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A Novel Green Approach for Lead Adsorption and Isotherm Evaluation

M. Dharsana* and J. Prakash Arul Jose*†
*Department of Civil Engineering, Noorul Islam Centre for Higher Education, Thuckalay, Kanyakumari, Tamilnadu, India
†Corresponding author: J. Prakash Arul Jose; joseprakash1430@gmail.com

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ABSTRACT

Environmental damage due to the discharge of organic pollutants and heavy metal toxins has become a major topic of concern for the past couple of years. Using just a natural adsorbent to solve wastewater concerns has lately gained popularity as an ecologically acceptable solution that encourages long-term growth. A range of approaches, including adsorption to the surface of agricultural leftovers, have been used to minimize heavy metals in an aqueous medium. Lead is amongst the most hazardous and widely discovered toxic substances in industrial waste. *Citrus limetta* peel powder, Banana peel powder, and Betel leaf powder were chosen as adsorbents in this study to absorb synthetic lead from an aqueous solution since they are low-cost materials. Our research aims to find natural bio-sorbents that can remove highly hazardous Pb^{2+} ions from aqueous solutions. The importance of contact time, concentrations, adsorbent-based dose, and pH in the adsorption process is investigated. The adsorption rate for betel leaves, *Citrus limetta* peel, and banana peel was 5, 10, 15, 20, and 25 $g.L^{-1}$. *Citrus limetta* peel (10 $g.L^{-1}$), banana peel (5 $g.L^{-1}$), and betel leaf (5 $g.L^{-1}$) provide the highest lead adsorption. Material characterization is used to determine the lead nitrate process in lead adsorption. The capacity of the lead-adsorbing substances to achieve adsorption equilibrium was assessed and estimated using linear Freundlich and Langmuir isotherms, with the experimental data fitting the Freundlich isotherm models.

INTRODUCTION

Heavy metal ions are employed in a wide range of industrial applications (Gunarathne et al. 2022). These businesses' wastewater discharges gradually leak some toxins into the atmosphere (Singh et al. 2022). Contamination by heavy metals water is a major ecological calamity due to its high toxicity and accumulation in food chains (Ashar et al. 2022). Unlike biological pollutants that are usually recyclable, heavy metals do not break down into safe end products. Heavy metals are hazardous to marine habitats, even at small concentrations (Brennecke et al. 2022). Heavy metals such as copper (Cu), chromium (Cr), mercury (Hg), and lead (Pb) are toxic to humans and the environment. Lead (Pb^{2+}) is a very hazardous heavy metal that can harm people and the environment (Ajiboye et al. 2021). It causes severe damage to both the nervous and immune systems (Munir et al. 2021) Pb^{2+} is utilized in various industrial processes, including battery production (Otieno et al. 2022). Electroplating (Shittu et al. 2017), petroleum and chemical operations (Védrine 2016), printing inks (Näsström et al. 2022), and biofuel production (Sentanuhady et al. 2021). Hazardous residues from the process sector, energy storage, pesticides, and pharmaceutical mixtures used for flavoring and seasonings contribute to lead in water (Jarvis & Fawell 2021). Adsorption is the most effective method for removing hazardous or heavy metals, and it gives the best results since it can be used to remove a wide range of hazardous elements (Anselmo et al. 2022). Commercial activated carbon (CAC) is the most widely used technology for removing dangerous ions due to its effectiveness and increased adsorption capacity; however, its usage remains limited due to the increased operating costs (Jose & Dharsana 2022). CAC also raises concerns about the need for regeneration and the challenges of isolating it from the effluent after its use (Shahrokhi-Shahraki 2021). Several researchers have focussed on discovering non-traditional alternative adsorbents to reduce the cost of pollution treatment.

Although it is common, most agricultural residues are conveniently utilized and thus do not necessarily require an advanced processing stage or design that empowers before execution. Restoration of these absorbents may not be a requirement, and the procedure requires minimal supervision. Some researchers have concentrated on using waste material as a sorbent (Kim & Singh 2022). Heavy metals can be removed using precipitation, phase separation, electrochemical treatment, and membrane filtration (Xu et al. 2022). However, only adsorption seems to become an economically feasible option at this time. The adsorbent is a unit process in residential and municipal sewage treatment operations and is becoming more common in today's plants (Kim et al. 2022). Contaminant adsorption has always been the subject of extensive research throughout the years, and it has been ongoing. Adsorbents such as chemical adsorbents (Hu et al. 2022, Alex et al. 2022a), zeolites (He et al. 2021), bio-adsorbent materials (Karimi et al. 2022), and nano-adsorbents (Janani et al. 2022), were suggested. However, due to isolation and regeneration costs, most recommended adsorbents have still yet to show cost-effectiveness; consequently, research into the best biosorbent metal ion removal continues. Several therapeutic approaches have been used in the past to eliminate lead from water. The use of cationic resin polite (Jose & Dharsana 2022), adsorbents using high alumina concentration bauxite, alkali ash particle permeable responsive barrier, use of particular lactic acid bacteria (Li et al. 2021), electro-coagulation process (Deng et al. 2021), adsorption using natural American bentonite (Gupt et al. 2022, Alex et al. 2021) and activated carbon (Hao et al. 2021), using iron nanoparticles (Vázquez-Guerrero et al. 2021), and using okra (Barasikina 2021), rice husk (Babazad et al. 2021), and sawdust are among these (Ibrahim et al. 2022).

Adsorption is the process of preventing adsorbents from moving around on the adsorbent. In the affluent approach, isotherm adsorption studies were used to predict the ability of specific adsorbents to remove contaminants. Water contamination arises from a lack of contemporary wastewater-treatment systems and a scarcity of competent workers, resulting in a shortage of drinkable water. The demand for low-cost wastewater treatment options prompted this research. Poor economic situations in several developing countries need low-cost water treatment options for afflicted areas, as traditional or advanced pollution removal, is impossible.

