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RESEARCH ARTICLE

Utilization of Response Surface Methodology to Optimize Ammoniacal Nitrogen Adsorption Efficiency from Palm Oil Mill Secondary Effluent Using Palm Empty Fruit Bunch as Adsorbent

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Abstract The disposal of ammonia-laden wastewater from the palm oil industry presents a significant environmental challenge leading to water quality deterioration and adverse effects on aquatic ecosystems. The conventional methods for ammonia removal are often costly and complex necessitating the exploration of alternative solutions. This study investigates the use of palm empty fruit bunch (EFB) biochar as a sustainable adsorbent for the removal of ammonia from palm oil mill secondary effluent (POMSE). Employing Response Surface Methodology (RSM) with a Central Composite Design (CCD), the study has optimized key parameters including adsorbent dosage, initial ammonia concentration and carbonization temperature. The results demonstrated that palm EFB biochar effectively reduced ammonia levels, achieving removal efficiencies of approximately 30% at a dosage of 2 g/L and up to 43% at 4 g/L under optimal conditions (initial ammonia concentration of 292.5 mg/L and carbonization temperature of 500°C). The adsorption process conformed to the Langmuir isotherm model, indicating monolayer adsorption on a homogeneous surface. Characterization techniques such as Scanning Electron Microscopy (SEM) and Fourier Transform Infrared (FTIR) spectroscopy confirmed the structural integrity and functional interactions of the biochar with ammonia molecules. Additionally, the study explored the regeneration capabilities of the palm EFB biochar, highlighting its potential for repeated use in wastewater treatment applications. This research underscores the viability of utilizing agricultural waste as an effective and sustainable solution for ammonia removal, contributing to environmental sustainability and resource recovery in the palm oil industry.

Keywords: Response Surface Methodology, Central Composite Design, Langmuir isotherm, palm EFB, palm oil mill secondary effluent.

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Introduction

Ammonia pollution in industrial wastewater is a critical environmental issue particularly in regions where agricultural and industrial activities dominate [1], [2]. Ammonia, predominantly found in ammoniacal nitrogen (NH_3-N) form is discharged into water systems from various industries including agriculture, food processing and chemical manufacturing [3]. Ammoniacal nitrogen exists in two primary forms which are un-ionized ammonia (NH_3) and ammonium ion (NH_4^+). The equilibrium between these two species is influenced by pH and temperature. At lower pH values that is below 7, ammonium ion (NH_4^+) predominates, remaining dissolved in water and posing minimal toxicity. However, at higher pH levels, the equilibrium shifts towards un-ionized ammonia (NH_3) which is volatile and highly toxic to aquatic life. This equilibrium is governed by the reaction in Equation 1.

$$NH_3 + H_2O \rightleftharpoons NH^{4+} + OH^- \quad \text{(Equation 1)}$$

The dissociation constant (pKa) for this reaction is approximately 9.25 at 25°C. This means that at pH 9.25, ammonia and ammonium exist in equal concentrations. Any increase in pH beyond this threshold leads to a higher proportion of un-ionized ammonia thus increasing the risk of ammonia volatilization and toxicity thus poses severe threats to aquatic ecosystems, public health and water quality [3]. For instance, ammonia contributes significantly to eutrophication where excessive nutrients in water bodies stimulate algal blooms [4]. These blooms result in a decline in water quality, increased water cloudiness and unpleasant odors as well as a significant drop in oxygen levels when the algae decompose, thereby harming aquatic life and reducing biodiversity [5].

Due to its adverse environmental effects, various treatment methods are employed to remove ammoniacal nitrogen from wastewater. One of the most effective biological methods is the nitrification-denitrification process. In this process, ammonia is first oxidized to nitrite (NO_2^-) by *Nitrosomonas* bacteria, followed by oxidation to nitrate (NO_3^-) by *Nitrobacter* bacteria [6].

$$NH^{4+} + O_2 \xrightarrow{\text{Nitrosomonas}} NO^{2-} + H_2O + H^+ \quad \text{(Equation 2)}$$

$$NO^{2-} + O_2 \xrightarrow{\text{Nitrobacter}} NO^{3-} \quad \text{(Equation 3)}$$

Subsequently, denitrification occurs under anoxic conditions where nitrate is reduced to nitrogen gas (N_2) by denitrifying bacteria which is then released into the atmosphere [6].

$$NO^{3-} + \text{Organic Carbon} \xrightarrow{\text{Denitrifiers}} N_2 + CO_2 + H_2O \quad \text{(Equation 4)}$$

Chemical methods include struvite precipitation and air stripping. Struvite precipitation involves the reaction of ammonium with magnesium and phosphate ions, results in the formation of magnesium ammonium phosphate ($MgNH_4PO_4 \bullet 6H_2O$) which can be removed from wastewater while air stripping converts NH_4^+ to NH_3 gas by raising the pH above 11, allowing volatilization.

$$NH^{4+} + Mg^{2+} + PO_4^{3-} \rightarrow MgNH_4PO_4 \bullet 6H_2O \quad \text{(Equation 5)}$$

Adsorption is another effective method, involving the attachment of ammonia onto solid surfaces. Adsorbents like activated carbon and chemically modified biochar interact with ammonia via functional groups such as hydroxyl and carboxyl.

Among emerging techniques, adsorption using natural and low-cost materials has attracted considerable attention for its simplicity, efficiency, and sustainability. Natural adsorbents, especially those derived from biomass, offer eco-friendly and economical alternatives to synthetic materials. Adsorption occurs through mechanisms like electrostatic attraction, ion exchange, and hydrogen bonding, and its effectiveness depends on surface area, porosity, and chemical functionality.

Palm empty fruit bunch (EFB), a by-product of the palm oil industry, has emerged as a particularly promising natural adsorbent. For every tonne of palm oil produced, approximately 1.5 tonnes of palm EFB are generated [7], [8]. This material, rich in cellulose, hemicellulose, and lignin, contains functional groups and a fibrous, porous structure that enhance its adsorption capabilities. Activation and chemical treatments such as with NaOH or H_3PO4 improve surface area, porosity and functional group availability, increasing its capacity to bind ammonium ions through ion exchange and other mechanisms. Palm EFB has shown impressive performance in previous studies. For example, up to 79.5% removal of ammonia nitrogen was achieved from natural rubber wastewater [9], and 66% recovery was reported from aquaculture effluent using palm EFB biochar [10]. These results are attributed to both the physical properties and chemical modifications that enhance the adsorptive performance of EFB.

Conventionally, palm EFB is either left to decompose in open piles or used as compost in agricultural fields [11], [12]. While composting can be useful, large-scale disposal of palm EFB still presents a significant waste management challenge. Furthermore, conventional disposal methods do not harness the full potential of palm EFB, especially considering the unique chemical and physical properties of the material that could make it highly effective in addressing some of the most pressing environmental issues such as water pollution and air quality [11], [12].

Despite the promising potential of palm EFB as an adsorbent, several challenges must be addressed before it can be widely implemented. One key issue is the inconsistency in material quality, as the properties of palm EFB can vary depending on its source and how it is processed. To address this, further research is needed to standardize preparation methods and enhance adsorption capacity through chemical treatments or activation. Another challenge is scaling up. While lab-scale studies show good results, more work is needed to evaluate large-scale production and how it can be effectively integrated into existing wastewater treatment systems.

To optimize the adsorption performance of palm EFB, several studies have employed thermal and chemical modifications. Carbonization, a thermal treatment process conducted at temperatures between 300°C and 700°C, significantly alters the microstructure of palm EFB. Higher carbonization temperatures lead to greater porosity and surface area, which improve adsorption efficiency. Response Surface Methodology (RSM) has been widely used to identify the optimal conditions for ammonia removal using palm EFB. Comparative studies have shown that palm EFB outperforms other agricultural waste-based adsorbents in ammonia removal. For example, while rice husk biochar is effective, its adsorption capacity is often lower than that of activated palm EFB due to differences in porosity and surface chemistry. Similarly, coconut husk-based adsorbents, though promising, may face limitations in availability and cost compared to palm EFB. These comparisons underscore the advantages of palm EFB as a practical and efficient solution for ammonia pollution in industrial wastewater.

