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Study of Provision of Cellulose Nanocrystal Derived From Palm Oil Empty Fruits Bunches (EFB)

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Abstract. Isolation and characterization of cellulose nanocrystal of alpha-cellulose from Empty Fruit Bunch (EFB) have been performed. Empty Fruit Bunch (EFB) was delignificated with 3.5% HNO_3 and $NaNO_2$, precipitated with17.5% NaOH, bleaching process with 10% H_2O_2. Nanocrystal was obtained through the hydrolysis of alpha-cellulose using 45% H_2SO_4. Nanocrystal surface structures cellulose was analyzed by Scanning Electron Microscopy (SEM) and the results show that the resulting cellulose Nanocrystal size was 79 nm. Analysis of thermal degradation using Thermal Gravimetric Analysis (TGA) shows that the cellulose nanocrystal was started at 160ºC decomposes. It shows the process of cellulose nanocrystal that isolated from alpha-cellulose has occurred.

Keywords: Empty Fruit Bunches Palm, Hydrolysis, Cellulose Nanocrystal

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1 Introduction

Palm oil plants (*Elaeis guinensis Jack*) come from Nigeria, West Africa. In fact, palm oil plants thrive outside their native areas, such as Malaysia, Indonesia, Thailand, and Papua New Guinea. It is even able to provide higher yields per hectare. Indonesia is a major producer of palm oil (Fauzi, 2003).

Palm oils produce waste that can provide great benefits for life, including organic fertilizer and as activated charcoal. One of the solid wastes of the palm oil industry is palm oil empty fruit bunches (EFB) (Steven, 2000). EFB is wrong one waste congested palm oil industry. EFB also produces fiber strong as ingredient filler in product fiber rubberized, including seat car, mattress, and board composite. Solid waste has a characteristic in its composition. The largest component in solid waste is cellulose. In addition to other components, although smaller, such as ash, hemicellulose, and lignin.

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Cellulose is a carbohydrate polymer composed of -D glucopyranose and consists of three hydroxy groups per anhydrous glucose making cellulose have a high degree of functionality (Siminar, 1990). Cellulose is a abundant biopolymer in nature that can be renewable, easily biodegradable, and also non-toxic. As a renewable material, cellulose and its derivatives can be studied well. The synthetic fibers are classified as cellulose (Bilmeyer, 1984). The isolated cellulose is not pure cellulose but for analytical purposes is sufficient to determine cellulose (Fengel, 1995). Cellulose I is the original form cellulose which is composed of α-cellulose and β-cellulose (Vander, 1994). Cellulose is less soluble in dilute acid and at high concentrations will be hydrolyzed to cellulose Cowd. 1991). Cellulose basic material has been used for more than 150 years in various applications, such as food, paper production, biomaterials, and in the health sector (Coffey et al., 1995).

Nanocrystals as materials that role in the manufacture of drugs that have been imported to Indonesia so the drug prices are relatively increasing. With this research on the isolation of nanocellulose, it can be seen the yield of cellulose obtained from palm oil empty friut bunches (EFB) by lignification, purification of alpha-cellulose, and hydrolysis with acid to precipitate nanocellulose. After that, it was characterized using scanning electronic microscopy (SEM). Isolation of nanocellulose from EFB was carried out by precipitation through acid hydrolysis using sulfuric acid at 45ºC for 30 minutes (Bledzki and Gassan, 1990).

In previous nanocrystal studies, hydrolysis with H_2SO_4 will experience a change in physical structure (fiber shape) at around 65% of H_2SO_4. Changes in structure and reaction patterns due to the influence of acid and temperature indicate that the main factors causing these changes. The structure of cellulose is the breakdown of hydrogen bonds. In hydrolysis with dilute acid, the hydrolysis reaction takes place to produce hydro cellulose with a lower degree of polymerization, but higher crystallinity. The hydrolysis of cellulose is strongly influenced by the degree of crystallinity and swelling of the cellulose. Its reactivity is also affected by its disintegration or decrystallization procedure. When the pre-treatment was carried out using 60% of H_2SO_4, the hydrolysis process was very slow. But the addition is above 65% of H_2SO_4, the most of the cellulose is dissolved. When this happens, it is diluted with water, so that part of the dissolved cellulose can be precipitated. From the results of this study, it can be concluded that circumstances of cellulose influence hydrolysis (Qianxiang et al., 2003).

Based on description above, author would like to conduct research about utililization of the palm oil waste as empty fruit bunch (EFB) to produce the cellulose nanocrystal (CNC).

