



Response Surface Optimization of Ultrasonic-Assisted *Nypa fruticans* Activated Carbon for Lead(II) Adsorption from Aqueous Solution

Muhammad Kamal Rusyaidi Mohtar¹, Inawati Othman^{1,2*}, Sumayya IK Mieta³,
Nor Azalina Rosli^{1,2}, Noor Hasyimah Rosman⁴, May Raksmei⁵

¹Department of Civil Engineering, University Malaysia Sarawak, Sarawak, Malaysia

²UNIMAS Water Centre (UWC), Faculty of Engineering, Universiti Malaysia Sarawak, Sarawak, Malaysia

³Department of Biomedical Sciences, Faculty of Health Sciences, University of Johannesburg, South Africa

⁴Department of Civil Engineering, Universiti Kebangsaan Malaysia, Selangor, Malaysia

⁵Department of Architectural Engineering, Institute of Technology of Cambodia, Cambodia

*Corresponding author: oinawati@unimas.my

ABSTRACT

Aims: The main objective of this study was to review and analyse the use of ultrasonically activated *Nypa fruticans* activated carbon (NFAC) for Pb(II) removal from aqueous solutions by applying Response Surface Methodology (RSM). **Methodology and results:** NFAC was synthesised from *Nypa fruticans* shells using potassium hydroxide (KOH) as an activating agent under ultrasonic treatment and characterised using SEM, BET surface area measurement, and FTIR spectroscopy. The optimal conditions of ultrasonic-assisted activation and adsorption variables were optimised using response surface methodology (RSM). The prime conditions for activation involved a 20 kHz ultrasonic frequency, 120 W ultrasonic power, and 15 min sonication, giving the highest BET surface area of 1328.74 m²/g. Through batch assay studies, the dose of adsorbent greatly affected the binding of Pb(II) ions. Equilibrium results were best fitted to the Langmuir isotherm model, indicating monolayer adsorption with a maximum saturated adsorption loading of 137.17 mg/g. The rate of reaction study showed that the pseudo-second-order kinetic model best represented the experimental results. **Conclusion, significance and impact study:** The ultrasonic-assisted activation using KOH greatly improved the performance of NFAC in Pb(II) adsorption, and hence the material can operate effectively as a feasible and promising removal agent for eliminating Pb(II) ions.

MANUSCRIPT HISTORY

- Received
January 2026
- Revised
May 2026
- Accepted
August 2026
- Available online
September 2026

KEYWORDS

- Activated carbon
- Nypa fruticans*
- Pb(II) adsorption
- Response surface methodology
- Ultrasonic-assisted activation

1. INTRODUCTION

The effects of fast-paced urbanisation and industrialisation are placing immense strain on our planet's freshwater sources. These effects are reflected in terms of water pollution that is prevalent around the globe. Heavy metals pose serious threats to freshwater sources due to their persistent and toxic characteristics. High concentrations of heavy metals have been detected in freshwater sources across the world. A surplus presence of heavy metals has been noted in Malaysia through liquid specimens collected from the Langat River catchment. These high concentrations could be attributed to both biogenic, geogenic and man-made sources in the form of industrial wastes, mining and urban runoffs (Kumar *et al.*, 2019; Ahmed *et al.*, 2020). Such pollution cases have also been recorded in other developing nations, indicating the prevalence of heavy metal pollution (Kumar *et al.*, 2025). One example is the River Dilimi in Nigeria, in which Ezekiel and Dikam (2020) found Pb concentrations 6,400% above the recommended permissible limits.

Heavy metals are toxic pollutants, posing serious health concerns to human beings even at low exposure. The main cause of heavy metal poisoning is contamination of water due to corrosion of pipelines or discharge by industries. According to the World Health Organization (2022), chronic exposure to Pb(II) leads to neurotoxic effects, poor cognitive functions, and cardiovascular problems. Furthermore, according to Gupta *et al.* (2019), heavy metals are responsible for considerable health hazards. Therefore, purification of lead (Pb) in polluted aqueous environments is an important ecological issue.

Adsorption is considered one of the highly effective ways of eliminating heavy metal ions from wastewaters, because of its ease, feasibility, and efficiency. The effectiveness of heavy metal ions' adsorption depends on several factors, such as specific area, pore diameter and functional moiety, along with characteristics of precursors of preparation and activation. The use of potassium hydroxide for chemical activation is an extremely efficient way of creating porosity and improving the ion-binding ability of organic-based carbonaceous adsorbent (Borghei *et al.*, 2021). Adsorption behaviour can usually be assessed using mathematical models, namely the Langmuir and Freundlich isotherms, as well as the pseudo-first-order and pseudo-second-order kinetic equations (Al-Ghouti & Da'ana, 2020; Kecili & Hussain, 2018).

Nypa fruticans, an abundant mangrove biomass in Southeast Asia, has attracted interest as a

potential precursor for activated carbon production. Previous studies have demonstrated its potential in producing activated carbon and removing contaminants such as iron and manganese. Still, its application for Pb(II) removal remains limited (Taslim *et al.*, 2021; Syauqiah *et al.*, 2020). Furthermore, while KOH activation is widely used to refine the surface attributes of biomass-derived carbons, studies have indicated that ultrasonic-assisted techniques can further improve pore development and adsorption performance. However, the combined application of ultrasonic-assisted KOH activation specifically for *Nypa fruticans*-based activated carbon has not been thoroughly researched (Nasseh *et al.*, 2021; Eslami *et al.*, 2020; Bora *et al.*, 2021). Moreover, systematic optimisation of both preparation conditions and adsorption parameters using Response Surface Methodology (RSM) is still limited.

Therefore, this study aimed to develop and evaluate ultrasonic-assisted KOH-activated carbon derived from *Nypa fruticans* for Pb(II) ion binding from aqueous solutions. Response Surface Methodology (RSM) was used for optimisation of the ions' uptake procedure. The steady state and kinetics of the ion-binding process were examined through the surface adsorption models and the rate reaction models, respectively.

2. RESEARCH METHODOLOGY

The current work is based on the synthesis of ultrasound-assisted KOH-activated carbon from *Nypa fruticans* shells for Pb(II) ion binding from aqueous solutions onto the surface of the adsorbent. Experimental techniques used in this study comprised three major stages, which are the synthesis and activation of NFAC, characterisation of the synthesised adsorbent, and assessment of its adsorption capacity by batch adsorption experiments. Surface morphological analysis, specific area and surface moiety analyses of the NFAC were performed to assess its physical properties. Correspondingly, adsorption studies along with optimisation were performed using Response Surface Methodology (RSM).

2.1 Preparation of NFAC

The shells of *Nypa fruticans* were collected, thoroughly washed, and dried in sunlight. The moisture was completely removed from the precursor through drying in an oven at 100 °C for 24 hours. The dried biomass was carbonised at 500 °C for one hour in a furnace and further

milled to produce carbon particles with dimensions ranging from 0.15 to 0.30 mm.