This study explores the treatment of palm EFB using alkaline and acidic solutions to optimize its ammonia adsorption capacity. Key variables, including palm EFB dosage, initial ammonia concentration in palm oil mill secondary effluent (POMSE), and carbonization temperature, were examined, with optimization achieved through response surface methodology (RSM). Additionally, the regeneration capabilities of the treated palm EFB were investigated. These factors were analyzed to identify the optimal conditions for effective ammonia removal. Utilizing palm EFB as an alternative adsorbent presents a sustainable and cost-effective solution, particularly for ammonia removal in anaerobic digestion systems. This innovative approach not only enhances wastewater treatment processes but also minimizes the environmental impact of both wastewater management and the palm oil industry. Furthermore, by repurposing agricultural waste like palm EFB as effective adsorbents, this study aligns with the principles of a circular economy supporting both environmental sustainability and economic resilience.

Materials and Methods

The palm empty fruit bunch (EFB) and palm oil mill secondary effluent (POMSE) were retrieved taken from Johor, Malaysia. Chemical composition of palm EFB is shown in Table 1.

Table 1. Chemical elements in palm empty fruit bunch (EFB)

Element	Weight % of Palm Empty Fruit Bunch (EFB)
Carbon	73.87
Potassium	6.65
Oxygen	17.28
Sodium	1.42
Calcium	0.78

While the chemical oxygen demand (COD), biological oxygen demand (BOD), ammonia content and heavy metal (if applicable) in POMSE as per shown in Table 2.

Table 2. Characteristics of POMSE

Parameters	Value	SD[a]	Effluent (Standard B)[b]
pH	8.2	0.9	5.5-9.0
COD (mg/L)	12500	1500	120
BOD (mg/L)	2327.48	1481.27	50
NH_3-N (mg/L)	423	33	20

The palm EFB was collected after an initial adsorption experiment where it effectively removed ammonia from palm oil mill secondary effluent (POMSE). The initial ammonia concentration in the POMSE was set at 195 mg/L, 292.5 mg/L, and 390 mg/L while the dosage of palm EFB used for adsorption was 2 g/L, 3 g/L and 4 g/L. Sodium hydroxide, NaOH (Merk, Germany) was used for alkalinization and as the regeneration solution.

Palm Empty Fruit Bunch (EFB) Treatment

The treatment of palm empty fruit bunch (EFB) involved careful preparation to optimize the adsorption capabilities. Palm EFB samples were sieved and ground into a fine powder. Producing finely ground particles was essential for maximizing the surface area of the samples thereby enhancing their adsorption efficiency and minimizing experimental and data variability. Fine particles also allow for a clearer analysis of the palm EFB morphology, ensuring that the palm EFB are sufficiently ground to achieve a powder-like consistency.

Following the grinding process, the powdered palm EFB samples underwent carbonization in a carbolite furnace at temperatures ranging from 300°C to 700°C, in intervals of 200°C. After the carbonization process, samples were stored in sealed bags in preparation for further treatment involving soaking in sodium hydroxide (NaOH). The NaOH solution is gradually released into an Erlenmeyer flask containing distilled water to achieve the desired 0.1M concentration. The use of 0.1M NaOH for alkaline treatment was selected based on previous studies indicating optimal disruption of lignocellulosic bonds and enhancement of active adsorption sites at this concentration without compromising biomass structure [13]. This setup ensures precision in preparing the alkaline treatment solution which crucial for maintaining consistency in the experimental process.

For the soaking process, 80 mL of the diluted 0.1 M NaOH solution was used in a 100 mL Erlenmeyer flask, with stirring maintained at 250 rpm for 8 hours using a magnetic stirrer. After the soaking process, the treated palm EFB samples were filtered through a 0.45 µm membrane and rinsed with distilled water to remove any remaining chemical residues. The samples were then dried in an oven at 60°C for 24 hours to eliminate excess moisture. Once dried, the samples were stored in sealed bags, ready for further characterization.

Adsorption Performance of Palm Empty Fruit Bunch (EFB) for Ammonia Removal in Palm Oil Mill Secondary Effluent (POMSE)

The treated palm empty fruit bunch (EFB) underwent adsorption study to evaluate its effectiveness in ammonia removal from palm oil mill secondary effluent (POMSE). The adsorption experiments focused on three primary parameters: the dosage of treated palm EFB (mg/L), the initial ammonia concentration in POMSE (mg/L), and the carbonization temperature of the palm EFB (°C). A series of 20 adsorption trials were conducted simultaneously following experimental conditions suggested by Design Expert-11 as presented in Table 3.

During the adsorption trials, samples were soaked in POMSE and subjected to shaking at 150 rpm for one hour using an orbital shaker. After the shaking process, the ammonia concentration in both POMSE and the synthetic ammonia solution was measured using a UV-Vis Spectrometer (HACH DR6000) with Nessler Reagent, according to HACH Method 8038. This approach allowed for precise quantification of the ammonia concentration before and after the adsorption process providing a basis for assessing the adsorption capacity of the treated palm EFB.

The adsorption capacity (Q_t) of the palm EFB was determined by analyzing the kinetic isotherms and reaction rates, which helped elucidate the relationship between the dosage of palm EFB and the amount of ammonia adsorbed. The value of Q_t was calculated using **Equation 6**, where C_o and C_e (mg/L) represent the initial and final ammonia concentrations respectively, *V* denotes the volume of POMSE or synthetic ammonia solution used and *M* refers to the mass of palm EFB. This critical analysis of the adsorption capacity aimed to provide insights into the effectiveness of treated palm EFB as a sustainable and efficient adsorbent for ammonia removal from wastewater.

$$Q_t = \frac{(C_o - C_t)\ x\ V}{M} \quad \text{(Equation 6)}$$

Optimization Study of Adsorption of Palm EFB for Ammonia Adsorption

In this study, the CCD matrix was generated using Design Expert-11 software based on the variables listed in Table 3, which defined the levels of the three key factors—adsorbent dosage (2, 3, 4 g/L), initial ammonia concentration (195, 292.5, 390 mg/L), and carbonization temperature (300, 500, 700 °C). The design included 8 factorial (cube) points representing all combinations of the factors at two levels (-1 and +1), 6 axial (star) points located at ±α levels to allow estimation of quadratic effects, 6 center points to provide an estimate of experimental error and to check for the presence of curvature in the response surface. The axial distance (α) was set to 1.682, ensuring the rotatability of the design, which allows for uniform precision in predictions at points equidistant from the design center. This resulted in a total of 14 experimental runs.

The runs were randomized to prevent biases and uncontrolled variability. The inclusion of replicated center points enabled validation of the predictive accuracy and assessment of experimental error. Validation through residual analysis and normal probability plots confirmed a good fit to the quadratic model. The goodness of fit of the model was assessed via the R^2 value which indicated how well the polynomial model described the experimental data.

Table 3 shows the results for the three relevant independent variables being used in the CCD: adsorbent dosage (in mg/L), initial ammonia concentration in POMSE (in mg/L) and carbonization temperature of palm EFB (°C). A total of 20 runs were done during the design as the equation of $20=2^k+2k+6$, which k indicates the number of factors (k=3) and 14 runs are enhanced while six replications were made to find the pure error in the experiments. Afterward, R^2 value was produced as an indicator for predicted and experimental percentage adsorption, thus it represented the quality of the fit of the polynomial model.

Table 3. Variables of Central Composite Design (CCD)

Level of Value	Amount of dosage (g/L)	Initial ammonia concentration (mg/L)	Carbonization temperature (°C)
-1	2	195	300
0	3	292.5	500
+1	4	390	700

Adsorption Isotherms

In this study, two equilibrium isotherms were used (Langmuir and Freundlich) to find the characteristics of ammonia adsorption on the adsorbents (palm EFB). For Langmuir isotherm of EFB, a linear form of Langmuir was used using Equation 7.

$$\frac{1}{qe} = \frac{1}{QbCe} + \frac{1}{Q} \quad \text{(Equation 7)}$$

which Ce is the equilibrium concentration of adsorbate (mg/L) and qe (x/m) is the amount of adsorbate in unit mass of adsorbent (mg/g). Then, Q (mg/g) and b (L/mg) are the Langmuir constants of adsorption capacity and its rate of adsorption. A straight line is got when 1/qe as the y-axis and 1/Ce is the x-axis, while the Q is the gradient of the line and b as the y-intercept. While for Freundlich Isotherm, Equation 8 was used.

$$\text{Log qe} = \log \text{K} + \frac{1}{n}\log \text{Ce} \quad \text{(Equation 8)}$$

where Ce is the equilibrium concentration of adsorbate (mg/L), qe is the amount of adsorbate in unit mass of adsorbent (mg/g), while K & n are the Freundlich constants.

