

KINETIC AND ISOTHERM ADSORPTION MODELLING IN AMMONIUM REMOVAL PROCESS

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Abstract- Adsorption is an entrapment process that occurs when an adsorbate (NH_4^+) is in direct contact with a solid (adsorbent). Adsorbents can be obtained from plants with high cellulose, such as pineapple crown, which contains 70-80% cellulose. This study aims to measure the absorption efficiency of the adsorbent from pineapple crown waste against ammonium waste and to evaluate the kinetics and isotherm adsorption model. This research begins with the pineapple crown adsorbent preparation process, then activated using NaOH, followed by the adsorption process of ammonium ions with various conditions of time and concentration. Adsorption efficiency was obtained at 15.59% at a concentration of 50 ppm for 90 minutes. In addition, an ammonia adsorption efficiency of 71.77% at a concentration of 25 ppm for 12 hours was obtained. The model that is suitable for the adsorption process is the first-order kinetic. A suitable isotherm model is the Toth model, which describes the adsorption of heterogeneous systems for high and low concentrations.

Keywords: Adsorption, Ammonium, Isotherm, Kinetic, Pineapple Crown.

1. INTRODUCTION

Industrial development in Indonesia every year causes an increase in environmental pollution. According to Statistics Indonesia, in 2018, environmental pollution in several regions in Indonesia showed the highest environmental pollution was water pollution at 73%. Water contamination can negatively affect the community's health in the vicinity of the industrial sector. Hence, the wastewater discharged was treated before release into the environment [1]. Ammonium (NH_4^+) comes from organic nitrogen, broken down by heterotrophic organisms. Ammonium is a pollutant that causes eutrophication and the growth of toxic algae in the waters, thereby destroying aquatic ecosystems [2]. The impact of ammonium pollution on health, when exposed to ammonium in humans for 30-60 minutes at a concentration of 50 ppm, can cause respiratory problems and irritation of the nose and throat mucous membranes.

Ammonium at a concentration of 5000 ppm can cause fatal effects to death [3].

The concentration of ammonium ions in wastewater can be reduced using several methods, such as adsorption, ammonia decomposition, chemical precipitation, and ion exchange [4]. However, the easiest and most effective way to do this is by using the adsorption method. The adsorption method is an adsorption process that occurs when a fluid (liquid or gas) is bound and in direct contact with a solid (adsorbent) so that the adsorbate (substance absorbed) is bound to the pores of the adsorbent establishing a thin layer, the characteristics of the thin film layers depend on the concentration of the layer produced, namely a layer on the solid surface [5]. Adsorbents can be made by utilizing plants or organic materials such as pineapple crowns.

Based on Statistics Indonesia data for 2019, pineapple production in the South Sulawesi region was 28.975 quintals. Pineapple crowns (*Ananas comosus*) have not been widely used by the public; so far, they have become waste. Meanwhile, the consumption of pineapples in the community is increasing, causing the accumulation of pineapple waste. One pineapple contains about 25-35% of the pineapple's total weight. Pineapple crown contains several components, such as 70-80% cellulose, 5-12% lignin, and hemicellulose, which contains carbonyl, carboxyl, and hydroxyl functional groups which can be used as precursors in the manufacture of activated carbon [6, 7]. Pineapple crown has been characterized as activated carbon using several activators such as H_3PO_4 [6,14]. In addition, activated carbon characterization was also carried out by comparing the activators NaOH, H_3PO_4 , and the surfactant Sodium Dodecylbenzene Sulfonate (SDS) [8]. Based on this, activated carbon produced from pineapple crown has proper characteristics as a bio adsorbent. Meanwhile, efforts to reduce the concentration of ammonium in wastewater have been studied using activated carbon as an adsorbent modified by the surfactant Sodium Dodecyl Sulfate (SDS), which shows an ammonium removal rate of 82% and the adsorption kinetics according to this adsorption process are pseudo-second-order kinetics and Freundlich isotherm [3].

