OPTIMIZATION AND ADSORPTION ANALYSIS OF LEAD REMOVAL USING MODIFIED MACROPHYTE BIO-ADSORBENT

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ABSTRACT: Lead contamination in wastewater presents a major environmental and public health concern due to its toxicity, persistence, and non-biodegradable nature. Although conventional treatment methods such as chemical precipitation and membrane filtration are commonly used, they often come with drawbacks including high operational costs, energy demands, and secondary pollution. As a sustainable alternative, this study investigates the potential of *Azolla*, an aquatic macrophyte, as a low-cost and eco-friendly bio-adsorbent for lead removal from contaminated water. The *Azolla* biomass was pre-treated with hydrochloric acid for five hours to improve its surface area and activate functional groups. Its lignocellulosic structure, rich in hydroxyl and carboxyl groups, supports effective lead adsorption via complexation and ion exchange mechanisms. A 2-Level Factorial Design was used to optimize key adsorption parameters including pH, contact time, and initial lead concentration. The highest removal efficiency of 93.69% was achieved at pH 10, a lead concentration of 250 ppm, and a contact time of 10 minutes. Adsorption isotherm analysis indicated that the process followed the Freundlich isotherm model, suggesting multilayer adsorption on a heterogeneous surface with multiple binding sites. These findings demonstrate the effectiveness of *Azolla* as a promising bio-adsorbent for lead removal. Further research is recommended to explore its long-term performance and scalability for industrial wastewater treatment applications.

KEY WORDS: *Aquatic macrophyte, Azolla, Adsorption, Lead removal, Lignocellulose*

1. INTRODUCTION

Dumped pollutants release heavy metals and cause harm to the environment and society's health. Removing heavy metals from water is a complicated task and requires constant attention and monitoring as heavy metals are major wastewater pollutants. One of the most hazardous metals is lead which is widely used especially in the industry that produces lead acid batteries, paints, and bangle industry. Leads posing significant environmental risk due to their cumulative toxicity and non-biodegradable nature [1]. They can cause carcinogenic, anemia, abdominal, muscle and joint pains, kidney problems and high blood pressure [2] . Conventional methods such as ion exchange, adsorption, chemical precipitation and membrane filtration require significant amounts of chemicals to facilitate the removal of metals, need specialized set-up equipment and infrastructure which can be both expensive and harmful to the environment and cause high initial operating [3] . Among that, adsorption method was reported as the most effective technique for heavy metals removal. It has low operating costs, high removal capacity, easy to implement and simple treatment process [4]. Adsorption is a surface phenomenon where molecules from fluid, either gas or liquid, accumulate on the surface of a solid or liquid at the interface between two phases. This results in a higher concentration of molecules at the interface compared to the bulk of the surrounding medium [5]. This has led to growing interest in biosorbents which are natural, low-cost, and eco-friendly materials for sustainable wastewater treatment. Biosorbents offer effective pollutant removal and align with green technology goals.

To support green technology development, aquatic macrophytes such as *Azolla*, a renewable and low-cost biomass material, were used as adsorbents. Macrophyte contains lignocellulosic plant cell walls, where the cellulose is surrounded by and linked to hemicellulose and lignin as shown in Fig. 1 [6]. In cellulosic biosorbents, adsorption mechanisms include metal-proton exchange, van der Waals forces, electrostatic interactions, surface complexation, and binding at reactive sites [7]. Studies show macrophytes can remove up to 99.4% of lead from wastewater [6]. Lignocellulose contains functional groups like carboxyl, hydroxyl, and thiol that help adsorb lead ions through electrostatic attractions as in Fig. 2 and make lignocellulose effective for lead removal [7].

