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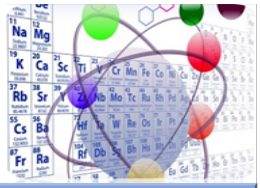

Effectiveness of Modified Empty Palm Oil Bunch-Based Activated Carbon for Reducing Ammonia Content in Palm Oil Wastewater

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ABSTRACT

Indonesia, particularly North Sumatra, is a major producer of palm oil, generating significant waste such as empty fruit bunches (EFB) and ammonia-rich wastewater. Ammonia pollution poses serious environmental risks if not treated properly. This study explores the potential of three types of adsorbents—Fe-Cu modified activated carbon, AC-Cu(TAC)₂ composite, and unmodified activated carbon—for ammonia adsorption. Activated carbon was synthesized from EFB and further modified using Fe-Cu and Cu(TAC)₂. Adsorption experiments were conducted with variations in adsorbent mass, contact time, and NH_4OH concentration. The results showed that Fe-Cu modified activated carbon had the highest ammonia removal efficiency (90%), while the AC-Cu(TAC)₂ composite had the highest adsorption capacity. Equilibrium analysis revealed that the AC-Cu(TAC)₂ composite fit both Langmuir and Freundlich isotherm models (R^2 = 1), suggesting both monolayer and heterogeneous adsorption. The unmodified activated carbon followed the Langmuir model (R^2 = 0.9498), while Fe-Cu modified activated carbon did not fit either model. Kinetic studies indicated that Fe-Cu modified activated carbon followed a pseudo-first-order model (R^2 = 0.9936), implying physisorption, whereas both AC-Cu(TAC)₂ and unmodified activated carbon followed pseudo-second-order models (R^2 = 0.9779 and 0.9869), indicating chemisorption. These findings highlight the promising potential of modified EFB-based adsorbents for ammonia removal in wastewater treatment applications.

Keywords: Adsorption, absorption efficiency, pseudo second order kinetics

1. INTRODUCTION

According to data from the Ministry of Agriculture, North Sumatra, Indonesia ranks among the top ten provinces for palm oil in 2021, with a production of 5.45 million tons of palm oil and an area of 1.54 million hectares (Directorate General of Estates, Ministry of Agriculture of the Republic of Indonesia, 2023). The harvest of fresh fruit bunches (FFB) from palm oil generates palm empty fruit bunch waste of 20-23%. Additionally, the production of one ton of Crude Palm Oil (CPO) can result in liquid waste from palm oil mills, known as Palm Oil Mill Effluent (POME), amounting to 2.5-3.5 m^3. Palm oil wastewater has a high ammonia content from the decomposition of proteins and fats by microorganisms (bacteria) (Sitepu et al.,

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2018). Ammonia is naturally produced from decayed protein in the decomposition of waste or organic matter (Daniel et al., 2020). If this is not managed properly, it will become a major problem in the future and cause environmental pollution (soil, water, and air) (Wafti et al., 2017). To reduce ammonia in aquatic biota, various methods can be employed such as ion exchange, adsorption, solvent extraction, and precipitation. The most effective and commonly used method is adsorption (Jasmidi et al., 2022).

Adsorption has become a popular new method for reducing waste and heavy metal content in water due to its simplicity and low cost, effectiveness and efficiency in terms of absorption processes, and the ability to be a regenerable adsorbent (Jasmidi et al., 2022). Empty Palm Oil Bunch (TKKS) consists of lignin, hemicellulose, and cellulose components. The high carbon and cellulose content in TKKS makes it a suitable candidate for application as an adsorbent (Thao et al., 2019). Currently, the techniques for utilizing and processing Empty Palm Oil Bunch (TKKS) focus on waste management and providing added value to the industry. Activated carbon as an adsorbent material is generally used in the adsorption process of organic compounds due to its high porosity, hydrophobicity which functions for surfactant stability in activated carbon modification, and its relatively low cost. However, the irregularity of pore size distribution and weak interaction between the adsorbate and its adsorbent (Munoz-Senmache et al., 2020).

The objective of this article is to investigate the equilibrium and kinetic properties of three different types of adsorbents, namely Fe-Cu modified activated carbon, the AC-Cu(TAC)$_2$ composite, and unmodified activated carbon.

2. EXPERIMENTAL

The method used in this research is as follows

2.1. Tools and Materials

The main material used in this research is Oil Palm Empty Fruit Bunch (EFB). The chemicals used include NH_4OH (Ammonium Hydroxide), H_3PO_4 (Phosphoric Acid), HCl (Hydrochloric Acid), TAC (Terephthalic Acid), HF (Hydrofluoric Acid), HNO_3 (Nitric Acid), $Cu(NO_3)_2$ (Copper(II) Nitrate), $FeSO_4$ (Ferrous Sulfate), $CuSO_4$ (Copper(II) Sulfate), Phenolphthalein Indicator (PP), distilled water (aquadest), and filter paper. The glassware and equipment used include burettes, stands, clamps, an analytical balance, a 200-mesh sieve, an oven, a furnace, a hot plate, and reflux apparatus.