For catalytic activation, 10 g of the carbonised material was mixed with 1 M KOH solution and stirred continuously for 1 hour. It was further filtered and oven-dried at 100 °C for 24 hours to obtain chemically activated carbon.

Ultrasonically-assisted activation involved the mixing of 10 g of carbonised material with 1 M KOH solution in an ultrasonic bath under different conditions of power (120 to 180 W) and treatment time (15 to 60 minutes) at a fixed frequency of 20 kHz. The activated carbon obtained was further desiccated in an oven at 100 °C for 24 hours.

2.2 Characterisation

In order to examine the surface texture of NFAC, scanning electron microscopy (SEM) analysis was operated by focusing on the structural and textural aspects of the material. The samples were attached onto a sticky carbon tape and analysed using a beam voltage of 15 kV.

Specific surface and pore diameter distribution of NFAC were determined via the Brunauer-Emmett-Teller (BET) method. Prior to analysis, a sample with a 0.2 g mass was heated at 300°C for 3 hours to remove any impurities from the samples.

The moieties on the surface of NFAC were identified through Fourier-Transform Infrared (FTIR). First, a pellet of NFAC was prepared using the KBr pellet method by grinding 1 mg of NFAC to 20 mg of KBr.

2.3 Batch Adsorption

Batch assays were performed to investigate the adsorption of Pb(II) using NFAC. The influence of various parameters such as the initial Pb(II) ion concentration (10–50 mg/L), adsorbent dosage (0.1–0.5 g), and interaction time cycles (30-150 minutes) was examined in order to find out how these parameters affected adsorption performance. In all of these investigations, an appropriate amount of Pb(II) solution was mixed with a certain amount of NFAC under stirred conditions, and the mixture was further investigated in terms of the binding performance of NFAC active sites after the adsorption operation. The RSM method was applied to optimise the adsorption process. In this regard, Minitab 17 software was used based on second-order polynomial models.

2.4 Experimental Design and RSM

Response surface methodology was utilised to determine the optimum conditions for the Pb(II) adsorption process. A total of 51 replicates were performed using NFAC with optimised surface area to investigate the influence of Pb(II) saturation, adsorbent dose, and interaction time cycle on the adsorption efficiency. Conditions for experiments were varied in accordance with the design of experiments. For proper boundary condition and nonlinear behaviour analysis, a custom RSM structure for five levels was chosen, along with enough degrees of freedom for quadratic curvature analysis. Table 1 shows the factor levels used in the 5-level custom response surface design. Data obtained were analysed based on a polynomial model in order to examine interaction and optimise the adsorption conditions. The ANOVA method was carried out to determine the validity of the model.

Table 1 Factor levels used in the 5-level custom response surface design

Factors	Unit	Coded (-2)	Coded (-1)	Coded (0)	Coded (1)	Coded (2)
Concentration	Ppm	10	20	30	40	50
Dose	g	0.1	0.2	0.3	0.4	0.5
Time	min	30	60	90	120	150

2.5 Adsorption Isotherm

The adsorption isotherm equation was used to determine the relationship between the equilibrium level of Pb(II) ions in the aqueous phase and their adsorption onto NFAC at a given temperature. The Langmuir isotherm equation was used to evaluate the adsorption in one layer on a homogeneous surface (Liu et al., 2019). The linear form of the Langmuir equation was obtained from Equation 1.

$$\frac{C_e}{q_e} = \frac{1}{Q_o b} + \frac{C_e}{Q_o} \quad (1)$$

The term q_e is the balanced adsorption loading (mg/g); C_e is the balanced concentration (mg/L); Q_o is the saturated adsorption loading in a single layer (mg/g), while b is the Langmuir constant (L/mg).

For heterogeneous adsorption and adsorption consisting of more than one layer on the surface, the Freundlich isotherm was employed to analyse the process (Al-Ghouti & Da'ana, 2020). The linear form of the Freundlich equation is expressed as shown in Equation 2.

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \quad (2)$$

Where the term K_F denotes the sorption loading, and $1/n$ represents the sorption binding strength and layering of the surface.

2.6 Adsorption Kinetics

Rate of reaction studies have been carried out to study the reaction dynamics of Pb(II) ions loading on NFAC, focusing on exposure time between the ions and the adsorbent under invariable parameters. Kinetic modelling provides valuable insights into the adsorption mechanism and identifies rate-controlling factors, such as surface interactions and mass transport (Kamal *et al.*, 2017). The pseudo- first-order (PFO) adsorption kinetics model assumes that the rate of occupation of adsorption sites is proportional to the number of vacant sites available. This equation can be written as in Equation 3.

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} t \quad (3)$$

Where q_e and q_t (mg/g) are the equilibrium and time-dependent adsorption capacities, respectively, while k_1 (min^{-1}) is the pseudo-first-order kinetics rate constant.

In the pseudo-second-order (PSO) kinetic equation, it assumes that the adsorption process involves chemisorption where there is interaction between the adsorbent and adsorbate due to valence forces, including electron sharing and/or electron exchange. Equation 4 represents the PSO kinetic equation:

$$t/q_t = \left(\frac{1}{k_2 q_e^2} \right) + \frac{t}{q_e} \quad (4)$$

Where q_e and q_t (mg/g) are equilibrium and time-dependent adsorption capacities, respectively, while k_2 (g/mg min) is the pseudo-second-order kinetics rate constant.

3. RESULTS AND DISCUSSION

In this section, the findings of the characterisation of NFAC, optimisation of adsorption of Pb(II) using response surface methodology (RSM), and the adsorption performance of NFAC by comparison, isotherms study and kinetic modelling are presented. The impact of ultrasound-assisted activation on the structural properties and adsorption performance of NFAC for Pb(II) ion adsorption is addressed.

3.1 Characterization of NFAC

3.1.1 SEM Results

Figures 1 and 2 illustrate the morphology of the NFAC samples. The non-activated KOH-activated carbon possessed a fairly compact honeycomb structure with an arithmetic mean pore size of about 6.67 μm . On the other hand, the ultrasonic-activated carbon possessed a defined honeycombed-like structure, having a relatively large average pore size of about 12 μm .

The use of ultrasound during the activation process increased porousness by increasing the surface expanse. The same results have been observed in other investigations on ultrasonically activated carbon, and this phenomenon has been attributed to cavitation during the ultrasonic-activation procedure (Nasseh *et al.*, 2021). A highly developed porous structure is essential in any adsorption process, as it facilitates diffusion into the pores and increases the availability of adsorption sites.

