Initial Characterization of Activated Charcoal from the Indigenous Ziziphus mauritiana Wood from Dryland of Sumbawa

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Received: Abstract: Ziziphus mauritiana is widely found in Sumbawa arid and semi-arid 15 June 2023 area. It is a drought tolerant plant which grows in areas with extreme conditions. While information of the utilisation of Bidara seeds as activated carbon is Revised: available, there are limited resources that use Bidara woods. Therefore, this study 10 August 2023 aims to characterize the activated charcoal derived from Z. mauritiana wood which was activated using 25%, 30% and 35% of H_2SO_4 and NaOH. The moisture content, ash content, volatile matter, fixed carbon, and iodine absorption capacity were Accepted: 10 September 2023 investigated and compared to the Indonesian National Standard (SNI. 06-3730-1995). The result showed that NaOH activated charcoal obtained higher quality Published: compared to the H₂SO₄ activated charcoal. The best activated charcoal was 27 September 2023 obtained from 35% of NaOH which has 1.19% moisture content, 13.21% ash content, 1.42% volatile matter, 84.73% fixed carbon, and 1892.40 mg/g iodine number. This study concludes that the characteristics of Z. mauritiana activated charcoal (except the ash content) comply with Indonesian National Standard and DOI: potentially can be developed as an adsorbent. 10.29303/jrpb.v11i2.536 Keywords: activated charcoal; sodium hydroxide; sulphuric acid; Ziziphus ISSN 2301-8119 mauritiana

INTRODUCTION

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Background

Bidara (*Ziziphus mauritiana*) is widely found in several regions in Indonesia, including Sumbawa, West Nusa Tenggara. The main characteristics of this plant are that it grows in dry and extreme conditions such as extreme temperature (>50°C), waterlogging, and low rainfall rate area which defined the plant as a drought and heat tolerant plant (Sakhanokho et al., 2022), (Riaz et al., 2021), (Oliveira et al., 2022). Sumbawa region is characteristically having a large landscape, surrounding by coastlines, and is having tropical climate which is the ideal condition for Bidara to grow (Pratiwi & Sativa, 2022). In general, Bidara trees are used as woodfire and a vital material for wooden boat (Latifah et al., 2019). In addition, the fruits and the leaves are also consumed as fresh fruits and tea, respectively. The woods of Bidara can also be utilized as the materials for activated charcoal production (Regti et al., 2017).

Activated charcoal is a carbonaceous porous material with high adsorption capacity due to its reasonably large pore size, high surface reactivity, and its high specific surface area (Gao et al., 2020), (Lempang et al., 2011). Hence, the charcoal adsorption capacity is influenced by the surface area and the pore size. Also, the presence of hydrocarbon or other components such as minerals, water, nitrogen, and sulphur residues on the surface of the charcoal can affect the adsorption capacity. Each carbonaceous material can be utilized to produce activated charcoal (Lempang et al., 2012). Nevertheless, the quality of activated charcoal is not just affected by the materials used but also influenced by the activating methods used to generate the charcoal.

Charcoal activation could be performed by employing several methods such as physical activation, chemical activation, and physicochemical activation. Physical activation employs oxidizing gas (carbon dioxide, oxygen, and water) pervaded to the charcoal, chemical activation employs chemical agent to impregnate the charcoal, meanwhile the physicochemical method combined the physical and chemical activation method (Jamilatun et al., 2016), (Balahmar et al., 2017), (Danish & Ahmad, 2018). Among the three methods, chemical activation was majorly studied from different materials and different purposes such as heavy metal adsorber (Mariana et al., 2021), cationic dyes scavenging adsorption (Khan et al., 2022), phenol adsorption (Lütke et al., 2019), and wastewater treatment in which removing the micropollutant (Joseph et al., 2020).

The advantageous of the chemical activator is that low energy and time requirement, highly specific microporosity, and high surface area (Naji & Tye, 2022). Several chemical agents that are intensively used are KHSO₅, H₃PO₄, ZnCl₂, NaOH, KOH, dan H₂SO₄ (Zhou et al., 2022). These chemicals react with the polysaccharides of the substrate which leads to the dehydration and elimination of hydrogen and oxygen, enhancing the oxidation, peptization, and dissolution of polysaccharides. The chemicals will also increase the carbonization due to the chemical thermal conductivity and enhance the volatile matter condensation. These activation mechanisms affect the pore size and the carbon structure of the charcoal.

Aims

The aim of this study is to initially characterized activated charcoal generated from Bidara woods which utilized chemical agents such as H_2SO_4 and NaOH with various concentrations to activate the charcoal. Several studies have been conducted on the production of activated charcoal from Bidara seeds such as an adsorptive scavenging of cationic dyes (Khan et al., 2022), methylene blue adsorption (Regti et al., 2017), energy storage applications (Ghimire et al., 2021), and the adsorption of copper ions (Massai et al., 2020). However, currently there are limited resources about the utilisation of Bidara woods as activated charcoal and its characteristics.

