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SURFACE AREA ANALYSIS OF ACTIVATED CARBON MATERIAL FROM PALM FROND WASTE USING DIFFERENT ACTIVATION AGENTS

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ABSTRACT

Carbon is one of the abundant elements obtained from organic matter, one of which is found in the fronds of oil palm trees which is found on the Kalimantan Island. This research uses local organic waste in East Kalimantan to be used as activated carbon material, palm frond waste. Activated carbon material is one of the materials that has a high surface area and is widely applied for adsorbent materials. This study aims to analyze and compare the surface area of activated carbon synthesized from palm frond waste using different activating agents. This research also analyzes the nature of the material structure, surface area, pore size and pore volume of activated carbon. The method used is the process of thermal activation and chemical activation to convert carbon into activated carbon with variations of activating agents, phosphoric acid (H_3PO_4) and sodium hydroxide (NaOH). XRD, BET-SAA and SEM-EDX characterization tests were conducted. The results obtained show that the amorphous structure of activated carbon has been formed and there is an increase in BET surface area due to the activation process. The surface area obtained for the Non-Activated Carbon (C) sample is 15,149 m^2/g, Activated Carbon-NaOH (AC-1) is 2,082 m^2/g, Activated Carbon-H_3PO_4 (AC-2) is 307,692 m^2/g. This shows that the best sample with the highest surface area is the AC-2 sample which uses the H_3PO_4 chemical activator solution.

Keywords: Activated Carbon, Activating Agent, Surface Area, Pore Size, Pore Volume

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INTRODUCTION

Kalimantan, one of the major islands in Indonesia, has abundant natural resources. One of the great potentials is the palm oil industry. Oil palm plantations have become one of the significant economic sectors in the region, accounting for the majority of palm oil exports and being an important source of income for local communities. With the potential of this large oil palm plantation, it will have an impact from the economic sector to the environmental conservation sector due to the large amount of waste produced [1]. However, the development of the palm oil industry has also caused environmental problems, especially related to palm frond waste. Palm frond waste is generally not optimally utilized and is often dumped or burned, causing environmental pollution.

Palm fronds have great potential as a natural material rich in carbon content. This abundant carbon content is a valuable asset that can be utilized to produce activated carbon. Activated carbon is a material that has a porous structure and large surface area, has the ability to absorb and bind various contaminants, including odors, synthetic dyes, heavy metals, and organic compounds. Therefore, processing palm frond waste into activated carbon through a thermal activation process is an attractive alternative in reducing the environmental impact of palm frond waste and at the same time increasing the added value of the waste [2].

Biomass is material derived from living things, including plants, animals and microbes. One of the wastes from biomass is palm frond which is still very little processed. Therefore, processing of palm frond waste is needed to increase its use value. One of the products that can be produced from palm fronds is activated carbon. Palm fronds have a high carbon content of 73.33%, so they have the potential to be used as raw material for making activated carbon [3]. Activated carbon is believed to be the most popular form of carbon and can be produced from natural sources of carbon-containing materials. Chemically, activated carbon is carbon that has been enhanced with micro-sized porosity. The surface area of activated carbon can reach 500-2000 m^2/g [4].

Several studies have successfully synthesized activated carbon materials from biomass using various types of chemical and thermal activation. Coconut shells have been used to synthesize activated carbon using activation temperatures of 600^0C and 1000^0C for 1 hour with $NaCO_3$ chemical activator resulting in pore sizes of 0.8-1 μm [5], while activated carbon from other coconut shells activated at 600^0C for 3 hours using NaOH obtained a surface area of 516 m^2/g [6]. Besides that, activated carbon has been synthesized from oily sludge using KOH agent and activation temperature 800^0C show the pore structure, high volume of micropores and surface area was 2,263 m^2/g [7]. The kenaf core was also used to produce activated carbon using H_3PO_4 as activator agent resulting in a surface area of 299.02 m^2/g [8], while activated carbon from date palm fronds using H_3PO_4 as activator agent produces a surface area of 1138 m^2/g [9]. The shell of the nipa fruit was activated using variation of temperatures (300^0C, 400^0C, 500^0C, 600^0C, and 700^0C), obtained the optimum temperature is 500^0C and has a surface area 27.39 m^2/g [10]. In addition, activated carbon have been synthesized from palm fronds by comparing the use of chemical activators Na_2CO_3 and NaCl. It was found that more pores were formed with greater pore cavity depth after using Na_2CO_3 than NaCl [11].

