

Characterization of Pineapple Peel Activated Carbon (*Ananas comosus*) as an Iron (Fe) Heavy Metal Adsorbent with NaOH and NH₄OH Activators

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ABSTRACT

The purpose of this study was to investigate the effect of strong and weak base activators on the adsorption properties of activated carbon derived from pineapple peel waste. Activated carbon prepared with NaOH and NH₄OH activators exhibited different physical characteristics; NaOH-activated carbon appeared darker and had a rougher texture compared to NH₄OH-activated carbon. The NaOH-activated carbon contained a moisture content of 8.2%, an ash content of 2.22%, and an iodine adsorption capacity of 393.75%. In contrast, the NH₄OH-activated carbon contained a moisture content of 9.9%, an ash content of 2.68%, and an iodine adsorption capacity of 382.41%. Atomic absorption spectrophotometry (AAS) analysis showed an adsorption capacity of 0.17 mg/L for NaOH-activated carbon and 0.18 mg/L for NH₄OH-activated carbon. Fourier Transform Infrared (FTIR) spectroscopy revealed that NaOH-activated carbon contained O-H (hydroxyl), C=C (aromatic), C-H (alkane), C-O (alcohol), and C-H (alkene) functional groups, while NH₄OH-activated carbon exhibited O-H (hydroxyl), C=C (aromatic), C-H (alkane), and C-H (alkene) groups.

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INTRODUCTION

Indonesia is home to a variety of fruits, one of which is pineapple. The pineapple plant (*Ananas comosus* (L.) Merr) is native to Brazil, Argentina, and Paraguay. Over time, it has spread widely across tropical regions worldwide. In Indonesia, pineapples were initially grown in backyard gardens, but later evolved into large-scale crops. Pineapple is widely favored for its delicious, refreshing, and slightly acidic taste [1]. According to data from the Central Statistics Agency in 2018, Indonesia's pineapple production reached 1.805.499 tons, with production waste comprising 27% of the total pineapple output [2].

Pineapple plants are commonly found throughout Indonesia, with nearly all regions cultivating them. In 2014, pineapple production reached 1.729.603 tons [1]. According to [2], pineapple peel contains 23.39% cellulose, 42.72% hemicellulose, and 4.03% lignin. The high cellulose content holds potential for the production of activated carbon, which can be used to remove various types of pollutants [3]. Cellulose, as one of the main compounds, has been widely explored as an adsorbent for binding or reducing heavy metal content, purifying water, and adsorbing colors and odors [2]. Pineapple peel contains cellulose, hemicellulose, and lignin

compounds, all of which can serve as sources of carbon [4]. The use of pineapple waste as an activated adsorbent has been proven to reduce pollutants in wastewater by up to 99.5% [5]. Activated carbon is used in various applications, such as adsorbing dyes [6]; [7]; [8]; heavy metals like Cd and Fe [9]; [10]; [11]; and gases [12].

Heavy metal pollution, including iron (Fe), is a significant issue that has both positive and negative impacts. Fe is the fourth most abundant metal in the Earth's crust [13]. Iron (Fe), with an atomic number of 26, an atomic mass of 55.85 g/mol, and a melting point of 1536 °C, forms a brownish suspension when present in water. This suspension tends to aggregate and settle at the bottom of water bodies [14]. The increase in heavy metal pollution is primarily due to industries and factories failing to properly manage waste, resulting in negative environmental impacts on water, soil, and air. Heavy metals can enter the human body through various routes, such as consumption of food or drink contaminated by industrial waste or heavy metals, or through the use of contaminated cookware. Additionally, pollution can occur via the respiratory tract by inhaling fumes from industrial effluents [15].

The production and development of activated carbon from various raw materials, such as coconut shells, different types of wood and bamboo, coal, and other high-carbon materials, has been widely explored. Pineapple peel is another potential material for producing activated carbon [16]; [2]. The process of making activated carbon involves two main stages: carbonization and activation. The process of making activated carbon involves two main stages: carbonization and activation. The quality of activated charcoal produced is influenced by several factors, including raw materials, temperature, activators, and production methods [2]. Its use for heavy metal adsorption has been widely studied [17].

Adsorption is a surface phenomenon in which a species accumulates at the boundary between a solid and a liquid phase. Adsorption occurs due to attractive forces between molecules. In general, adsorption refers to the process of transferring specific components from the fluid phase (solution) to the surface of a solid substance that functions as an adsorbent [18]. Factors that affect adsorption include concentration, surface area, temperature, particle size, pH, and contact time. Adsorption is selective, meaning only certain solutes or solvents are adsorbed [19]. The amount of substance adsorbed depends on the concentration of the solute, and the relationship between the amount of substance adsorbed and the equilibrium concentration is known as isothermal adsorption. The purpose of this study is to investigate the effects of strong and weak alkaline activators on the adsorption capacity of activated carbon produced from pineapple peel waste [20].

