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Characterization by infrared spectroscopy (FTIR) of corncob powder

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ABSTRAK

Corn cobs contains 40-45% cellulose, 30-35% hemicellulose, and 10-20% lignin, while corn cobs ash contains more than 60% silica with small amounts of metal elements. Silica in corn cob powder can be isolated thermally and nonthermally. Nonthermally silica can be separated with HCl. This research was conducted to isolate silica from corncobs using HCl with variations in calcination temperature and holding time. The silica obtained was then characterized by bonding and its structure using infrared spectroscopy (FTIR). The steps taken were preparing the tools and materials, then preparing the corncob powder, and then preparing silica with HCL and FTIR, as well as data processing and analysis. This study concludes that corncob powder produces silica by isolating it using HCL at 70 ºC for 3 hours and calcining at 800 ºC with a holding time of 4 hours. Based on its FTIR characterization, samples soaked in HCL at 70 ºC resulted in absorption caused by several factors, including single NH, CH, OH, triple bonds, double bonds, and single bonds. This is reinforced by the FTIR results, where the peaks are in regions 1, 2, 3, and 4.

Keywords: Calcination, FTIR, insulation, nonthermal, silica

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INTRODUCTION

Corn cobs are one of the lignocellulosic wastes that are widely available in Indonesia. Lignocellulosic waste is agricultural waste containing cellulose, hemicellulose, and lignin. Each is a compound that can be converted into other compounds biologically. *Cellulose* is a carbon source that microorganisms can use as a substrate in fermentation to produce products with high economic value (Simanjuntak, 2014).

Corn cobs are a great source of carbohydrates with a hemicellulose content of 30.91%; alpha-cellulose of 26.81%; lignin of 15.52%; carbon of 39.80%; nitrogen 2.12%; and 8.38% water content (Prasetyawati, 2015).

So far, corn cobs waste has been underutilized or the utilization of corncob waste still needs to be improved. Most corn cob waste is only used as a livestock feed additive or as a firewood substitute. This makes corn cob waste very potential to be applied as an organic filler (Mushlihah, 2013).

According to (Shofiyanto, 2008), the characteristics and composition of corn cobs can be seen in table 1 below:

Table 1. Characteristics and Composition of Corn Cobs

Content	%	Nutrient Amount	%
Water	9,4	Proteins, N x 6,25	2,5
celllulose	41	Fat, ester, dll	0,5
Hemicellulose	36	Coarse fiber	32
Xylan	30	Ash	1,5
Lignin	6	Free Nitrogen Extract	53,5
Pectin	3	*Neutral detergen fiber*	83
starch	0,014	Total nutrient can be digested	42

(Sumber: Shofiyanto, 2008)

Silica is a chemical compound SiO_2 which has been widely used as a dye (Ke et al, 2016), in medicine (Hacene et al, 2016), electronic devices (Sharma et al, 2016), ceramics (Nien et al, 2016), catalyst (Liu et al, 2016), and catalyst support materials (Ewing et al., 2016). This is because silica has properties proven to have high stability, chemical flexibility, and biocompatibility, which play an essential role in various fields (Nandiyanto et al., 2014). Silica in nature can be obtained from minerals and vegetable materials. Research on silica obtained from vegetable materials has been carried out by (Rafiee et al., 2012) obtained nano silica from rice husks, and Rahman et al., 2015) obtained mesoporous silica from bagasse ash.

Apart from rice husks and bagasse, corn cobs are an alternative source of silica from vegetable materials. Corn cob contains 40-45% cellulose, 30-35% hemicellulose, and 10-20% lignin (Velmurugan et al., 2015), while corncob ash contains more than 60% silica with small amounts of metal elements (Adesanya & Raheem, 2009). Silica in corn cob powder can be isolated thermally and nonthermally. Nonthermally silica can be isolated with HCl (Roschat et al., 2016). This research was conducted to isolate silica from corncobs using HCl with variations in calcination temperature and holding time. The silica obtained was then characterized by bonding and structure using infrared spectroscopy.

