Synthesis and Characterization of Nano Activated Carbon of Corn Cob Charcoal as an Adsorbent of Health Masks

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Abstract. Corn is widely grown by Indonesians as a carbohydrate-producing food. In every major harvest, a lot of corn waste, such as the cobs, has not been used properly. Therefore, in this study, corn cobs will be used as one of the adsorbent materials for health masks. Corn cobs are synthesized into activated carbon with variations in carbonization temperature to form a structure with the ability as an adsorbent. Corn cobs were synthesized using the coprecipitation method of HCl and NH_4OH followed by the carbonization process with temperature variations of 100°C and 400°C with a holding time of 5 hours. Material characterization using DTA TG (Differential Thermal Analysis-Thermogravimetry), XRD (X-ray Diffraction), FTIR (Transform Infrared Spectrometer), and SEM-EDX (Scanning Electron Microscope-Energy Dispersive X-ray). The results from DTA-TGA show that at temperatures above 250°C there is no mass addition and it is constant up to a temperature of 400°C. So that it becomes an indicator that the desired phase is formed. The X-ray diffraction pattern of activated carbon corn cob charcoal powder with a carbonization temperature of 100°C has an amorphous structure and the carbonization temperature of 400°C corresponds to the phase crystal structure graphite C hexagonal with a crystal size of 100 nm. From the results of the FTIR test, it is shown that the typical components of activated carbon are O-H, C-O, C=C, and C-H functional groups. The C=C group of the aromatic ring is a typical compound of activated carbon. And the morphology of the SEM test results of activated carbon corn cob charcoal powder at a carbonization temperature of 100°C shows the distribution of small particles with an average particle size of 200 nm and at a carbonization temperature of 400°C shows a more even distribution of particles and the surface area increases with a particle size on average is 100 nm. So that the activated carbon nanocrystals of corncob charcoal powder as a result of this study can potentially be used as an adsorbent in the manufacture of health masks.

INTRODUCTION

Corn cobs are part of corn waste that has not been used properly. From the results of field observations, it was found that corn waste in the form of husks, stalks and cobs was not appropriately utilized. Dried corn waste is only thrown away or burned, while the young leaves and stems are used as animal feed. The structural components of corn cobs consist of cellulose (41%), hemicellulose (36%) and lignin (6%). So, corn cobs are potentially and effectively used for the carbon production, besides that the ash content contained in corn cobs is also relatively low, namely 1.50%[1].

Carbon is a material that has various morphologies, including colloidal carbon[2], nanotube[3], fullerence[4], graphite[5], graphene[6], nanofiber[7], porous carbon[8], and activated carbon[9]. Activated carbon is a compound carbon or charcoal material specially treated to obtain high adsorption power. The structure of activated carbon and graphite has the same form, namely HCP (Hexagonal Close-Peaked). Activated carbon can adsorb certain gases and chemical compounds, or its adsorption properties are selective, depending on the size or volume of the pores and surface area. The absorption of activated carbon is extensive, which is 25-100% by weight of activated carbon[10]. Many studies have been conducted

on the use of activated carbon as an adsorbent. One of them is done by adding activated carbon to the porous material, namely ceramics, which have small cavities so that the fluid can enter the membrane. In addition, ceramics are used as filters because they are relatively resistant to high-temperature changes, corrosion, and contamination of other materials[11].

Nanoparticles have potential in the application of nanotechnology development. In some developing countries, nanoscale materials are mainly carried out in technology development to modify materials with the desired size, structure, and properties more effectively and efficiently[12]. Activation of nano-carbon can be influenced by factors including carbonization temperature, type of activator, activator concentration, and carbonization time. A suitable activator for lignocellulosic materials is an acidic activator, such as $ZnCl_2$, HCl, and H_3PO_4, compared to an alkaline activator-like KOH[13]. Research conducted by Guler produced activated carbon from tea dregs, increasing carbonization temperature and longer carbonization time[13]. The carbonization temperature of 400°C for 30 minutes has 62.24%, at a temperature of 800ºC for 30 minutes, it produces 72.14%, while at a temperature of 800ºC for 120 minutes, it produces 82.54%. Carbon has a surface area wider and becomes stable if the size is nano[14]. To obtain nanoparticles, many coprecipitation methods have been developed on natural materials, such as rocks, mining materials, and other inorganic materials. But it has not developed on corn cobs with a high cellulose compound content, 41%[15], where cellulose is a source of carbon[16].

Further research is needed to characterize activated carbon nanoparticles from corn cobs as an adsorbent for mask materials. In this study, corncob-activated carbon as an adsorbent in the mask material will be carried out. Activated carbon with temperature variations will be characterized so that the structure formed can be known.

