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Optimizing Temperature and Time for Producing Coconut Shell Activated Charcoal to Reduce Free Fatty Acids in Used Cooking Oil

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ABSTRACT

Activated charcoal is a type of carbon that has a high capacity to absorb anions, cations, and molecules in both organic and inorganic compounds, whether in solutions or gases. This research aims to determine the effect of temperature variation and carbonization time on optimizing the production of activated charcoal from coconut shells as an environmentally friendly solution to reduce the acid value in used cooking oil. The activated charcoal production process in this study uses carbonization with temperature variations of 200°C, 250°C, 300°C, and 350°C, and carbonization times of 1 hour, 1.5 hours, 2 hours, 2.5 hours, and 3 hours. The best results for activated charcoal are obtained at 350°C and 3 hours, with moisture content of 0.811%, volatile matter of 5.926%, ash content of 1%, and other parameters that meet the Indonesian National Standard (SNI) for activated charcoal, such as fixed carbon content, iodine absorption capacity in accordance with SNI-06-3730-1995, and an acid value that also meets the standard (SNI 01-3741-2013).

INTRODUCTION

As one of the countries located in the tropical region, Indonesia is known as one of the largest coconut producers in the world. This is evidenced by the vast coconut plantation area in Indonesia, which reached 3.31 million hectares in 2023, with approximately 74.6% of it being productive plants (Central Statistics Agency, 2024). The potential of the coconut tree (Cocos nucifera L.) is enormous because almost every part of the plant can be utilized for various human needs. The coconut fruit, consisting of the husk, shell, flesh, and coconut water, can all be processed into industrial products without any part going to waste (Sangadji, et al., 2022).

Although coconuts have great profitable potential, their processing produces waste in the form of coconut shells, which are often considered trash despite being beneficial. Coconut shell waste, whether from processing industries or households, is generally discarded without being utilized. Although classified as organic waste, coconut shells are difficult to decompose by microorganisms due to their hard nature. Additionally, their large and heavy size causes waste accumulation when discarded (Arfadiani, et al., 2022). Given that coconut shells contain 26.6% cellulose, 29.4% lignin, 27.7% pentose, 4.2% solvent, 3.5% anhydrous uronates, 0.62% ash, 0.11% nitrogen, and 8.01% water, these characteristics indicate that coconut shells have great potential as raw material for producing activated charcoal (Suhartana, et al., 2007).

The issue of abundant waste cooking oil also needs attention. Used cooking oil is oil that has been reused several times (around four times), resulting in decreased quality. Fat in food should not contain more than 50% free fatty acids (Winarsih, et al., 2007). However, used cooking oil generally has a much higher free fatty acid content than unsaturated fat. One solution to reduce

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the fatty acids in used cooking oil is to use activated charcoal as an absorbent medium to lower the free fatty acid content (Adaawiyah, et al., 2022).

Based on previous research by (Jumiati, et al., 2024), which serves as a reference for this study titled "Purification of Used Cooking Oil Using Coconut Shell Activated Carbon with Physical Activation," the research used constant variables such as a 7-day drying time, 5-hour carbonization time, activation temperature of 900°C, 1-hour activation time, 100 Mesh sieve size, 110°C activated charcoal drying temperature, 2-hour drying time, and 5-hour carbonization time. The results showed that activated carbon from coconut shells had a moisture content of 13.2%, ash content of 2.1%, volatile matter of 17.9%, and carbon content of 80.0%, meeting the quality standards for activated charcoal as per SNI 06-3730-1995. After the purification process, the used cooking oil had a good smell and taste, normal color, moisture content of 0.06%, and free fatty acids of 0.15%, meeting the palm cooking oil quality standards according to SNI 7709:2019. The purification process also resulted in oil with a clearer color, a 60% reduction in moisture content, and a 63.34% reduction in free fatty acids, making the used cooking oil suitable for reuse. Therefore, the innovation of this research is to produce activated charcoal from coconut shells through experimental methods with direct observational analysis to reduce the acid content in used cooking oil, thereby improving the oil's quality.

