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# Utilization of zeolite H-MOR based on bagasse ash silica as a catalyst for the hydrolysis reaction of cassava peel cellulose for glucose production

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**Abstract.** Bagasse and cassava peel are waste that can be increased economic value. In this study, we have successfully synthesized zeolite H-MOR from sugarcane bagasse ash (SCBA) silica using the Steam-Assisted Crystallization (SAC) method at 170°C for 120 hours and determined its catalytic activity on the hydrolysis of cassava peel starch to produce glucose. H-MOR synthesized using LUDOX (commercial silica) was used as a comparison. The results showed that the MOR zeolite synthesized using SCBA and LUDOX silica had a crystallinity of 92.12% and 81.17%, respectively. The cellulose content in cassava peel flour is 57.8%. Optimization of the catalytic test variable showed that the hydrolysis of cellulose occurred at 140°C for 4 hours with a 1: 1 ratio of catalyst and substrate. The glucose concentrations obtained from cellulose hydrolysis using zeolite H-MOR catalyst from SCBA and Ludox were 398.5 ppm and 237.45 ppm with conversion degrees of 60.4% and 61.2%, respectively.

**Keyword:** glucose, H-MOR, hierarchically porosity, sugarcane bagasse ash, and steam-assisted crystallization

## 1. Introduction

Cassava production in Indonesia during 1980-2016 tended to increase. The Province of Lampung is the center for the largest cassava harvest in Indonesia with a total production of 27.17% [1]. Cassava processing will produce cassava peel waste which can have an impact on the environment [2]. However, polysaccharides in cassava peels (starch, cellulose, and hemicellulose) can be hydrolyzed



to produce monosaccharides such as glucose to produce fuels such as bioethanol [3] as well as high quality chemicals such as 5-HMF (5-Hydroxy-Methyl-Furfural), an acid levulinate, and others [4]. Thus, the process of hydrolysis of cellulose to glucose is often said to be the first step into the biorefinery field [5]. The nature of cellulose which is a crystalline polymer with high chemical stability causes a slow hydrolysis reaction of cellulose so that a catalyst is needed to accelerate the reaction [6].

Hydrolysis reactions usually use enzymes as catalysts. This is due to the high selectivity of the enzyme, but the reaction takes a long time and is expensive [7]. Homogeneous catalysts provide good catalytic activity at low cost but less efficient because they produce a mixture that is difficult to separate and causes large amounts of waste [8]. Heterogeneous catalysts are widely used to overcome the limitations of the two types of catalysts both economically and their impact on the environment [9]. One of the heterogeneous catalysts that is often used is zeolite [10].

Zeolite is a crystalline aluminosilicate material with regular micropores (<2nm) composed of  $\text{SiO}_4$  and  $\text{AlO}_4$  tetrahedral. Nowadays more than 200 types of synthetic zeolites have been discovered with pore sizes depending on the structure of the framework. Due to its unique properties including large surface area, high chemical and thermal stability, zeolite's adjustable form selectivity and acidity has been widely used as a catalyst in various chemical processes such as transesterification, cracking, isomerization, alkylation [11], including the hydrolysis of cellulose. Mordenite is a synthetic zeolite that is commonly used in various catalytic reactions. Zeolite MOR can be synthesized without the addition of templates and using natural silica from Sugarcane Bagasse Ash (SCBA) thereby minimizing production costs [12].

In this study, the catalytic activity of zeolite MOR was tested using silica from bagasse ash (SCBA) in the hydrolysis reaction of cassava peel cellulose and compared its catalytic activity with zeolite MOR synthesized using LUDOX commercial silica. The catalytic test variables carried out included temperature, time and amount of catalyst. To increase glucose production, efforts were made to increase the solubility and decrease the degree of crystallinity of cellulose using the mix-milling technique.

## 2. Experimental Methods

### 2.1 Materials

The starting materials used in this research are bagasse from PT. Adi Karya Gemilang at Terbanggi Besar, LUDOX (HS-40) (colloidal silica, Sigma Aldrich), sodium hydroxide (NaOH, Merck), hydroxide (Al (OH)<sub>3</sub>, Sigma-Aldrich), nitric acid (HNO<sub>3</sub>, Sigma- Aldrich), cassava peel, sulfuric acid (H<sub>2</sub>SO<sub>4</sub>, Sigma-Aldrich), calcium carbonate (CaCO<sub>3</sub>, Sigma-Aldrich), ammonium nitrate (NH<sub>4</sub>NO<sub>3</sub>, Merck), and deionized water (H<sub>2</sub>O). All the commercial reactants were in reagent grade and used without further purification.

