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Effect of Pre-Treatment and KOH Concentration on The Characteristics of Rice Husk Active Charcoal

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ABSTRACT

Rice husk as organic waste can be used as raw material for activated charcoal. The quality of activated charcoal is influence by the ash content in the raw material, which can inhibit the development of carbon pores and the concentration of chemical compounds during chemical activation. The purpose of this study was to determine the best treatment from pre-treatment using NaOH and KOH concentrations of 10, 20, and 30% on rice husk charcoal activation to produce activated charcoal with the best morphological and proximate characteristics. The method used in this study is an experimental laboratory approach for the characterization and activation of rice husk charcoal. The results showed that the lowest water content of activated charcoal was obtained with N30 treatment, namely non-pre-treatment with activation using 30% KOH and P30 treatment (pre-treatment and 30% KOH), namely 1.23 and 1. 47%. Activated charcoal with NaOH pre-treatment all had lower ash content compared to non-pre-treated activated charcoal and commercial charcoal. The morphological characteristics showed that the development of activated charcoal pores of rice husk with pre-treatment was better than non-pre-treatment, which was indicated by the number of pores in a certain area unit. Increasing the concentration of KOH resulted in a decrease in the water content and ash content as well as an increase in the porosity of the activated charcoal of rice husk.

INTRODUCTION

Activated charcoal is solid carbon produced through the use of lignocellulosic components in the material, then the carbon contained is activated to create reactive pores to react with the adsorbate (Altintig et al. 2015). Activated charcoal is known to be made from a variety of organic wastes such as coconut shells, peanut husks, banana peels, mangosteen shells, candlenut shells, palm kernel shells, rice husks, and so on. Different precursor raw materials can provide different adsorption power due to differences in the structure of the raw materials (Tadda et al. 2016).

The characteristics of the raw materials determine the quality of activated charcoal. Rice husk is classified as a waste that is rich in lignocellulose content as an organic component and a carbon source for the manufacture of activated charcoal, which is 72-85%, which includes lignin, cellulose, and hemicellulose (Altintig et al. 2015; Chen et al. 2013; Uddin & Rahman, 2017). According to Daffala et al. (2012), the carbon content of rice husk is known to be quite high, which is around 41.16% wt. Carbon, as the main element of activated charcoal with a porous structure, makes activated charcoal from rice husks used to adsorb liquid, gas, organic, and inorganic chemical compounds (Rohmah & Redjeki, 2014; Altintig et al. 2015). The potential of rice husk charcoal as raw material for activated charcoal production is supported by the abundance of rice husk production, which reaches 20% of the total mass of rice plants (Ng et al. 2019).

The ash content of rice husks is known to be higher than other basic materials such as wood, bagasse, and coconut shells, which is around 3.4-17% (Menya et al. 2018a; Daffala et al. 2012; Bushra & Remya, 2020). Masoud et al. (2012) reported that most of the ash content in rice husks is silica (SiO2) which is 94.5-96.34% of the ash content. The high mineral ash content in activated carbon precursor material is reported to be able to inhibit the development of carbon pores. The formation of pores that are not optimal will have an impact on decreasing the adsorption capacity and mechanical strength of the activated carbon produced (Yeganeh et al. 2006).

Pre-treatment is a preliminary process using alkaline or acid solutions whose purpose is to reduce inorganic compounds such as silica as mineral ash and carbonates to obtain activated carbon with high purity (Daffala et al. 2020). This pre-treatment is also known as the leaching process. This process is reported to be able to reduce ash content up to 92-98% (mass) (Menya et al. 2018b). The release of silica, in addition to increasing the purity of the resulting carbon, also causes the formation of new pores (Hieu et al. 2015; Li et al. 2015). Li et al. (2015) in their study showed that the removal of silica by alkaline treatment using NaOH led to an increase in the formation of pores and an expansion of the carbon surface. In the process, the removal of silica through the formation of Na_2SiO_3 is very easy because it is soluble in water so it can be removed from the material through water washing (Menya et al. 2018).

The activation process is also one of the determinants of the quality of the activated charcoal produced. The carbon activation process is the stage of developing charcoal pores to produce a porous solid (Ma et al. 2017). Activation of carbon removes tar resulting from the carbonization process, frees atoms, water, gases, and hydrogen from the surface that covers the pores causing the opening of the pores (Haryono et al. 2012; Dewi et al. 2020). Chemical activation has been widely applied to the manufacture of activated charcoal. Chemicals as dehydrating agents will work to inhibit the formation of volatile components such as tar to produce porosity effectively, which also increases the surface area and high adsorption capacity compared to physical activation (Shrestha et al. 2019; Alam et al. 2020). Chemical activation of carbon can be done using various types of chemicals, for example, KOH. The application of KOH on carbon activation by Linares-Solano et al. (2012) showed that the surface area and the formation of the carbon pores formed were influenced by the concentration of KOH.