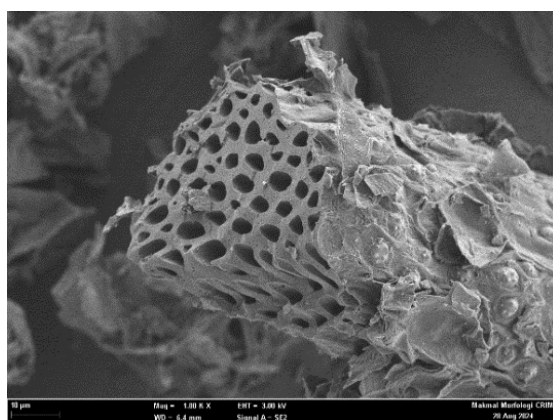


Figure 1 SEM of *Nypa fruticans* carbon activated with KOH at 1,000x magnification

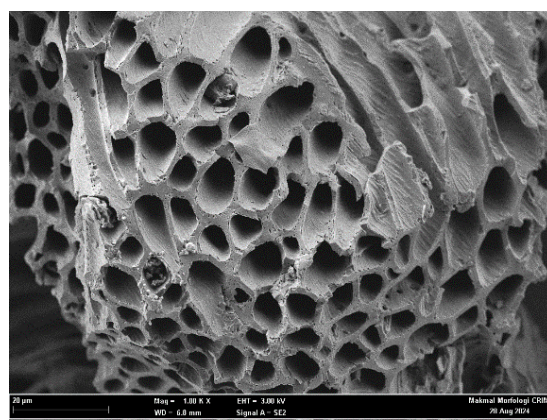


Figure 2 SEM of *Nypa fruticans* carbon activated with KOH assisted with sonification at 1,000x magnification

3.1.2 FTIR Analysis

The FTIR spectra of *Nypa fruticans* raw carbon, KOH-activated carbon, and ultrasonically-assisted NFAC are shown in Figures 3, 4, and 5, respectively. The raw carbon showed intense broad peaks centred at 3,200-3,550 cm^{-1} , which can be ascribed to the O-H stretching band of hydroxyl moieties and moisture. Moreover, peaks in the regions 1,203-1,273 cm^{-1} and 1,392-1,585 cm^{-1} were correlated to C-O stretching vibration and aromatic C=C mode, respectively, thus demonstrating the existence of lignocellulose-containing functional groups.

For the FTIR spectra of KOH-activated carbon, a reduction in the peak intensity for the O-H stretching was observed, implying a partial removal of oxygen-containing functional groups because of KOH activation. The existence of the peak at 1,585 cm^{-1} implies the existence of stable aromatic structures, whereas peaks at 798 cm^{-1} and 1,072 cm^{-1} indicate some structural changes in the carbon matrix.

FTIR analysis for ultrasonic-assisted NFAC showed the presence and development of broad peaks of O-H stretching vibrations (3,244-3,550 cm^{-1}) together with C-H stretching vibrations (2,850-2,950 cm^{-1}). Peaks ranging from 1,171 to 1,269 cm^{-1} represent C-O stretching vibrations. Thus, there is evidence of a high saturation of surface oxygen moieties, which help to adsorb metal ions. Similar results have been achieved by scientists in studies of ultrasound-assisted activation (Eslami *et al.*, 2020).

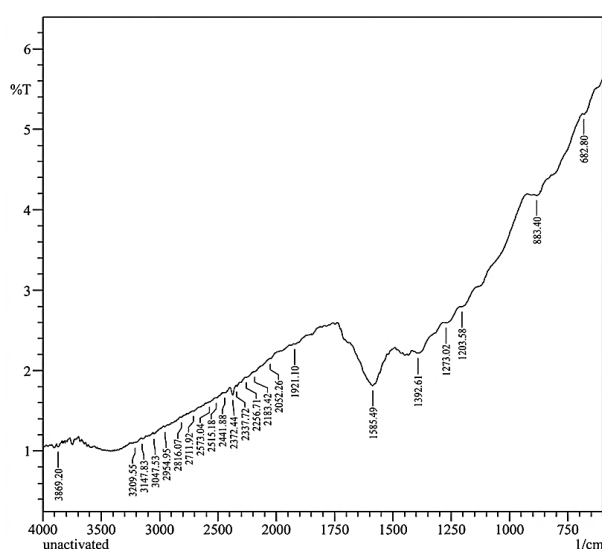


Figure 3 FTIR spectra of *Nypa fruticans* raw carbon

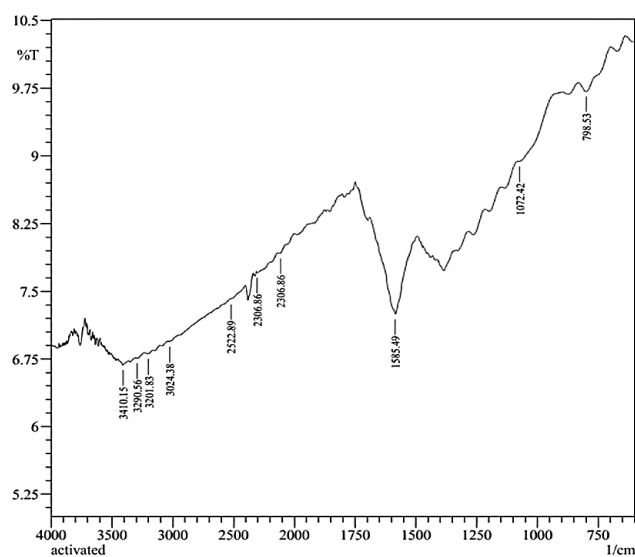


Figure 4 FTIR spectra of KOH-activated carbon

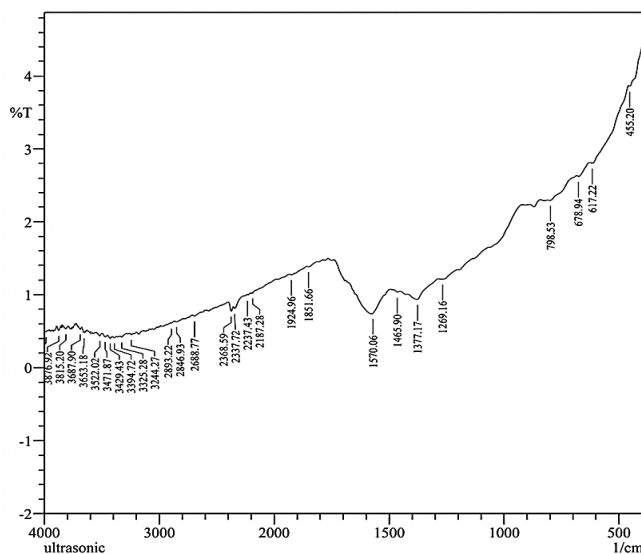


Figure 5 FTIR spectra of ultrasonically assisted NFAC

3.1.3 Surface Area

The BET analysis presented in Table 2 indicates that ultrasonic-assisted activation significantly expanded the surface area of NFAC. Initially, the surface boundary of the raw material was quite low, measuring only 0.17 m²/g. However, activation without ultrasound raised the surface area to 555.61 m²/g. The prime conditions for ultrasonic activation were proved to be 20 kHz, 120 W,

and 15 minutes, resulting in a maximum surface area of 1328.74 m²/g. This exceptionally high surface area sample was selected for further adsorption tests.