Based on several previous studies, it shows that natural materials or biomass are proven to have high carbon content and can be synthesized into activated carbon material. Has been shown different types of activator agents and different activation temperatures, as well as different surface area results. This study uses palm frond waste (*Elaeis guineensis jacq*) as the basic material for making activated carbon by comparing the effect of using different activating agents, there were NaOH and H_3PO_4 and also using thermal activation at 500^0C with a shorter holding time, thus obtain a simpler method for producing activated carbon material. Furthermore, this research aims to synthesize and identify the material structure, surface area, pore size and pore volume of activated carbon which will be compared with non-activated carbon (without activation process).

METHOD

Experimental Method

This research was used a simple experimental method to synthesize activated carbon material. Activated carbon was synthesized using solvothermal method with chemical activation and thermal activation. The raw material used was palm frond waste (*Elaeis guineensis jacq*) obtained from Ibu Kota Nusantara (IKN) palm oil plantation area, East Kalimantan. *Sodium Hydroxide* (NaOH) PA *Merck KGaA* 1 M and *Phosphoric Acid* (H_3PO_4) PA *Merck* 83% were used as activating agents. *Aquadest* and *Hydrochloric Acid* (HCl) *Merck* PA were used for the pH neutralization process.

The synthesis of activated carbon consists of four procedures, there are carbonization, chemical activation, thermal activation and pH neutralization. Carbonization was carried out to convert palm fronds into charcoal/carbon using a furnace with a temperature of 600°C for 3 hours. Chemical activation was carried out by mixing carbon powder with NaOH and H_3PO_4 solutions respectively with a mass ratio of 1:3 grams. The solution that has been mixed will be soaked for 24 hours in a closed container. Thermal activation was carried out after chemical activation process for both solutions. Thermal activation was used a furnace with a temperature of 500^0C for 1 hour.

The last process is pH neutralization, the activated carbon powder is washed with HCl solution and distilled water until a neutral pH condition is obtained. The samples were dried at 120 ^{0}C for 30 minutes to remove moisture content. The final sample was obtained in powder which is consisted of 3 samples there are C (pure carbon), AC-1 (activated carbon with NaOH agent) and AC-2 (activated carbon with H_3PO_4 agent).

Instrumentations

X-Ray Diffraction (XRD) characterization was used to identify the structure of the material. The XRD analysis was performed by Bruker Eco D8 Advance using Cu-Kα (λ=1.5405 Å) radiation source, operating under a voltage of 40 kV and a current of 25 mA. The diffraction angle (2θ) is used from 10º to 80º.

Scanning Electron Microscopy (SEM) characterization was performed to determine the morphological structure with *Energy Dispersive X-Ray* (EDX) analysis to determine the elements contained in the material. The SEM-EDX analysis was performed by Phenom ProX, operating under a voltage 1.5 kV.

Surface Area Analysis (SAA) characterization with *Brunauer-Emmet-Teller* (BET) full isotherm method was used to analyze the pore size, surface area and pore volume of the material. The BET-SAA analysis was performed by *Quantachrome Instrument Nova Touch* LX4 using Nitrogen (N_2) adsorbate at 77.35 K. All samples were degassed at 300^0C for 3 hours.

RESULTS AND DISCUSSION

Activated carbon materials have been successfully synthesized using several methods with variation of activator solutions. The activated carbon material in this study was synthesized from palm frond waste obtained from oil palm plantations in the IKN area of East Kalimantan. The material synthesis process is carried out in several steps, including the dehydration process, carbonization, chemical activation, thermal activation, and pH neutralization. The final sample obtained is activated carbon powder. The process of synthesizing activated carbon material from palm frond waste are shown in Figure 1.

