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CHARACTERIZATION OF CARBON NANOCRISTRAL STRUCTURE BASED ON CORN COB CHARCOAL

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ABSTRACT

Carbon has an amorphous structure and a crystalline structure. The amorphous structure of carbon is usually found in charcoal, while the crystalline structure of carbon can be obtained from heat treatment. In the present study, the synthesis of carbon nanocrystals based on corn cob charcoal was successfully carried out. The synthesis began with the carbonization process of corn cobs to produce charcoal. Corn cob charcoal powder was then put into 80 mL of HCl solution and stirred using a magnetic stirrer by a speed of 750 rpm at room temperature and 80 mL of NH4OH solution was titrated into it. After the synthesis, the carbon powder was calcined at 400°C and activated using PEG 2000 template. The samples were tested using XRD (X-ray Diffraction) and SEM-EDX (Scanning Electron Microscope-Energy Dispersive X-ray). The carbon component (C) from the EDX test after the synthesis and carbonization process had an atomic percentage of 56.89% and increased by 81.06 % after PEG 2000 activation. The results of the X-ray diffraction pattern show that in all samples a broad and weak diffraction pattern was the characteristic of amorphous carbon. However, on carbon heated for 5 hours at 400°C and the addition of PEG 2000 activator, the crystal structure pattern with higher diffraction peaks was obtained and the peaks of diffraction were matched with CIF data 9008569 from phase C Graphite which had a space group P of 63 mc. SEM data on the morphology of the material showed that after receiving PEG activator, the carbon particles were split into smaller ones so that it increased in surface area and showed fairly even distribution of pores which was also seen in the surface morphology of the carbon.

Keywords: Charcoal powder; corn cobs; nanocrystal; structure.

Introduction

Corn cobs are an important part of corn because of their high benefits, one of which is potential to overcome air pollution problems such as heavy metals.[1,2] Most of the constituents of cob cell walls consist of hemicellulose, protein structure, lignin, tannin content, to cellulose which is not easily broken or soluble in water.[3] The composition and content of corn cobs are 0.014% starch; 1.5% ash; 3% Pectin; 6% Lignins; 9.6% water; 36% Hemicellulose; and 41% Cellulose.[4]

Nanotechnology has potential in developing health, food systems, textiles, agriculture, materials, communications, energy sectors, and information technology has been investigated in a number of developing countries at the nanoscale. The material modification can be performed to create materials that have the desired size, structure, and properties more effectively and efficiently.[5] Materials can be modified and controlled in terms of shape, size, surface functionalization, and chemical properties if it's all in the form of nanoparticles. This makes the uniqueness of the nanoparticle material.[6] Activated carbon with a pore size of less than 100 nm is known as carbon nano porous. Carbon nanopores physically consist

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of a solid material containing carbon (matrix) and empty cavities (pores). The activation process can increase the number and size of pores. The activation process can be done in several ways including physics, chemistry, and templates.[7] The activator functions to oxidize surface molecules so that carbon undergoes changes in physical and chemical properties with an increase in surface area and better absorption ability.

Carbon can be produced from organic materials through the process of carbonization and pyrolysis combustion.[8] The process of heating without oxygen in carbonization is carried out to be able to decompose organic compounds. Therefore, carbon volatile components can be removed and its element can be retained.[9,10] Charcoal that is removed volatile components and water will produce impure carbon.[11,12] Activated carbon is different from ordinary charcoal. The surface area produced by activated carbon can be used as an absorbent material. Any form of crystal structure, the equal crystal structure is Hexagonal Close-Peaked (HCP) formed on graphite and activated carbon. However, the graphite crystal structure is more regular than that of activated carbon.[13] Graphite can be formed from corn cobs heated by the carbonization method at a certain temperature.[14] However, each carbonization process for the formed carbon phase needs to be studied further.

Carbon nanoscale has been successfully fabricated from corncob charcoal using HCl and NH_4OH activator. Besides being used as an activator, carbonization was also carried out at 400°C for 5 hours which resulted in a particle size of 250 nm.[15] In the preparation of nano powder size and crystal formation can also be obtained from the addition of PEG to the sample.[16]

The synthesis method begins with the carbonization process. The first stage is heating which is carried out at low temperatures to reduce the moisture content and destroy the crystalline structure. In the second stage high temperature is used to convert the carbon source into carbon particles.[17] whereas, nanoparticles can be obtained from the coprecipitation method. Activation is done using a PEG template. The structure of activated carbon was tested using X-Ray Diffraction (XRD) and SEM-EDX.