Utilization of rice husk as the activated charcoal adsorbent can be a solution for processing rice husk waste. This study aims to determine the best treatment from pre-treatment using NaOH and KOH concentrations of 10, 20, and 30% on rice husk charcoal activation to produce activated charcoal with the best morphological and proximate characteristics.

Previous research has shown that activated charcoal can be made from various organic wastes such as coconut shells, peanut husks, banana skins, hazelnut shells, and rice husks, with characteristics varying depending on the raw material and activation method used Tadda et al.. Several studies have revealed that the carbon content in rice husks is quite high, at around 41.16% by weight, making it a potential raw material for activated charcoal Daffala et al.. However, the main challenge in using rice husks is the high ash content, especially in the form of silica, which can inhibit carbon pore development and reduce adsorption power Menya et al.. Therefore, previous research has explored pre-treatment methods using alkaline solutions such as NaOH to reduce ash content and increase the number and size of pores in the resulting activated charcoal Hieu et al.; Li et al..

In addition to pre-treatment, the chemical activation method with potassium hydroxide (KOH) has also been widely studied to improve the quality of activated charcoal. Linares-Solano et al. showed that activation with KOH can increase surface area and carbon pore formation. Another study by (X. Ma et al., 2017)also found that the chemical activation process can remove tar and volatile substances from the carbon surface, thus increasing its porosity. In research using KOH activation, the higher the KOH concentration, the higher the resulting porosity and adsorption capacity (Wester et al., 2019). However, there are still differences in the results of various studies on the optimal KOH concentration to achieve the best characteristics of rice husk activated charcoal.

Although there has been a lot of research on making activated charcoal from rice husks using chemical activation, there are still some aspects that have not been studied in depth, especially related to the role of the combination of NaOH pre-treatment and activation with various concentrations of KOH on the characteristics of the resulting activated charcoal. Some previous studies have only focused on one method, either pre-treatment or activation, without comprehensively exploring the interaction between the two. In addition, there have not been many studies that systematically compare the effects of pre-treatment on the physical and chemical characteristics of rice husk activated charcoal when applied with different KOH concentration variations. Therefore, further research is still needed to identify the best treatment combination that produces activated charcoal with optimal characteristics.

The uniqueness of this research lies in the systematic approach in evaluating the effect of the combination of pre-treatment using NaOH and variations in KOH concentration on the characteristics of activated charcoal from rice husks. This research not only compares the results of activation with and without pre-treatment but also explores how increasing the concentration of KOH contributes to the porosity, moisture content, and ash content of the resulting activated charcoal. Thus, this study provides new insights into the relationship between pre-treatment and chemical activation that can be used to optimize the production process of rice husk-based activated charcoal, which has not been widely discussed in previous studies.

This study aims to determine the best treatment in the activation process of rice husk charcoal with a combination of NaOH pre-treatment and variations in KOH concentration to produce activated charcoal with optimal morphological and proximate characteristics. The results of this study are expected to provide benefits in developing a more effective and efficient method of producing activated charcoal based on rice husks. In practical terms, this research can be a reference for the biomass waste processing industry in producing high-quality activated charcoal products that can be used in various applications, such as liquid waste and gas adsorption, the pharmaceutical industry, and water purification. In addition, this research also contributes to the reduction of rice husk waste by utilizing it as a product with high economic value.

METHOD

The method used in this study is an experimental laboratory approach for the characterization and activation of rice husk charcoal. The research process consists of several main stages, including charcoal preparation, pre-treatment, activation using potassium hydroxide (KOH) at various concentrations, and characterization of the activated charcoal. Initially, rice husk charcoal was ground using a disk mill and sieved to a size of 100 mesh before undergoing pre-treatment with a 1M sodium hydroxide (NaOH) solution to enhance activation efficiency. The pre-treated charcoal was then soaked in KOH solutions with concentrations of 10%, 20%, and 30% for 2 hours, followed by activation through heating in a kiln at 360°C for 90 minutes. After activation, characterization was carried out using various analytical methods. Moisture content was analyzed based on a modified method from the Indonesian National Standard (SNI) No. 06-3730-1995, where the sample was heated in an oven at 105°C for 3 hours and calculated using the wet-basis moisture content formula. Ash content was determined through a similar method, involving heating in a kiln at 750°C for 6 hours, and calculated based on the mass difference before and after heating. The surface morphology and pore size were analyzed using a Zeiss Evo MA10 Scanning Electron Microscope (SEM), with sample preparation involving gold coating using a Q150R ES sputter coater before observation at various magnifications (100x, 500x, 1000x, 2000x, and 5000x). The pore size was calculated based on the average pore diameter observed in the SEM images. Overall, this method allows for detailed characterization of the activated charcoal, providing insights into its physical structure and adsorptive properties.

