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Production and characterization of nanocrystalline cellulose from palm empty fruit bunch fiber

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Abstract. The main objective of this research is to utilize fiber waste from palm empty fruit bunch provided very abundant in Aceh. For that needs to be studied and researched other benefits are more valuable. Among them is a source of raw material to produce nanocrystalline cellulose (NCC), a material in nanometer dimensions which can be used as fillers or reinforcing a matrix in order to provide bio nanocomposites. NCC isolated from palm empty fruit bunch through the hydrolysis of sulfuric acid, using a solvent mixture of DMAC/LiCl, were passed on activated dialysis membrane after centrifuged to release it from solvent. NCC will be characterized include aspect ratio, morphology, thermal, structural test, and several other physical parameters. The end product is characterized by means of thermogravimetric analysis (TGA), Infrared Spectroscopy (FTIR), X-Ray Diffraction (XRD), Differential Scanning Calorimetry (DSC) and Scanning Electron Microscopy (SEM). From XRD and FTIR analysis showed that the nanocrystalline cellulose formed was Nano cellulose I with an average size of 72 nm. DSC and TGA thermal test showed that the glass transition, melting point, and decomposition temperature of NCC were 109.59 °C, 191.28 ° C, 210 ºC, respectively.

1. Introduction
Cellulose is a sustainable and renewable natural material. Nanocomposite fibers and materials nanoselulosa attracted much interest due to extraordinary changes in the nature [1]. As reinforcement in composite material components, this natural fiber has its advantages such as a renewable, recyclable and can be biodegradable in the environment. In addition, natural fibers have good mechanical properties and less expensive than synthetic fibers. Simultaneously, production of crude palm oil (CPO) in the ground water is estimated to reach 13.6 million tons per year and as a result of the processing side of fiber to be obtained from the solid waste munitions and fiber seed, reached 20 million tons per year [2]. These properties make nano fibers are very promising materials for the composites industry, automotive materials, pulp and paper, electronics and other industries.

2. Methods

2.1. Material
Palm empty fruit bunches (EFB) fibers from Palm Oil Research Center, Medan (Indonesia). Reagents used are: nitric acid, sodium nitrite, sodium sulphite, sodium hydroxide, sodium hypochlorite, hydrochloride acid, sulphuric asid, dimethyl acetamide, litium chloride (analytical grade).title.

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2.2. Methods

- Provision of Oil Palm Fiber Empty Bunches (EFB)
- Delignification process-Bleaching [3]
- Hydrolysis processes (manufacture of microcrystalline cellulose) [4]
- Dispersion process (Making Nanocellulose) surname [5]

3. Results and discussions

Figure 1 shows the physical aspects of the original EFB fiber and figure 2 after bleaching with peroxide solution and alkaline solution.

Figure 1. Original empty fruit bunch fiber.

Figure 2. Empty fruit bunch fiber after bleaching.

XRD analysis performed to determine the crystallinity, also called the degree of order. X-ray diffraction pattern of fibers and bleached EFB after a microcrystalline fiber is shown in figure 3 and 4.

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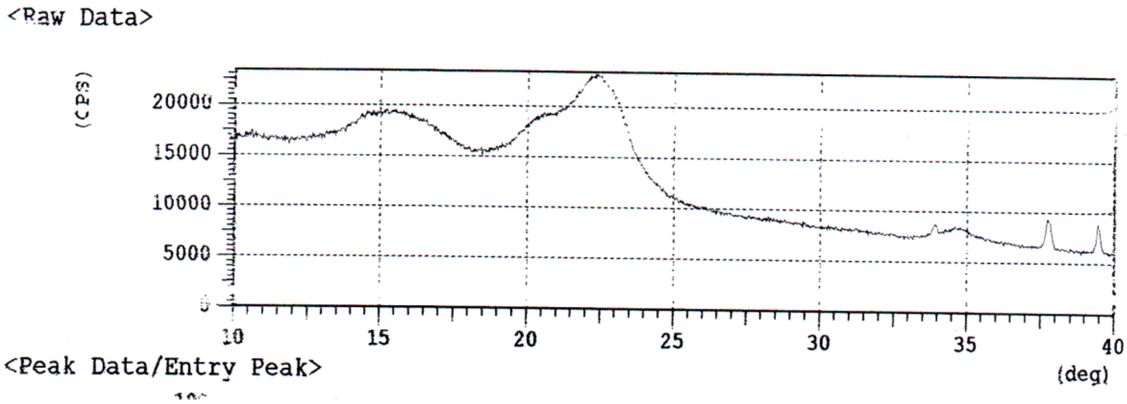

Figure 3. X-ray diffraction pattern of fibers and bleached EFB.

