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ADSORPTION OF LEAD FROM CONTAMINATED WATER USING BIOSORBENT

UPORABA BIOABSORBENTA ZA ABSORPCIJO SVINCA IZ ONEČIŠČENE VODE

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For the past few years, environmental degradation caused by the discharge of heavy metals and organic contaminants has been a major source of worry. Using a natural adsorbent to tackle wastewater problems has recently been viewed as an environmentally friendly move that promotes sustainable development. Heavy metals have indeed been reduced in aqueous solutions using a variety of methods, include adsorption to the surfaces of agricultural residues. Lead is among the most toxic and common heavy metals found in industrial wastewater. In this research, adsorbents selected are moringa leaves, rice husk and coconut fibers, which are very low-cost materials, employed for the lead exclusion from industrial wastes. Thus, our study aims to investigate the ability of natural bio-sorbents to remove very toxic Pb^{2+} ions from aqueous solutions. Contact time, concentration, adsorbent-based dosage and pH were all evaluated as important factors in the adsorption mechanism. The adsorption efficiency was discovered to be pH dependent, rising as the solution pH was increased in the ranges of 2.5 to 6.5. After 120 min, the equilibrium state was reached, and the optimum removal rate was obtained with a 1.5 g adsorbent loaded weight. The adsorption equilibrium capability of the lead-adsorbing materials was evaluated and estimated utilizing linear Freundlich and Langmuir isotherms, with the experimental results fitting the Freundlich isotherm models.

Keywords: biosorbents, isotherm model, lead, Langmuir, removal

V zadnjem obdobju povzroča degradacija okolja vse večjo skrb in pozornost. Glavni problem je odlaganje težkih kovin in različnih organskih nečistoč. Z uporabo naravnih absorbentov, ki so okolju prijazni bi se lahko uspešno lotili reševanja problemov onesnaženja voda. Količino težkih kovin v vodnih raztopinah je v resnici že možno dokaj zmanjšati z uporabo različnih metod, vključno z absorpcijo površin kmetijskih ostankov. Svinec je ena iz med najbolj toksičnih težkih kovin, ki se lahko nahaja v industrijskih odplakah. V članku avtorji predstavljajo raziskavo v katero so vključili izbrane bio absorbente in sicer liste drevesa moringa, riževe lupine in kokosova vlakna. To so relativno ceneni bio materiali, ki se jih lahko uporabi za izločanje svinca iz industrijskih odplak. V raziskavi so avtorji ugotavljali sposobnost izbranih naravnih bio absorbentov za odstranitev zelo toksičnih ionov Pb^{2+} iz vodnih raztopin. Kot pomembne dejavnike za ovrednotenje mehanizmov absorpcije so izbrali in zasledovali kontaktni čas, koncentracijo, dozo absorbenta in pH vrednost. Ugotovili so, da je učinkovitost absorpcije odvisna od zmanjševanja kislosti oziroma pH raztopine v območju med pH je 2,5 in 6,5. Po 120 min je bilo doseženo ravnotežno stanje in ugotovljena optimalna hitrost absorbcije, ki je bila 1,5 g na izbrano maso absorbenta. Avtorji so ravnotežno absorpcijsko kapaciteto izbranih materialov ovrednotili in ocenili z uporabo linearnih Freundlich-Langmuirjevih izoterm s prilagoditvijo raziskovalnih rezultatov na izotermne Freundlichove modele.

Ključne besede: bio absorbent, izotermalni model, svinec, Langmuirjev model absorpcije, odstranitev

1 INTRODUCTION

Heavy metals are used in industry applications in a multitude of sectors. These companies' wastewater effluents eventually release a few of those pollutants into the atmosphere. Owing to their acute toxicity and buildup in food chains, heavy-metal poisoning of water is an important ecological disaster. Unlike organic contaminants, that are most likely recyclable, heavy metals do not degrade to safe final products. Even at low concentrations, heavy metals are harmful to marine environments. Copper (Cu), chromium (Cr), mercury (Hg) and lead (Pb) are the heavy metals harmful to humans and the environment. The human body may absorb, store, and collect these metals, resulting in erythrocyte disintegration, vomiting, salivation, diarrhea, muscle cramps, renal deterioration, persistent pulmonary difficulties, and skeletal deformities. Lead (Pb^{2+}) is a highly toxic heavy metal that poses a risk to human and environmental health.[1] The nervous and immunological systems are both severely harmed by it. Pb^{2+} is used in industrial operations such as battery manufacture, electroplating, petrochemical operations, printing pigments and biofuels. The process industry's harmful byproducts, energy storage, insecticides, plastic water systems, food, beverages, ointments, and pharmaceutical concoctions for flavoring and seasoning are all sources of the lead in water.