2.2. Research Procedures

The procedures in this study consist of several stages, including:

2.2.1. Synthesis of Activated Carbon from Oil Palm Empty Fruit Bunch (EFB)

The oil palm empty fruit bunch biosorbent is carbonized using a furnace at 500°C for 2 minutes. It is then activated using phosphoric acid (H_3PO_4) for 24 hours.

2.2.2. Modification of Activated Carbon with Fe-Cu

30 grams of activated carbon are added into 150 mL of a Fe and Cu solution with a 1:1 ratio. The mixture is then heated for 6 hours, cooled, and filtered. The residue is then placed in an oven at 105°C for 2 hours.

2.2.3. Activated Carbon – Cu(TAC)$_2$ Composite

The modification of activated carbon with MOFs Cu(TAC)$_2$ is carried out by soaking a mixture of terephthalic acid, ethanol, and activated carbon in a ratio of 1:10:0.6 for 24 hours. The mixture is then combined with the MOFs Cu(TAC) solution and refluxed at 105°C for 8 hours.

2.2.4. NH_4OH Adsorption Using Activated Carbon

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Ammonia adsorption is conducted with three variations: mass, concentration, and contact time. For the mass variation, 30 mL of 0.3 M NH_4OH is added to 0.5 g, 1 g, 2 g, 4 g, and 8 g of activated carbon. To determine the optimal concentration, 0.1 M, 0.2 M, 0.3 M, 0.4 M, and 0.5 M NH_4OH are used. To evaluate time efficiency, contact times of 15, 30, 45, 60, and 75 minutes are tested.

2.2.5. NH_4OH Adsorption Using Fe-Cu Modified Activated Carbon

Adsorption using Fe-Cu modified activated carbon is also conducted with three variations. First, to evaluate the effect of adsorbent mass, 0.5 g, 1 g, 2 g, 4 g, and 8 g of Fe-Cu modified activated carbon are added to a beaker glass, followed by 30 mL of NH_4OH. The mixture is stirred at 125 rpm for 45 minutes, filtered, and the filtrate is titrated with HCl. To determine the optimal concentration, 0.1 M, 0.2 M, 0.3 M, 0.4 M, 0.5 M, 0.6 M, and 0.7 M NH_4OH solutions are used. For contact time variation, durations of 15, 30, 60, and 90 minutes are applied.

2.2.6. NH_4OH Adsorption Using Activated Carbon – $Cu(TAC)_2$

Ammonia adsorption is conducted with three variations: mass, concentration, and contact time. For the mass variation, 30 mL of 3 M NH_4OH is added to 0.5 g, 1 g, 2 g, 4 g, and 8 g of the composite. To determine the optimal concentration, 1 M, 2 M, 3 M, 4 M, and 5 M NH_4OH solutions are used. For contact time variation, durations of 15, 30, 45, 60, and 75 minutes are tested.

3. RESULTS AND DISCUSSION

3.1. Analysis of the Adsorption Results of Types of Activated Carbon from Empty Palm Fruit Bunches Based on Their Adsorption Efficiency (%)

The results of the efficiency analysis of each adsorption process show that the higher the efficiency (%), the better the absorption process.

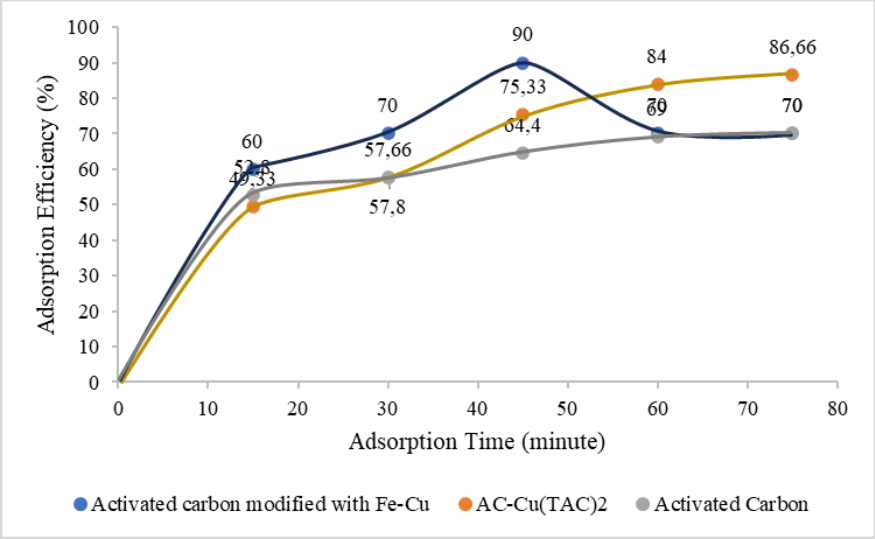

Figure 1. Adsorption Efficiency Under Different Mass Variations (%) of Activated Carbon Modified with Fe-Cu, Activated Carbon - $Cu(TAC)_2$, Activated Carbon