Regeneration of Used Palm Empty Fruit Bunch (EFB) Adsorbent

After ammonia adsorption, the regeneration procedure on the used adsorbent was carried out using 0.1M hydrochloric acid (HCl). First, 0.1M HCl solution was prepared by diluting concentrated HCl in distilled water to serve as the desorbing agent for removing the adsorbed ammonia from the palm EFB surface. Regeneration with 0.1M HCl was employed to effectively desorb ammonia ions while maintaining adsorbent stability and reusability, consistent with prior reports favoring mild acid concentrations for biochar regeneration [14]. The used palm EFB was then immersed in the prepared HCl solution, ensuring that it was fully submerged to maximize the contact between the adsorbent and the acid. The EFB was allowed to soak in the solution for 3 hours.

Once the soaking process was completed, the palm EFB was separated from the acid solution through filtration or decantation. The filtrate collected contained the desorbed ammonia. Following this, the palm EFB was washed thoroughly with distilled water several times to remove any residual HCl or ammonia that remained on its surface. The washing process was continued until the pH of the rinse water reached neutral, indicating the complete removal of acid residues. Finally, the washed palm EFB was dried in an oven at 80ºC to remove any residual moisture. Once dried, the regenerated palm EFB was ready for reuse in further ammonia adsorption processes.

Results and Discussion

Surface Charge and Particle Size Characterization of Palm Empty Fruit Bunch (EFB) and NaOH-Treated Palm Empty Fruit Bunch (EFB)

Surface charges of the palm empty fruit bunch were assessed through zeta potential as depicted in Figure 1 and Figure 2. From Figure 1, the zeta potential of untreated palm EFB was -21.6 mV showing moderate stability in the suspension. Following the NaOH treatment, the zeta potential in Figure 3 became more negative at -38.7 mV which shows stronger particle repulsion and better colloidal stability. The NaOH treatment likely introduced additional negatively charged groups such as hydroxyl or carboxyl groups onto the surface of the palm EFB.

In addition to changes in zeta potential, the variability in surface charge distribution was also assessed through zeta potential deviation. The untreated EFB and NaOH-treated EFB exhibited a deviation of 5.89 mV and 9.18 mV respectively. This increase in variability is due to chemical modification introduced structural or compositional differences across the treated EFB particles and possibly due to uneven surface functionalization or variations in the extent of ion exchange.

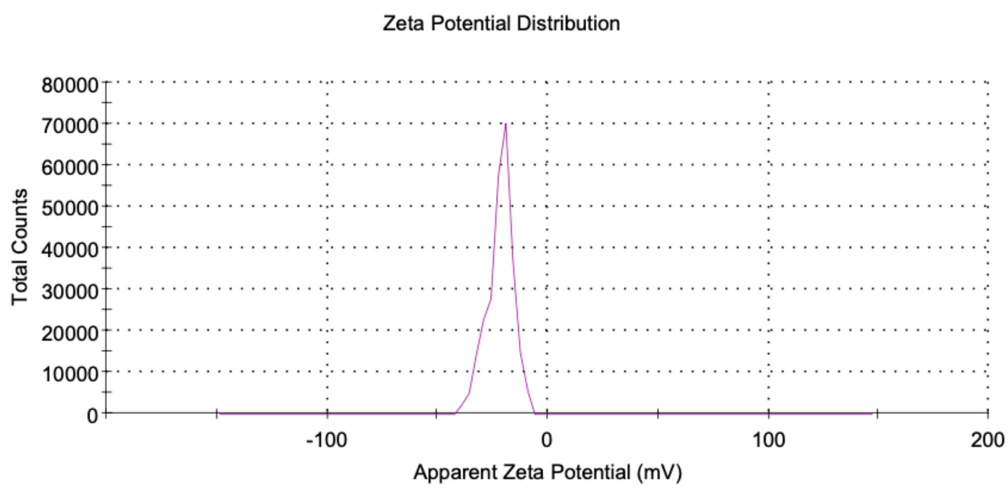


Figure 1. Zeta potential distribution of palm empty fruit bunch (EFB)

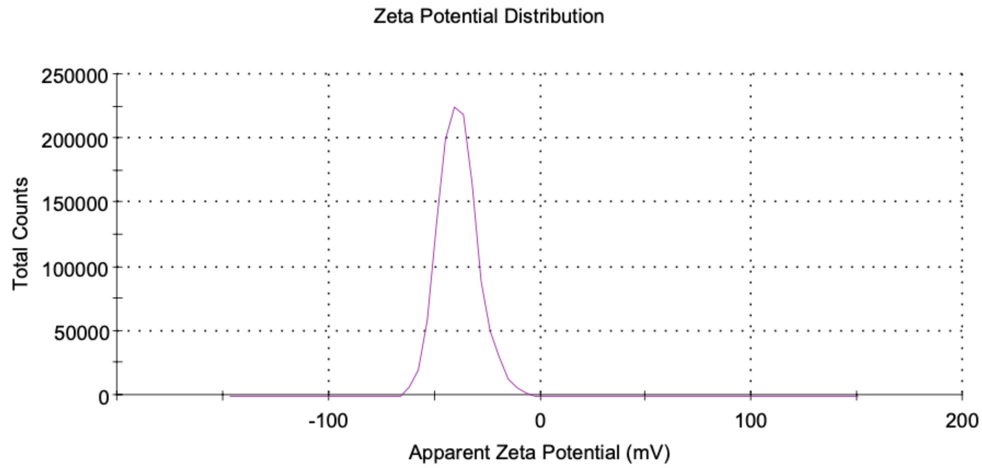


Figure 2. Zeta potential distribution of NaOH treated palm empty fruit bunch (EFB)

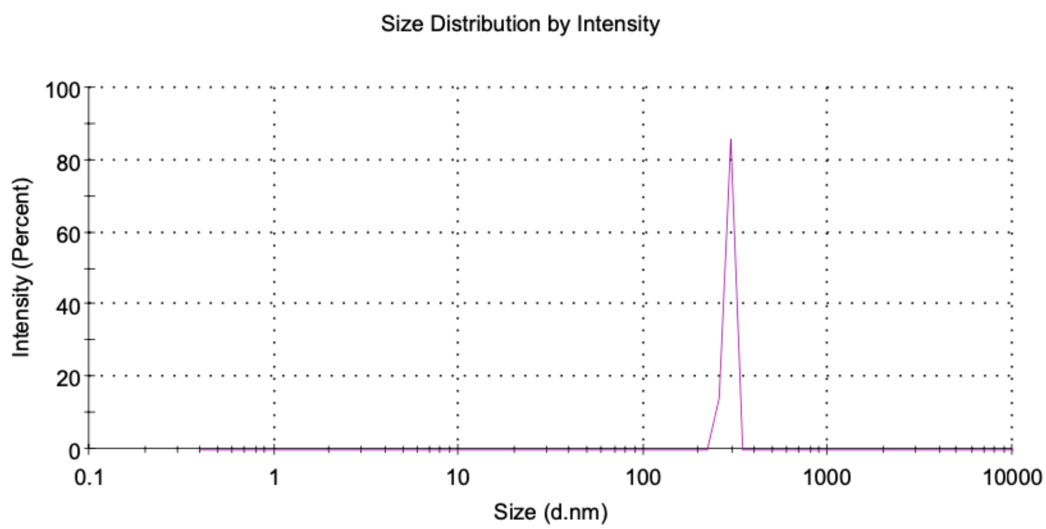


Figure 3. Particle size distribution of NaOH treated palm empty fruit bunch (EFB)

Figure 3 presents the particle size distribution of NaOH treated palm empty fruit bunch (EFB). The sample predominantly consists of particles with a hydrodynamic size of approximately 615.1 nm, representing the dominant particle size peak. However, the Polydispersity Index (PDI) of 0.972 indicates a high level of heterogeneity in the particle size distribution. This high PDI value suggests the presence of larger aggregates or polydisperse regions not distinctly resolved in the distribution plot.

The Z-average diameter, measured at 3469 nm, is notably larger than the dominant peak size. This can be explained by the intensity-weighted nature of the Z-average diameter measurement, which places greater emphasis on larger particles or aggregates, thereby increasing the overall average size reported by dynamic light scattering (DLS).

Structural and Morphology of Palm Empty Fruit Bunch (EFB)

The structural of palm empty fruit bunch (EFB) was determined by x-ray diffraction (XRD) analysis. XRD patterns in Figure 4 compare the structural differences between (a) untreated palm empty fruit bunch (EFB) and (b) NaOH-treated and carbonized palm EFB at 500ºC. Broader and less-defined peaks were shown in untreated palm EFB indicate the presence amorphous cellulose, hemicellulose and lignin which are the main organic components of biomass [15]. The untreated palm EFB also consists predominantly of disordered regions with only limited crystalline domains as observed at the broad peak around 2θ = 20–25° showing the semicrystalline structure likely due to the disruption of the ordered structure of the cellulose during processing. Indeed, the weak peak intensities confirm the dominance of amorphous phases with only minor contributions from crystalline regions.