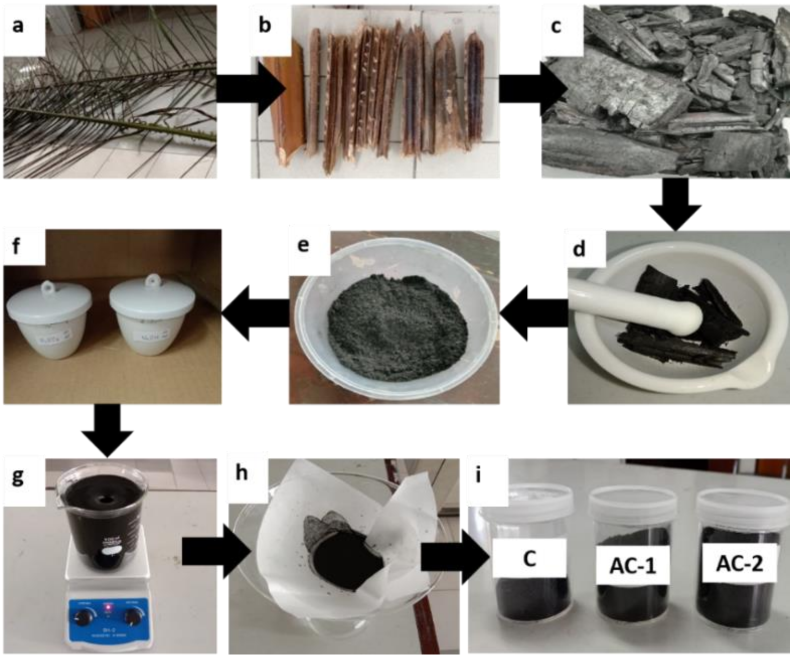


Figure 1. Synthesis process of activated carbon consists of (a) raw material from palm frond waste; (b) cutting process; (c) carbonization process; (d) mashing process; (e) charcoal powder; (f) activation process; (g) pH neutralization process; (h) filtration process; and (i) sample results C, AC-1, AC-2.