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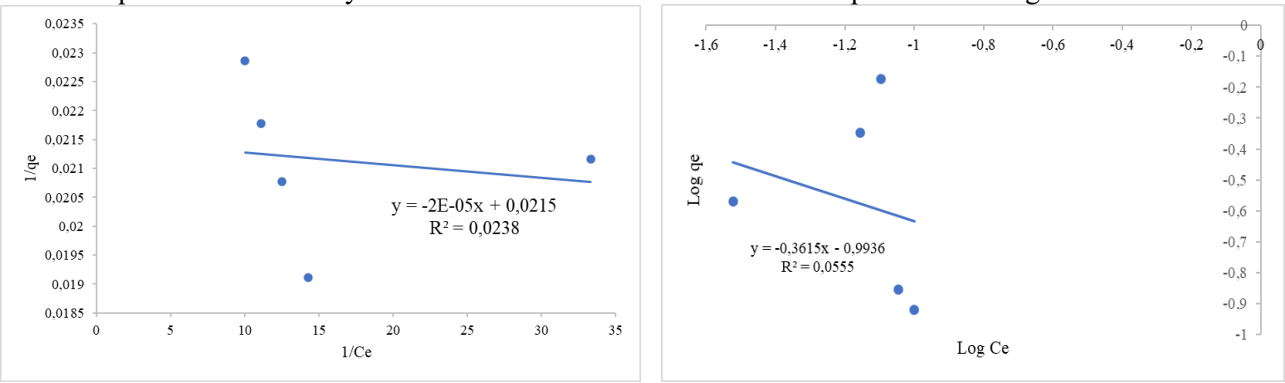
The figure 1 illustrates the adsorption efficiency of ammonia (NH_4OH) over different adsorption times using three types of adsorbents: activated carbon modified with Fe-Cu, AC-Cu(TAC)$_2$ composite, and unmodified activated carbon. Initially, all three adsorbents show a rapid increase in adsorption efficiency within the first 10 minutes. The Fe-Cu modified activated carbon reaches about 60% efficiency, AC-Cu(TAC)$_2$ reaches approximately 52.8%, and unmodified activated carbon achieves around 49.33%. As the adsorption time increases to 30 minutes, the Fe-Cu modified activated carbon continues to outperform the others, reaching 70%, followed by AC-Cu(TAC)$_2$ at 57.66% and unmodified activated carbon at 57.8%. At 45 minutes, the Fe-Cu modified activated carbon attains its peak efficiency of 90%, which is the highest among all. The AC-Cu(TAC)$_2$ composite also shows a significant increase to 75.33%, while unmodified activated carbon reaches 64.4%. Beyond 45 minutes, the Fe-Cu modified activated carbon's efficiency slightly declines to 70% at 70 minutes, whereas AC-Cu(TAC)$_2$ continues to increase, achieving the highest efficiency of 86.66% at 75 minutes. The unmodified activated carbon remains relatively stable, reaching 70% at 75 minutes. This trend indicates that while Fe-Cu modified activated carbon exhibits the fastest and highest initial adsorption efficiency, AC-Cu(TAC)$_2$ ultimately reaches a comparable or higher efficiency given a longer adsorption time. Unmodified activated carbon shows the lowest and most stable performance throughout the tested periods.

3.2. Analysis of Adsorption Equilibrium Properties

Adsorption isotherm analysis was conducted to evaluate the characteristics and adsorption capacities of three types of adsorbents: Fe-Cu modified activated carbon, AC-Cu(TAC)$_2$ composite, and unmodified activated carbon. Using isotherm models such as Langmuir and Freundlich, this study aims to understand how each adsorbent interacts with the adsorbate (NH_4OH) under equilibrium conditions. The results of this analysis provide insights into the adsorption efficiency, maximum adsorption capacity, and surface behavior of each adsorbent. A comparison of the three adsorbents also reveals the effect of surface modification on their adsorption performance.

(a) Adsorption Isotherm On Fe-Cu Modified Activated Carbon

The adsorption isotherm analysis of Fe-Cu modified activated carbon is presented in Figure 2 below.