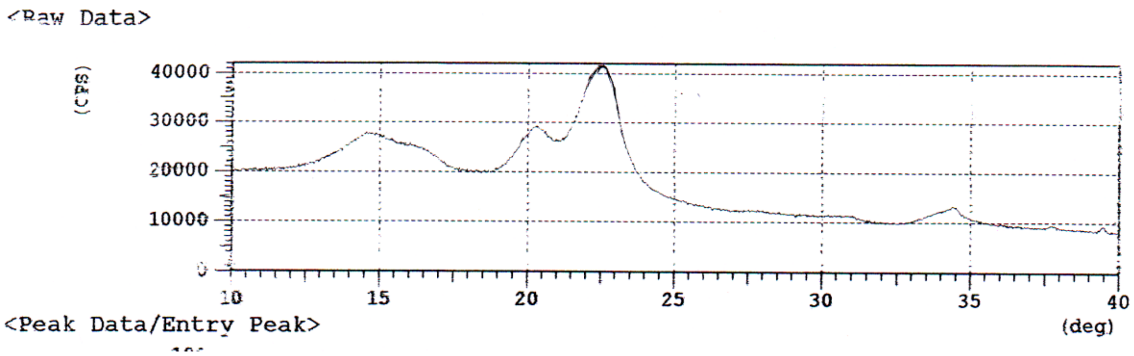

Figure 4. X-ray diffraction pattern of microcrystalline fiber.

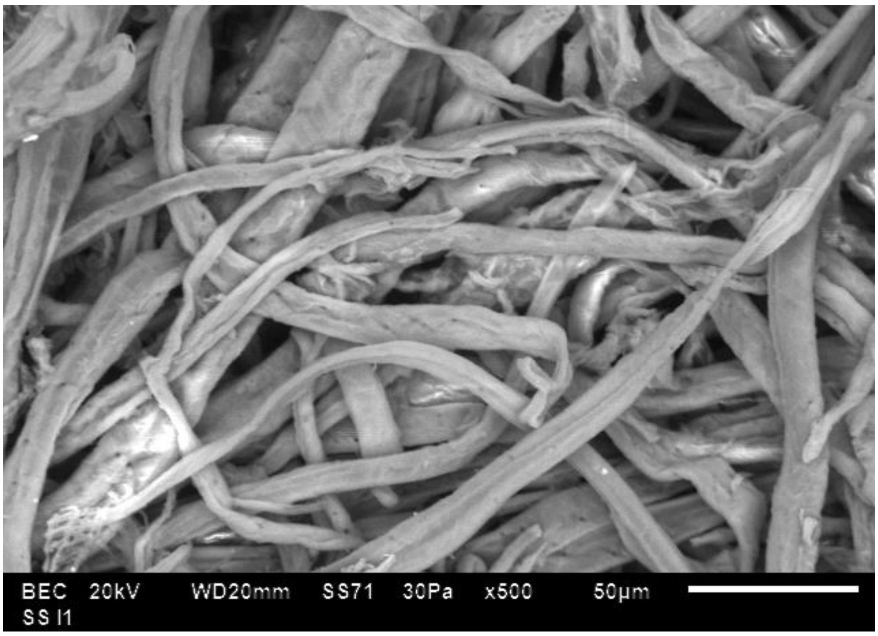

Figure 5. Morphology crystalline EFB.

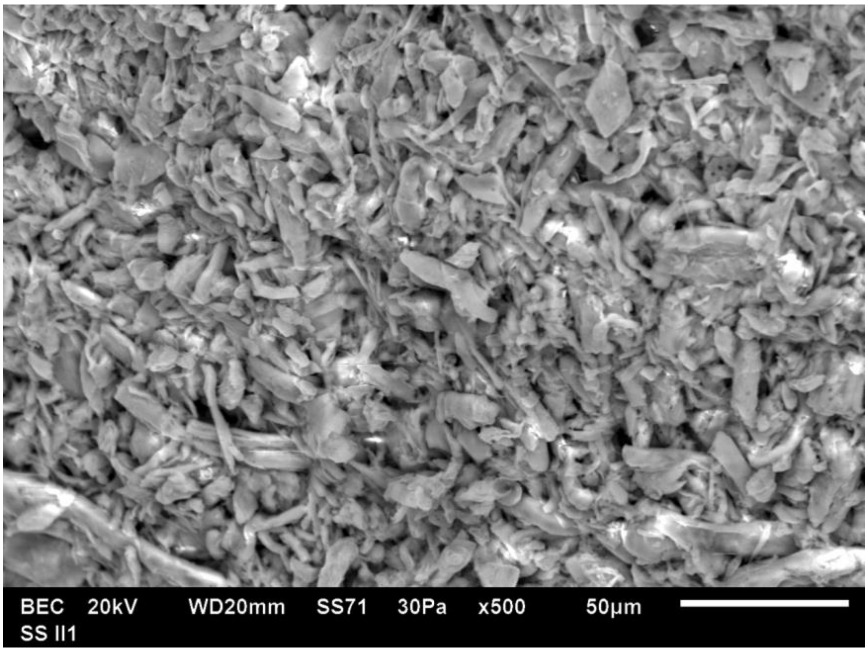

Figure 6. Morphology microcrystalline EFB.

