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Effect of Calcination Temperature on the Synthesis of Silica from Bagasse

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ABSTRACT

Bagasse is the solid waste derived from the sugar-making prosess which is a potential source of silica. In this study, extraction of silica from bagasse was carried out in the following steps: pretreatment of bagasse using HCl solution, followed by calcination at varying temperatures (700ºC, 800ºC, and 900°C). Furthermore, extraction using NaOH solution and precipitation using HCl. Silica characteristics were obtained using X-Ray Diffraction (XRD), Scanning Electron Microscopy-Electron Dispersive Spectrometry (SEM-EDS), and Brunauer, Emmett and Teller (BET) surface area. The results showed that calcination temperature affected the characteristics of the silica produced. The silica extracted at 700°C produced an amorphous phase with a broad peak at an angle of 2θ = 20-24°. It contained the most considerable silica content and surface area, 42.46% and 796.89 cm^2/g, respectively. The extracted silica had an average diameter of 5.67 mm and a pore volume of 1.184 cm^3/g.

Keywords: silica, bagasse, calcination temperature

INTRODUCTION

Bagasse is one of the wastes generated from the production of sugar. It is estimated that as much as 0,4 million tons/year of bagasse is produced in Indonesia[1]. It takes a large space to dispose of solid waste, and waste disposal harms the environment, especially soil and water [2]. The utilization of this waste is still limited. Some of the bagasse is used as fuel for heating the boiler. In this process, 8-10% of solid waste is produced in the form of ash [3]. Bagasse ash cannot be used as fertilizer because it is low in nutrients and even increases heavy metals concentration in the soil, for example Al_2O_3, Fe_2O_3, MnO, Cr_2O_3, NiO [4,5]. The research by Norsuraya [6] showed that bagasse ash contained 53.10% silica. Other studies have found that silica content in bagasse ash around 50-78% [7,8,9]. Thus, bagasse has the potential as a silica source to increase its economic value by replacing commercial silica, which is quite expensive.

Silica has several properties, such as high thermal stability, low bulk density, and low thermal conductivity [10,11]. Several silica applications include catalysts, cosmetics, ceramics, cement, pharmaceuticals, and detergents [5,12]. Silica is also commonly applied as a precursor for the synthesis of inorganic and organometal substances, as a coating for electronic and optical materials, as a filler for composites, and as a filler in photochromic pigment [13,14]. Silica has a large pore and surface area that allowed it to be utilized as an adsorbent, such as

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removing methylene blue dye, Janus Green B, Reactive Black 5, and dimethyl phthalate [15]. The silica surface can also be modified to produce specific properties, such as modifying the silica with amino to act as a congo red dye adsorbent [16]. Other studies have shown that quaternary ammonium salts modified silica has antibacterial properties in the biomedical field [17].

Silica can be extracted from biomaterials using several methods, including precipitation using acid [18], ion exchange resin [19], ablation and calcination then followed by treatment using alkaline solutions [20], sol-gel followed by adsorption using activated carbon [5]. Researchers generally extract silica out of bagasse ash from sugar factory waste. In this research, silica extraction was carried out from bagasse. The extraction stage includes acid treatment of bagasse followed by the calcination process. The next process is treatment with NaOH solution, then continue with precipitation using HCl solution. This method is relatively straightforward and likely successful. One of the factors that are affecting extraction success is calcination temperature. Research on silica extraction from rice husks with calcination temperature variations of 400-900°C shows that the optimum temperature is at 500°C, where the soluble fraction of silica is most significant [21]. Calcination temperature affects the type of phase, the percentage of silica produced, and the surface area [5,22]. SiO_2 from bagasse ash with acid treatment and calcined at a temperature of 500ºC produced 63.122% [23], at a calcination temperature of 600ºC and 1000ºC obtained 70.97% [23] and 87.63% [24], respectively. The optimum calcination temperature depends on the material used. The calcination temperatures used in this study were 700ºC, 800ºC, and 900°C. The resulting silica was characterized using XRD, SEM-EDS, and BET surface area.

EXPERIMENT

Chemicals and instrumentation

The chemicals used include HCl, NaOH, silica products from Merck (Germany), bagasse obtained from sugar factory waste in the province of South Sumatra, Indonesia, deionized water, and distilled water.

