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Characterization quality and chemical surface of Indonesian coal-based activated carbon produced in the continuity process

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Preparation and characterization of bamboo based activated carbon prepared by hydrochloric acid

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Structural characterization of $\text{LiCr}_x\text{Mn}_{2-x}\text{O}_4$ prepared by simple reflux-microwave irradiation technique

[Dyah Purwaningsih](#); [Hari Sutrisno](#); [Handy Riantana](#)

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The effect of polymerization temperature on polyurethane synthesis using *Calophyllum inophyllum* oil-based polyol

[Dedy Suhendra](#); [Putri Maulida Sholiha](#); [Erin Ryantin Gunawan](#); [Sri Seno Handayani](#)

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[Erin Ryantin Gunawan](#); [Eka Kusmiati](#); [Dedy Suhendra](#); [Murniati](#)

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[Gani Purwiandono](#); [Puji Lestari](#); [Kazuhiro Manseki](#); [Takashi Sugiura](#)

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Preparation and characterization of bamboo based activated carbon prepared by hydrochloric acid

Wiwik Dahani; Irfan Marwanza; Riskaviana Kurniawati; Fadhilah; Oka Shinta Sekar Kirana; Nor Farida; Muhammad Rasyid Abdullah; Ade Irma Rozafia; Naufal Madani Saputra; Ratna Ediaty; Djoko Hartanto 

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Preparation and Characterization of Bamboo Based Activated Carbon Prepared by Hydrochloric Acid

Wiwik Dahani¹, Irfan Marwanza¹, Riskaviana Kurniawati¹, Fadhilah¹, Oka Shinta Sekar Kirana², Nor Farida², Muhammad Rasyid Abdullah², Ade Irma Rozafia², Naufal Madani Saputra¹, Ratna Ediaty², and Djoko Hartanto^{2,a)}

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Abstract. This research develop an activation process for preparing high surface area bamboo-based activated carbon using hydrochloric acid in low concentration. Bamboo-based activated carbon was prepared using hydrochloric acid as the activating agent. The ratio between carbon bamboo and HCl (w/v) is 1:10, with a surface area of 434.2808 m²/g. After being synthesized, activated carbon was characterized using SEM and gas sorption analyzer (BET and BJH methods). From analysis, activated carbon from bamboo contains a pore within a pore diameter of 3.2209 nm and a V_{total} of 0.247298 cm³/g. This research shows that low concentration hydrochloric acid (0,1 mol/L)

INTRODUCTION

Activated carbon is an effective adsorbent that has been widely utilized for the removal of numerous pollutants [1] [2]. The advantageous characteristics of activated carbon are related to its large surface area, well-developed internal structure, and several surface functional groups [3]. Various carbonaceous resources, such as coal, lignite, wood, and various agricultural byproducts, can be utilized as carbon precursors [4]. Due to their low cost, renewability, and widespread occurrence, agricultural by-products have received increased attention for producing activated carbon in recent years [5]. Some agricultural waste can be used as activated carbon precursors, like bamboo [6]; this agricultural waste utilization is an action to reduce environmental pollution [4]. Bamboo is a cheap and abundant agricultural by-product in tropical countries like Indonesia [7]. However, its utilization has yet to be fully explored [5]. The production of value-added products such as activated carbon will enlarge its application and help to deal with the emergent wastewater treatment challenge in Indonesia. Generally, physical or chemical activation processes are used to prepare activated carbon [8][9]. Chemical activation occurs with the impregnation of the primary material with activating agents such as H₃PO₄, KOH, or ZnCl₂ [10], followed by thermal activation to create the pore structure [11]. Chemical activation generally results in a higher carbon yield and a more fully developed pore structure than physical activation [12][13][14]. In general, alkalis' chemical activation involves a solid–solid or solid–liquid reaction involving hydroxide reduction and carbon oxidation to produce porosity [15] [16]. The surface functional group of activated carbon can be controlled through chemical activation or post-treatment [16]. Changing the activation temperature, activated carbon matrix, or acid/base impregnation makes it possible to modify the pore structure of activated carbon [17] [18].