Chemisorption, membrane separation, ion exchange, biosorption, and adsorption are some of the ways for removing hazardous or heavy metals from aqueous solutions. Adsorption is the recommended approach for removing hazardous or heavy metals, and it produces the best outcomes because it can be used to eliminate a variety of dangerous elements. Due to its efficacy and higher adsorption capacity, commercial activated carbon (CAC)

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is the most extensively utilized method for eliminating of hazardous metals; nevertheless, its application is still restricted due to higher operating costs.[2] CAC also raises issues about the requirement for regeneration and the difficulties of separating it from the effluent after usage. To lower the cost of pollution remediation, many studies have focused on identifying non-traditional alternate adsorbents. Non-hazardous wastes from industry, farming, and bio sorbents all seem to be examples of lower-cost adsorbents.

Some studies have focused on using a waste product as a sorbent even though:

- it is prevalent,
- most kinds of agricultural wastes are easily utilized and therefore do not necessitate a complex processing step or model that enables before implementation,
- rejuvenation of these adsorbent materials may not be a requisite,
- the procedure requires less maintenance and supervision.

Nevertheless, by the need for post-use disposal, the application of these low-cost options for pollution management is currently restricted. The usage of moringa leaves, rice husk, and coconut fibers for wastewater remediation is a winning strategy since it not only converts trash into a useful resource, but it also eliminates garbage from being burned on-site, reduces waste production, and lowers CO_2 emissions. The comparatively lower adsorbing capability of bio-sorbents is indeed a serious barrier. Nevertheless, in certain circumstances, activating, surface functionalization, and sonication can boost adsorption capability. Due to their microporous architectures and extensive diffusion paths through solid materials of adsorbents, activated carbons derived from plant sources frequently suffer from lower adsorption rates.[3] The most significant quality to examine when evaluating the adsorption of metal ions from effluent is a higher adsorption ability, which would be connected to a larger surface area to volume ratio.

Precipitation, solvent evaporation, ion exchange, and membrane filtration are all ways for removing heavy metals, but only adsorption appears to be an economically viable choice at the present time. Adsorbent as a unit process in residential water and municipal wastewater treatment operations is becoming increasingly prevalent in current plants. Over the years, a considerable study has been done on heavy metal ion adsorption, but it is still ongoing. Chemical adsorbents, zeolites, bio-adsorbent materials, and nano-adsorbents are some of the adsorbents that were suggested.[4] Nevertheless, most suggested adsorbents have not yet proven to be cost-effective owing to separation and regenerating; therefore, research into the optimal adsorbent for heavy metal ion elimination remains. In the past, several therapy methods have been utilized in the elimination of lead in water. These contain the use of catonic resin purolite, adsorption using high alumina content bauxite, alkali ash material permeable reactive barrier, the use of specific lactic acid bacteria, electrocoagulation process, adsorption using natural American bentonite and activated carbon, using iron nanoparticles, using okra, rice hush, maize and saw dust. The usage of adsorption methods in the management of heavy-metal wastewater has gained a lot of popularity. Adsorption is the prevention of the movement of adsorbates on the adsorbent. Isotherm adsorption studies have been employed in the effluent method to forecast the capability of certain adsorbents to eliminate pollutants.

The lack of modern wastewater-treatment technologies coupled with the unavailability of skilled personnel, results in water contamination and consequent upon the unavailability of potable water. The need for low-cost wastewater-treatment alternatives necessitated this study. In certain developing nations, poor economic conditions necessitate lower-cost water-treatment options for impacted communities, as conventional or sophisticated removing contaminants is not feasible. Plant-based substances for treating water are ecologically responsible, expensive, non-toxic, easy to conduct, as well as the residual produced is easily disposed of even though they are available nearby.[5]