The expansion of the adsorbent surface was due to the process of cavitation since cavitation led to improved puncture and piercing of the activation agents on the carbon surface and helped in creating uniform honeycombed-like pores. Previous studies have found similar findings for ultrasonic activation (Bora *et al.*, 2021; Rahayu *et al.*, 2020).

However, increasing the sonication exposure cycle and ultrasonic power led to a reduction in the surface expansion due to the crushing of pores from high-magnitude bubble collapse energy. This trend was specifically seen in the case of 180 W power, with a reduction in surface expansion of 562.71 m²/g to 630.01 m²/g. Additionally, the temperature of the ultrasonic bath increased depending on the duration and power of the treatment, reaching as high as 87 °C under prolonged treatment conditions. This temperature change may have contributed to cavitation effects and reduced the surface area with increased treatment duration.

Table 2 BET surface area of NFAC

Sample	Frequency (kHz)	Time (minutes)	Power (W)	BET Surface Area (m ² /g)
Raw	-	-	-	0.17
Control	-	-	-	555.61
1	20	15	120	1328.74
2	20	30	120	625.22
3	20	45	120	591.20
4	20	60	120	578.17
5	20	15	180	630.01
6	20	30	180	620.42
7	20	45	180	588.43
8	20	60	180	562.71

3.2 RSM Analysis of Pb (II) Removal

The Response Surface Methodology (RSM) technique was employed to study the effects of initial Pb(II) concentration, contact time, and adsorbent amount on adsorption efficiency using ultrasonic-assisted NFAC. A total of 51 experiments were conducted based on the design matrix, and the amount of Pb(II) ions bound to active binding pores was measured using Atomic Absorption Spectroscopy (AAS). The precision of the established model was verified through

ANOVA with the results provided in Table 3.

The relation between the operating variables and the response was modelled by a full second-order polynomial model. The mathematical model for the removal efficiency of Pb(II) in terms of un-coded variables is shown in Eq. (5).

$$\begin{aligned}
 & \text{Pb(II) removal} \\
 &= 7.97 + 1.714 \text{ Pb(II) concentration} - 0.0229 \text{ Time} \\
 &+ 16.6 \text{ Dosage} - 0.0198 \text{ Pb(II) concentration} \\
 &\quad * \text{ Pb(II) concentration} + 0.0001 \text{ Time} * \text{ Time} \\
 &- 21.3 \text{ Dosage} * \text{ Dosage} \\
 &- 0.0003 \text{ Pb(II) concentration} * \text{ Time} \\
 &- 0.909 \text{ Pb(II) concentration} * \text{ Dosage} \\
 &- 0.0346 \text{ Time} * \text{ Dosage}
 \end{aligned} \tag{5}$$

The model was statistically robust ($F=17.18$; $p = 0.01$), which means that selected parameters are responsible for the process of Pb(II) ions binding on the surface of NFAC. As far as linear parameters were concerned, as indicated by the positive linear regression coefficient (+16.6), adsorbent dosage was shown to have the most significant control on Pb(II) sorption ($F = 51.66$), followed by initial concentration ($F=34.83$) and contact time ($F=3.07$). It can be concluded that adsorbent dosage plays a vital role in the process of ion binding efficiency.

Furthermore, the quadratic coefficient (Pb^2) was also significant ($F=36.46$; $p=0.01$), and it could be associated with non-proportionality caused by site exhaustion of existing pores with metal ions at increased concentrations. At the same time, quadratic coefficients for contact time (t^2) and dosage (C^2) were not significant.

The relation between adsorbent dose and initial concentration of Pb was statistically significant, owing to the small value of p ($p=0.01$). This shows that NFAC dosage is highly dependent on the driving force of the initial Pb concentration gradient. However, for interactions where contact time was involved, there was statistical insignificance, implying that contact time works independently to attain kinetic equilibrium.

Regarding the R^2 coefficients, the values obtained ($R^2=79.04\%$; adjusted $R^2=74.44\%$; predicted $R^2=69.94\%$) indicate decent reliability. In Response Surface Methodology (RSM), a difference of less than 20% between the values of adjusted R^2 and predicted R^2 is considered to

be good agreement, which means that there is no problem of overfitting of the developed model and it has good predictive ability. The difference between the values of adjusted R^2 and predicted R^2 is 4.50%, which demonstrates excellent validity and good predictive ability of the developed model.

This finding also correlates well with the results obtained by other studies on environmental adsorption processes. For example, in the work conducted by Delnavaz and Kazemimofrad (2020), the values of adjusted R^2 and predicted R^2 were 89.60% and 72.30%, respectively, and the difference between the two was 17.30% in the case of dye adsorption using nano-zero-valent iron. Similarly, Abdmunaf Atta *et al.* (2022) and Norouzi and Nezamzadeh-Ejhieh (2023) recorded differences of 3.05% and 5.27%, respectively, in the case of adsorption using adsorbents made up of rice husk and binary photocatalysts. Thus, despite the natural variability that exists in biomass-based adsorbents, the present finding supports the fact that the developed RSM model has a decent fit and predictive ability.

Table 3 ANOVA of Pb(II) adsorption

Source	DF	Adjusted SS	Adjusted MS	F-Value	P-Value
Model	9	2,569.28	285.476	17.18	0.01
Linear	3	1,506.35	502.115	30.21	0.01
conc	1	578.88	578.884	34.83	0.01
time	1	51.01	51.014	3.07	0.08
dose	1	858.55	858.554	51.66	0.01
Square	3	612.89	204.297	12.29	0.01
conc*conc	1	605.95	605.950	36.46	0.01
time*time	1	1.64	1.642	0.10	0.75
dose*dose	1	6.91	6.908	0.42	0.52
2-Way Interaction	3	250.06	83.353	5.01	0.05
conc*time	1	3.15	3.152	0.19	0.66
conc*dose	1	241.62	241.619	14.54	0.01
time*dose	1	2.83	2.831	0.17	0.68

3.2.1 Surface Plot for Pb Removal

Tri-variate response surface plateaus (Figure 6A-E) show the combined effects of initial Pb(II) concentration, contact time, and adsorbent dosage on the percentage of Pb(II) ions binding on the site of the NFAC. The interplay between initial concentration and adsorbent dosage was

assessed at different time cycles, ranging from 30 min to 150 min.