Infrared spectroscopy is an instrument used to measure the absorbance of infrared radiation at various wave numbers. Most infrared spectra record wavenumber or frequency versus %T. If a compound absorbs radiation at a specific wave number, the intensity of the radiation transmitted by the sample will decrease. This decreases %T and appears in a spectrum as a dip, called the absorption band (Ciesielski, 1999). The use of infrared spectroscopy is used to identify a compound. This is because the FTIR spectrum of a compound is very distinctive, meaning that different compounds will have different spectra. The vibration of chemical bonds in a molecule causes an absorption band almost entirely in the IR region of 4000-450 cm-1 (Silverstein et al., 1981).

The atoms of the molecules move in different ways but always at a certain energy level. The vibrational energy range for organic molecules must match that of infrared radiation with a wave number of 1200-4000 cm-1. There are two kinds of molecular vibrations: stretching and bending. Identifying the characteristic absorption bands caused by various functional groups is the basis for interpreting the infrared spectrum. The O-H bond of the carboxyl group is absorbed in the region from 2500 to 3300 cm-1, and the C=O bond is shown between 1710 to 1750 cm-1. Only vibrations that produce rhythmic changes in the polar moment are observed in the infrared (Tarisma, 2015).

Based on the description in the background that has been explained, the research problem can be formulated: how to characterize the results of infrared spectroscopy for corncobs.

The research aims to characterize the results of infrared spectroscopy for corncobs.

RESEARCH METHODS

This research will use tools and materials, including an infrared spectrometer (Nicolet Avatar 360 FT-IR: Fourier Transform Infra Red), magnetic stirrer, hot plate, oven, furnace, analytical balance, 100 mesh sieve, and glassware. The materials used in this study were corn cobs (Zea Mays Saccharata) of the Bonanza variety obtained from the Bojonegoro area, Bojonegoro Regency), filter paper, distilled water, and 37% HCl (Merck).

The first step was preparing the tools and materials, then preparing the corncob powder, then preparing the silica with HCL and FTIR, then processing the data, analyzing it, and discussing it.

Corn cobs obtained from the Bojonegoro area were washed and dried in the sun until completely dry. The corncobs were chopped and baked in the oven at 60ºC for 12 hours. Dry corn cobs were mashed and sieved through a 100-mesh sieve.

Then the silica preparation method was adopted from the research of Roschat et al. 2016 with several modifications. The fine dry powder was weighed as much as 90 grams and divided into three equal parts in a 250 mL beaker. Each powder was washed with distilled water and filtered. The precipitate was dried in an oven for 24 hours and labeled A (corncob powder without acid treatment), B (corncob powder soaked in HCl at room temperature), and C (corncob powder soaked in HCl at 60ºC). STB and C were added with 3 M HCl, each 200 mL. B was stirred with a magnetic stirrer at room temperature, while C was stirred at 60˚C for 3 hours. The powder is washed with distilled water and filtered. The precipitate obtained was dried at a temperature of 110˚C. A, B, and C were calcined at 800˚C for 4 hours. Characterization using FTIR after data processing, analysis, and discussion.

RESULTS AND DISCUSSION

Thermal and non-thermal methods can remove compounds other than silica contained in corn cobs. The thermal method is carried out by burning at high temperatures on corncobs to obtain silica. Research by (Rafiee et al., 2012) regarding the optimization and synthesis of nano silica from rice husk, using a variation of combustion temperature of 500 ºC and 700 ºC, shows that at 700 ºC, it is whiter than at 500 ºC. In Gladys Ayu's research, 2017, corncob powder was thermally treated at a temperature of 800 ºC for 4 hours, and this research will use a temperature of 800 ºC and a holding time of 4 hours as well. This calcination process is necessary because calcination compounds other than silica can be decomposed and lost.