RESEARCH METHOD

Preparation Stage

- Preparation of research tools and materials
- Cleaning of coconut shells from the fruit flesh
- Drying using an oven at 110°C for 1 hour.

Carbonization Stage

- Place 100 grams of coconut shells that have been oven-dried into a furnace at temperatures of 200°C, 250°C, 300°C, and 350°C, with carbonization times of 1 hour, 1.5 hours, 2 hours, 2.5 hours, and 3 hours
- After carbonization, the charcoal is cooled in a desiccator. Then, measure its moisture content, volatile matter, and carbon content.
- Determining the moisture content by weighing 1 gram of charcoal, then reheating it at 105°C for 1 hour
- Determining the volatile matter and carbon content by weighing 1 gram of charcoal, then heating it at 950°C for 15 minutes and cooling it in a desiccator
- Determining the moisture content, carbon content, ash content, and volatile matter according to the procedures in the SNI Standard No.06-3730-1995.

Activation Stage

- Grind the charcoal to a size between 20-40 Mesh, then soak it in a chemical activator NaOH with a 2% concentration, using 200 mL per 100 grams of sample for two hours
- Separate the charcoal from the activator solution by filtration, then dry it by heating at 350°C for 1 hour.

Used Cooking Oil Treatment with Activated Charcoal

- The purification process of the used cooking oil is carried out by mixing the oil with 25% activated coconut shell charcoal by mass of the used cooking oil, after first weighing the ingredients
- The materials are then mixed in a beaker using a Hot Plate Magnetic Stirrer at 100°C for 30 minutes with a rotation speed of 120 rpm
- Next, filter the oil using an Erlenmeyer flask, a glass funnel, and Whatman filter paper to obtain the purified cooking oil sample
- After that, determine the acid value by testing 10-15 grams of the purified oil with activated charcoal in an Erlenmeyer flask, adding 50 mL of 95% alcohol and three drops of phenolphthalein as an indicator

- Finally, titrate with 0.1 N NaOH until the solution turns a stable pink color, then record the volume and calculate the acid value.

RESULTS AND DISCUSSION

Moisture Content

The results of the moisture content test on activated charcoal from coconut shells showed significant variations based on the treatment of temperature and carbonization time. According to the SNI 06-3730-1995 standard, the maximum allowable moisture content for activated charcoal is 10%. In this study, the moisture content ranged from 0.811% to 1.835%, which fully meets the applicable SNI standards. The sample with a temperature treatment of 350°C for 3 hours resulted in the lowest moisture content of 0.811%, while the highest moisture content was found in the activated charcoal treated at 250°C for 1 hour, which was 1.835%. A graph of the moisture content test results against the temperature and time variables is shown in Figure 1 below.