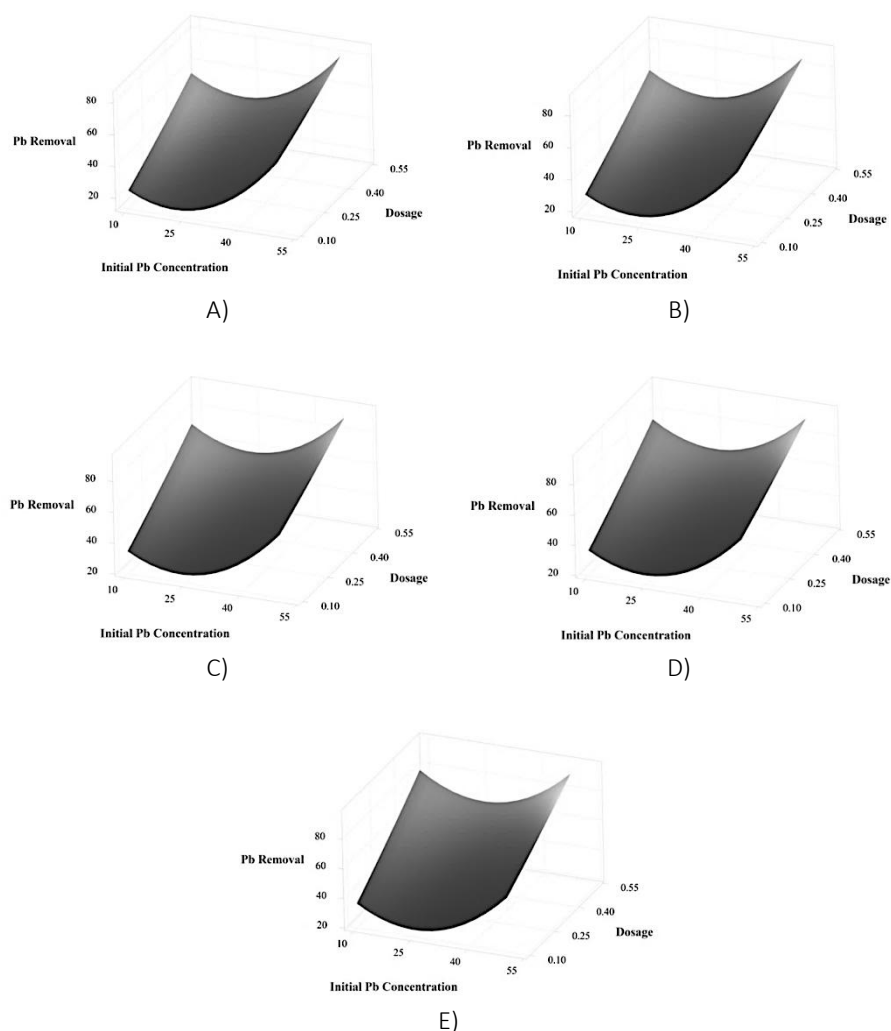


Figure 6 Response surface plots showing the effects of initial Pb(II) concentration and adsorbent dosage on Pb(II) removal efficiency at different contact times: A) 30 min, B) 60 min, C) 90 min, D) 120 min, and E) 150 min

Overall, the plateaus illustrate a similar pattern regardless of the chosen contact time since it has only a minimal bearing on the studied factors. The increase in Pb(II) ion binding takes place simultaneously with the rise in initial concentration and dosage of the NFAC. From the plateaus, the notable bearing of the adsorbent dosage correlates with the ANOVA results and shows that the number of NFAC active binding pores significantly affects the binding of ions.

The 60-minute contact time exhibited the peak response surface among the evaluated time

times, suggesting that the adsorption system was in steady state, beyond which no significant improvement in removal efficiency is observed as the binding sites are fully occupied. Pratiwi and Prinajati (2018) also indicated a similar tendency for the contact time factor. Pb removal increased with the increase in contact time, but after 60 minutes, the amount of ions bound to the active sites was negligible. For instance, at pH 4, the removal of Pb rose from 99.60% at 30 minutes to 99.78% at 60 minutes and 99.81% at 90 minutes, while at pH 6, the removal of Pb was found to rise from 98.25% to 99.63% and 99.79%. Though the experiment was carried out under varied pH levels, the same plateauing effect of removal was seen with an increase in contact time.

From a mechanistic perspective, the increase in Pb(II) sorption with contact cycles is attributed to the incremental binding of available active sites, while a higher adsorbent dosage expands the number of active binding pores available for adsorption. The graph observed after 60 minutes indicates that adsorption equilibrium has been reached under the studied conditions.

3.3 Comparative Analysis of KOH-Activated and Ultrasonic Assisted NFAC

Based on Figures 7 and 8, NFAC activated using ultrasonic technology showed a consistent increase in Pb(II) removal efficiency compared to the KOH-activated NFAC across all dosages of the adsorbents. When 0.1 g of adsorbent was used, the removal of Pb(II) using ultrasonic-assisted NFAC achieved an efficiency of 46.6%, while only 4.7% efficiency was achieved using KOH-activated NFAC. The result implies that the ultrasonic activation process significantly increased the adsorption efficiency of NFAC in situations where adsorbent dosage is lower. At lower dosage, surface modification exerts a strong influence on the binding process of Pb(II) ions.

Increasing the adsorbent dose from 0.3 to 0.5 g improved the efficiency of removing Pb(II). However, ultrasonic-assisted NFAC still showed superior performance with 74.9% removal efficiency against 70.2% achieved by KOH-activated NFAC. The performance difference became smaller as the dosage of adsorbents increased, implying that adsorption is less limited by surface modifications and more by mass transfer issues.

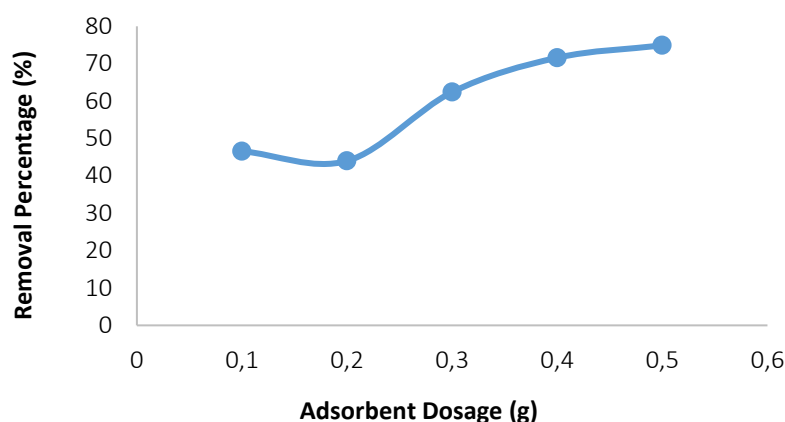


Figure 7 Effect of adsorbent dosage of ultrasonic-assisted KOH-activated NFAC

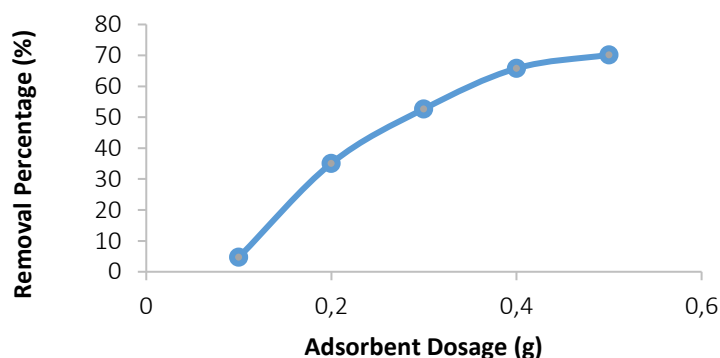


Figure 8 Effect of adsorbent dosage of KOH-activated NFAC

Equilibrium adsorption capacity (q_e) also indicates the superior performance of ultrasonic-assisted NFAC over KOH-activated NFAC under similar conditions. Improved efficiency in ultrasonic-assisted NFAC could be explained by efficient surface modification and surface site availability resulting from the ultrasonic activation process.