(b) Isotherm Langmuir **(c)** Isotherm Freundlich

Figure 2. Adsorption Isotherm On Fe-Cu Modified Activated Carbon,
(a) Isotherm Langmuir (b) Isotherm Freundlich

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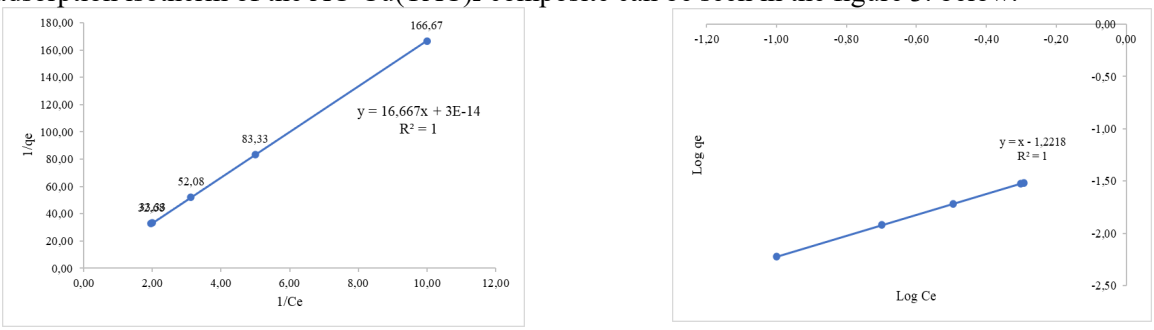
Figure 2. presents the Langmuir isotherm plot, while Figure (b) shows the Freundlich isotherm plot for the adsorption process. Based on the coefficient of determination (R^2), the Freundlich model gives **R^2 = 0.0555**, which is slightly higher than the Langmuir model with **R^2 = 0.0238**. However, both R^2 values are very low, indicating that **neither the Langmuir or Freundlich isotherm fits well** for describing the adsorption behavior observed in this experiment. In the Langmuir plot, the relationship between 1/qe and 1/Ce shows a very weak decreasing trend (as indicated by the near-zero slope), suggesting a poor fit to the Langmuir monolayer adsorption model. In the Freundlich plot, the distribution of data points (log qe vs. log Ce) also shows a poor alignment with the regression line. This implies that the adsorption process may not follow an ideal monolayer (Langmuir) or heterogeneous multilayer (Freundlich) mechanism, and could be influenced by other factors such as non-uniform surface characteristics, weak adsorbate–adsorbent interactions, or insufficient data points.

(b) Adsorption Isotherm on AC-Cu(TAC)$_2$ Composite

The adsorption isotherm of the AC-Cu(TAC)$_2$ composite can be seen in the figure 3. below.



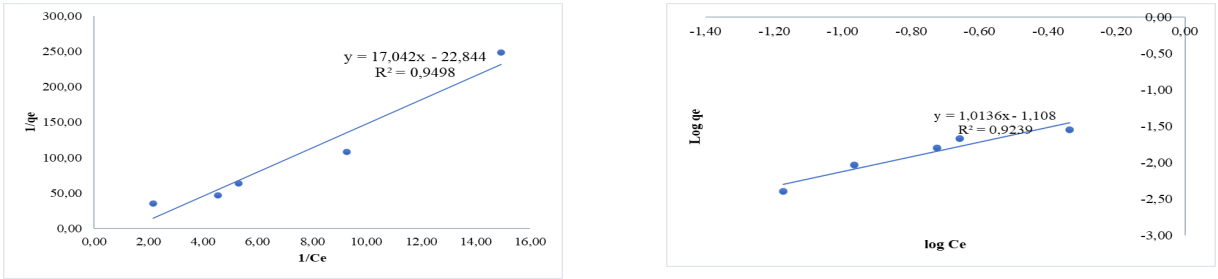
(a) Isotherm Langmuir (b) Isotherm Freunlich

Figure 3. Adsorption Isotherm of The AC-Cu(TAC)$_2$ Composite,
(a) Isotherm Langmuir (b) Isotherm Freundlich

In figure 3.(a), the data show a linear relationship between 1/qe and 1/Ce, with the regression equation y = 16.667x + 3E-14 and an R^2 value of 1. This perfect R^2 indicates that the Langmuir model fits the adsorption data extremely well, suggesting that the adsorption occurs as a monolayer on a homogeneous surface, with no interactions between adsorbed molecules. In figure 3. (b) linear relationship is also observed between log qe and log Ce, with the equation y = x - 1.2218 and again R^2 = 1. This perfect fit indicates that the Freundlich model also accurately describes the adsorption process, which implies a heterogeneous surface and the possibility of multilayer adsorption, with adsorption sites of varying energy.

(c) Adsorption Isotherm on Activated Carbon

The adsorption isotherm analysis of unmodified activated carbon can be seen in Figure 4.



(a) Isotherm Langmuir (b) Isotherm Freundlich

Figure 4. Adsorption Isotherm of The Activated Carbon, (a) Isotherm Langmuir (b) Isotherm Freundlich