Materials

The materials used in this research are rice husk charcoal, sodium hydroxide (NaOH), potassium hydroxide (KOH), buffer solution pH 4 and pH 7, HNO_3 0.5 M. The tools used in this research are a disk mill machine, 100 mesh sieve machine, oven, pH meter, Scanning Electron Microscope (SEM) Zeiss Evo MA10, desiccator, furnace, hotplate heater, Atomic Absorption Spectrophotometry (AAS) Shimadzu AA-700, magnetic stirrer.

Charcoal preparation and charcoal pre-treatment

Rice husk charcoal was pulverized using a disk mill and then sieved to a size of 100 mesh. Rice husk charcoal which has been reduced in size, was then weighed each by 200 g and put into a beaker. Rice husk charcoal was mixed with 500 mL 1M NaOH solution and stirred using a magnetic stirrer for 20 minutes at room temperature. Filtered and rinsed using distilled water. Rice husk charcoal that has been given pre-treatment was then dried using an oven at a temperature of 105 °C for 6 h. After cooling, the pretreated rice husk charcoal that had been dried was then stored in a dry and tight storage area to then be activated using KOH and compared with rice husk charcoal without pre-treatment.

Rice husk charcoal activation

Potassium hydroxide (KOH) activator solution was used with various concentrations of 10%, 20%, and 30%. Then 200 g of rice husk charcoals were soaked each with KOH according to the concentration treatment for 2 h. After soaking, the rice husk charcoal was then filtered and rinsed using distilled water and 0.5M nitric acid until the pH was neutral. Then the activation was carried out using a kiln by heating it from room temperature to 360 °C and then maintained for 90 minutes. Activated charcoal that has been heated and then stored in a glass jar for storage. Rice husk-activated charcoal with pre-treatment and activated with 10, 20, and 30% KOH is symbolized by P10, P20, and P30, while charcoal without pre-treatment is symbolized by N10, N20, and N30. Rice husk-activated charcoal was characterized by SEM, moisture content, and ash content.

Characterization of rice husk-activated charcoal

Water content

The water content (wb) of the adsorbent was analyzed using a modified method of measuring the water content of SNI No. 06-3730- 1995 (BSN, 1995). Weighed as much as 1 g of activated charcoal and placed it into an aluminum/porcelain cup. Activated charcoal was then heated using an oven at 105°C for 3 hours. The activated charcoal was then cooled in a desiccator for 15 minutes. Activated charcoal samples were weighed using an analytical balance. The activated charcoal moisture content was calculated using the following wet-basis moisture content equation:

$$\text{Water content}=\frac{Ws-(Wc{+}s+Wc)}{Ws}\times 100\%$$

with:

Ws = sample weight (g)

Wc+s = weight of cup and sample after oven (g)

Wc = weight of empty cup (g)

Ash content

The ash content test refers to the modified method of SNI No. 06-3730-1995(BSN, 1995). Weigh the mass of the empty porcelain cup to be used. A total of 1 g of activated carbon was weighed and placed into a porcelain dish, and the mass of the cup containing the sample was weighed before being heated. Heated in a kiln using a temperature of 750 °C for 6 h. The sample was cooled in a desiccator for 15 minutes. The cup containing the sample that has been heated is then weighed, and the ash content was calculated using the following equation:

$$A=\frac{Wc+s-Wc}{Ws} \times 100$$

with:
Wc = mass of empty cup (g)
Ws = sample mass (g)
Wc+s = mass of cup + ash sample (g)

Surface morphology and pore size

Morphological and pore size analysis of activated carbon was carried out by characterization using the Zeiss Evo MA10 Scanning Electron Microscope (SEM). The sample to be analyzed is prepared as much as 1 g. Prepared stainless stub as a specimen. The sample was glued using carbon tape on top of the stainless stub. The sample was then coated with gold using a sputter coater Q150R ES. The coated sample is then placed into the specimen holder, and the stainless stub is tightened using the bolts on the specimen holder. The specimen holder is inserted into the chamber for later analysis. Vacuum and gun conditioning was carried out. After conditioning, the morphology and pore size analysis of the samples was carried out with magnifications of 100, 500, 1000, 2000, and 5000x.

$$\text{Calculation of Pore Size} = \frac{D1+D2}{2}$$

RESULTS AND DISCUSSION

Water content

Moisture content is one of the proximate characteristics of activated charcoal that determines the quality of activated charcoal produced. The water content of activated charcoal is defined as the amount of water in normal conditions that are physically bound to activated charcoal (Gunorubon & Kekpugile, 2018). The determination of water content is intended to determine the hygroscopicity of activated charcoal. The high affinity for water also causes activated charcoal to have a high water content (Sriatun et al. 2020). Generally, to reduce high water content in biomass, high temperatures and a long time are used in the carbonization and activation processes (Menya et al. 2018b; Janković et al. 2019). The results of measuring the moisture content (wb) of rice husk-activated charcoal and the comparison sample in this study are presented in the data below.

