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Characterization of Active Carbon Pineapple Skin (*Ananas comosus*) as Absorbent of Heavy Metal Copper (Cu) with NaOH and NH_4OH Activators

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ABSTRACT

This research aims to evaluate the potential of pineapple peel as a raw material for making activated carbon, as well as comparing the effectiveness of NaOH and NH4OH activators in the activation process. Activated carbon is produced through a carbonization and chemical activation process, then its quality is tested based on the Indonesian National Standard (SNI) 06-3730-1995. The research results show that activated carbon from pineapple peel meets SNI standards in terms of ash content and water content, with each not exceeding the maximum limits of 10% and 15%. The yield of activated carbon activated with NH_4OH (87.5%) was higher than NaOH (79.4%). In addition, the water content of activated carbon with NH_4OH (1.07%) is lower than NaOH (1.42%). Although the iodine absorption capacity of activated carbon from pineapple peel is still below the minimum SNI standard (750 mg/g), NH_4OH shows better results in adsorption of the heavy metal Cu (0.0041 mg/g) than NaOH (0.0015 mg/g). The FTIR spectrum shows the presence of diverse functional groups, indicating complex chemical interactions. Based on the results of this research, pineapple peel has good potential as a raw material for activated carbon, with NH_4OH as a more effective activator in improving the quality and adsorption ability of activated carbon.

Keywords: Activated carbon, pineapple peel, activator

I. INTRODUCTION

Indonesia is the fifth largest pineapple producing country in the world after Thailand, Costa Rica, Brazil, and the Philippines. The food industry in Indonesia processes this fruit into new products and then produces waste that causes environmental problems. Data from the Central Statistics Agency in 2018, pineapple production in 2018 was 1,805,449 tons and pineapple skin waste was 27% of the total pineapple production (Nurhayati, 2014). Pineapple skin waste is underutilized and simply thrown away, even though the cellulose, hemicellulose and lignin compounds contained in pineapple skin have the potential to be a source of carbon (Novia and Putra, 2022).

The demand for activated carbon will continue to increase by 9% per year until 2014, activated carbon is estimated at 1.7 million tons per year (Esterlita and Herlina, 2015). Activated carbon is a porous material containing 85-95% carbon with a large surface area consisting of free carbon elements and each is covalently bonded (Permatasari et al. 2014). Rusli (2023), regarding the manufacture of activated carbon from pineapple skin as an absorbent of heavy metal pb with a comparison of NaOH and HCl activators, it states that pineapple skin has the potential as a raw material for activated carbon as indicated by parameters in accordance with the SNI 06-3730-1995 standard.

Environmental pollution due to industrial waste is a serious problem that is getting worse along with the growth of human civilization and technological advances. One type of significant pollution is pollution by heavy metal copper (Cu). Pollution by heavy metal copper can occur because industrial waste containing copper is dumped into the environment without proper handling. Research on the characterization of pineapple skin activated carbon as an absorbent of heavy metal copper (Cu) using NaOH and NH_4OH activators aims to overcome pollution problems because this activator is able to attract and bind water molecules around it, including those trapped in the pores of activated carbon. Based on this description, research was conducted by utilizing pineapple skin waste into a product with economic value, namely activated carbon as an absorbent of heavy metal Cu.

II. METHODS

A. Time and Place of Research

This research was conducted in February-March 2024 at the Chemistry Laboratory of Muhammadiyah University of Bulukumba, the Laboratory of the Environmental and Forestry Service of Bulukumba Regency, the Laboratory of the Bantaeng Environmental Service and the Chemistry Laboratory of the Faculty of Science & Technology, UIN Alauddin Makassar.

B. Tools and Materials

The tools used in this study were Atomic Absorption Spectrometry (Thermo Scientific), Fourier Transform Infrared (ThermoScietific Nicloet iS_{10}), furnace (Branstead Thermolyne), oven (Cosmos), analytical balance (Osuka), desiccator (Thermo scientific), burette, scale pipette, dropper pipette, Erlenmeyer flask (Pyrex), beaker (Pyrex), porcelain cup (Herma), stirring rod (Iwaki), mortar, knife and sieve.

The materials used in this study were pineapple skin (Ananas comosus (L) merr), sodium hydroxide (NaOH) 1 M, ammonium hydroxide (NH_4OH) 1 M, iodine (I_2) 0.1 N, starch indicator 1%, sodium thiosulfate ($Na_2S_2O_3$) 0.1 N, distilled water (H_2O), copper powder (Cu).

C. Work Procedures

1. Sampling

   A 1 kg pineapple skin sample was taken from a fruit trader in Bulukumba Regency.

