

# Characterization for Post-treatment Effect of Bagasse Ash for Silica Extraction

Patcharin Worathanakul, Wisaroot Payubnop, and Akhapon Muangpet

**Abstract**—Utilization of bagasse ash for silica sources is one of the most common application for agricultural wastes and valuable biomass byproducts in sugar milling. The high percentage silica content from bagasse ash was used as silica source for sodium silicate solution. Different heating temperature, time and acid treatment were studied for silica extraction. The silica was characterized using various techniques including X-ray fluorescence, X-ray diffraction, Scanning electron microscopy, and Fourier Transform Infrared Spectroscopy method. The synthesis conditions were optimized to obtain the bagasse ash with the maximum silica content. The silica content of 91.57 percent was achieved from heating of bagasse ash at 600°C for 3 hours under oxygen feeding and HCl treatment. The result can be used as value added for bagasse ash utilization and minimize the environmental impact of disposal problems.

**Keywords**—Bagasse ash, synthesis, silica, extraction, post-treatment.

## I. INTRODUCTION

**B**AGASSE is a cellulose fiber remaining after the extraction of the sugar-bearing juice from sugarcane. Biomass is an important source of energy in tropical countries like Thailand [1, 4]. Bagasse ash is one of the biomass sources and valuable byproducts in sugar milling that often uses bagasse as a primary fuel source to supply all the needs of energy to move the plants. Burning bagasse as an energy source yields its ash, considered as a waste causing disposal problems [5]. It is well-known that bagasse ash is an alternative source of energy with high silica content [3, 5]. Several studies have investigated bagasse ash potential applications such as producing silica gel as adsorbent, raw material for ceramic, cements and concrete additives, catalyst, cosmetics, paint and coating, etc [2, 3, and 5]. based on its characteristic. The silica contents of bagasse and its ash are varied depending on the type of soil and harvesting [2]. The objectives of this work were to synthesize and characterize the bagasse ash obtained under factory processing conditions. The effects of temperature and time of heating as well as the acid treatments of sugar cane bagasse were determined.

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Synthesized silica with different conditions including chemical and physical analysis was characterized. The extracted silica from this study can be added value of silica source for many applications of bagasse ash.

## II. EXPERIMENTAL

### A. Raw Material Preparation

Sugarcane bagasse, from local market and bagasse ash from sugarcane bagasse burnt in factory boilers, main raw materials, were used in this study. Before any treatment, the sugarcane bagasse was dried, resized with refiner 2850 rpm (Condux type LHM 20/16, Condux-Werk, Wolfgang bei Hanau, Germany), and screened through a sieve of 20-mesh size for 2 hours to eliminate the larger bagasse size. Then the pretreatment step with 1 M and 3 M hydrochloric acid of those samples at 100 °C for 2 hours was carried out. The samples were washed with distilled water until neutral condition (pH meter, METTLER TOLEDO) was achieved. The samples were then dried in the oven (Mettmert, UL 40, Germany). The acid treatment helped to dealuminate the bagasse ash and to remove iron to the certain extent for adjusting raw material quality. After acid treatments, sugarcane bagasse and bagasse ash were treated in the furnace (NABER 2804 Lilienthal Bremen Program Controller C8) under different temperatures at 600°C, 700 °C and 800°C and different heating time of 1 hour, 2 hours and 3 hours without and with oxygen constantly flow rate feed to the furnace. The highly silica content from processing conditions was analyzed and reported using the characterization techniques to use as silica sources for silica compound synthesis.

### B. Silica Characterization

**X-ray Fluorescence Spectroscopy (XRF)** Chemical analysis was determined by using X-ray Fluorescence (XRF HORIBA, MESA 500-WX-ray fluorescence spectrometry with 15/50 keV X-ray tube. Quantitative analysis of silica content with no preparatory work such as calibration using standard samples and the pre-registration of a standard spectrum was performed.

**X-ray Powder Diffraction (XRD)** Phase identification of silica extracted were determined by X-ray powder diffraction Phillips PW 1830/40 was used to determine the samples, using Cu-K $\alpha$ 1 radiation with  $\lambda = 1.5406 \text{ \AA}$ , generator tension 40 kV, generator current 30 mA in the range 5-70° 2 $\theta$  and at a rate of 0.01°/min.

**Scanning Electron Microscopy (SEM)** Surface structure of the sample, shape and particle size were taken on scanning electron microscopy (SEM) for three-dimensional appearance. The powder sample was sprinkled on a carbon sticky tab of aluminum specimen mount and coated with a nanometer-thick layer of gold (Au) using a sputter coater before being examined and photographs SEMs. Phillips XL30 SEM with 15-20 kV energy) and the Edward Scancoat six sputter coater (Edwards Laboratories, Milpitas, CA) for sputtering Au were used to determine synthesized silica.