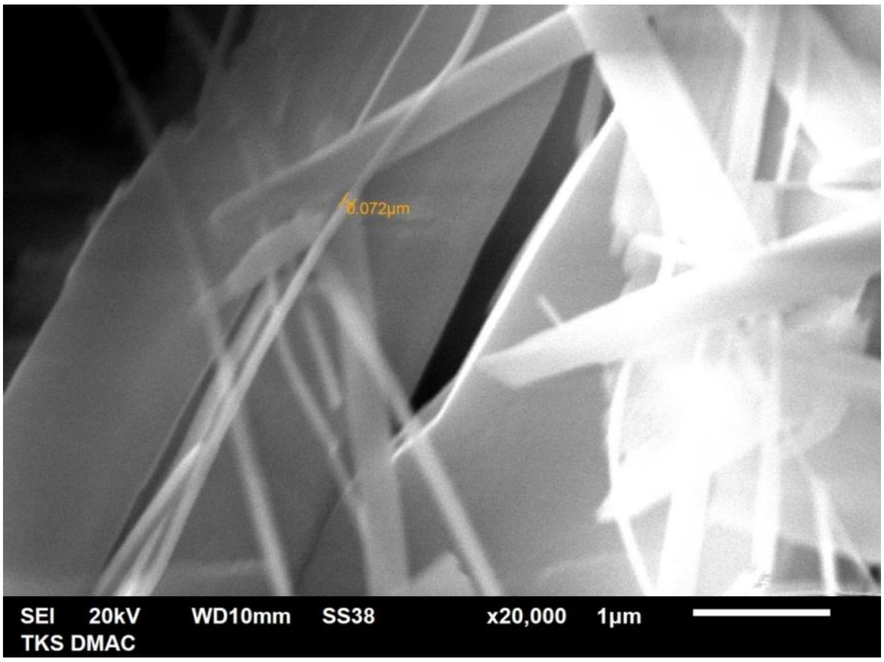

Figure 7. Morfologi nanocrystalline EFB.

Both difraktogram featuring a mixture of cellulose polymorphs (typical peaks at 15^O and 22.6^O) [6]. The percentage of crystallinity for bleached fibers after TKS was 75%, whereas for microcrystalline was 86%. Increase in crystallinity of cellulose fibers with microcrystalline cellulose bleaching results showed that the hydrolysis process in the formation of microcrystalline cellulose is complete [7,8].

Figure 5 shows the morphology of cellulose fibers with SEM EFB. 5 is an image of microcrystalline cellulose, microcrystalline image 6 hydrolysis results with sulfuric acid and microcrystalline image 7 is treated with lithium chloride-dimethyl asetamida.

Results after acid hydrolysis of microcrystalline cellulose is 30% of initial weight. This is because the remaining amorphous microcrystalline separated during the hydrolysis. Also seen that the microcrystalline released during the process of washing / neutralization.

Meanwhile, the handling of microcrystalline cellulose in lithium chloride-dimethyl asetamida deliver nearly three times the initial weight of microcrystalline, the possibility of compound formation between microcrystalline cellulose with dimethyl asetamida / lithium chloride.

4. Conclusion

EFB fiber can be a source of cellulose for the manufacture of microcrystalline cellulose. EFB fibers can be bleached with solvents that are less aggressive to the environment (and alkaline peroxide solution). From the results of the XRD analysis, it appears that the resulting fibers are pure mikroselulosa (there is only one peak), while from the SEM analysis, the resulting fibers measuring 72 nanometers, but has not been uniform.

Need for additional research to optimal operating conditions, in order to obtain the cellulose fibers from EFB fibers in a uniform nano size.

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References

[1] Ibrahim M M, El-Zawawy W K and Nassar M A 2010 *Synthesis and characterization of PVA / nanospherical cellulose particle films*, Carbohydrate Polymers 79*,* 694-699s

[2] Darnoko A S and Sutarta I A S 2006 Pabrik Kompos di Pabrik Sawit *Tabloid Sinar Tani* **9**

[3] Klemm D, Heublein B, Fink H P and Bohn A 2005, Cellulose: fascinating biopolymer and sustainable raw material *Angewandte chemie international edition* **44**(22) 3358-3393

[4] Ohwoavworhua F O and Adelakun T A 2005 Phosphoric acid-mediated depolymerization and decrystallization of α-Cellulose obtained from corn cob: preparation of low crystallinity cellulose and some physicochemical properties *Tropical Journal of Pharmaceutical Research* **4**(2) 509-516

[5] Bondeson D, Kvien I and Oksman K 2006 Strategies for preparation of cellulose whiskers from microcrystalline cellulose as reinforcement in nanocomposites *American Chemical Society*

[6] Klemm D, Kramer F, Moritz S, Linstrom T, Ankerfors M, Gray D, Dorris A 2011 Nanocelluloses: A new family of nature-based materials *Angew. Chem. Int. Ed.* **50** 5438-5466

[7] Ioelovich M 2012 Optomal condition for isolationof nanocrystaline cellulose particles, Nanoscience and Nanotechnology **2**(2) 9-13

[8] Adriana 2018 Proses pembuatan nanokristal selulosa dari tandan kosong kelapa sawit, Paten IDP000050091