2. Preparation of Pineapple Skin Samples

   The pineapple skin is washed clean with running water. Then, the pineapple skin is cut into small pieces. Next, the pineapple skin is dried using an oven at a temperature of 230°C for 1 hour.

3. Activated Carbon Production and Carbonization

   The dried pineapple skin was carbonated using a furnace at a temperature of 600°C for 5 minutes. Furthermore, sieving was carried out to reduce the size of the pineapple skin particles, then soaked in 8 mL of 1 M NaOH solution and 1 M NH_4OH solution mixed with distilled water (H_2O) for 12 hours for the activation process. After soaking, the activated carbon was dried again using a furnace at a temperature of 600°C for 5 minutes. Determination of Yield The yield of activated carbon is produced from the activation process using strong and weak base solutions. Determination of yield aims to determine the amount of activated carbon produced from the carbonization and activation processes.

4. Determination of water content

   As much as 1 gram of activated carbon was weighed and then stored in a porcelain cup whose weight had previously been known. Furthermore, the activated carbon was dried in an oven at a temperature of 105°C until it reached a constant weight. The activated carbon was cooled in a desiccator for 15 minutes and reweighed until it reached a constant weight.

5. Determination of ash content

   As much as 1 gram of activated charcoal was weighed, then put into a furnace at a temperature of 600°C for 3 hours. After that, the activated charcoal was put into a desiccator for 30 minutes and then weighed to obtain a constant weight.

6. Determination of iodine number

   As much as 0.5 grams of activated charcoal that has been weighed is put into a bottle, then added with 50 mL of 0.1 N iodine solution and the mixture is stirred using a stirring rod for 15 minutes. Furthermore, filtration is carried out to separate the filtrate. The filtered filtrate is put into an Erlenmeyer flask as much as 10 mL and titrated using 0.1 N sodium thiosulfate solution. If the yellow color has disappeared during titration, 1% starch indicator is added and then titrated again until the blue color disappears.

7. Activated carbon absorption on copper solution

   The process of activated carbon absorption in copper solution was carried out by preparing 0.3 grams of Cu powder, then mixed with 100 mL of distilled water. Furthermore, 1 gram of pineapple skin activated carbon was added to 100 mL of diluted Cu solution and stirred until the solution and activated carbon were evenly mixed, then left for 48 hours. The same process was carried out with activated carbon that had been activated using different activators, namely 1 M NaOH and NH_4OH. Analysis was carried out using an AAS instrument.

8. Characterization of activated carbon

   Characterization was carried out using FTIR testing at a wavelength of 400-4000 nm to determine the functional groups of activated carbon from pineapple skin.

D. Data Analysis

1. Determination of yield

   Level Referring to the research of Sofyan et al. (2020), the yield level of pineapple skin activated carbon can be determined using the following formula:

$$\text{Yield (\%)} = \frac{a}{b} \times 100\%$$

Information:

a = weight of carbon

b = weight of material

2. Determination of water content

Referring to Rusli (2023), to find out the water content in activated carbon, it can be calculated using the formula:

$$\text{Water content (\%)} = \frac{\text{B.Sebelum Pemanasan} - \text{B.Setelah pemanasan}}{\text{Bobot Sampel}} \times 100\%$$

3. Determination of ash content

The equation that can be used in determining ash content is as follows:

$$\text{Water content (\%)} = \frac{\text{B.Sebelum Pengabuan} - \text{B.Setelah pengabuan}}{\text{Bobot Sampel}} \times 100\%$$

4. Determination of iodine absorption capacity

The iodine absorption capacity can be calculated using the following equation:

$$\text{Iodine absorption capacity (\%)} = \frac{\frac{(V \times N\ Na2S2O3)}{N\ Iodin} \times 12{,}6}{w}$$

Information:
V = volume of $Na_2S_2O_3$ (mL)
N = normality $Na_2S_2O_3$ (N)
12.6 = amount of iodine corresponding to 1 mL of 1 N $Na_2S_2O_3$ solution
w = sample mass (grams)

III. RESULTS AND DISCUSSION

A. Activated Carbon

Carbon active Which produced from skin pineapple with activator NaOH and NH_4OH show difference physical, where activated carbon activated with NaOH appears rougher compared to that activated with NH_4OH which appears smoother.

