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Effect of Activation Temperature and H₃PO₄ Concentration on Activated Carbon from Asian Palmyra Palm Fronds (*Borassus Flabellifer* Linn)

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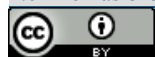
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ABSTRACT

Asian palmyra palm (*lontar*) fronds are a byproduct generated from the asian palmyra palm plant. To add value to these fronds, they can be converted into activated charcoal. This study aims to determine the properties of activated charcoal derived from asian palmyra palm fronds and the effects of activation temperature and chemical concentration on these properties. The activation process was conducted using H₃PO₄ solution with two concentration variations, 10% and 20%, for 24 hours, and three temperature variations, 600°C, 700°C, and 800°C, each for 60 minutes. The data obtained were analyzed using R software version 4.3.1. The effects of the variation factors in H₃PO₄ concentration and activation temperature on the yield and characteristics of the activated charcoal were calculated using two-way ANOVA with a 95% confidence level. The properties of the activated charcoal produced met the SNI 06-3730-1995 standard for technical activated charcoal, with yield, moisture content, ash content, volatile matter content, fixed carbon content, and iodine adsorption values of 80.41%, 0.42%, 9.35%, 8.28%, 82.38%, and 668.13 mg/g, respectively. The best properties of activated charcoal were obtained from the activation treatment at 600°C and 20% H₃PO₄ concentration

INTRODUCTION

The Asian palmyra palm (*Borassus flabellifer* Linn) is a type of palm widely distributed in arid regions of Indonesia, such as the coastal areas of East Java, the eastern part of Central Java, Madura, Bali, West Nusa Tenggara, East Nusa Tenggara, and Sulawesi (Nasri et al., 2017). This plant has many benefits, including its fruit, leaves, fronds, and trunk. Among all parts of the plant, the fronds have the highest biomass. A single trunk of the Asian palmyra palm can produce up to 30-40 fronds (Saduk et al., 2018).

Fronds of the Asian palmyra palm were previously used as writing media, substituting for paper (Tambunan, 2010), but this practice has now ceased. Traditionally, old palm fronds have been used as building materials and firewood, while young fronds can be made into brushes, brooms, and other household items (Nasri et al., 2017). Currently, the utilization of Asian palmyra palm fronds is still considered suboptimal; many old fronds are left to fall to the ground and become waste. To address this issue, solutions are needed to ensure that palm fronds can be used optimally and have economic value.

The fronds of the Asian palmyra palm consist of an outer layer containing silica and an inner part composed of fibers. As a lignocellulosic material, the fronds are porous and contain 68.94% cellulose, 5.37% lignin, 14.03% hemicellulose, and 0.6% wax (Masury et al., 2018). Based on their chemical composition, the value of these fronds can be enhanced by converting them into carbon and activating them to become activated carbon for use as an adsorbent.

Activated carbon or activated charcoal can be made from carbon-containing materials, such as lignocellulosic waste, including agricultural waste. Activated carbon has low density and a porous structure, and it falls into the category of non-graphitic carbon due to its large internal surface area and amorphous structure. This study aims to determine the properties of activated charcoal from Asian palmyra palm fronds and the effects of activation temperature and H₃PO₄ concentration on these properties. The production of activated carbon was carried out using physical and chemical activation methods. The characteristics of the resulting activated charcoal will be evaluated based on SNI No. 06-3730-1995.

METHODS

A. Preparation of Raw Materials

The raw materials used in this study were fronds of the Asian palmyra palm obtained from Dasan Anyar Village, Jereweh District, West Sumbawa, West Nusa Tenggara. The chemicals used included H₃PO₄, 0.1 N iodine solution, and sodium thiosulfate (Na₂S₂O₃) 0.1 N. Equipment used in this study included 60-mesh and 80-mesh sieves, plastic trays, porcelain cups, glass desiccators, porcelain mortars, filter paper, pH meter, dropper pipettes, furnaces, drum kiln furnaces, and ovens.