Plant-based compounds for water treatment are environmentally friendly, inexpensive, non-toxic, and simple to use, with the residual created being disposed of even though they are readily available (Rastogi et al. 2008). Numerous organic chemicals have been studied as low-cost water treatment coagulants. Using bio-sorbents in water filtering fits well with environmental stability since waste material is reused to lessen created in an environmentally advantageous manner (Giri et al. 2022). Adsorption was assumed to be a critical method for eliminating toxins whenever these biomass materials were used for water treatment. Even though many types of research conducted have concentrated on the use of natural materials as water treatment agents to remove various impurities, little is known about the mechanisms of operation in the method of treatment (Elkhaleefa et al. 2021, Thakare et al. 2021, Dineshkumar et al. 2022, Alex et al. 2022b). The removal process of one heavy metal may differ from that of others. As a result, instead of concentrating on biomass use, it is critical to comprehend the factors contributing to the removal of specific metal ions from an aqueous solution. Adsorption is a regularly used strategy for isolating and purifying lead ions due to its substantially improved metal-binding capacity. It could promote human health by improving the quality of treated wastewater quality.

Furthermore, the activation chemicals are easily recyclable and reusable (Zhang et al. 2022). Carbon activation using phosphoric acid is often used to saturate lignocellulose substances, including coir and timber, on a big scale. Phosphoric acid increases yield by modifying the pyrolysis process, which includes the disintegration of lignocellulosic materials, depolymerization, dryness, and component distribution (Mirmohammadmakki et al. 2022). Phosphoric acid has become extremely popular as the acid recovery duration has improved.

The goal of this study was to use organic substances such as *Citrus limetta* peel powder, Banana peel powder, and Betel leaves powder to remove Pb^{2+} ions from an aqueous system via a biosorption method. These are organic resources with a large adsorption ability for heavy metal removal from wastewater (Chawla et al. 2022, Sharma et al. 2021). The purpose of this study was to see whether the existing adsorption isotherm technique could effectively define the behavior and therapeutic efficiency of *Citrus limetta* peel powder, Banana peel powder, and Betel leaves powder in removing Pb and to learn much

Table 1: Biosorbents for lead removal.

Biosorbent	Metal removed	References
Spirogyra	Lead	(Kumar & Oommen 2012)
Red microalgae	Lead	(Anastopoulos & Kyzas 2015)
Caster leaf powder	Lead	(Al Rmalli et al, 2008)
Punica granatum L. peels	Lead	(Ay et al. 2012)
Green algae waste biomass	Lead	(Jalali et al. 2002)
Cucumis melo	Lead	(Akar et al. 2012)
A.Leucocephala bark	Lead	(Munagapati et al. 2010)
Maple sawdust	Lead	(Hossain et al. 2014)
Tea leaves	Lead	(Ahluwalia & Goyal 2005)
Waste tea leaves	Lead	(Mohammed et al. 2016)
Palm kernel husk	Lead	(Baby et al. 2019)
Rice husk and fly ash	Lead	(Syuhadah & Rohasliney 2012)
Trichoderma reesi	Lead	(Ng et al. 2013)

about the adsorption mechanism of a particular heavy metal described by the ideas. Table 1 shows some research works which use biosorbents for lead removal.

THE OBJECTIVES OF THE WORK

The main intentions of this research study are given below.

- To identify an efficient method to remove lead from synthetic and industrial wastewater.
- To carry out the experimental study using three adsorbents, namely *Citrus limetta* peel, Banana peel, and Betel Leaf, on both synthetic and industrial wastewater.
- To analyze the characterization of the bio adsorbents and identify the optimizing pH, contact time, and adsorbent dose.

MATERIALS AND METHODS

Green Adsorbents Used in This Study

This research uses natural adsorbents like *Citrus limetta* peel powder, Banana peel powder, and Betel leaves powder to absorb the synthetic lead from an aqueous solution. The technical details of the green absorbent materials used in this study are shown in Table 2.

Table 2: Scientific details of green absorbents used.

GREEN ADSORBENTS USED		
Citrus limetta	Banana	Betel
• Kingdom: Plantae • Order: Sapindales • Family: Rutaceae • Genus: *Citrus* • Species: *C. limetta*	• Kingdom: Plantae • Order: Zingiberales • Family: Musaceae • Genus: *Musa*	• Kingdom: Plantae • Order: Piperales • Family: Piperaceae • Genus: *Piper* • Species: *P. betel*

Preparation of Adsorbent

Citrus limetta peel, Betel leaf, and Banana peel were washed with distilled water and dried in the sun for 7 days. After sundried, it was dried in an oven for two hours at 150°C. After that, it was ground in the mill as a *Citrus limetta* peel powder, depicted in Fig. 1**.** The adsorbent dosage was selected as 5, 10, 15, 20, and 25 $g.L^{-1}$ for Betel leaf, banana peel, and *Citrus limetta* peel.

Preparation of Stock Solution

The synthetic stock solution is prepared by dissolving the lead Nitrate salt in various concentrations in the range of 5 $g.L^{-1}$, 10 $g.L^{-1}$, and 15 $g.L^{-1}$ from the literature study, and the volume of the solution is made up to 1000 mL. The details of the salts and the amount used are tabulated in Table 3.

Table 3: Preparation of stock solution.

Metal	Complex salt	Formula	Molecular weight	Amount used [$g.L^{-1}$]
Lead	Lead Nitrate	$Pb(NO_3)_2$	331.2 $g.mol^{-1}$	5 10 15

Methodology and Characterization

Characterization is performed to study the different attributes of created adsorbents from *Citrus limetta* peel powder, Banana peel powder, and Betel Leaf powder. Synthetic water is the stock solution prepared in the laboratory using lead Nitrate at various concentrations. Industrial water is collected from the river Cauvery, Karur District, Tamilnadu. The characterization is critical when another material is produced as an adsorbent for the expulsion of particular contamination, as depicted in Fig. 2.