The main purpose of this study is to develop an activation process for preparing high surface area bamboo-based activated carbon. The carbon yield was given attention as it was necessary for practical application. The activated carbons' surface physical and chemical properties have been characterized using low-temperature N₂ adsorption and surface morphology.

EXPERIMENTAL SECTION

Materials

The materials in this study are Indonesia's bamboo *Gigantochloa pseudoarundinacea*, sodium hydroxide (NaOH), hydrochloric acid (HCl), and distilled water.

Preparation of Bamboo Carbon

Bamboo carbon was carried out by the pyrolysis method. Bamboo are cleaned using distilled water and bamboo were put into the furnace and pyrolyzed for 6 hours at 450 °C in a vacuum. This step obtained the bamboo carbon.

Bamboo Carbon Activation

The bamboo carbon was grounded with a pestle and mortar and then sieved using a 100-mesh sieve. Two grams of bamboo carbon were added with NaOH 0.1 mol/L. The mixture was heated on a hotplate at 85 °C and stirred at 100 rpm for 3 hours. Then, filtered through filter paper until the filtrate and residue were separated. Residue or carbon was dried in an oven at 105 °C for 2 hours. After drying, HCl 0.1 mol/L was added, heated at 65 °C and stirred at 100 rpm for 3 hours. Then, the mixture was filtered, and the residue was washed with hot distilled water. After that, calcined in the muffle furnace at 500 °C for 2 hours. This step obtained bamboo activated carbon.

Material Characterizations

The surface physical properties of the activated carbons were characterized with a Micromeritics ASAP 2020, using N₂ as the adsorbate. The surface area (S_{BET}) was calculated with the BET equation, and the pore volume (V_T) was obtained from the adsorption isotherm at $P/P_0 = 0.95$. The micropore area (S_{mic}) and volume (V_{mic}) were obtained using the t -plot method. The mesopore volume (V_{mes}) was the deduction of V_{mic} from V_T , and the mesopore area (S_{mes}) was the deduction of S_{mic} from S_{BET} . The structural and morphology of carbon before and after activation were analyzed with scanning electron microscopy analysis at 2.5 kV and 20 μ A.

RESULTS AND DISCUSSION

Bamboo Carbon and Bamboo Activated Carbon

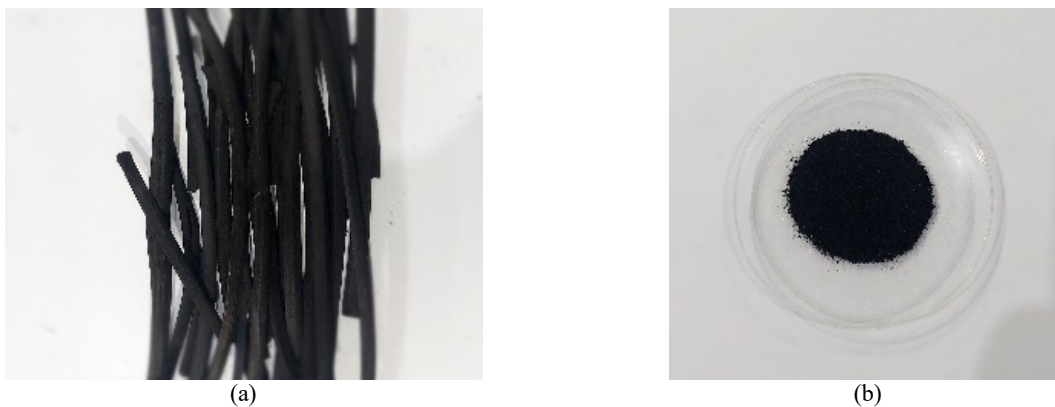


FIGURE 1. (a) Bamboo Carbon, (b) Bamboo Activated Carbon

The bamboo has been pyrolyzed, shown in Fig. 1 (a), called bamboo carbon. Then bamboo carbon was activated using sodium hydroxide (NaOH) and penetrated further into the porous structure of the bamboo carbon [2]. After NaOH activation, the remaining chemical reagents and impurities or tar are separated by filtering to obtain better carbon pore size [5]. The bamboo carbon was reactivated using hydrochloric acid (HCl) to degrade the organic compounds formed during the carbonization process, reduce the tar formation, and dehydrate water trapped in the

carbon cavities to obtain a larger pore size [6], [7]. After activation, activated carbon was washed using distilled water to remove impurities and dried at 105 °C for 24 hours, then calcined at 500 °C for 2 hours to increase the surface area and pore. Bamboo activated carbon is shown in Fig. 1 (b).