**Fourier Transform Infrared Spectroscopy (FTIR)** The surface chemical bonding was determined by Fourier transform infrared spectroscopy (FTIR, Perkin Elmer, Spectrum One, USA). Diffuse reflectance infrared fourier transform (DRIFT) technique was used for all samples to identify types of chemical bonds (functional groups). The spectral resolution was  $4\text{ cm}^{-1}$  and 256 scans were coded into the mid infrared region ( $4000\text{-}400\text{ cm}^{-1}$ ) [6].

### III. RESULTS AND DISCUSSION

#### A. Effect of Temperature and Time

The silica content increased with increasing temperature was shown in XRF result (Table I). The silica contents were 19.42%, 21.05% and 27.98% when the temperatures were  $600^\circ\text{C}$ ,  $700^\circ\text{C}$ , and  $800^\circ\text{C}$ , respectively. The high temperature helped eliminate impurities in bagasse ash as well. The result from the heat treatment without oxygen feed at  $800^\circ\text{C}$  was used to study further into the effect of heating time. For 3 hours holding time in furnace, silica content was slightly increased to 29.85% as shown in Table I.

TABLE I  
COMPOSITION OF BAGASSE ASH BEFORE ACID TREATMENT WITHOUT OXYGEN FEEDING IN THE FURNACE WITH DIFFERENT TEMPERATURES AND TIME

Component	Mass% for 3 hours heating time			
	Mass% for 2 hours heating time	600 °C	700 °C	800 °C
SiO <sub>2</sub>	19.417	21.053	27.979	29.849
K <sub>2</sub> O	35.036	30.499	24.301	23.990
P <sub>2</sub> O <sub>5</sub>	12.428	12.722	12.241	12.043
SO <sub>3</sub>	10.969	13.141	14.734	13.242
CaO	14.482	15.129	13.721	13.307
MgO	2.766	3.24	2.598	2.598
Mn <sub>2</sub> O <sub>3</sub>	1.236	1.338	1.332	1.303
Fe <sub>2</sub> O <sub>3</sub>	1.884	1.287	1.768	1.812
Al <sub>2</sub> O <sub>3</sub>	0.973	0.956	0.661	1.262
Other	0.809	0.635	0.665	0.594

#### B. Effect of Acid Treatment and Oxygen Feeding

However, in the presence of oxygen (95%) feed constant flow rate and after acid treatment, the result shows the high percentage of silica contents changed to 89.04% at  $800^\circ\text{C}$  for 3 hours of bagasse ash (Table II).

TABLE II  
SILICA CONTENT WITH ACID TREATING AND OXYGEN FEEDING IN THE FURNACE FOR 3 HOURS HEATING TIME

Component	Mass %		
	before acid treatment		after acid treatment
	without O <sub>2</sub>	with O <sub>2</sub>	with oxygen
SiO <sub>2</sub>	29.849	75.142	89.037
K <sub>2</sub> O	23.99	0.902	2.134
P <sub>2</sub> O <sub>5</sub>	12.043	1.200	1.687
SO <sub>3</sub>	13.242	3.740	0.330
CaO	13.307	16.141	2.549
Mn <sub>2</sub> O <sub>3</sub>	1.303	0.047	0.153
Fe <sub>2</sub> O <sub>3</sub>	1.812	1.413	1.969
Al <sub>2</sub> O <sub>3</sub>	1.262	0.613	0.791
Other	3.192	0.802	0.791

#### C. Phase and Morphology Analysis

The result from 1 M and 3 M hydrochloric acid treatment experiment is presented in Fig. 1. From Fig. 1, it shows that XRD peaks at  $2\theta=20$ , and is obvious for 1 M acid treatment of bagasse ash.

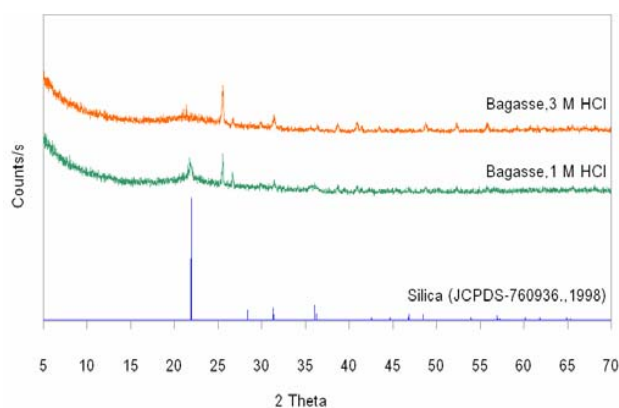


Fig. 1 Effect of acid concentration treatment on phase analysis

Moreover, the phase analysis was analyzed for the effect of oxygen feeding and the result shows that the highly crystal phase of samples occurred in the presence of oxygen (Fig. 2).

