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Research Article

Characterization of Active Carbon from Coconut Shell using X-Ray Diffraction (X-RD) and SEM-EDX Techniques

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Abstract

Activated carbon is produced from the coconut shell through physical and chemical activation. The pyrolysis method was employed in this research for physical activation at an optimum temperature of 600°C and 1,000°C, for chemical activation immersed using 10% Na_2CO_3 activating agent. This research has produced two samples, namely the physical activation of 1,000°C and the physics-chemical activation of Na_2CO_3. The X-Ray Diffraction (X-RD) spectrum of activated carbon in the samples 1,000°C and Na_2CO_3 contained silicate minerals, iron ore and quartz, respectively, and it showed the formation of carbon and graphite structures in the hkl (002) and (100) planes. Through Scherrer's method, the average size of the Na_2CO_3 crystals sample is 15.03 nm and the sample crystal sample of 1,000°C is 54.53 nm; the size of the Nano-scale crystals was formed when the temperature increases ≥ 600° C. The X-RD resulted the percentage of elemental content carbon phase volume fraction (Fv) and impurity (I) in the 1,000°C sample of 75.61%, 24.39% and the Na_2CO_3 sample of 77.87%, 22.13% . These results indicate that the carbon content in chemical activation is much better than the physics activation. SEM results with magnification of 5,000x, it is very clear the porosity formed of the 10 µm picture size are 0.8 µm in Na_2CO_3 sample and 1.00 µm in 1,000°C sample.

Keywords: *active carbon, coconut shell, SEM-EDX, X-RD*

Karakterisasi Karbon Aktif Disiapkan dari Tempurung Kelapa Menggunakan Teknik X-Ray Diffraction (X-RD) dan SEM-EDX

Abstrak

Karbon aktif dihasilkan dari tempurung kelapa melalui aktivasi fisika dan kimia. Metode pirolisis digunakan untuk aktivasi fisika pada suhu optimum 600°C dan 1000°C, untuk aktivasi kimia yang direndam menggunakan 10% Na_2CO_3 sebagai zat aktivasi. Hasil penelitian ini menhasilkan dua sampel yaitu aktivasi fisika 1000°C dan aktivasi fisika-kimia Na_2CO_3. Sinar spektrum X-Ray Diffraction (X-RD) karbon aktif dalam sampel 1000°C dan Na_2CO_3 mengandung mineral silikat, bijih besi dan kuarsa, masing-masing, dan menunjukkan pembentukan struktur karbon dan grafit dalam bidang hkl (002) dan (100), metode Scherrer ukuran rata-rata kristal sampel Na_2CO_3 adalah 15,03 nm dan sampel kristal 1000°C adalah 54,53 nm, ukuran kristal berskala nano terbentuk ketika suhu meningkat ≥ 600°C. Hasil persentase X-RD kandungan unsur fasa karbon fraksi volume (Fv) dan ketidakmurnian (I) pada sampel 1000°C adalah 75,61%, 24,39% dan sampel Na_2CO_3 adalah 77,87%, 22,13%. Hasil ini menunjukkan

bahwa kandungan karbon dalam aktivasi kimia jauh lebih baik daripada aktivasi fisika. Hasil SEM dengan perbesaran 5000 x, ukuran gambar 10 µm sangat jelas porositas yang terbentuk adalah 0,8 µm pada Na_2CO_3 dan 1,00 µm pada sampel 1000°C.