Soaking using HCL is a preparation process to produce silica in corn cob powder. HCL immersion is a non-thermal method because HCL can reduce organic compounds and other inorganic compounds besides silica, such as Mg, Na, K, and Ca (Umeda et al., 2009), found in corn cobs. Three powders are used: powder without HCL, HCl powder at room temperature, and HCL powder at 70 ºC. This was done to determine the effect of HCl and the optimum immersion temperature in obtaining silica from corn cobs.

Results

The following are the steps for reading the FTIR spectra:

First, determine the X-axis and Y-axis of the spectrum. The X-axis of the IR spectrum is labeled as “wavenumber,” and the numbers range from 400 on the far right and 4,000 on the far left. The X-axis provides absorption numbers. The Y axis is labeled as “percent transmittance,” and the numbers range from 0 at the bottom and 100 at the top.

Second, determine the characteristics of the peaks in the IR spectrum. All infrared spectra contain multiple peaks. Next, look at the available group area data needed to reach the spectrum.

Third, determine the spectrum region where the characteristic peak is present. The IR spectrum can be separated into four regions. The first region ranges from 4,000 to 2,500. The second area ranges from 2,500 to 2,000. The three regions range from 2,000 to 1,500. The fourth region ranges from 1,500 to 400.

Fourth, determine the functional group absorbed in the first region. If the spectrum has characteristic peaks in the range of 4,000 to 2,500, the peaks correspond to absorptions caused by NH, CH, and single OH bonds.

Fifth, determine the functional groups absorbed in the second area. If the spectrum has a characteristic peak in the range of 2500 to 2000, the peak corresponds to the absorption caused by the triple bond.

Sixth, determine the functional group absorbed in the third region. If the spectrum has characteristic peaks in the 2,000 to 1,500, the peaks correspond to absorptions caused by the C = O, C = N, and C = C double bonds.

Seventh, compare the peaks in the fourth region to those in the other four regions of the IR spectrum. The fourth is known as the IR spectrum's fingerprint region and contains many absorption peaks that account for a wide variety of single bonds. If all the peaks in the IR spectrum, including those in the fourth region, are identical to the other peaks in the spectrum, then the two compounds can be believed to be identical.

After synthesizing corncobs, corncob powder was obtained. This powder was then observed for its silica absorption band using FT-IR at wave numbers 4000 – 400 cm-1.

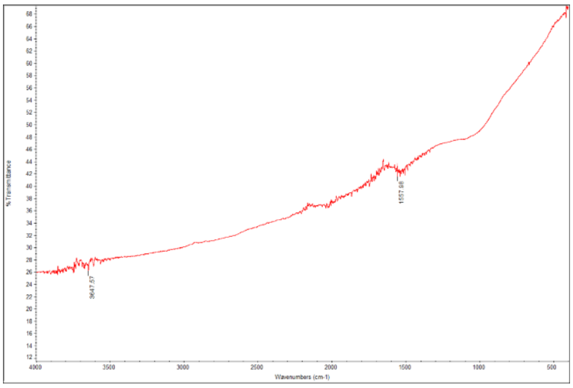

Figure 1. FTIR Results of Sample A

Figure 1. shows the FTIR results of sample A, namely the sample without HCL immersion treatment. The visible peak results are at a wavelength of 3647.57 and 1557.98 cm-1.

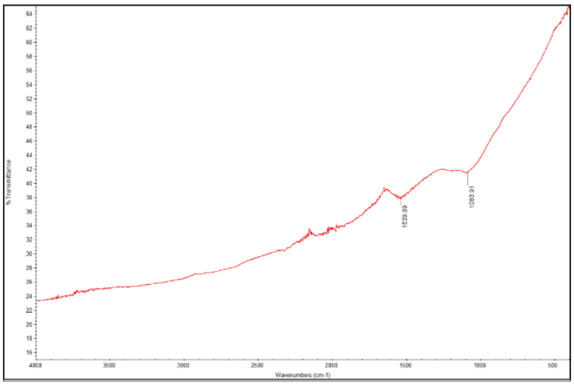

Figure 2. FTIR Results of Sample B

Figure 2 shows the FTIR results of sample B, namely samples treated with HCL immersion at room temperature. The visible peak results are at a wavelength of 1539.89 and 1083.91 cm-1.