In contrast, the NaOH-treated and carbonized palm EFB shows significantly sharper and more intense peaks particularly around 2θ = 25–30°, 40–50° and beyond. These peaks are characteristic of carbonaceous structures suggesting the formation of a more ordered graphitic or turbostratic carbon phase. This is consistent with findings from a study that noted distinct peak patterns in XRD are characteristic of h-graphite and turbostratic carbon where turbostratic carbon typically exhibits broadened and less defined peaks due to disorder in the stacking of graphene layers [16]. The ordered structures identified in both studies suggest that higher temperatures can enhance the crystallinity of carbonaceous materials. Improved crystallinity can significantly affect the adsorption properties of these materials, particularly for ammonium ions. More ordered graphite or turbostratic carbon provides a larger surface area and more accessible active sites for ion adsorption which potentially enhances the capacity of the adsorbent for ammonium ion uptake [16]. Furthermore, the increase in crystallinity is due to the removal of hemicellulose and lignin during the NaOH treatment, which enhances the exposure of the cellulose crystalline regions. The overall transformation from broad amorphous peaks to well-defined crystalline peaks indicates a structural transition from an organic biopolymer-rich material to a more carbon-dominant and ordered structure.

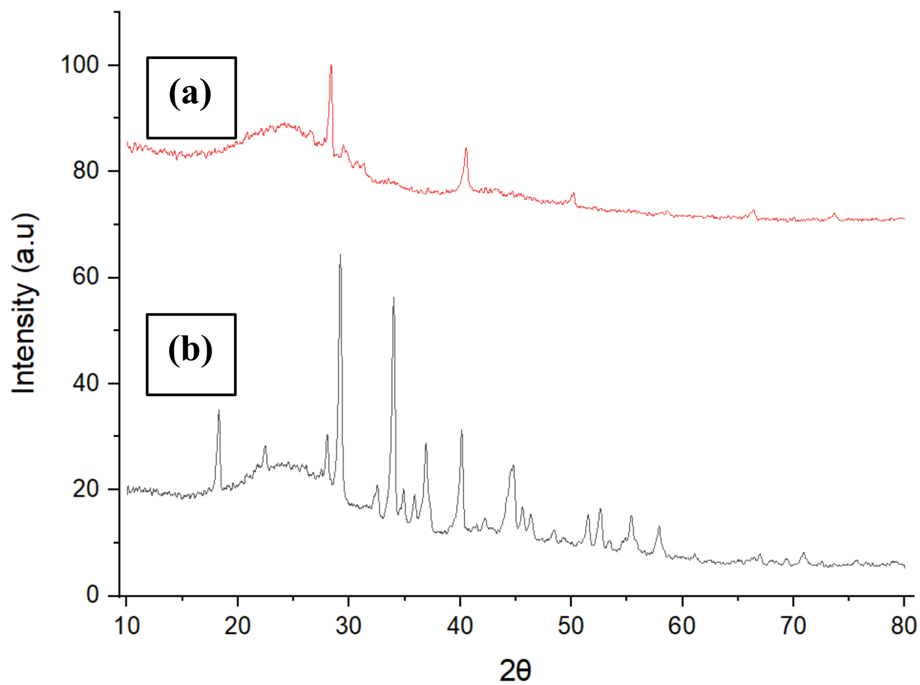


Figure 4. XRD plot of (a) untreated palm empty fruit bunch (EFB) and (b) NaOH treated and carbonized at 500°C palm empty fruit bunch (EFB)

The morphology of palm empty fruit bunch (EFB) treated with NaOH and carbonized at various temperatures (300°C, 500°C, and 700°C) were determined by scanning electron microscopy (SEM) and showed distinct structural changes that enhanced its potential for adsorption of ammonia. As shown in Figure 5 (a), at 300°C, NaOH treatment induced a more irregular pore structure with wider and unevenly distributed pores. This was due to the dissolution of hemicellulose and the disruption of the lignin structure resulting in a rougher surface morphology increased to 500°C as shown in Figure 5 (b), the pore structure became more pronounced, indicating enhanced removal of organic components and a significant increase in surface area. While in Figure 5 (c), at 700°C, NaOH treatment led to a highly porous and fragmented structure, characterized by larger void spaces and an extensively developed porous network. This transformation was enhanced by the introduction of hydroxyl functional groups during NaOH treatment which increased the potential for ammonia adsorption.

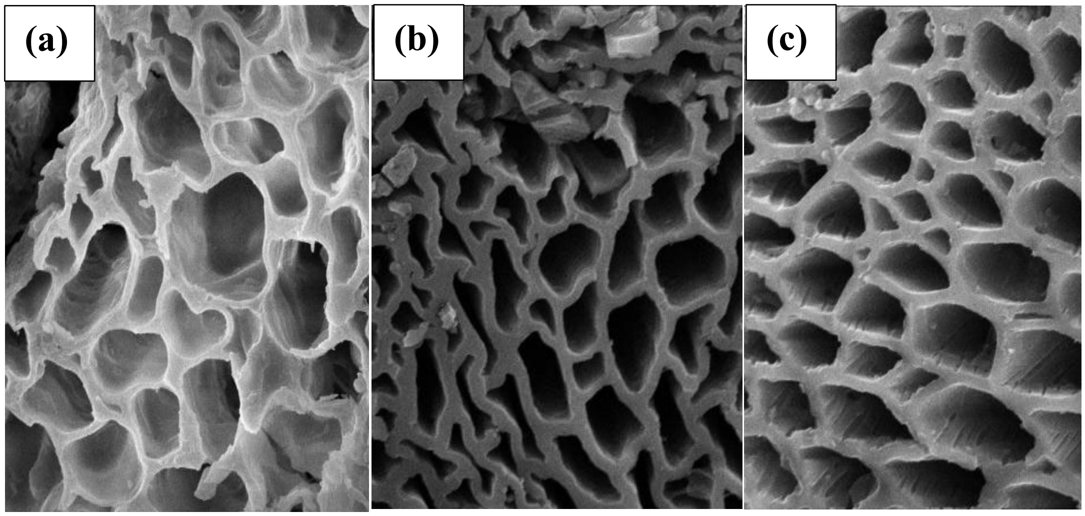


Figure 5. SEM Images for EFB treated with 0.1M NaOH (a) 300°C, (b) 500°C and (c) 700°C

The alkali treatment disrupts the lignocellulosic structure of palm EFB by breaking ester bonds and glycosidic linkages effectively removing lignin and hemicellulose [15]. This process exposes the cellulose matrix and creates a porous structure with a significantly larger surface area. Furthermore, the treatment improves the availability of active adsorption sites such as hydroxyl (-OH) and carboxyl (-COOH) groups which play a critical role in binding ammonium ions (NH_4^+). The alkaline environment created by NaOH deprotonates these functional groups increasing their negative charge and enhancing their attraction to positively charged species like NH_4^+. The chemical reaction governing this mechanism can be summarized as shown in Equation 9 and Figure 6. Additionally, the introduction of hydroxyl groups increases the hydrophilic nature of the surface, making it more favorable for ammonia adsorption in aqueous environments.