However, using SDS as an activator has a relatively higher price than other activators, such as NaOH, which can produce an area of 338.92 m²/g [7]. So it is necessary to study the ability to absorb ammonium using activated carbon with a cheaper activator. Besides that, the activated carbon used comes from pineapple crown waste. This study aimed to determine the optimum capacity of bio-adsorbent of NaOH-activated pineapple crowns in adsorbing ammonium in wastewater and to evaluate the kinetics and isotherm models in the adsorption process to remove ammonium.

2. MATERIALS AND METHODS

2.1. Materials

Pineapple crown waste obtained from Local Market in Makassar, NaOH p.a Merck, NH₄Cl Merck, Nessler Reagent made of KI (Merck), HgCl (Merck) and NaOH (Merck).

2.2. Instrumentation

The UV-Vis Spectrofotometer Shimadzu 1800 was to measure the energy absorption to be converted into ammonium concentration using a standard solution curve. Forrier Transform InfraRed (FTIR) (Shimadzu, IRPrestige 21) was to identification the fungsional groups, Scanning Electron Microscopy (SEM) (Hitachi SU 3500) was to analyze the morphology of bio adsorbant

2.3. Procedure

2.3.1. Preparing of Bio Adsorbent

Before the carbonization process, the pineapple crown waste was first reduced to about 3 cm and then pyrolyzed at 4000 °C for approximately 6 hours to obtain carbon with a yield of 42.12%. The carbon produced by the pyrolysis process is crushed and filtered to a size of 100 mesh. After that, the carbon was activated using 2% NaOH with a carbon to 2% NaOH ratio of 25:200 (g/ml) for 150 minutes. Activated carbon is dried at 110 °C for 150 minutes. The dry activated carbon was stored in a desiccator for 30 minutes.

2.3.2. Adsorption Kinetics

In a reactor, the adsorption procedure was completed with a mass ratio of adsorbent to ammonium waste volume of 1:500 (g/ml). Adsorption kinetics was carried out at an ammonium concentration of 50 ppm with a stirring speed of 180 rpm at various contact times (0, 30, 45, 60, 75, and 90 minutes). Then, 10 mL of ammonium solution was pipetted and filtered for each contact time interval. The resulting filtrate was then tested for its adsorption ability of NH₄⁺ with a UV-Vis spectrometer using the Nesler method at a wavelength of 469 nm.

2.3.3. Adsorption Isotherm

The NH₄⁺ adsorption isotherm process was carried out by mixing the adsorbent into the ammonium solution in the reactor with the mass ratio of the adsorbent: the volume of waste ammonium being 1:500 (gr/mL). Adsorption isotherms were carried out at concentrations (0, 25, 50, 75, 100, and 125 ppm) with 180 rpm of stirring at room temperature for 12 hours. After that, filtering is done. The resulting filtrate was then tested for its adsorption ability of NH₄⁺ with a UV-Vis spectrometer using the Nesler method at a wavelength of 469 nm.

2.4. Data Analysis

2.4.1. Ability Adsorption

The ability of the bio adsorbent to adsorb ammonia waste were calculated using the equation:

$$\%Adsorption = \left(\frac{C_o - C_a}{C_o} \right) \times 100\% \quad (1)$$

where, C₀ initial concentration of NH₄⁺ (mg/L), and C_a is NH₄⁺ concentration following the adsorption process (mg/L).

2.4.2 Kinetics and Isotherm Studies Adsorption

Kinetic data processing adsorption were calculated following the equations:

- First Order Kinetic Model:

$$\ln C_A = k_1 t + C_{Ao} \quad (2)$$

- Second Order Kinetic Model:

$$\frac{1}{C_A} - \frac{1}{C_{Ao}} = k_1 t \quad (3)$$

- Pseudo-First Order Kinetic Model (PFO):

$$\frac{dq_t}{dt} = k_{p1} (q_e - q_t) \quad (4)$$

- Pseudo-Second Order Kinetic Model (PSO):

$$\frac{dq_t}{dt} = k_{p2} (q_e - q_t)^2 \quad (5)$$

Isotherm data processing adsorption were calculated following the equation:

- Langmuir isotherm

$$q_e = q_m \frac{K_L C_e}{1 + K_L C_e} \quad (6)$$

- Freundlich isotherm

$$q_e = K_f C_e^{1/n} \quad (7)$$

- Redlich-Peterson isotherm

$$q_e = \frac{K_{RP}}{1 + \alpha C_e^g} \quad (8)$$

- Toth isotherm

$$q_e = \frac{K_T C_e}{(\alpha T + C_e^z)^{1/z}} \quad (9)$$

- Khan isotherm

$$q_e = \frac{q_m K C_e}{(1 + K C_e)^n} \quad (10)$$

3. RESULTS AND DISCUSSION

3.1. Effect of Adsorption Time

An essential step in the adsorption process is figuring out the adsorption rate [9]. Research that has been carried out experimentally based on the effect of time as a variable with pineapple crown waste adsorbents that have been activated using NaOH can show the efficiency of ammonium adsorption as a function of time Figure 1.

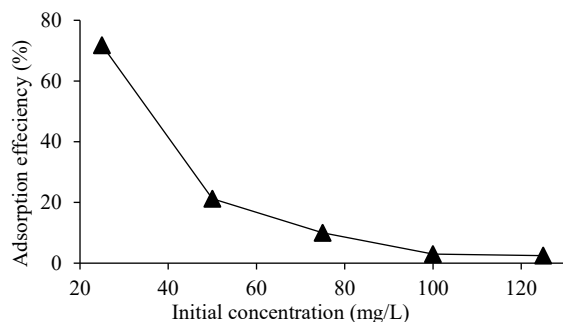


Figure 1. Ammonium removal efficiency (%) at various times

Figure 1 describes that the uptake of NH_4^+ rises as contact time increases. The optimum adsorption efficiency occurred at 90 minutes of 15.59%. The longer the adsorption time between the adsorbent and NH_4^+ , the more NH_4^+ is adsorbed by the bio adsorbent because more active sites on the adsorbent bind to NH_4^+ ions. The adsorbed NH_4^+ ion increased because the opportunity for activated carbon particles from pineapple crown waste to interact with NH_4^+ was increasing. After the optimum adsorption capacity is reached, the pineapple crown waste adsorbent is no longer able to bind or retain NH_4^+ , so NH_4^+ is released from the adsorbent, which causes a decrease in the adsorption capacity of NH_4^+ ions [10].

3.2. Initial Concentration of NH_4^+

Evaluation of effect of NH_4^+ initial concentration on adsorption efficiency is shown in Figure 2. The initial concentration of the adsorbate affects the adsorption process because it can provide effective binding strength to overcome adsorption disturbances that occur from the liquid phase (adsorbate) to the adsorbent's surface. Therefore, effect of NH_4^+ adsorbate's concentration on the adsorption process was evaluated at 0, 25, 50, 75, 100 and 125 mg/L with 0.2 gram of adsorbent during a contact time of 12 hours. Based on Figure 2, it can be seen that there was a process of decreasing the rate of removal of NH_4^+ along with an increase in the initial concentration

from 71.77% to 2.97%. Previous studies stated that increasing the initial concentration of the adsorbate could overcome the adsorption disturbance and increase the percentage of adsorption. However, in this study, there was decrease due to factors that affected the adsorption process between NH_4^+ ions in the liquid phase and the solid phase adsorbate [9].

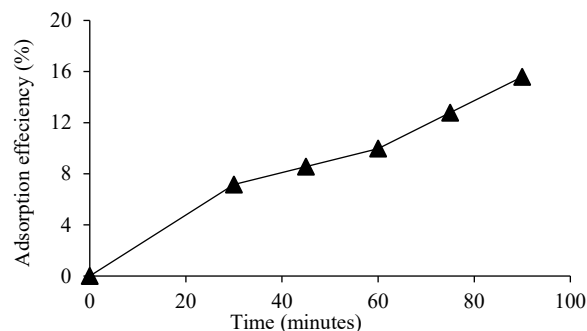


Figure 2. Ammonium removal efficiency (%) at various initial concentrations of NH_4^+