Kata Kunci: *karbon aktif, SEM-EDX, tempurung kelapa, XRD*

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I. INTRODUCTION

Coconut shell as a raw material in several occasion is used to be activated into active carbon [1]. Based on the data in the Asian region, in particular Southeast Asia,is a region with a considerable amount of coconut production with four countries as its main producers, India, Indonesia, The Philippines and Sri Lanka contributing up to 78% of coconut production in the world [2]. Regarding the active carbon production, Indonesia is now one of the major exporting countries of active carbon [3]; the activated carbon currently has been widely studied across the world [2, 4, 5]. One of the advantages of the proposed materials of coconut shell is that the active carbon has been popular as the most effective and useful adsorbent to remove pollutants from contaminated gas and fluid flow. This characteristic is because by nature the active carbon has a large active surface area which can provide a well-developed porous structure of adsorption capacity and good mechanical properties [6]. The fundamental substance used for the production of activated carbon is carbon-rich organic matter [7]. The development of methods of using waste materials as activated carbon is desirable to be a solution in waste utilization such as seeds of *Jatsrophas*, corncobs, coconut shell, palm fiber and sawdust. These sources have been proven very well to be converted into activated carbon because of their hard and strong texture due to the high lignin and carbon content, and on the other hand, consisting of low ash content inside these materials [4, 5]. To produce the activated carbon from the coconut shell, in particular, pyrolysis method is employed, i.e. the process of coconut shell into charcoal followed by the activation process. The activation process is divided into two methods, namely physical and chemical activations. The process of physical activation is obtained by carbonization with oxidizing gas or carbon dioxide at high temperatures of 400-1,000°C. For chemical activation, the material has to be soaked into chemical solutions such as $ZnCl_2$, Na_2CO_3, KOH and KCl with a certain concentration [8]. The activated carbon is believed to produce good carbon crystal structures and amorphous structures that are irregularly stacked by carbon rings and beneficial to produce an adsorbed slit, which is 97% pure carbon [8, 9]. The activation process is carried out through some procedures of mixing the basic material with the activation reagent, and the mixture is heated under atmospheric pressure inert. This process is usually performed at lower temperatures and times compared to the physical activation process. The surface area and the resulting porosity are better than

chemical activation; therefore, this research utilized coconut shell as the main ingredient in generating the activated carbon and observe produced crystal structure by using X-ray diffraction and surface porosity of activated carbon material. Scanning Electron Microscopy - Energy Dispersive X-ray Spectroscopy (SEM-EDX) is employed to observe the physical morphology of surfaces of the samples and analyze elemental compositions, including visible light elements such as carbon, nitrogen and oxygen [10-13].

II. RESEARCH METHOD

Raw material

Coconut shell is selected for the manufacture of the activated carbon. The material was cleaned with distilled water three times in order to remove dust and dirt on the shells. The coconut shell sample was then dried in the oven at 110ºC for 24 hours to remove surface moisture and then milled to the estimated size of 5-10 mm. The analysis to determine the level of volatile and carbon fixed as well as to measure the composition of each element was conducted through similar method which has been done as stated in previous research conducted by Hidayu et al.

Activated Carbon

Physical carbon activation of the coconut shell was carried out by inserting the coconut shell into a heated pyrolysis reactor at 600º C and 1,000 ºC for one hour. Carbon shelled coconut at 600 ºC was processed by chemical activation and soaked in 10% Na_2CO_3 solution then stored for 24 hours at room temperature. The two samples of physical activation of 1,000 ºC and physics-chemical activation Na_2CO_3 were produced. After the activation processes were completed, activated carbon was then cleaned with distilled water and dried by oven at 100 °C for thirty minutes. After that, sieving was done using ASTM Standard Test Sieve with the size of 70-200 Mesh. The sieve model used consisted of three sieve arrangements; sifted samples, then taken and characterized.

III. RESULTS AND DISCUSSION

X-RD analysis

The XRD spectrum of the activated carbon of Figure 1 shows the diffraction peaks formed on the samples of 1,000 °C and Na_2CO_3 at the angles of $2\theta = 27.5^o$, 37.9^o, and 57.7^o in the hkl (101), (004), and (211) plots, respectively, silicate minerals, iron ore and quartz. At the rest of the other peaks, there are sodalite, analcime and sodium silicate located at 44.5^o. The existence of those materials were either due to the contaminated samples during the preparation or the quality of the coconut shell samples were not quite good. [9]. Meanwhile, the widespread peaks around 24.4^o and 44.3^o indicate the formation of carbon and graphite structures in the (hkl) plane of (002) and (100). Both of the active carbon samples showed that two diffraction peaks are located at the diffraction angle of 57.7° which explains the presence of amorphous phases that are irregularly stacked by carbon rings and useful for generating an adsorbed gap. In the Na_2CO_3 sample, the diffraction peak was observed at 44.5^o, this was due to the presence of the Sodium (Na) species used during the activation process [6]. After the pyrolysis was done, both samples have two broad diffraction peaks and can be attributed to the presence of carbon and graphite which is in line with the previous studies by Kushwaha et al. and Rani et al. [14-16]. In order to measure the size of the crystal of the two diffraction peaks, such calculation should be undertaken to the unknown parameter L (crystal size), the value $\Delta\theta = FWHM\,(\beta)$, which is roughly half of $2\Delta\theta$. The Scherrer's equation predicts the size of the grains or the crystals whether the crystals are smaller than 1,000 Å or 100 nm. The simplest way to derive the Scherrer's equation