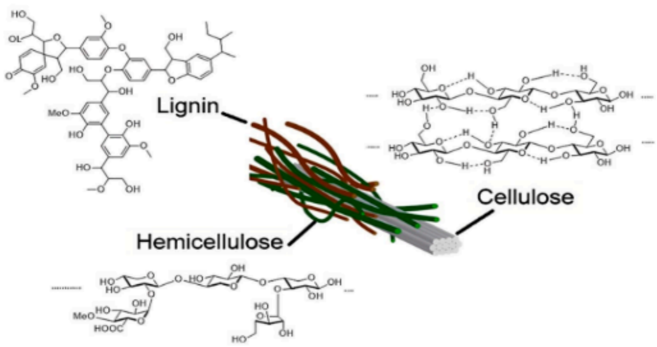


Fig. 1: Chemical structure of cellulose, hemicellulose and lignin (Source: [6])

Fig. 2: Schematic chemical reaction of lead ion and cellulose

The adsorption process is influenced by several key factors, including pH, temperature, adsorbate dosage, and contact time [8]. The pH of the solution significantly affects the adsorption efficiency, as low pH levels result in high hydrogen ion concentrations that compete with metal ions for active sites on the adsorbent, reducing adsorption capacity [9][10]. Temperature also plays a crucial role; in endothermic reactions, higher temperatures enhance adsorption by supplying the energy needed to overcome activation barriers, while in exothermic reactions, increased temperature may weaken the interaction between adsorbent and adsorbate, lowering efficiency [9]. The amount of adsorbate used also affect the adsorption capacity, higher initial lead concentrations enhanced the percentage removal due to increased mass transfer [11]. Contact time impacts how long the adsorbate is in contact with the adsorbent, with longer durations allowing more adsorption until equilibrium is reached [12].Optimizing these parameters is crucial for maximizing pollutant removal and ensuring effective performance of the biosorbent system. These parameter ranges were based on findings by Priyantha et al. [13].

Despite significant advancements in biosorption, a critical research gap remains in understanding the adsorption mechanism and process optimization when using macrophytes as biosorbents. Although previous studies have explored biosorption technologies for wastewater treatment, limited work has specifically focused on the efficiency and adsorption mechanism of macrophytes for lead ion removal as paper discussed by Al-Baldawi et al. [14] and Saralegui et al. [6].

Therefore, this study focuses on the development of a chemically modified macrophyte. Laboratory-scale batch adsorption aims to investigate the efficacy of modified macrophytes as cellulosic biosorbents for the removal of lead from synthetic wastewater. A series of experiments were conducted using 2-level factorial, following the design of experiments (DOE) generated by using Design-Expert 6.0.8 systematically vary key parameters including pH, initial lead concentration (ppm), and adsorption contact time (min) to determine their optimum condition for adsorption and impact on the adsorption process and to do the statistical analysis. Once the optimum condition is achieved, a series of experiments were carried out to determine the adsorption isotherm models to understand the adsorption behavior and mechanism of the process. The predominant retention of Pb in the root system enhances its applicability for rhizofiltration. Together with its rapid biomass production, ease of cultivation, and nitrogen-fixing ability, *Azolla* represents a promising and cost-effective candidate for integrated wastewater treatment systems, particularly in resource-limited and decentralized environments [15].

2. MATERIALS AND METHODS

2.1. Materials

Fresh *Azolla microphylla* was harvested in Selangor, Malaysia. Lead nitrate was purchased from R&M chemicals. All other reagents were commercial products of analytical grade. Deionized water was used for samples preparation. The experiments for process optimization and statistical analysis were designed using Design Expert® software version 6.0.8 (STAT-EASE Inc., Minneapolis, USA). Fresh *Azolla* can be illustrated in Fig. 3.