(a) (b)

Figure 1. (a) Carbon active activator NaOH, (b) Carbon active activator NH_4OH

The differences in the characteristics of activated carbon produced from activation using NaOH and NH_4OH can be explained by the nature of each activator. NaOH, as a strong base, functions more aggressively in the activation process. carbon. Activity from NaOH influence material standard with method more aggressive, resulting in an activated carbon structure that has larger pores. This process significantly increases the internal surface area of activated carbon, making it more effective in adsorbing small molecules and increasing the absorption capacity (Sadiyah et al. 2020).

On the other hand, NH_4OH, as a weak base, produces activated carbon with a finer structure and smaller pores. The activity of NH_4OH is lighter than that of NaOH. Although NH_4OH produces a structure which more fine, This often time leading on level water which more lower and higher ash content in the final product. This shows that although NH_4OH is less effective in create an activated carbon structure with pores big, He still produce carbon active Which stable and purity good (Desiani, 2021).

B. Activated Carbon Yield

Determination of yield is a procedure used to identify the amount of activated carbon after activation. Determination of yield is done by weighing comparison weight between charcoal active before carbonized and after carbonization (Guntama et al. 2023) (Table 1).

Table 1. Yield (%) of Carbon Active

Type Activator	Weight Sample (g)	Yield (%)
NaOH	11.2	79.4
NH_4OH	11.2	87.5

Table 1 shows the yield (%) of activated carbon activated using two types of activators, namely NaOH and NH_4OH . Both initial samples have the same weight, namely 11.2 grams. The results show that the activation yield using NaOH is 79.4%, while the yield with NH_4OH is 87.5%. Yield This important in process production carbon active Because show efficiency conversion material standard become product end. From the data the, seen that activation with NH_4OH produce yield which is higher (87.5%) compared to NaOH (79.4%). NH_4OH, with yield more tall (87.5%) compared to NaOH (79.4%), showed better efficiency in producing activated carbon under the same test conditions.

C. Water Content

Determination of water content in activated carbon is an important step in evaluating its quality and characteristics. Water content describes the hygroscopic properties of activated carbon, which is the ability of carbon to absorb or interact with water. Determination The water content in this study was carried out on pineapple skin charcoal activated with NaOH and NH_4OH (Table 2).

Table 2. Level Water (%) Activated Carbon

Activator Types	Sample Weight (g)	Sample Weight Before Heating (g)	Sample Weight After Heating (g)	Water content (%)
NaOH	11.2	1.06	0.9	1.42
NH_4OH	11.2	1.02	0.9	1.07

Table 2 show data about level water (%) on carbon active activated using NaOH and NH_4OH. In the sample activated with NaOH, the initial sample weight was 11.2 grams, with a weight before heating of 1.06 grams and after heating it became 0.9 grams, so that the water content obtained was 1.42%. Meanwhile, in the sample activated with NH_4OH, the initial sample weight was 11.2 grams, the weight before heating was 1.02 grams and after heating it became 0.9 grams, resulting in a water content of 1.07%. From this data, it can be seen that activated carbon activated with NaOH own level water A little more tall compared to with Which activated using NH_4OH. This may indicate that NaOH is more effective in absorbing water or has a greater affinity for water than NH_4OH.

The Indonesian National Standard (SNI) for activated carbon, namely SNI 06-3730-1995, which stipulates the maximum water content in activated carbon. active by 15%, both samples have water content far below the maximum limit (Aisyah, 2019). This indicates that both activation method, using either NaOH or NH_4OH, produce carbon active with quality Which Good in matter level water.

D. Ash Level

Determination of ash content in activated carbon is an important process in the characterization of activated carbon which aims to determine the amount of residue remaining after the activated carbon sample is burned at high temperatures. The Indonesian National Standard (SNI) 06-3730-1995 on technical activated carbon stipulates that the maximum total ash content in activated carbon should not exceed 10% (Oko *et al.,* 2021). Ash content is an important parameter in evaluating quality charcoal active Because affect the absorption capacity and success in certain applications (Sahara *et al.,* 2017). To ensure quality charcoal active Which produced, results characterization need compared to with standards Which has set, like Which set up in SNI 06-3730-1995 about technical activated charcoal (Table 4.3).

Table 3. Level ash (%) Activated Carbon

Activator Types	Sample Weight (g)	Weight Sample Before Ashing (g)	Weight Sample After Ashing (g)	Content (%)
NaOH	11.2	1	0.45	4.91
NH_4OH	11,2	1	0,42	5,17

Table 3 shows the results of the ash content analysis of activated carbon processed using NaOH and NH_4OH activators Based on the data in the table, activated carbon processed with NaOH has an ash content of 4.91%, while Which processed with NH_4OH own level ash as big as 5.17%. Indonesian National Standard (SNI) 06-3730-1995 stipulates that the maximum total ash content in activated carbon should not exceed 10%. Thus, both types of activated carbon treated with NaOH and NH_4OH meet the SNI standard, because the ash content produced is 4.91% and 5.17% respectively, both of which are below the maximum limit of 10%.