Characterization of Bamboo Carbon and Bamboo Activated Carbon

Activation of carbon from the activation bamboo using hydrochloric acid (HCl) has been successfully prepared. SEM analysis was used to determine the surface morphology of pyrolyzed bamboo carbon before and after activation. Characteristics of the morphology of pyrolyzed carbon before activation are shown in Fig. 2. It shows that there are small pores on the surface of the bamboo carbon.

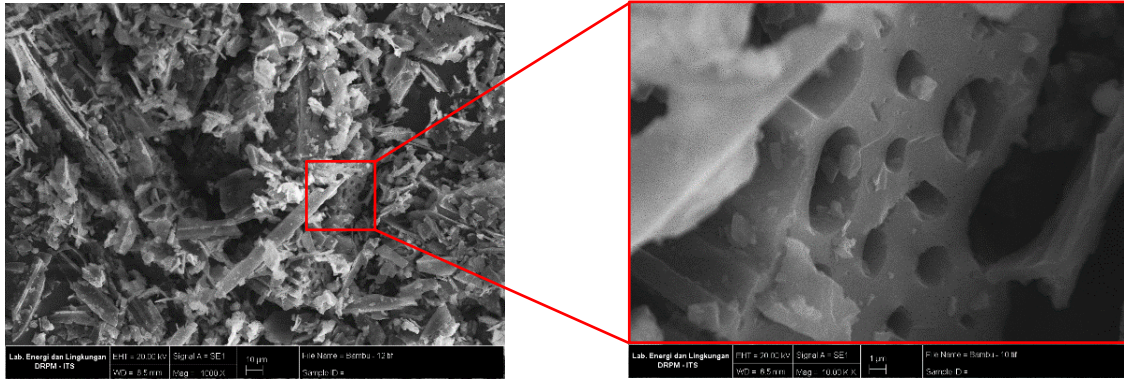


FIGURE 2. Surface Morphologies of Bamboo Carbon

After the pyrolyzed process, bamboo carbon was activated using hydrochloric acid (HCl). The bamboo activated carbon shows the pore with channel structure associated with the cellulose fibres. It is a suitable morphology for adsorption because it can diffuse into the activated carbon pores and has a high surface area. During pyrolysis, most of the non-carbon compounds are removed with the remaining polymer base framework, resulting in carbon that retains the precursor's shape and forms the initial carbon porosity. Micrographs show that considerable porosity is present in bamboo carbon after the activation process in the form of an interconnected network which accounts for the high BET surface area (S_{BET}) pore volume and pore diameter of activated carbon. Surface morphologies of bamboo activated carbon are shown in Fig. 3.