RESEARCH METHODS

Research Location

This research was conducted in February-March 2024 at the Chemistry Laboratory of the Faculty of Natural Sciences, Muhammadiyah University, Bulukumba, the Laboratory of the Bulukumba Regency Environment and Forestry Agency, and the Laboratory of the Bantaeng Regency Environment Agency.

Materials and Equipment

The tools used in this study include Atomic Absorption Spectrometry (Thermo Scientific), Fourier Transform Infrared (Thermo Scientific Nicolet iS10), a furnace (Branstead Thermolyne), oven, analytical balance, desiccant, burette, volumetric pipette, dropper pipette, Erlenmeyer flask, beaker, porcelain cup, stirring rod, mortar, knife, and sieve.

The materials used in this study are pineapple peel waste (*Ananas comosus* (L.) Merr), sodium hydroxide (NaOH), ammonium hydroxide (NH₄OH) 1 M, iodine (I) 0.1 N, starch indicator 1%, sodium thiosulfate (Na₂S₂O₃) 0.1 N, and distilled water (H₂O).

Research Procedure

The procedure began with the collection of a 1 kg sample of pineapple peel from the Bangsalayya area, Kindang District, Bulukumba Regency. The sample was thoroughly washed, cut into small pieces, and then dried in an oven at 230°C for one hour. The carbonization process was carried out by heating the pineapple peel in a furnace at 600°C for five minutes. Subsequently, the carbonized charcoal was soaked in an activator solution (NaOH and NH₄OH) for 12 hours to complete the activation process, and then dried again in the furnace at the same temperature.

Data Analysis

This study employed several characterization parameters for the activated carbon. The yield was determined by calculating the ratio of the mass of activated carbon to the initial mass of the material, while the moisture and ash contents were determined based on the weight difference before and after heating. The iodine absorption was measured using the iodometric titration method, which also serves as an indicator of the surface area and microporosity of the activated carbon. The adsorption of activated carbon towards Fe metal was tested by mixing 1 gram of activated carbon with an Fe solution, allowing it to react for 48 hours, followed by analysis with Atomic Absorption Spectroscopy (AAS) to determine adsorption efficiency. The identification of functional groups in the activated carbon was conducted using Fourier Transform Infrared (FTIR) spectroscopy, aiming to identify the chemical bonds responsible for the adsorption properties of each activated carbon sample.

Characterization of activated carbon involved measuring several key parameters, including yield, moisture content, ash content, iodine absorption, and surface area. The activated carbon yield was calculated to assess the efficiency of the carbonization and activation processes using the formula.

$$Yield(\%) = \left(\frac{a}{b}\right) \times 100\% \quad (1)$$

where A represents the weight of the obtained activated carbon, and B is the initial weight of the raw material.

The moisture content of the activated carbon is determined using the oven heating method and is calculated using the following formula:

$$Water\ content\ (\%) = \left(\frac{a - b}{a}\right) \times 100\% \quad (2)$$

where a is the weight of the sample before heating (in grams), and b is the weight of the sample after heating (in grams).

The determination of ash content follows the methodology outlined in the study by Pratiwi et al. (2022). The equations used to calculate ash content are as follows:

$$Ash\ content\ (\%) = \left(\frac{ash\ weight}{activated\ carbon\ weight}\right) \times 100\% \quad (3)$$

The iodine absorption is determined using the following equation:

$$Iodine\ absorption\ capacity\ (\%) = \frac{V \times N\ Na_2S_2O_3}{N\ iodine} \times 12.6 \quad (4)$$

Description:

- V = volume of Na₂S₂O₃ (mL)
- N = normality of Na₂S₂O₃ (N)
- 12.6 = amount of iodine corresponding to 1 mL of 1 N Na₂S₂O₃ solution
- w = sample mass (grams).

The surface area determination can be calculated as follows:

$$S = \frac{xm.N.a}{M} \quad (5)$$

RESULTS AND DISCUSSION

The production of activated carbon involves several stages, namely carbonization and activation. The carbonization process is a phase that enhances the carbon content by removing non-carbon species, while the activation process involves treating the carbon with chemical solutions such as NaOH and NH₄OH to activate it.