3.4 Adsorption Isotherm

Adsorption isotherms for Pb(II) ions on NFAC activated by the ultrasonic-assisted technique were performed using Langmuir and Freundlich adsorption isotherms. Equilibrium adsorption data acquired at varying Pb(II) ion concentrations in the range of 10-50 mg/L with a constant contact time of 60 min and an adsorbent dose of 0.1 g were fitted according to both isotherm equations

and used to plot the models. Figures 9 and 10 show the Langmuir model and Freundlich model, respectively.

Out of the two models, Langmuir exhibited a higher correlation to the experimental data ($R^2 = 0.97$) compared to Freundlich ($R^2 = 0.88$), with saturated adsorption loading, $Q_{\max}$ of 137.17 mg/g. The dimensionless separation factor, R_L of 0.75, indicates favourable adsorption of Pb(II).

The relatively high coefficient of correlation with the Langmuir model indicates monolayer adsorption on a limited number of similar active sites. Conversely, the Freundlich isotherm suggests some surface non-uniformity due to the n value equal to 1.08, which corresponds to favourable adsorption conditions. Therefore, higher correlation with the Langmuir isotherm indicates that the adsorption process forms a single layer on the surface of the NFAC.

Table 4 illustrates a comparison between the maximum monolayer adsorption capacity of the synthesized ultrasonic-assisted NFAC and those of several other activated carbons derived from biomass, as described in recent literature. The NFAC demonstrated a maximum monolayer adsorption capacity of 137.17 mg/g, which is better than several agricultural waste adsorbents such as kiwi peel (0.16 mg/g), Albizia lebbeck seeds (0.23 mg/g), Reptonia buxifolia seeds (51.00 mg/g), and sugarcane bagasse (60.24 mg/g). Additionally, NFAC showed better adsorption capacity compared to mangrove waste activated carbon (112.50 mg/g). The higher adsorption capacity of NFAC is due to the ultrasonically modified structure of NFAC during preparation, leading to an increase in the surface area, hence more sites for Pb(II) adsorption.

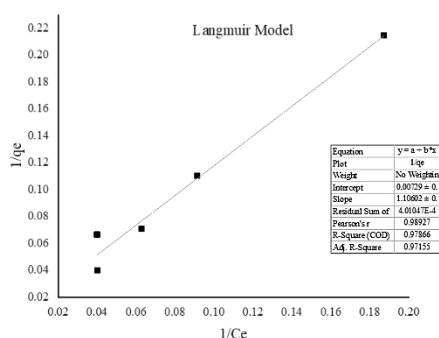


Figure 9 Langmuir model

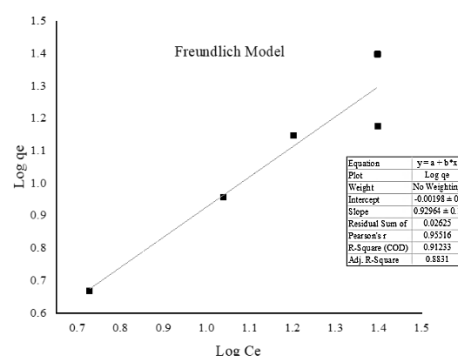


Figure 10 Freundlich model

Table 4 Adsorption capacity comparison

Organic Waste Derived Activated Carbon Used for Lead Removal	Lead Adsorption Capacity (mg/g)	References
Kiwi Peel Activated Carbon	0.16	(Rahimnejad <i>et al.</i> , 2018)
Albizia lebbeck Seed Activated Carbon and Chinaberry Seed Activated Carbon	0.23 and 0.29	(Ullah <i>et al.</i> , 2020)
Solitary Fishtail Palm Seed Activated Carbon	42.9	(Ravulapalli & Ravindhranath, 2018)
Reptonia buxifolia Seed Activated Carbon	51.0	(Bilal <i>et al.</i> , 2020)
Sugarcane Bagasse Activated Carbon	60.24	(Somyanonthanakun <i>et al.</i> , 2023)
Pistachio Wood Activated Carbon	190.2	(Sajjadi <i>et al.</i> , 2019)
Nypa fruticans Activated Carbon (NFAC)	137.17	This study

3.5 Adsorption Kinetics

Adsorption kinetics were examined using the PFO and PSO models to determine the governing mechanism that limits the binding of Pb(II) ions onto NFAC. PFO is associated with physical adsorption, while PSO is associated with chemisorption due to electron exchange or sharing between adsorbates and adsorbents (Wang *et al.*, 2022). Studies on kinetics were performed under conditions of an initial concentration of Pb(II) ions equal to 10 mg/L, a dosage of adsorbent of 0.5 g, a solution volume of 100 mL, and contact time varying from 15 to 150 minutes. Under these operating conditions, the experimental equilibrium adsorption capacity ($q_{e,exp}$) was determined to be 1.02 mg/g. As shown in Figure 11, PFO yielded an R^2 equal to 0.90, where $q_e = 4.37$ mg/g and $k_1 = 9.0 \times 10^{-6} \text{ min}^{-1}$. The PFO model significantly overestimated the actual equilibrium capacity, suggesting that the model is not adequate to describe the overall kinetic process.