Palm EFB (Lignocellulose) + NaOH → Activated Palm EFB (Cellulose – rich structure) + Lignin /Impurities removed (Equation 9)

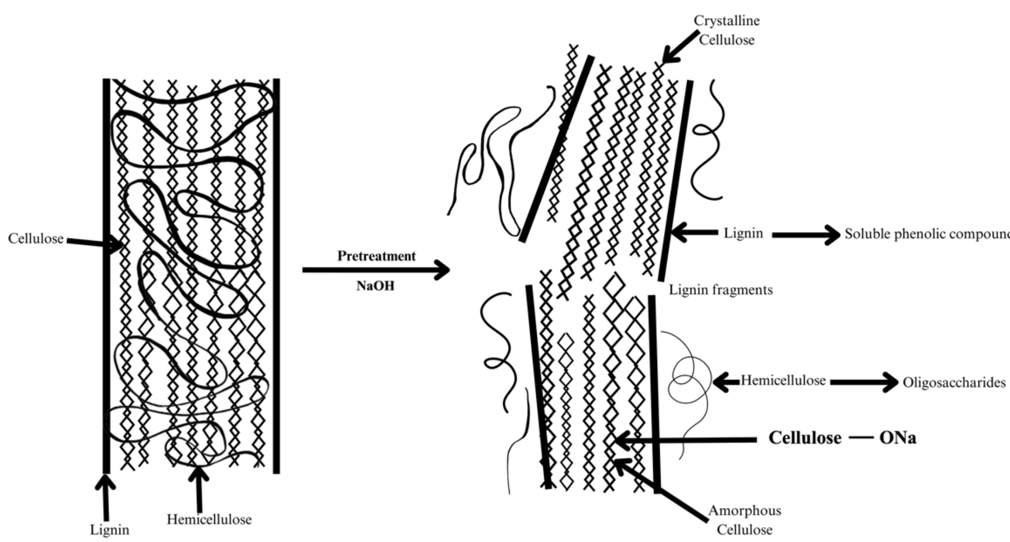


Figure 6. Structural transformation of palm EFB biomass via NaOH pretreatment

After ammonia adsorption, the SEM image as shown in Figure 7 revealed that many of the previously open pores were partially or completely obstructed with ammonia deposits, giving the surface a rough and obstructed appearance. Figure 8 of the EDX spectrum further confirmed the adsorption of ammonia molecules, as evidenced by an increase in the nitrogen content after adsorption. This result indicated the successful capture of ammonia molecules through physical interactions such as van der Waals forces filling the pores and chemical interactions with functional groups like hydroxyl and carboxyl groups. These interactions, verified by the elemental composition from the EDX spectrum contributed to the high adsorption efficiency.

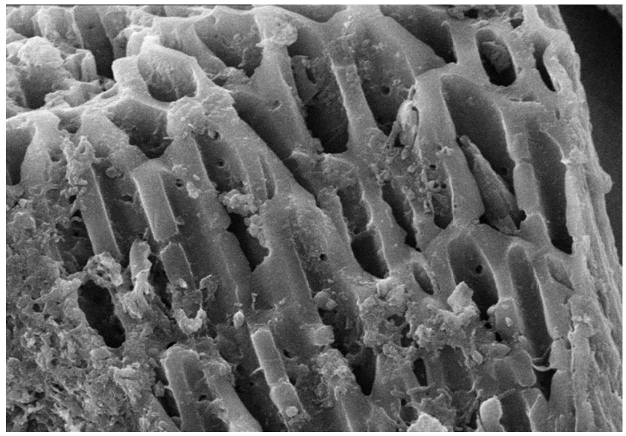
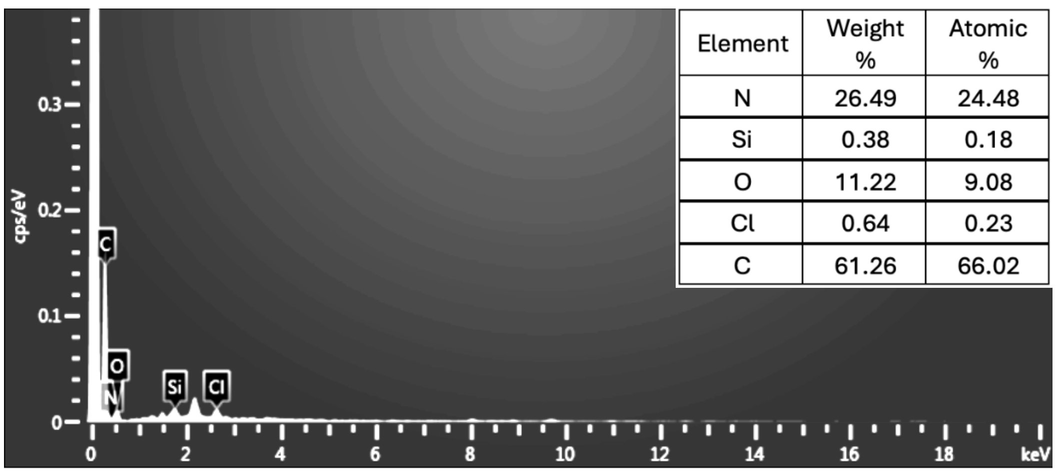


Figure 7. SEM image of after ammonia adsorption of NaOH treated palm empty fruit bunch and carbonized at 500°C



Element	Weight %	Atomic %
N	26.49	24.48
Si	0.38	0.18
O	11.22	9.08
Cl	0.64	0.23
C	61.26	66.02

Figure 8. EDX spectra of after ammonia adsorption of NaOH treated palm empty fruit bunch and carbonized at 500°C

Following regeneration with HCl treatment, as shown in Figure 9, the palm EFB morphology was significantly restored. The surface appeared cleaner, with reopened pores and minimal obstruction compared to the post-adsorption state. The regeneration process effectively desorbed the ammonia by protonating the adsorbed molecules, converting them into soluble ammonium ions that were easily removed. This restored the surface and the active adsorption sites, maintaining the honeycomb-like structure and ensuring that the EFB could be reused efficiently for future ammonia adsorption cycles.

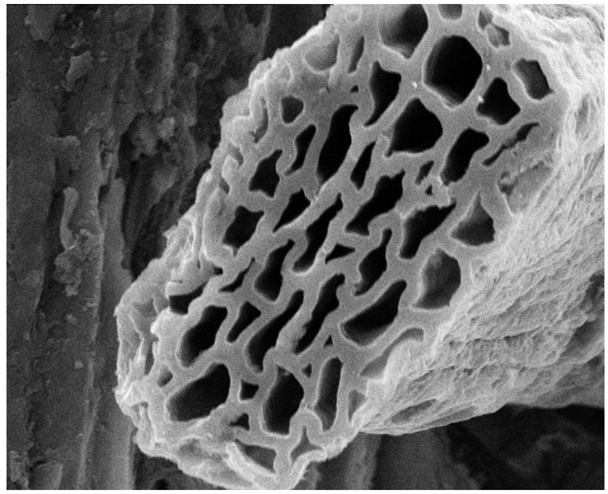


Figure 9. SEM image of regenerated palm EFB

In summary, the SEM analysis depicted after the NaOH treatment, carbonization, adsorption, and regeneration processes have altered and restored the morphology of palm EFB. These changes enhanced the effectiveness of the material to be used for removal of ammonia while maintaining its potential for reuse, highlighting its suitability as a sustainable adsorbent.

Functional Group Analysis

The FTIR spectra in Figure 10 illustrate the functional groups present in palm EFB before ammonia adsorption, after adsorption, and following regeneration.

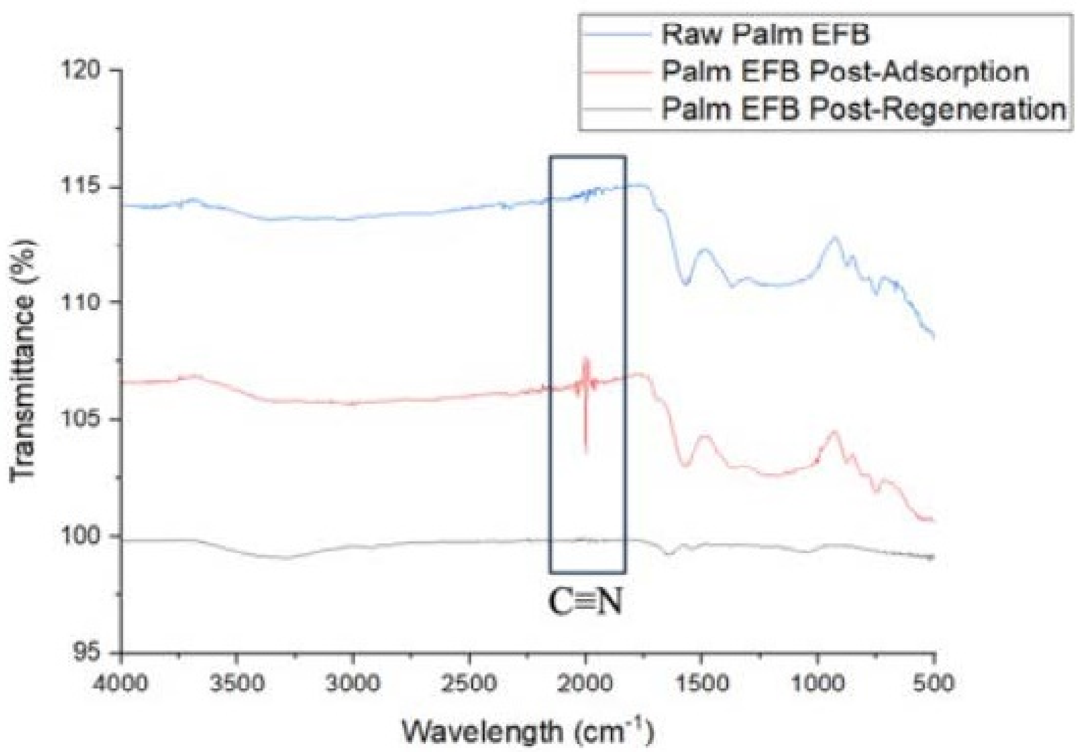


Figure 10. FTIR spectrum of palm EFB treated with NaOH at 500°C (a) before ammonia adsorption (b) after ammonia adsorption (c) after regeneration

The raw palm EFB (Figure 10(a)) shows characteristic peaks such as the broad O–H stretching band around 3300 cm^{-1} and carbonyl (C=O) stretching vibrations near 1730 cm^{-1} and 1630^{-1} cm, typical of lignocellulosic materials.