### 2.2 Silica Extraction from Bagasse

Bagasse was immersed in 2.5% HNO<sub>3</sub> and heated at 80°C for 1 hour, rinsed with distilled water and oven-dried at 100°C. The dry bagasse is burned using a furnace at a temperature of 600°C for 5 minutes to produce bagasse ash (SCBA). Silica extraction from bagasse ash was preceded by dissolving SCBA in 2 M NaOH and heated at 80°C to boiling for 1 hour. The mixture was left at room temperature for 24 hours and filtered to obtain a brownish yellow filtrate. The filtrate was added with 10% HNO<sub>3</sub> solution dropwise to form a hydrogel at pH 7. The hydrogel formed was left for 48 hours to form a gel, dried at 80°C for 24 hours. The silica obtained was then characterized and used as a precursor for the synthesis of MOR zeolite.

### 2.3 Synthesis of Zeolite MOR Using the Steam-Assisted Crystallization (SAC) Method

Zeolite MOR was synthesized using silica of bagasse ash and LUDOX with a molar composition of 1 SiO<sub>2</sub>: 0.229 NaOH: 30 H<sub>2</sub>O: 0.184 Al<sub>2</sub>O<sub>3</sub> [13]. The mixture was stirred and heated at 60°C until a dry gel was obtained. 2 g of dry gel is put into a small Teflon and placed in a larger teflon containing 10 mL of water. The larger teflon was put into autoclave stainless steel and heated at 170°C for 120 hours. The resulting solid was washed and dried at 90°C. the resulting product was calcined at a temperature of 550 °C for 6 hours. The zeolite samples were ion exchanged using 2 M NH<sub>4</sub>NO<sub>3</sub> solution and subsequently followed by calcination at 550 °C for 6 hours to obtain protonated zeolite (H-MOR).

### 2.4 Characterizations

Silica SCBA and zeolite MOR were characterized using X-ray diffraction (XRD), X-ray fluorescence (XRF), Fourier-transform infrared spectroscopy (FTIR), Scanning Electron Microscope (SEM), N<sub>2</sub> physisorption isotherm [14].

### 2.5 Catalyst Activity Test

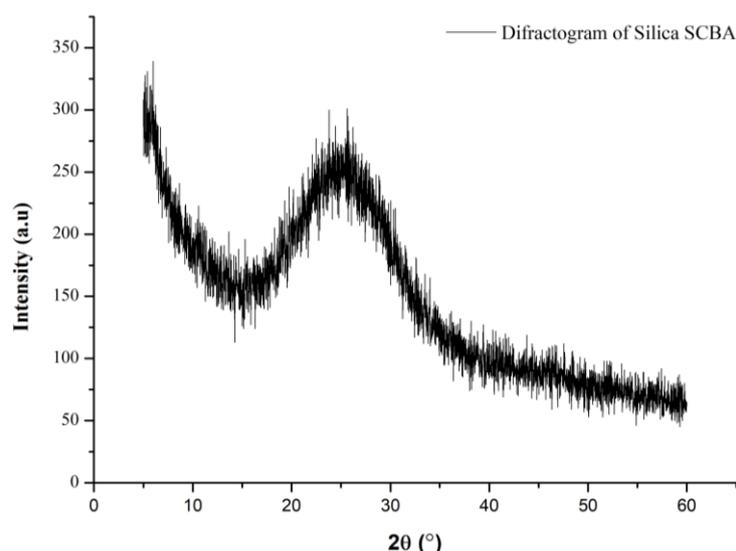
#### 2.5.1 Hydrolysis Reaction

0.03 g H-MOR (SCBA and ludox) is mixed with 0.05 g of cellulose flour. The mixture was then crushed using a mortar and pestle for 24 hours and put into Teflon containing 5 mL of distilled water [15]. The hydrolysis reaction was carried out at 140°C for 3 hours. The hydrolyzed mixture was cooled and separated from the precipitate using filter paper. The glucose content in the filtrate was analyzed using UV-Vis at a wavelength of 540 nm using the Mendels method. Optimization of the catalytic test variables carried out included variations in temperature, time and amount of catalyst.

