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Zeolite Synthesis Based Silica from *Saccharum officinarum L.* with Black Stem Using the Hydrothermal Method

Muhamad Redi, Intan Rizki Widya Astuti, Restu Agung Ramadhani, Baiq Nova Sopiana, Farhana Nida'ul Adhya', Hidayatun Nikmah, Sudirman*, Baiq Nila Sari Ningsih

Chemistry Study Program, University of Mataram, Mataram, Indonesia
*E-mail: sudirman28@unram.ac.id

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Abstract: *Saccharum officinarum L.* has a high amount of silica content, which can be used as the main component of zeolite synthesis. Silica was isolated from *Saccharum officinarum L.* bagasse with NaOH and HCl. This research aims to synthesize zeolite using silica from *Saccharum officinarum L.* with black stem and alumina from aluminium foil. The *Saccharum officinarum L.* used in this research was sourced from Lombok Island, West Nusa Tenggara Province, Indonesia. Silica was sourced from *Saccharum officinarum L.* using the sol-gel method. The silica from the isolation of dregs ash obtained 20 g of silica with a high percentage (yield) of 50%. This study found that 5.29 g of aluminium foil produced 14.29 g of alumina with the addition of 22.50 g of Na_2CO_3. The zeolite obtained was 3.88 g with a SiO_2:Al_2O_3 ratio of 5:3. From this study, the percent (%) of obtained zeolite was 48.5% from the weight of the silica and alumina produced. The zeolite results synthesized from *Saccharum officinarum L.* ash were characterized using FTIR. Based on the FTIR results, absorption was found at wave numbers (cm^{-1}) 960, 729, and 668. The zeolite obtained was pure white and had a FTIR spectrum similar to that of Zeolite X from previous research. It can be predicted that the zeolite obtained from this research has been formed. This research is expected to be useful in increasing the effectiveness of the silica extraction process from *Saccharum officinarum L.* with black stem. Further analysis using XRD and SEM is needed to determine the characteristics of the zeolite produced.

Keywords: Alumina; *Saccharum officinarum L.* with Black Stem; FTIR; Silica; Zeolite.

Introduction

Sugarcane (*Saccharum officinarum L.*) is a plant often used as a globally significant producer of sugar and bioenergy [1]. Saccharum plays a substantial role in the world economy as a producer of international sugar commodities. Bagasse is often used as fertilizer or even thrown into landfills, causing increasing environmental problems [2]. Waste is physically disturbing, and causes smell and air pollution around industrial plants. It is necessary to handle Saccharum waste so that it can be put to good use to produce products that are not only economical but also environmentally friendly.

Saccharum bagasse is an agricultural waste that has the potential to be used as a source of natural silica [3]. Saccharum bagasse contains significant amounts of carbon and silica. A simple NaOH fusion method and water dilution can recover the silica content to produce a sodium silicate solution. Then, the solution obtained can be used for various material preparations, such as silica materials and zeolite synthesis [4]. The silica content in Saccharum bagasse based on mass percentage was found to be 76.168%, 76.292%, and 77.286% [5]. This high silica content can be used as the main component in the synthesis of zeolite with the addition of alumina. Alumina can be produced from aluminium materials such as aluminium foil waste (Aluminum Foil Waste/AFW). The method used is precipitation with HCl 5M. Aluminium shows rich variations in coordination depending on temperature, moisture content, and zeolite [6]. Factors that can affect the extraction of alumina are the process of mixing the solution between NaOH and $Ca(OH)_2$ [7]. Alumina functions as an abrasive and absorbent [8].

Zeolite is a mineral material with good hydrothermal stability and is widely used in various industrial fields [9]. Zeolite can be formed into natural and synthetic zeolite based on its formation. Natural zeolites have been widely used for air purification, catalysts, or adsorbents, and they are activated first to decrease their availability in nature. Zeolite can be activated with mineral acids [10]. Three types of natural zeolite function as absorbents for safranin dye pollutants from air heulandite zeolite, clinoptilolite zeolite, and phillipsite zeolite [11].