Methods

2.1 Synthesis of corn cob powder

Corn cob powder was obtained by drying of corn cobs at the temperature of 100°C which had previously been cleaned of corn kernels. Corn cobs were ground to a fine powder measuring 200 mesh. Corn cobs powder weighed as much as 10 grams put into a 500 mL beaker glass. Then 80 mL of HCL solution as activator was added. The mixture was stirred at room temperature for 20 minutes at a speed of 750 rpm. Next step, dissolving 80 ml of NH_4OH solution by titration into the mixture. Next, stirring was carried out at room temperature for 30 minutes at a speed of 720 rpm. The next stage was the washing process using aquadest to pH 7 on the stirring mixture. After that, the filtration process as carried out by taking the sample precipitate and followed by the drying process by heating it at a temperature of 100°C in an oven until it became powder. Then the calcination temperature at 100°C and 400°C was carried out.

2.2 Fabrication of PEG/carbon composite

At the stage of mixing the activated carbon sample with Polyethylene Glycol (PEG) 2000. PEG as a non-ionic surfactant capable of absorbing fine particles in solution through hydrogen bonding and electrostatic attraction. 1 gram of PEG 2000 and 40 ml of distilled water were mixed and heated at 90°C at 700 rpm. The process of stirring the solution was carried out for 30 minutes. Next, 20 ml of PEG 2000 solution was mixed with 3 grams of activated carbon sample which had been heated at 400°C and stirred for 1 hour at 750 rpm. The precipitate obtained from the filtering process was dried into powder.

2.3 Analysis of carbon

Phase of activated carbon samples was analyzed after the coprecipitation process and the mixing process of activated carbon with PEG 2000. The characterization test used

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XRD at an angle of 2θ 10°-90° with a step size of 0.02. Based on the diffraction pattern data, the phase identification process was carried by the references matching and determining the lattice parameters using Rietica software. The SEM-EDX test was used to determine the constituent elements and the morphology of activated carbon.

Result and Discussion

3.1 EDX Analysis

After synthesis, EDX test was carried to find out the percentage of components. From the EDX results, the highest atomic percentage in corn cob charcoal was found as carbon (C). Corn cob charcoal powder heated at 100°C has a carbon atom percentage (C) of 72.69%. The abatement in the percentage value of carbon atoms (C) by 56.89% occurred after the carbonization process with a holding time of 5 hours at a temperature of 400°C. The decrease in the percentage of atomic value is due to oxygen binding to carbon so that it can tune out the total mass of corn cob charcoal. Corn cob charcoal powder heated for 5 hours at 400°C and received PEG 2000 template activation had an increased percentage of carbon atoms (C) by 81.06%. An increase in the percentage of carbon atoms occurred when PEG activator was added. The carbon particles split into smaller ones and show an enhancement in surface area. With the increasing surface area, the distribution of carbon atoms becomes more even so there is an enhancement in the percentage of carbon atoms on the surface. As for inorganic components have contents in the corn cob charcoal are Aluminum (Al), Oxygen (O), Silica (Si), Potassium (K), and Magnesium (Mg).

3.2 XRD Test Results

The test results using XRD show the diffraction pattern of activated carbon corn cob charcoal in Figure 1.

Table 1. EDX test results on corn cob charcoal powder (a) heated at 100°C, (b) heated for 5 hours at 400°C (c) heated for 5 hours at 400°C with PEG 2000 template activation

Elements	a		b		c	
	Mass (%)	Atom (%)	Mass (%)	Atom (%)	Mass (%)	Atom (%)
C	64.63	72.69	45.70	56.89	74.29	81.06
O	28.85	24.36	38.75	36.21	20.19	16.54
Other elements	06.52	02.95	15.55	06.90	05.52	02.40

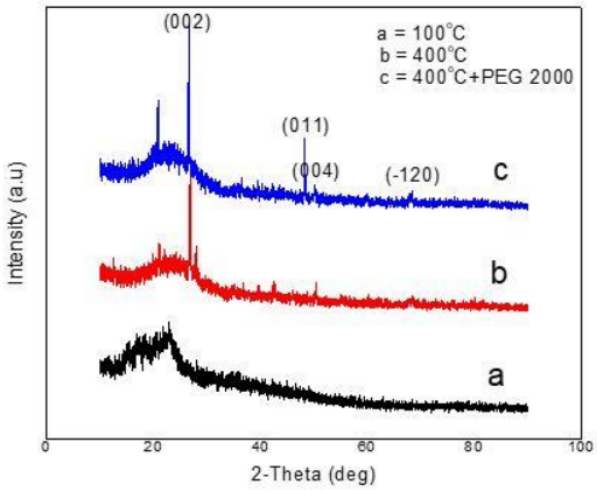

Figure 1. X-ray diffraction pattern on corn cob charcoal powder (a) heated at 100°C, (b) heated for 5 hours at 400°C (c) heated for 5 hours at 400°C with PEG 2000 template activation