After ammonia adsorption (Figure 10(b)), a new peak appears at approximately 2200 cm^{-1}. This band is attributed to adsorbed ammonia species interacting with the surface functional groups of palm EFB. These interactions may include hydrogen bonding or coordination involving ammonia or ammonium ions with hydroxyl and carbonyl groups, causing vibrational modes to shift into this region. Such spectral features are consistent with ammonia adsorption mechanisms reported for bio-based adsorbents.

Notably, the intensities of other characteristic peaks, including the O–H stretching band, remain largely unchanged after adsorption. This indicates that the overall lignocellulosic structure of palm EFB is preserved during the process.

Following regeneration (Figure 10(c)), the 2200 cm^{-1} peak significantly diminishes, demonstrating that the adsorbed ammonia has been effectively removed. The regenerated spectrum closely resembles that of the raw palm EFB confirming the restoration of the chemical structure of the material and its suitability for reuse in subsequent adsorption cycles.

Analysis of Variance (ANOVA)

A central composite design (CCD) was employed to optimize the experimental conditions for ammonia removal efficiency from palm oil mill secondary effluent (POMSE) using palm empty fruit bunch (EFB) as an adsorbent. Table 4 presents the ammonia concentration values removed by the adsorbent (palm EFB) after a 60-minute adsorption period. An analysis of variance (ANOVA) was conducted, as shown in Table 5, to evaluate the significance of the model. For a model to be considered statistically significant, the probability value ($P > F$) must be less than 0.05, while values exceeding 0.1000 indicate a lack of significance.

The low $P > F$ values (<0.0001) observed for palm EFB suggest that the model terms are highly significant, validating the reliability of the predictive model in estimating the ammonia removal efficiency. Additionally, the adequate precision values for the palm EFB model (22.6454) exceed the minimum threshold of 4, confirming that the signal-to-noise ratio is sufficient to provide adequate model discrimination. This high adequate precision demonstrates that the model is robust and can be confidently used for further optimization of ammonia removal from POMSE using palm EFB as an eco-friendly adsorbent.

Table 4. Response values for CCD

Run	Factor ***A*** Adsorbent Dosage, (g/L)	Factor ***B*** Initial Ammonia Concentration (mg/L)	Factor ***C*** Carbonization Temperature (°C)	Response (Palm EFB)	
				Ammonia Reduction (mg/L)	% Removal
1	3	195	300	132	32.31
2	3	390	700	246	36.92
3	4	292.5	700	153	47.69
4	4	390	500	249	36.15
5	4	195	500	101	48.21
6	3	390	300	231	40.77
7	3	292.5	500	186	36.41
8	3	292.5	500	177	39.49
9	3	292.5	500	180	38.46
10	3	292.5	500	189	35.38
11	3	292.5	500	204	30.26
12	2	195	500	147	24.62
13	2	292.5	700	225	23.08
14	3	292.5	500	201	31.28
15	4	292.5	300	156	46.67
16	3	292.5	500	195	33.33
17	3	292.5	500	207	29.23
18	3	195	700	117	40.00
19	2	292.5	300	243	16.92
20	2	390	500	258	33.85

Table 5. Significant ANOVA results for palm EFB

Source	Sum of square	Degree of freedom	Mean square	*F*-value	*p* value (Prob>*F*)
Model	36065.68	5	7213.14	36.13	< 0.0001
A	5724.50	1	5724.50	28.67	0.0003
B	29646.13	1	29646.13	148.5	< 0.0001
C	55.13	1	55.13	0.2761	0.6344
AB	342.25	1	342.25	1.71	0.2115
B^2	297.68	1	297.68	1.49	0.2442
Residual	2794.88	14	199.63	-	-
Lack of fit	1923.00	7	274.71	2.21	0.1592
Pure error	871.88	7	124.55	-	-

The lack of fit analysis for the model, indicated by a *p*-value of 0.1592 for palm EFB is greater than 0.05 suggesting that the lack of fit is not significant. A non-significant lack of fit implies that the design model sufficiently accounts for systematic variations within the dataset. The coefficient of determination (R^2) for ammonia removal by palm EFB is 0.9281, demonstrating that over 92% of the total variation in the data is explained by the independent variables with only 8% of the variation remaining unexplained. These results as presented in Table 5, indicate that for palm EFB, the degrees of freedom associated with the residuals are significant at the 5% confidence level, confirming that the model provides an adequate representation of the relationship between the variables and responses.

The predicted value of 36065.68 (Table 5) further highlights significant model terms specifically *A*, *B*, *AB*, and B^2 identifying initial ammonia concentration as the dominant factor in this study (*F*-value = 29646.13). According to Equation 10, only two independent variables: dosage and initial ammonia concentration, exhibit a substantial impact on the efficacy of palm EFB in removing ammonia from POMSE. The high *F*-values associated with initial ammonia concentration underscore the importance of this factor when optimizing the adsorption efficiency of ammonia removal by palm EFB.

$$\text{Ammonia adsorption (palm EFB)} = +193.00 - 26.75A + 60.88B + 9.25AB - 7.88B^2 \qquad \text{(Equation 10)}$$

The intercept term (193.00 mg/L) represents the modeled adsorption at the central, coded zero values of the independent variables specifically, an adsorbent dosage of 3 g/L and initial ammonia concentration of 292.5 mg/L, as defined in the Central Composite Design (CCD). Notably, carbonization temperature (C), despite being a key experimental factor ranging from 300°C to 700°C, is excluded from the final regression model. This exclusion results from statistical analysis via ANOVA, which identified C as statistically insignificant (p-value > 0.05) for explaining variability in ammonia adsorption within the studied parameter range. While carbonization temperature is known to influence physical and chemical properties of biochar, in this study it did not show a meaningful effect on the adsorption outcome compared to adsorbent dosage and initial ammonia concentration.

The exclusion of C enhances model simplicity and predictive reliability, as demonstrated by a high coefficient of determination (R2=0.9281), indicating that over 92% of response variability is accounted for by the included variables. Practically, this suggests that, under the tested conditions, manipulating dosage and initial ammonia concentration yields the most significant impact on improving ammonia removal efficiency with palm EFB.

However, it should be acknowledged that although C was not statistically significant as a main effect, potential indirect or interaction effects, or influences outside the studied temperature range, may still affect performance. Further studies with broader temperature ranges or more complex modeling may be warranted if temperature-dependent properties are of interest.

Normal probability plots of the studentized residuals and the plot showing the predicted versus actual values were employed to evaluate the adequacy of the model. It is shown by a normal distribution where the samples are illustrated in a straight line. In Figure 11, it shows that some scatterings occur in the normal probability model that indicating a normally distributed model. Lastly, the predicted values for palm EFB as shown in Figure 12 is agreed with the actual values that can be used to find the design space.