Meanwhile, synthetic zeolite is a zeolite developed in a laboratory to meet needs in various fields. One of the synthetic zeolites that has been widely developed is zeolite X. Zeolite X is a type of zeolite with a low SiO_2:Al_2O_3 molar ratio. Zeolite X is commonly used in the waste processing industry. This is because zeolite X has good stability, many pores, and a large surface area [12]. Zeolite Zeolite X has a ratio value of 2:3 (SiO_2:Al_2O_3). Zeolite with primary minerals containing alumina in the residue can reduce the alumina extraction ratio [13].

NaY zeolite was successfully developed from bagasse at an optimal crystallization temperature of 298.15 K. The SEM test results showed the process of forming spherical NaY zeolite crystals. The size of NaY zeolite crystals decreases with decreasing crystallization temperature, resulting in perfect CO_2 adsorption energy. The

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size and shape of the NaY zeolite crystals did not change after modification, which was higher in surface area, micropore volume, and pore volume than pure zeolite [14]. The characterization of the zeolite made from 25 L hydrothermal proved that the process was good, using geochemical methods as a result of the apparent activation energy reaction at a temperature range of 477-531 K, which were identified to be 2.16 and 46.61 kJ/mol, respectively. In this study, it was proposed that hydrothermal technology be used for the comprehensive utilization of silicates [15].

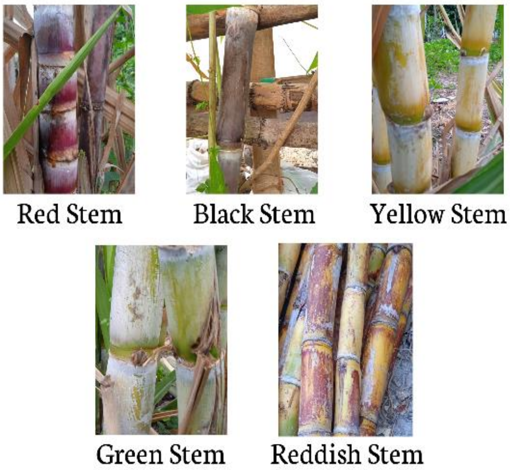

Figure 1. Variability of the stem external colour of the sugarcane (*Saccharum officinarum L.*) cultivars collected in Lombok, Indonesia

This research aims to synthesize and characterize zeolite from sugarcane biomass with black stem as a source of silica. In this research, silica was obtained using the sol-gel method. This method was chosen because the process takes place at low temperatures, is relatively easy, can produce products with high purity and homogeneity, and can be applied in all conditions [16]. Zeolite synthesis was carried out using the hydrothermal method to get a good level of crystallinity when compared to the sol-gel method [17]. Sugarcane (*Saccharum officinarum L.*) has a variety of external stem colours, namely black, wine red, green, yellow, red, reddish, and dark green stems [1]. The diversity of outer stem colours in sugarcane cultivars is shown in Figure 1. To our knowledge, previous research was only carried out using *Saccharum officinarum L.* with green stems.

Meanwhile, research using *Saccharum officinarum L.* with a black stem has not been widely carried out. The difference in silica yield values (%) between *Saccharum officinarum L.* with a black stem from this research and a green stem from previous research must be studied further. This underlies the need to research advanced zeolite synthesis using silica from *Saccharum officinarum L.* with a black stem, while alumina was sourced from aluminium foil. The results of this zeolite synthesis were then characterized using Fourier Transform Infrared Spectroscopy (FTIR) of the resulting SiO_2.