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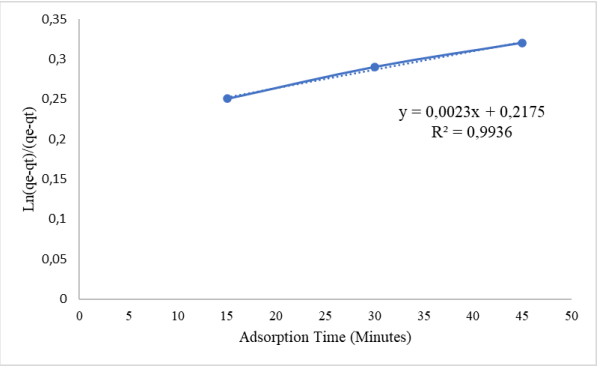
The adsorption isotherm analysis of unmodified activated carbon is presented in Figure 4, which includes both the Langmuir and Freundlich models. In the Langmuir isotherm (Figure 4a), a linear relationship between 1/qe and 1/Ce is observed, with the regression equation y = 17.042x – 22.844 and a determination coefficient (R^2) of 0.9498. This high R^2 value indicates that the Langmuir model fits the adsorption data well, suggesting that the adsorption occurs as a monolayer on a relatively homogeneous surface. Meanwhile, the Freundlich isotherm (Figure 4b) shows a linear relationship between log qe and log Ce, with the regression equation y = 1.0136x – 1.108 and an R^2 value of 0.9239. This also demonstrates a good fit, implying that the adsorption process takes place on a heterogeneous surface and may involve multilayer adsorption. Although both models fit the data reasonably well, the slightly higher R^2 value of the Langmuir model suggests that the adsorption mechanism of unmodified activated carbon is more consistent with monolayer coverage on a uniform surface.

3.3. Analysis of Adsorption Kinetic Properties

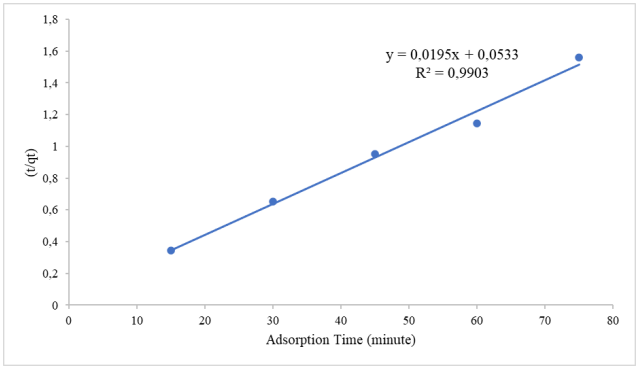
Here is the pseudo-order kinetic analysis for the three types of adsorbents.

a) Pseudo-Order Kinetic Analysis on Fe-Cu Modified Activated Carbon

The pseudo-order kinetic analysis was conducted on Fe-Cu modified activated carbon to understand the adsorption mechanism and rate. This analysis helps determine whether the adsorption process follows a pseudo-first-order or pseudo-second-order kinetic model, which describes how quickly the adsorbate molecules are captured by the adsorbent surface over time. The results provide insight into the efficiency and behavior of the Fe-Cu modified activated carbon in removing contaminants like ammonia.



(a) Pseudo First Order



(b) Pseudo Second Order

Figure 5. Pseudo-Order Kinetic Analysis on Fe-Cu Modified Activated Carbon,
(a) Pseudo First Order (b) Pseudo Second Order

The figure above illustrates the adsorption kinetics analysis of Fe-Cu modified activated carbon using both pseudo-first-order and pseudo-second-order models. In Figure (a), the linear relationship between ln(qe–qt) and adsorption time represents the pseudo-first-order kinetics, yielding the regression equation **y = 0.0023x + 0.2175** with a coefficient of determination **R^2 = 0.9936**. This high R^2 value indicates that the pseudo-first-order model fits the experimental data very well. In contrast, Figure (b) shows the pseudo-second-order model, where a linear plot of t/qt versus adsorption time produces the equation **y = 0.0195x + 0.0533** with **R^2 = 0.9903**. Although both models show excellent correlation with the data, the slightly higher R^2 value in the pseudo-first-order model suggests a better fit. Therefore, it can be concluded that the

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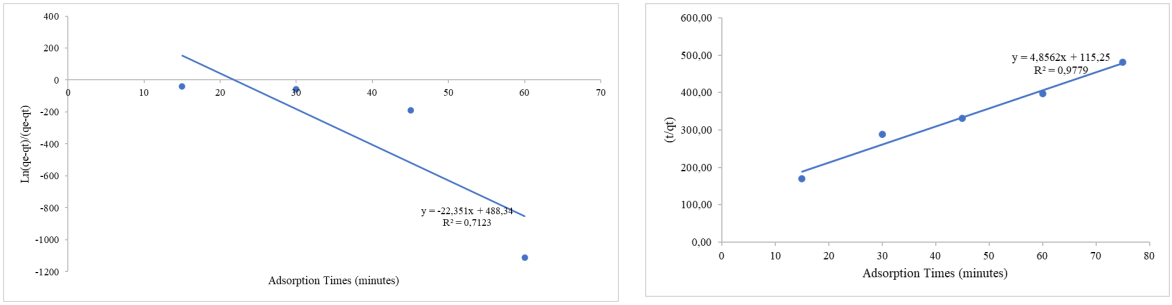
adsorption of NH_4OH onto Fe-Cu modified activated carbon follows pseudo-first-order kinetics more closely, implying that the rate of adsorption is mainly influenced by the number of available active sites rather than the concentration of the adsorbate in the solution.

b) **Pseudo-Order Kinetic Analysis on AC-Cu(TAC)$_2$**

The adsorption kinetics of NH_4OH onto the AC-Cu(TAC)$_2$ composite adsorbent are analyzed using pseudo-first-order and pseudo-second-order kinetic models. The purpose of this analysis is to determine the rate-controlling mechanism and to evaluate which kinetic model best describes the adsorption process on AC-Cu(TAC)$_2$.