E. Iodine Absorption Capacity

The iodine test aims to determine the ability of activated charcoal to absorb small molecular weight molecules and substances in the liquid phase. The higher the iodine number, the better the ability of activated charcoal to absorb small molecules or substances in the liquid phase (Guntama et al., 2023) (Table 4).

Table 4. Absorption Capacity Iodine Carbon Active

Activator Types	Volume $Na_2S_2O_3$ (mL)	Power Absorb Iodine (mg/g)
NaOH	2.87	393.75
NH_4OH	3.05	382.41

Analysis absorption capacity iodine is commonly used methods to evaluate ability charcoal active in absorb substances chemistry. on table above, the analysis results show that activated charcoal activated with NaOH has an iodine absorption capacity of 393.75 mg/g, while charcoal activated with NH_4OH has a lower iodine absorption capacity, which is 382.41

mg/g. This difference causes a discrepancy with the Indonesian national standard (SNI) 06-3730-1995, which stipulates that a good iodine absorption value for activated charcoal is at least 750 mg/g (Kusdarini *et al.*, 2017).

This discrepancy can be caused by several factors. One of them is the difference in the analysis method used. Although the iodometric titration method is a common method used to measure iodine absorption, the results obtained can be influenced by factors such as the accuracy of laboratory techniques, the use of quality chemicals and experimental conditions. Further research may be needed to better understand the factors that cause discrepancies between the analysis results and the SNI standard.

F. Power Absorb Carbon Active To Heavy Metal Cu

Table 5. Power Absorb Carbon Active to Copper (Cu)

Kode Sampel	Nilai Rata-Rata Absorben (mg/g)	[Cu] (mg/L)
NaOH	0.0015	0.13
NH_4OH	0.0041	0.80

The table above shows a comparison of the absorption capacity of activated carbon. with NaOH and NH_4OH to copper (Cu). Carbon active with NaOH activator has an average absorption value of 0.0015 mg/g at a Cu concentration of 0.13 mg/L. Meanwhile, activated carbon activated with NH_4OH shows the average value higher absorption, namely 0.0041 mg/g at a Cu concentration of 0.80 mg/L.

This difference shows that activated carbon is activated with NH4OH own ability absorption Which more good compared to with which is activated with NaOH. In addition That, mark absorption Which more tall on carbon active Which activated with NH_4OH shows that this chemical activation process is more effective in increasing the absorption capacity of activated carbon against heavy metals. This is important because high absorption capacity is one of the main parameters in determining quality and utility carbon active in application environment, such as the treatment of wastewater contaminated by heavy metals.

G. Group Function

The data obtained from FTIR are used to understand how the activation process affects the chemical structure of activated carbon, as well as to identify functional groups that may play a role in adsorption activity. The infrared spectra of activated charcoal can be seen in the following table:

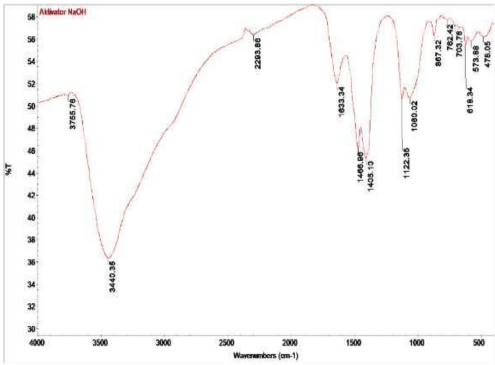
Table 6. FTIR from Carbon Active Which Activated with NaOH

No.	Puncak (cm^{-1})	Gugus Fungsi	Jenis Vibrasi	Interpretasi
1	3440.35	O- H	Stretching	Alcohol or water absorbed
2	2293.86	C≡C or C≡N	Stretching	Compound with triple bond
3	1633.34	NH or C=C	Bending or Stretching	Amide or Alkene
4	1468.98	CH_2	Bending	Chain hydrocarbon
5	1405.10	CH_3	Bending	Chain hydrocarbon