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2 Materials and Methods

2.1 Equipments

In this study, the equipment used were glassware, analytical balance, oven, universal pH indicator, thermometer, hot plate, magnetic stirrer, Whatman no. 1 filter paper, desiccator, centrifugator, a set of TGA, and SEM spectroscopy.

2.2 Materials

The materials used were palmoil emtry frut bunch (EFB), NaOH, H_2O_2, aquadest, HNO_3, H_2SO_4, $NaNO_2$, $NaSO_3$, NaOCl aquabidest, and dialysis membrane of 0.22 µm.

2.3 Procedure

2.3.1 Preparation of Empty Palm Oil Bunch (TKS) Powder

EFB was washed and soaked in water for 2 hours. Then dried under sun for 2 days. Next, the fibers were cut to obtain fine fibers.

2.3.2 Isolation of α-Cellulose from Palm Oil Empty Fruit Bunches

A total of 75 g of EFB fiber was put into a glass beaker, then 1 L of 3.5% HNO_3 and 10 mg $NaNO_2$ were added, and then heated on a hot plate at 90ºC for 2 hours. After that, it was filtered and washed until the filtrate was neutral. Furthermore, it was digested with 750 mL of a solution containing 2% NaOH and 2% Na_2SO_3 at 50ºC for 1 hour. Then dregs was filtered and the were washed until pH was neutral. Furthermore, the bleaching process was carried out with 250 mL of 1.75% NaOCl solution at a boiling temperature for 30 minutes. Then dregs were filtered and washed until the pH of the filtrate was neutral. After that, α-cellulose was purified from the sample with 500 mL of 17.5% NaOH solution at 80ºC for 30 minutes. Then filtered, and washed until the filtrate is neutral. Next, bleaching was followed with 10% H_2O_2 at 60ºC in the oven for 1 hour. Then kept in a desiccator.

2.3.3 Isolation of Cellulose Nanocrystals from α-Cellulose

A total of 1 g of α-cellulose was dissolved in 25 mL of 45% H_2SO_4 at 45ºC for 45 minutes. Then cooled and added with 25 mL of aquabidest, then left for one night to form a suspension. The suspension formed was centrifuged at 10000 rpm for 20 minutes until the pH was neutral. Then, it was ultrasonicated for 10 minutes, after that it was put into a dialysis membrane and soaked in 100 mL of aquabidest, and the left for 4 days while stirring. Then the aquabidest was evaporated at 70ºC to obtain cellulose nanocrystals (CNC).

2.3.4. Morphological Test using Scanning Electron Microscopy (SEM)

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The use of SEM was begun by gluing the sample with a stab made of palladium specimen metal. Then, the sample was cleaned with a blower and coated with gold and palladium in a dispatcher machine with a pressure of 1492 x 10^{-2} atm. The sample is then put into a special room and then irradiated with a 10 kV until release the electron beam so that the sample emits secondary electrons and bounced electrons which can be detected and the detector scienter is then amplified with an electrical circuit that causes a CRT (Chatode Ray Tube) image to appear. Shooting is done after selecting a certain part of the object (sample) and the desired magnification so that a good and clear photo is obtained.

2.3.5 Degradation Thermal Test Using Thermogravimetry Analysis (TGA)

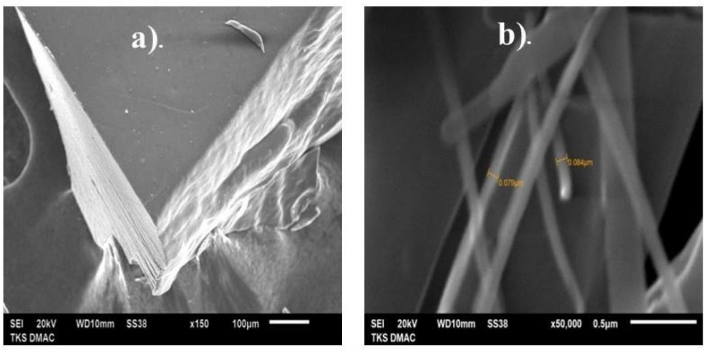
The sample was weighed as much as 12.272 mg and heated from room temperature to 612ºC with heating rate up 10ºC/min. The analysis was conducted with raised temperature sample by gradually and determine heavy to temperature change. The temperature in method testing reaches 650ºC or more. The change of mass of sample is consequence of heating process and can be determined directly from a successful thermogram. After the data was obtained, it can be determined its decomposition.

3 RESULT AND DISCUSSION

The results of this study yellow-brownish pulp. The final process was conducted bleaching using 10% of H_2O_2. The cellulose produced from the process has white pulp and the dried in the oven at 60ºC.