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Analysis of synthesized corn cob charcoal powder included heating at a temperature of 100°C in Figure 1(a) to form an amorphous carbon structure at an angle of 2θ = 10°-27° with a peak angle of 2θ = 14.85°. Broad and weak diffraction pattern are characteristic of amorphous carbon. Figure 1(b) is a diffraction pattern of activated carbon from corn cob charcoal powder heated for 5 hours at 400°C and showed a higher diffraction peak and the peak match of the diffraction results obtained with CIF data 9008569 from phase C Graphite which has a space group P of 63 mc at position 2θ= 26.603°, 44.666°, 54.749°, 77.699°. In addition, the activated carbon of corn cob charcoal powder heated for 5 hours at 400°C showed an increase in the degree of crystallinity. Figure 3(c) is a diffraction pattern of activated carbon from corn cob charcoal powder heated for 5 hours at 400°C and received PEG 2000 template activation and shows the previous peak has a higher intensity than the carbon peak heated for 5 hours at 400°C. The suitability of the diffraction peak with CIF data 9008569 from phase C Graphite has a space group P of 63 mc at position 2θ= 26.603°, 44.666°, 54.749°, 77.699°. From Figure 1, the hkl (002) and (001) showed an increasing of intensity which indicated the degree of crystallinity. The increasing of intensity in sample (b) of activated nanocarbon was caused by the addition of HCl and NH4OH activators as well as carbonization. Furthermore, the addition of PEG surfactant also increased the intensity of hkl (002) and (011) which was shown in the diffraction pattern of the sample (c). the PEG can increase the intensity of the crystals formed because the PEG has the ability to reduce and prevent the appearance of clumps between particles [16]. In addition, the peak shift appeared and were caused by the atomic activation energy in increasing variations in carbonization temperature.

The formed phase and lattice parameters of the crystal structure can be obtained from the XRD test results. Rietica is the software used for analysis. Lattice parameters can be seen in samples that have a degree of crystallinity. This shows the lattice parameters on the activated carbon sample of corn cob charcoal powder heated for 5 hours at 400°C and corn cob charcoal activated carbon heated for 5 hours at 400°C plus PEG 2000 template in Table 2.

Table 2. Results of Rietica Analysis of Corn cob Charcoal Powder

Lattice Parameters	400°C 5 hours	400°C 5 hours + PEG 2000
a=b (Å)	2.481	2.459
c (Å)	6.807	6.701
Vol. cell (Å^3)	35.428	35.109

Rietica analysis showed that the phases of corn cob charcoal powder heated for 5 hours at 400°C and corn cob charcoal powder heated for 5 hours at 400°C with the addition of PEG 2000 activator resulted in a, b and c of lattice parameters. This is in accordance with the resulting diffraction peak, i.e a shift caused by increasing the heating temperature so that the atoms making up the activated carbon vibrate and then are bound and transferred to the other side. This causes the energy of these atoms to exceed the activation energy and substitution occurs moving towards the vacancy.[18]

The analysis using Rietica software indicated that the corn cob charcoal powder phase heated for 5 hours at 400°C was compatible with c graphite which has a hexagonal structure. The lattice parameter shows the size of the cell volume of the sample was 0,035428 nm^3. Corn cob charcoal powder heated for 5 hours at 400°C with the addition of peg 2000 activator has a match with c graphite which has a hexagonal structure with a cell volume size of 0.035109 nm^3. The size of the cell volume in the sample added with PEG 2000 activator was smaller. This can be caused by the addition of peg activator which can break down the carbon size into smaller ones. PEG activator functions to oxidize surface molecules so that carbon undergoes changes in physical and chemical properties with increasing surface

area and better absorption ability. This indicates that the activated carbon phase of corn cob charcoal powder heated for 5 hours at 400°C and corn cob charcoal powder heated for 5 hours at 400°C with the addition of peg 2000 activator has a degree of crystallinity in nanocrystal size. To be able to ascertain the size of the crystals in the sample, the analysis was carried out using the MAUD device.