Fig. 3: Fresh untreated *Azolla microphylla*

2.2 Experimental procedure

2.2.1. Synthesis of bio-adsorbent

Synthesis of bio-adsorbent were done according to Nasrol et al. [16] with slight modification. *Azolla* plants were dried in an oven at 105 °C for 24 hours. The dried biomass was then crushed and sieved to obtain particles sized between 1–2 mm by using the blender and steel sieve. Subsequently, the biomass was treated with 0.1M HCl for five hours to optimize its adsorption properties by purifying and activating the material. Then, the sample were thoroughly washed with distilled water and dried in a shaded environment. The bio-adsorbent were stored at room temperature prior to use as in Fig. 4 below:

Fig. 4: Dried and treated *A. microphylla*

2.2.2. Design of experiment

The optimization of batch adsorption experiments was designed by using a 2-level factorial design with four center points, resulting in 12 runs as determined by Design of Experiment (DOE) statistical software (Stat-Ease, Inc., Minneapolis, USA). Each experiment was replicated three times, and the final concentrations of lead were measured as response. The experimental variables, including pH, initial lead concentration, and adsorption contact time, were summarized in Table 1.

Table 1: Range and levels of categorial factors by DOE

Variables	Unit	Type	Low Actual	High Actual	Low Coded	High Coded
pH		Numeric	4	10	-1	1
Initial lead concentration	ppm	Numeric	100	250	-1	1
Contact time	min	Numeric	10	70	-1	1

2.2.3. Batch adsorption

For the optimization adsorption experiment, 1000 ppm of synthetic wastewater stock solution was prepared by diluting a lead nitrate in deionized water. The adsorption experiments were conducted under different initial lead concentration (100 ppm, 175 ppm, and 250 ppm) by diluting lead stock solution in a 100 ml conical flask. Then for each solution, the pH was adjusted until the required pH values (pH 4, pH 7, and pH 10) using either NaOH (1 M) or HCl (1 M). The prepared *Azolla* of approximately 0.3 g was weighed and put in contact with 100 ml of the solutions containing lead and the mixtures were agitated using a laboratory shaker continuously with a constant speed of 180 rpm. After a pre-determined contact time (10min, 40min, and 70 min) the treated samples were collected and analyzed using a UV-Vis spectrophotometer to measure absorbance to determine the equilibrium concentration. The percentage of lead removed was calculated using Eq. 1.

$$\%removal = \frac{C_0 - C_t}{C_t} \times 100 \quad (1)$$

Where C_0 and C_t are the initial and equilibrium concentrations of lead in the solution, (ppm), respectively.

2.2.4. Adsorption Isotherm

Adsorption isotherm analysis was conducted to determine the behavior of lead ion adsorption onto the cellulosic macrophyte adsorbent. Six different initial lead concentrations (150 ppm, 250 ppm, 450 ppm, 200 ppm, 550 ppm, and 750 ppm) were prepared, and 0.3 g amount of macrophyte was added to each 100 mL conical flask containing lead synthetic wastewater. Then, the solution was shaken continuously using a laboratory shaker with constant speed of 180 rpm and fixed pH (pH 10) for 10 minutes at room temperature. These adsorption isotherm experiments were performed under the optimal conditions obtained. After the desired contact time, the treated samples were collected, and the absorbance was measured using a UV-Vis spectrophotometer.

Two widely used isotherm models, Langmuir and Freundlich, were applied to investigate the behavior of the adsorption process. The linearized equation of Langmuir and Freundlich model were used to calculate the linear regression values (R^2) [13]. The Langmuir isotherm model is given as in Eq. (2).

$$\frac{C_e}{q_e} = \frac{1}{bq_{max}} + \frac{C_e}{q_{max}} \quad (2)$$

where, q_e denotes the quantity of adsorbate adsorbed in mg/g, C_e denotes the equilibrium concentration (ppm), b is the adsorption equilibrium constant (L/mg) which is related to the adsorption energy, and q_{max} (mg/g) is the maximum adsorption capacity.

Second model is Freundlich isotherm model, which is expressed in a linear form as shown in Eq. (3).

$$\ln q_e = \frac{1}{n} \ln C_e + \ln K_f \quad (3)$$

where K_F is an adsorption capacity indicator and $1/n$ are an intensity of adsorption.

Adsorption equilibrium is determined from the uptake-time data. For both isotherm models, linear regression values (R^2) were calculated in search of the best-fitting model in describing the adsorption process. High value of R^2 (~1.0) shows that adsorption process fitted better in that particular isotherm model.