METHODOLOGY

The materials used in this study were corn cob charcoal powder, aqua dest, HCl, and NH_4OH. Sample preparation was carried out by drying the corn cobs and cleaning the fiberes until only the corn cobs were left. The corn cobs are then burned until they become charcoal. Corncob charcoal is powdered using a blender and mortar and then sieved with a 200mesh sieve. Corncob charcoal powder is made into a powder so that the heat distribution when the carbonization process starts is more even or homogeneous.

In the preparation process, samples of sifted corncob powder and corncob charcoal powder were weighed 10 grams each, mixed with 80 ml of HCl, and then stirred for 20 minutes. After 20 minutes, NH_4OH was titrated to 40 ml and kept in the stirrer for 30 minutes. Once that is done, wash the precipitate with distilled water until pH 7. Next carbonization process, charcoal powder raw corn cob was dried (drying) in advance at 110°C. The method of drying uses an oven. Corncob charcoal powder nanoparticles were then carbonized at 100°C and 400°C with a holding time of 5 hours in a furnace in free air. Through this process, we can be seen the content of the carbon phase formed in it.

Characterization of materials using DTA TG (Differential Thermal Analysis-Thermogravimetry) for testing thermal properties, XRD (X-ray Diffractometer) for determining the structure of the material, FTIR (Transform Infrared Spectrometer) for determining functional groups, and SEM-EDX (Scanning Electron Microscope-Energy Dispersive X-ray) for determining morphology and elements contained in the material.

RESULT AND DISCUSSION

DTA TG Test Results

In this study, the DTA TG test was used to determine the amount of temperature of the activated carbon phase of corncob charcoal powder was formed. The graph of the DTA TG test results is shown in Figure 1.

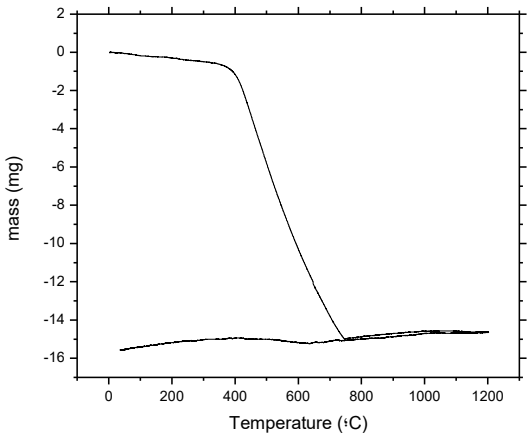


FIGURE 1. Graph of DTA-TG Test Results for Corn Cob Charcoal Powder.

Figure 1 shows a graph of the endoderm process at temperatures between 0-200°C. In the endoderm process of 0-200°C, there was a reduction in mass by requiring energy to release water into the material. There was a reduction in abundance from the unwanted phases. Then, when the temperature is above 250°C, there was no decrease in mass, and it was constant up to a temperature of 400°C. So, this was an indicator that the desired phase was formed. However, at a temperature of 400-700 C, there was a drastic decrease in mass. This was also expressed by Demiral in their research on variations in carbonization temperatures of 400°C, 500°C, and 600°C. His research found that the surface area of activated carbon increases below 500°C because more volatile compounds are released as the temperature increases. It helps open the pores and increase the surface area. Meanwhile, the pores widen at temperatures above 500°C, which destroys the already formed pore structure[17,18]. From several previous studies and the results of the DTA TG test, this study used variations in carbonization temperature at 100°C and 400°C for 5 hours.

XRD Test Results

The XRD profile of the activated carbon sample of corncob charcoal powder as an adsorbent is shown in Figure 2. In Figure 2, It can be observed that the diffractogram of the activated carbon synthesis sample of corncob charcoal powder has the appearance of a broad diffraction background, varying background intensity, and the absence of sharp peaks. Figure 2 (a) shows the diffraction pattern of activated carbon charcoal powder corncob-heated at 100°C which has a broad peak that indicates amorphous structures. Figure 2 (b) is an active carbon powder diffraction pattern corncob charcoal-heated temperature of 400°C for 5 hours shows the diffraction peaks are higher.