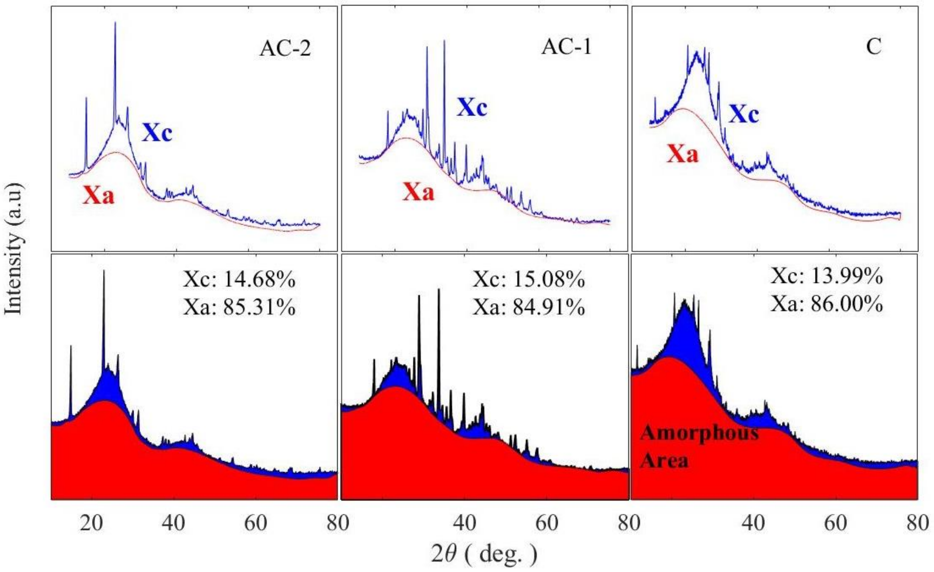
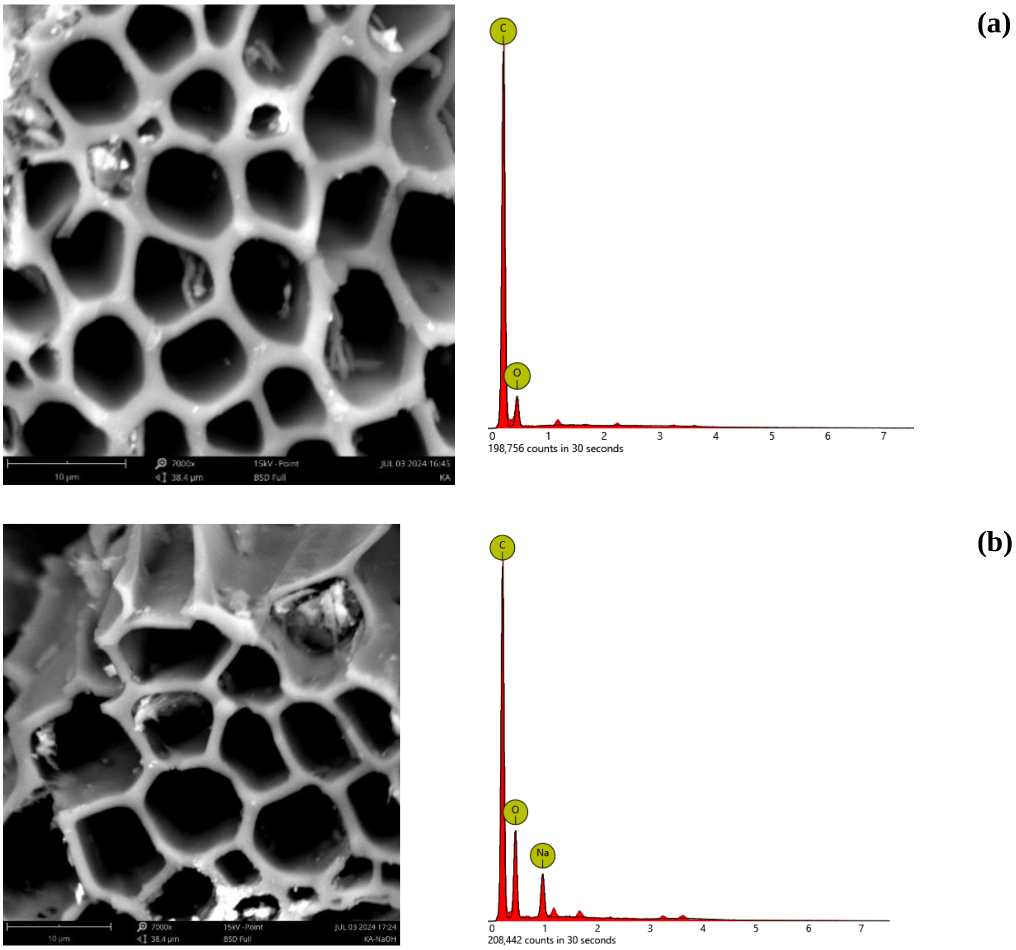


Figure 2. XRD pattern with amorphous index (Xa) and crystallinity index (Xc) of C (pure carbon), AC-1 (activated carbon with NaOH agent) and AC-2 (activated carbon with H_3PO_4 agent).

The structural analysis of activated carbon was obtained from the *X-Ray Diffraction* (XRD) characterization results. The results of XRD are shown in Figure 2. The XRD graph provides information about diffraction angle (θ) and the intensity of the diffraction peak. The XRD pattern of all sample show a broad diffraction background which reflects the amorphous structure of non-activated carbon and activated carbon [12]. The results of the three samples show different graphical characteristics, but there are similarities in the wide peaks between θ = 20^0-30^0 and 43^0. The wide peak shows the characteristics of activated carbon which has an amorphous phase [8]. Figure 2 also shows the amorphous and crystallinity index of three samples.

