

Hydrolysis of Eichhornia Crassipes to Glucose over Sulfonated Active Carbon Catalyst

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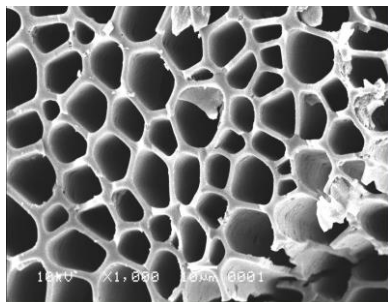
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GRAPHICAL ABSTRACT



ABSTRACT

Enzymatic process and acid hydrolysis process are common process for conversion of cellulose to glucose. Unfortunately, the two processes are expensive process and corrosive process. Hence, the new process, that use sulfonated active carbon catalyst is important to developing. The sulfonated active carbon is made from carbonated coconut shell under temperature at 350°C. After carbonation, sulfonated active carbon soaked under sulphate acid 96% at 150°C until 15 hours. The result is then washed and dried, and tested catalyst characteristics in the form of H⁺ capacity, pore size catalysts by used BET surface area, functional groups by used FTIR, and morphology catalyst structure by used SEM. Catalyst performance was tested in an autoclave reactor through a hydrothermal process with difference of the catalyst amount and temperature. The results showed that the test characteristics of H⁺ capacity is 2.95 mmol/g, the pore size is 29 m²/gr. FTIR analyze showed that the presence of sulfonate groups read at a wavelength of vibration 1750 cm⁻¹ and 1379 cm⁻¹. By SEM analyze showed that the morphological structure of sulfonated active carbon is more open than other catalyst. By testing catalyst, the highest conversion of glucose is 87.2 %.

Key words: Cellulose, Glucose, eceng gondok, sulfonated active carbon

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1. INTRODUCTION

Biomass has the potential to be rich in lignocellulosic materials. Today in the developed countries started to do research and development there's even down to the level of production how to convert biomass materials into ethanol (Alico, et.al. 1982). Bioethanol is expected to gradually become an alternative fuel stations. As we know that gasoline is obtained from the refining of crude oil, while crude oil prices tend to fluctuate, while imports of crude oil and processed products to meet domestic demand also increased, it takes strategic measures to conserve conventional energy reserves, maintain availability of energy, and reduce foreign exchange expenditure of this sector (Kosaric, et.al., 1981).

Cellulose is made into a stage of cellulosic ethanol is converted into glucose, and glucose is converted to ethanol. The technology has been used for this is the process of enzymatic and acid hydrolysis. Both of these technologies are still having technical problems, ie for the price enzymatic enzymes are expensive and vulnerable to changes in operating conditions, while acid hydrolysis process is corrosive and cause waste, requiring the development of new technologies to improve the technical issues.

Acid hydrolysis proceeds in three steps. The reaction is initiated by a proton from the acid to interact

quickly with glycosidic oxygen linking two sugar units, thus forming bonds conjugate acid. The split of the CO bond and the acid conjugated to cyclic carbonium ion then broke into sugar free and carbonyl groups are not stable. After the addition of water fast, carbonium intermidiet into sugar, as it re-released acid group H⁺, and so on.

To solve the technical problems of the solid acid catalyst are made sulfonated activated carbon catalyst. This catalyst is made by reacting activated carbon with acid sulfuric acid. The group which containing sulfonate group H⁺ could as acid site. The technology which used sulfonated activated carbon is a new new technology. This technology is being developed and studied in Japan and other countries. From several studies of this catalyst are conclude that this catalyst is capable to transforming cellulose into glucose. Onda, et al. (2008) studied the ability of some solid catalysts to convert the cellulose into glucose. The results showed sulfonated activated carbon has the best performance that the highest conversions are obtained. The sulfonated activated carbon catalyst used from sugar-based materials, which converted into carbon by pyrolysis process (T > 300°C). The sulfonation of carbon use sulfuric acid solution at a temperature of 150°C (Onda et al, 2008).

The carbon which used as catalyst support should be in the hydrocarbons polyaromatik structure form. In this structure, the catalyst will be allow for attached of

sulfonate groups. Hence, the carbonization process carried at 400°C to form a structure poliaromatik (Mochida et al, 2006). The advantages of activated carbon as catalyst support are acid and alkaline resistant, high temperature and high pressure resistant, inert, easy recycling (recovery), and has a large surface area (Mochida et al, 2006).

The process which use of sulfonated activated carbon catalysts has no corrosive process. The spent this catalyst are complete separation easily by filtration, and the separated this catalyst can be reactivated. So the product does not produce waste catalyst. The purpose of this research is to study the potential of Eichhornia Crassipes (Eceng Gondok) as cellulose source for produce glucose.

2. METHODOLOGY

The activated carbon was made from coconut shells, which was carbonized at temperature of 350°C. The activated carbon was reduced in size to 60 mesh and was soaked in 96% sulfuric acid solution for 15 hours at a temperature of 150°C. The catalyst was washed and dried. The characterization of catalyst was carried by H⁺ capacity, BET, FTIR, and SEM.

The hydrolysis process was carried by autoclave. The dimension of autoclave equipment has volume of 250 ml, height of 11 cm, made of SS 304 (Figure 1). Before hydrolysis, the cellulose material was heated at a temperature of 105°C and was reduced in size to 60 mesh.