RESEARCH METHODS

Reagent and Materials

Bidara (*Ziziphus mauritiana*) woods and stems were collected from Moyo Hulu district, Sumbawa regency, West Nusa Tenggara. Other materials used were NaOH (Sigma Aldrich, 98%), H₂SO₄ (Sigma Aldrich 98%), iodine 0.1N (Sigma Aldrich, 99%), deionized water, sodium thiosulfate 0.1N (Sigma Aldrich, 99%), and amylum 1% (Sigma Aldrich).

Methods

Activated Charcoal Preparation

Bidara wood was pyrolyzed conventionally then crushed into 1-2 cm. The charcoal then impregnated according to the method in (Tan et al., 2014), (Neolaka et al., 2021) with modifications. The sample was soaked in sodium hydroxide and sulphuric acid with 25%,

30%, 35% each in 10:50 (w/v) ratio (10g charcoal: 50ml of the chemical activator). The impregnation was performed for 24 hours in ambient temperatures then filtered and washed in running water to remove the chemical residues. The precipitate was heated in a furnace at 500°C for 2 hours to obtain activated charcoal (AC).

Proximate Test

The AC moisture content was measured in dry basis by the modified method in (Suryajaya et al., 2020), (Fansyuri et al., 2023). The AC sample was heated at 105°C for 1 hour or until a constant weight difference was observed, then it was subsided to ambient temperature in a desiccator. The moisture content is expressed in percentage using following formula (Fansyuri et al., 2023):

 $Mc(\%) = \frac{M_1 - M_2}{M_1} x \ 100\%$ (1)

Where Mc is moisture content, M_1 is the initial weight of sample and M_2 is the final weight of sample.

The AC ash content was also measured by the modified method in (Suryajaya et al., 2020). The remaining sample from Mc measurement was heated at 815°C for 1 hour and subsequently subsided in desiccator. The ash content is also expressed in percentage using the formula in Equation (1).

The volatile matter (Vm) of the sample was also obtained by the modified method in (Suryajaya et al., 2020). The remaining AC from ash measurement was heated at 959°C for 7 minutes then the relative weight differences were measured using formula in Equation (1).

The fixed carbon (FC) percentage expressed the amount of non-volatile carbon remaining in the charcoal. It is calculated by the total difference of moisture content, ash content, and volatile matter content using following formula:

FC (%) =100-(moisture (%) +*Ash* (%) +*Vm* (%).....(2)

Iodine Absorption Capacity

The AC absorption capacity was determined by the ability of the charcoal to absorb iodine according to the method in (Lestari et al., 2019) with modifications. 0.5g of activated charcoal was added into 30ml of iodine 0.1N solution, then shaken for 15 minutes at room temperature and filtered. The filtrate (10ml) was titrated using 0.1N sodium thiosulfate (Na₂S₂O₃) solution. Two to three drops of 1% of amylum solution then pipetted into the titrated sample (a very light yellow) and continued to be titrated until the end point (clear solution). Iodine absorption is expressed in mg/g using following formula:

$$Ia = \frac{A\frac{BxC_1}{C_2}x_{126.93xfp}}{g \ sample}$$
(3)

Where Ia is Iodine Absorption, A is the volume of the sample (ml), B is sodium thiosulfate volume used in titration (ml), C_1 is the concentration of sodium thiosulfate (N), C_2 is the concentration of iodine (N), fp is dilution factor, and 126.93 is the level of iodine in 1 ml of sodium thiosulfate.

The data obtained were statistically analyzed using a one-way analysis of variance test (ANOVA) which was performed using Minitab 7. All data were expressed as the mean \pm standard error of the mean (SEM). The significant difference then measured using Tukey's test with 5% of the α (Alpha). The data were also compared to the Indonesian National Standard (SNI 06-3730-1995).

RESULTS AND DISCUSSION The Effect of NaOH and H₂SO₄ on the Proximate of Activated Charcoal Moisture Content

The moisture content of the activated charcoal which was activated using the sodium hydroxide and sulphuric acid was ranged around 0.36%-1.19% and 1.06%-4.43%, respectively. The statistical analysis showed that the moisture content of the activated charcoal was significantly affected by the variation of the chemical agent concentrations which was also indicated by the escalated moisture content.

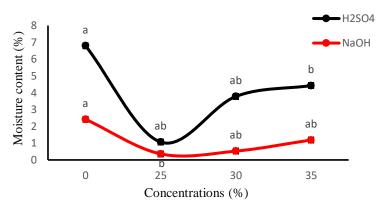


Figure 1. Moisture content with the concentration of chemical activator NaOH and H₂SO₄. (Numbers with the same letters show no significant difference (at the level of 0.05) of different concentration in each curve).