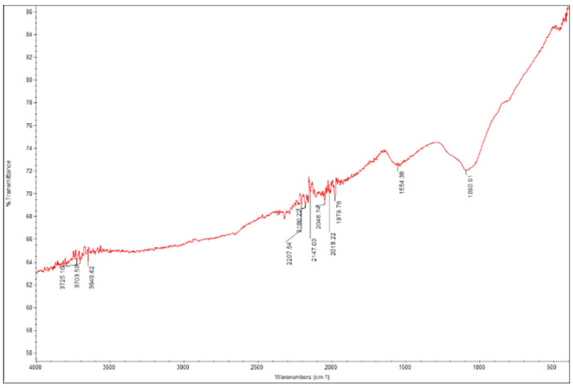

Figure 3. FTIR Results of Sample C

Figure 3 shows sample C's FTIR results, namely samples treated with HCL immersion at 70 ºC. The visible peak results are at a wavelength of 3725.16; 3703.5; 3649.62; 2207.54; 2147.03; 2018,22; 1554.38; and 1090.91 cm-1.

The following is a table for the transmittance peaks of the three samples and the classification of the areas that cause absorption.

Table 2. Peak Transmittance FTIR Result

Region	Sample A	Sample B	Sample C
			3725,16
1			3703,5
	3647,57		3649,62
			2207,54
2			2147,03
			2018,22
3	1557,98	1539,89	1554,38
4		1083,91	1090,91

From the table, it can be seen that sample A, namely the sample that was not immersed in HCL, belongs to regions 1 and 3. This means that sample A gets absorption caused by a single NH, CH, or OH in region one and has a double bond. Sample B or samples with immersion at room temperature belong to regions 3 and 4. Sample B has absorption caused by double bonds and has single bonds, namely two identical compounds. Whereas for sample C, the sample with HCL immersion at 70 ºC belongs to regions 1, 2, 3, and 4. Sample C has all the causes of absorption from absorption caused by NH, CH, and single OH bonds, then absorption of triple bonds, absorption of double bonds, and absorption of single bonds where the two compounds are identical. This means that it can be concluded that HCL and heating can change or even increase the cause of the absorption of corncob powder.

The three samples tested by FTIR are then combined using the origin application.

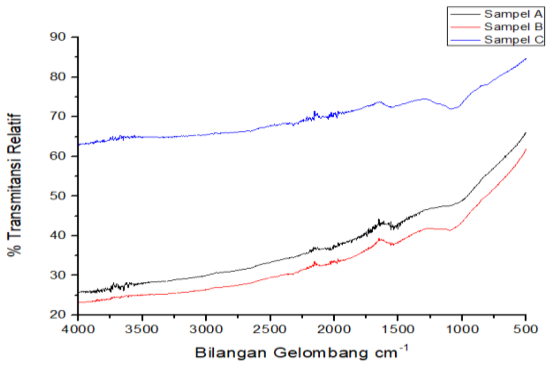

Figure 4. Margin FTIR Result with Origin

CONCLUSION

Corn cobs powder produces silica by isolating it using HCL at a temperature of 70 ºC for 3 hours and calcining at 800 ºC with a holding time of 4 hours. Based on its characterization using FTIR, samples soaked in HCL at 70 ºC resulted in absorption caused by several factors, including single NH, CH, OH, triple bonds, double bonds, and single bonds. This is reinforced by the FTIR results, where the peaks are in regions 1, 2, 3, and 4.

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