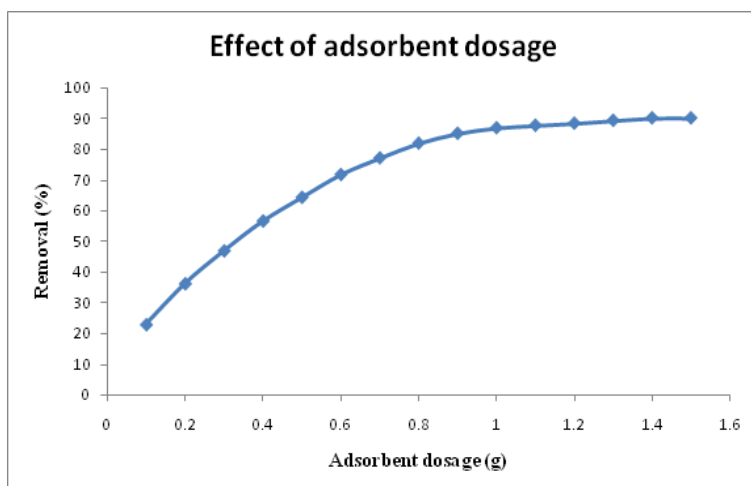

Figure 5. Effect of adsorbent dose on the adsorption of Pb(II) onto TLG.

4.6 Adsorption isotherm studies

Determination of the isotherm is essential for the analysis of the adsorption characteristics. The equilibrium batch adsorption data were fitted by two simple but practically useful isotherm- the Langmuir and Freundlich isotherms.

4.6.1 Langmuir model

The following are the results of the equilibrium investigations for Pb(II) adsorption onto TLG that conform to the Langmuir model [22]:

$$\frac{C_e}{q_e} = \frac{1}{bK_L} + \frac{C_e}{b} \tag{2}$$

where C_e is the equilibrium concentration (mg/l), q_e is the quantity of Pb(II) ions adsorbed per unit weight of the adsorbent (mg/g), K_L is the Langmuir equilibrium constant (l/mg), and b is the quantity of adsorbate necessary to produce a monolayer (mg/g). So, as illustrated in Figure 7, a plot of C_e/q_e vs C_e should be a straight line with a slope and an intercept. The Pb(II) ion adsorption data on TLG was successfully matched to the Langmuir isotherm, according to the correlation coefficient (R^2) value of (0.996). Since the Langmuir equation assumes that the

surface is homogeneous, it is possible that the homogeneous distribution of active sites on the adsorbent surface is the reason why the Langmuir isotherm closely matches the experimental results. The total adsorption capacity of the adsorbent for lead(II) is determined by the maximum adsorption capacity (q_e) of the adsorbent as estimated from the Langmuir isotherm equation.In Table 3, the estimated values of the constants K_L and b are shown. For the removal of Pb(II) from aqueous solutions, the Langmuir adsorption capacity is particularly effective.

In a batch adsorption process, the Langmuir isotherm can be used to predict whether a sorption system is favourable or unfavourable. The separation factor (R_L), a dimensionless constant that is a crucial component of the Langmuir isotherm, was employed for this. Its definition is given by the equation below:

$$R_L = \frac{1}{1 + K_L C_i} \tag{3}$$

where R_L stands for separation factor, C_i for initial metal ion concentration, and K_L for Langmuir constant. Positive isotherm is represented by R_L values between 0 and 1. Adsorption is still beneficial even for larger initial metal concentrations, according to R_L values (separation factor R_L value of 0.819-0.312) determined for lead(II) adsorption.
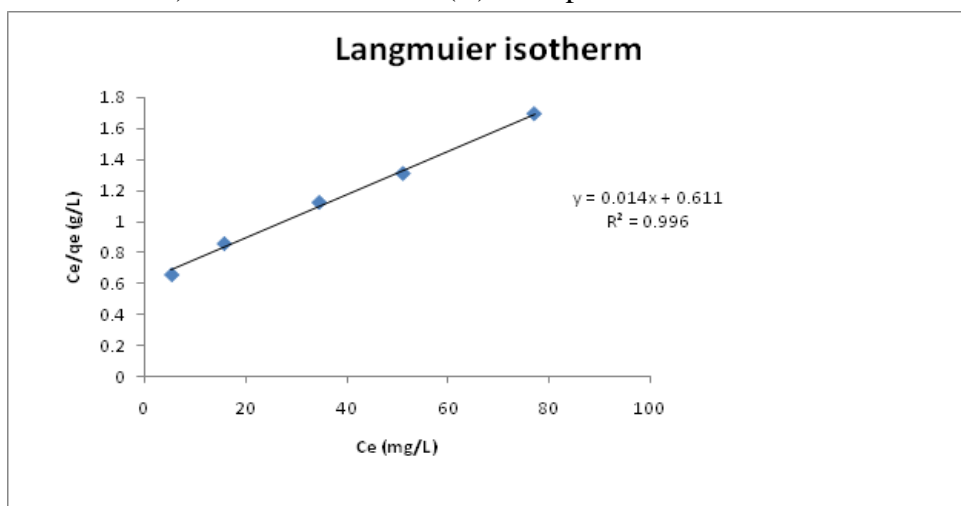

Figure 6. Langmuir plot Ce/qe versus Ce.

4.6.2 Freundlich model

The Freundlich isotherm is an empirical expression based on biosorption on a heterogeneous surface. The Freundlich model is represented by the equation [23]:

$$q_e = (K_F)(C_e^{1/n}) \tag{4}$$

$$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e \tag{5}$$

where q_e is the amount of Pb(II) ions adsorbed per specific amount of the adsorbent (mg/g), C_e is the equilibrium concentration (mg/l), and K_F is the Freundlich equilibrium constant. So, as illustrated in Figure 6, a plot of ln q_e vs ln C_e should be a straight line with a slope and an intercept. The Freundlich model and the lead(II) adsorption's linear regression coefficients (R^2 =.991) also appeared to be in agreement. However, the Freundlich constant n does not support it;

Table 3 showed that n is less than 1.0, indicating that the Freundlich model is unfavourable. Since the absorption on the adsorbent is not favoured when the Freundlich constant n is less than 1, this condition exists [24].
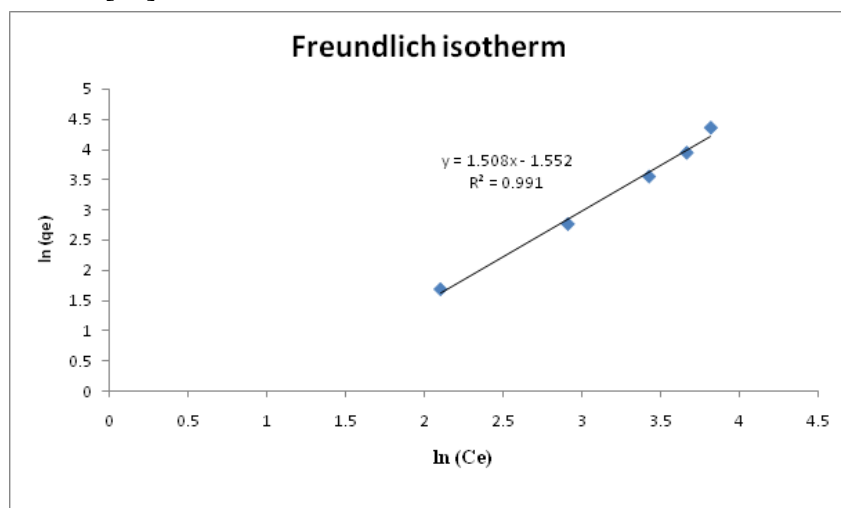

Figure 7. Fruendlich plot ln qe versus ln Ce.

Table 3. Langmuir and Freundlich adsorption isotherm constants for Pb(II) ions on lemon grass.

Isotherm	Constants		
Langmuir	b (mg/g)	K_L (l/mg)	R^2
	71.42	0.022	0.996
Freundlich	K_F (mg/g)	n	R^2
	4.72	0.663	0.991

4.7 Kinetic studies

The best operating parameters for a full scale batch operation must be chosen based on knowledge of the kinetics of pollutant uptake. Two kinetic models pseudo-first-order and pseudo-second-order have been used to analyse the experimental data in order to understand the adsorption kinetics of lead(II) ions. Correlation coefficient (R^2) was used to represent the degree of agreement between experimental data and values predicted by the model.