is to take a derivative from Bragg's Law, $\lambda = 2d \sin\theta$, with a thickness of $\Delta d = L$, Scherrer's equation is written. As the values of λ (wavelength) and K were close to unit value (0.9), Scherrer's equation can be stated as follows:

$$L = \frac{0.9\lambda}{\beta.cos\theta} \quad (1)$$

To observe the evolution of carbon and graphite phase growth (C), the percentage of volume fraction (Fv) was calculated based on the X-RD spectrum, the peak intensity of carbon phase diffraction $I(fn)$, and the intensity of the entire peak of the diffraction (I total) using the following formula:

$$Fv(fn) = \frac{I(fn)}{Itotal} \quad (2)$$

To know the amount of impurities (I) that may exist, the following formula was used [10]:

$$I = 100\ \% - Fv \quad (3)$$

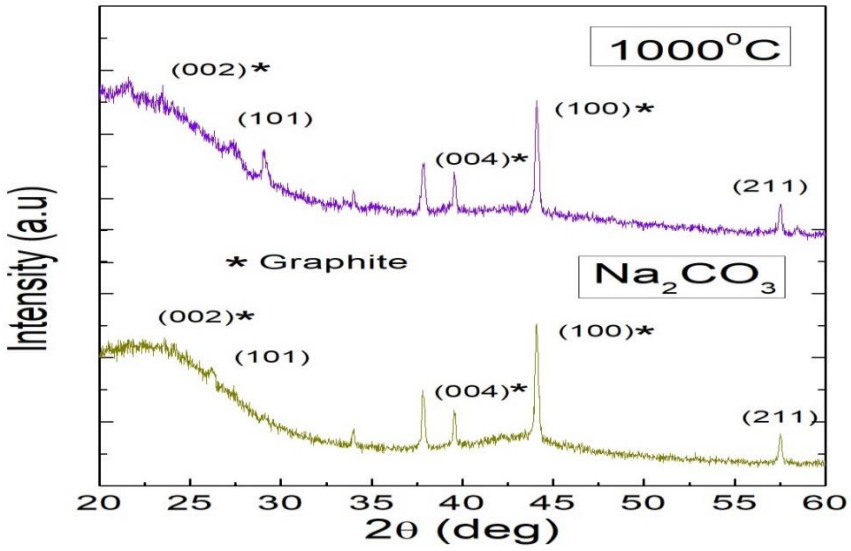

Figure 1. X-ray Diffraction Patterns of the Activated Carbons of Na_2CO_3 and 1,000 °C (* Content: Carbon and Graphite)

Table 1. Crystal Size by Scherrer's Method

Activated Carbon	2θ (deg)	hkl.	FWHM/β (radiant)	Scherrer's Method	
				Crystal size (nm)	Average size of crystal (nm)
Na_2CO_3	21.66	100	0.0209	6.74	15.03
	27.30	101	0.0168	8.48	
	38.70	004	0.0084	17.54	
	44.86	100	0.0067	22.38	
	54.34	211	0.0078	19.99	
1,000° C	24.10	100	0.0091	15.62	54.53
	27.33	101	0.0023	61.71	
	37.82	004	0.0032	46.22	
	43.46	100	0.0025	58.58	
	57.24	211	0.0017	90.51	

Table 2. Percentage of Element Content by XRD Technique

Activated Carbon	Percentage (%)	
	Volume Fraction (*Fv*)	Impurity (*I*)
1,000ºC	75.61	24.39
Na_2CO_3	77.87	22.13