FTIR Analysis

The Fourier transforms infrared spectroscopy (FTIR), performed in the wavelength range of 4000-500cm^{-1}, to analyze the available functional groups. The chemical nature of bio adsorbents and functional groups on the surface of

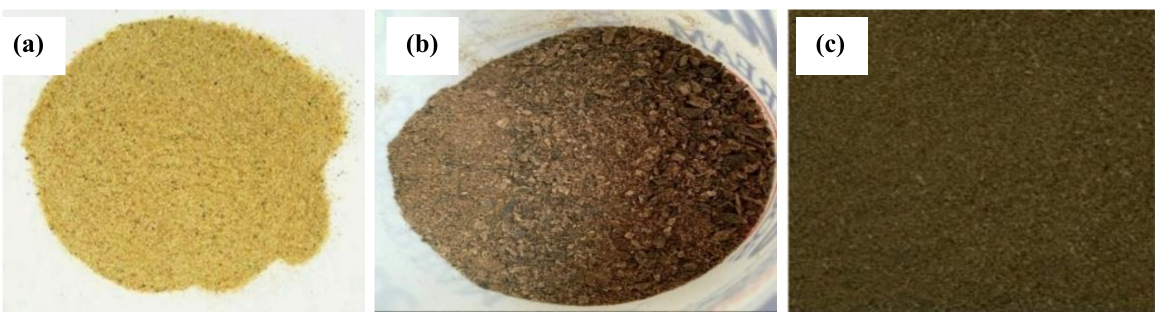

Fig. 1: (a) *Citrus limetta* peel powder, (b) Banana peel powder, and (c) Betel leaf powder.

Fig. 2. Methodology of study.

biosorbents were predicted using Fourier Transform Infrared spectroscopy (FTIR). The batch adsorption test information is utilized to assess different parameters with the assistance of various isotherm models accessible in the literature.

Isotherm Models

To comprehend the circulation of the metal particles in the fluid and solid stages at balance, the distinctive isotherm models must be fitted with experimental equilibrium data information. The present work fits the information with the Langmuir Freundlich isotherm model, depicted in Fig. 3.

Langmuir Isotherm

This model suggests monolayer coverage and continuous adsorbate-surface binding energy. The model is given by equation 1:

$$q_e = \frac{K \cdot Q_a^0 \cdot C_e}{1 + K \cdot C_e} \quad \ldots(1)$$

Where Ce is the equilibrium concentration of the adsorbate ($mg.L^{-1}$), qe is the amount of adsorbate adsorbed per unit mass of adsorbent ($mg.g^{-1}$), Qo and a are Langmuir constants related to adsorption capacity and rate of adsorption, respectively. Qao represents the maximal adsorption efficiency (g $solute.g^{-1}$ adsorbent). $L.mg^{-1}$ is the unit of measurement for K.

BET (Brunauer, Emmett and Teller) Isotherm

This is a multi-layer, more general model. It is assumed that each layer has a Langmuir isotherm and that no translocation happens between them. Apart from the first layer, this assumes that each layer has the same adsorption energy, as shown in equation 2.

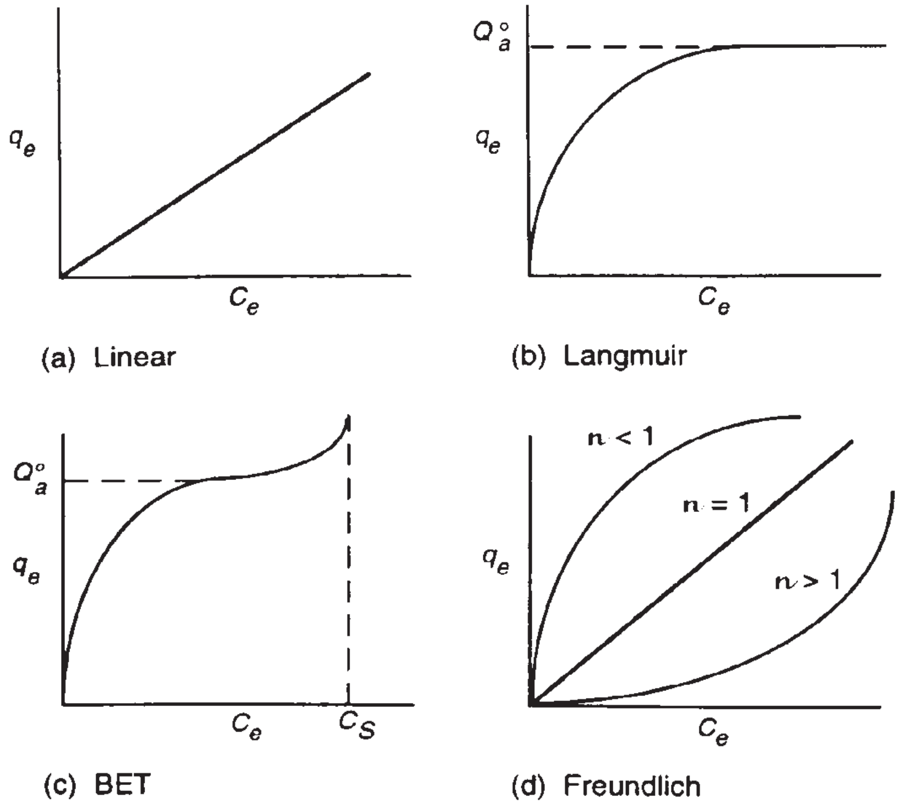

Fig. 3: Isotherm models.

$$q_e = \frac{K_B \cdot C_e \cdot Q_a^0}{(C_S - C_e)\{1 + (K_B - 1)(C_e / C_S)\}} \quad \ldots(2)$$

where qe is the measure of adsorbate adsorbed per unit mass of adsorbent (mg.g^{-1}), Ce is the equilibrium concentration of the adsorbate (mg.L^{-1}), Kf and *n* are Freundlich constants with *n* giving an indication of how suitable the adsorption process.