Silica from bagasse produced was characterized using X-Ray Diffraction (XRD) Rigaku Miniflex 600 (λ=1.5406 Å, 40 Kv, and 40 mA) to determine the crystalline structure. Analysis of morphology and element composition was carried out utilizing Scanning Electron Microscopy-Energy Dispersive X-Ray Spectrometry (SEM-EDS Tescan Vega 3). Meanwhile, the surface area was analyzed using the surface analyzer Quantachrome Nova 4200 series instrument based on N_2 adsorption at 77K calculated using the BET method (Brunauer-Emmet -Teller).

Procedure

Silica Extraction from Sugarcane Bagasse

The bagasse was cut into pieces about one centimeter in size, washed with distilled water, and oven-dried at 110°C overnight. Afterwards, as much as 250 g of bagasse is added 0.1 M HCl as much as 500 mL, stirred using a shaker for 3 hours. The mixture was filtered, the bagasse was washed with deionized water until neutral, then dried using an oven at 110°C overnight [25]. Bagasse was calcined using a furnace with a heating speed of 15°C/minute for 2 hours with variations in calcination temperature, 700ºC, 800ºC, and 900ºC. The calcined bagasse ash was milled and sieved with a 200 mesh sieve.

A total of 6 g of bagasse ash was added into 100 mL of 2.0 M NaOH, stirred for 15 minutes, and filtered using filter paper. The filtrate was titrated using 2.0 M HCl to form a gel.

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Titration is carried out until the pH is 7. The resulting gel was left to stand for 5 hours, then washed with distilled water. After that, the gel was dried in an oven at 110°C for 5 hours. Silica powder was obtained after drying.

RESULT AND DISCUSSION

All silica extraction from bagasse is carried out in several stages. The initial stage is washing the bagasse with HCl solution, which aims to remove several minerals. Leaching using HCl is effective in reducing metallic impurities in the material [26]. Another study showed that silica extraction from bagasse ash with pretreatment using HCl obtained more silica content when compared without treatment [22]. The reactions that occur include the following [14].

$$Al_2O_3(s) + 6HCl(aq) \rightarrow 2AlCl_3(aq) + 3H_2O(l) \quad (1)$$
$$CaO)s) + 2HCl(aq) \rightarrow CaCl_2(aq) + H_2O(l) \quad (2)$$
$$MgO(s) + 2HCl(aq) \rightarrow MgCl_2(aq) + H_2O(l) \quad (3)$$
$$K_2O(s) + 2HCl)aq) \rightarrow 2KCl(aq) + 3H_2O(l) \quad (4)$$

The calcination process is carried out at temperatures of 700°C, 800°C, and 900°C. This process converts the bagasse into bagasse ash. The ashing process affects the amount of silica that can be extracted and the phase formed. Next, the bagasse ash was reacted with NaOH. The reaction is as follows [21].

$$SiO_2 + NaOH \rightarrow Na_2SiO_3 + H_2O \quad (5)$$

The greater the NaOH concentration used, the more silica that can be extracted. The research conducted by Megawati [3] showed that 2.0 M NaOH was the optimum concentration for silica extraction from bagasse ash. Likewise, other studies' show that 1.0 M NaOH concentration could extract 91% silica while the NaOH concentration less than 0.1 M was unable to extract silica [11]. The Na_2SiO_3 solution was titrated using HCl to form a silica solid. The reaction that occurs is [5]:

$$Na_2SiO_3 + HCl \rightarrow SiO_2 + NaCl + H_2O \quad (6)$$

Figure 1 shows the silica extracted at various calcination temperatures. The silica extracted at 700°C shows a white color, while the higher calcination temperature (900°C), the color tends to be darker. The higher of temperature, the phase change from amorphous to crystalline.

Figure 1. The silica extracted at a calcination temperature of (a)700°C, (b)800°C and (c)900°C

The XRD spectra of the extracted silica are shown in Figure 2. The silica with calcination temperatures of 700ºC, 800ºC, and 900°C showed an amorphous phase, with an angle of 2θ of 20-24°. The extracted silica has similarities to commercial silica. A study of Komangklang [25] showed that silica extracted from bagasse by two methods, calcination and without calcination, both produced silica with an amorphous phase. However, the peak intensity of silica produced by calcination is higher than without calcination. The diffraction angle widening indicates an amorphous phase that shows a porous material [27]. In the amorphous phase, the arrangement of atoms in the molecule is in a random state and an irregular pattern [28].