Pineapple peel activated carbon treated with NaOH and NH₄OH activators exhibits distinct physical characteristics. These differences are evident in terms of color and texture, with NaOH-activated carbon being darker and having a rougher texture compared to NH₄OH-activated carbon. This variation is attributed to the differing properties of each activator. NaOH is a strong base with a pH of 11, while NH₄OH is a weak base with a pH range of 8-11. The pH influences the surface charge of the adsorbent, the degree of ionization, and the species absorbed during the adsorption process, thereby affecting the physical and chemical properties of the activated carbon [21].

The yield of activated carbon derived from pineapple peel is presented in Table 1 as follows:

Table 1. Yield (%) of activated carbon

Sample code	Sample weight (g)	Yield (%)
NaOH	18.8	37.34
NH ₄ OH	18.8	34.28

The yield (%) of activated carbon is presented in Table 4.1, which shows a yield of 37.34% for NaOH and 34.28% for NH₄OH. It is observed that the yield from the strong base, NaOH, is higher than that from the weak base, NH₄OH. This difference can be attributed to the loss of some activated carbon during the sifting and filtering processes, leading to variations in the weight of each yield. This finding is consistent with previous research [22].

The moisture content of the pineapple peel activated carbon is shown in Table 2 as follows:

Table 2. Moisture content of activated carbon

Sample code	Sample weight (g)	Sample weight before heating (g)	Sample weight after heating (g)	Moisture content (%)
NaOH	11.24	1.06	0.9	8.2
NH ₄ OH	11.24	1.02	0.9	9.9

The test results indicated that the moisture content of activated carbon with NaOH as the activator was 8.2%, while for NH₄OH it was 9.9%. This difference in moisture content can be attributed to the loss of particles during the soaking process, which is influenced by the distinct

properties of each activator. Despite this variation, both activators still meet the standard set by SNI 06-3703-1955, which specifies a maximum moisture content of 15% [23].

The ash content of the pineapple peel activated carbon is presented in Table 3 as follows:

Table 3. Levels of activated carbon ash

Sample code	Sample weight (g)	Sample weight before incineration (g)	Sample weight after incineration (g)	Moisture content (%)
NaOH	11.24	1	0.58	2.22
NH ₄ OH	11.24	1	0.49	2.26

The calculation results for the ash content (%) in activated carbon derived from pineapple peel showed values of 2.22% for NaOH and 2.26% for NH₄OH. These results are considered good and comply with the SNI standard, which specifies a maximum of 10%. According to Rusli's research (2023), the difference in ash content for each activator is attributed to variations in the functional groups of the activated carbon [23].

The calculation for determining the iodine content is as follows:

$$\begin{aligned} \text{Iodine absorption Capacity (\%)} \text{ NaOH} &= \frac{3.125 \times 1 \text{ N} \times}{0.1 \text{ N}} \times 12.6 \times 1 \\ &= 31.25 \times 12.6 \times 1 \\ &= 393.75 \% \end{aligned}$$

The calculation of iodine absorption capacity in NH₄OH is as follows:

$$\begin{aligned} \text{Iodin (\%)} \text{ NaOH} &= \frac{\left(\frac{V \times N \text{ Na}_2\text{S}_2\text{O}_3}{N_{\text{iodin}}} \right)}{0.1 \text{ N}} \times 12.6 \\ \text{Iodine absorption Capacity (\%)} \text{ NaOH} &= \frac{3.035 \times 1 \text{ N}}{0.1 \text{ N}} \times 12.6 \times 1 \\ &= 30.33 \times 12.6 \times 1 \\ &= 382.41 \% \end{aligned}$$

The results of the iodine absorption capacity calculation in activated carbon with NH₄OH and NaOH activators showed similar values. However, the second set of absorption capacity results did not comply with the SNI standard, as the absorption rate was below the required 750% mg/g.

The following are the test results for the absorption of pineapple peel activated carbon with NH₄OH and NaOH activators towards Fe metal, as determined by Atomic Absorption Spectroscopy (AAS).

Table 4. Carbon to iron (Fe) absorption

Sample code	Average absorbance value (mg/L)	[Fe] (mg/L)
NH ₄ OH	0.18	0.18
NaOH	0.17	0.17

The table above shows that activated carbon with NH₄OH activators has an average adsorption value of 0.18 (mg/L), while activated carbon with NaOH activators has an average

value of 0.17 (mg/L). The difference in concentration for each activator affects the adsorption capacity of the activated carbon. As shown in the table, the absorption capacity of NH_4OH -activated carbon is higher than that of NaOH -activated carbon. This difference is attributed to variations in surface area, pore size, molecular size, molecular polarity, chemical composition, pH, and temperature between the two activators [23]. Therefore, it can be concluded that NH_4OH activators exhibit better adsorption performance compared to NaOH activators.