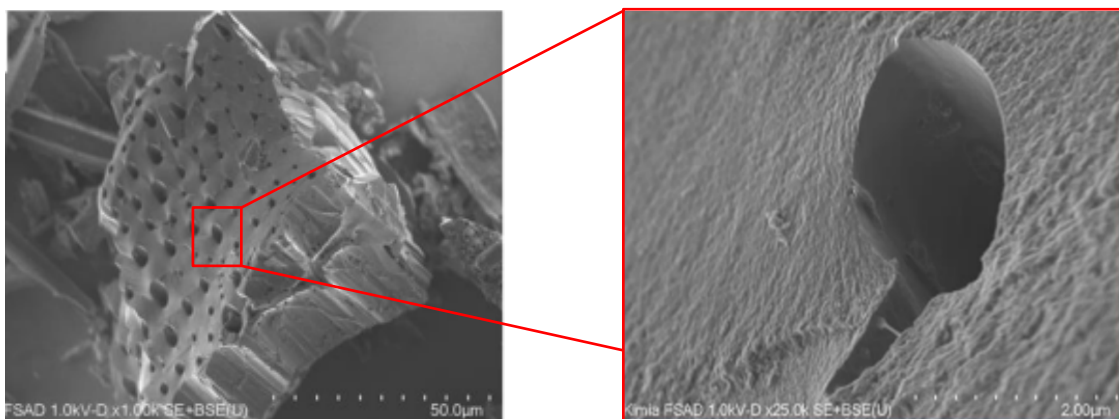


FIGURE 3. Surface Morphologies of Bamboo Activated Carbon

Nitrogen physisorption analysis determined the material surface area and pore size through BET and BJH methods. The analysis results show that bamboo carbon before activation only has a small surface area of 4.7970 m²/g. In addition, the total pore volume and diameter were 0.006154 cm³/g; 1.7078 nm, respectively. This small surface area indicates that the remaining organic molecules (tar form) have not been removed. It is expected that after the activation

process using sodium hydroxide, the surface area increase above 300 m²/g. The pore size of bamboo carbon is shown in Fig. 4.