Freundlich Isotherm

The Freundlich model is used for the special case of multiple surface energies (particularly useful for contaminated materials) in which the frequency term, KF, fluctuates as a factor of surface coverage n, and K_F is a specific constant, as shown in equation 3.

$$q_e = K_F * C_e^{1/n} \quad \ldots(3)$$

Physicochemical Properties of the Adsorbents

The adsorbents *Citrus limetta* peel, Banana peel, and Betel Leaf are characterized in terms of

- pH
- moisture content
- Ash content

Determination of pH

The standard analysis of pH for three adsorbents was tested. 1g of all three adsorbents was weighed and moved into a glass beaker. The adsorbent was mixed with 100mL of distilled water was measured and added to the sample of powdered adsorbents in the beaker, and stirred for one hour. The pH for all three adsorbents is represented in the graph.

Determination of Moisture Content

A crucible was dried out and cooled in a desiccator, and weighed. The powdered adsorbents were transferred into the crucible and weighed (W_i). The crucible and sample were then dried out in a hot air oven at 105°C, and it was considered as (W_f), as given in equation 4. The result is represented in the graph.

$$\text{Moisture content (\%)} = \frac{W_i - W_f}{W_i} * 100 \quad \ldots(4)$$

Determination of Ash Content

A crucible was preheated in a muffle furnace at 500°C, cooled in a desiccator, and 1g of adsorbents were transferred into the crucible and reweighed. Then the sample was kept in a muffle-shaped furnace and preheated to 500°C. It was cooled in desiccators, and the weight was taken. The result is represented in the graph, and equation 5 represents it.

$$\text{Ash content (\%)} = \frac{\text{Ash weight on dry x 100}}{\text{Oven weight dry}} \quad ...(5)$$

RESULT AND DISCUSSION

Effect of Parameters in Synthetic Wastewater

Batch adsorption studies were directed to improve critical exploratory parameters which can influence the efficiency of containment removal. The activated biosorbents are pressed mechanically and applied in water treatment. Press cake was soaked with water overnight to get the remaining compounds, and the clean press cake was used in this experimental work. The moisture content was calculated to get the correct mass for the biosorbent added to water. The press cake of biosorbents was added to each beaker with the sample solution. The stirring speed was set at 200 rpm for 4 minutes and 40 rpm for 30 minutes. All treatments were done as triplicates, and at the end of each optimization, solutions were allowed 60 min for settling and filtered using filter papers. Finally, the remaining heavy metals concentration of each solution was measured using Thermo Fisher Scientific iCE 3500 AAS Atomic Absorption Spectrometer

Effect of Initial Concentration

The impact of initial lead concentration in addition to the measure of adsorbate adsorbed per unit weight of adsorbent. From Fig. 4, it is demonstrated clearly that the removal efficiency of *Citrus limetta* is 77% at an initial concentration, Banana peel is 82.7% at an initial concentration, and Betel leaf is 52.98% at an initial concentration of 5 g.L^{-1}, as depicted in Fig. 4.

Effect of Adsorbent Dosage

The impact of the adsorbent dose on the expulsion of lead is depicted in Fig. 5, with an initial concentration of 5 g.L^{-1} for *Citrus limetta* peel and 7.5 g.L^{-1} for Banana peel and Betel Leaf. It is seen that the maximum removal efficiency of lead is 73.4%, 81.7%, and 53%, which is found at an optimum dosage of 30 g.L^{-1} for *Citrus limetta* peel, 15 g.L^{-1} for Banana peel, and Betel Leaf.

Effect of pH

The impact of pH on the adsorption of lead particles is shown in Fig. 6 with steady introductory fixation and adsorbent measurement. The proportion of adsorption was the least in the case of pH 2 anyway. It increased with expanding pH. The most extraordinary adsorption occurs at pH 6 for Banana peel and pH 7 for *Citrus limetta* peel and betel leaf.

Effect of Contact Time

The impact of contact time on the expulsion of lead ions is shown in Fig. 7 by maintaining other optimization parameters at constant magnitude. The percentage removal of lead is attained within 100mins for *Citrus limetta* peel powder and Betel Leaf, while it takes about 140 mins for Banana peel powder. This data implies a better adsorption efficiency

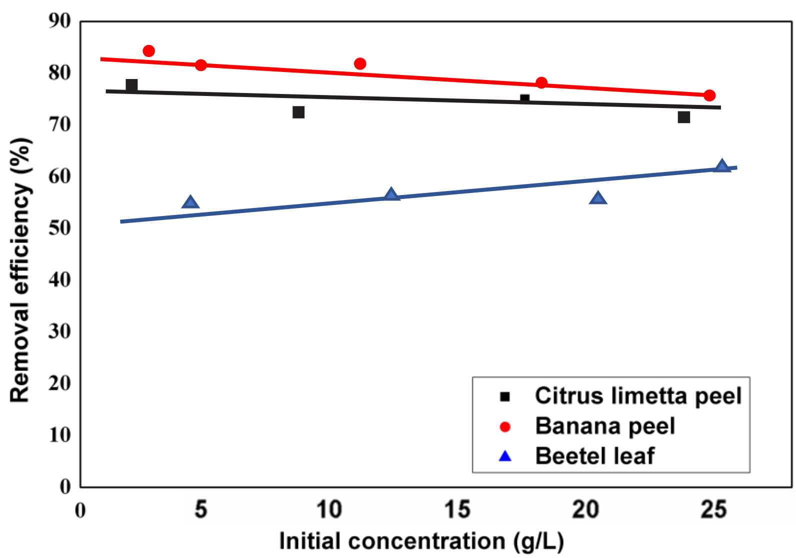

Fig. 4: Impact of initial concentration on the expulsion of lead by *Citrus limetta* peel powder, Banana peel powder, and Betel Leaf powder.