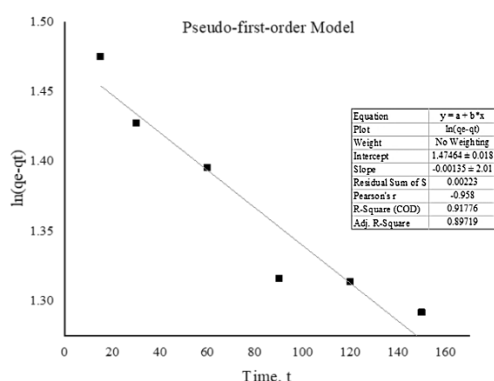


Figure 11 PFO model

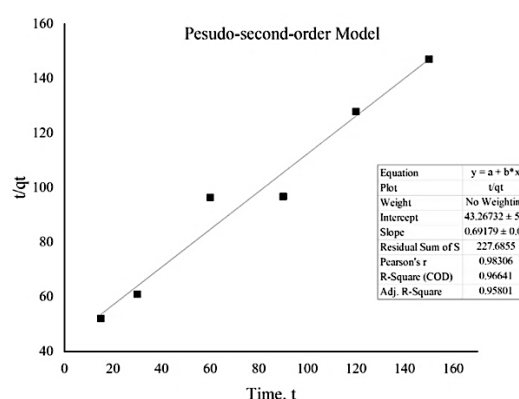


Figure 12 PSO model

On the other hand, in Figure 12, PSO showed a higher degree of correlation with experimental values ($R^2=0.96$), k_2 equal to $0.01 \text{ g mg}^{-1} \text{ min}^{-1}$ with q_e calculated at 1.45 mg/g that aligns closely with the experimental equilibrium adsorption capacity. Higher correlation indicates that the PSO model proficiently characterises the kinetic binding of Pb(II) ions on the NFAC active surface. This indicates that adsorption could possibly be affected by strong surface interactions. Previous studies have indicated that the PSO model characterises adsorption processes more accurately than other models when it comes to heavy metal adsorption on activated carbon (Igbemi *et al.*, 2021; El-Sheetta *et al.*, 2022; Taye *et al.*, 2023).

4. CONCLUSION

This research highlights the possibility of using ultrasonic-assisted KOH-activated Nypa fruticans activated carbon (NFAC) for Pb(II) removal from aqueous solutions. The ultrasonic-assisted activation process has greatly improved the physicochemical properties of NFAC. The BET specific surface area increased to $1328.74 \text{ m}^2/\text{g}$, compared to the $555.61 \text{ m}^2/\text{g}$ obtained from KOH-activated NFAC through the optimisation of sonication parameters at 20 kHz, 120 W, and 15 minutes. Based on Response Surface Methodology (RSM) analysis, adsorbent dosage was identified as the parameter that highly affected the percentage removal efficiency of Pb(II), with maximum removal achieved at 74.9%.

The results of the isotherm analysis proved that the Langmuir isotherm model ($R^2 = 0.97$) is appropriate for describing the adsorption reaction, indicating that adsorption occurs mainly through monolayer formation with a saturated adsorption loading of 137.17 mg/g . Meanwhile, rate of reaction studies showed that the PSO kinetics equation was more suitable ($R^2 = 0.96$) compared to PFO kinetics because adsorption was governed by strong surface interactions. The results show that NFAC produced via ultrasound activation is a feasible and promising adsorbent for binding Pb(II) ions from aqueous solution. However, this study is limited by its reliance on synthetic single-metal solutions, a lack application to real wastewater, and the absence of thermodynamic and regeneration studies. To address these gaps, future studies should further explore multi-meter system, adsorbent's reusability and application in real surface water.

5. ACKNOWLEDGMENT

The authors would like to acknowledge Universiti Malaysia Sarawak (UNIMAS) for the support

provided for this publication.

REFERENCES

- Abdmunaf Atta, H., Hummadi, K. K., & M-Ridha, M. J. 2022. The application of response surface methodology and Design-Expert® for analysis of ciprofloxacin removal from aqueous solution using raw rice husk: kinetic and isotherm studies. *Desalination and Water Treatment*. 248: 203-216. <https://doi.org/10.5004/dwt.2022.28074>.
- Ahmed, M. F., Mokhtar, M. B., Alam, L., Mohamed, C. A. R., & Ta, G. C. 2020. Investigating the status of cadmium, chromium and lead in the drinking water supply chain to ensure drinking water quality in Malaysia. *Water (Switzerland)*. 12(10): 1-26. <https://doi.org/10.3390/w12102653>.
- Al-Ghouti, M. A., & Da'ana, D. A. 2020. Guidelines for the use and interpretation of adsorption isotherm models: A review. *Journal of Hazardous Materials*. 393: 122383. <https://doi.org/10.1016/j.jhazmat.2020.122383>.
- Bilal, M., Ali, J., Hussain, N., Umar, M., Shujah, S., & Ahmad, D. (2020). Removal of Pb(II) from wastewater using activated carbon prepared from the seeds of *Reptonia buxifolia*. *Journal of the Serbian Chemical Society*. 85(2): 265-277. <https://doi.org/10.2298/JSC181108001B>.
- Borghei, S. A., Zare, M. H., Ahmadi, M., Sadeghi, M. H., Marjani, A., Shirazian, S., & Ghadiri, M. 2021. Synthesis of multi-application activated carbon from oak seeds by KOH activation for methylene blue adsorption and electrochemical supercapacitor electrode. *Arabian Journal of Chemistry*. 14(2): 102958. <https://doi.org/10.1016/j.arabjc.2020.102958>.
- Bora, M., Benoy, S. M., Tamuly, J., & Saikia, B. K. 2021. Ultrasonic-assisted chemical synthesis of activated carbon from low-quality subbituminous coal and its preliminary evaluation towards supercapacitor applications. *Journal of Environmental Chemical Engineering*. 9(1). <https://doi.org/10.1016/j.jece.2020.104986>.
- Delnavaz, M., & Kazemimofrad, Z. (2020). Nano zerovalent iron (NZVI) adsorption performance on acidic dye 36 removal: Optimization of effective factors, isotherm and kinetic study. *Environmental Progress and Sustainable Energy*. 39(4). <https://doi.org/10.1002/ep.13349>.
- El-Sheeti, M. A., Goher, M. E., Abd El-Moghny, M. G., & El-Deab, M. S. 2022. Efficient elimination of Pb(II) ions from aqueous solutions using magnetic Fe₃O₄-nanoparticles/activated carbon derived from agricultural waste. *Desalination and Water Treatment*. 258: 241-260. <https://doi.org/10.5004/DWT.2022.28365>.
- Eslami, H., Khosravi, R., Miri, M. R., Gholami, A., Ghahramani, E., & Khosravi, A. 2020. Facile preparation of porous activated carbon under ultrasonic assistance for the methylene blue removal from aqueous environment: Characterization, isothermal, kinetic and thermodynamic studies. *Materials Research Express*. 7(1). <https://doi.org/10.1088/2053-1591/ab69d1>.