Figure 1 also shows that the moisture content of the charcoal that was activated using 25% of NaOH (0.360%) was lower than that of the charcoal without NaOH activation (2.026%). Similarly, the moisture content of the activated charcoal using 35% of H₂SO₄ (4.426%) was significantly lower than that of charcoal without H₂SO₄ (6.796%). This was because the higher the concentration of NaOH and H₂SO₄ the more oxygen complexes that deteriorated during the activation process which subsequently increased the polarity of the charcoal.

These results also showed that the moisture content of the charcoal has reached the Indonesian National Standard for charcoal quality (moisture content less than 15%). The moisture content of this Bidara activated charcoal was lower than that of the charcoal from langsat shell (*Lansium domesticum Corr*) (8.48%) (Yunanda & Kurniawati, 2021). However, the moisture content of activated Bidara charcoal was higher than that of the activated Mangrove woods charcoal (0.11-0.38%) (Budianto et al., 2019).

Ash Content

The ash content indicates the carbon proportion of the activated charcoal in which low ash content is desirable due to its indirectly proportional relationship with the carbon content (Ajala et al., 2022). The difference of each concentration in NaOH and H_2SO_4 affects the ash content of the activated charcoal. Figure 2 shows that while the ash content of the NaOH activated charcoal was around 13.21%-20.79%, the ash content of H_2SO_4 activated charcoal was around 31.24%-31.27%. In addition, the figure also showed significant differences between the high ash content of activated charcoal without chemical activation (37.1%) and that of using NaOH using 25% (13.21%) and 30% (18.2%) of NaOH. Meanwhile, the ash content of activated charcoal without H_2SO_4 activation (70.09%) was significantly higher than that of charcoal activated using 25% (31.24%) and 35% of H_2SO_4 (31.72%).

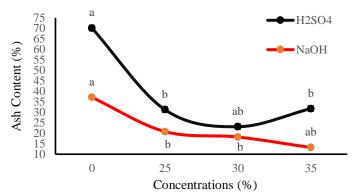


Figure 2. Ash content with the concentration of chemical activator NaOH and H₂SO₄ (Numbers with the same letters show no significant difference (at the level of 0.05) of different concentration in each curve)

Figure 2 also shows the fluctuated and the decreased ash content of activated charcoal activated using H_2SO_4 and NaOH, respectively. The ash content variation was caused by the incomplete oxidation during chemical activation which cause the mineral such as magnesium, sodium, and calcium attached to the pores of the charcoal (Jamilatun et al., 2016). These minerals were nondischargeable during the heating process in the furnace (Permatasari et al., 2014).

These results also showed that the ash content of the charcoal has exceeded the Indonesian National Standard for charcoal quality (ash content is maximum 10%). The ash content of this Bidara activated charcoal was higher than that of the activated charcoal produced from Beringin wood (5.99%) and Ketapang wood (3.36%) (de Meira et al., 2021).

Volatile Matter

Figure 3. shows the volatile matter content of the activated charcoal which was activated using the sodium hydroxide and sulphuric acid ranged around 1.42%-2.22% and 14.67%-19.65%. The low volatile matter indicates that high amount of minerals that was released during the activation process and subsequently increased the quality of the activated charcoal (7) (Hammerton et al., 2018).

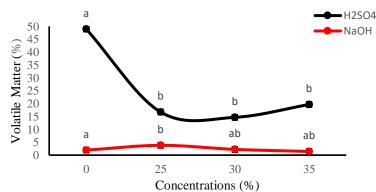


Figure 3. Volatile matter with the concentration of chemical activator NaOH and H2SO4 (Numbers with the same letters show no significant difference (at the level of 0.05) of different concentration in each curve).

Figure 3 shows that there was a significant difference of the volatile matter content that was activated using various concentrations of chemical agents. The result indicated that the increasing of NaOH concentration tends to decrease the volatile matter. This was because the increasing of the NaOH concentration is directly proportional to the increasing of the solution

temperature. Hence, this increased the polarity of the activated carbon which subsequently enhanced the degradation of organic substrate during the heating process.

While the volatile matter was increased due to the NaOH concentration differences, the volatile matter of the activated charcoal activated using H_2SO_4 was increased except in the concentration of 30%. This was because the higher the sulphuric acid concentration the lower the volatility of the mineral during the heating process because sulphuric acid formed a strong bond with the carbonization residues (Fernianti, 2018).