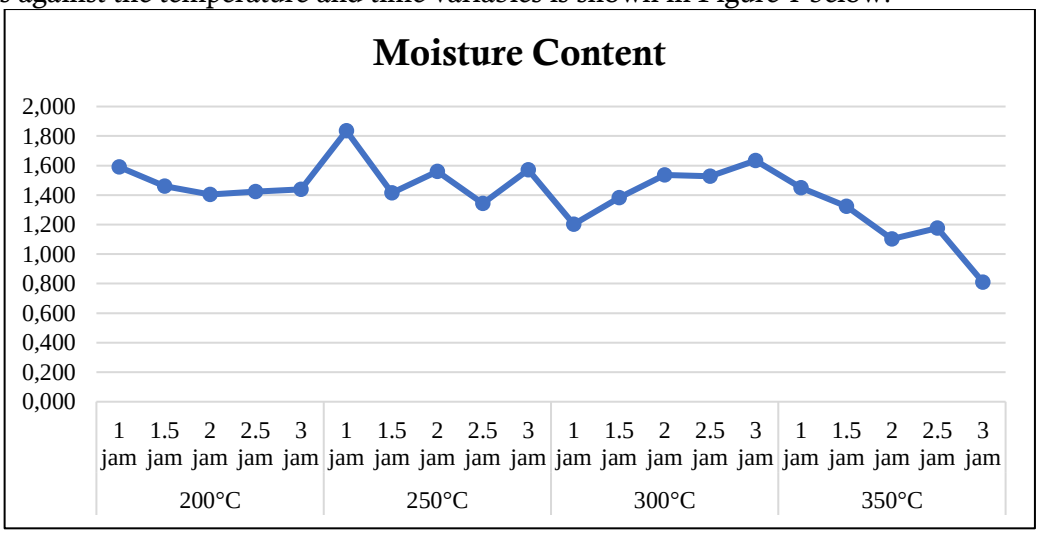

Figure 1. Graph of Moisture Content Test Results

One of the chemical properties that affects the quality of activated charcoal is its moisture content. Controlling moisture content is crucial because it affects the performance of activated charcoal as an absorbent. The lower the moisture content, the better the dimensional stability of the charcoal, indicating that most of the water in the raw material has evaporated during the activation process. Low moisture content also increases the adsorption capacity of activated charcoal. Adjusting the time and temperature during the production process can help control the moisture content, ensuring that the product meets the standards (Rahman, et al., 2018).

The research shows that the high moisture content in charcoal is caused by coconut shell charcoal particles that have not undergone complete carbonization. This results in low density, making the charcoal more prone to absorbing moisture from the air and still containing water that has not fully evaporated, leading to the high moisture content in the activated charcoal.

Volatile Matter

The determination of volatile matter content aims to identify the amount of compounds that have not evaporated during the carbonization and activation processes but will evaporate at a temperature of 950°C. The volatile matter content can be seen in the graph below (Maulana, et al., 2017). A graph of the volatile matter analysis test results against the temperature and time variables is shown in Figure 2 below.

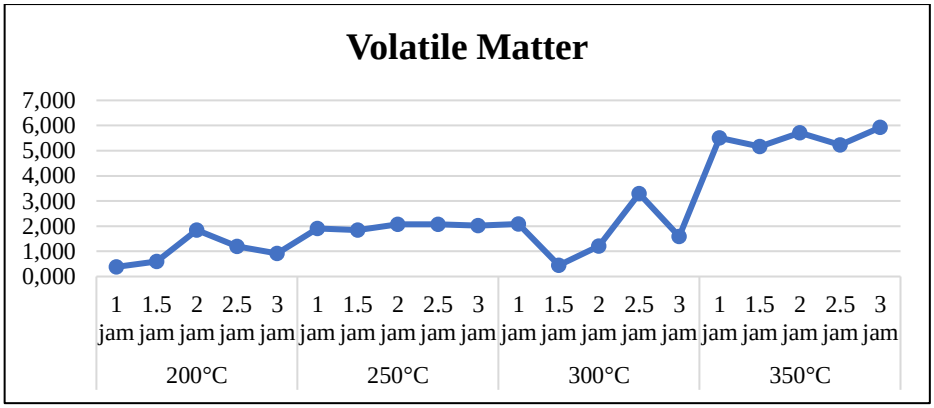

Figure 2. Graph of Volatile Matter Test Results

The graph above shows that the highest volatile matter content was achieved in the biobriquette processed at 350°C for 3 hours, with a percentage of 5.926%. Meanwhile, the lowest volatile matter content was obtained from the treatment at 300°C for 1 hour, at 0.384%. Overall, all samples met the quality standards for activated charcoal according to SNI No. 06-3730-1995, which sets the maximum limit for volatile matter content at 15%. The volatile matter analysis results represent the decomposition of compounds in activated carbon that occur due to heating during the carbonization process and are not part of the components that make up the activated carbon itself.

Ash Content

The analysis of ash content is a measurement of the weight percentage of mineral oxides such as silicon, sulfur, calcium, and other elements in small amounts in the carbon. A graph of the ash content analysis test results against the temperature and time variables is shown in Figure 3 below.