Based on the results of the Scherrer's method in Table 1, the average size of the Na_2CO_3 sample crystals is 15.03 nm and the size of 1,000ºC sample crystal is 54.53 nm. The size of the Nano-scale crystals was formed when the temperature rises ≥ 600 °C, despite the fact it was less affected than the higher temperature increase. In other words, the experimental activation energy for the formation of Nano-scale crystals can occur on the baseline of ≥ 600 °C [17]. The results of table 2 are the analysis of the X-ray diffraction spectrum, the percentage of element content of volume fraction (*Fv*) and impurity (*I*) of carbon phase in the 1,000ºC sample is 75.61 % and 24.39 %, and the percentage for Na_2CO_3 sample is 77.87 %, 22.13% and 8.8%, this means that the carbon content of further chemical activation is good.

SEM-EDX Analysis

Scanning Electron Microscopy - Energy Dispersive X-ray Spectroscopy (SEM-EDX) analysis was employed to observe the physical morphology of sample surfaces and to analyze the element compositions, including the visible light elements such as carbon, nitrogen and oxygen. EDX detector equipped with ultra-thin element light windows that detect elements with atomic numbers > 4 [17]. The EDX technique also was utilized in the analysis on the contamination because the presence of particular substances, and such contamination may affect some issues, i.e. the quality of the content [18]. Figure 2 shows the SEM morphology of the activated carbon microstructure of Na_2CO_3 and 1,000 ºC with the magnification of 3,000 times, a 10 µm image size. For Figure 2 (a) Na_2CO_3, the pore size formed is 0.8 µm, and around the area, there is also another porosity which explains that the carbon activation result is successful, and for Figure 2 (b) 1,000oC, the porosity is clearly visible at the size of 1.00 µm, and around the area there are also other porosities proving that carbon activation is successful. Figure 2 shows that the activation stage produces a large external surface with sufficient pores [19]. The results of the EDX spectrum in Table 3 show the presence of elemental contents of Al, Ca, Fe, K, Mg, Na, O, Si, Ti, content (wt.%), respectively for Na_2CO_3 samples of 5.22%, 10.59%, 3.31%, 11.82%, 3.70%, 17.45%, 34.99%, 11.89%, 1.07%, 72.50%, and for 1,000 ºC of 7.62%, 3.93%, 3.65%, -%, 3.21%, 5.41%, 38.55%, 18.69%, -%, 74%. Element Ti (Titanium) in energy beam, i.e. 4.50 KeV and 0, the 50 KeV presents in the Na_2CO_3 sample indicates the width of the various metals that allows the material to be dispersed [20, 21].

Table 3. Elements detected on the EDX technique

Activated Carbon	Energy Dispersive X-ray Spectroscopy (wt. %)								
	Al	Ca	Fe	K	Mg	Na	O	Si	Ti
Na_2CO_3	5.22	10.59	3.31	11.82	3.70	17.45	34.99	11.89	1.07
1,000 ºC	7.62	3.93	3.65	-	3.21	5.41	38.55	18.69	-