Table 1. The water content of rice husk-activated charcoal

No	Sample	Average moisture content (%wb)
1	Commercial Activated Charcoal	4.29 ± 0.088
2	Rice Husk Charcoal	6.35 ±1.427
3	Silica Dregs	12.66 ± 0.019
4	P10	2.26 ± 0.158
5	P20	1.96 ± 0.230
6	P30	1.47 ± 0.100
7	N10	4.47 ± 0.158
8	N20	4.39 ± 0.248
9	N30	1.23 ± 0.135

Note: P for Pre-Treatment: Soaking with 1M NaOH, followed by the percentage of KOH added
N for Non Pre-Treatment, followed by the percentage of KOH added

Based on Table 1, it can be seen that the moisture content of rice husk charcoal as raw material without treatment has moisture content of 6.35%. The trend of decreasing the water content of rice husk-activated charcoal occurred along with the increase in the concentration of KOH used. The lowest water content was obtained at 1.23% on non-pre-treatment activated charcoal with a 30% KOH concentration. This result is not much different from the water content of activated charcoal of rice husk with pre-treatment activated using 30% KOH, which has a water content of 1.47%. When compared with the comparison sample, namely rice husk charcoal (without activation), silica dregs, and commercial activated charcoal, the moisture content of rice husk-activated charcoal in this study obtained lower levels. The entire moisture content of the rice husk-activated charcoal sample has met the standards set out in SNI No. 06-3730-1995; namely, the maximum water content of powdered activated charcoal is 15%, and the water content obtained in this study is below 15%. According to Zulkania et al. (2018), the lower the water content, the higher the adsorption capacity of activated charcoal will be.

The water content in activated charcoal is expected to be contained at the lowest possible level. This decrease was attributed to the chemically activated charcoal treatment. This is because of the chemical activator compound KOH as a dehydrator which causes a decrease in the water content of activated charcoal after the activation process. In its function as an activator, the concentration of KOH is said to have an effect on reducing the water content, whereas the increase in the concentration of the activator causes the water content to decrease. This is because the chemical activator will bind to the water molecules on the charcoal, resulting in a decrease in the water content of the activated charcoal (Laos & Selan, 2016; Gunorubon & Kekpugile, 2018). In addition, according to Khalid et al. (2016), the higher the concentration of the activator, the larger the water molecules pushed out of the pores by the chemical activator. In line with this statement, the results of this study also showed a trend of decreasing the water content of activated charcoal in rice husks, both with pre-treatment and non-pre-treatment due to the increase in KOH concentration. The application of KOH in this study has provided a reduction in the moisture content of rice husk-activated charcoal compared to the precursor, which has a moisture content of 6.35%. The function of chemical activators in reducing water content is also proven in research by Gunorubon & Kekpugile (2018), which compared physical and chemical activation methods on periwinkle shell charcoal, and the results showed that the water content of activated charcoal produced by chemical activation with alkaline compounds was lower than that of physically activated charcoal.

Based on Table 1, it is also found that activated charcoal samples with pre-treatment tend to give lower water content of charcoal compared to activated charcoal from non-pre-treated rice husks. The tendency of the lower pre-treatment rice husk-activated charcoal moisture content could be due to the loss of inorganic compounds due to immersion in NaOH solution. Asadi et al. (2008) so that the formation of pores by the activator is maximized, and water molecules are more volatile during the activation process. Based on the results of this study, the lowest water content was found in the non-pre-treatment activated charcoal sample with 30% KOH concentration (N30) which was 1.23% compared to the pre-treated rice husk-activated charcoal (P30). This correlates with the hygroscopic nature of NaOH. The remaining mineral residues of NaOH left in the charcoal after the pre-treatment process can be inserted into the rice husk charcoal matrix, which causes the activated charcoal of rice husks as a result of pre-treatment to have the ability to absorb moisture from the surrounding environment (Alias et al. 2017). In addition, the hygroscopic power of activated charcoal is also related to the concentration of chemical compounds used. The dissolution of mineral components due to pre-treatment and the use of high concentrations of KOH causes the formation of pores to increase, thereby increasing the hygroscopic power of charcoal to water vapor in the environment (Puspitasari et al. 2021).

Ash content

Ash is defined as a non-volatile component of metal oxides, so it cannot evaporate during the carbonization process. At the time of carbonization, the combustion of organic components produces oxygen which can then trigger an oxidation reaction and produce ash (Arneli et al. 2017). The determination of ash content is intended to determine the levels of metal oxides present in activated charcoal (Kembaren et al. 2018). Ash content is an important quality parameter for activated charcoal. This refers to the influence of ash content on the surface area characteristics of the activated charcoal produced. High ash content can cause the surface area of activated charcoal to decrease due to inhibited pore development (Airport et al. 2020). In addition, the ash content also affects the adsorption power of activated charcoal through the reaction that occurs between the chemical activator compound and silica in mineral ash so that the activator is not able to activate charcoal optimally and is unable to produce activated charcoal with high adsorption power (Menya et al. 2018b). The decrease in adsorption power is also associated with the activated charcoal surface covered by ash, thereby reducing the active site for adsorption (Kim et al. 2015). Therefore, it is important to know the ash content of activated charcoal because it is correlated with the formation of pores and the adsorption capacity of activated charcoal.