## 3. Results and discussion

### 3.1 Silica from Sugarcane Bagasse Ash (SCBA)

The structure and phase of SCBA silica were determined by XRD at  $2\theta = 5^\circ - 60^\circ$  as shown in Figure 1. The amorphous phase is characterized by peaks extending around  $2\theta = 22^\circ$  [16].



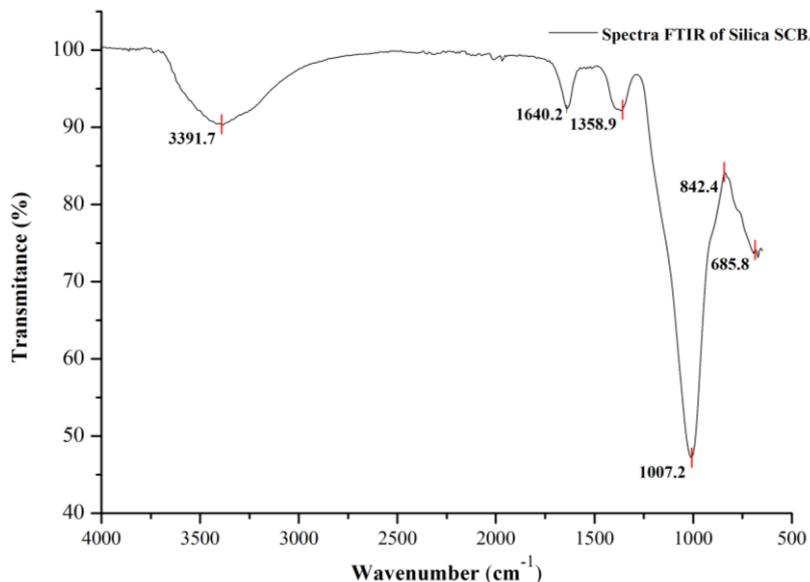
**Figure 1.** Diffractogram of SCBA silica

The diffractogram in Figure confirmed that the typical peaks of quartz silica were not detected in the SCBA silica. The amorphous structure of silica makes it easy to rearrange the structure into a zeolite framework [17]. The results of XRF observation that the SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> content in the extracted silica products were 79.47% and 18.13%, respectively. The composition of the compounds in the extracted silica is shown in Table 1.

**Table 1.** Chemical composition of SCBA silica

Compound	Composition (%)
SiO <sub>2</sub>	79.477
MgO	0.483
Al <sub>2</sub> O <sub>3</sub>	18.135
P <sub>2</sub> O <sub>5</sub>	1.243
K <sub>2</sub> O	0.05
CaO	0.321
TiO <sub>2</sub>	0.006
Cr <sub>2</sub> O <sub>3</sub>	0.008
MnO	0.001
Fe <sub>2</sub> O <sub>3</sub>	0.11
ZnO	0.085
Ga <sub>2</sub> O <sub>3</sub>	0.001
Ag <sub>2</sub> O	0.063

The functional groups in SCBA were identified using FTIR as shown in Figure 2. Silanol (Si-OH) and siloxane (Si-O-Si) functional groups are the main functional groups of SCBA silica. The OH group from Si-OH appeared at the wave number 3391.7 cm<sup>-1</sup> and the Si-O-Si asymmetric stretching vibrations were 1640.2 cm<sup>-1</sup> and 1007.2 cm<sup>-1</sup>. The wave number 685.8 cm<sup>-1</sup> is identified as symmetric Si-O-Si.