Figure 2 (a) activated carbon of corncob charcoal powder shows an amorphous structure corresponding to the phase of C Graphite with secondary data PDF (Powder Diffraction File) 96-901-2234. In Figure 2 (b), activated carbon charcoal powder corncob heated temperature of 400°C for 5 hours diffraction results obtained peak conformance with 9,011,577 CIF data from phase C Graphite which has a space group P 63 / m m c at position 2θ= 26,79°, 43,47°, 57,00°. In addition to the activated carbon charcoal powder, the corncob heated temperature of 400°C for 5 hours showing an increase in the degree of crystallinity in the sample. This indicates that the activation and carbonization treatment affects the formation of activated carbon and the degree of crystallinity.

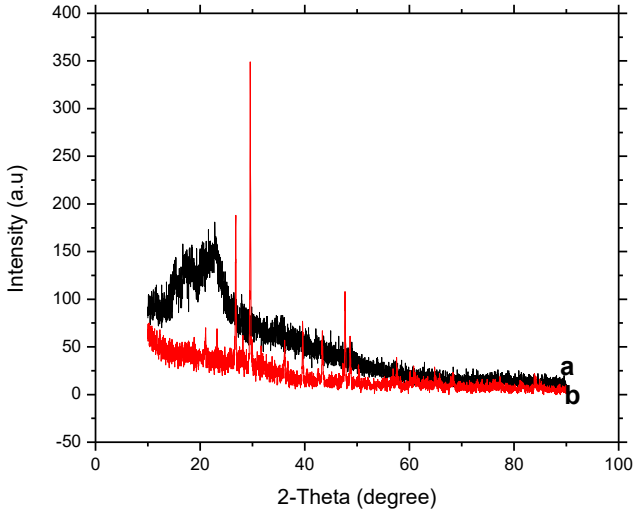


FIGURE 2. X-Ray Diffraction Pattern on (a) corncob charcoal powder heated at 100°C and (b) corn cob charcoal powder heated at 400°C for 5 hours.

For the analysis of the lattice parameters on the sample, the device used *Rietica*. Lattice parameters can be seen in samples that have a degree of crystallinity. So here can be shown the lattice parameters on the activated carbon sample of corncob charcoal powder heated at 400°C for 5 hours in Table 1.

TABEL 1. Results of *Rietica* Analysis

Parameters of Grating	Corn Cob Charcoal Powder 400°C 1 Hour
a=b (Å)	2.44
c (Å)	6.56
Vol. cell	33.85 ± 0.47

From the analysis results using *Rietica*, it was shown that the corncob charcoal powder phase heated at 400°Cf or 5 hours was compatible with *C* Graphite which has a hexagonal structure. From the lattice parameters, the size of the cell volume of the sample is 33.845280 ± 0.465609 Å. This may indicate that the phase-activated carbon charcoal powder corncob heated at 400°C for 5 hours has a degree of crystallinity in the nanocrystal size. To ascertain the size of the crystals in the sample, an analysis was carried out using the *MAUD* device.

Crytal Size Results

The MAUD software was used to analyze the crystal size of the sample of activated carbs from charcoal powder[19]. To be able to explore the crystal size, the sample must have a crystalline structure. In the activated carbon samples, corncob charcoal powder was heated at 100°C showed an amorphous structure. So, it can be analyzed with the crystal size MAUD device is activated carbon samples corncob charcoal powder which is heated to 400°C for 5 hours are shown in Table 2.

TABEL 2. Size of activated carbon crystals of corn cob charcoal powder heated at 400°C for 5 hours

Corn cob charcoal powder 400°C 1 Hour	Result
Size (Å)	1000.017
Micro strain (x 10^{-4})	2.046
Size distribution (x 10^{-4})	5.253

The MAUD analysis results showed that the crystal size of activated carbon from corn cob charcoal powder heated at 400°C for 5 hours had an s of 100 nm. This indicates 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 broader and more stable surface area[13]. so that nano activated carbon is better used as an adsorbent.

FTIR Test Results

FTIR (*Transform Infrared Spectrometer*) can determine the functional groups in each corncob activated carbon based on the absorption peaks produced in Figure 3.