Research Methods

Materials and Tools

The materials used in this study included *Saccharum officinarum L.* with black stem ash, sodium hydroxide (NaOH 5 M) with a ratio of 1:5, concentrated hydrochloric acid (HCl), hot distilled water (H_2O), sodium carbonate (Na_2CO_3), aluminium foil (Al_2O_3), purified water, and hydrochloric acid (HCl). The tools used in this study were Erlenmeyer, hot plate, vacuum butcher, magnetic stirrer, mortar, pestle, weighing, oven, petri dish, porcelain cup, furnace, plastic wrap, autoclave, porcelain crucible, Bucher funnel, Whatman filter paper, burettes, pipettes, pH sticks, and vials.

Sample preparations

The raw materials for this research were *Saccharum officinarum L.* with black stem and aluminium foil. Materials for silica extraction from Saccharum waste are prepared based on modified previous research methods [18]. Saccharum waste that has been squeezed is cleaned and then dried conventionally in the sun. Once dry, Saccharum is burned using a furnace above 500^{o}C and checked every 1 hour until the bagasse turns ash. The ash obtained is then separated from impurities by sieving. Once smooth, the ash is then stored for the silica extraction process. The aluminium foil sample was prepared based on previous research methods [7]. The aluminium foil samples were cut into small pieces of around 10x10 mm to facilitate the alumina synthesis process.

Silica Extraction

Silica extraction was carried out using the sol-gel method, referring to previous studies [19]. This process is carried out by adding a solution of 200 mL of NaOH to an Erlenmeyer, which already contains 40 g of Saccharum ash. The mixture was then homogenized and heated for 30 minutes at 120°C using a hot plate. To obtain the filtrate, the mixture that has been cooled is then filtered using a butcher vacuum with one layer of filter paper. The next process is the pickling process. The resulting filtrate was added with concentrated HCl to pH 7, left for 24 hours, stirred with a magnetic stirrer, and then filtered. The silica obtained was washed with hot distilled water up to pH 7 to remove impurities. The silica was then dried for 18 hours at 85°C. After drying, the silica is crushed using a mortar until smooth. Next, the silica obtained is weighed to determine the percent yield obtained using **Equation 1** [20].

$$R = \frac{\mathrm{WS}}{WA} \times 100\% \qquad \text{(Eq. 1)}$$

Information :
R: Yield (%)
WS: Weight of silica (g)
WA: Weight of ash (g)

Alumina Synthesis

The synthesis of alumina refers to previous studies [7] carried out through the precipitation method or deposition with a concentration of hydrochloric acid. A 5.29 g of aluminium foil is cut into small pieces and 22.5 grams of sodium carbonate (Na_2CO_3) powder. The aluminium foil was dissolved in the 31.34% 6M HCl solution, which had previously been prepared by dilution to obtain an aluminium chloride ($AlCl_3$) solution. 25 mL of distilled water was added to the mixture, then filtered using a vacuum with 3 layers of Whatman filter paper to obtain precipitate and filtrate. The filtrate was added with 22.5 grams of sodium carbonate (Na_2CO_3) and stirred with a magnetic stirrer until it hardened into a coarse gel. The mixture was then rinsed with 600 mL of distilled water and waited for it to settle. The next process is decantation, or separating the mix between solution and solid. The precipitate is put into a petri dish and then baked in the oven for 2 hours at 150°C to get $Al(OH)_3$. The oven results were transferred to a porcelain cup and calcined using a furnace for 2 hours at 600 °C to obtain alumina (Al_2O_3).

Zeolite Synthesis

Zeolite synthesis was carried out using the hydrothermal method, which refers to previous research [17] which has been modified. Sodium silicate solution (Na_2SiO_3) made from 5 g of SiO_2 and mixed with 20 mL of 8 M NaOH. The mixture was heated for 3 hours at 50°C, covered with plastic wrap and stirred with a magnetic stirrer. Sodium aluminate solution ($NaAlO_3$) is prepared from 3 g of Al_2O_3 mixed with 10 mL of 8 M NaOH. The mixture was heated for 1 hour at 50°C and stirred using a magnetic stirrer. The sodium aluminate solution ($NaAlO_3$) is slowly added to the slightly clear sodium silicate (Na_2SiO_3) solution, and the magnetic stirrer is removed from the solution. The poured solution was stirred slowly until homogeneous for 3 hours, and then a white gel was formed. The gel formed was then put into the autoclave and heated at 150°C for 10 hours. The formed zeolite was placed in a porcelain crucible and then heated in the oven for 14 hours at 150°C. The ya oven results were crushed with a mortar and pestle until smooth. The zeolite was weighed, and the percent yield (g/g) was calculated from the ratio of the weight of the calcined zeolite to the dry weight of Al_2O_3 and SiO_2 in the synthesis solution based on the following Equations 2 [21].