**Figure 2.** FTIR spectra of Silica SCBA*3.2 Zeolite Mordenite*

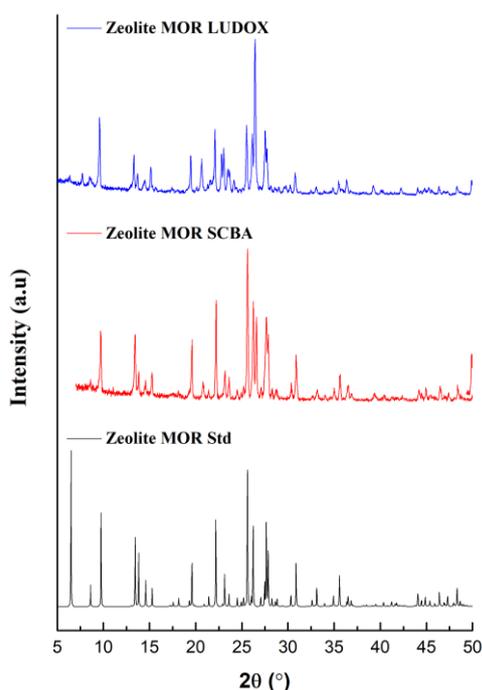
The synthesis of zeolite MOR was carried out using the Steam Assisted Crystallization (SAC) method where the crystallization of MOR zeolite was assisted by water vapor [18]. Water vapor will be absorbed into the intermediate gel pores thereby facilitating crystallization by increasing diffusion, controlling the distance between precursors, assisting migration, and participating in crystal growth.

The formation of a crystal nucleus (nucleation) and crystal growth are the two most important steps in the crystallization of zeolites. During the induction period, rearrangement of the aluminosilicate amorphous gel will occur and form a small amount of crystal core as long as the precursor mixture is heated. The synthesis process includes a series of chemical reactions that occur in the liquid and solid phases through hydrolysis / dissolution reactions, and condensation [19].

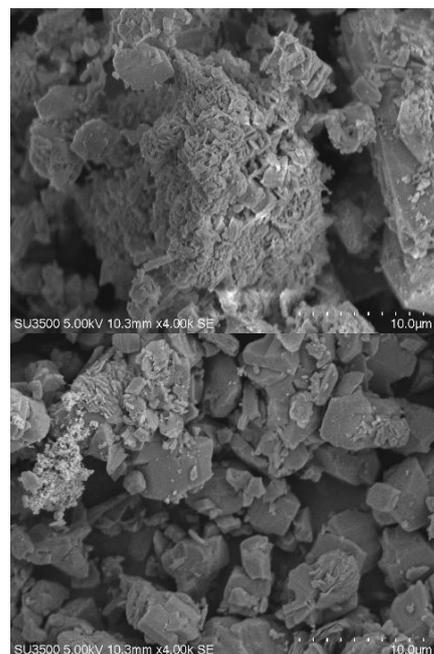
During the dry gel formation process, a number of small clusters of primary building blocks are formed as a result of the reaction between the silicate anion  $[\text{SiO}_4]^-$  and  $[\text{Al}(\text{OH})_4]^-$  where this aluminate ion will give a negative charge  $[\text{Al}(\text{OH})_4]^-$  tetrahedral. In alkaline solution and combine to form a  $\text{Na}^+$  cation stabilized zeolite aluminosilicate framework. The high concentration of reactants in xerogels allows the formation of a crystal nucleus from the combination of a number of small clusters of primary building blocks [20-21]. In the next crystallization process, the gel will shrink while others become zeolite nucleus and grow rapidly by consuming nutrients (precursors) to become nanocrystallites [22-23]. After orienting each other, these nanocrystals then form a mesoporous network [13, 18].

Figure 2 shows that the zeolite-MOR synthesis has been successfully carried out. The relative intensity which represents the crystallinity level of each sample [24] shows that zeolite-MOR LUDOX and zeolite-MOR SCBA have crystallinity levels of 81.17% and 92.12%, respectively. Both samples have relatively high crystallinity, which is shown by the XRD diffractogram that both have good peak sharpness and fairly flat baseline. Hu et al (2008) also succeeded in synthesizing zeolite-MOR with a crystallinity of 91%.

Zeolite MOR has a crystallite size of 50-200 nm [25]. In this study, the crystallite sizes of zeolite-MOR LUDOX and zeolite-MOR SCBA were 148.75 nm and 216.72 nm, respectively. Crystallite size is obtained using the Scherer equation and is the average overall particle size versus individual crystallite size. The difference in crystal size is caused by the difference in the rate of nucleation that occurs. The faster the nucleation rate occurs, the smaller zeolite will be produced [26].



**Figure 3.** Diffractogram from ZeolitMOR (a) Standard (b) SCBA (c) LUDOX



**Figure 4.** The morphology of hierarchical pore MOR zeolite based on silica SCBA

Zeolite MOR (SCBA) and zeolite-MOR (LUDOX) which have been identified as having a crystal phase through XRD characterization are further characterized using SEM to see the morphology of the samples shown in Figures 3 and 4.