According to Alauhdin's research (2021), the absorption spectrum data are shown in Table 5 as follows [24].

Table 5. Absorption regions of the infrared spectrum.

Group	Compound Type	Absorption Region (cm^{-1})
C-H	Alkana	2850-2960, 1350-1470
C-H	Alkena	3020-3080, 675-870
C-H	Aromatik	3000-3100, 675-870
$\text{C}\equiv\text{H}$	Alkuna	3300
$\text{C}=\text{C}$	Alkena	1640-1680
$\text{C}=\text{C}$	Aromatik (cincin)	1500-1600
C-O	Alkohol, eter, asam karboksilat, ester	1080-1300
$\text{C}=\text{O}$	Aldehid, keton, asam karboksilat ester	1690-1760
O-H	Alkohol, fenol (monomer)	3610-3640
O-H	Alkohol, fenol	3000-3600
O-H	Asam karboksilat	3000-3600
N-H	Amina	3310-3500
C-N	Amina	1180-1360
$-\text{NO}_2$	Nitro	1515-1560, 1345-1385

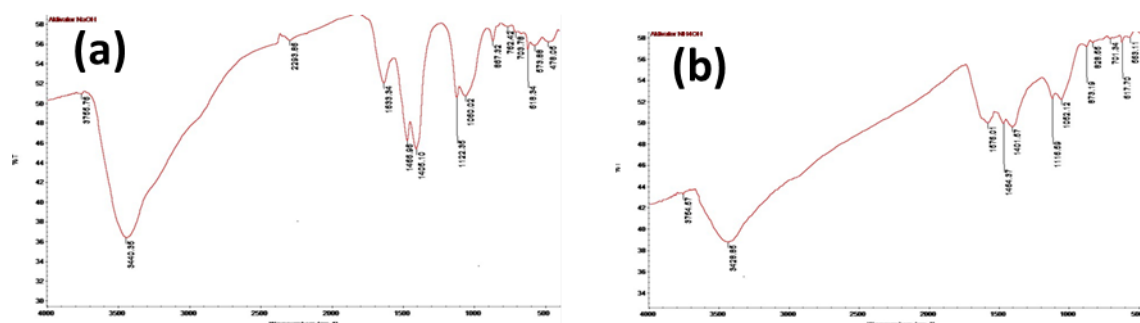


Figure 2. Infrared spectrum of activated carbon with sodium hydroxide (NaOH) activator (a) and ammonium hydroxide (NH_4OH) activator (b).

The results of FTIR analysis of pineapple peel activated carbon using NaOH as the activator showed the presence of O-H bonds (3440 cm^{-1}), aromatic C=C bonds (1633 cm^{-1}), C-H bonds ($1466\text{--}1405\text{ cm}^{-1}$), C-O (alcohol) bonds (1060 cm^{-1}), and C-H (alkene) bonds (867 cm^{-1} , 762 cm^{-1} , and 703 cm^{-1}). Meanwhile, the FTIR analysis of pineapple peel activated carbon using NH_4OH as the activator revealed the presence of O-H bonds (3428 cm^{-1}), aromatic C=C bonds (1576 cm^{-1}), C-H bonds (1464 cm^{-1} and 1401 cm^{-1}), and C-H (alkene) bonds (873 cm^{-1} , 828 cm^{-1} , and 701 cm^{-1}). These results indicate that activated carbon prepared with NaOH and NH_4OH activators contains nearly identical functional groups. Activated carbon treated with NaOH typically shows an increase in hydroxyl and oxygen groups, while carbon treated with NH_4OH tends to exhibit an increase in nitrogen-containing groups and relatively fewer hydroxyl signals. However, the presence or absence of OH groups depends on the process parameters [25]; [26].

CONCLUSION

The process of producing activated carbon from pineapple peel (*Ananas comosus*) was carried out by activating the carbon with strong and weak base activators, followed by characterization using Fourier Transform Infrared Spectroscopy (FTIR). The adsorption capacity of pineapple peel activated carbon against iron (Fe) was 0.17 mg/L for NaOH activation and 0.18 mg/L for NH_4OH activation. The iodine absorption of the activated carbon was 393.75% for NaOH and 382.41% for NH_4OH . The moisture content of activated carbon with NaOH activator was 8.2% , while with NH_4OH activator it was 9.9% . The ash content of the activated carbon with NaOH activator was 2.22% , while with NH_4OH it was 2.68% . These results comply with the SNI standard, which specifies a maximum of 10% .

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