A number of natural-based compounds have been investigated as lower-cost coagulants for water treatment. Because waste material is recycled to minimize produced in an environmentally beneficial manner, the usage of bio-sorbents in water filtration fits well to the area of environmental stability. Whenever these kinds of biomass materials were utilized for water treatment, adsorption was thought to be a key process for removing contaminants.[6] Despite the fact that research studies have focused a lot of emphasis on the use of organic fibers as water purification agencies to eliminate types of contaminants, there is still an insufficient knowledge of the mechanisms at work in the treatment method. One heavy metal's removal technique may differ from that of another. As a result, rather than focusing on the utilization of biomass, it is important for understanding the mechanisms that contribute to the elimination of a particular heavy metal from an aqueous phase. Because of its highly enhanced metal-binding capability, adsorption is a commonly utilized approach for the selective extraction of lead ions, and could improve human health by enhancing the treated wastewater quality. Moreover, the activation compounds could be easily recycled and reused. Carbon activation with phosphoric acid is commonly employed in large-scale uses to saturate lignocellulose materials like coir and wood. Phosphoric acid boosts the yield by altering the pyrolysis process disintegration of lignocellulosic materials along with depolymerization, dryness, and distribution of components.[7,8] Because of the advancements in the acid recovery period, phosphoric acid has grown increasingly popular in recent years. As previously said, finding lower-cost material to make activated carbon is naturally a major undertaking.[9,10]

Figure 1: Raw moringa leaves, rice husk and coconut fiber

The aim of the current research was designed to eliminate Pb^{2+} ions from an aqueous solution through a biosorption process by employing biological materials like moringa leaves, rice husk and coconut fibers, as shown in **Figure 1**. Moringa leaves are a natural material with a high adsorption capacity for eliminating heavy metals from wastewaters. The use of moringa leaves, rice husk, and coconut fibers as adsorbents for removing heavy metals are discussed in this article. These are low-cost amino acid-containing materials. The amino acid is a key component of functional groups that assist in heavy-metal elimination via metal ion exchanges or complexation that was influenced primarily via pH, bio-sorbent dosage, and contact duration. The goal of this research was to see if extant adsorption isotherm method can accurately define the behavior and therapeutic effectiveness of the moringa leaves, rice husk, and coconut fibers in expelling Pb, as well as to learn more about the adsorption process of a specific heavy metal explained by the concepts.

2 EXPERIMENTAL PART

2.1 Preparation of precursors

The ground water samples containing Pb were collected from the river Cauvery, Karur District, Tamilnadu. The heavy metals of Cu, Cd, Fe, and Pb were higher in the river-water samples. The pH of the solution is found to be 5.4. The concentration of metals in the river samples is shown in **Table 1**. The chemicals employed in the study contain, HCl, H_3PO_4, KOH and NaOH were of analytical grade and attained from Sigma-Aldrich (Australia) and utilized without further refinement. The pH of the water specimens was adjusted by employing either 0.1-M HCl and 0.1-M NaOH. The moringa leaves, rice husk and coconut fibres were collected from Kanya-Kumari District, Tamil Nadu, India, as it is readily available in villages.

2.2 Processing of activated carbon (ACC)

The leaves of moringa were washed by deionized water for 5 min to remove any suspended materials on their surface and desiccated in an oven at 70 °C for 72 h. The dried samples were ground using a domestic blender and separated over a 200-μm stainless-steel sieve. The biosorbent samples were stored in glass bottles for further studies.

To eliminate dust and grime, the rice husk was cleaned and rinsed with deionized water. The rice husks were then steeped in hot water for 1 h, then dried in sunlight for 4 h before being dried again with an oven dryer at 120 °C for 1 h. After drying, the products were pulverized in a ball mill and sieved on a 1-mm mesh size. Lastly, rice husks were roasted for 3 h at 120 °C before being kept in a desiccator. Several grams of rice husk were digested in a 0.5-M (molarity) NaOH solution. This was performed to separate silicates from rice husks, enabling adsorption procedures to function more smoothly later. Sodium silicate is formed when NaOH enters the cellulose framework and interacts with the silica in the rice husk even during the setup process. The introduction of NaOH during activating will create a few additional sites on the adsorbent's surfaces, increasing adsorption capability. The rice husk and NaOH combination is then agitated at room temperature for 60 min at 80 min^{-1} revolutions. The mixture of NaOH and rice husk was then rinsed with distilled water many times until it reached a pH of ± 7 and drying for 2 h at 50 °C. 1 g of neutralized rice husk was combined for 2 h at 80 min^{-1} with 100 cm^3 of 0.5 M HCl, then drying for 3 h at 50 °C. After that, the altered rice husks were rinsed with distilled water to eliminate excess HCl and re-dried at 50 °C for 24 h. For more research, the bio sorbent specimens were housed in glass jars.