$$R = \frac{ZP}{SP + AP} \times 100\% \qquad \text{(Eq. 2)}$$

Information:
R: Yield (%)
ZP: Zeolite produce (gram)
SP: Silica produce (gram)
AP: Alumina produce (gram)

Results and Discussion

Alumina Content of Aluminum Foil

The aluminium foil used in this research is usually used for cooking or wrapping food. When AFW is dissolved with HCl, it will produce a product in the form of $AlCl_3$ where the solution is then vacuumed and washed to obtain $AlCl_3$ which is cleaner and purer from impurities. This reaction will also produce hydrogen, so it must be done outdoors or using air ventilation. $Al(OH)_3$ is then formed from the response described in the method and is characterized by the formation of a white precipitate. Alumina is then formed by heating $Al(OH)_3$ and releasing H_2O.

Meanwhile, the calcination temperature used in this research was 600°C. The goal is to obtain a larger alumina surface area (m^2g^{-1}). This refers to previous research [7], which showed that calcination temperatures of 550°C and 675°C obtained a higher alumina surface area than at a temperature of 800°C. This study found that 5.29 g of aluminium foil produced 14.29 g of alumina with the addition of 22.50 g of Na_2CO_3. The amount of alumina produced increases as the acid concentration increases and will become zero when the acid concentration is equal to 1M. Calcination temperature significantly influences the surface area of the alumina produced [7].

Silica Content of Saccharum Biomass

This research obtained a silica weight of 20 g with white granules. The results showed that the percentage by weight of silica in the samples treated with the addition of acid was higher. This is because the HCl addition method helps remove impurities from the Saccharum ash. The results obtained are similar to previous research [3] about sugar cane as a renewable source of silica for the synthesis of SBA-15 using concentrated acid in silica extraction so that it can increase the weight percentage of silica in the ash. The weight percentage of silica obtained in previous research [3] was 53.10%, whereas the research used *Saccharum officinarum L.* with a green stem.

In comparison, the amount of acid solution used in this study was higher than in the latest study [3]. These different methods only aim to remove impurities. The results obtained and their levels are presented in Figure 2.

(a) Silica (20g) (b) Alumina (14,29g) (c) Zeolite (3,88g)

Figure 2. Product Results in grams

The calculation of the silica yield content refers to Equation 1 so that the percentage (%) silica reduction obtained is 50%. The yield value obtained is less than that of the latest research [3], which yielded 53.10%. This could happen because the method used in previous research used acid treatment to produce higher silica purity. Meanwhile, compared with another research [4], which obtained a yield (%) of 49.98%, the value obtained was higher. This is because this research used Saccharum (bagasse) ash through a combustion process, whereas the previous study [4] used Saccharum (bagasse) fly ash. This shows that sugarcane bagasse ash has a greater silica content than sugarcane fly ash.

Table 1. The Silica Obtained from Previous Studies

Research	Treatment / Method	Parameter	
		Yield (%)	Physical Appearance
[3]	Pyrolysis and acid treatment method	53.10	
[4]	Sol-gel hydrothermal method	49.98	White granules
[22]	Acid treatment, ion exchange treatment, and washing with de-mineralized water	50.00	

The data from the table shows that silica from *Saccharum officinarum L.* with a black stem has a fairly high yield compared to *Saccharum officinarum L.* with a green stem.