(a) Pseudo First Order (b) Pseudo Second Order

Figure 6. Pseudo-Order Kinetic Analysis on AC-Cu(TAC)$_2$
(a) Pseudo First Order (b) Pseudo Second Order

The figure above presents the pseudo-order kinetic analysis of adsorption on the AC-Cu(TAC)$_2$ composite. Two models are compared: pseudo-first-order Figure 6. (a) and pseudo-second-order Figure 6. (b). In the pseudo-first-order model Figure 6. (a), the plot of ln(qe–qt) versus adsorption time shows a linear regression equation of $y = –22.351x + 488.5$ with a coefficient of determination $R^2 = 0.7123$. This relatively low R^2 value indicates a poor fit between the model and the experimental data, suggesting that the pseudo-first-order kinetic model is not suitable for describing the adsorption behavior of AC-Cu(TAC)$_2$.

On the other hand, the pseudo-second-order model (Figure b) provides a better linear correlation with the regression equation $y = 4.8562x + 115.25$ and a high R^2 value of 0.9779. This strong correlation suggests that the adsorption of NH_4OH onto AC-Cu(TAC)$_2$ follows pseudo-second-order kinetics, meaning the adsorption process is likely controlled by chemisorption involving valence forces through sharing or exchange of electrons between adsorbent and adsorbate.

The interaction between ammonia (NH_3) molecules and the AC–Cu(TAC)$_2$ composite occurs mainly through the functional groups located on the surface of the TAC ligand. These functional groups play a key role in facilitating the adsorption process. Ammonia, being a neutral molecule, possesses a lone pair of electrons on its nitrogen atom. This lone pair allows NH_3 to form hydrogen bonds with polar functional groups, particularly the carboxylate group ($–COO^–$), which are present on the surface of the composite. Hydrogen bonding is one of the primary mechanisms that enables NH_3 molecules to be held onto the composite surface. In addition to hydrogen bonding, the polar nature of the NH_3 molecule also supports dipole-dipole interactions with charged or polar functional groups on the composite surface. These dipole interactions contribute further to the overall adsorption capacity and selectivity of the composite toward NH_3 molecules. Moreover, the porous structure of the composite, which is enhanced by the presence of activated

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carbon, significantly increases the surface area available for adsorption. This high surface area, along with the interconnected pore structure, provides multiple pathways for NH_3 molecules to diffuse easily toward active adsorption sites within the material. As a result, the composite not only offers physical space for adsorption but also promotes efficient mass transfer. Overall, the combined effects of hydrogen bonding, dipole-dipole interactions, and the high surface area provided by the porous structure result in an effective adsorption process. It is important to note that this adsorption mechanism relies mostly on physical and non-covalent chemical interactions, without requiring direct coordination or interaction with metal ions as active centers. This makes the AC–$Cu(TAC)_2$ composite a highly efficient and versatile adsorbent for ammonia removal. For further clarification, refer to the mechanism shown below in Figure 7.