The amorphous index (Xa) calculated for all samples - C (86.00%), AC-1 (84.91%), and AC-2 (85.31%) - clearly indicate a significant predominance of the amorphous phase. Therefore, although the observed peaks indicate the presence of small crystalline regions, the majority of the material structure is amorphous. This high amorphous content justifies the overall classification of the sample as largely amorphous, with the understanding that a crystalline component may be presented but not dominant aspect [13].



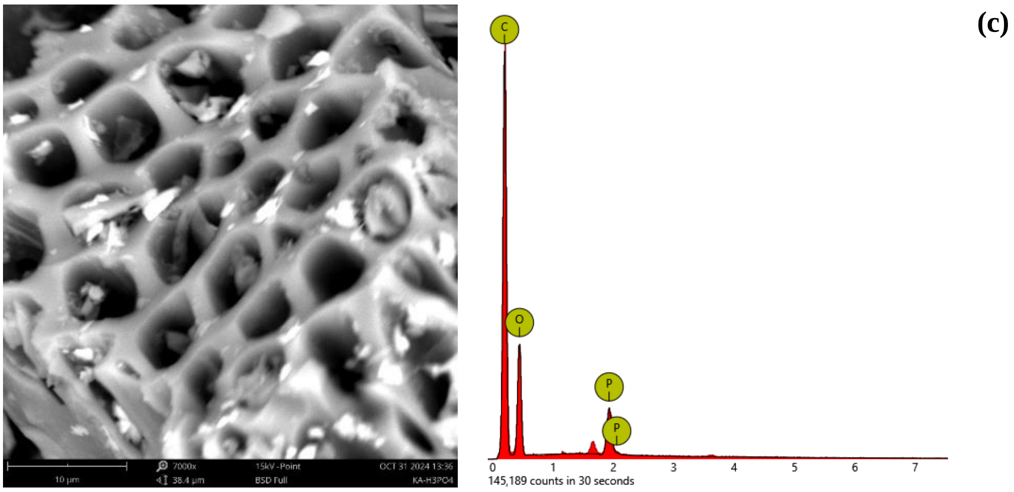


Figure 3. The morphological structure through SEM and EDX element detected of samples at 7000x magnification (a) C (b) AC-1, and (c) AC-2

XRD pattern of samples AC-1 and AC-2 were found that there are some impurities in crystal peaks. The impurities might come from the activating agent which are NaOH and H_3PO_4, where the NaOH crystal peaks were found at around 2θ $(34^0$-$35^0)$ [14] and the H_3PO_4 peak was at around 2θ $(16^0, 22^0, 26^0)$ [15]. This impurity crystal peaks were due to the incomplete washing process of the activated carbon sample, which causes the activator material to remain on the sample [16]. In addition, the presence of several sharp peaks on the XRD pattern indicates the presence of other elements that are crystalline [17], the presence of these peaks is in accordance with the SEM-EDX results in the next discussion.

In addition, the BET results on pore size offer additional context for the observed XRD characteristics. The activated carbons, AC-1 and AC-2, show very different pore sizes compared to the non-activated (C). AC-1, with a large average pore size of 12.73 nm, and AC-2, with a very small average pore size of 1.19 nm, still showed a high degree of amorphousness according to the Segal method. This suggests that the activation process, although dramatically changing the porosity and surface area, mainly produces or maintains a disordered atomic arrangement[18-19] .

Scanning Electron Microscopy (SEM) is an electron microscope that uses electron beams to observe the physical morphology of the sample [20]. Analysis of the pore surface structure was carried out using a scanning electron microscope (SEM). This characterization provides the surface morphology material due to changes in carbonization and activation process [21]. The result of surface morphology from *Scanning Electron Microscopy* (SEM) and *Energy Dispersive X-Ray* (EDX) component are shown in Figure 3. The results show the presence of porous surfaces by the presence of hollow hole which is indicated by the difference in color contrast from the SEM images.