3. RESULTS AND DISCUSSION

3.1 Optimization of Adsorption

The results of the 12 batch adsorption runs are summarized in Table 2. The maximum lead removal efficiency of 93.69% was achieved within 10 minutes at pH 10 with an initial lead concentration of 250 ppm. The highest removal efficiency of this study is close to the findings reported by Saralegui et al. [6], who achieved 99.4% lead removal using *Azolla*. In contrast, the lowest removal efficiency of 52.76% was observed at the lowest pH (pH 4) and the longest contact time. In this study, pH, contact time, and initial Pb(II) concentration were treated as independent variables, while lead removal efficiency (%) was considered the response variable.

The effect of pH is highly impactful to the adsorption process. In this study, the lowest percentage removal occurred at the low pH and the adsorption improved at a higher pH. The reduced removal efficiency at low pH can be attributed to the high concentration of hydrogen ions (H^+), which compete with lead ions for the available active sites on the surface of the adsorbent, thereby limiting lead uptake. At a higher pH, the interaction between functional groups of the adsorbent and lead ions is very high as the hydrogen ions concentration was very low in an alkaline solution [16] as depicted in Fig. 5. From Table 2, when pH was low (pH 4), the lower efficiency of 58.22% was recorded with the same initial concentration and contact time.

Table 2: Removal Efficiency of Lead for 12 runs of Experiments

Run	pH	Contact time (min)	Initial Lead Concentration (ppm)	Absorbance (nm)	Final Concentration (ppm)	Removal Efficiency (%)
1	10.00	70.00	250.00	0.643	23.64	90.54
2	4.00	70.00	250	3.212	118.08	52.76
3	10.00	70.00	100.00	0.419	15.40	84.6
4	10.00	10.00	100.00	0.407	14.96	85.04
5	4.00	70.00	100.00	0.58	21.32	78.68
6	7.00	40.00	175.00	0.42	15.44	91.18
7	4.00	10.00	250.00	2.841	104.45	58.22
8	4.00	10.00	100.00	0.486	17.87	82.13
9	7.00	40.00	175.00	0.45	16.54	90.55
10	10.00	10.00	250.00	0.429	15.77	93.69
11	7.00	40.00	175.00	0.45	16.54	90.55
12	7.00	40.00	175.00	0.42	15.44	91.18