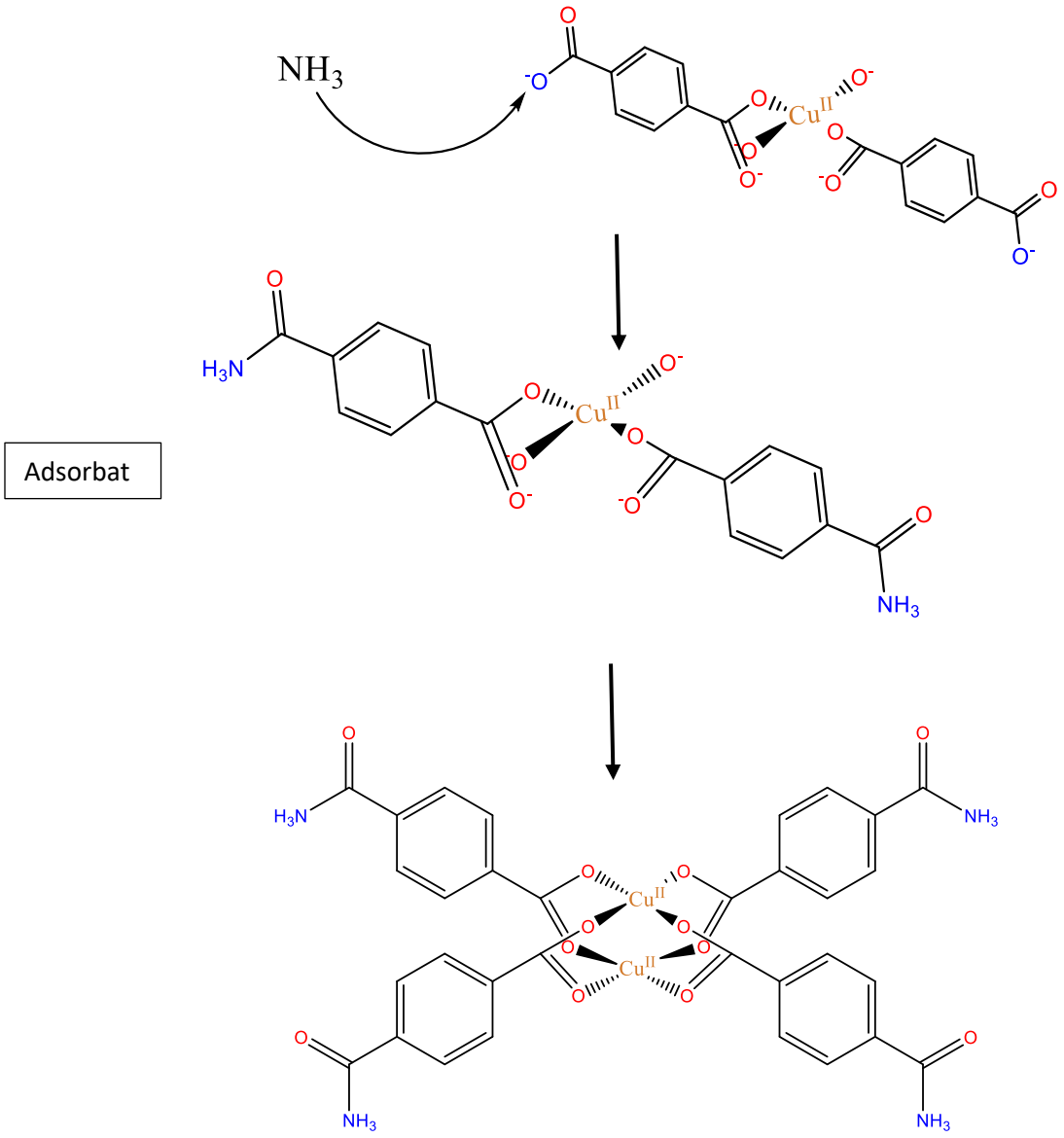

Figure 7. Adsorption Mechanism of Ammonia with AC-$Cu(TAC)_2$

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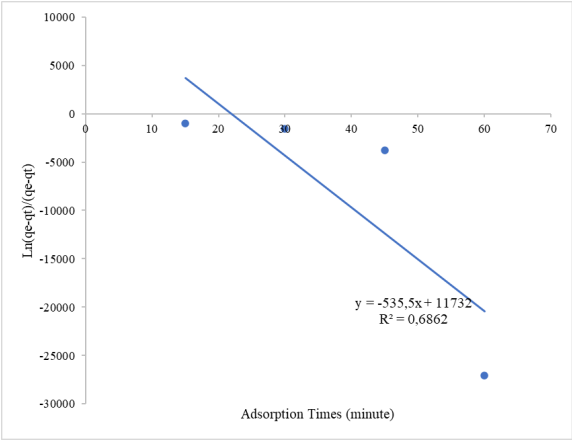
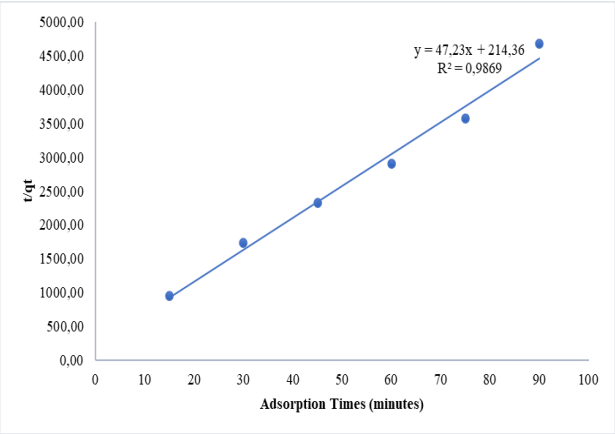
c) **Pseudo-Order Kinetic Analysis on Activated Carbon**

The adsorption kinetics of NH_4OH onto unmodified activated carbon are analyzed using pseudo-first-order and pseudo-second-order kinetic models. This analysis is conducted to determine the rate and mechanism of adsorption, as well as to identify which kinetic model provides the best fit for describing the behavior of ammonia adsorption on the activated carbon surface. The pseudo-order kinetic analysis of activated carbon can be seen in Figure 8.