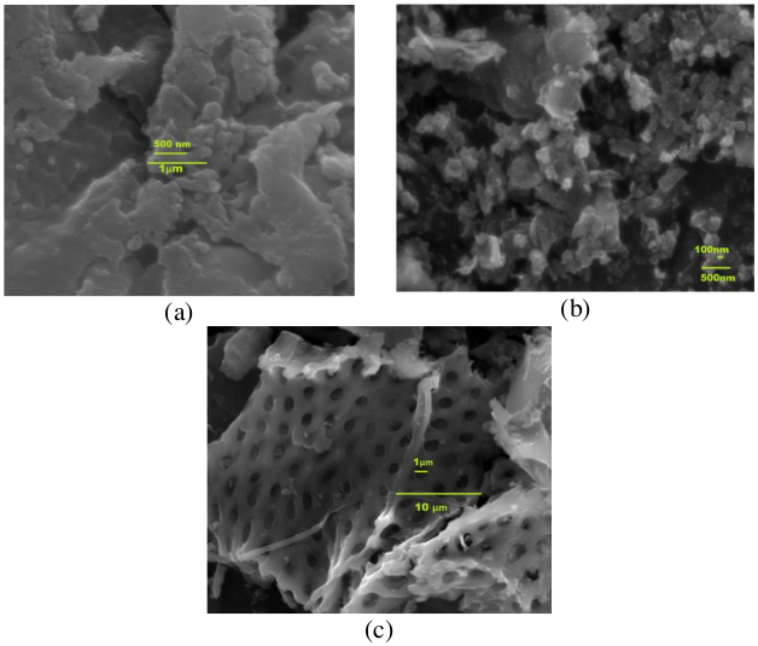
3.3 Crystal Size Results

Analysis of crystal size on samples of activated carbohydrate from corn cobs charcoal powder was performed by using MAUD software.[19] In order to analyze the crystal size, the sample must have a crystalline structure. The activated carbon sample of corn cob charcoal powder heated at 100°C showed an amorphous structure. Thus, samples that can be analysed in crystal size with the MAUD device were activated carbon samples of corn cob charcoal powder heated for 5 hours at 400°C and corn cob charcoal powder heated for 5 hours at 400°C with the addition of PEG 2000 activator as shown in Table 3.

MAUD analysis showed that the crystal size of activated carbon from corn cob charcoal powder heated for 5 hours at 400°C had a size of 1015.858 Å or ± 100 nm. Meanwhile, corn cob charcoal powder heated for 5 hours at 400°C with the addition of PEG 2000 activator has a size of 1087.037 Å or ± 100 nm. This shows that the carbonization temperature, type of activator and carbonization time are factors for the formation of carbon nano. The size of the carbon in the form of nano will make the carbon have a wider and more stable surface area.[20] Therefore, nano activated carbon is better used as an adsorbent.

Table 3. Crystal Size of Activated Carbon Corn cob Powder

Parameter	400°C 5 hours	400°C 5 hours + PEG 2000
Size (Å)	1015.858	1087.037
Microstrain	0.031	0.033
Size distribution	5.743	2.658



(a) (b)

(c)

Figure 2. SEM results on corn cob charcoal powder (a) heated at 100°C, (b) heated at 400°C for 5 hours, (c) heated for 5 hours at 400°C and activated PEG 2000 template

3.4 SEM Analysis

SEM testing was carried out to determine the morphology of the particles of activated carbon from corn cob charcoal powder as shown in Figure 2.

From the SEM image in Figure 2(a) the carbon heated at a temperature of 100°C had a larger particle size than that of carbonized for 5 hours at a temperature of 400°C (Figure 2(b)). The morphology of activated carbon from corn cob charcoal powder heated for 5 hours at 400°C showed particle size with an average size of 100 nm and a more even distribution of particles.

When carbonization at 400°C for 5 hours received the addition of PEG 2000 activator, the pore distribution was even and can be seen quantitatively as shown in Figure 2(c). This shows that the addition of PEG 2000 activator serves to oxidize surface molecules so that the carbon changes its physical and chemical properties with the addition of surface area and better absorption ability.

Conclusion

The carbon component (C) from the EDX test after the synthesis and carbonization process had an atomic percentage of 56.89% and an increase of 81.06 % after PEG 2000 was activated. The results of the X-ray diffraction pattern showed that all samples showed a broad and weak diffraction pattern and is a characteristic feature amorphous carbon. However, on carbon heated for 5 hours at 400°C and the addition of PEG 2000 activator, the crystal structure pattern was seen with higher diffraction peaks. In terms of carbon morphology, after being given PEG activator, the carbon particles split into smaller ones so that the surface area increased. The morphology of the pore distribution that is fairly even on the carbon surface is also evident from the SEM results.

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