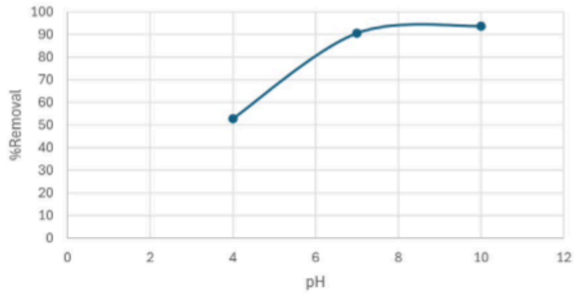


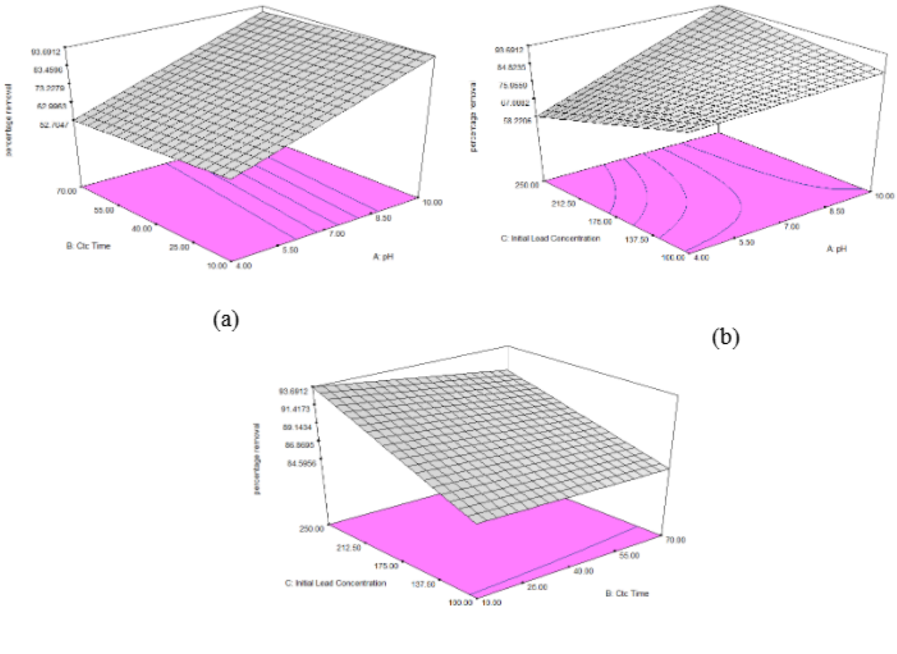
Fig. 5: Effect of pH on lead removal efficiency

Furthermore, from the observations, the results indicate that shorter contact times between *Azolla* and lead significantly enhance adsorption efficiency. This is due to the more availability of active sites on the *Azolla* surface for metal ion binding at the initial stage of adsorption [17]. Moreover, a higher initial concentration of lead improved the percentage removal [18]. This is attributed to the steeper concentration gradient, which facilitates mass transfer and increases the likelihood of collisions between lead ions and the hydroxyl functional group on the adsorbent surface [7].

The result of this study is consistent with the findings reported by Lawal, 2017 [11], who investigated the use of black walnut seed husk (WSH) as a biosorbent for the removal of lead ion from aqueous solutions. Their study revealed similar trends, further validating the current findings. It was demonstrated that several factors including pH, initial metal ion concentration, and contact time significantly influenced the biosorption process. At lower pH values, lead adsorption was notably reduced due to the competitive interaction between hydrogen ions and lead ions for the active binding sites on the bio adsorbent surface. This competition limits the availability of sites for lead ions, thereby decreasing adsorption efficiency. Additionally, their study showed that increasing initial concentration of lead ions enhanced the percentage of removal. This was attributed to improved mass transfer dynamics and a greater probability of interaction between metal ions and active sites on the adsorbent. Furthermore, a rapid uptake phase was observed within the first 10 minutes of the adsorption process, during which approximately 80% of the lead ions were removed. Equilibrium was achieved at around 60 minutes, indicating that the adsorption occurred swiftly and reached a stable state in a relatively short period. These findings underscore the effectiveness of WSH as a biosorbent and align closely with the outcomes reported in the present study.

A three-dimensional (3D) response surface plot was used to analyze the interaction of key variables affecting lead removal efficiency as in Fig. 6.

In Fig. 6(a), the combined effect of pH and contact time is shown while keeping the initial lead concentration constant at its highest level. Although a higher initial lead concentration generally improves removal efficiency, however, low pH and high contact time limit the extent of this improvement because of the competing ions such as hydrogen ions are also attached to the active sites. Fig. 6(b) shows that increasing pH and initial lead concentration enhances the removal efficiency. However, this scenario happens when the contact time is shorter as it is at the initial stage of the adsorption process [17] and increasing contact time might limit the adsorption process efficiency. Finally, Fig. 6(c) shows that the highest lead removal efficiency is achieved at the lowest pH, high initial lead concentration, and short contact time because of high collision between heavy metals and adsorbent [18] and low competing ions.



(c)

Fig. 6: Response surface plot as the function of (a) pH and contact time (b) pH and initial lead concentration (c) contact time and initial lead concentration

3.2 Statistical Analysis of Batch Adsorption

The results of the analysis of variance (ANOVA) were generated by Design-Expert software (Stat-Ease, Inc.USA: version 6.0.8). The relationship between experimental parameters and percentage removal were evaluated by generating diagnostic plots.