To produce activated charcoal with high absorption, an alkaline solution in the leaching process or in this study called pre-treatment is known to be able to reduce the mineral ash content of rice husks. NaOH is said to be able to reduce the ash content mainly through the binding of silica components in rice husks with sodium (Menya et al. 2018b). NaOH solution has been widely used in the pre-treatment of activated carbon raw materials; it is mentioned because of its effective performance, cost and process temperature required is low, and the reaction time required is quite short (Ahmadi et al. 2016). The results of determining the ash content of rice husk-activated charcoal and comparison samples are presented in Table 2.

Based on Table 2, the ash content of rice husk charcoal as raw material for activated charcoal is 45.65%. The high content of ash as an inorganic component in carbonized raw materials is caused by the formation of residues resulting from the combustion of organic matter or biomass at high temperatures (Li et al. 2020). The ash content of rice husk charcoal is not much different from the ash content of commercial activated charcoal as comparison, which is 44.56%, both of which have not been able to meet the national standard of activated charcoal ash content. The activation treatment in this study resulted in a decrease in ash content to 19.00-31.31%. The high ash content in the comparison sample of silica dregs is in line with the shape of the sample, which is ash. Based on Table 8, all samples of activated charcoal with a particle size of 100 mesh had lower ash content than commercial activated charcoal in the form of granules. According to Fauzia & Purnama (2021), the difference in particle size of activated charcoal can give a difference in ash content. It was further stated that the low ash content due to the small size of activated charcoal particles allowed melting to occur when heated at a temperature of 650 °C.

Table2. Ash content analysis

No	Sample	Average ash content (%)
1	Commercial Activated Charcoal	44.56 ± 0.040
2	Rice Husk Charcoal	45.65 ± 0.051
3	Silica Dregs	62.50 ± 0.625
4	P10	28.52 ± 1.105
5	P20	19.25 ± 0.465
6	P30	19.00 ± 0.623
7	N10	31.31 ± 0.101

8	N20	28.53 ± 2.135
9	N30	23.46 ± 1.269

The data above shows that the ash content decreased along with the increase in KOH concentration, where the lowest ash content was obtained through pre-treatment and 30% KOH concentration (P30) which was 19.0%. This trend of decreasing ash content is similar to the research by Paryanto et al. (2019), which activates mangrove waste using various concentrations of KOH; the results show the higher the concentration of KOH used, the lower the ash content of the activated charcoal produced. The decrease in ash content due to an increase in concentration is said to occur because the more concentrated the concentration of the activator, the more the surface of the charcoal is filled with an activator which causes the ash content in the pores to be pushed out and released (Khalid et al. 2016). A similar trend was also found by Saad et al. (2020), which activates rice husk charcoal using NaOH, which reduces the ash content from 19.11% to 13.09%. Owabor & Iyaomolere (2013) stated that the trend of decreasing ash content could be caused by the low rate of charcoal burning in each impregnation ratio. According to Henning & Schäfer (1993), impregnation creates synergism between carbon and chemical activator compounds by optimizing the properties of activated carbon.