Coconut coir was chopped into small pieces and cleaned in hot distilled water before being oven roasted for 12 h at 110 °C. The coconut coir was then filtrated through with a 1 mm screen after it had been washed. About 25 g of sieved coir was cooked for a few hours after being handled with 500 mL of 50 % phosphoric acid. The phosphoric active ACC was then oven baked at 100 °C for 48 h before being pyrolyzed at 450 °C for 1 h. Allowing the specimen to approach room temperature, it must have been thoroughly cleaned with distilled water before being treated with lower concentration KOH to eliminate any residual phosphoric acid till the pH of the water reached the 6.0–7.0 level. The ACC then was baked at 90 °C for 4 h. The bio-sorbent samples were stored in glass bottles for further studies.

2.3 Press-cake preparation

The activated moringa leaves, rice husk and coconut coir were pressed mechanically and examined in water therapy. The residual chemicals were extracted from the press cake by soaking it in water overnight, as well as the clean pressing cake was employed in this research. To achieve the proper mass for the bio sorbent applied to water, the water content was determined.

2.4 Batch adsorption study

For 4 min, the stirring rate was set to 200 min^{-1}, then 40 min^{-1} for 30 min. All therapies were carried out in triplicate, and after each optimization, the solutions were left to settle for 60 min before being filtered with filter papers. Lastly, the residual heavy metals concentration of each solution was examined by employing Thermo Fisher Scientific iCE 3500 AAS Atomic Absorption Spectrometer and shown in **Table 1**.

Table 1: Initial and final concentration of heavy metals in samples after inserting press cake

Metal	Initial (mg/L)	Final (mg/L)	Removal (%)
Pb	1.718	0	100
Cu	1.64	0.29	82.31
Cd	1.306	0.035	98
Fe	1.054	0.019	98

3 RESULTS AND DISCUSSION

3.1 Influence of pH on the elimination of Pb^{2+}

Figure 2 exemplifies the variation in the elimination efficacy of Pb^{2+} versus the final pH of solutions comprising moringa leaves, rice husk and coconut coir. An amounts of metal removal by moringa leaves, rice husk and coconut coir were significantly exaggerated by changing the pH of a solution. At a lower pH ranging from 2 to 3.5, adsorption of Pb^{2+} utilizing moringa leaves, rice husk and coconut coir was relatively lesser ranging from 12 % to 45 %. At greater pH (from 3.5 to 6), the amount of Pb^{2+} elimination enlarged remarkably nearby 100 %. This relation among the pH and the quantity of Pb^{2+} uptake through moringa leaves, rice husk and coconut coir can be accredited to the circumstance that at lower pH values, the surface functional assemblies, essentially carboxylic acid functional assemblies of the bio sorbent, get protonated through H^+ ions, results in a net positive charge on the exterior of the bio-sorbent, as explicated by Equation (1). Thus, this makes a repulsive force among Pb^{2+} cations and the positively charged exterior of the rice husk, impeding the biosorption method.

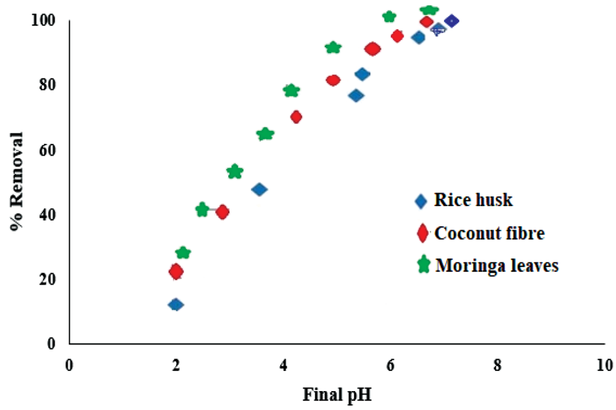

Figure 2: End pH of the solution versus the elimination amount of Pb^{2+} by moringa, rice husk and coconut fiber