B. Preparation of Activated Charcoal

The fronds of the Asian palmyra palm were first dried under sunlight to reduce moisture content. The dried fronds were then carbonized using a drum kiln furnace at a temperature of 400°C for approximately 2 hours. The resulting charcoal was ground into powder using a porcelain mortar and sieved to obtain charcoal powder with a particle size passing through a 60-mesh sieve and retained on an 80-mesh sieve. The production of activated charcoal was carried out through chemical and physical activation. The sieved charcoal powder was chemically activated using H₃PO₄ solution with two concentrations, 10% and 20%, for 24 hours (Manurung et al., 2017). After washing the charcoal thoroughly with deionized water until reaching a neutral pH, it was dried and reactivated at three temperature variations: 600°C, 700°C, and 800°C, each for 60 minutes using a furnace (Wibowo et al., 2011). Each treatment was combined to produce 6 treatment combinations and replicated 3 times. Thus, the total number of samples used was 18.

C. Testing of Activated Charcoal

The yield of activated charcoal is determined by comparing the weight of the activated charcoal produced with the weight of the charcoal before activation. The yield is calculated using the equation:

$$\text{Yield (\%)} = \frac{\text{Weight of charcoal after activation (g)}}{\text{Weight of charcoal before activation (g)}} \times 100$$

Testing of activated charcoal refers to the Indonesian National Standard (SNI) 06-3730-1995, including parameters such as moisture content, ash content, volatile matter content, fixed carbon content, and iodine adsorption capacity. Each parameter is calculated using equations (2) to (6) as follows:

$$\text{Moisture Content (\%)} = \frac{\text{Initial sample weight (g)} - \text{Weight of oven - dried sample (g)}}{\text{Initial sample weight (g)}} \times 100$$

$$\text{Ash Content (\%)} = \frac{\text{Weight of ash (g)}}{\text{Weight of oven - dried sample (g)}} \times 100\%$$

$$\text{Volatile Matter Content (\%)} = \frac{\text{Weight of oven - dried sample (g)} - \text{Weight of residual sample (g)}}{\text{Weight of oven - dried sample (g)}} \times 100\%$$

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

$$\text{Iodine Adsorption Capacity} \left(\frac{\text{mg}}{\text{g}} \right) = \frac{10 - \text{Molarity of Na}_2\text{S}_2\text{O}_3 (0,1) \times \text{ml of Thio for titration}}{\frac{\text{Molarity of Iodine (0,1002)}}{\text{Initial Sample Weight (g)}}}$$

D. Statistical Analysis

All data obtained were analyzed using R software version 4.3.1. The influence of the variation factors of H₃PO₄ concentration and activation temperature on the yield and characteristics of activated charcoal was calculated using two-way

analysis of variance (two-way ANOVA) with a 95% confidence level. Subsequently, factors showing significant effects were further tested using Tukey's Honestly Significant Difference (HSD) test by comparing the difference between parameter values and the HSD value obtained to determine different treatment levels.

RESULTS AND DISCUSSION

A. Yield

Activated charcoal yield represents the amount of active charcoal in every gram of activated charcoal. The calculation of activated charcoal yield is conducted to determine the amount of active charcoal produced after the activation process from charcoal to active charcoal (Rahman et al., 2018). The activated charcoal yield from Asian palmyra palm fronds is presented in Table 1.

Table 1. Average Yield of Active Charcoal from Asian Palmyra Palm Fronds (%)

Temperature (°C)	H ₃ PO ₄ Concentration (%)		Mean
	10	20	
600	75.43	78.19	76.81 ^{ns}
700	78.39	81.84	80.11 ^{ns}
800	86.23	82.40	84.31 ^{ns}
Mean	80.07 ^{ns}	80.81 ^{ns}	80.41

Note: Ns, Denotes Non-Significant Difference

The average yield of activated charcoal from Asian palmyra palm fronds ranges from 75.43% to 86.23%, with an overall average yield of 80.41%. The highest yield was obtained from the treatment at 800°C with 10% H₃PO₄ concentration, while the lowest was obtained from the treatment at 600°C with 10% H₃PO₄ concentration. The average yield of activated charcoal from Asian palmyra palm fronds falls into the high category as it exceeds 80%. This yield is significantly higher compared to the yield of active charcoal from coconut shells, which is approximately 49.58% (Rahman et al., 2018), and from beach *calophyllum* shells, which is 80.80% (Sudrajat et al., 2005).