Table 3: Result of Analysis of Variance (ANOVA)

Source	Sum of Squares	df	Mean square	F-value	p-value	
Model	1541.82	7	220.26	1663.52	< 0.0001	significant
A	842.01	1	842.01	6359.29	< 0.0001	
B	19.53	1	19.53	147.51	0.0012	
C	155.06	1	155.06	1171.11	< 0.0001	
AB	3.54	1	3.54	26.75	0.0140	
AC	518.85	1	518.85	3918.60	< 0.0001	
BC	2.77	1	2.77	20.91	0.0196	
ABC	0.062	1	0.62	0.47	0.5421	
Curvature	426.97	1	426.97	3224.71	< 0.0001	significant
Pure Error	0.40	3	0.13			
Cor Total	1969.19	11				

As summarized in Table 3, the model is statistically significant. The model's F-value of 1663.52 suggests a strong fit, with only a 0.01% probability that such a high value could result from random variation or noise. P-values less than 0.0500 indicate significant model terms. In this study, the terms A (pH), B (contact time), C (initial lead concentration), AB, AC, and BC are all statistically significant. On the other hand, model terms with P-values greater than 0.1000 are considered not significant. The curvature F-value of 3224.71 further confirms the presence of significant curvature in the design space. This means there is a notable difference between the average responses at the center points and those at the factorial points, with only a 0.01% likelihood that such a result is due to random error. All statistical values from the batch adsorption design were displayed in Table 4.

Table 4: Statistical values of batch adsorption

Std. Dev.	0.36	R-Squared	0.9997
Mean	82.43	Adj R-Squared	0.9991
C.V.	0.44	Pred R-Squared	N/A
PRESS	N/A	Adeq Precision	129.873

The model shows a high level of accuracy, as indicated by the coefficient of determination (R^2) value of 0.9997, confirming that it fits the experimental data very well. The adjusted R^2 value of 0.9991 is also very close to the R^2, indicating the model's robustness and the significant influence of the selected variables. Although the predicted R^2 and PRESS statistics were not reported, this was due to a leverage value of 1.000, suggesting that one data point had a strong influence on the model. However, this case was not explored further, as the main objective of the study was to ensure a high R^2 value. Importantly, the model's Adequate Precision value of 129.873, far exceeding the minimum threshold of 4, indicates a strong signal-to-noise ratio. This confirms that the model has excellent precision and is reliable for navigating and optimizing within the experimental design space.

For optimization experiment, the goal to find the highest percentage removal was achieved. Three parametric factors was varied and the software computed that the most desirable condition was at pH of 10, 10 minutes of contact time and 250ppm initial lead concentration. The experimental result from Table 2 showed the highest lead removal of 93.69% was achieved. This is very close to the predicted removal percentage which is 93.68% with the percentage difference between experimental and computed was 0.004% as shown in Fig. 7. This difference was considered very small and acceptable. The desirability value was 1.000, indicating that the model is reliable and suitable for predicting lead removal under the given condition.