Because there were insufficient active sites on the adsorbent when the NH_4^+ concentration was high, the adsorption efficiency reduced as the initial NH_4^+ concentration increased. In this case, the mass of the adsorbent used is relatively low. In contrast, the concentration is relatively high, causing the adsorbent to saturate quickly, and the adsorption process cannot be carried out optimally [11]. Figure 2 shows the optimum adsorption percentage at 25 ppm NH_4^+ concentration. Under these optimum conditions, a maximum interaction process occurs between NH_4^+ ions and the active site of the adsorbent [12]. Based on these data it can be seen that the pineapple crown waste adsorbent with NaOH activator can adsorb NH_4^+ waste at a low concentration of 25 ppm.

3.3. Determination of Adsorption Kinetics Model

Adsorption kinetics describes the rate, which includes adsorption time and rate. The rate of adsorption that occurs on the adsorbent to the adsorbate is expressed in the adsorption rate studies which can be done by predicting the adsorption kinetics modeling. The kinetic model is used to ascertain the adsorption process's rate and kinetic analysis also helps to explore the control mechanism in the adsorption process. The physical or chemical characteristics of the adsorbent being utilized determine the adsorption mechanism and the process of transporting molecules during adsorption [13, 14].

Table 1. The Parameter Values of Adsorption Kinetics Model

Kinetic Models	Equation	SSE	k	Unit
First Order (FO)	$\frac{-dc}{dt} = kc$	0.1278	1.8×10^{-3}	1/min
Second Order (SO)	$\frac{-dc}{dt} = kc^2$	0.1286	4.0×10^{-6}	g/mg min
Pseudo First Order (PFO)	$\frac{dq_t}{dt} = k_1 (q_e - q_t)$	0.3541	2.1×10^{-2}	1/min
Pseudo Second Order (PSO)	$\frac{dq_t}{dt} = k_2 (q_e - q_t)^2$	22.065	2.49×10^{-1}	g/mg min

Determination of the kinetic model is obtained from the equation that has been calculated by comparing the kinetic model through analysis of Sum Square error (SSE) values and the adsorption constant, which can be seen in Table 1 and Figure 3. The optimal model for the adsorption process in this study was determined via SSE analysis of various kinetic models; specifically, a first-order kinetic model with an SSE value of 0.1278 was obtained. This shows that the adsorption kinetics process occurs singly, where the adsorbent is only able to adsorb NH_4^+ the pores of the adsorbent surface. There are some NH_4^+ ions adsorbed, so that NH_4^+ ions in the liquid phase (waste solution) experience a reduction. This is evidenced by a comparison between the initial and final concentration values of first-order NH_4^+ ions [15, 16].

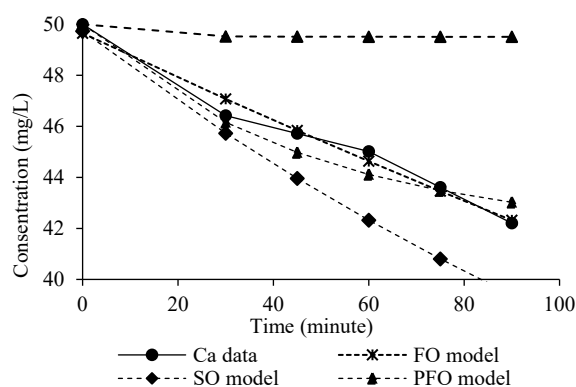


Figure 3. Adsorption kinetic test result shown along with kinetic models

Based on previous research on the adsorption of NH_4^+ with natural zeolite, the appropriate kinetic model was determined from the smallest value analysis (SSE) of 0.000154, which is a pseudo first order kinetic model with an indication that NH_4^+ adsorbed by natural zeolite is a surface adsorption.

