Synthesis and Characterization of Silica Gel from Corn Cob Skin and Cob Waste

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Abstract. The waste of husks and corn cobs that relatively abundant in Indonesia is mainly used as animal feed. Its utilization is still not maximized and pollutes the environment a lot. Corn husks and cobs contain silica, which can be used as a raw material in the manufacture of SiO2 silica gel. This study aims to obtain SiO2 from corn husk and cob ash using the sol- gel extraction method to determine the effect of ashing temperature and mass ratio of corn husks and cobs and the characteristics of silica gel. The method used in this study is the sol-gel method. The characterization of silica gel utilizes FTIR (Fourier Transform Infra-Red) and XRF (X-ray fluorescence) as a process. The result indicates that the FTIR shows an absorption pattern of silica with silanol (Si-OH) and siloxane (Si-O-Si) groups, with an amorphous silica phase at an ashing temperature of 750 °C and crystalline silica at an ashing temperature of 850 °C. XRF results determine that the yield of Si and SiO2 produced in corn husks tends to be higher than corn cobs and at an ashing temperature of 750 °C tends to be higher than 850 °C.

Keywords: Silica-gel · Corn-Husk · Corn-Cob · Ashing · Temperature · Mass-Ratio

1 Introduction

The main food commodity in Indonesia after rice is corn. Corn is a type of grain food crop (serilia) from the grass family (Arianingrum, R. 2014). Because it is a staple food substitute for rice, corn is included in food waste where this corn waste consists of corn husks and corn cobs.

Waste husks and corn cobs have been used as animal feed by the community, but their utilization has not been maximized. About 40% of corn plant waste is the cob (Susilowati, 2011) and about 20% of corn waste is the husk (Nurilah, 2017). In 2015, it is known that at least 7,844,974 tons of corn cobs and 3,922,487 tons of corn husks will become waste (BPS, 2015). Corn cobs have a chemical composition of 10.9% water, 36.48% cellulose, 28.86% hemicellulose, 3.16% lignin and 20.6% silica (Erviana et al,

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2013). While the chemical composition of corn husk includes 15% lignin, 5.09% ash, 4.57% alcohol-cyclohexane, and 44.08% cellulose (Fagbemigun et al, 2014). Corn cobs if ashed have a fairly large content of about 67.41% (Hidayat et al. 2015). Corn husk ash content has a SiO2 content of 47.40% (Wijiawati, 2016). The presence of silica content in corn cobs and corn husk waste can be utilized into various kinds, one of which is silica gel. Silica gel has many uses, including as an adsorbent or as a stationary phase in chromatography (Sulastri et al., 2010). The source of silica gel produced from coal ash and quartz sand is decreasing, because it is not renewable. Therefore, corn cob and husk waste can be used as an alternative source of basic materials in the manufacture of renewable and more economical silica gel (Okoronkwo et al, 2013).

In a previous study by Okoronkwo, silica gel on corn cobs using the sol gel (SiO2) method was 52.32% (Okoronkwo et al, 2013). Silica gel synthesis from corn cob ash percentage of silica content (SiO2) was 90.6% (Chanadee et al. 2016). Silica in corn husk ash was 47.40% and with the sol-gel method the silica content (SiO2) was 97.09% (Wijiawati, 2016). The use of an acidic solution with a low pH will produce a lot of silanol groups, the more silanol groups, the more the ability to bind water molecules through hydrogen bonds (Kristianingrum et al., 2011).

2 Research Methodology

2.1 Ingredient

The materials used in this study were corn cob and husk waste, NaOH, HCl.

2.2 Research Procedure

2.2.1 Pretreatment

The raw materials in the form of waste skin and corn cobs were given physical treatment including washing, drying under the sun, burning and ashing at temperatures of 750 °C and 850 °C.

2.2.2 Preparation of Silicate Solution (Na2SiO3)

The results of the initial treatment in the form of waste ash of corn husks and cobs as much as 10 g with variations of corn cobs and husks (1:0; 0:1; 1:1; 2:1; 1:2) were put into a 250 mL baker glass with the addition of 3M NaOH. as much as 60 ml. The solution was then stirred using a magnetic stirrer and heated at a temperature of 60 °C for 1 h.

2.2.3 Silica Gel Manufacturing

The extracted solution is (Na2SiO3) and the residue is then filtered with filter paper, then the Sodium Silicate (Na2SiO3) solution is taken. Na2SiO3 was stirred using a magnetic stirrer and added with 3M HCl solution little by little to form a gel.