(a) Pseudo First Order



(b) Pseudo Second Order

Figure 8. Pseudo-Order Kinetic Analysis on Activated Carbon
(a) Pseudo First Order (b) Pseudo Second Order

Figure 8 shows the linearized graphs of the ammonia adsorption kinetics on activated carbon, analyzed using both the pseudo-first-order model (a) and the pseudo-second-order model (b). In Figure 8. (a), which represents the pseudo-first-order model, the obtained regression equation is **y = –535.5x + 11732** with a determination coefficient **R^2 = 0.6862**. This low R^2 value indicates a poor fit of the model to the experimental data, suggesting that the pseudo-first-order model does not appropriately describe the ammonia adsorption mechanism on activated carbon. In contrast, Figure (b) presents the pseudo-second-order kinetic model with a regression equation of **y = 47.23x + 214.36** and a much higher R^2 value of **0.9869**, which is very close to 1. This strong correlation indicates that the pseudo-second-order model provides a significantly better fit to the experimental data. Based on the comparison of R^2 values from both models, it can be concluded that the adsorption of ammonia onto activated carbon is more accurately described by the pseudo-second-order kinetics. This suggests that the adsorption rate is more influenced by chemical interactions between the ammonia molecules and the active surface sites of the carbon, indicating that the mechanism tends toward **chemisorption**.

The influence of the pore structure of the adsorbent can also be discussed in terms of pore size distribution, pore stability, and pore blockage by surface functional groups. Pore size distribution and void volume are critical parameters that govern the adsorption of NH_3/NH_4^+. According to (Bashkova and Bandosz, 2014), adsorbents for NH_3/NH_4^+ generally perform better when they have well-developed pore structures, with pore sizes comparable to that of NH_3/NH_4^+ (around 3 Å). This is important because the small size of NH_3/NH_4^+ leads to very weak retention forces on conventional adsorbents like zeolites and activated carbon (Vikrant et al., 2017). Therefore, pore modification strategies have been used to enhance NH_3

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adsorption, especially in materials such as MOFs, which can be finely tuned to have microporous dimensions matching those of NH_3/NH_4^+ (Vikrant et al., 2017).

In general, the adsorption capacity of activated carbon for NH_4^+ depends on both its **physical properties** (such as surface area) and **chemical properties** (such as the presence of surface functional groups). However, research by (Yu et al,. 2016) found that specific surface area, pore volume, and microscopic structure of activated carbon are not always directly correlated with NH_4^+ adsorption capacity. Instead, (Yang et al., 2018) reported that **chemical bonding** and **electrostatic interactions** between NH_4^+ ions and surface functional groups are the main mechanisms responsible for adsorption. This implies that the role of surface functional groups is more critical than the overall surface area of the carbon itself.

Furthermore, a rapid adsorption process is desirable for materials to selectively adsorb NH_4^+ in the presence of various other pollutants. According to (Tu et al., 2019), the presence of surface functional groups and a well-developed porous structure enhances the fast adsorption of NH_4^+, which is a small-sized ion, compared to larger and more non-polar molecules.

Overall, **pseudo-second-order kinetics** consistently show better correlation in NH_4^+ adsorption processes. As stated by (Alshameri et al., 2018), the good agreement with the pseudo-second-order model suggests that the process involves three main steps: (i) diffusion of NH_4^+ from the liquid phase to the liquid-solid interface, (ii) movement of NH_4^+ from the interface to the solid surface, and (iii) diffusion of NH_4^+ into the pores of the adsorbent particles.

4.CONCLUSION

The purpose of this study was to evaluate the equilibrium and kinetic properties of three types of adsorbents: Fe-Cu modified activated carbon, the AC-$Cu(TAC)_2$ composite, and unmodified activated carbon. The findings revealed that Fe-Cu modified activated carbon achieved the highest ammonia removal efficiency, reaching up to 90%, while the highest amount of ammonia adsorbed was observed in the AC-$Cu(TAC)_2$ composite.

In terms of adsorption equilibrium, each adsorbent showed different characteristics. The Fe-Cu modified activated carbon did not fit either the Langmuir or Freundlich isotherm models, indicating a more complex adsorption behavior. In contrast, the AC-$Cu(TAC)_2$ composite fit both the Langmuir and Freundlich models exceptionally well, with a correlation coefficient (R^2) of 1, suggesting both monolayer and heterogeneous surface adsorption. Meanwhile, unmodified activated carbon followed the Langmuir isotherm model with an R^2 value of 0.9498, indicating that the adsorption occurs in a monolayer on a homogeneous surface.

Regarding kinetic behavior, the adsorbents also exhibited distinct properties. The Fe-Cu modified activated carbon followed the pseudo-first-order model with a high R^2 value of 0.9936, implying that the adsorption process was dominated by physisorption. The AC-$Cu(TAC)_2$ composite followed the pseudo-second-order model with an R^2 of 0.9779, indicating a chemisorption mechanism. Similarly, unmodified activated carbon also fit the pseudo-second-order model, with an R^2 of 0.9869, supporting the occurrence of chemisorption as the main mechanism.

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