3.4. Determination of the Isotherm Model Adsorption

The adsorption and desorption of pollutants from the adsorbent constitute the two phases of the adsorption process. The term "adsorption isotherm" refers to a condition of dynamic equilibrium characterized by equal rates of two processes. Adsorption isotherms are utilized under specific conditions to examine the relationship between equilibrium adsorption capacity and equilibrium pollutant concentrations. (fixed temperature and pH) [10]. The isotherm describes the interaction of NH_4^+ ions with the active adsorbent site and has a fundamental function for enhancing adsorption conditions. When the equilibrium of adsorption occurs, the number of NH_4^+ ions adsorbed on the surface of the adsorbent is equal to the quantity of NH_4^+ ions released [12]. Adsorption isotherm analysis was conducted to determine the right equilibrium model for research data. So that, each adsorbent and adsorbed material (adsorbate) have a different adsorption equilibrium. The determination of the adsorption isotherm model depends on R^2 degree's which is close to 1. The value of R^2 indicates the model's suitability to the data obtained.

The adsorption isotherm model used in evaluating the research data uses the Langmuir, Freundlich, Khan, Redlich-Peterson (RP) and Toth models. Adsorption isotherm for each model can be obtained by plotting the final concentration of NH_4^+ (C_e) vs adsorption capacity (q_e). Data from the evaluation of the adsorption isotherm model are shown in Table 2, while the graph of the evaluation of the data isotherm model is presented in Figure 4.

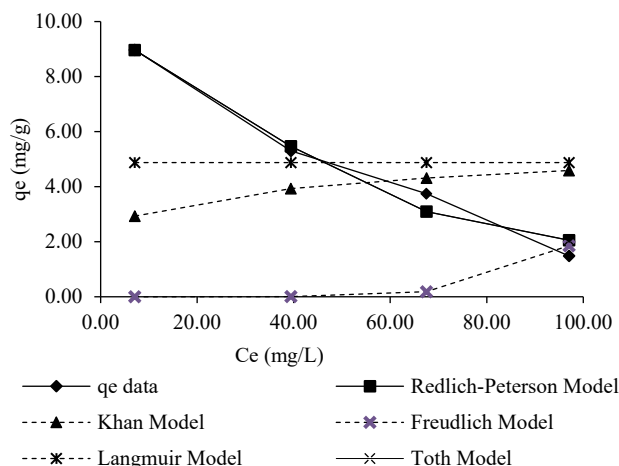


Figure 4. Adsorption isotherm test results shown along with isotherm models

Table 2 and Figure 4 show the parameter data and curve fitting of the adsorption isotherm model. Based on the parameter data, the isotherm model that corresponding to the adsorption process of NH_4^+ ions is the Redlich-Peterson and Toth model. This is evidenced by the R^2 value, which is close to 1, and the lowest SSE is the sa in both models of 0.9841 and 0.7768. The Redlich Peterson isotherm model is a three-parameter isotherm model that can be used in the adsorption process of homogeneous or heterogeneous systems. The g value in the Redlich Peterson equation indicates the isotherm model where the degree of g must be in the range of 0 and 1. When the value of $g = 0$, this model becomes the Henry isotherm model, while the degree of $g = 1$ becomes the Langmuir isotherm model [14, 15]. Referring to the data Table 2 the g value obtained by the RP isotherm model is not in the range of 0 and 1. The g value obtained is 2.173. This indicates that the Redlich Peterson model is not suitable to be applied to the NH_4^+ ion adsorption process with pineapple crown waste bio adsorbent. The Toth isotherm model is a development of the Langmuir isotherm model in the adsorption process of heterogeneous systems that can be applied at low or high concentrations. The z value in the Toth isotherm model indicates the heterogeneity of the adsorption system. If the value of $z = 1$, this model will be the Langmuir isotherm model. Meanwhile, if the z value > 1 indicates that the adsorption process has an increasingly heterogeneous system [8]. The z value obtained in Table 2 is $2.173 > 1$ indicating that the adsorption process of NH_4^+ ions with pineapple crown waste bio adsorbent occurs heterogeneously. Based on Table 2 and Figure 4 show that the best pineapple crown waste bio adsorbent used is the Toth isotherm model.