The porous surface of the samples indicated that carbon is a material that has high porosity. However, the shape and size of the pores in all samples are still classified as non-homogeneous and has an irregular shape. This can be caused by the raw material used are from organic samples. Besides that, it can also be caused by the charcoal smoothing process which is done manually using a mortar pestle. In addition to the porous structure, the morphological structure of all samples also shows a hollow structure caused by the opening of pores due to the evaporation process of cellulose components resulting in larger cavities with a tight arrangement [2]. The surfaces of all samples have the pores which are in different sizes and shapes. The pore development on the surfaces of the activated carbon resulted from the evaporation of the activator solution during carbonization, leaving space previously occupied by the activator solution [22]. Chemical and thermal activation processes can lead to the formation of voids that aid in the decomposition of volatile matter from biomass. Thermal activation at 500^0C can produce in more pores and substantial volatile matter removal [8]. With the thermal activation process using a temperature of 500^0C causes the formation of cavities due to the influence of heat so that the decomposition of organic compounds from palm fronds occurs [23].

Furthermore, the EDX results show a number of elements contained in each sample. In addition, to determine the percentage of concentration ratio between elements in each sample, the atomic and element weight percentage value have been presented in Table 1.

Table 1. The elemental concentration in atomic and weight percentage of samples with 7000x magnification.

Sample	Element			Concentration (%)	
	Number	Symbol	Name	Atomic	Weight
C	6	C	Carbon	83.39	79.04
	8	O	Oxygen	16.61	20.96
AC-1	6	C	Carbon	71.39	64.35
	8	O	Oxygen	26.14	31.39
	11	Na	Sodium	2.47	4.26
AC-2	6	C	Carbon	73.71	65.72
	8	O	Oxygen	23.47	27.87
	15	P	Phosphorus	2.49	5.71

The results obtained show that the concentration of carbon atoms in all samples has a high concentration compared to other atoms. In addition to carbon atoms, the EDX characterization results indicated the presence of other atoms such as Oxygen (O), Sodium (Na) and Phosphorus (P). In sample AC-1 (activated carbon with NaOH activation) shows the presence of Na atoms in the sample, because the carbon sample was soaked and activated using NaOH solution. The Na content that is still in the sample can be caused by the neutralization and washing in synthesize process might not take place optimally, so there are still Na atoms in the sample composition. This can also be seen from the XRD pattern results in Figure 1 which shows a high peak in the AC-1 sample which is indicated by Na atoms. Furthermore, in the AC-2 there were P atoms in the sample which is caused by the chemical activation process using H_3PO_4 solution. The remaining phosphate atoms in the sample can be caused by the relatively low temperature and holding time of thermal activation, so that it has not been maximized in removing other elements in the carbon sample.

The surface area and pore size analysis of the materials were obtained from *Brunauer-Emmet-Teller* (BET) full isotherm characterization. The BET-SAA is a characterization to identify the surface properties of a porous material, such as surface area, pore size, and pore volume [6]. The BET-SAA characterization results are shown with the N_2 Adsorption-Desorption Isotherm curve in Figure 4.

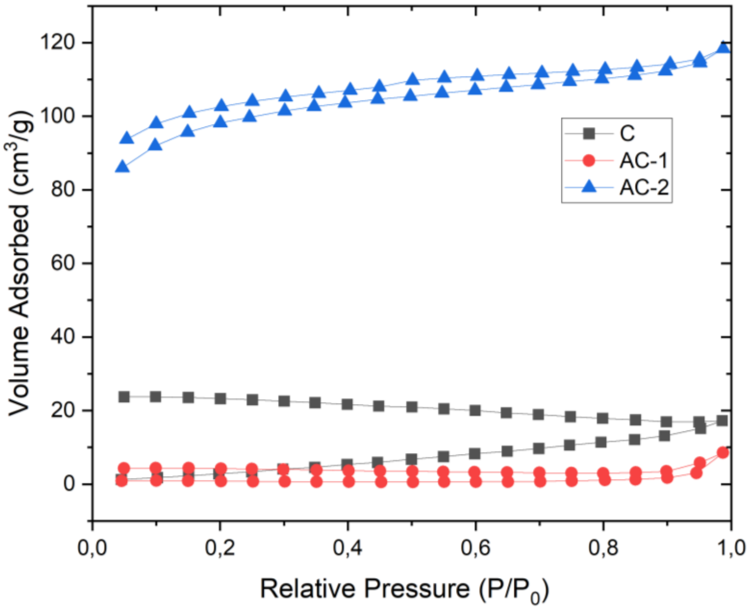


Figure 4. N_2 Adsorption-Desorption Isotherm Curves of samples C, AC-1 and AC-2.