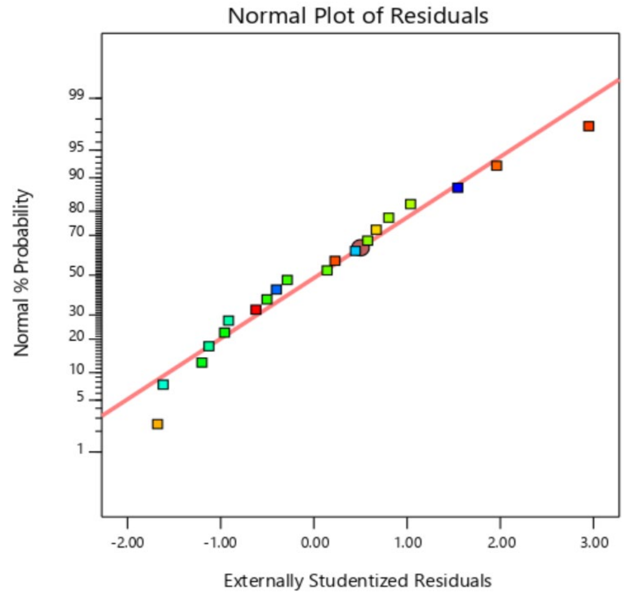


Figure 11. Normal probability plot of the studentized residuals for ammonia removal by Design Expert plot

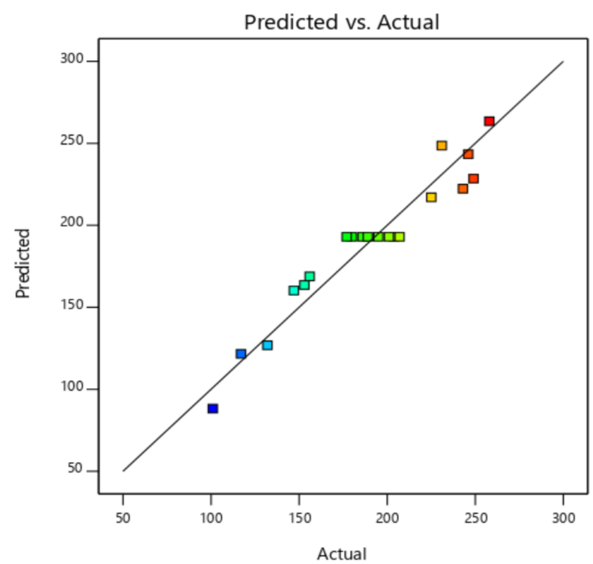


Figure 12. Comparison between the predicted vs actual values for ammonia using palm EFB by Design Expert plot

The Removal Efficiency of Ammonia by Palm Empty Fruit Bunch (EFB)

The interaction between independent variables and the response was evaluated using contour plots and three-dimensional response surfaces generated from the quadratic polynomial model. Figure 13 illustrates the influence of adsorbent dosage (palm EFB) (A) and initial ammonia concentration in POMSE (B) on ammonia removal efficiency, as well as their combined effect when using palm EFB as the adsorbent. Based on Table 5, the initial ammonia concentration (B) shows a stronger impact (148.50) on ammonia removal efficiency compared to adsorbent dosage (A), which has an effect size of 28.67.

Figure 13 further demonstrates that the highest ammonia removal efficiency (101 mg/L) is achieved at an adsorbent dosage (A) of 4 g/L. Conversely, the minimum predicted efficiency occurs at the lowest values of A and the highest initial ammonia concentration of POMSE (B). The interaction between A and B, represented by a positive coefficient (+9.25AB) in the predictive equation, indicates a synergistic interaction, suggesting that simultaneous increases in both dosage and initial ammonia concentration can enhance ammonia removal efficiency.

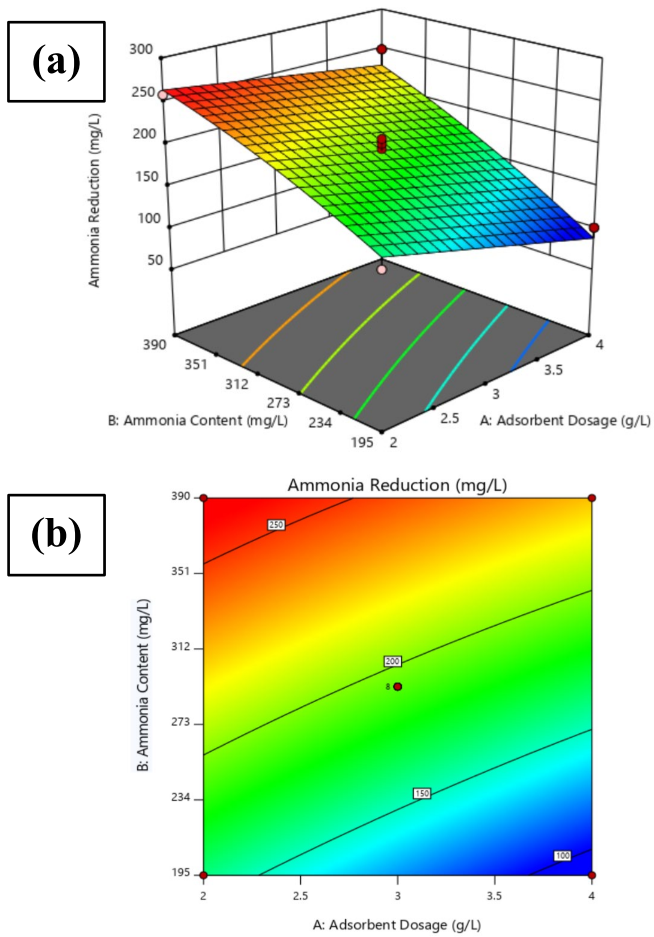


Figure 13. The effect of adsorbent dosage and initial ammonia concentration on ammonia removal in POMSE (a) response surface 3D plot, (b) contour plot

The study also revealed that the adsorbent (palm EFB) demonstrated greater efficiency in adsorbing ammonia in highly diluted (low ammonia concentration) POMSE conditions. Future investigations would benefit from a comparative analysis with other natural adsorbents to evaluate relative adsorption efficiencies more comprehensively.

Optimization of Experimental Methods

The ammonia adsorption findings suggest that palm empty fruit bunch (EFB) could achieve improved ammonia adsorption efficiency if the corresponding optimum conditions are determined. Using Design Expert-11 software, the optimal range for each experimental parameter: adsorbent dosage, initial ammonia concentration and carbonization temperature was identified within the experimental constraints set for this study. For optimization purposes, the study selected the top two predicted conditions for the palm EFB adsorbent. Table 6 presents the optimized ammonia reduction capacities using palm EFB as an adsorbent. Under optimized conditions, the efficiency of palm EFB for ammonia removal was predicted to reach 203.6 mg/L (Solution 1) and 222.4 mg/L (Solution 2) at a dosage of 2 g/L with actual values measured slightly higher at 212 mg/L (Solution 1) and 231 mg/L (Solution 2). Similarly, at a dosage of 4 g/L, predicted values were 168.9 mg/L (Solution 3) and 163.7 mg/L (Solution 4), closely aligned with actual measurements of 164 mg/L (Solution 3) and 167 mg/L (Solution 4). The data demonstrate strong agreement with predicted values, with percent deviations below 5% confirming the reliability of the model.

These findings indicate that palm EFB is a suitable and effective adsorbent for ammonia removal from POMSE, displaying consistent and high adsorption efficiency. The low deviation from predicted values further emphasizes the potential of palm EFB as a sustainable alternative to synthetic adsorbents in wastewater treatment applications.

Table 6. Optimization of ammonia reduction by Palm EFB.

Solution No.	Adsorbent Dosage (g/L)	Ini. Ammonia Conc. (mg/L)	Carbonization Temp. (°C)	Final Ammonia Conc. (mg/L)		
				Predicted	Actual	% deviation
1	2	292.5	500	203.6	212	4.1
2	2	292.5	500	222.4	231	3.9
3	4	292.5	500	168.9	164	2.9
4	4	292.5	500	163.7	167	2.0

Adsorption Isotherms

As shown in Table 7 and Figure 14, the adsorption behavior of the EFB adsorbent aligns more closely with the Langmuir isotherm (R^2 = 0.9447) compared to the Freundlich isotherm (R^2 = 0.8949). This result suggests that ammonia adsorption occurs predominantly on homogeneous surfaces forming a monolayer as described by the Langmuir model. In contrast, the Freundlich isotherm which describes adsorption on heterogeneous surfaces is less applicable in this context. Furthermore, the dimensionless equilibrium parameter for EFB, as shown in Table 7, is 0.6976. This value indicates the favorability of the adsorption isotherm, providing further validation in line with Equation 11.

$$R_L = \frac{1}{1 + bC_0} \quad \text{(Equation 11)}$$

where b is the Langmuir constant and C_0 is the initial concentration of ammonia in POMSE. The R_L value indicate if it is unfavourable ($R_L > 1$), linear ($R_L = 1$), favourable ($0 < R_L > 1$) and irreversible ($R_L = 0$), thus indicating the EFB was in favourable process of adsorption.

Table 7. Langmuir and Freundlich isotherm for Palm EFB.