Table 2. The Parameter Values of Adsorption Isotherm Model

No.	Isotherm Models	Equality	Isotherm Parameters	R^2
1	Redlich Peterson (RP)	$q_e = \frac{K_{RP}C_e}{1 + aC_e^g}$	$K_{RP} = 1,586 \text{ L/g}$ $a = 0.003558 \text{ L/mg}, g = 2.173$ $SSE = 0.7768$	0.9841
2	Khan	$q_e = \frac{q_m b_K C_e}{(1 + b_K C_e)^{a_K}}$	$q_m = 0.8677 \text{ mg/g}$ $b_K = 177 \text{ L/g}, a_K = 0.8292$ $SSE = 48.3299$	0.0089
3	Freundlich	$q_e = k_F C_e^{1/n}$	$K_F = 4,579 \times 10^{-13} \text{ L.mg/g}$ $n = 0.1576$ $SSE = 121.3544$	0.8436
4	Langmuir	$q_e = \frac{q_m k_l C_e}{1 + k_l C_e}$	$q_m = 4.876 \text{ mg/g}$ $K_l = 83200 \text{ L/g}$ $SSE = 29.7445$	0.3900
5.	Toth	$q_e = \frac{K_T C_e}{(a_T + C_e^z)^{1/z}}$	$K_T = 969 \text{ mg/g}$ $a_T = 281.1, z = 2.173$ $SSE = 0.7768$	0.9841

The adsorption isotherm of NH_4^+ ion with pineapple crown waste adsorbent uses the Toth isotherm model. This model includes a monolayer adsorption isotherm so that the pineapple crown waste adsorbent has a one-layer active site that can adsorb NH_4^+ ions on the surface of the adsorbent. The adsorbent's active area is obtained from the activation process with NaOH activator. If the adsorbent's active area on the surface has been covered by NH_4^+ ions, then the adsorbent cannot be adsorbed. The Toth isotherm model is included in the adsorption process of heterogeneous systems, where active sites on the adsorbent

are not evenly distributed. So there are several adsorbent pores that have smaller active sites than the maximum adsorbent active site on the other side. This resulted in the adsorbed NH_4^+ ions spreading unevenly on the adsorbent surface of pineapple crown waste. However, the surface of the adsorbent can still maximally absorb NH_4^+ ions [17]. This research used pineapple crown waste adsorbent with NaOH activator to adsorb NH_4^+ adsorbate to obtain the Toth model as an appropriate isotherm model. This shows that the type of adsorbents and activators used can produce different adsorption isotherm models.