2.3 Silica Gel Analysis

Parameters observed were the presence of silanol and siloxane groups in silica gel as an indication of the presence of silica in corn cobs and husks, as well as the number of elements and concentrations in silica gel on cobs and corn husks. Then, a characterization utilize the FTIR (Fourier Transform Infrared) & XRF (X-ray fluorescence) analysis. The reaction kinetics in the manufacture of silica gel:

$$SiO2(S) + residue(s) + 2\ NaOH(aq)\ Na2SiO3(aq) + H2O(l)$$
$$+ residue(s)\ Na2SiO3(aq) + H2O(l) + 2\ HCl(l)\ SiO2.nH2O$$
$$+ 2\ NaCl$$

The process to be carried out in this research are described in the following stages sequentially:

1) Preparation of Ash from corn cobs and husks

   – burned in the furnace at a temperature of 750 °C & 850 °C for 4 h
   – 80 mesh ash sieving

2) Preparation of Sodium Silicate Solution

   – stirring on a magnetic stirrer at a temperature of 60 °C for 1 h
   – filtering

3) Silica Gel Manufacturing

   – stirring and adding 3M HCL little by little until it reaches a pH of 7
   – drying silica gel in the oven at 120 °C for 2 h

3 Result and Discussion

3.1 FTIR (Fourier Transform Infrared) Analysis

The use of FTIR spectroscopy aims to determine the functional group of a compound and identify the compound and determine the molecular structure. The results of the isolation of silica gel from corncob waste at an ashing temperature of 750 °C contained several group vibrations as shown in Fig. 1.

The results of the isolation of silica gel from corncob waste at an ashing temperature of 850 °C there are several group vibrations as shown in Fig. 2.

The results of the interpretation of silica gel FTIR spectra at an ashing temperature of 750 °C can be seen in Table 1.

The results of the interpretation of the infrared spectra of silica gel at an ashing temperature of 850 °C can be seen in Table 2.

Characterization with FTIR was carried out in the wave number range of 400–4000 cm-1. Silica absorption patterns that appear generally are silanol (≡Si-OH) and

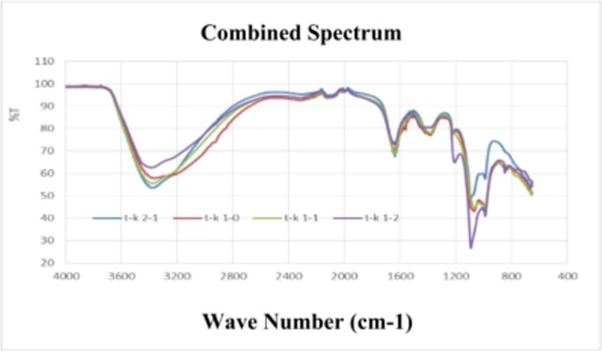

Fig. 1. FTIR of Silica synthesized from corn cobs 750 °C

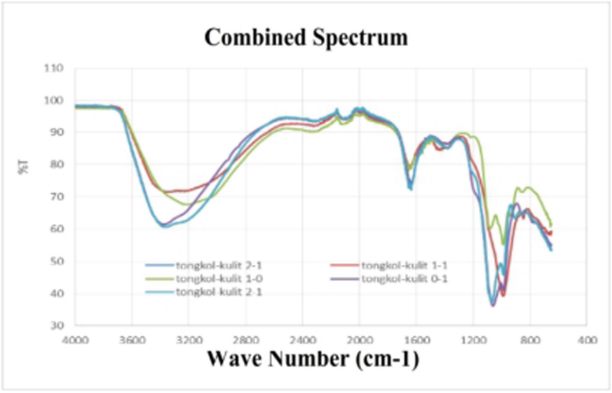

Fig. 2. FTIR Spectra of Silica synthesized from corn cobs 850 °C

siloxane (≡ Si-O-Si≡) groups. The absorption pattern of the synthesized silica in Figs. 4 and 5 shows that the absorption with an absorption band in the 620–900 cm-1 region indicates a range of OH groups in silanol (Si-OH) [30]. The absorption band in the area 1075–1095 cm-1 is the absorption band from the asymmetric stretching vibration of the Si-O group on the siloxane (Si-O-Si) group (Nadiyanto et al., 2019). The wide band in the wave number region of 3250–3650 cm-1 is the absorption band from the vibrations of the -OH hydroxy group on the silanol (Si-OH) group. The data shows that the absorption bands that appear in the FTIR spectra of the synthesized silica are silanol groups (Si-OH) and siloxane groups (Si-O-Si).

3.2 XRF (X-Ray Fluorescence) Analysis

Silica isolated was analyzed by XRF to determine and determine the percentage of purity of SiO2 and the elements and compounds contained in it as impurities. The results of the XRF analysis showed that several compounds were detected in the sample, which

Table 1. Spectrum of corn cob-husk (T:K) silica FTIR results 750 °C

No	Wave Number (cm-1)				
	T/K (0:1)	T/K (1:0)	T/K (1:1)	T/K (1:2)	T/K (2:1)
1	969.60	861.01 985.88	989.60	836.106 989.608	985.89
2	1086.5	1069.75	1064.1	1090.25 1207.66	1090.3
3	1388.4	1388.43	1379.1	1407.07	1226.3 1395.9
4	1653.0	1636.3	1636.3	1634.44	1640.03
5	2325.9	2081.7 2277.4	2098.5 2294.2	2087.31 2303.49	2083.4 2299.8
6	3399.3	3268.9	3347.2	3384.42	3350.9

Table 2. Spectrum of corn cob-husk (T:K) silica FTIR results 850 °C.