The yield of activated charcoal from Asian palmyra palm fronds increases with the rise in temperature and H₃PO₄ concentration (Table 1). The addition of H₃PO₄ concentration tends to increase the yield of activated charcoal. The utilization of H₃PO₄ can slow down the oxidation process rate and protect the active charcoal when exposed to high temperatures, thereby reducing the amount of material burned during the activation process (Hartoyo and Pari, 1993). However, based on the variance analysis results of this study, it is indicated that temperature and concentration do not significantly affect the yield of activated charcoal, nor does the interaction between temperature and concentration. Therefore, in the production of activated charcoal using Asian palmyra palm fronds,

it can be carried out by combining chemical activation using 10% H₃PO₄ concentration at the lowest temperature, i.e., 600°C.

B. Characteristics of Activated Charcoal

1. Moisture Content

Moisture content measurement aims to determine the hygroscopic properties of activated charcoal. The moisture content value is influenced by water vapor molecules present in the hexagonal lattice of activated charcoal, especially during the cooling process (Pari et al., 2001). The average moisture content of Asian palmyra palm fronds activated charcoal is presented in Table 2. The average moisture content of activated charcoal is 0.42%, which meets the standard of SNI 06-3730-

1995 ($\leq 10\%$). However, compared to several previous studies with average moisture content values ranging from 4-11% (Pari et al., 2006; Suryani et al., 2018; Dewi et al., 2020; Norma et al., 2022), the moisture content value from this study is exceptionally low. Pari (2003) stated that the low moisture content of activated charcoal indicates the success of the activating agent in binding water molecules contained in the material and the loss of free water and bound water content that occurs during the carbonization process of the activated charcoal raw material. This is also proven by the research findings of Dewi et al. (2020), which show that the higher the concentration of the solution, the lower the moisture content produced.

Table 2. Average Moisture Content of Activated Charcoal from Asian Palmyra Palm Fronds (%)

Temperature (°C)	H3PO4 Concentration (%)		Mean
	10	20	
600	0,76	0,43	0,59 ^{ns}
700	0,23	0,17	0,20 ^{ns}
800	0,62	0,34	0,48 ^{ns}
Mean	0,53 ^{ns}	0,31 ^{ns}	0,42

Note: Ns, Denotes Non-Significant Difference

The results of this study indicate that the lowest moisture content value is produced from the combination treatment of 700°C temperature with 20% H₃PO₄ concentration, which is 0.17% (Table 2). Meanwhile, the highest value is obtained from the treatment of 600°C temperature with 10% H₃PO₄ concentration, which is 0.71% (Table 2). Although the values produced differ for each treatment combination, neither temperature, concentration, nor the combination of both factors affect the moisture content value. The variance test results ($\alpha = 0.05$) show no difference in moisture content values for all treatments given ($p\text{-value} > 0.05$). This result is similar to previous research conducted by Rahman et al. (2018), which shows that the type of raw material, concentration, and their interactions do not significantly affect the moisture content.

2. Volatile Matter Content

Table 3 shows the volatile matter content values of activated charcoal from Asian palmyra palm fronds. The volatile matter content ranges from 4.00% to 16.93%, with an average volatile matter content of 9.35%. The highest volatile matter content is obtained from the treatment at 600°C with H₃PO₄ concentration of 10%, while the lowest volatile matter content is obtained from the treatment at 800°C with H₃PO₄ concentration of 20%. The volatile matter content values in this study meet the standard of SNI 06-3730-1995 with a required moisture content of $\leq 25\%$. The average volatile matter content of activated charcoal from Asian palmyra palm fronds is lower compared to previous studies with various types and concentrations of treatments, which have volatile matter contents ranging from 32.70% to 36.33% (Maulana et al., 2017; Rahman et al., 2018; Adawi et al., 2021).