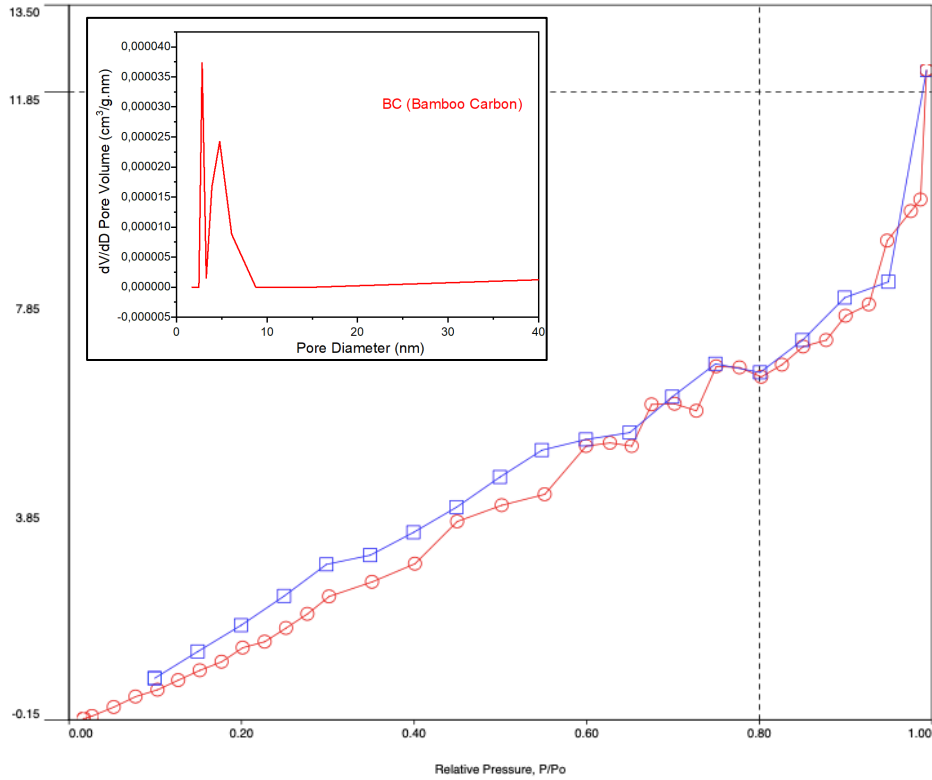


FIGURE 4. Nitrogen Gas Adsorption-Desorption Isotherm of Bamboo Carbon

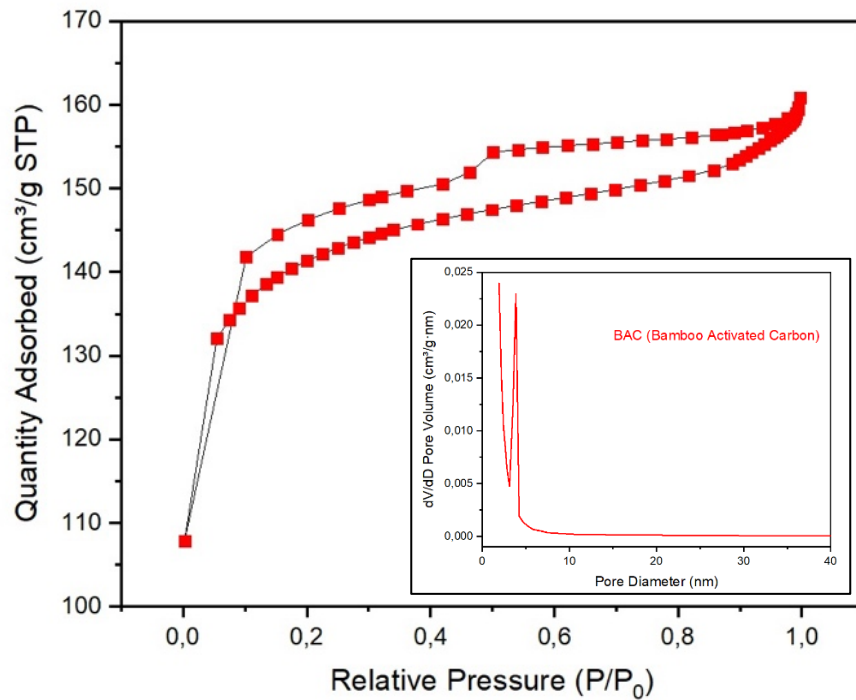


FIGURE 5. Nitrogen Gas Adsorption-Desorption Isotherm of Bamboo Activated Carbon

Bamboo carbon after activation also determined using nitrogen physisorption analysis. The analysis results show that bamboo activated carbon has type V pore, larger surface area, total pore volume, also pore diameter than bamboo carbon before activation. The bamboo activated carbon has surface area, total pore volume, and diameter were 434.2808 m²/g; 0.247298 cm³/g; 3.2209 nm, respectively. The pore size of bamboo activated carbon is shown in Fig. 5.

Table 1 below shows the differences between bamboo carbon and bamboo activated carbon. From table 1, it shows that bamboo carbon after activation increased surface area, total pore volume, and pore diameter. This can happen because the bamboo carbon was activated using hydrochloric acid (HCl) to degrade the organic compounds formed during the carbonization process, reduce the tar formation, and dehydrate water trapped in the carbon cavities to obtain a larger pore size [6], [7].

TABLE 1. Nitrogen Gas Adsorption-Desorption Isotherm of Bamboo Carbon and Bamboo Activated Carbon

Material	S _{BET} (m ² /g)	Volume Total (cm ³ /g)	Pore Diameter (Å)	Pore Diameter (nm)
Bamboo Carbon	4.797	0.006154	17.078	1.7078
Bamboo Activated Carbon	434.2808	0.247298	32.209	3.2209

CONCLUSION

This research has succeeded in synthesizing carbon from bamboo through pyrolysis and activation using hydrochloric acid to produce bamboo activated carbon. SEM and nitrogen physisorption characterized the results. SEM showed that the bamboo carbon formed larger pores after activation from those before it was activated. The nitrogen adsorption-desorption of bamboo carbon before activation showed a surface area of 4.7970 m²/g, total pore volume of 0.006154 cm³/g, and pore diameter of 1.7078 nm. After activation, bamboo carbon shows surface area, total pore volume, and pore diameter were 434.2808 m²/g; 0.247298 cm³/g; 3.2209 nm, respectively.

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The materials in this study are Indonesia's bamboo *Gigantochloa pseudoarundinacea*, sodium hydroxide (NaOH), hydrochloric acid (HCl), and distilled water.

Preparation of Bamboo Carbon

Bamboo carbon was carried out by the pyrolysis method. Bamboo are cleaned using distilled water and bamboo were put into the furnace and pyrolyzed for 6 hours at 450 °C in a vacuum. This step obtained the bamboo carbon.

Bamboo Carbon Activation

The bamboo carbon was grounded with a pestle and mortar and then sieved using a 100-mesh sieve. Two grams of bamboo carbon were added with NaOH 0.1 mol/L. The mixture was heated on a hotplate at 85 °C and stirred at 100 rpm for 3 hours. Then, filtered through filter paper until the filtrate and residue were separated. Residue or carbon was dried in an oven at 105 °C for 2 hours. After drying, HCl 0.1 mol/L was added, heated at 65 °C and stirred at 100 rpm for 3 hours. Then, the mixture was filtered, and the residue was washed with hot distilled water. After that, calcined in the muffle furnace at 500 °C for 2 hours. This step obtained bamboo activated carbon.