No	Wave Number (cm-1)				
	T/K (0:1)	T/K (1:0)	T/K (1:1)	T/K (1:2)	T/K (2:1)
1	964.02	991,47	993.34	964.02	898.29 987.75
2	1079.1	1084,7	-	1058.6	1079.1
3	1386.6	1388,4	1429.1	-	-
4	1653.1	1653,1	1669.9	1640.1	1636.1
5	2066.8 2266.7	2102,2 2335,2	2120.9 2316.5	2087.3 2325.9	2113.4 2290.4
6	3324.8	3149,6	3274.5	3354.6	3321.1

are presented in Table 3 and Table 4 with temperature variations of 750 °C and 850 °C, respectively.

The Si results in Tables 3 and 4 show the tendency of corn husks to produce a more dominant Si content than corn cobs. Corn husk ash has a higher percentage of silica (Si) than corn cob ash. Corn husk ash has 64.62% silica, while corn cob ash only has 52.32% silica (Rofiqoh, 2020). In the Table 3, the results of XRF characterization at an ashing temperature of 750 °C show that the yield of cobs at a T:K ratio of 1:0 is greater than that of corn husks at a T:K ratio of 0:1. This is because the 0:1 T:K ratio has a smaller pH or tends to be acidic which can be seen from the large Cl concentration in Table 3. At a low pH the resulting Si will decrease and will increase the K value. combined, even with high Cl levels, the resulting Si will be a lot.

Table 3. XRF characterization at ashing temperature of 750 °C

Element	Concentration (%)				
	T:K (1:0)	T:K (1:1)	T:K (0:1)	T:K (1:2)	T:K (2:1)
Si	2.87	2.43	1.22	3.71	2.14
P	0.85	2.13	0.39	0.45	2.36
S	1.74	1.57	0.47	0.81	1.13
Cl	62.28	53.87	68.31	70.47	60.84
K	31.91	39.61	29.07	24.23	33.12
Ag	0.35	0.39	0.54	0.33	0.41
Total	**100**	**100**	**100**	**100**	**100**

Table 4. XRF Characterization at 850 °C ashing temperature

Element	Concentration (%)				
	T:K (1:0)	T:K (1:1)	T:K (0:1)	T:K (1:2)	T:K (2:1)
Si	0,87	2.41	3.86	2.70	1.55
P	2,11	1.21	0.87	0.59	2.46
S	0,23	1.09	2.48	1.12	1.24
Cl	72,08	66.30	58.32	68.18	65.38
K	24,18	28.55	34.09	27.08	28.95
Ag	0,44	0.43	0.38	0.33	0.42
Total	**100**	**100**	**100**	**100**	**100**

Table 5. Characterization Results of XRF Compounds at ashing temperatures of 750 °C and 850 °C

Element	Concentration (%)				
	T:K (1:0)	T:K (1:1)	T:K (0:1)	T:K (1:2)	T:K (2:1)
SiO2 (750 °C)	5.84	4.87	2.55	7.641	4.35
SiO2 (850 °C)	1.80	4.94	7.74	5.56	3.17

SiO2 compounds at different ashing temperatures are shown in Table 5. Table 5 shows the ashing temperature of 750 °C showing the highest SiO2 results obtained at a ratio of 1:2 corn cobs and husks, which is 7.641%. In contrast to the results of the ashing

temperature of 850 °C which showed the highest SiO2 results at a 0:1 ratio of cobs and corn, 7.74%.

SiO2 at a temperature of 850 °C has been transformed from amorphous to crystalline silica. This transformation occurs due to combustion carried out above 800 °C. The amorphous and crystalline phases contained in this silica ash can affect the solubility of silica which can increase and decrease the yield of xerogel silica and in the amorphous phase the silica yield is large (Chanadee, et al. 2016).

In Table 5, each of these SiO2 ratios is different, causing the obtained SiO2 to have different values. Table 4.5 at a temperature of 850 °C with a ratio of corn cobs and husks 1:0 has a small value of 1.80% this is due to the large concentration of Cl.

4 Conclusion

Silica gel from husk and corn cobs waste ash showed the presence of silanol groups (Si-OH) with an absorption band of 620–900 cm-1 and siloxane groups (Si-O-Si) with an absorption band of 1075–1095 cm-1. The ashing temperature affects the phase of silica gel, namely when the ashing temperature is 750 °C the amorphous phase is in the amorphous phase, while at 850 °C the crystalline phase is at a temperature. The ratio mass ratio affects the amount of SiO2 obtained, the highest results are variations in the ash of cob waste and corn husks 1:2 with a SiO2 yield of 7.641%.

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