4.7.1 Pseudo-First-Order Model

Based on solid capacity, Lagergren's [25] pseudo-first order rate model is often phrased as follows:

$$\log(q_e - q_t) = (\log q_e) - \left(\frac{k_1}{2.303}\right) t \tag{6}$$

where q_t is the quantity of solute adsorbed at any time (mg/g), q_e is the amount of solute adsorbed at equilibrium per unit weight of adsorbent, and k_1 is the adsorption constant. The

pseudo-first-order kinetic model that is most frequently used is this one. For lead (II) ions, the values of k_1 were determined from the plots of log(qe qt) versus time (Figure 8). Correlation coefficients (R^2) and constant k_1 have been computed. Table 4 shows that there is poor agreement between the experimental values of q_{exp} and the estimated theoretical values. Therefore, modelling the adsorption of lead(II) onto TLG using the pseudo-first-order model is not appropriate. The obtained correlation coefficients (R^2) values were not very high.
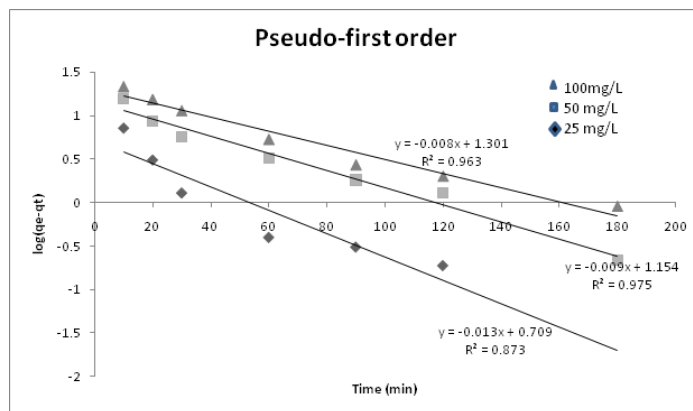

Figure 8. Pseudo-first order kinetic model plot of different concentrations of lead.

4.7.2 Pseudo-Second-Order Model

We tested the pseudo-second order model [26] to explain the adsorption data as the pseudo-first order model did not exactly meet our expectations. This can be said to be:

$$\frac{t}{q_t} = \left(\frac{1}{k_2 q_e^2}\right) + \left(\frac{1}{q_e}\right) t \qquad (7)$$

Where the relationship between t/qt and time should be linear, and qe (mg/g) and k_2 (g/mg.min) can be calculated from the slope and intercept of the plot (Figure 9). Table 4 lists the k_2 (g/mg.min) and q_e (mg/g) calculated from the model along with the relevant correlation coefficient values. Regression coefficients that is all higher than 0.99 are produced when a pseudo second-order model is applied. In addition, as shown in Table 4, the experimental and estimated values of q_e are fairly near. As a result, the pseudo second-order kinetic model is a good choice for simulating the lead (II) adsorption curves onto TLG.
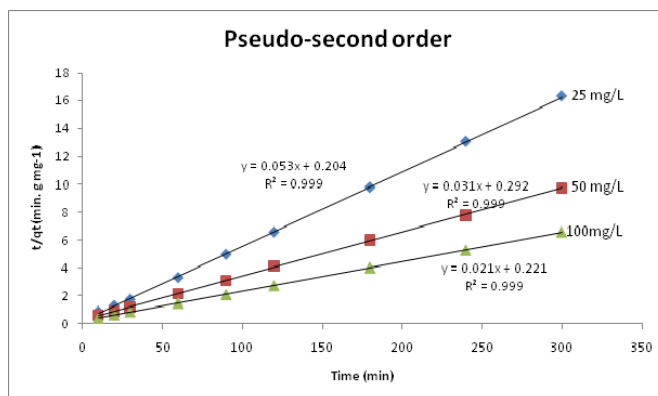

Figure 9. Pseudo-second order kinetic model plot of different concentrations of lead.