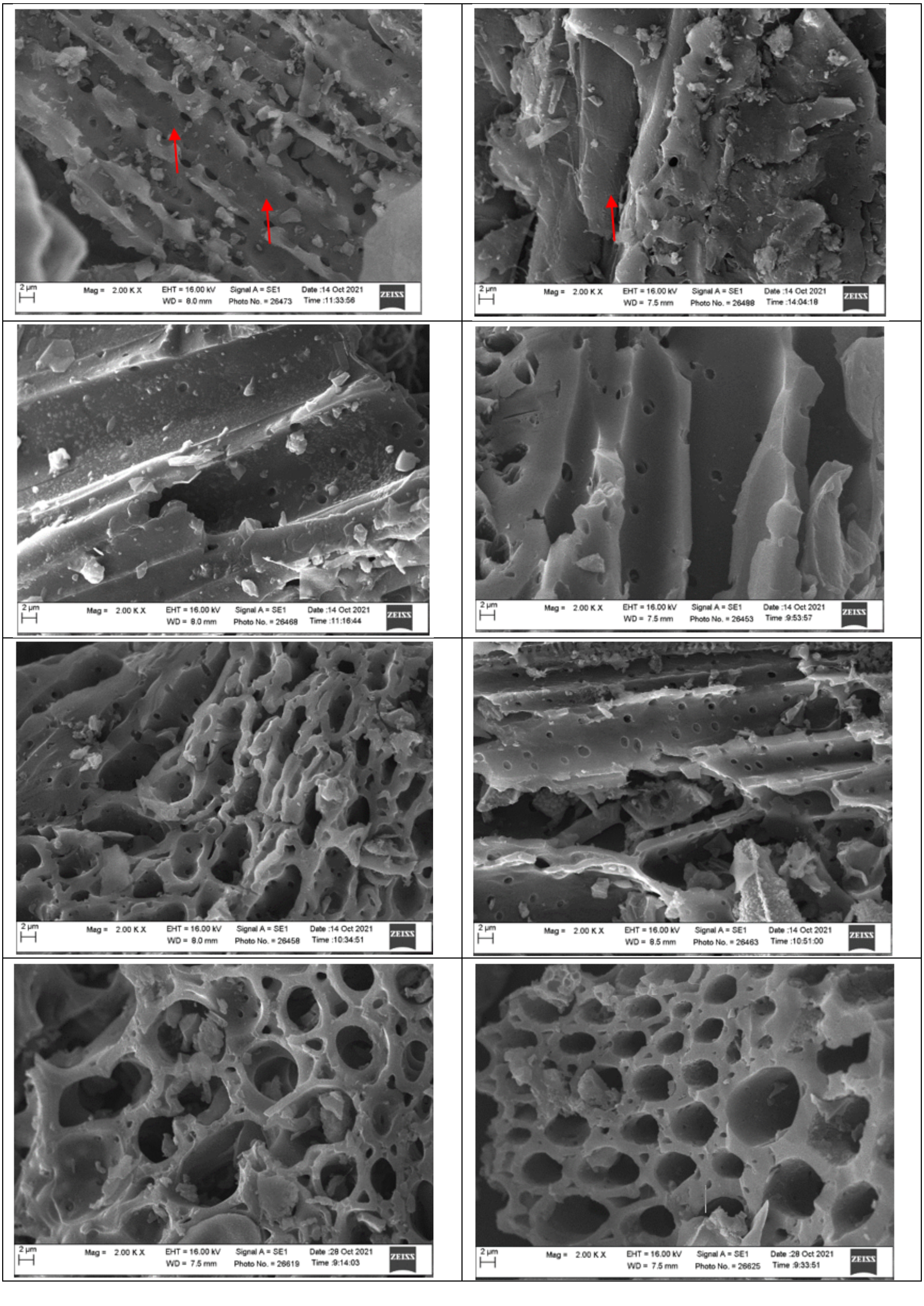
The decrease in ash content is also possible due to the washing treatment. In this study, washing treatment with distilled water was applied after pre-treatment with NaOH and washing using nitric acid and distilled water was applied after soaking the charcoal in KOH. Invention Iroba et al. (2013) showed the effect of washing after pre-treatment and chemical activation, where washing can reduce ash content up to 25-55%, depending on the ratio of impregnation between charcoal and chemical compounds NaOH. Iroba et al. (2013) explained that the washing treatment immediately after chemical treatment resulted in the loss of solid lignin and dissolved lignin. This finding is supported by another finding that applies pre-treatment with NaOH to activated charcoal from cassava peels. The results show a lower ash content due to the low lignin content in the charcoal (Kayiwa et al. 2021). In addition, research by Park et al. (2019) showed that there was a difference in the effectiveness of washing between washing with water and washing using acetic acid on charcoal after activation. His test on N_2 adsorption proved that activated charcoal which was activated using KOH and rinsed using acetic acid had a higher adsorption power than activated charcoal, which was rinsed using only water. Washing using an acid solution is further said to be able to reduce the activator residue, and ash formed from the activation process and clean the pores of the activated charcoal (Park et al. 2019). Lee et al. (2020) showed in their research that pre-treatment of activated charcoal raw materials by washing using distilled water was only able to remove water-soluble components.

Another result obtained is that the ash content of activated charcoal of rice husk with pre-treatment tends to be lower than that of activated charcoal of non-pre-treated rice husk, which is around 19.00-28.52%. This shows that pre-treatment affects the ash content of activated charcoal in rice husks. The decrease in ash content due to pre-treatment is based on the reaction of the formation of sodium silica which is then removed by washing (Menya et al. 2018a). Yang et al. (2021) added that the decrease in ash occurred after treatment with alkali. The decrease in ash content encourages the formation of pores and increases the average pore size of the carbon.