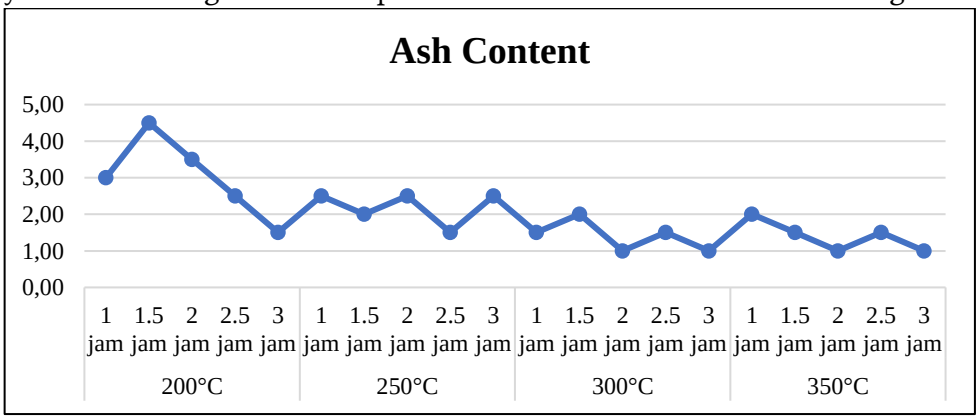

Figure 3. Graph of Ash Content Test Results

Based on the graph, the highest ash content was recorded in the activated charcoal processed at 200°C for 1.5 hours, with a percentage of 4.5%. Meanwhile, the lowest ash content was produced by the activated charcoal treated at 300°C for 3 hours, at 1.00%. From the ash content test results, only a few samples met the SNI 06-3730-1995 standard. The graph shows fluctuations in ash content. High ash content is caused by incomplete combustion, leaving behind ash residue. This is influenced by the inefficiency of the equipment used in the carbonization process, as the temperature not reaching the optimal combustion point results in an incomplete carbonization process.

Carbon Content

Bound carbon content is the amount of pure carbon contained in charcoal. The measurement of carbon content is carried out to determine the percentage of pure carbon present in the activated charcoal. In this study, the bound carbon content in the activated charcoal has met the expected quality standards in accordance with SNI No. 06-3730-1995. According to Nurhayati (1974), a

good carbonization process results in charcoal with higher carbon content. Based on the requirements of SNI-06-3730-1995, the minimum bound carbon content is 79% (Maulana, et al., 2017). A graph of the bound carbon content analysis test results against the temperature and time variables is shown in Figure 4 below.

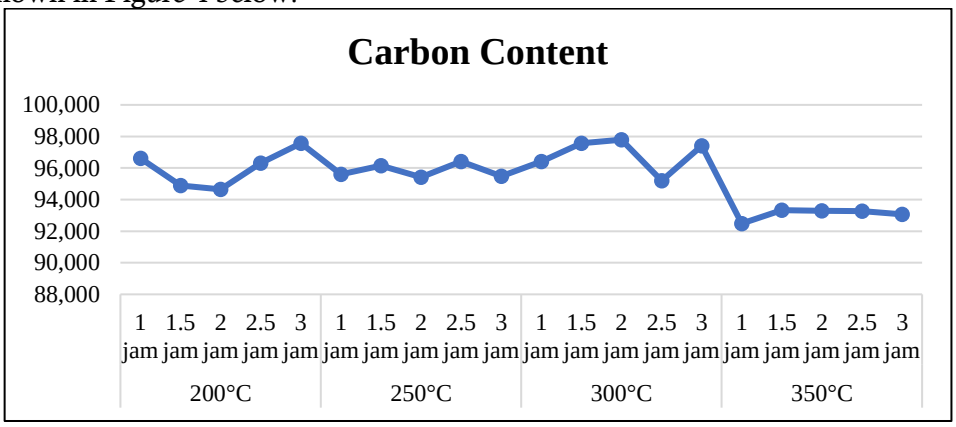

Figure 4. Graph of Bound Carbon Content Test Results

The analysis graph of bound carbon content in the activated charcoal from this study ranges from 92.485% to 97.578%. It shows that the highest bound carbon content was recorded in the activated charcoal processed at 200°C for 3 hours, with a percentage of 97.578%. Meanwhile, the lowest bound carbon content was produced by the activated charcoal treated at 350°C for 1 hour, at 92.485%. Based on the bound carbon content test results, all samples met the SNI 06-3730-1995 standard. This is related to the low ash content and volatile matter, which results in higher bound carbon content in the activated charcoal. As the ash content and volatile matter decrease, the amount of pure carbon in the activated charcoal increases. This condition may also be due to an optimal carbonization process. According to Nurhayati (1974), a well-conducted carbonization process will produce charcoal with higher carbon content.