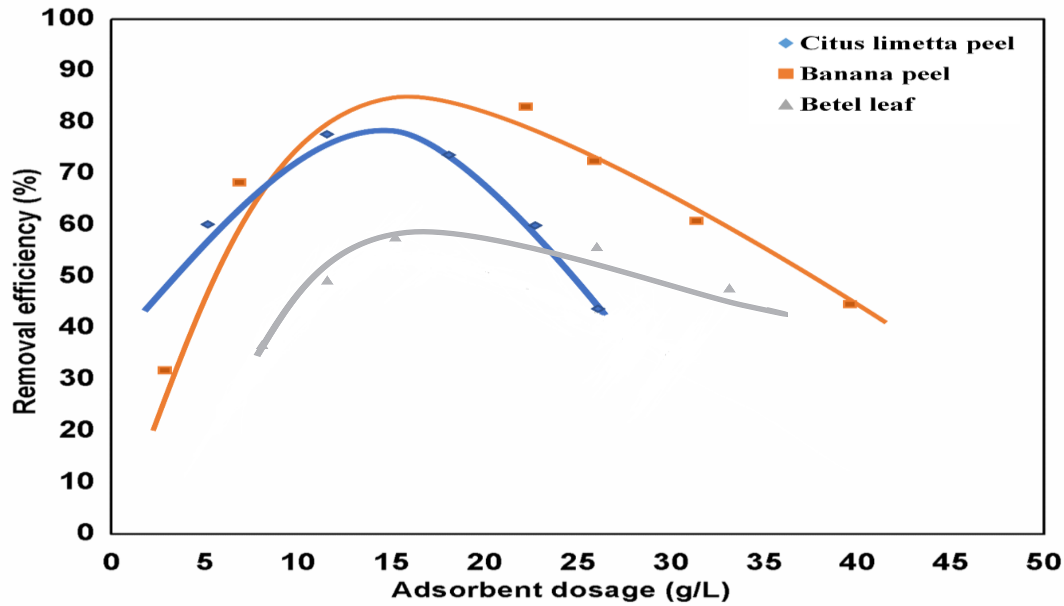

Fig. 5: Impact of adsorbent dose on the expulsion of lead by *Citrus limetta* peel powder, Banana peel powder, and Betel Leaf powder.

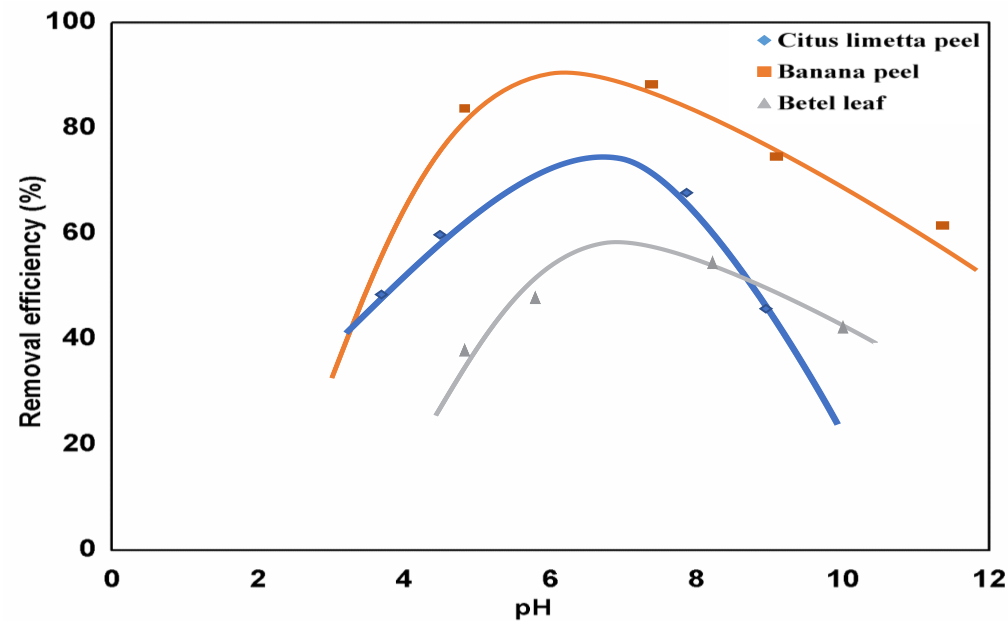

Fig. 6: Impact of pH on the expulsion of lead by *Citrus limetta* peel powder, Banana peel powder, and Betel Leaf powder.

for Banana peel when compared to *Citrus limetta* peel and Betel leaf powder.

Adsorption Isothermal Studies

At equilibrium conditions, the adsorption isotherm is an equation depicting the transfer of adsorbed species from the liquid solution to the adsorption phase. Adsorption isotherm provides essential models in the description of adsorption behavior. It describes how the adsorbate interacts with the adsorbent and offers an explanation of the nature and mechanism of the adsorption process. When the adsorption reaction reaches an equilibrium state, the adsorption isotherm can indicate the distribution of adsorbate molecules between the solid phase and the liquid phase. Equilibrium