Material Characterizations

The surface physical properties of the activated carbons were characterized with a Micromeritics ASAP 2020, using N₂ as the adsorbate. The surface area (S_{BET}) was calculated with the BET equation, and the pore volume (V_{T}) was obtained from the adsorption isotherm at $P/P_0 = 0.95$. The micropore area (S_{mic}) and volume (V_{mic}) were obtained using the t -plot method. The mesopore volume (V_{mes}) was the deduction of V_{mic} from V_{T} , and the mesopore area (S_{mes}) was the deduction of S_{mic} from S_{BET} . The structural and morphology of carbon before and after activation were analyzed with scanning electron microscopy analysis at 2.5 kV and 20 μ A.

RESULTS AND DISCUSSION

Bamboo Carbon and Bamboo Activated Carbon

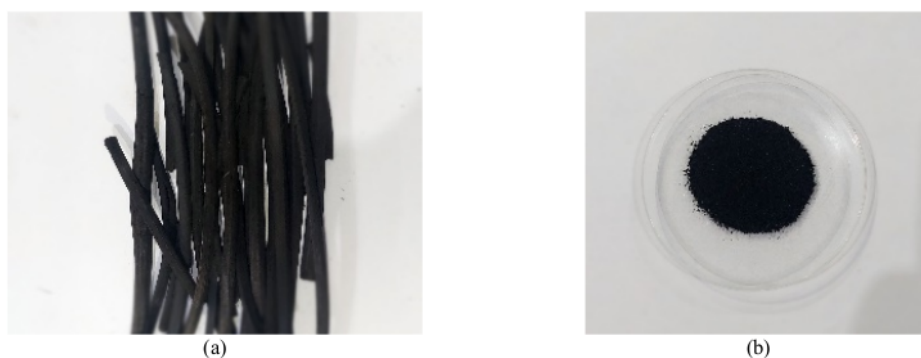


FIGURE 1. (a) Bamboo Carbon, (b) Bamboo Activated Carbon

The bamboo has been pyrolyzed, shown in Fig. 1 (a), called bamboo carbon. Then bamboo carbon was activated using sodium hydroxide (NaOH) and penetrated further into the porous structure of the bamboo carbon [2]. After NaOH activation, the remaining chemical reagents and impurities or tar are separated by filtering to obtain better carbon pore size [5]. The bamboo carbon was reactivated using hydrochloric acid (HCl) to degrade the organic compounds formed during the carbonization process, reduce the tar formation, and dehydrate water trapped in the

carbon cavities to obtain a larger pore size [6], [7]. After activation, activated carbon was washed using distilled water to remove impurities and dried at 105 °C for 24 hours, then calcined at 500 °C for 2 hours to increase the surface area and pore. Bamboo activated carbon is shown in Fig. 1 (b).

Characterization of Bamboo Carbon and Bamboo Activated Carbon

Activation of carbon from the activation bamboo using hydrochloric acid (HCl) has been successfully prepared. SEM analysis was used to determine the surface morphology of pyrolyzed bamboo carbon before and after activation. Characteristics of the morphology of pyrolyzed carbon before activation are shown in Fig. 2. It shows that there are small pores on the surface of the bamboo carbon.