Cellulose nanocrystals isolated from α-cellulose were clear needle-shaped crystals. The manufacture of cellulose nanocrystals consists of several stages, namely hydrolysis, neutralization by centrifugation process, and isolation of cellulose nanocrystals using membrane dialysis. The hydrolysis process was carried out using 45% of H_2SO_4. A concentration of 45% was required to obtain good cellulose nanocrystals, if using a concentrated one, it will be hydrolyzed to glucose. Centrifugation in this process was required so that the resulting nanocrystals become neutral after being hydrolyzed with acid. Neutralization process using aquabidest was to produce pure nanocrystals.

The next process was the isolation of cellulose nanocrystals using a dialysis membrane,



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accompanied by immersion in aquabidest and using a stirrer which aims to get the nanocrystals out of the membrane faster. This process was performed 4 to 8 days. After that, evaporation was carried out to obtain the desired nanocrystal. Then an analysis was carried out using Scanning Electron Microscopy (SEM) to determine the surface size of the nanocrystals and thermal analysis using TGA to determine the decomposition temperature of cellulose nanocrystals obtained from the research results.

Figure 1. The SEM morphology of CNC with magnification a). 150x and b). 50000x

Figure 1 shows that the surface structure of cellulose nanocrystals. The SEM analysis results obtained, in figure (1.a) at 150x magnification, which indicated there was a group of agglomerated crystals. While in figure (1.b) at magnification of 50000x, which shown the single crystals are in nano-size, which was between 79 to 84 nm.

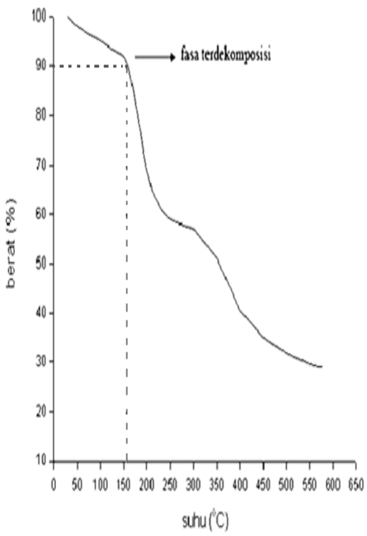

Figure 2. TGA Graph

In addition, figure 2 shows the change in weight indicating that the cellulose nanocrystals began to decompose at 160ºC. The weight loss was a function of temperature. There were two peaks obtained from the thermogravimetric curve generated from the study. The first peak ranges from 150ºC to 200ºC and the second peaks between 300ºC to 350ºC. Cellulose degraded at a temperature of 160ºC and have 28% of residue.

4 Conclusion

Based on the data obtained in this study, it can be concluded that:

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1. The α-cellulose obtained was white pulp which was done through delignification, precipitation, and bleaching process. The cellulose nanocrystal was isolated by hydrolysis process using 45% of H_2SO_4 produced clear crystal needle.

2. Analysis of surface structure of cellulose nanocrystal was performed using scanning electron microscopy shows that the CNC obtained was 79 nm and the degradation thermal analysis was conduted by thermogravimetry analysis (TGA).

References

Bilmeyer, F. W., 1984. Textbook of Polymer Science. Third Edition. New York: John Willey & Sons, Inc.

Bledzki, A-K, Gassan J., 1990. Composites Reinforced with Cellulose Based Fibres. Prog Polym Sci.

Coffey, D.G., D. A. Bell and A. Handerson. 1995. Cellulose and Cellulose Derivates. New York.

Cowd, M. A., 1991. Kimia Polimer. Bandung: ITB.

Fauzi, I. Y., 2003. Kelapa Sawit. Jakarta: Penebar Swadaya.

Fengel, D., 1995. Kimia Ultra Struktur Reaksi – Reaksi. Cetakan Pertama. Yogyakarta: UGM Press.

Steven, M. 2000. Kimia Polimer. Cetakan Pertama. Jakarta: Pradya Paramitha.

Suminar, S. 1990. Kimia Kayu, Dirjen Pendidikan Tinggi Pusat Antar Universitas Ilmu Hayat. Bogor: IPB.

Vander Hart, D. C., 1984. Study of Macromolecules in Native Cellulose Using Solid-State NMR. Macromolecules

Qianxiang, Y. Y. Lee, Par O. Petterson and Robbert W., 2003. Heterogeneous Aspect of Acid Hydrolysis of α-Cellulose. Auburn: University Auburn.