Table 3. Average Volatile Matter Content of Activated Charcoal from Asian Palmyra Palm Fronds (%)

Temperature (°C)	H ₃ PO ₄ Concentration (%)		Mean
	10	20	
600	16,39	6,71	11,55 ^{ns}
700	9,77	5,18	7,48 ^{ns}
800	14,02	4,00	9,01 ^{ns}
Mean	13,40 ^a	5,30 ^b	9,35

Note: Ns, Denotes Non-Significant Difference. Different Letters Behind the Numbers Indicate Significant Differences Based on ANOVA Test With $\alpha = 0.05$

The volatile matter content value indicates the adsorption capacity of activated charcoal. A high volatile matter content will reduce the adsorption capacity of activated charcoal. Conversely, a low volatile matter content is influenced by an increase in activation temperature. When the temperature is high, there is a release of adsorbed compounds, resulting in a decrease in adsorption capacity. Compounds released during the adsorption process include CO₂, CO, CH₄, and H₄ (Lano et al., 2020). Previous research with similar raw materials by Kurniawan & Sutapa (2020) showed that the interaction between temperature and concentration significantly affects the volatile matter content. However, in this study, the variance analysis results ($\alpha = 0.05$) indicate that only the H₃PO₄ concentration affects the volatile matter content, while the activation temperature and the interaction between H₃PO₄ concentration and activation temperature do not have an effect.

Pari et al. (2006) stated that the high or low volatile matter content indicates the presence of active charcoal surface still covered by non-carbon compounds, thus affecting its adsorption capacity. Based on Table 3, the higher the concentration of H₃PO₄ provided, the lower the volatile matter content. This result contradicts the findings of Sudrajat et al. (2005), where an increase in H₃PO₄ concentration tended to increase the volatile matter content. This is because H₃PO₄ functions as a

protector from heat. If the concentration of H₃PO₄ is high, it can reduce the sulfur and nitrogen content that evaporates during combustion at a temperature of 950°C (Sudrajat et al., 2005). Thus, it can result in a high volatile matter content.

3. Ash Content

The ash content is the weight percentage of oxide minerals in carbon such as silicon, sulfur, and calcium (Kalensun et al., 2018). The ash content of activated charcoal from Asian palmyra palm frond ranges from 6.33% to 9.33%, with an average ash content of 8.28% (Table 4). These values already comply with the SNI 06-3730-1995 standard, which requires ash content to be $\leq 25\%$. The average ash content of activated charcoal from Asian palmyra palm frond is higher compared to that of activated charcoal from coconut shell, which is around 2.73% (Suryani et al., 2015; Dewi et al., 2020), and lower compared to activated charcoal from nyamplung shell, which is around 22.4% (Jamilatun, 2015). Research by Sahara (2017) indicated that phosphoric acid could cause corrosion in metals. This is because during the washing process of activated charcoal, soaking it in phosphoric acid (H₃PO₄) still leaves 1-2% phosphorus (P) on the surface of the activated carbon that cannot be removed. Metals contained in charcoal will corrode during activation using phosphoric acid, resulting in a lower ash content of activated charcoal (Yue et al., 2003).

Table 4. Average Ash Content Values of Activated Charcoal from Asian Palmyra Palm Frond (%)

Temperature (°C)	H3PO4 Concentration (%)			Mean
	10	20		
600	7,15	6,33	6,74 ^{ns}	
700	8,48	9,08	8,78 ^{ns}	
800	9,33	9,29	9,31 ^{ns}	
Mean	8,32 ^{ns}	8,23 ^{ns}	8,28	

Note: Ns, Denotes Non-Significant Difference

Table 4 shows that the highest ash content was obtained from the treatment with a temperature of 800°C and H₃PO₄ concentration of 10%, while the lowest ash content was obtained from the treatment with a temperature of 600°C and H₃PO₄ concentration of 20%. Scroder (2006) stated that the increase in carbonization temperature can trigger the oxidation of most of the volatile matter. However, conversely, ash remains un-oxidized. Thus, un-oxidized ash tends to cover the pores, which tends to reduce the ash content value. However, the analysis of variance in this study indicates that temperature and concentration did not significantly affect the ash content of activated charcoal, as well as the interaction between temperature and concentration.

4. Fixed Carbon Content

The fixed carbon content indicates the amount of non-volatile carbon in the activated carbon

(Nurhilal et al., 2020). The average fixed carbon content is presented in Table 5. The fixed carbon content ranges from 76.46% to 86.96%, with an average of 82.38%. The highest fixed carbon content is obtained from the treatment at 800°C with H₃PO₄ (20%), while the lowest is obtained from the treatment at 600°C with H₃PO₄ (10%). The average fixed carbon content of the Asian palmyra palm frond activated carbon is higher compared to the fixed carbon content of activated carbon from thorny bamboo branches, approximately 36.33% (Adawi et al., 2021), and lower compared to activated carbon from pinecones, approximately 91.46% (Kalensun et al., 2012). The fixed carbon content of the Asian palmyra palm frond activated carbon produced in this study meets the standard required in SNI 06-3730-1995, which is ≤65%.