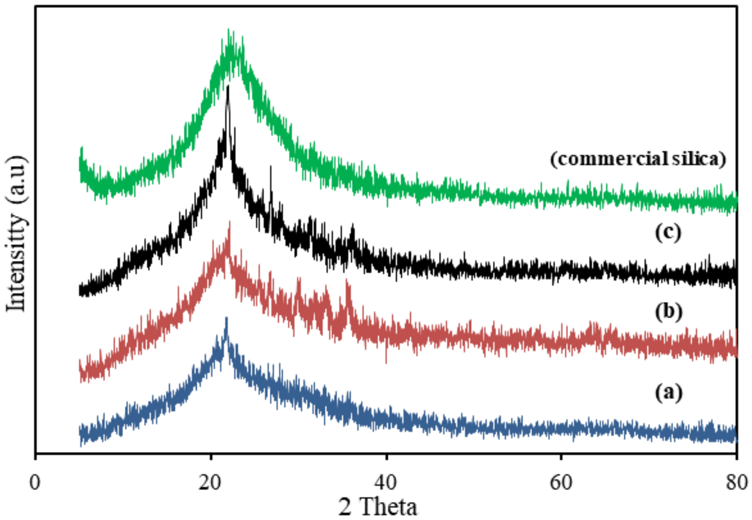

Figure 2. X-ray diffraction of silica at a calcination temperature of (a)700ºC, (b)800ºC, (c)900ºC and commercial silica

The increase in calcination temperature produces higher peak intensity. The observation indicates more phase change from amorphous to crystalline at higher temperatures. The silica extraction from rice husks showed that at a calcination temperature of 1000°C, a crystalline phase was formed, which was identified as cristobalite and tridymite [21]. This study indicates that the silica phase conforms to the ICDD 00-001-0424 standard card, namely the silica-cristoballite phase. Table 1 shows the silica crystal sizes calculated using the Scherrer equation. The extracted silica has a crystal size in the nanometer scale (<100 nm). The smallest crystal size was produced at 700°C. The increase in temperature is also increasing the crystal size. Another study showed similar results, namely silica from rice husks, the crystal size increased at a calcination temperature of 500-900°C [26].

Table 1. Crystallite size of silica

Calcination temperature (ºC)	Peak position (2θ)	Crystallite size (nm)
700	21.62	2.36
800	22.09	2.42
900	21.94	7.55
Commercial silica	21.26	3.46

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The morphology of the extracted silica is shown with a magnification of 10,000x in Figure 3. The three extracted silicas appear to be fibrous macro structures with different pore sizes. The silica extracted at a calcination temperature of 700ºC has a denser surface than 800ºC and 900ºC. The determination of the silica composition using EDS is shown in Table 2. The silica produced at calcination temperatures of 700°C and 800°C has a not much different composition, namely 42.46% and 42.04%. Meanwhile, silica produced with calcination at 900°C showed less content, 31.35%. The calcination process causes the decomposition of hydrocarbons and some phosphorus, potassium, and aluminum compounds, which melt at a temperature of 240-490°C [29].