These results showed that the volatile matter content has met the SNI standard in the charcoal activated using both NaOH and H_2SO_4 (less than 25%). However, this study indicated that the volatile matter of the activated charcoal which was impregnated using NaOH solution was lower than that of H_2SO_4 . In addition, this volatile matter was lower than that of Tectona grandis activated charcoal (3.46%) (Erawati & Afifah, 2018).

Fixed Carbon

The use of different concentrations of NaOH and H_2SO_4 showed significant difference of the fixed carbon content of the activated charcoal generated from Bidara. Figure 4 shows that fixed carbon of the charcoal which was activated using NaOH and H_2SO_4 was around 74.73%-84.73% and 44.21%-58.44%, respectively. The fixed carbon value was increased as the concentration of NaOH increased, while fluctuated value was observed as the concentration of H_2SO_4 increased. In addition, the fixed carbon measured from the NaOH activated charcoal was higher compared to that of H_2SO_4 . This was because NaOH oxidized the mineral residues which were attached to the carbon material of the charcoal. The higher the fixed carbon of an activated charcoal, the higher its absorption capacity (Rahman et al., 2020).

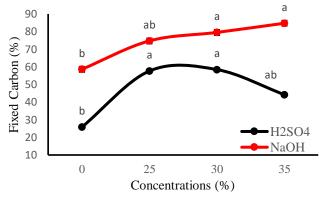


Figure 4. Fixed carbon with the concentration of chemical activator NaOH and H₂SO₄ (Numbers with the same letters show no significant difference (at the level of 0.05) of different concentration in each curve)

The result also showed significant difference of fixed carbon content between charcoal without chemical activation and the chemically activated charcoal in both chemical treatments. Nevertheless, while the fixed carbon of NaOH has met the SNI standard (exceed 65%), the fixed carbon of H₂SO₄ activated charcoal was lower than the SNI standard. The fixed carbon content of the NaOH activated charcoal were higher compared to the fixed carbon of activated charcoal produced from *Cenostigma pluviosum* (69.83%), *Ficus benjamina* (71.30%), *Handroanthus sp.* (73.23%), *Licania tomentosa* (67.34%), *Nectandra megapotamica* (73.64%), *Schinus mole* (68.80%), *Terminalia catappa* (72.29%) (de Meira et al., 2021). Meanwhile, the fixed carbon of H₂SO₄ activated charcoal was lower than that of the mentioned activated charcoal.

Iodine Number

Figure 5 illustrates that the iodine adsorption by the H_2SO_4 and NaOH activated charcoal was around 1523.16 mg/g-2538.60 mg/g and 1015.44 mg/g-1892.40mg/g,

respectively. The results showed that there was a significant difference of iodine number between the NaOH and H₂SO₄ activated charcoal. Also, the iodine number of the NaOH activated charcoal fluctuated, while the iodine number of H₂SO₄ activated charcoal gradually increased as the concentration increased. The high iodine adsorption capacity was caused by the removal of chemical residues during the heating process (Pandia & Warman, 2017) and the bond formed between the adsorbent and the adsorbate which strengthen the adsorption process (Efiyanti et al., 2022). In addition, the high adsorption capacity was caused by the detachment of C and H bonding causing the carbon crystallite to either form new pores or expand the pore sizes (Rahman et al., 2020).

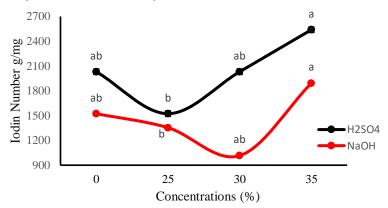


Figure 5. Iodine number with the concentration of chemical activator NaOH and H₂SO₄ (Numbers with the same letters show no significant difference (at the level of 0.05) of different concentration in each curve)

The results also indicated that the iodine number has met the SNI standard (iodine number more than 750 mg/g) in both NaOH and H_2SO_4 activated charcoal. This result was higher compared to the iodine number of mangrove activated charcoal (769.075-1.019mg/g) (Budianto et al., 2019).

Conclusion

This study concluded that the activated charcoal impregnated using 35% of NaOH obtained the most preeminent characteristics such as moisture content, ash content, volatile matter, fixed carbon, and iodine number which was 1.19%, 13.21%, 1.42%, 84.73%, and 1892.40 mg/g, respectively. Meanwhile, the most outstanding characteristics of H₂SO₄ activated charcoal was obtained using 30% of H₂SO₄ which was 3.78%, 23.10%, 14.67%, 58.44%, 2030.88 mg/g of moisture content, ash content, volatile matter, fixed carbon, and iodine number, respectively. This study also concluded that the NaOH activated carbon can be potentially developed as an adsorbent due to its reasonably high quality by which also met the Indonesian National Standard for activated charcoal quality.

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CONFLICT OF INTEREST

The Authors declares that there is no conflict of interest.

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