Adsorption Capacity for I^2

The determination of the adsorption capacity of activated charcoal for I^2 aims to measure the ability of the charcoal to adsorb colored substances or impurities. The I^2 adsorption test results show that the more optimal the carbonization process, the higher the I^2 adsorption capacity. This is related to the increase in bound carbon content, especially with the use of NaOH activator. However, in this study, there were still samples of activated charcoal that did not meet the SNI-06-3730-1995 standard, which requires a minimum I^2 adsorption capacity of 750 mg/g or 75% (Maulana, et al., 2017). A graph of the activated charcoal I2 adsorption test results against temperature and time variables is shown in Figure 5 below.

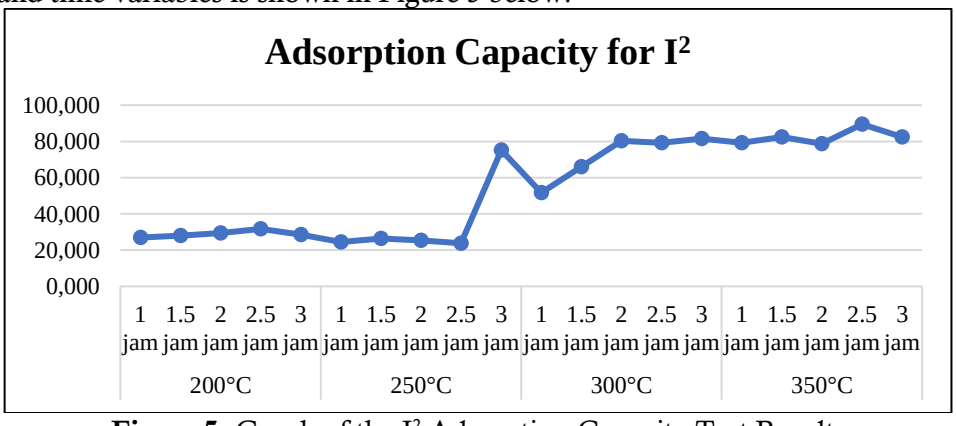

Figure 5. Graph of the I^2 Adsorption Capacity Test Results

Based on the adsorption capacity of activated charcoal against I^2, the highest adsorption capacity was recorded for activated charcoal processed at 350°C for 2.5 hours, with a percentage of 89.465%. On the other hand, the lowest adsorption capacity of activated charcoal against I^2 was

produced by activated charcoal treated at 250°C for 2.5 hours, with a value of 23.794%. The results of the I^2 adsorption test in this study indicate that some samples still do not meet the SNI-06-3730-1995 standard, which requires a minimum of 750 mg/g. This is due to the low content of volatile matter in the activated charcoal, which leads to a low I^2 adsorption capacity. Therefore, the lower the content of volatile matter, the lower the carbon content, as less carbon results in fewer pores being formed. This, in turn, affects the surface area of the activated charcoal, leading to a lower I^2 adsorption capacity.

Reduction of Acid Value in Used Cooking Oil

The reduction of acid value in used cooking oil through the adsorption process using activated coconut shell charcoal at a specified temperature will be influenced by the applied variables. The analysis graph of the reduction in acid value in used cooking oil against the temperature and time variables of activated charcoal is shown in Figure 6 below.