- Ezekiel, O., & Dikam, K. I. 2020. Assessment of Concentration Status of Some Heavy Metals in Water along River Dilimi, Jos North, Plateau State-Nigeria. *Indonesian Journal of Urban and Environmental Technology*. 4(1): 28-44. <https://doi.org/10.25105/urbanenvirotech.v4i1.6768>.
- Gupta, A., Hussain, N., & Pandey, B. 2019. A critical study about the toxicity of lead and the precautionary measures: A review. *Indian Journal of Scientific Research*. 10(1): 205. <https://doi.org/10.32606/IJSR.V10.I1.00032>.
- Igbemi, I. A., Nwaogazie, I. L., Akaranta, O., & Abu, G. O. 2021. Adsorption dynamics of agricultural waste activated carbon in water quality improvement. *European Journal of Engineering and Technology Research*. 6(7): 11-19. <https://doi.org/10.24018/ejeng.2021.6.7.2526>.
- Kamal, M. A., Bibi, S., Bokhari, S. W., Siddique, A. H., & Yasin, T. 2017. Synthesis and adsorptive characteristics of novel chitosan/graphene oxide nanocomposite for dye uptake. *Reactive and Functional Polymers*. 110: 21-29. <https://doi.org/10.1016/j.reactfunctpolym.2016.11.002>.
- Kecili, R., & Hussain, C. M. 2018. Mechanism of adsorption on nanomaterials. In *Nanomaterials in Chromatography: Current Trends in Chromatographic Research Technology and Techniques*. Elsevier. pp. 89-115. <https://doi.org/10.1016/B978-0-12-812792-6.00004-2>.
- Kumar, A., Kumar, V., Thakur, M., Singh, K., Jasrotia, R., Kumar, R., & Radziemska, M. 2025. Global perspectives on lead contamination and health risks in surface water, rice grains, and soils. *Land Degradation and Development*. John Wiley and Sons Ltd. <https://doi.org/10.1002/ldr.5510>.
- Kumar, V., Parihar, R. D., Sharma, A., Bakshi, P., Singh Sidhu, G. P., Bali, A. S., Karaouzas, I., Bhardwaj, R., Thukral, A. K., Gyasi-Agyei, Y., & Rodrigo-Comino, J. 2019. Global evaluation of heavy metal content in surface water bodies: A meta-analysis using heavy metal pollution indices and multivariate statistical analyses. *Chemosphere*. 236: 124364. <https://doi.org/10.1016/j.chemosphere.2019.124364>.
- Liu, L., Luo, X.-B., Ding, L., & Luo, S.-L. 2019. Application of nanotechnology in the removal of heavy metal from water. In *Nanomaterials for the Removal of Pollutants and Resource Reutilization*. Elsevier. pp. 83-147. <https://doi.org/10.1016/B978-0-12-814837-2.00004-4>.
- Nasseh, N., Khosravi, R., Rumman, G. A., Ghadirian, M., Eslami, H., Khoshnamvand, M., Al-Musawi, T. J., & Khosravi, A. 2021. Adsorption of Cr(VI) ions onto powdered activated carbon synthesized from Peganum harmala seeds by ultrasonic waves activation. *Environmental Technology and Innovation*. 21. <https://doi.org/10.1016/j.eti.2020.101277>.
- Norouzi, A., & Nezamzadeh-Ejhieh, A. 2023. Investigation of the simultaneous interactions of experimental variables and mechanism pathway in the photodegradation of methylene blue by binary ZnO/Cu₂O photocatalyst. *Materials Research Bulletin*. 164. <https://doi.org/10.1016/j.materresbull.2023.112237>.

- Pratiwi, R., & Prinajati, P. D. 2018. Adsorption for lead removal by chitosan from shrimp shells. *Indonesian Journal of Urban and Environmental Technology*. 2(1): 35-46. <https://doi.org/10.25105/urbanenvirotech.v2i1.3554>.
- Rahayu, W., Muthomimah, S., Sumari, S., & Nazriati, N. 2020. Effect of sonication time on characteristics of synthesized silica aerogel activated carbon nanocomposite based on bagasse ash. *IOP Conference Series: Materials Science and Engineering*. 833(1): 012088. <https://doi.org/10.1088/1757-899X/833/1/012088>.
- Rahimnejad, M., Pirzadeh, K., Mahdavi, I., & Peyghambarzadeh, S. M. 2018. Pb (..) Removal from Aqueous Solution by Adsorption on Activated Carbon Ffrom Kiwi Peel. *Environmental Engineering and Management Journal*. 17(6): 1293-1300. <https://doi.org/10.30638/eemj.2018.128>.
- Ravulapalli, S., & Kunta, R. 2018. Removal of lead (II) from wastewater using active carbon of Caryota urens seeds and its embedded calcium alginate beads as adsorbents. *Journal of Environmental Chemical Engineering*. 6(4): 4298-4309. <https://doi.org/10.1016/j.jece.2018.06.033>.
- Sajjadi, S.-A., Meknati, A., Lima, E. C., Dotto, G. L., Mendoza-Castillo, D. I., Anastopoulos, I., Alakhras, F., Unuabonah, E. I., Singh, P., & Hosseini-Bandegharaei, A. 2019. A novel route for preparation of chemically activated carbon from pistachio wood for highly efficient Pb(II) sorption. *Journal of Environmental Management*. 236: 34-44. <https://doi.org/10.1016/j.jenvman.2019.01.087>.
- Somyanonthanakun, W., Greszta, A., Roberts, A. J., & Thongmee, S. 2023. Sugarcane Bagasse-Derived Activated Carbon as a Potential Material for Lead Ions Removal from Aqueous Solution and Supercapacitor Energy Storage Application. *Sustainability (Switzerland)*. 15(6). <https://doi.org/10.3390/su15065566>.
- Syauqiah, I., Elma, M., Mailani, D. P., & Pratiwi, N. 2020. Activated carbon from Nypa (Nypa fruticans) leaves applied for the Fe and Mn removal. *IOP Conference Series: Materials Science and Engineering*. 980(1): 012073. <https://doi.org/10.1088/1757-899X/980/1/012073>.
- Taslim, Iriany, Bani, O., Audina, E., & Hidayat, R. 2021. Preparation of activated carbon-based catalyst from nipa palm (Nypa fruticans) shell modified with KOH for biodiesel synthesis. *IOP Conference Series: Earth and Environmental Science*. 912(1): 012094. <https://doi.org/10.1088/1755-1315/912/1/012094>.
- Taye, A., Mehretie, S., & Admassie, S. 2023. Adsorption of Lead(II) ions using KOH-activated carbon derived from water hyacinth. *Bulletin of the Chemical Society of Ethiopia*. 37(6): 1369-1382. <https://doi.org/10.4314/bcse.v37i6.6>.
- Ullah, M., Nazir, R., Khan, M., Khan, W., Shah, M., Afridi, S. G., & Zada, A. 2020. The effective removal of heavy metals from water by activated carbon adsorbents of Albizia Lebbeck and

Melia azedarach seed shells. *Soil and Water Research*. 15(1): 30-37.
<https://doi.org/10.17221/212/2018-SWR>.

Wang, T., Jiang, M., Yu, X., Niu, N., & Chen, L. 2022. Application of lignin adsorbent in wastewater treatment: A review. *Separation and Purification Technology*. Elsevier B.V.
<https://doi.org/10.1016/j.seppur.2022.122116>.

World Health Organization. 2022. Lead in drinking-water: Health risks, monitoring and corrective actions. World Health Organization.
<https://www.who.int/publications/i/item/9789240020863>.