Figure .1 Autoclave equipment

Testing of catalyst was done with variable catalyst weight (i.e. 0-4 gram) and reaction temperature (i.e. 130-170°C). The sample weight of 2 gram and aquadest volume of 200 cm every run of experiment. Analysis of glucose products use the method Antrone (9,10-Dihydro-9-0xanthracene / C₁₄H₁₀O) 0.1% in sulfuric acid. Preparation of standard curve of total sugar made by dissolving 0.1 g of glucose in 100 mL of distilled water standards to obtain a concentration of 1000 ppm. The solution was then diluted with distilled water to obtain concentrations of 0 (control), 40, 80; 120; 160; 200 ppm. Each solution is then added to 5 mL of reagent Anthrone,

then closed and mixed thoroughly. Once placed in a water bath at 100°C for 12 minutes, and cooled with water, and then carried out on the spectrometer readings absorbance maximum of 630 nm wavelength is the relationship between the absorbance to mg glucose. Determination of total sugar concentration contained in the sample was performed at 1 mL sample was diluted in a test tube in the same way as in the manufacture of a standard curve.

3. RESULTS AND DISCUSSION

The result of catalyst characterization indicated that the number of H⁺ in the catalyst is 2.95 mmol / g. This is mean that every 1 g of the catalyst contained 2.95 mmol of H⁺ ions. The H⁺ ion is bounded to the sulfonate group which is expected to act as a catalyst in the process of hydrolysis to break down cellulose into glucose, as shown Figure 2 (Onda, at.al., 2008). From BET analyze, the surface area of the catalyst is 29 m²/g. The surface area of the catalyst is expected to play a role in the interaction of active centers on the catalyst surface with cellulose (Mochida et al 2006).

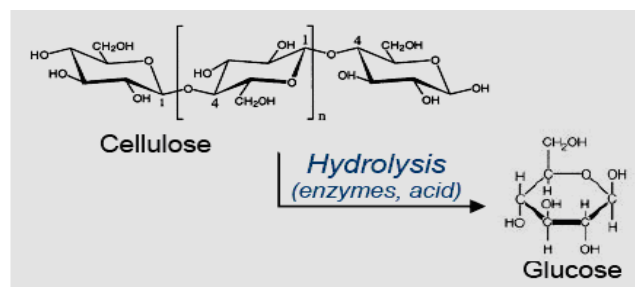
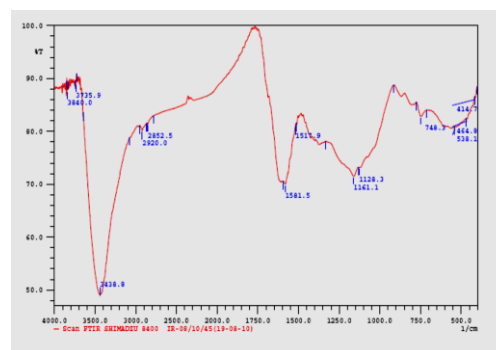
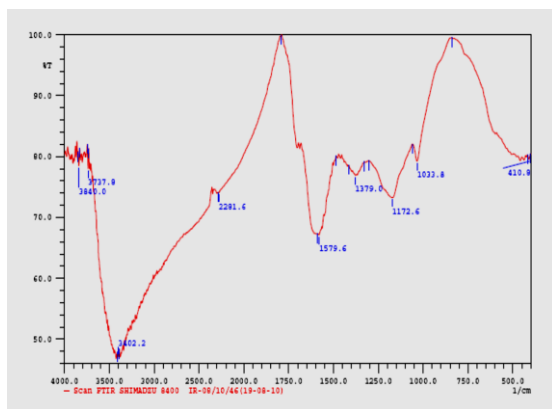


Figure 2. Reaction of break down cellulose into glucose (Onda, at.al., 2008)

The FTIR analyzed results was showed on Figure 3. The bands peak at a wavelength of $\lambda = 1750 \text{ cm}^{-1}$ and $\lambda = 1379 \text{ cm}^{-1}$ of sulfonated activated carbon is difference with activated catalyst. At those wavelengths detected the presence of sulfonate groups SO₃H. This result indicated that activated carbon after sulfonation containing sulfonate groups as part of an active site of the activated carbon catalyst.



(a)



(b)

4. CONCLUSION

Silicon Nitride thin films were deposited using a commercial RF magnetron sputtering system following the non reactive sputtering routine. The influence of sputtering pressure was investigated on films` properties like growth rate, refractive index, surface roughness, surface morphology, chemical bonding and microstructure.

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The result showed that the sputtering pressure has an imperative effect on the properties of magnetron sputtered thin films. Good quality and near stoichiometric silicon nitride thin films with minimum surface value of 1.2 nm, at growth rate 7.03 nm/min were obtained under 3 mTorr sputtering pressure.

The refractive index, surface roughness, excess silicon content and stress of deposited thin films increased linearly with the sputtering pressure. However, an optimum sputtering pressure value was observed during the characterization for growth rate. The growth rate was increased until that optimum value and then started decreasing due to back diffusion and scattering effect.

As a future work, the stoichiometry of silicon nitride thin films produced by RF magnetron sputtering can be adjusted by bleeding nitrogen gas during the deposition but this could affect the growth rate of thin films.

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