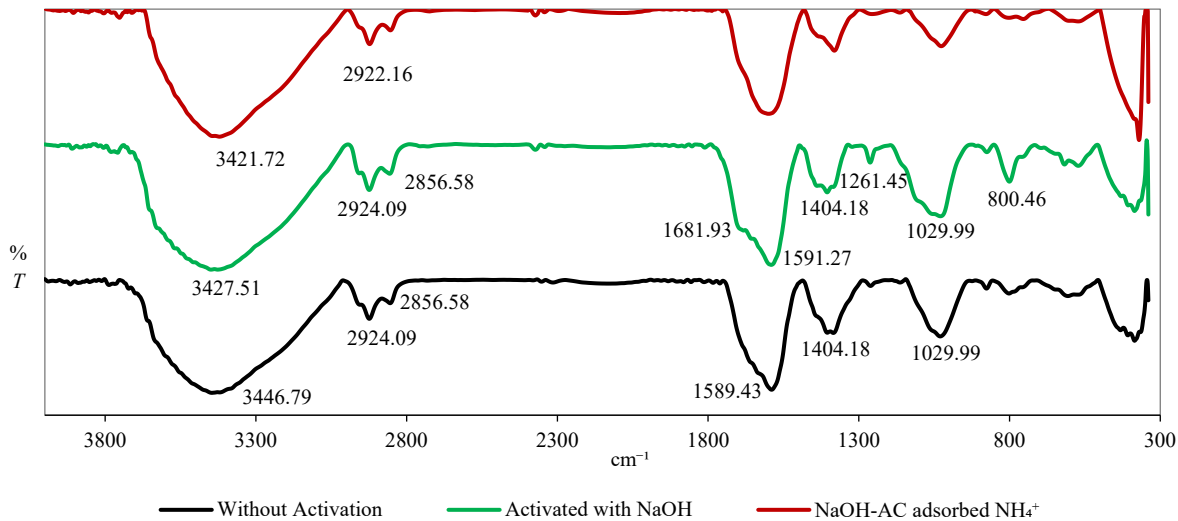


Figure 5. FTIR Spectra of Adsorbent

3.5. The Functional Groups of Adsorbent

The infrared spectrum presented in Figure 5 was observed by comparing the shifting of peaks, the disappearance of peaks and the formation of new peaks as a result of the activation and adsorption treatments. The infrared spectrum for the pineapple crown waste adsorbent before activation (AC) shows absorption wavelengths in the 3446.79 cm^{-1} area which indicates the presence of free -OH bonds, this peak also appears in the infrared spectrum for NaOH-AC and NaOH-AC adsorbed NH_4^+ . A new peak appearing in the 1681.91 cm^{-1} area indicating the presence of an aromatic $\text{C}=\text{C}$ bond in activated carbon.

This proves that there is an increase in aromatic compounds due to an activation process where aromatic compounds are an indication of the hexagonal structure of adsorbent. In addition, the appearance of a new peak also occurs in the 1261.45 cm^{-1} area which indicates a C-O-C bond together with a peak in the 3427.51 cm^{-1} area indicating the presence of a Carboxyl Acid Group bond. The infrared spectrum of NaOH-AC- NH_4^+ shows a peak shift and the appearance of a new peak at a wavelength of 1381.03 cm^{-1} which indicates a Nitrate bond is formed (NO_3^-). This peak is strengthened by the area of 1597.06 cm^{-1} which indicates the asymmetric $\text{NH}_3^+\text{-N-H}$ bond [18, 19].