The mineral content of ash in rice husks can vary, one of which is influenced by geographical location (Benassi et al. 2015). In addition, the higher temperature and activation time can increase the ash content in activated charcoal (Bedia et al. 2018). The concentration of NaOH solution in the pre-treatment also affects the amount of reduction in the ash content of activated charcoal in rice husks. The higher the ash content in the raw material, the greater the concentration of NaOH required. The ash content of rice husk varieties depends on factors such as the source of water used to irrigate rice (Menya et al. 2018). The level of ash content reduction through pre-treatment is also influenced by the temperature and time of impregnation. The higher the temperature used can cause the ash content to increase, while the longer the impregnation time can produce a high level of reduction in the ash content because the longer the reaction time between the carbon and the pre-treatment agent (Kundu et al. 2015).

Morphological analysis

The adsorbent's morphological characterization was conducted using SEM analysis. In this study, data were obtained in the form of morphological appearance, average pore size, and the number distribution of pores in a surface area. This data is necessary to observe the effect of pre-treatment and variations in KOH concentration on the formation of carbon pores. According to Abdullah & Mohammed (2019), SEM can effectively analyze in nanometer and micrometer scales on organic and inorganic material. Compared to a light microscope, SEM is better able to see objects down to the molecular and atomic level using a much greater magnification. The working principle of SEM is to shoot an electron beam at the specimen, which will produce a signal containing information that is then detected and converted into an image as a result of the interaction between the electron beam and the atoms in the specimen (Kannan, 2018). The surface morphology and the size of the charcoal pores using SEM can be seen in Figure 1.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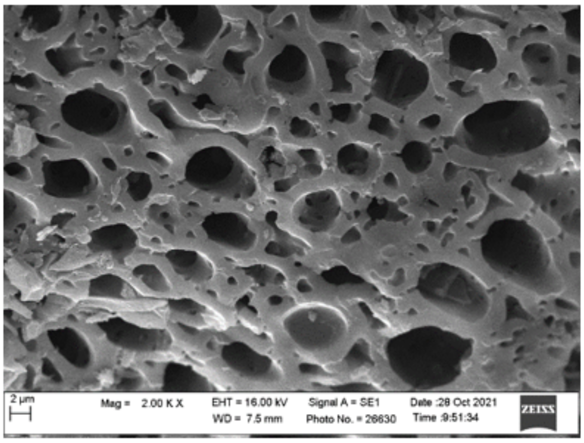

Figure 1. Morphological appearance at 2000x magnification a) Rice husk charcoal, b) Commercial charcoal, c) Silica residue, d) Activated charcoal P10, e) Activated charcoal P20, f) Activated charcoal P30, g) Activated charcoal N10, h) Activated charcoal N20, and i) Activated charcoal N30

Table 3. Average pore size & number of pores per unit area

No	Sample	Average pore size (nm)	Number of pores (within 30x30 m)
1	Commercial Activated Charcoal	1373.0	1
2	Rice Husk Charcoal	1482.5	13
3	Silica Dregs	1316.2	12
4	P10	1812.4	13
5	P20	1293.9	39
6	P30	1129.4	39
7	N10	836.0	8
8	N20	491.1	19
9	N30	627.0	28

Fig. 1(a) shows rice husk charcoal has pores formed as a result of the carbonization process, but there are still many clogged pores and a lot of impurities on the carbon surface. This is in line with Hanum et al. (2017) that carbonized charcoal still contains a lot of impurities. In addition to silica, the clogging of carbon pores can be caused by tar (Hagemann et al. 2018). According to Benassi et al. (2015), the external morphology of rice husks has an irregular shape resembling a parallel cone. It is known to be high in silica content, while the internal surface with low silica content has a smooth internal surface. In addition, Figs. 1(a), (b), and (c) show the impurities present on the carbon surface. These impurities can be in the form of hydrocarbon components, tar, or other components formed during the carbonization process. The impurity components will be oxidized and eroded by chemical activator compounds during carbon activation to support the formation and widening of carbon pores (Rampe & Tiwow, 2018).

Fig. 1(c) shows The morphological structure of the silica dregs is shown. According to Vieira et al., (2014), rice husk silica an amorphous structure. The presence of pores in the silica dregs sample is possible because the sample contains a number of lignocelluloses which then undergo combustion and chemical treatment with KOH solution, which causes the formation of pores. In addition, the commercial activated charcoal shown in Fig. 1(b) at 2000x magnification found only one open pore and the surface was filled with particles such as impurities in rice husk charcoal. The commercial activated charcoal observed was granular activated charcoal, in contrast to rice husk charcoal and rice husk-activated charcoal in this study which was made of 100 mesh size before activation treatment. This gives a high possibility of the influence of charcoal size, where the smaller charcoal particle size will have better characteristics after the activation process because the contact surface between the charcoal and the activator will be wider and more pores can be formed. According to Erdoğan et al. (2016) the smaller the particle size of the charcoal to be activated, the more optimal the interaction between the activator and the raw materials.