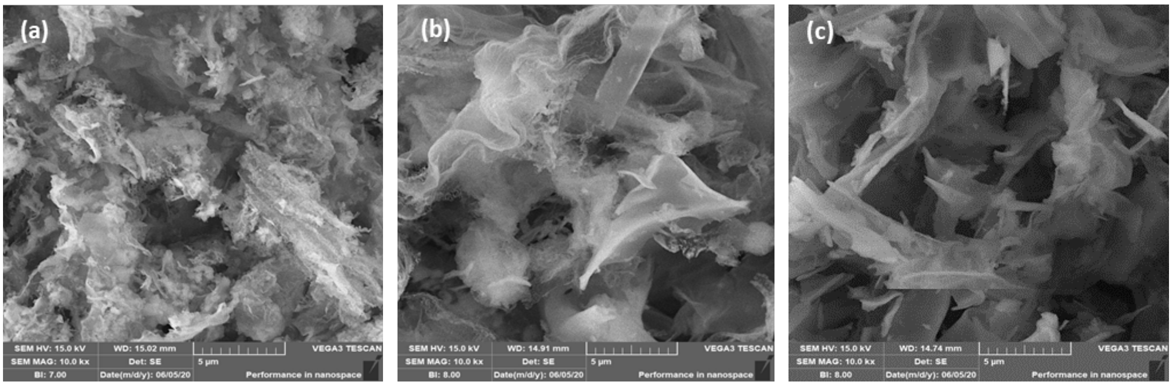

Figure 3. SEM image silica extracted at a calcination temperature of (a)700ºC, (b)800ºC, (c)900ºC

The percentage of silica extracted from rice husks with a calcination temperature of 500°C is more significant than 400°C, 450°C, 500°C, and 900°C. Silica with a calcination temperature of 500ºC has the most soluble fraction and an amorphous phase. Silica with the amorphous phase is more reactive than the crystalline phase so that it forms more Na_2SiO_3 when reacted with NaOH. At a high temperature, the silica crystalline phase is dominated so that it does not react with NaOH to form Na_2SiO_3 [21]. Other research showed that Baggase ash burning out at a temperature of 500ºC for 3 hours obtained SiO_2 of 63.122% [10]. Other researchers found that the percentage of SiO_2 was 70.97% at a calcination temperature of 600ºC [23] and a calcination temperature of 1000ºC obtained 87.63% [24]. In this study (Table 2), the highest Si amount was produced from the extraction of calcined bagasse at a temperature of 700ºC was 42.46% or SiO_2 of 90.98%. SiO_2 content in this study was more significant than obtained by other researchers. The best calcination temperature depends on the type of biomaterial [10]. Other elements such as Al, Ca, Mg, and others are still present in small amounts. In general, the extracted silica from plant material still contains small amounts of these elements in the form of oxides [10,26,30].

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Table 2. Chemical composition of silica extracted from bagasse

Element	Mass (%)		
	700°C	800°C	900°C
O	48.80	50.72	53.99
Si	42.46	42.04	31.35
Al	1.70	2.32	3.12
Ca	1.97	1.72	2.29
Mg	1.09	0.85	1.87
K	0.34	0.52	1.47
Na	0.24	0.50	1.45
Fe	1.38	0.61	2.36
Cr	0.32	-	0.38
P	0.81	0.48	0.65
S	0.33	0.04	0.16
Other	0.56	0.20	0.91

BET analysis utilizing nitrogen adsorbate was carried out to obtain data on the surface area, pore diameter, and pore volume. The analysis results are presented in Table 3. Silica extracted at calcination temperature 700°C has the largest surface area than 800°C and 900°C. An increase in temperature indicates a phase change from amorphous to crystalline and an increase in crystal size. The crystal size will further decrease the surface area due to the nucleation and growth of the crystal. When the crystal particles grow, they fill in the empty pores, which reduces the surface area. Similar results were obtained in the silica obtained from rice husk ash, where the surface area at a calcination temperature of 700°C was more significant than 800°C [31]. Synthesis of mesoporous silica from rice husk with a calcination temperature of 600°C using a 1.25% Cetyl trimethyl ammonium bromide template produced an amorphous silica phase and a surface area of 768.947 m^2/g [32]. The surface area is smaller than the results of this study, namely 796.89 cm^2/g.

Table 3. The surface area, pore diameter, and pore volume of silica

Calcinanation temperature	Surface area (m^2/g)	Average pore diameter (nm)	Pore volume (cm^3/g)
700°C	796.89	5.67	1.184
800°C	758.43	6.12	1.126
900°C	546.91	6.89	1.121

CONCLUSION

This research has been successfully extracting silica powder from bagasse. The silica extraction stages include pretreatment of bagasse using HCl, followed by calcination of bagasse with temperature calcination of 700°C, 800°C, and 900°C, continued by extraction using NaOH and precipitation using HCl solution. The calcination temperature has been shown to influence the characteristics of the silica produced. The silica extracted with calcination temperature of 700°C had an amorphous phase with the highest content and surface area, respectively 42.46% and 796.89 m^2/g.

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