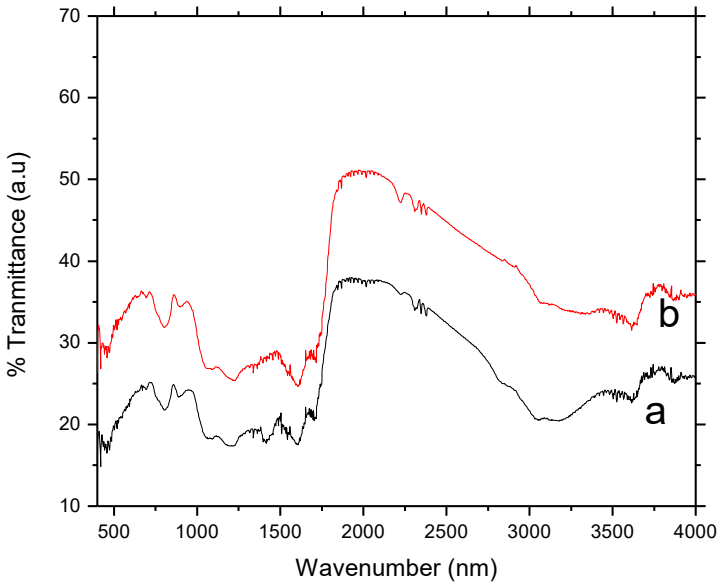


FIGURE 3. FTIR spectra on (a) corn cob charcoal powder heated at 100°C and (b) corn cob charcoal powder heated at 400°C for 5 hours.

The resulting spectrum pattern is the absorption of vibrations in the cell. From the result of the spectrum pattern, several absorption results of the functional groups of activated carbon are shown in Table 3. Table 3 shows the FTIR spectra analysis on activated carbon which indicates the presence of functional groups OH, CO, C=C, and CH, which are the components that make up activated carbon[20]. There was a shift in wave number and a change in intensity between the spectrum pattern of the synthesis of corn cob charcoal powder heated at 100°C and the synthesis of corn cob charcoal powder heated at 400ºC for 5 hours. At wave numbers, corncob charcoal powder was heated to 100°C in the wave number area 3207,62 - 3419,79 cm^{-1} indicating the presence of hydroxyl OH groups with an intensity of 34,637 – 34,7. Meanwhile, in corn cob charcoal powder synthesized and heated at 400°C for 5 hours, there was an OH functional group in the wave number region of 2980,02 and 3209,55 cm^{-1} with an intensity of 46,763 and 47,437. The OH functional group of corncob charcoal powder heated 400°C for 5 hours is greater than that of corn cob charcoal powder heated 100°C. Besides the OH group, there is also a CH group at wave number 2929,87 cm^{-1} with an intensity of 38.638 in powder corncob charcoal at 100°C and at wave numbers 2875,86, 1460,11, and 1417,68 cm^{-1} with an intensity 47,633, 26,272, and 27,101 on corn cob charcoal powder heated to 400°C for 5 hours. In carbon charcoal powder corncob nanocrystal synthesized and dried at 100°C (Table 3), it also appears strain C=C bonds are included in the range of wave numbers 1514,12 and 1606,7 cm^{-1}. In contrast, the corn cob charcoal nanocrystal powders are heated at 400°C for 5 hours to appear in functional groups C=C in the range of 1795.73 to 2260.57 cm^{-1} wave-number. The absorption peak indicates the presence of a C=C group from the aromatic ring which is a typical compound of activated carbon. The C=C functional group can be formed by the decomposition of the CH bond and the C=O group conjugated with an aromatic ring[21].

TABLE 3. The results of the suitability of the value of the wave number to the type of functional group

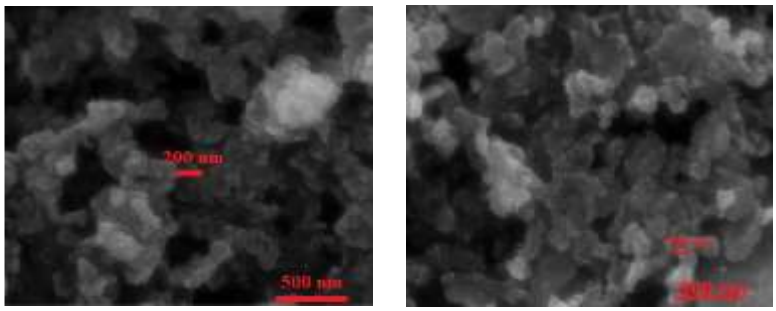
Corncob Charcoal Powder Heated to 100°C				Corncob charcoal powder heated to a temperature of 400°C for 5 Hours			
Wave number (Cm^{-1})	**Intensity**	**Bond**	**Functional Group**	**Wave number (Cm^{-1})**	**Intensity**	**Bond**	**Functional Group**
611,43	35,7	=C-H out of bending	*Alkena*	696,3	30,962	=C-H out of bending	*Alkene*
759,95	41,121	=C-H out of bending	*Alkena*	779,24	31,087	=C-H out of bending	*Alkene*
860,25	45,086	=C-H out of bending	*Alkena*	875,68	27,48	=C-H out of bending	*Alkene*
1072,42	30,901	C-O	Alcohol & Phenol	1033,85	20,953	C-O	Alcohol & Phenol
1153,43	33,365	C-O	Alcohol & Phenol	1417,68	27,101	C-H	Alkane
1242,16	37,347	C-O	Alcohol & Phenol	1460,11	26,272	C-H	Alkane
1514,12	44,633	C=C	Aromatic ring	1795,73	47,811	C=C	Aromatics
1606,7	44,816	C=C	Aromatic ring	2140,99	62,721	C=C	Alkyne
2929,87	38,638	C-H	Alkane	2260,57	60,949	C=C	Alkyne
3207,62	35,981	O-H	Alcohol & Phenol	2513,25	53,875	O-H	Carboxylate Acid
3246,2	34,637	O-H	Alcohol & Phenol	2875,86	47,633	C-H	Alkane
3284,77	33,583	O-H	Alcohol & Phenol	2980,02	46,763	O-H	Carboxylic Acid
3305,99	33,357	O-H	Alcohol & Phenol	3209,55	47,437	O-H	Alcohol & Phenol
3419,79	34,7	O-H	Alcohol & Phenol				