Figs. 1(d), (e), (f), (g), (h), and (i) show the differences from unactivated rice husk charcoal, where irregular cavities are formed, the pores look more open, the distribution of more pores, which also shows better characteristics compared to other comparison samples. According to Sirimuangjinda et al. (2013), the reaction of the adsorbent material with chemicals as activators causes the formation of an irregular cavity structure. The findings of this morphological appearance are supported by similar findings of rice husk-activated charcoal in the study by Scapin et al. (2021) which applied K_2CO_3 as a chemical activator, where the morphology of rice husk-activated charcoal showed the presence of small pores which were also found in large pores on the surface (Figs. g, h, i) and flat structures (Fig. e).

The number of pore distributions is supported by the data in Table 3, where rice husk charcoal activated using KOH tends to have a larger number of pores. In line with research results, Paryanto et al. (2019) that activated carbon using KOH gives the formation of more pores compared to unactivated carbon. Heidarinejad et al. (2020) and Hosseini et al. (2017) explained that the insertion of potassium metal from KOH into the internal structure of the carbon matrix and the release of volatile compounds during activation caused the formation of carbon porosity. An increase in porosity is also reported to occur because during the pyrolysis process, the release of volatile components causes the opening of new pores and the widening of the pores (Menya et al. 2018a). Other analysis results show that there is a correlation between the concentration of activator and the number of pores formed, where the higher the concentration of KOH, the more pores produced. This is indicated by the carbon pores of the rice husk-activated charcoal sample with 30% KOH concentration in a unit area 30x30 more than rice husk-activated charcoal with 10% KOH concentration. Similar findings were reported by Van & Thu (2019) that the results showed that the higher the concentration of alkaline activator used, the more pores formed. A similar trend was also found by Tejada et al. (2017) where the ratio of 1:4 activator solution causes more pore formation on the carbon surface than the ratio of 1:3.

Observation of the morphology of activated charcoal also obtained data on the average pore size. Calculation of the average pore size was obtained by taking 3 samples of pores at random at 5000x magnification. Measurement of each pore produces 2 sizes (D1 and D2); this is due to the size of the pores, which tend to be oval (not perfectly round), so it is necessary to take 2 diagonal pore sizes. The final pore size was obtained from the average of the 3 calculated pore sizes. Based on the average pore size, rice husk-activated charcoal has an average pore size that tends to decrease from the average pore size of rice husk charcoal. The downward trend in the average pore size also occurred in a study conducted by Khalid et al. (2016), where the application of KOH as an activator resulted in a decrease in the average pore size with increasing KOH concentration. The smaller pore size with increasing activator concentration illustrates the wider surface area of activated charcoal formed. This will have an impact on increasing the adsorption capacity of the adsorbate (Prastuti et al. 2019). The increase in pore size due to an increase in activator concentration can occur because high activator concentrations can cause an expansion reaction in the pores (Chaisit et al. 2020). In addition, from Table 10 it is also known that the average pore size of pre-treated rice husk-activated charcoal tends to be larger than that of non-pre-treated rice husk-activated charcoal at all KOH concentrations. This is due to the effect of the pre-treatment process using NaOH. According to Yang et al. (2021) and Mustaqim et al. (2021), pre-treatment using NaOH gives a tendency to form pores of larger size by removing ash so that the pores are more open.

Rice husk-activated charcoal with pre-treatment compared with non-pre-treatment rice husk-activated charcoal has a number of pores that tend to be more. In addition, it was seen that the pretreated rice husk-activated charcoal formed pores to deep crevices, while the non-pre-treatment rice husk-activated charcoal tended to form a porous structure, and only a few pores were formed, as well as pores doesn't look fully open. The mechanism of the opening of pores is explained through the opening of new pores as well as the widening of existing pores (Budi et al. 2016). The difference in morphology of rice husk-activated charcoal in this study could be caused by the provision of pre-treatment which reduces the mineral ash component, namely silica in rice husk charcoal. It was also mentioned by Kaur et al. (2020) that pre-treatment with base or acid could dissolve inorganic components so that porosity is formed optimally. In addition, the formation of activated charcoal pores can be influenced by the precursor raw material, the temperature used in the process, the impregnation ratio, and the atmospheric conditions at the time of the activation process (Kwiatkowski & Broniek, 2017).

CONCLUSION

The use of pre-treatment and variations in the concentration of KOH gave different characteristics to the activated charcoal of rice husk compared to the non-pre-treatment treatment. The lowest water content was achieved in the N30 sample, which was 1.23%, and the entire water content of activated charcoal had met the SNI standard. Pre-treatment had a significant effect on reducing the ash content of activated charcoal. The ash content of activated charcoal that was pre-treated with NaOH was lower than that of activated charcoal without pre-treatment. Based on its morphology, the activated charcoal of rice husk, which was given pre-treatment showed better porosity properties. The highest KOH concentration of 30% resulted in the best characteristics of water content, ash content, and morphology of rice husk-activated charcoal compared to concentrations of 10 and 20% and better characteristics than unactivated rice husk charcoal and commercial activated charcoal

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