Langmuir isotherm	
Q (mg/g)	0.00400
b (L/mg)	0.00179
R^2	0.94468
R_L	0.6976
Freundlich isotherm	
K (mg/g) $(L/g)^{1/n}$	2.55941E-05
1/n	0.69773
R^2	0.89495

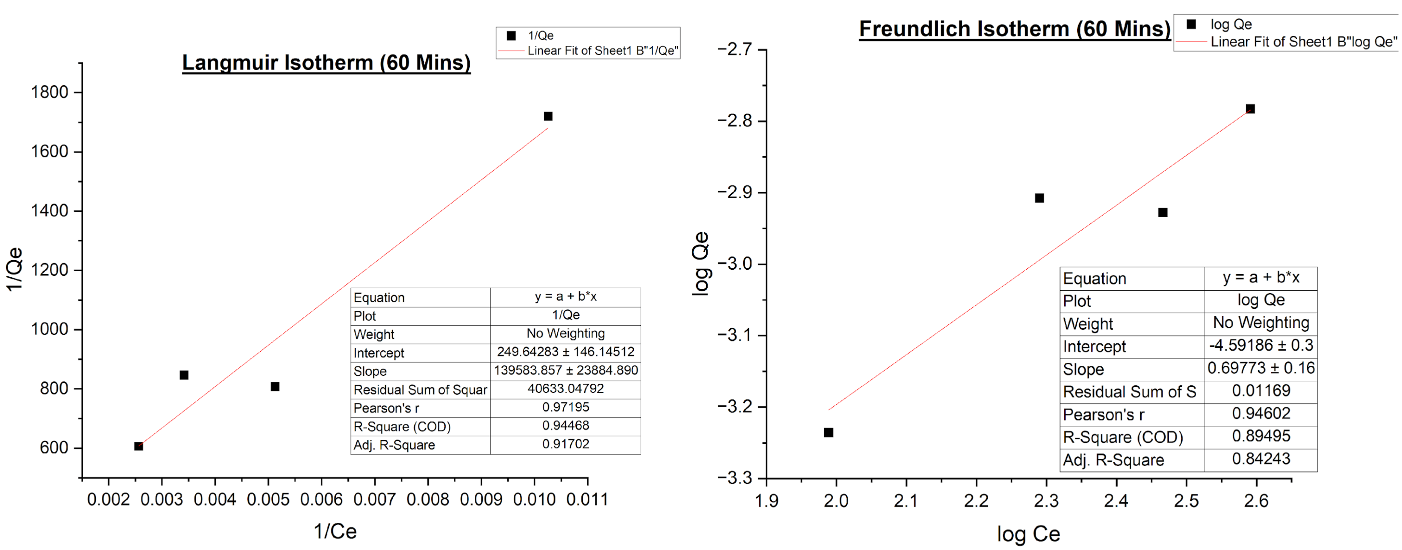


Figure 14. Langmuir and Freundlich Isotherm for EFB

Regeneration Performance

The adsorption results demonstrated that the most favorable conditions for ammonia adsorption were achieved using an adsorbent dosage of 4 g/L and a carbonization temperature of 500°C. Under these conditions, the predicted final ammonia concentration (203.6 mg/L) closely matched the actual experimental value (212 mg/L), with a percentage deviation of only 4.1%. This indicates that the carbonized palm EFB at these parameters had an optimal surface area, porosity, and distribution of active sites for ammonia capture. The adsorbent's high surface area and microporous structure, resulting from carbonization at 500°C, facilitated effective ammonia adsorption by providing sufficient adsorption sites and enhancing physical interaction between ammonia molecules and the adsorbent.

Following this optimization, the regeneration study was performed to evaluate the reusability of the adsorbent. The results revealed that after regeneration, only 2 mg/L of ammonia was desorbed into the solution, corresponding to a desorption efficiency of 2.27%. The low desorption efficiency of only 2.27% after treatment with 0.1M HCl implying that the vast majority of adsorbed ammonia remains strongly retained on the adsorbent surface. This limited desorption can be critically attributed to several intertwined physicochemical factors related to the nature of the adsorbent, the adsorption mechanisms, and the regeneration method employed.

Firstly, carbonization at 500°C substantially modifies the surface chemistry of palm EFB, producing a biochar matrix with a high degree of aromaticity, microporosity, and acidic functional groups such as carboxyl (-COOH) and phenolic (-OH) groups. These acidic sites strongly favor ammonium ion (NH_4^+) adsorption through ion exchange, hydrogen bonding, and complexation mechanisms. The formation of stable ammonium salts or surface complexes limits the reversibility of the adsorption process, particularly under mild desorption conditions using dilute hydrochloric acid. The presence of these strong chemisorptive interactions explains why simple acid washing removes only a small fraction of adsorbed ammonia [1].

Secondly, the enhanced micropore structure and high surface area induced by carbonization, while beneficial for adsorption capacity, can also hinder desorption. Ammonia molecules may penetrate into narrow micropores and become physically entrapped or strongly adsorbed via van der Waals forces and pore-filling interactions, requiring more aggressive regeneration conditions (e.g., higher acid strength, alkaline or thermal treatments) to be released [1]. The observed low desorption contrasts with adsorbents carbonized at lower temperatures or prepared via physical activation, which generally show higher desorption due to predominance of physisorption rather than chemisorption.

Thirdly, competing ions present in palm oil mill secondary effluent (POMSE) such as potassium (K^+) and magnesium (Mg^{2+}) may form multi-ion complexes with the biochar surface, further stabilizing ammonium adsorption sites and impeding desorption. Such sites may require stronger chemical disruption than the mild acid employed in this study.

The low desorption efficiency found here is consistent with other reports on biochar-based ammonia adsorbents. For example, studies on rice husk biochar reported desorption efficiencies of less than 10% with acid washing, while alkaline or thermal regeneration methods achieved higher desorption but at the expense of partial adsorbent degradation and capacity loss [1]. Similarly, coconut husk biochar exhibited limited ammonia recovery using acidic desorption, highlighting a general trend for strongly chemisorptive agricultural biochars.

While strong ammonia binding increases adsorption stability and reduces leaching risk, it presents challenges for sustainable adsorbent reuse since regenerability is key for cost-effective wastewater treatment. Mild regeneration agents like 0.1M HCl are insufficient to break the strong chemical bonds or displace ammonia from micropores, necessitating exploration of alternative regeneration strategies. These could include alkaline treatment to disrupt ammonium complexes, thermal regeneration at controlled temperatures to desorb ammonia without structural damage, or combinatorial chemical-thermal methods. Additionally, surface modifications aimed at reducing acid group density or tailoring pore size distribution might balance adsorption capacity with improved desorption.

In conclusion, the low desorption efficiency of ammonia from palm EFB biochar under mild acidic regeneration is fundamentally due to a predominance of strong chemisorption and physical entrapment combined with the inherent surface chemistry generated by carbonization. This is a commonly observed phenomenon among high-temperature carbonized agricultural biochars and highlights a critical trade-off between adsorption strength and adsorbent regenerability. Future work must prioritize advanced regeneration techniques and adsorbent design optimization to render palm EFB biochar a reusable and sustainable ammonia adsorbent for industrial wastewater treatment.

Conclusions

In conclusion, this study successfully demonstrates the potential of palm empty fruit bunch (EFB) as an effective adsorbent for the removal of ammoniacal nitrogen from palm oil mill secondary effluent (POMSE). Utilizing Response Surface Methodology (RSM) with a central composite design (CCD), the study has identified optimal conditions for ammonia adsorption and reveals that an increase in palm EFB dosage significantly enhances removal efficiency. Specifically at a dosage of 4 g/L, approximately 43% of ammonia was removed from a concentration of 292.5 mg/L at a carbonization temperature of 500^{o}C. The adsorption process was characterized by the Langmuir isotherm model indicating a monolayer adsorption on a homogeneous surface which underscores the effectiveness of palm EFB biochar in treating POMSE. Scanning Electron Microscopy (SEM) analysis confirmed the successful interaction between ammonia molecules and the active sites on the biochar surface while Fourier Transform Infrared (FTIR) spectroscopy indicated that the structural integrity of the palm EFB was maintained throughout the adsorption process. Furthermore, the regeneration of the biochar was effective allowing for its reuse in subsequent adsorption without significant degradation. This study not only highlights the feasibility of using agricultural waste as a sustainable adsorbent for wastewater treatment but also laying the groundwork for potential future processes aimed at ammonia recovery and resource reuse. Future work should focus on scaling up this process and exploring the long-term performance and regeneration of palm EFB in various wastewater treatment.

Conflicts of Interest

The author(s) declare(s) that there is no conflict of interest regarding the publication of this paper.

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