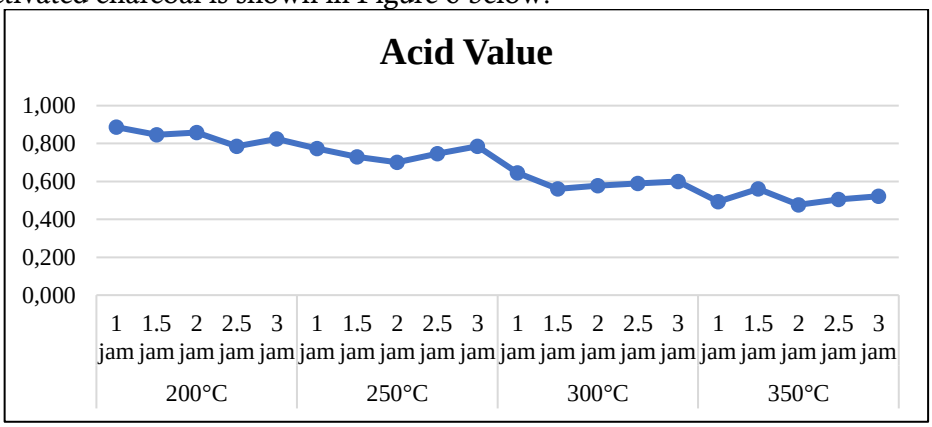

Figure 6. Graph of the Analysis Results for the Reduction of Acid Value in Used Cooking Oil

The graph of the analysis results for the reduction of acid value in used cooking oil shows that the activated carbon with the lowest test result refers to the treatment variable at a temperature of 350°C for 1 hour, with an acid value of 0.494 mgNaOH/g, which is in accordance with the applicable SNI standard of a maximum of 0.600 mgNaOH/g. Meanwhile, the highest reduction in acid value in used cooking oil is achieved by activated carbon treated under the variable of 200°C for 1 hour, with a value of 0.886 mgNaOH/g, which still does not meet the (SNI 01-3741-2013) standard for the quality of oil after refining.

The quality of activated carbon derived from coconut shell raw material, with the treatment of activator, temperature, and time during carbonization, can be analyzed through various physical and chemical property tests. The tests include the measurement of moisture content, ash content, volatile matter, bound carbon content, and iodine adsorption analysis. This evaluation process aims to assess the performance and potential use of activated carbon for reducing the acid value in used cooking oil, while also ensuring that the product meets the established quality standards for activated carbon.

Based on previous research (Jumiati, et al., 2024), which serves as a reference for this study titled "Purification of Used Cooking Oil Using Coconut Shell Activated Carbon with Physical Activation," the study used fixed variables such as a drying time of 7 days, a carbonization time of 5 hours, an activation temperature of 900°C, an activation time of 1 hour, a 100 Mesh sieve size, an active carbon drying temperature of 110°C, a drying time of 2 hours, and a carbonization time of 5 hours. The results showed that coconut shell activated carbon had a moisture content of 13.2%, an ash content of 2.1%, a volatile matter content of 17.9%, and a carbon content of 80.0%, meeting the activated carbon quality standards of SNI 06-3730-1995. After the purification process, the used cooking oil had a good smell and taste, normal color, a moisture content of 0.06%, and a free fatty acid content of 0.15%, meeting the palm oil quality standards based on SNI 7709:2019.

FORMULA PRESENTATION

Moisture Content

The determination of moisture content is to measure how much water can be evaporated so that the water bound to the activated carbon does not clog its pores. The procedure for determining moisture content follows the Indonesian National Standard (SNI) 06–3730-1995 regarding the quality requirements and testing of activated carbon. A 1-gram sample of charcoal is dried in an oven at approximately 110°C for 2 hours until its weight stabilizes. After that, the sample is cooled in a desiccator until its weight remains constant, and the moisture content is calculated as a percentage (%). The moisture content is calculated using equation (1):

$$\text{Moisture Content (\%)} = \frac{\text{sample examp - sample weight}}{\text{sample examp}} \times 100\% \quad (1)$$

Ash content

Ash content is the residue left after combustion that no longer contains elements or carbon values. The procedure for determining ash content follows the Indonesian National Standard (SNI) 06–3730-1995 regarding the quality requirements and testing of activated carbon. A crucible containing the sample with its moisture and volatile matter content already determined is used to measure the ash content. The crucible is gradually heated from room temperature to 900°C in a furnace for 2 hours. After that, the crucible is cooled in a desiccator until its weight stabilizes, and then it is weighed. The ash content is calculated using equation (2):

$$\text{Ash Content (\%)} = \frac{\text{ash weight (g)}}{\text{sample weight (g)}} \times 100\% \quad (2)$$