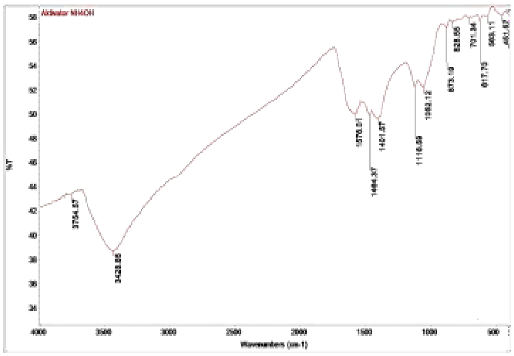
6	1122.35	CO or CO- C	Stretching	Alcohol, Esther, or Ether
7	1060.02	CO or CO- C	Stretching	Alcohol, Esther, or Ether
8	887.32	Aromatic or Aliphatic	Bending	Structure aromatic or long carbon chain
9	782.42	Aromatic or Aliphatic	Bending	Structure aromatic or long carbon chain
10	703.78	Aromatic or Aliphatic	Bending	Structure aromatic or long carbon chain
11	618.34	Aromatic or Aliphatic	Bending	Structure aromatic or long carbon chain
12	573.88	Aromatic or Aliphatic	Bending	Structure aromatic or long carbon chain

Table 7. FTIR from Carbon Active which Activated with NH4OH

No.	Peak (cm^{-1})	Group Function	Type Vibration	Interpretation
1	3428.66	O- H	Stretching	Alcohol or absorbed water
2	1578.01	NH or C=C	Bending or Stretching	Amide or Alkene
3	1464.37	CH_2	Bending	Chain hydrocarbon
4	1401.57	CH_3	Bending	Chain hydrocarbon
5	1116.59	CO or CO- C	Stretching	Alcohol, Esther, or Ether
6	1052.12	CO or CO- C	Stretching	Alcohol, Esther, or Ether
7	873.19	Aromatic or Aliphatic	Bending	Structure aromatic or chain long carbon
8	828.55	Aromatic or Aliphatic		Structure aromatic or chain long carbon
9	701.34	Aromatic or Aliphatic	Bending	Structure aromatic or chain long carbon
10	617.70	Aromatic or Aliphatic	Bending	Structure aromatic or chain long carbon
11	563.11	Aromatic or Aliphatic	Bending	Structure aromatic or chain long carbon



(a)



(b)

Picture 2. (a) FTIR NaOH activator and (b) FTIR activator NH_4OH

Based on results analysis FTIR, carbon active Which activated with NaOH and NH_4OH show various group function Which indicates complex chemical composition. Activated carbon activated with NaOH showed significant peaks at 3440.35 cm^{-1} (OH), 2293.86 cm^{-1} (C≡C or C≡N), 1633.34 cm^{-1} (NH or C=C), 1468.98 cm^{-1} and 1405.10 cm^{-1} (CH_2 and CH_3), as well as 1122.35 cm^{-1} and 1060.02 cm^{-1} (CO or COC). Besides That, there is peaks at 887.32 cm^{-1}, 782.42 cm^{-1}, 703.78 cm^{-1}, 618.34 cm^{-1} and 573.88 cm^{-1} which indicate aromatic and aliphatic bond vibrations.

Meanwhile, activated carbon activated with NH_4OH showed a peak on 3428.66 cm^{-1} (OH), 1578.01 cm^{-1} (NH or C=C), 1464.37 cm^{-1} and 1401.57 cm^{-1} (CH_2 and CH_3), as well as 1116.59 cm^{-1} and 1052.12 cm^{-1} (CO or C- OC), with peak addition on 873.19 cm^{-1}, 828.55 cm^{-1}, 701.34 cm^{-1}, 617.70 cm^{-1} and 563.11 cm^{-1}.

Similarities main between second activator is existence group hydroxyl, bond double two, chain hydrocarbons and structure aromatic or aliphatic. However, NaOH produces more alcohol groups and triple bonds, indicating its effectiveness in producing activated carbon with a more complex chemical structure than NH_4OH. This is supported by the fact that language strong can increase formation pores and expand surface area of activated carbon. This is because strong bases such as NaOH can change the carbon structure more effectively, produce more clusters functional and chemical bonds that contribute to higher absorption capacity (Kumar and Reddy, 2018).

IV. CONCLUSION

Based on the results of the research that has been conducted, the following conclusions can be drawn:

1. Pineapple peel has the potential as a raw material for activated carbon as indicated by parameters that comply with the SNI 06-3730-1995 standard.
2. Activated carbon from pineapple skin is able to absorb the heavy metal Cu, with NaOH activator (0.13 mg/g) and NH_40H activator (0.80 mg/g).
3. NH_4OH is more effective than NaOH, producing higher yield, lower water content and better Cu absorption capacity, although its iodine absorption capacity is slightly lower.

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