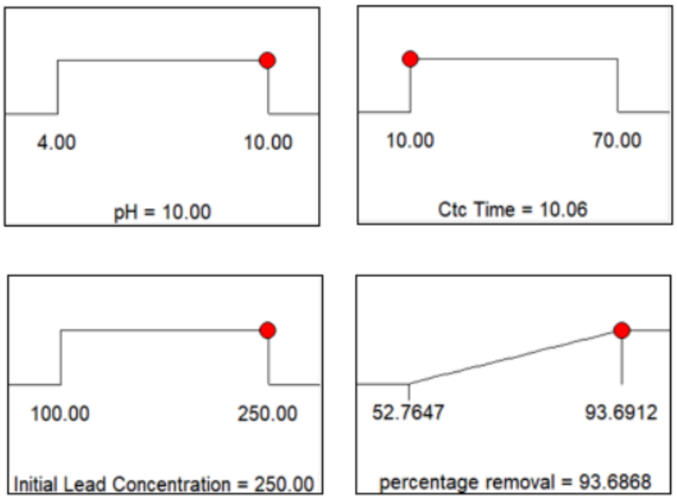
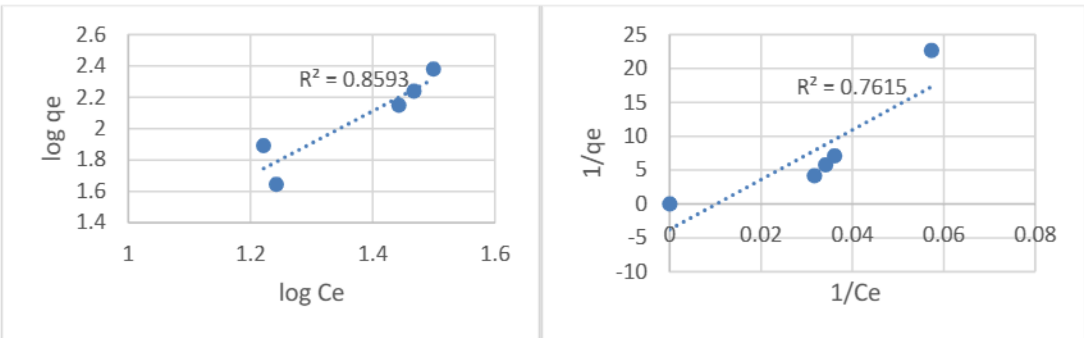


Fig. 7: The desirability effect for Lead adsorption on Azolla Macrophyte adsorbent

3.3 Adsorption Isotherm

The adsorption behavior of lead onto *Azolla* was investigated by comparing two common isotherm model (Langmuir and Freundlich). The removal efficiency was calculated, and the calculation of two isotherm were presented as in Table 4. The linearized plot of Freundlich isotherm and Langmuir isotherm were plotted in Fig. 8(a) and 8(b), respectively.



(a) (b)

Fig. 8: (a) Plot of Freundlich Isotherm (b) Plot of Langmuir Isotherm

Table 6: Estimated Isotherm Parameters

Isotherm model	**Langmuir**			**Freundlich**		
Estimated isotherm parameters	Q_{max} (mg/g)	K_L (L/mg)	R^2	*1/n*	K_F (mg/g)	R^2
	2.12857	0.0125	0.7264	2.0532	0.173	0.8593

High linear regression (R^2) represents the adsorption process is well-fitted with the Freundlich isotherm model at value of R^2 is 0.8593 compared to Langmuir isotherm model at value of R^2 0.7615 as shown in Table 6. The ideal fit of the equilibrium data from Freundlich isotherm expression predicted the multilayer adsorption capacity of lead onto *Azolla* was found to be 2.13 mg/g.

These findings are consistent with previous research, a study by Al-Baldawi et al. [14] involving yellow dye removal using magnetite-modified *Azolla* also confirmed that the adsorption behaviour followed the Freundlich isotherm Similarly, as reported by Lawal [11], the removal of lead ion from aqueous solution using Black WSH as bio adsorbent well-fitted with the Freundlich isotherm compared to Langmuir and Temkin where it supports the uptake of metal ions occurs on a heterogenous surface by multilayer adsorption.

4. CONCLUSION

The results of the 12 batch adsorption runs are summarized in Table 2. The maximum lead removal efficiency of 93.69% was achieved within 10 minutes at pH 10 with an initial lead concentration of 250 ppm, as identified through the Design of Experiments (DOE) approach. This is very close to the predicted removal percentage which is 93.68% with the percentage difference between experimental and computed was 0.004%. This difference was considered very small and acceptable. The adsorption study fitted well in Freundlich isotherm and the maximum adsorption capacity of led onto *Azolla* was 2.13 mg/g. The results confirmed the potential of *Azolla*, a natural aquatic plant, as an effective and low-cost bio-adsorbent for removing lead from wastewater

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