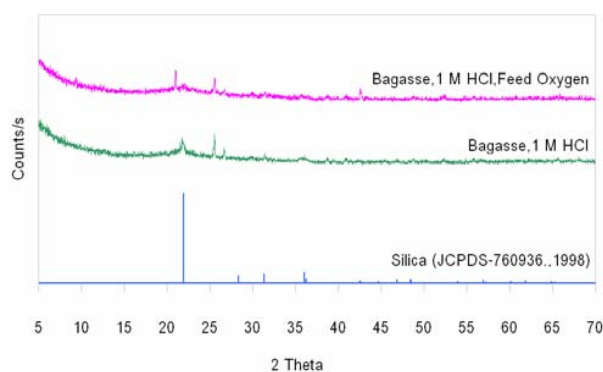


Fig. 2 Comparison between bagasse ash with and without oxygen feeding on phase analysis

Upon heating at  $800^\circ\text{C}$  for 3 hours, the morphology of processed bagasse ash was observed as the spherical shape of

silica particles and agglomerated as seen from SEM photograph in Fig. 3.

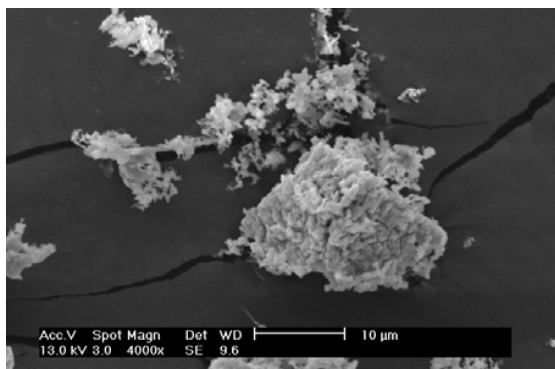


Fig. 3 SEM of Bagasse ash under 800°C for 3 hours

#### D. Chemical Bonding from FTIR

Fig. 4 shows FTIR spectrum in the region of 4000-400  $\text{cm}^{-1}$  for the sample of bagasse ash and sodium silicate prepared from silica source extracted at 800°C for 3 hours. The peaks at 466 are assigned to Si-O bending, 791  $\text{cm}^{-1}$  and 1090  $\text{cm}^{-1}$  from vibration in symmetric and asymmetric of  $\leftarrow\text{O-T}\rightarrow\leftarrow\text{O}$  stretching (T=Si, Al) functional group in tetrahedral arrangement, respectively [7]. Absorbed water bonding and Si-OH are shown at 3000  $\text{cm}^{-1}$  [7].

Moreover, sodium silicate solution is indicated in the wave number of 980  $\text{cm}^{-1}$ , 1432  $\text{cm}^{-1}$ , 1655  $\text{cm}^{-1}$  and 3305  $\text{cm}^{-1}$ , peak at 980  $\text{cm}^{-1}$  is indicated to the vibration in asymmetric of  $\text{SiO}_4$  stretching functional group, 1432  $\text{cm}^{-1}$  is carbonyl group [8], 1655  $\text{cm}^{-1}$  is absorption O-H stretching [7] and at 3305  $\text{cm}^{-1}$  for Si-OH and adsorbed water functional group [9].

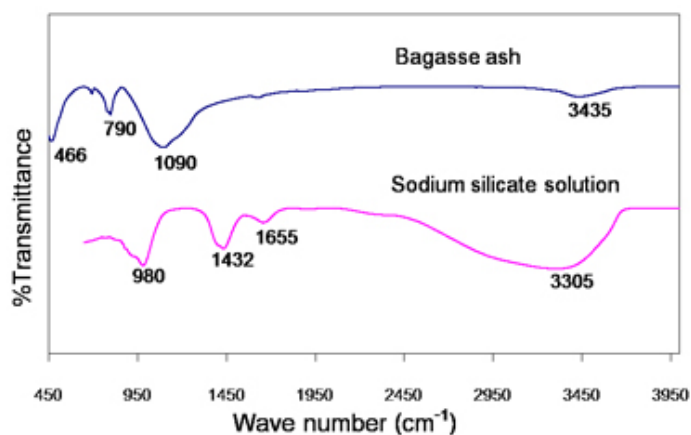


Fig. 4 FTIR spectrum of bagasse ash under 800°C for 3 hours and sodium silicate solution prepared

However, characterization of silica extracted was shown. The condition for acid treatment is the main importance for silica extraction; therefore the temperature of heating of 600 °C for 3 hours was successfully used instead of 800 °C for 3 hours for energy consumption. The silica content of 91.57

percent was achieved from heating of bagasse ash at 600°C for 3 hours under oxygen feeding and HCl treatment.

#### IV. CONCLUSION

Silica has been successfully extracted from bagasse ash. Mass compositions from XRF, XRD spectrum peak, morphology and chemical bonding have been reported. The highly silica content can be used for silica compound preparation and can be minimize the environmental impact of problems for bagasse ash disposal.

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