Moreover, the rise in the H^+ ions generate a competition among them and the metal ions over the energetic sorption spots on the exterior of the bio sorbents.

$$R - COOH + H^+ \rightarrow R - COOH_2^+ \quad (1)$$

The surface functional subunits, on the other side, are stimulated as the pH rises. The carboxylic acid group deprotonate and disintegrate into carboxylate anions (R-COO_) at greater pH values that happens in the pH levels of 3.5–5.5, which corresponds to the acid dissociation constant (pKa) of carboxylic acids. The negative charges interface of a rice straw inevitably causes an electrostatic attraction seen between positive ions of Pb^{2+} and the negative charges interface of the bio sorbents, facilitating the biosorption. Furthermore, as the pH of the solution rises, the concentration of H^+ ions drops, reducing competition among H^+ ions and Pb^{2+} for binding sites. Furthermore, the pH of a solution disturbs the solubility of metal ions; hence, when the pH rises, the solubility of metal ions falls, making the sorption process easier. The rise in percent elimination at pH > 6 is mostly owing to precipitation, as seen in **Figure 2**, since insoluble metal species of Pb^{2+} develop at pH > 6. All the Pb^{2+} removal is obtained by activated moringa leaves adsorbent. Activated coconut fiber and rice husk shows Pb^{2+} removal rate above 98 %.

3.2 Effect of concentration on the elimination of Pb^{2+}

The proportion of lead removed is greatly influenced by the original levels of lead. The instantaneous relationship among the lead concentration as well as the accessible binding affinity on an adsorbent surface determines the influence of the first Pb^{2+} concentration factor. The amount of lead removed generally reduces as the initial lead concentration rises, which might be owing to the saturated adsorption sites on the adsorbent, or because the adsorption rate improved as the original lead level. There are unfilled active spots on the surface of the adsorbent at lower concentrations, and then when the initial dye concentration rises, the adsorption sites needed for Pb^{2+} adsorption would be unavailable. The rate of re-

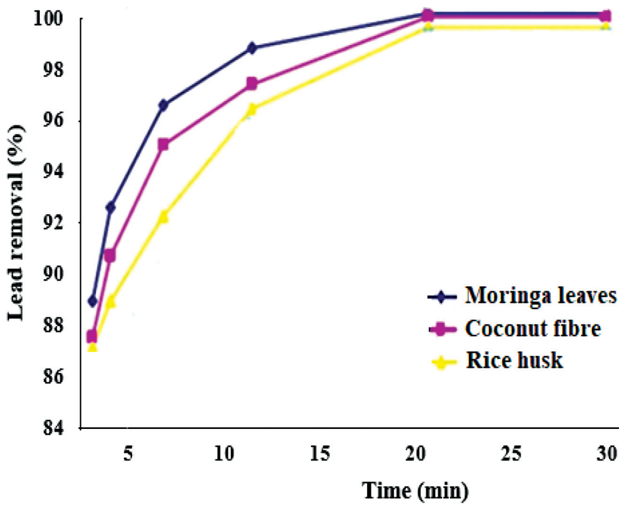

Figure 3: Percentage removal of Pb^{2+} by moringa, rice husk and coconut fibre

moval of Pb^{2+} by the moringa leaves, rice husk and coconut fiber are shown in **Figure 3**.

3.3 Impact of contact period on the elimination of Pb^{2+}

As shown in **Figure 4**, the contact period was changed from 15 min to 120 min to see how it affected the lead elimination. Initially, when the contact duration improved, the lead extraction efficiency improved as well. This could be due to the fact that metal binding period on the active surface of adsorbent rises. The extreme lead elimination efficacy was identified as 100 % at 90 min for moringa leaves and 98.7 % at 90 min for rice husk and 98.2 % at 90 min for coconut fiber, respectively.