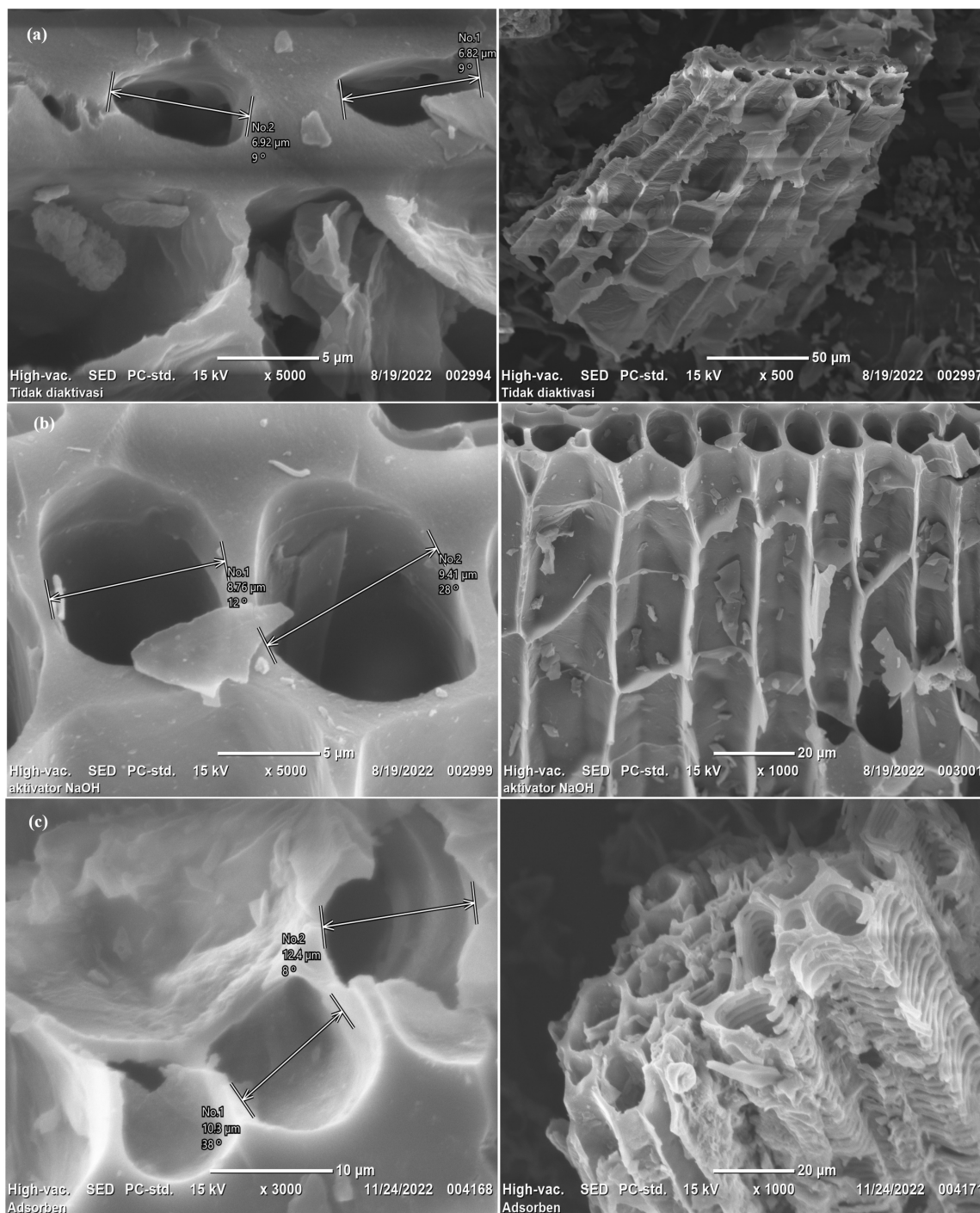


Figure 6. The Morphology of Adsorbent After the adsorption with NH_4^+ (a) Before activation; (b) Activated with NaOH; (c) After the Adsorption with NH_4^+

3.6. The Morphology Structure of Adsorbent

In order to get an idea of the microscopic structure of the bio adsorbent surface, the surface of bio adsorbent was determined by scanning electron microscopy (SEM). Based on Figures 4 and 6 there is a significant difference in the surface morphology of the adsorbent before and after activation with NaOH. At 5000x magnification, the pore diameter of the adsorbent before activation was 6.82-6.92 μm , while after activation, the pore diameter increased to 8.76-9.42 μm . This shows that the activation process can increase the pore diameter, besides that morphological

changes can also be seen at 1000x magnification where the activated adsorbent has a neater (hexagonal) pore structure. Changes in the pore structure can occur due to the diffusion of CO_2 which is the result of the reaction of NaOH and carbon into the carbon wall [7].

The difference in pore morphology was also seen significantly on the surface of the adsorbent that had gone through the ammonium adsorption process. Where, the resulting pore morphology is broken and irregular due to the presence of ammonium on the pore surface [20, 21].

4. CONCLUSIONS

Based on the results of this study, it can be concluded that the adsorption efficiency of NH_4^+ ions on the effect of time at a concentration of 50 ppm obtained the best adsorption optimum time of 90 minutes with an efficiency of 15.59%. At the same time, the adsorption efficiency of NH_4^+ ions on the effect of concentration at 12 hours of contact time obtained an optimum adsorption concentration of 25 ppm with an efficiency of 71.77%. The adsorption kinetics model suitable for the adsorption of NH_4^+ ion adsorbent from pineapple canopy waste is a first-order model with adsorption kinetics constant of 0.0016 gr/mg. While the most suitable adsorption isotherm model for adsorption of NH_4^+ ions by pineapple canopy waste adsorbent is the toth adsorption isotherm model where the parameter values obtained are k values of 969 mg/g; at is 281.1; z is 2.173.

ACKNOWLEDGEMENTS

The authors acknowledged that this work was funded and supported by Institute for Research and Development of Resources, Muslim University of Indonesia, Makassar, Indonesia with grant number 1971/A.03/LP2S-UMI/IX/2022

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