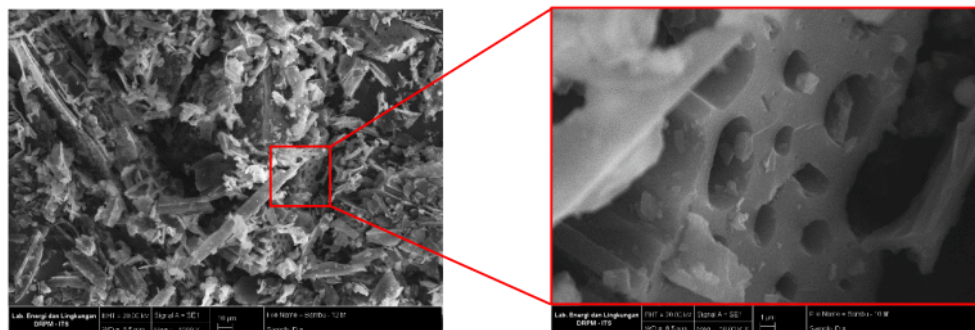


FIGURE 2. Surface Morphologies of Bamboo Carbon

After the pyrolyzed process, bamboo carbon was activated using hydrochloric acid (HCl). The bamboo activated carbon shows the pore with channel structure associated with the cellulose fibres. It is a suitable morphology for adsorption because it can diffuse into the activated carbon pores and has a high surface area. During pyrolysis, most of the non-carbon compounds are removed with the remaining polymer base framework, resulting in carbon that retains the precursor's shape and forms the initial carbon porosity. Micrographs show that considerable porosity is present in bamboo carbon after the activation process in the form of an interconnected network which accounts for the high BET surface area (S_{BET}) pore volume and pore diameter of activated carbon. Surface morphologies of bamboo activated carbon are shown in Fig. 3.

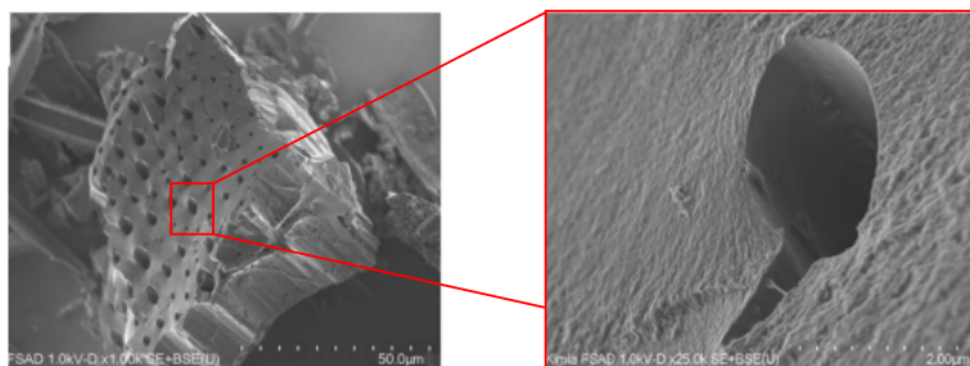


FIGURE 3. Surface Morphologies of Bamboo Activated Carbon

Nitrogen physisorption analysis determined the material surface area and pore size through BET and BJH methods. The analysis results show that bamboo carbon before activation only has a small surface area of 4.7970 m²/g. In addition, the total pore volume and diameter were 0.006154 cm³/g; 1.7078 nm, respectively. This small surface area indicates that the remaining organic molecules (tar form) have not been removed. It is expected that after the activation

process using sodium hydroxide, the surface area increase above 300 m²/g. The pore size of bamboo carbon is shown in Fig. 4.