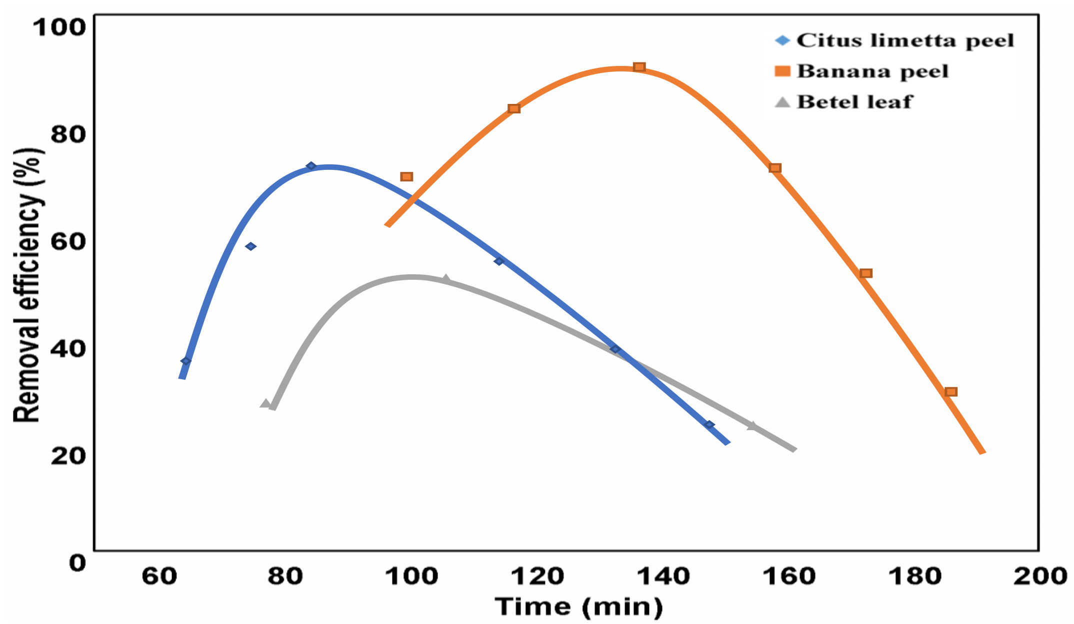

Fig. 7: Impact of contact time on the expulsion of lead by *Citrus limetta* peel powder, Banana peel powder, and Betel Leaf powder.

isotherm data obtained from the different models provide essential information on adsorption mechanisms and the surface properties and affinities of the adsorbent. Therefore, establishing the most appropriate correlation of equilibrium curves is essential to optimize the conditions for designing adsorption systems. In this present work, Langmuir and Freundlich's isotherms were employed to investigate the adsorption behavior.

The following characteristics, listed in Table 4, were used to evaluate the appropriateness of the equation given to explain the adsorption process.

Adsorption Equilibrium Isotherm Result

Freundlich Model

The following results are obtained from experimental data of the Freundlich model

(a) Freundlich model for Betel leaves: The Freundlich isotherm model for the adsorption of lead by Betel leaves powder is expressed as R^2 =0.9873, n = 0.71, 1/n = 1.408, K_f ($1.mg^{-1}$) = 0.132. Fig. 8 depicts the Freundlich isotherm model for the adsorption of lead by Betel leaves powder. It should be noted that the experimental values have a high coefficient of the relation between logarithmic values.

(b) Freundlich model for Citrus limetta peel: The Freundlich adsorption isotherm model of lead by *Citrus limetta* peel powder is expressed as R^2 = 0.9937, n = 5.052, 1/n = 0.198, K_f ($1.mg^{-1}$) = 1.268, depicted in Fig. 9. It exposed that the experimental values have high logarithmic correlation coefficients.

(c) Freundlich model for Banana peel: The Freundlich adsorption isotherm model of lead by Banana peel powder is expressed as R^2 = 0.9866, n = 0.284, 1/n = 3.52, K_f ($1.mg^{-1}$) = 0.073, depicted in Fig. 10. It proved that the experimental values have a high correlation between logarithmic co-efficient.

Langmuir Model

The following results are obtained from experimental data of the Langmuir model

(a) Langmuir model for Betel leaves: The Langmuir isotherm model for adsorption of lead by Betel leaves powder is expressed as R^2 = 0.9891, Q^O = 0.683, b = 0.544, depicted in Fig. 11. The experimental

Table 4: Adsorption isotherm studies.

Isotherms	Parameters	*Citrus limetta* peel powder	Betel leaves powder	Banana peel powder
Langmuir	R^2	0.9877	0.9891	0.9966
	Q^0 [$mg.g^{-1}$]	1.994	0.683	2.704
	b [$1.mg^{-1}$]	2.863	0.544	5.116
Freundlich	R^2	0.9937	0.9873	0.9865
	N	5.052	0.71	0.284
	1/n	0.198	1.408	3.52
	K_f [$1.mg^{-1}$]	1.268	0.132	0.073

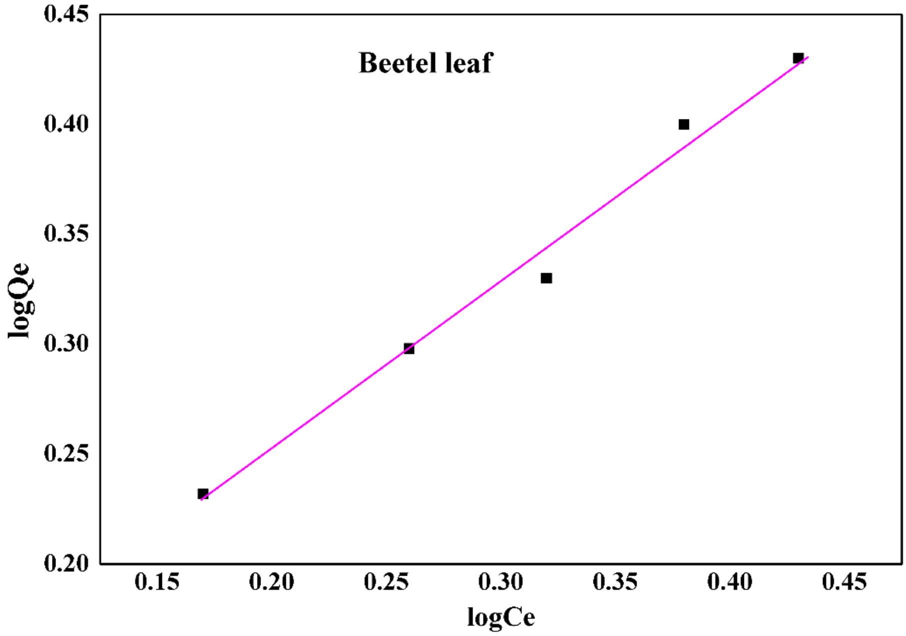

Fig. 8: Freundlich adsorption isotherm model of lead by Betel leaves powder.