3.4 Impact of adsorbent dosage on the elimination of Pb^{2+}

The adsorbent dosage was changed from 5 g/mL to 40 g/mL to see how effective it was at removing lead. **Figure 5** shows that at 25 g/mL, removal varied from 37 % to 100 % for moringa leaves, 41 % to 98 % for coconut fiber, and 38.4 % to 98 % for rice husk. Owing to the accessibility of more binding sites over the surface area of the adsorbent materials, the rate of lead extraction improved when the adsorbent dosage was adjusted from 5 g/mL to 15 gm/L, making metal (lead) penetrating simpler to the adsorbent surface. The removal efficiency reduced as the adsorbent dosage was enhanced to 25 g/mL, as well as the rate of elimination remained nearly constant up to 40 g/mL. Leading to a shortage of surface area and overlapping active surface area, the rates of lead elimination slow at increasing adsorbent doses. The optimal dosage was 25 g/mL, having a lead removal efficacy 100 %, 98.7 % and 98.2 % for moringa leaves, rice husk and coconut, respectively; indicating moringa leaves is an improved adsorbent amongst all the three verified adsorbents.

3.5 Adsorption isotherms

The adsorption isotherm (AI) is a useful model for describing adsorption behavior. It explores the purpose and mode of the adsorption method by describing how the adsorbate reacts well with the adsorbent. The AI can show the dissemination of adsorbate compounds among the solid and liquid stages when the adsorption mechanism achieves equilibrium. The equilibrium isotherm data generated from the various studies provide critical information on adsorption process as well as the adsorbent's surface characteristics and affinity. As a result, selecting a suitable correlation of equilibrium curves is critical for optimizing the parameters for building adsorption systems. Langmuir and Freundlich isotherms were employed to analyze the adsorption behavior throughout this study. **Figure 6** shows that the findings fit well to the Freundlich isotherm, indicating that lead ion adsorption on moringa leaves, coconut fiber, and rice husk is heterogeneous multilayer adsorption. Moreover, the "n" value of the Freundlich isotherm, which is greater than 1, indicates that the method is physisorption, as indicated in **Table 2**.

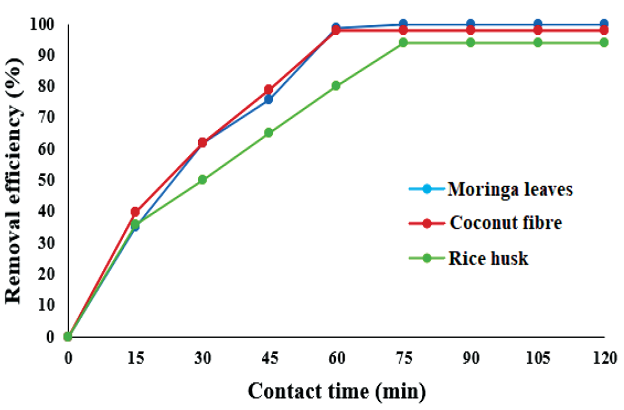

Figure 4: Impact of contact period on the lead elimination

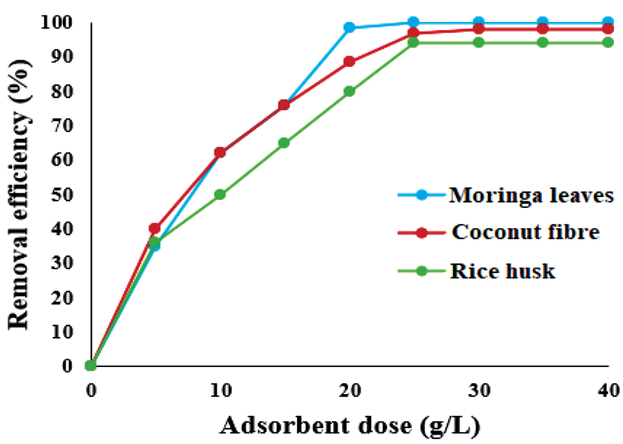

Figure 5: Impact of adsorbent dose on the lead elimination

Table 3: Adsorption factors of the Langmuir and Freundlich isotherms at room temperature for the adsorption of Pb^{2+} on moringa leaves, coconut fiber and rice husk

Biosorbants	Langmuir constant			Freundlich constant		
	q_{max} [b]	K/(L/mg)[b]	$R^{2a.b}$	K_f/(mg/g)[b]	n[b]	$R^{2\ a.b}$
Moringa leaves	41.05	0.0107	0.9626	3.4397	2.6690	0.9995
Coconut fibre	39.07	0.0104	0.9444	3.1286	2.4721	0.9893
Rice husk	37.98	0.0101	0.9189	2.9985	2.1997	0.9641

[a] R^2 is regression coefficient

[b] K, K_f, q_{mzx}, n, values and the nonlinear regression correlations for Langmuir isotherm study and Freundlich isotherm study were evaluated through nonlinear regression study