The isotherm curve is a curve that describes the N_2 adsorption-desorption process of the sample that forms a hysteresis loop [24]. The adsorption isotherm was used to evaluate the gas adsorbed at different relative pressures (P/P_0), where P is the gas vapor pressure, and P_0 is the saturation pressure of the adsorbent [6]. Based on the isotherm curves obtained from the three samples, different curve shapes and volumes of N_2 adsorbed were obtained. In curves AC-1 (red) and AC-2 (blue), the volume of N_2 absorbed (*Adsorption*) is almost equal to the volume released (*Desorption*), while for curve C (black) the volume of N_2 released (*Desorption*) is not equal to the volume absorbed (*Adsorption*). The difference in volume in N_2 Adsorption-Desorption could be due to the presence of N_2 gas trapped in the pores of the sample (C). Based on Figure 7, the AC-2 sample has a larger adsorbed volume (cm^3/g) value than the C and AC-1 samples. Furthermore, the comparison of surface area, particle radius, pore size and pore volume are shown in Table 2.

Table 2. Comparison of the BET-SAA characteristics of non-activated carbon (C) and activated carbon (AC-1 & AC-2).

Sample	**BET Surface Area (m^2/g)**	**Particle Radius (nm)**	**Pore Size (nm)**	**Pore Volume (cm^3/g)**
C	15.1492	90.014	3.5297	0.026736
AC-1	2.08211	654.93	12.737	0.013260
AC-2	307.692	4.4318	1.1934	0.18360

The activation process on carbon material is proven to increase the surface area as shown by sample AC-2 with the highest surface area value of 307.692 m^2/g compared to AC-1. Thus AC-2 sample activated using H_3PO_4 is able to produce high surface area, smallest pore size and largest pore volume compared to non-activated carbon (C) and activated carbon with NaOH activation (AC-1). This is in accordance with the N_2 Adsorption-Desorption Isotherm Curves in Figure 7. The difference in surface area of the samples can be caused by the type of chemical activator used as a representative of acidic, salt, and basic properties [6]. Activating agents contribute to creating more new pores and enlarging existing pores [25]. The greater the surface area of the activated carbon adsorbent, the greater the potential adsorption capacity of the adsorbent. This is because the activation process causes the opening of the pores of the activated carbon adsorbent, increasing the pore volume and diameter formed during the carbonization and activation process [26].

Further obtained, AC-1 sample showed a decrease in surface area and an increase in pore size from the non-activated carbon (C) sample to the activated carbon sample. This could be due to the presence of salt/sodium elements from the activation process that fill or get trapped in the pores of the carbon and as a result of the incomplete washing or neutralization process. Table 1 shows that the surface area and pore volume of activated carbon prepared by phosphoric acid are significantly higher than activated by sodium hydroxide. In terms of pore size distribution, C and AC-1 samples are mainly based on mesopores (2-50 nm) while AC-2 sample is classified into micropores (<2 nm) [27].

CONCLUSION

Activated carbon material has been successfully synthesized from palm frond waste using chemical and thermal activation with different activating agents. XRD characterization show that all samples have an amorphous carbon phase with the formation of wide peaks. The morphology from SEM Images shows a porous surface with the presence of hollow hole on the samples. The results of EDX show that overall, the carbon element is the highest concentration in all samples. The results of the BET-Surface Area Analyzer show the best surface area characteristics in AC-2 is 307.692 m^2/g which has significantly increased the surface area of non-activated carbon. Furthermore, it was found that the activation of H_3PO_4 agent was better in increasing the surface area than the activation of NaOH agent.

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