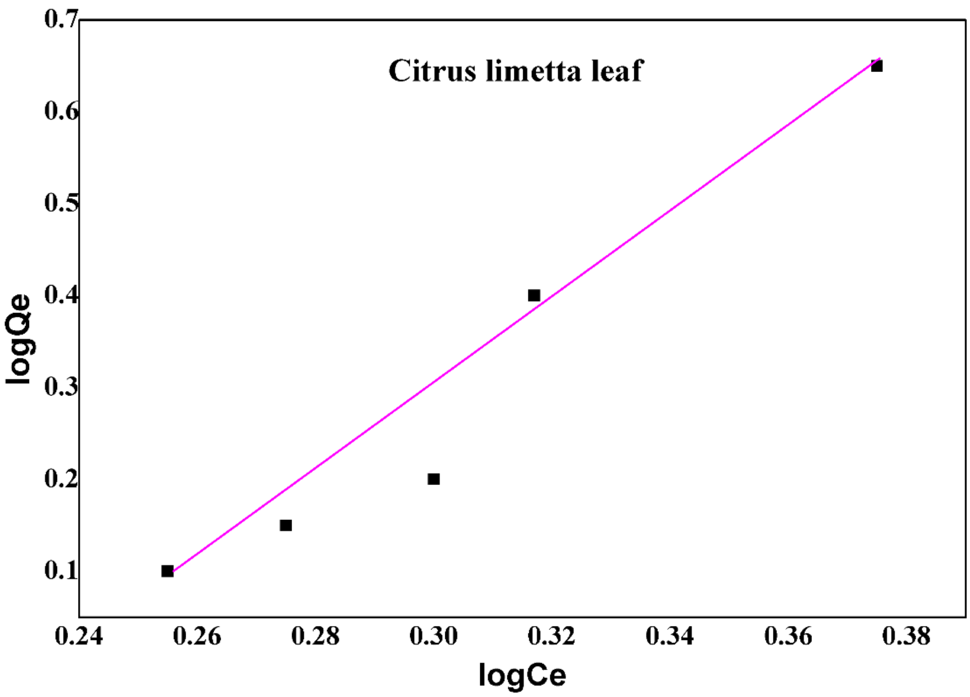

Fig. 9: Freundlich isotherm model for adsorption of lead by *Citrus limetta* peel powder.

values contain strong logarithmic (C_e/Q_e) correlation coefficients, which should be noted.

(b) Langmuir model for Citrus limetta peel: The Langmuir isotherm model for the adsorption of lead by *Citrus limetta* peel powder is expressed as $R^2 = 0.9$, Q^O =, b=, depicted in Fig. 12. The C_e/ Q_e values expose the same trend of betel leaves.

(c) Langmuir model for Banana peel: The Langmuir isotherm model for the adsorption of lead by Banana peel powder is expressed as $R^2 = 0.9966$, $Q^O = 2.704$, b = 5.116, as depicted in Fig. 13. High logarithmic correlation (C_e/Q_e) coefficients between the experimental values should be noted.

CONCLUSION

From this study, the following results can be concluded.

- In this work, the adsorption of lead from synthetic and industrial water was investigated using low-cost adsorbents such as *Citrus limetta* peel powder, Banana peel powder, and betel leaf powder were successfully developed.
- The adsorbent dosage was selected as 5, 10, 15, 20, and 25 $g.L^{-1}$ for Betel leaf, banana peel, and *Citrus limetta* peel.

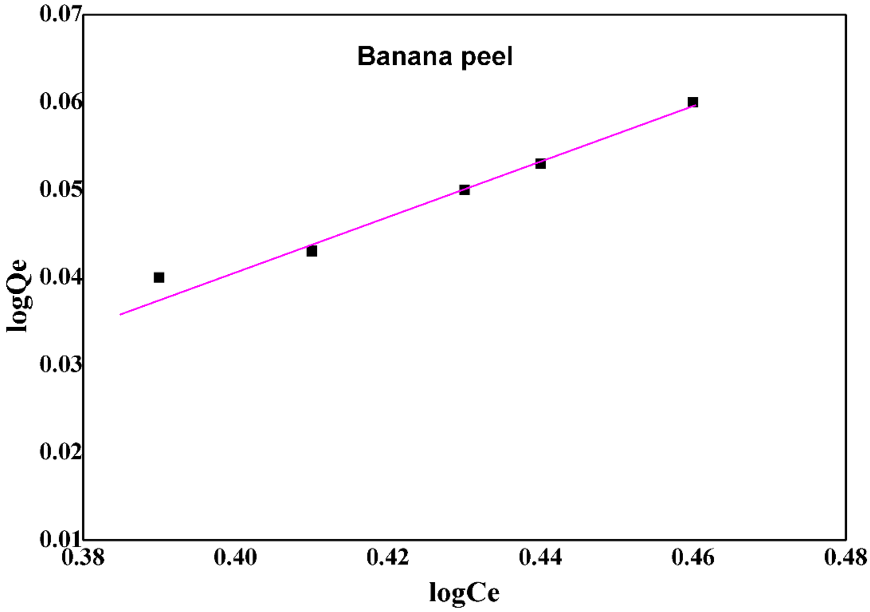

Fig. 10: Freundlich adsorption isotherm model of lead by Banana peel powder.