Volatile Matter

The determination of volatile matter content is intended to measure how much material or compound has not evaporated during the carbonization and activation processes. The procedure for determining volatile matter content refers to the Indonesian National Standard (SNI) 06–3730-1995 regarding the quality requirements and testing of activated carbon. A porcelain crucible containing the sample from the moisture determination is sealed and tied with nichrome wire. The crucible is then placed in an electric furnace at 950°C for 6 minutes. Prior to this, pre-heating is done on the flat part for 2 minutes and on the base of the furnace for 3 minutes. After the evaporation process is completed, the crucible is cooled in a desiccator until its weight stabilizes, then it is weighed. The volatile matter content is calculated using equation (3):

$$\text{Volatile Matter (\%)} = \frac{\text{sample examp - sample weight}}{\text{sample examp}} \times 100\% \quad (3)$$

Carbon Content

The procedure for determining the bound carbon content follows the Indonesian National Standard (SNI) 06–3730-1995 regarding the quality requirements and testing of activated carbon. Bound carbon is the portion of carbon stored in the material, excluding the components of moisture, volatile matter, and ash. The carbon content is calculated using equation (4):

$$\text{Carbon Content (\%)} = 100\% - (\text{Volatile Matter} + \text{Ash Content})\% \quad (4)$$

Adsorption Capacity for I^2

The procedure to determine the iodine adsorption follows the Indonesian National Standard (SNI) 06–3730-1995 regarding the quality requirements and testing of activated carbon. Weigh 1 g of activated carbon and heat it at 105°C for 1 hour. Then, cool the activated carbon in a desiccator for 30 minutes, and mix it with 100 mL of 0.1 N iodine solution. Stir for 15 minutes and let it stand for another 15 minutes. The solution is then filtered, and 25 mL of the filtrate is taken for titration. The filtrate containing the residual iodine is titrated with 0.1 N sodium thiosulfate solution until a faint yellow color appears. Then, 1 mL of 1% starch solution is added as an indicator to facilitate

the observation of the endpoint, marked by the disappearance of the blue color. To obtain the adsorption value, it can be calculated using equation (5):

$$\text{Adsoption Capacity for } I^2\,(mg/g) = \frac{(V_1N_1\text{-}V_2N_2) \times 12{,}693 \times fp}{W} \quad (5)$$

Acid Value

The measurement of the free fatty acid value present in the oil. Its determination can be performed using the titration method. The acid value analysis is conducted based on SNI 3741:2013. The acid value analysis procedure begins by weighing 10-50 g of the sample into an Erlenmeyer flask, then dissolving it in 50 mL of 95% neutral alcohol. The solution is then titrated using 0.1 N NaOH with phenolphthalein (PP) as the indicator. The titration is carried out until the solution turns pink, the titration volume is recorded, and the acid value is calculated. To calculate the acid value, the following formula is used:

$$\text{Acid Value} = \frac{5{,}61 \times \text{volume NaOH} \times \text{N NaOH}}{\text{sample weight}} \quad (6)$$

CONCLUSION AND SUGGESTIONS

Conclusion

Based on the research on the production of activated carbon from coconut shells with a chemical activation process using NaOH, it can be concluded that one of the key factors in determining the quality of activated carbon is the optimal treatment of temperature and time. The chemical activation method using NaOH also influences the ability of the activated carbon in the I^2 adsorption test and the reduction of acid value in used cooking oil. The best treatment for the carbonization temperature and time in activated carbon is at 350°C for 3 hours, with results showing 0.811% moisture content, 5.926% Volatile Matter, 1% ash content, as well as other test parameters that meet the activated carbon standards according to SNI 06-3730-1995, such as bound carbon content and I^2 adsorption capacity, and finally, the acid value that also meets the standard (SNI 01-3741-2013).

Suggestions

The treatment of temperature and time during carbonization with different variations can affect the analysis results. Therefore, to achieve optimal results in the production of activated carbon using the chemical activation method, it is recommended to use higher temperatures and longer carbonization times, so that the quality of the resulting activated carbon is improved.

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