Table 4. Pseudo-First order and pseudo-second-order models for adsorption of Pb(II) ions on lemon grass.

Initial Conc. (mg/l)	$q_{e.exp.}$ (mg/g)	Pseudo first order			Pseudo second order		
		k_1 (1/min)	q_e (mg/g)	R^2	k_2 (g/ mg. min)	q_e (mg/g)	R^2
25	8.2	0.013	5.11	0.873	0.0137	8.86	0.999
50	30.8	0.009	14.25	0.975	0.329	32.25	0.999
100	45.5	0.008	19.99	0.963	1.996	47.61	0.999

4.8 Desorption studies

Studies on desorption were done to see if it was possible to recover TLG and Pb(II) ions. 20 mL of a 0.01-0.2M HCl solution in a conical flask were filled with 0.1g of adsorbent, which was then agitated for 4 hours at room temperature (30–10C). The rate of desorption rose initially as the concentration of hydrochloric acid increased before becoming practically steady Figure 10. The maximum lead recovery percentage for TLG with 0.15 M HCl solutions was 98%. The metal-loaded adsorbents serve as a cation exchanger during the desorption process when H+ ions take the place of Pb(II) ions. The use of TLG as an adsorbent in the practical applications of treating industrial effluents (Pb(II) ions containing effluents collected from a medium sized battery industry at Kanpur, India) makes it possible to recover the adsorbed lead and maintain the adsorbent's repeatability. Under ideal circumstances, it has been utilised to remove lead ions using natural biosorbent in batch processing. Following the guidelines of the Indian Standard for the discharge of Pb(II)-containing wastewater (IS 10500 19920), this study was conducted after treatment when the effluent content was found to be less than 0.1 mg/l.

4.9 Comparison studies

Table 5 compares the Pb(II) ion adsorption capacities of the described adsorbents with those of other sorbents reported in the literature. The structure and functional groups of the sorbents, as well as their capacity as biosorbents, are major sorbent characteristics.

Table 5 . Comparison of removal of Pb(II) on LG with other adsorbents**.**

Adsorbent	Capacity (mg/g)
Lemon grass [This work]	**47.61**
Grass clippings[15]	29.05
Coir fibers[15]	52.03
Mango leaves[15]	31.54
Peanut hulls[15]	69.75
Rice husk[15]	31.13

Tea leaves discarded[15]	35.89
Teak saw dust[15]	40.70
Rice stem[15]	49.57

4.10 Mechanisms for Pb (II) adsorption

The current work demonstrates that ion-exchange and complexation process, results in Pb(II) being adsorbed by the TLG at roughly pH 5.0. After Pb(II) adsorption, the TLG's -OH stretching vibration bands (3396cm1) are widened and moved to a higher wave number (3405.37 cm1). This shows that after adsorption, stronger intra- and intermolecular hydrogen bonds have formed in the TLG. The basicity of the donor group, or the availability of the electron, is a key factor in determining the stability of the complexes for metal ions; the higher the basicity, the more stable the complex is [27]. Despite the donor group's weak basicity, lead is known to form extremely stable complexes; fixing is dependent on the metal ion's chemistry and ligand affinity. Additionally, fixing capacities depend on the metals adsorbed; they are larger for Pb(II) because of its high affinity for the hydroxyl functionalities on the substrate. One of the main adsorption methods for Pb(II) ions to lemon grass was carried out by ion exchange mechanism.

5. Conclusions

Pb(II) ions from aqueous solutions and effluent from the battery sector have been bound to lemon grass in a batch procedure. The absorption of lead (II) is very pH-dependent. At pH 5, the highest sorption capacity was discovered. You can think about 240 minutes as the time needed to attain the equilibrium and sorption kinetics. Since lemon grass (TLG) has a variety of binding sites, the sorption kinetics of lead(II) onto it are described by the pseudo-second-order model. The Langmuir isotherm equation could be used to match the equilibrium data. The oxygen atoms in the cellulose carboxylic groups of lemon grass (TLG) can be linked to the sorption capacity. This research shows that the oxygen in the cellulosic groups, specifically CH-OH, is creating compounds with lead.

Acknowledgement

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