Characteristics of Silica with FTIR

Figure 3 shows the spectrum of FTIR results for silica from Saccharum. The function of FTIR is to determine the functional groups of silica obtained. Figure 3 shows absorption in the wavelength region of 3391 cm^{-1}, 1632 cm^{-1}, 1061 cm^{-1}, and around 790 cm^{-1}. Identification in Table 1 shows that silica has Si-O-Si groups. This is due to the addition of acid, which will protonate the siloxy group (Si-O-) and cause it to become silanol (Si-OH). Siloxy groups then attack the silanol groups formed to form siloxane bonds (Si-O-Si), as evidenced by absorption at wave number 1061 cm^{-1}. It was also obtained that the SiO_2 spectrum produced in this study had similarities with the reference SiO_2 spectrum [19], as shown in Table 2. It can be seen from the absorption band with a wavelength of 3391 cm^{-1} that there is a wide absorption, which indicates the stretching vibration of the -OH group of Si-OH. This is supported by previous research [23], where the absorption band at 3480 indicates vibrations from the OH group. The presence of the OH group is again confirmed by the appearance of absorption at wave number 1632 cm-1, which suggests the existence of bending vibrations in the -OH group from Si-OH [24]. This is in line with previous research, which shows that there is absorption at a wave number of 3468.01 cm^{-1} and 3471.87 $cm^{-1,}$ which shows the -OH stretching vibration in Si-OH and an absorption band of 1639.49 $cm^{-1,}$ which indicates the bending vibration of the OH group [19].

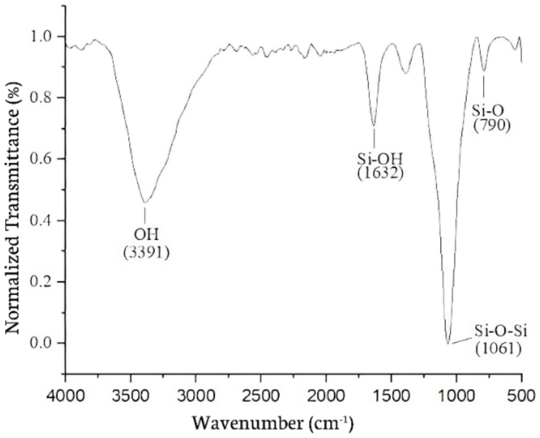

Figure 3. SiO_2 FTIR of Saccharum

The 1061 cm^{-1} wave absorption indicates the bending vibration of Si-O-Si [24]. In FTIR spectroscopy, each compound has a unique IR spectral absorption band due to the fingerprint spectroscopy's nature. The band absorption data in FTIR shows that silica has been formed from the ash of *Saccharum officinarum L.* with a black stem.

Table 2. Silica FTIR Absorption Bands

Wave Number (cm^{-1})		Interpretation [19]
Silica [19]	Sample	
3468.01 and 3471.87	3391	Stretching vibration of the O-H group from the Si-OH bond
1639.49	1632	Bending vibration of the O-H group from the Si-OH bond
1091.71 and 1095.57	1061	Stretching vibration of $Si-O^-$ from the Si-O-Si bond
800.46	790	Asymmetric stretching vibration of $Si-O^-$ from the Si-O-Si bond

Zeolite Synthesis

The formation of zeolite starts from reacting Na_2SiO_3 and $NaAlO_3$. The gel formed during the zeolite formation process is a stable meta phase, which then moves or migrates ions to reach the final stable phase of the reaction, namely the formation of zeolite crystals. The dissolved amorphous gel will experience structural rearrangement due to the heating process, which forms the embryonic crystal nucleus phase at the crystal formation stage. Then, the amorphous gel slowly forms embryonic crystal nuclei, and crystal growth occurs until the amorphous gel runs out so that crystals form stable [25].