Table 5. Average Fixed Carbon Content of Asian Palmyra Palm Frond Activated Carbon (%)

Temperature (°C)	H3PO4 Concentration (%)		Mean
	10	20	
600	76,46	86,96	81,71 ^{ns}
700	81,75	85,74	83,74 ^{ns}
800	76,65	86,71	81,68 ^{ns}
Mean	78,28 ^b	86,47 ^a	82,38

Note: Ns, Denotes Non-Significant Difference. Different Letters Behind the Numbers Indicate Significant Differences Based on ANOVA Test With $\alpha = 0.05$

The analysis of variance in this study indicates that only the concentration factor significantly affects the fixed carbon content of the activated carbon (Table 5). The high fixed carbon content is influenced by the concentration level. The higher the concentration, the higher the fixed carbon content contained in the activated carbon (Maulana et al.,

2017; Novia, 2020). Additionally, the fixed carbon content is also influenced by the cellulose content in the raw material; the higher the cellulose content, the higher the purity of carbon in the activated carbon (Pane & Hamzah, 2018).

5. Iodine Adsorption Capacity

The determination of activated carbon's iodine adsorption capacity aims to assess its ability to absorb colored and odorous solutions (Budiman et al., 2018). Activation time, activator ratio, and concentration can influence the iodine adsorption capacity of activated carbon. According to Jamilatun & Setyawan (2014), increasing the concentration of the activated carbon activator tends to enhance its iodine adsorption capacity. The iodine adsorption capacity of Asian palmyra palm frond activated carbon is presented in Table 6. The iodine adsorption

capacity values of frond activated carbon range from 641.34 mg/g to 714.4 mg/g, with an average iodine adsorption capacity of 668.13 mg/g. The average iodine adsorption capacity of Asian palmyra palm frond activated carbon is higher compared to that of betel nut activated carbon at around 377.56 mg/g (Dewi et al., 2020), and lower compared to activated carbon from wood waste at around 1013.23 mg/g (Rahman et al., 2018). Nonetheless, this study meets the requirements of SNI 06-3730-1995 with an iodine adsorption capacity stipulated as ≤ 750 mg/g.

Table 6. Average Iodine Adsorption Capacity Values of Asian Palmyra Palm Frond Activated Carbon (mg/g)

Temperature (°C)	H3PO4 Concentration (%)		Mean
	10	20	
600	661,04	648,64	654,84 ^{ns}
700	714,14	668,80	691,47 ^{ns}
800	641,34	674,81	658,08 ^{ns}
Mean	672,18 ^{ns}	664,08 ^{ns}	668,13

Note: Ns, Denotes Non-Significant Difference

The iodine adsorption capacity of activated carbon reflects the formation of micro porous structures. Conversely, a low adsorption capacity indicates fewer micro porous structures, resulting in lower iodine adsorption (Pari, 1999; Lempang & Mody, 2011). The adsorption capacity of activated carbon for iodine correlates with its surface area. A higher iodine number indicates greater ability to adsorb solutes or dissolved substances (Rahman et al., 2018). The analysis of variance in this study indicates that temperature and concentration have no significant effect on the iodine adsorption capacity of activated carbon, as well as the interaction between temperature and concentration. The variance test results ($\alpha = 0.05$) show no significant difference among all treatment groups regarding iodine adsorption values (p-value < 0.05).

CONCLUSION

1. Activated charcoal from Asian palmyra palm fronds has an average yield of 80.41%, with moisture content, ash content, volatile matter content, fixed carbon content, and iodine adsorption capacity values of 0.42%, 9.35%, 8.28%, 82.38%, and 668.13 mg/g, respectively.
2. The characteristics of activated charcoal produced from Asian palmyra palm fronds meet the standard of SNI 06-3730-1995. The best-quality activated charcoal is obtained from the activation treatment at 600°C with 20% H₃PO₄ concentration.

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