Because linear adsorption is defined as $n = 1$, chemical adsorption was defined as $n = 1$, and physical adsorption was defined as $n > 1$. The production of van der Waals bonds was thought to be responsible for the physisorption procedure. The kinetics analysis indicates that the adsorption mechanism is heavily favored by chemisorption. However, the adsorption process generally conforms the Freundlich isotherm theory to a $n = 2.21$, indicating that physisorption is used. Nevertheless, it can be hypothesized that engaged bio-sorbents adsorb lead ions via the van der Waal and covalent bonds, or that the very first adsorption layer occurs via covalent bonds, with subsequent layers occurring via van der Waal and covalent bonds.

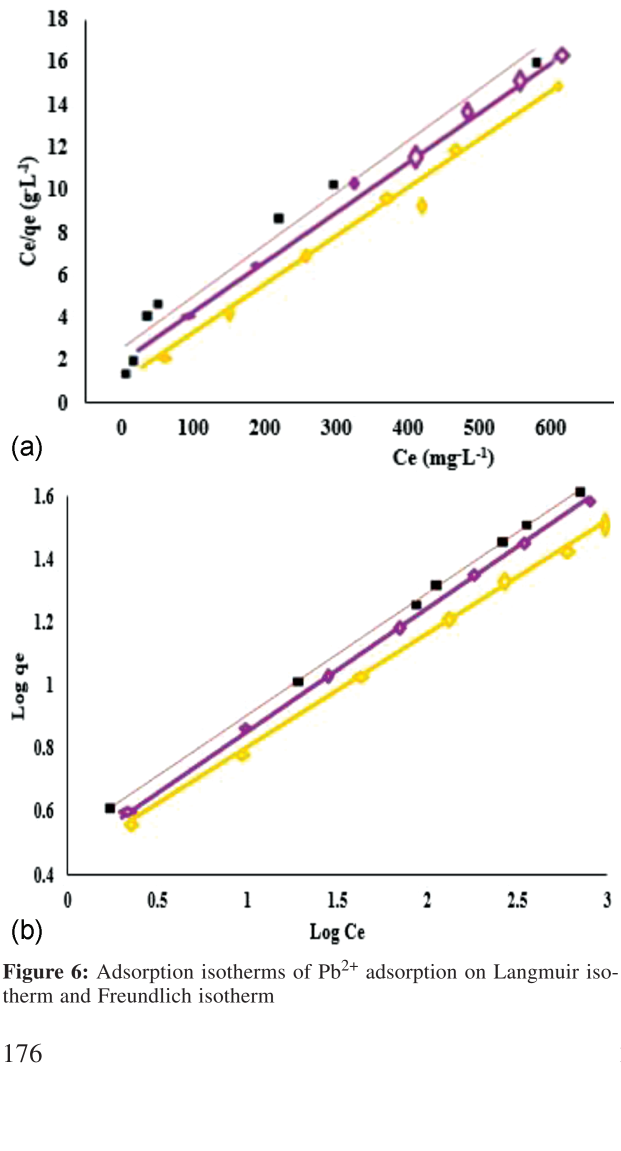

Figure 6: Adsorption isotherms of Pb^{2+} adsorption on Langmuir isotherm and Freundlich isotherm

4 CONCLUSIONS

In this paper we report on how we used activated moringa leaves, coconut fiber, and rice husk to eliminate lead ions from a tainted aqueous solution. The overall goal of this study is to create and use micro-porous activated adsorbent materials for effective lead-ion elimination from the aqueous stage. This research gives an ideal stage to change the moringa leaves, coconut fibre and rice husk, which is highly abundant in Kanya Kumari, Tamil Nadu, into activated carbon that can be commercialized for the elimination of Pb^{2+} impurities from the aqueous stage. The data obtained are well fixed with the Freundlich isotherm study that defined the adsorption process as heterogeneous multilayer adsorption. As per the research, it was determined that the manufactured activated carbons are possible candidate use at eliminating Pb^{2+} in sewage water to avert ecological impurity and related health hazards. Different chemicals were added to these adsorbents to improve their adsorption capability. However, the adsorbent was employed in this investigation without any chemical treatment or modification. As a result, in future investigations, its efficiency can be improved and assessed by chemically treating the adsorbent.

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