The presence of OH and CO bonds indicates that the activated carbon produced tends to be more polar. Thus the activated carbon produced can be used as an adsorbent for substances that are typical to be opposite such as for the purification of water, sugar, alcohol, or as an absorbent for formaldehyde emissions[22]. So, activated carbon nanocrystals of corn cob charcoal powder can be potentially an adsorbent in the manufacture of health masks.

The wave number region 1072.42-1242.16 cm^{-1} indicates the presence of CO carbonyl alcohol and phenol groups. =CH functional groups alkene on the corn cob charcoal powder appears in the range of 611.43 to 860.25 cm^{-1} wave number while the corn cob charcoal powder is heated to 400°C for 5 hours to appear on the wave number range from 696,3-875,68 cm^{-1}. The distribution spectrum pattern resulting from the synthesis of the corn cob charcoal powder is heated at 100°C with corn cob charcoal powder heated to 400°C for 5 hours showed a shift wave number and a big difference in intensity. This is due to the addition of an HCl activator and the effect of carbonization heating temperature.

SEM Test Results

Testing on Scanning Electron Microscopy was carried out to determine the morphology of the particles from activated carbon corncob charcoal powder, as shown in Figure 4.



(a) (b)

FIGURE 4. SEM test results on (a) corn cob charcoal powder heated at 100°C and (b) corn cob charcoal powder heated at 400°C for 5 hours with a magnification of 100,000x.

Figure 4 (a) is an activated carbon of corn cob charcoal powder heated at 100°C which shows the distribution of small particles with an average particle size of 200 nm. This has seen the formation of activated carbon nanoparticles, but the distribution of the particles is still uneven, and there are still many large pores. Figure 4 (b) is an activated carbon charcoal powder morphology corncob heated to 400°C for 5 hours showing that distribute particles are more evenly distributed with an average particle size of 100 nm. An increase in carbonization temperature and holding time shows that the pores are open, and the surface area is increased. So, this indicates that the carbonization temperature, type of activator, and carbonization time are factors for the formation of nano carbon.

CONCLUSION

The results of the DTA TGA showed that at temperatures above 250°C, there was no decrease in mass and was constant up to a temperature of 400°C. So, this was an indicator that the desired phase was formed. X-ray diffraction pattern of activated carbon charcoal powder corncob heated at 100°C showed an amorphous structure corresponding to the phase *C Graphite* with secondary data PDF (*Powder Diffraction File*) 96-901-2234. While on activated carbon charcoal powder corncob, the heated temperature of 400°C for 5 hours showed an increase in the degree of crystallinity obtained suitability peak results diffraction data CIF 9011577 of *C Graphite* phase which has the space group P 63/m m c at position $2\theta = 26{,}79^0, 43{,}47^0, 57{,}00^0$. The analysis results using MAUD showed that the crystal size of activated carbon from corn cob charcoal powder heated at 400°C for 5 hours had a length of 100 nm. The results of the FTIR spectra analysis on activated carbon showed the presence of functional groups O-H, C-O, C=C, and C-H which are the components that make up activated carbon. The C=C group of the aromatic ring is a typical compound of activated carbon. Morphology of SEM test results Activated carbon of corn cob charcoal powder was heated at 100°C showing the distribution of small particles with an average particle size of 200 nm. While the activated carbon of corn cob charcoal powder heated at 400°C for 5 hours showed a more even distribution of the particles. The surface area increased with an average particle size of 100 nm. So, activated carbon nanocrystals of corn cob charcoal powder can be potentially an adsorbent in the manufacture of health masks.

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