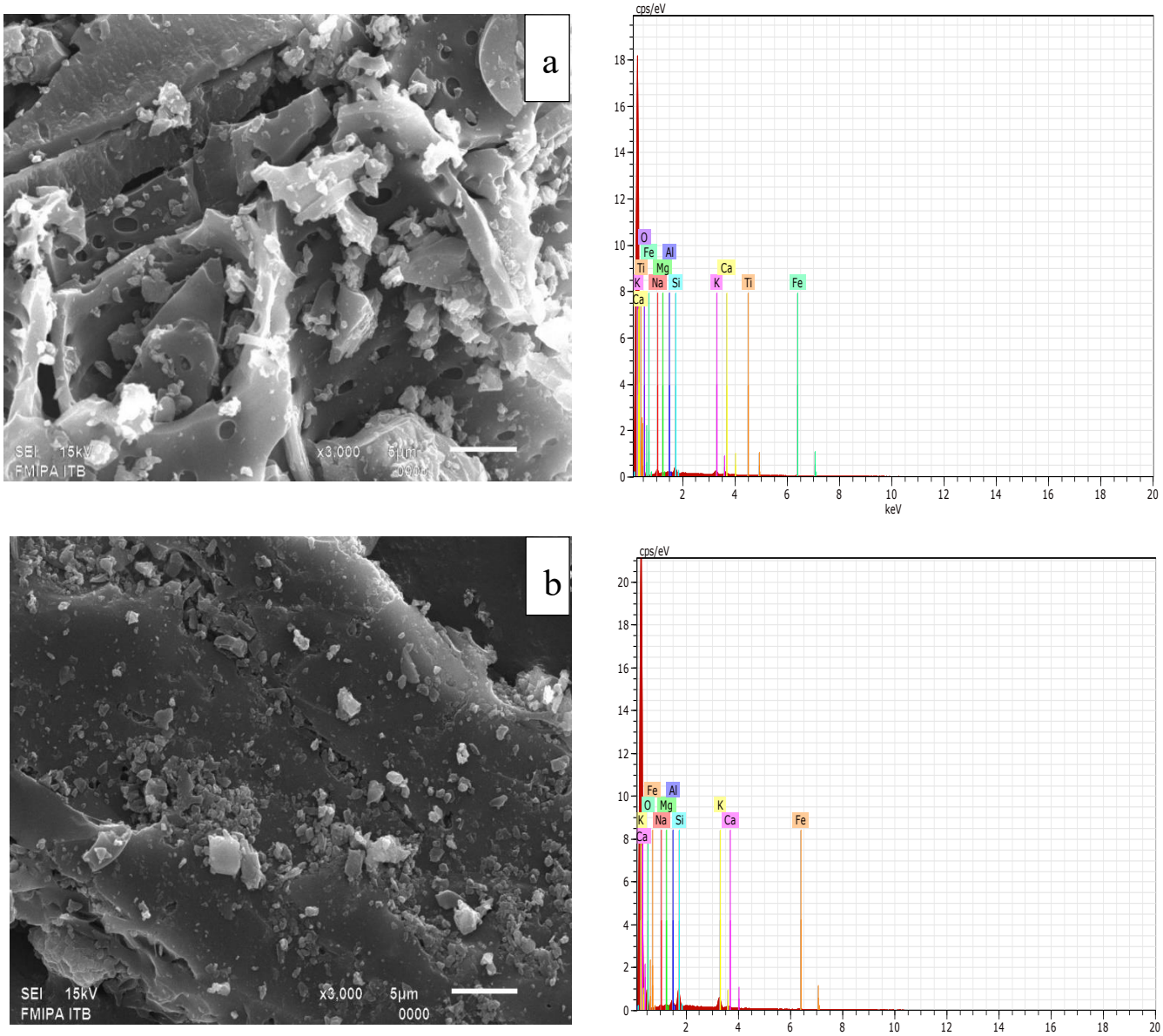

Figure 2. SEM morphology with the magnification of 3,000 times and EDX Spectrum analysis: (a) Na_2CO_3 and (b) 1,000ºC

IV. CONCLUSION

The XRD spectra of the activated carbon in 1,000 ºC and Na_2CO_3 samples contained some elements, respectively e.g. silicate minerals, iron ore and quartz. Other peaks indicate the formation of carbon and graphite structures in the hkl plane (002) and (100). By the Scherrer's method, the average size of the Na_2CO_3 sample crystal is 15.0285 nm and the average size of the 1,000 ºC sample crystal is 54.53 nm. The size of Nano-scale crystals is formed when the temperature increases at the level of ≥ 600° C. The percentage of the volume fraction (*Fv*) of carbon content of the 1,000ºC sample is 75.61 % and the Na_2CO_3 sample is 77.87 %. This percentage means the carbon content of chemical activation is much better than in that physical activation. The results of SEM with the magnification of 3,000 times of 10 µm image size, it is very clear that the porosity in Na_2CO_3 sample is 0.8 µm and in 1,000º C sample is 1.00 µm, proving that the carbon activation processes in this research are successful.

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