Some of the zeolite-MOR morphologies that have been reported are needle-like, fiber-like, rod-like, prism-like and spherical like crystals [23, 27]. The morphology of the LUDOX-MOR zeolite sample shown in Figure 4 has the same short, fused rod shape as reported by Zhang et al (2011) with the addition of a sphere of non-uniform size. Stem crystals like this are common in the synthesis of zeolite-MOR based on colloidal silica because the form of  $\text{SiO}_2$  hydrate in colloidal silica has been well dispersed [18].

### 3.3 Catalyst Activity Test

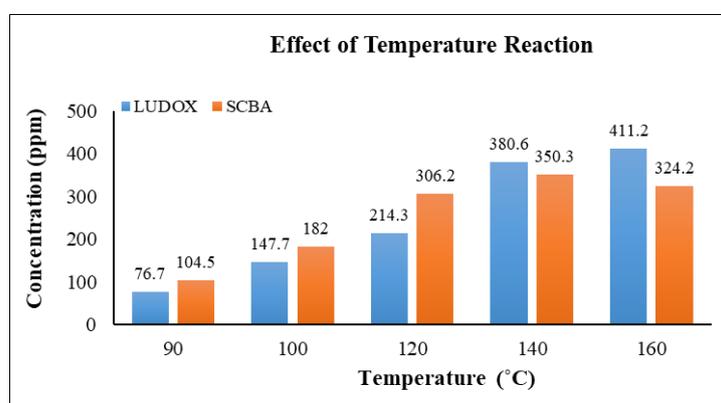
#### 3.3.1 The hydrolysis reaction

The hydrolysis reaction of cellulose was carried out using 0.05 g of cellulose and 0.03 g of H-MOR zeolite catalyst using 5 mL of distilled water. The results of hydrolysis of zeolite H-MOR (LUDOX) and H-MOR (SCBA) in the form of glucose filtrate as much as 2.5 mL and 2.7 mL with residual deposits of 0.06 g and 0.07 g. The conversion percentages of cellulose for each catalyst were 28.12% and 12.14%. The reducing sugar concentration produced with the LUDOX H-MOR zeolite catalyst was 370.16 mg / L, while the SCBA H-MOR zeolite only produced a reducing sugar concentration of 227.8 mg / L. It is known that the amount of cellulose converted with the help of zeolite H-MOR SCBA produces more by-products so that the glucose concentration is lower.

#### 3.3.2 Determination of the optimum temperature

The results showed that the concentration of reducing sugars increased with increasing reaction temperature when using LUDOX zeolite-MOR as a catalyst. Meanwhile, the hydrolysis reaction

catalyzed by zeolite MOR SCBA shows a decrease in catalytic activity at 160 °C as seen in Figure 5. At lower temperatures (90°C, 100°C, and 120°C) the  $\beta$ -1 bond breaks, 4 glycosidic in cellulose does not occur completely so that its conversion to glucose has not been fully achieved. Meanwhile, if the reaction is carried out at too high a temperature (160°C) the reducing sugar products formed tend to dehydrate and form byproducts such as levulinic acid, formic acid, and 5-HMF [15] on zeolite-MOR SCBA reducing sugar concentrations experiencing a decrease even though the% conversion was obtained at a temperature of 160°C managed to reach 53%. The hydrolysis reaction using zeolite-MOR LUDOX still experienced an increase in reducing sugar concentration along with the increase in temperature, but the increase was not significant and there was a lower% conversion at high temperatures, namely 32.8% from the previous 65.6%, so it can be said that LUDOX zeolite-MOR is not able to run optimally at too high temperature. Based on the results obtained, the optimum temperature that is good for use in the hydrolysis of cellulose using zeolite H-MOR is 140°C.