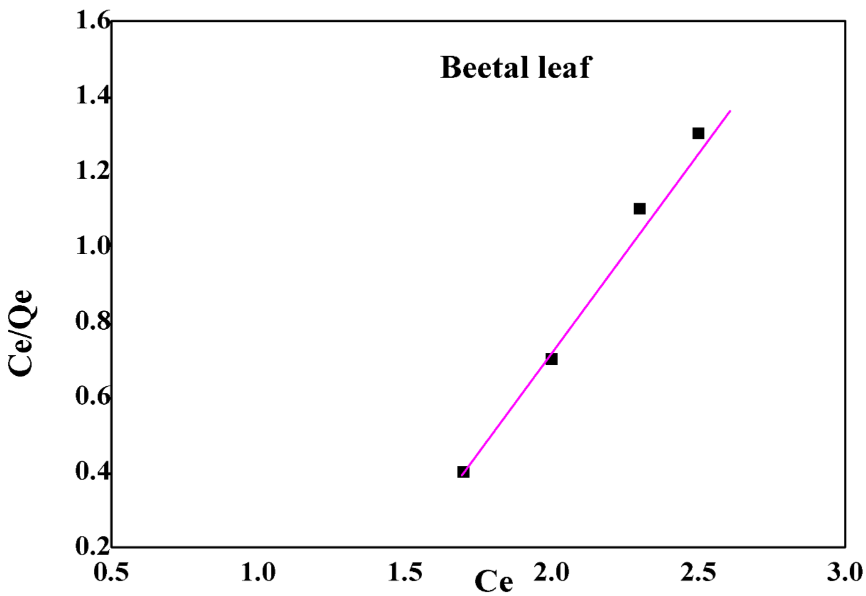

Fig. 11: Langmuir isotherm model for adsorption of lead by Betel leaves powder.

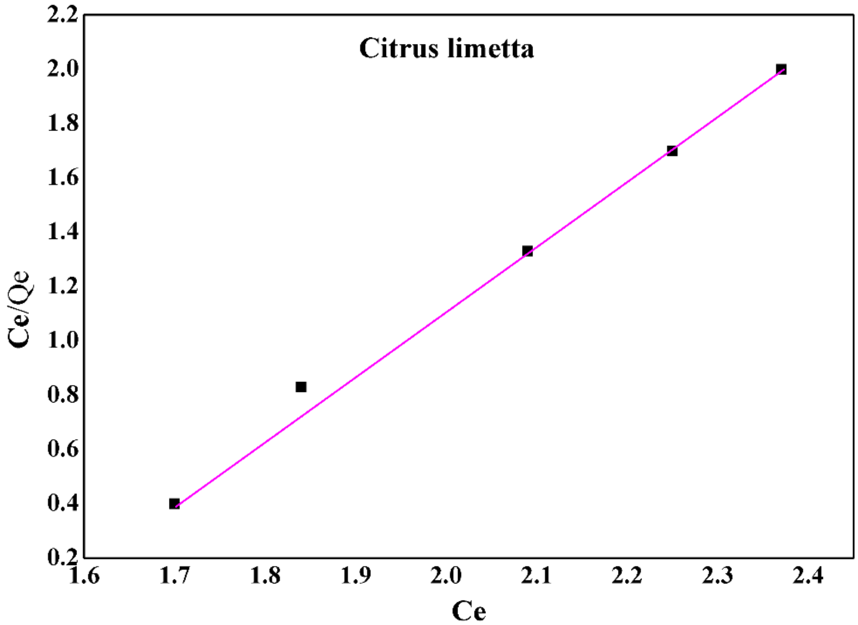

Fig. 12: Langmuir isotherm model for adsorption of lead by *Citrus limetta* peel.

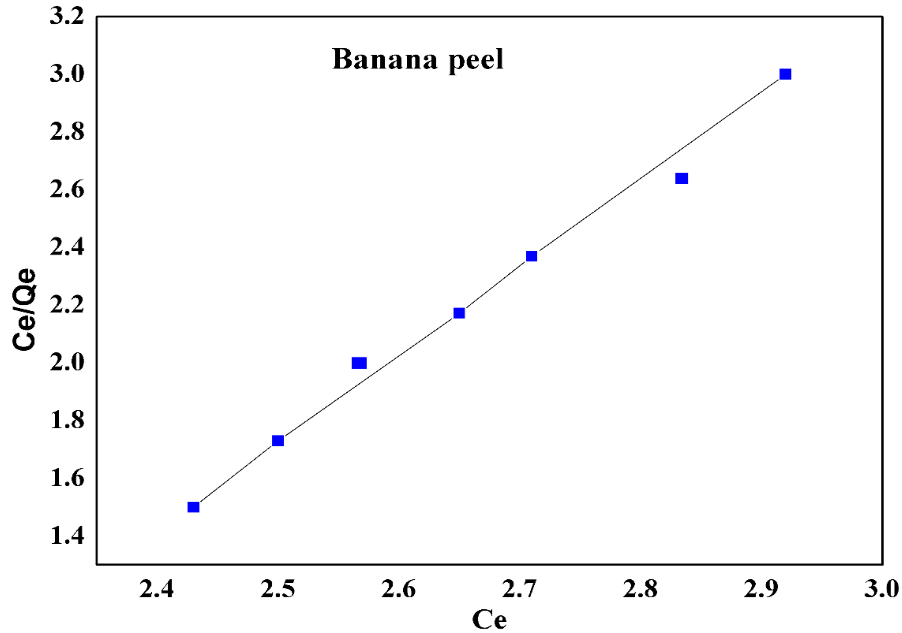

Fig. 13. Langmuir isotherm model for adsorption of lead by Banana peel.

- The result indicated that the percentage removal of lead was considerably affected by initial lead concentration, amount of adsorbent dose, pH value, and mixing contact time. The results showed that the removal percentage increased with an increasing amount of adsorbent dosage.
- The equilibrium adsorption of metal ion Lead was well-fitted by Langmuir and Freundlich's adsorption isotherm model.
- From these experimental results, the Langmuir is better fitted ($R^2 = 0.999$) for *Citrus limetta* peel powder, ($R^2 = 0.993$) Banana peel powder, and ($R^2 = 0.961$) betel leaf powder.

FUTURE SCOPE

The research study can be continued to advance studies in the following fields. The developed adsorbent may be further utilized to eliminate lead from industrial wastewater, and their process parameters can be studied. The results of the adsorption activity of adsorbents can be compared with both synthetic and industrial wastewater can be done. This work can be further developed on a large scale for use in Industries, and a comparison can be made by adding two adsorbents simultaneously.

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