The zeolite obtained in this research was 3.88 g, as shown in Figure 2. Calculations using Equation 2 produce the yield (%) of zeolite, around 48.5% of the 5 grams of silica and 3 grams of alumina produced (5:3 ratio of SiO_2:Al_2O_3). The hydrothermal method in this research functions in the crystallization process where the zeolite crystals obtained are clean white. The resulting zeolite will then be further analyzed using FTIR to identify the vibrations of its functional groups.

FTIR Analysis of Zeolite Synthesis

The FTIR spectrum for zeolite synthesis displays the absorption at wave number presented in Figure 4. Based on the absorption bands obtained, the SiO_2 spectrum produced in this study is similar to the reference zeolite spectrum [12], as evidenced by the difference in wavenumber between the study results and the reference (Table 3). The FTIR data obtained shows that there is absorption at a wavelength range of 960 cm^{-1}, indicating absorption from the Si-O group's strain. This aligns with previous research [1] showing an absorption band at 975 cm-1, indicating a Si-O group's presence. At a wavelength range of 729 cm^{-1}, Al-O bending vibrations detected absorption. The absorption band with a wavelength of 668 cm^{-1} indicates the presence of symmetrical stretching vibration absorption of O-Si-O or O-Al-O. This is in line with previous research [26]. This was further clarified by the next research [12], which stated the existence of Si-O asymmetric vibrations at a wave number of 690 cm^{-1}.

Based on Figure 4, it can be observed that OH groups were not detected in the resulting zeolite. The loss of OH in the absorption band is due to the water content of the zeolite having been greatly reduced due to heating carried out for 14 hours at a temperature of 150ºC. This also identifies the zeolite obtained in this study as similar to Zeolite X from other research [12]. Zeolite from silica *Saccharum officinarum L.* with a black stem has been successfully formed.

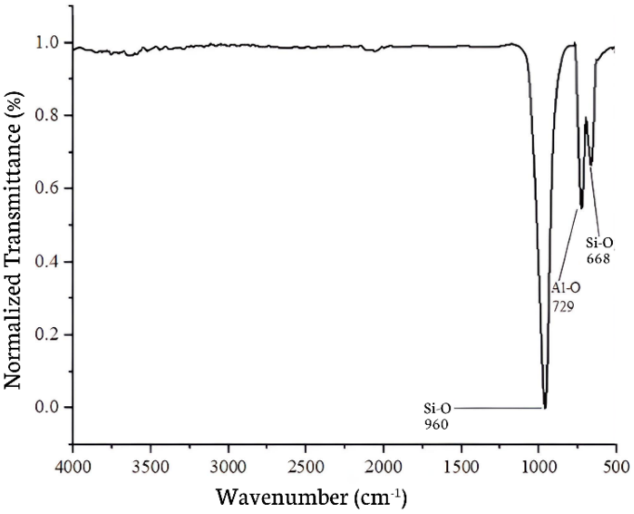

Figure 4. Zeolite FTIR of Saccharum

Table 3. Reference of Zeolite FTIR Absorption Bands

Wave Number (cm^{-1})		Interpretation
Zeolite [12]	Sample	
975	960	Stretching vibration Si-O [27]
790	729	Bending vibration Al-O-Si [28]
690	668	Asymmetric stretching vibrations of O-Si-O and O-Al-O [29]

Conclusion

Zeolite synthesis has been successfully done using silica from sugarcane bagasse ash and alumina from aluminium foil. The weight of the ash used was 20 grams and produced a silica yield percentage of 50%. Meanwhile, alumina obtained from the synthesis of aluminium foil (5.29 grams) produces alumina weighing 14.29 grams. The zeolite obtained from the hydrothermal process was 3.88 grams, with the SiO_2:Al_2O_3 ratio used being 5:3. The weight of silica and alumina obtained gave a zeolite yield percentage of 48.5%. The zeolite obtained was pure white and had an FTIR spectrum similar to Zeolite X from previous research. This research is expected to be useful in increasing the effectiveness of the silica extraction process from *Saccharum officinarum L.* with black stem.

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