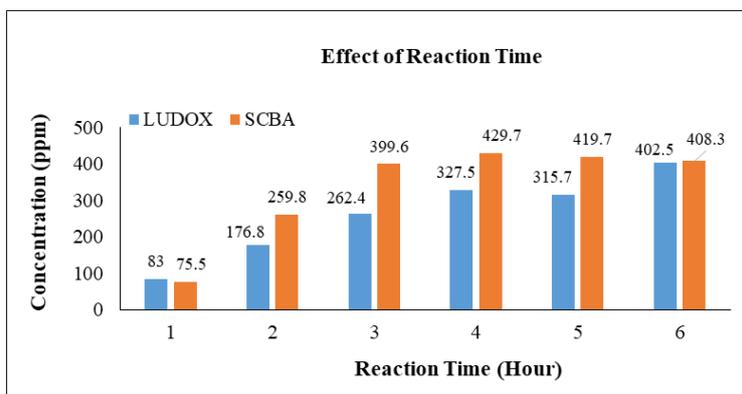


**Figure 5.** The effect of the temperature of the hydrolysis reaction of cassava peel cellulose on the resulting glucose concentration

### 3.3.3 Determination of the optimum time

The hydrolysis reaction catalyzed by the zeolite H-MOR LUDOX showed an increase in the reducing sugar concentration and the percent conversion until the reaction time was 6 hours. However, the increase in concentration did not show a significant difference at 4 hours of reaction. This is in accordance with the findings of Lanzafame et al (2019) where an increase in reaction time will increase the productivity of cellulose conversion, but it can also decrease the cellulose yield of glucose products.

The increase in the percentage of cellulose conversion in the hydrolysis reaction using the Zeolite H-MOR SCBA catalyst was accompanied by a decrease in the concentration of reducing sugars over the reaction time of 4 hours. The reaction time that is too long results in a decrease in the stability of the sugar products formed and leads to further decomposition reactions into 5-HMF products and levulinic acid [28].

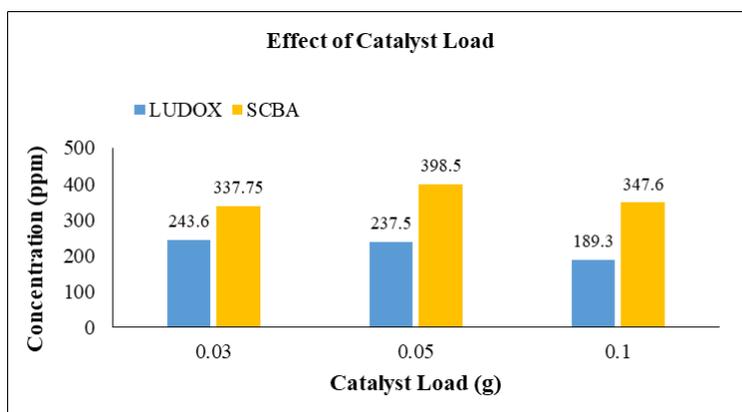


**Gambar 6.** Effect of hydrolysis reaction time of cassava peel cellulose on the resulting glucose concentration

3.3.4 *Determination of the optimum amount of catalyst*

In determining the optimum amount of catalyst, 0.05 g cellulose is mixed with 0.03 g; 0.05 g; and 0.1 g of catalyst so that the catalyst / cellulose ratio is (0.6: 1); (1: 1); and (2: 1). The hydrolysis reaction catalyzed by zeolite H-MOR SCBA produces optimum glucose when using a catalyst as much as 0.05 g (the ratio of catalyst to cellulose is 1: 1). Meanwhile, the hydrolysis reaction catalyzed by zeolite H-MOR (Ludox) showed a decrease in catalytic activity with an increase in the amount of catalyst (figure 7). However, the resulting activity is lower when compared to the use of the H-MOR catalyst from SCBA.

Increasing the amount of catalyst does not lead to a relevant increase in the contact area [29]. Basically, the excess zeolite will cover the first zeolite layer which has previously been in contact with cellulose fibers. Likewise with the glucose units that are formed which in turn, in the presence of excess zeolite can catalyze further transformation of the glucose units. The differences in catalytic activity possessed by LUDOX H-MOR zeolites and SCBA H-MOR zeolites can occur from many factors, especially from the interaction between catalyst and cellulose which is influenced by the morphology and surface area of the catalyst, causing differences in the ratio of cellulose and the optimum catalyst used.



**Figure 7.** The effect of the amount of catalyst on the hydrolysis reaction of cassava peel cellulose on the resulting glucose concentration

#### 4. Conclusion

The results showed that zeolite-MOR SCBA has been successfully synthesized using aluminosilicate sources derived from bagasse ash. The hierarchical pore H-MOR zeolite catalyst based on commercial silica LUDOX and SCBA silica has good catalytic activity in the cellulose hydrolysis reaction with optimum conditions obtained at 140 °C for 4 hours with a catalyst and cellulose ratio of 1: 1.

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