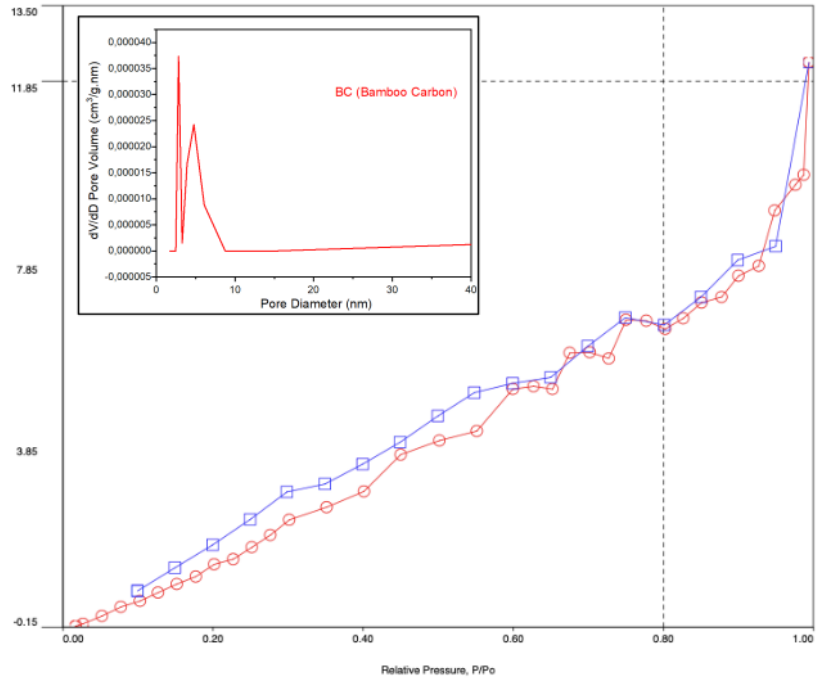


FIGURE 4. Nitrogen Gas Adsorption-Desorption Isotherm of Bamboo Carbon

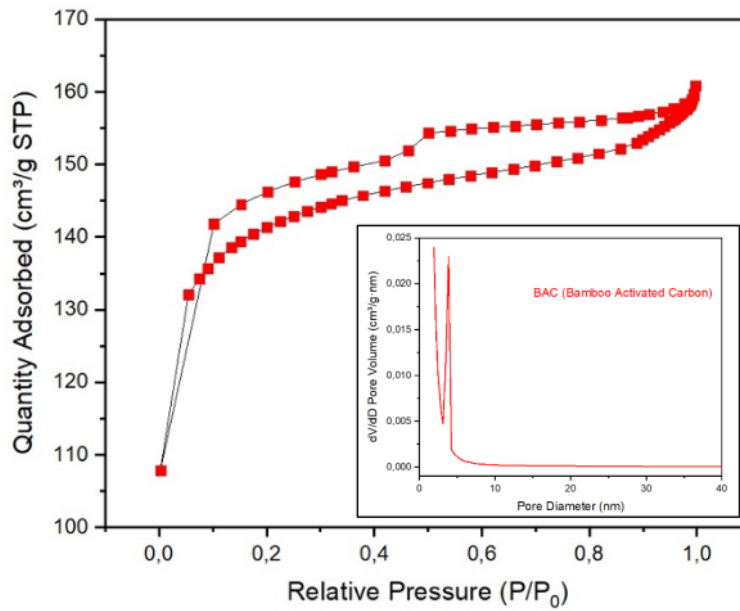


FIGURE 5. Nitrogen Gas Adsorption-Desorption Isotherm of Bamboo Activated Carbon

Bamboo carbon after activation also determined using nitrogen physisorption analysis. The analysis results show that bamboo activated carbon has type V pore, larger surface area, total pore volume, also pore diameter than bamboo carbon before activation. The bamboo activated carbon has surface area, total pore volume, and diameter were 434.2808 m²/g; 0.247298 cm³/g; 3.2209 nm, respectively. The pore size of bamboo activated carbon is shown in Fig. 5.

Table 1 below shows the differences between bamboo carbon and bamboo activated carbon. From table 1, it shows that bamboo carbon after activation increased surface area, total pore volume, and pore diameter. This can happen because the bamboo carbon was activated using hydrochloric acid (HCl) to degrade the organic compounds formed during the carbonization process, reduce the tar formation, and dehydrate water trapped in the carbon cavities to obtain a larger pore size [6], [7].

TABLE 1. Nitrogen Gas Adsorption-Desorption Isotherm of Bamboo Carbon and Bamboo Activated Carbon

Material	S _{BET} (m ² /g)	Volume Total (cm ³ /g)	Pore Diameter (Å)	Pore Diameter (nm)
Bamboo Carbon	4.797	0.006154	17.078	1.7078
Bamboo Activated Carbon	434.2808	0.247298	32.209	32209

CONCLUSION

This research has succeeded in synthesizing carbon from bamboo through pyrolysis and activation using hydrochloric acid to produce bamboo activated carbon. SEM and nitrogen physisorption characterized the results. SEM showed that the bamboo carbon formed larger pores after activation than those before it was activated. The nitrogen adsorption-desorption of bamboo carbon before activation showed a surface area of 4.7970 m², total pore volume of 0.006154 cm³/g, and pore diameter of 1.7078 nm. After activation, bamboo carbon shows surface area, total pore volume, and pore diameter were 434.2808 m²/g; 0.247298 cm³/g; 3.2209 nm, respectively.

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