Facile preparation of cellulose nanocrystals of banana trunk fiber via acid hydrolysis method

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ABSTRACT – Lignocellulosic fibers have been received an intense attention to many researchers due to their tremendous advantages. The aim of this study is to prepare cellulose nanocrystals (CNC) that derived from banana trunk fibre (BTF) via acid hydrolysis method. The effect of hydrolysis time and temperature correlation is studied in order to find the optimum conditions of CNC of BTF which are possessed good crystallinity in their crystal structure. Crystallinity index and crystallite size is determined and evaluated by using x-ray diffraction spectrometer (XRD). Thus, the CNC has been successfully produced with the optimum condition at 90 ˚C for 30 minutes by the 31.73 % crystallinity index and 5.47 nm of the crystallite size which act as a sustainable source due to the cheap, eco-friendly and its availability while preventing pollution to the environment that caused by BTF wastes.

1. INTRODUCTION

Banana plant (Musaceae) become the second largest crop in the world which contribute about 16 % of the world's fruit production and being cultivated over 130 tropical countries. Banana fibre is mainly extracted from its trunk, leaves, and rachis. As others lignocellulosic, banana fibre has attracted the attention due to its useful contribution in many applications such as in paper making [1-2], textile, biodegradable ropes, bags and natural sorbent [3]. Moreover, there is study that considered making the banana fibre as the reinforcing material in polymer matrix due to its good mechanical properties [4].

In recent years, lignocellulosic fibre has gained the attention and become the main source in producing the cellulose nanocrystals (CNC) due to their tremendous advantages properties such as renewable and low cost [5]. Banana trunk fibre (BTF) is one of the lignocellulosic fibre that have been used for many applications. However, the utilization of BTF as the starting material for CNC has not been much exploited. Therefore, this opens the opportunity to maximize the renewable sources of CNC instead by using other common lignocellulosic fibres such as sisal, kenaf, hemp, flax, grass and many more.

The production of CNC differs based on its cellulose sources, types and application. CNC can be obtained by using chemical treatment or by the combination, mechanical and chemical method. Acid hydrolysis is the famous used method that used in producing a stable colloidal suspension of CNC [6].

The objective of this current work is to prepare and characterize the cellulose nanocrystals (CNC) that derived from banana trunk fibre (BTF) using acid hydrolysis (H_2SO_4) method by different hydrolysis time and temperature. The CNC obtained will be further analysed and characterized by using x-ray diffraction microscopy (XRD).

2. METHODOLOGY

The powder fiber was obtained by crushing the dried banana trunk chips by using industrial blender. About 500 g of bleached banana trunk powder was treated with 1.5 L of 6 wt.% of NaOH, at room temperature for 24 hours. After that, the mixture was filtered by cloth sieve and washed with distilled water for several times until the pH is 7. The resultant mixture is then being put on the tray and dried in the drying oven at 50 ºC for 24 hours. As the result, hard, thick, pieces of banana trunk alkali treated formed. After that, the hard, pieces of the alkali treated banana trunk were crushed again by using industrial blender to obtain a powder of alkali treated banana trunk fiber.

The acid hydrolysis was carried out by using 40 wt.% of H_2SO_4 at different hydrolysis time and temperature. The ratio of pulp-to-acid was fixed at 1:15. About 3 g of alkali treated banana trunk powder was mixed with 45 ml of H_2SO_4. The experiments were conducted in different hydrolysis temperature and time conditions. The mixture was steadily stirred by the magnetic stirrer until the hydrolysis time was finished. The hydrolyzed cellulose was dialyzed a few times by using distilled water to a constant pH 7. Finally, the crystal structure of cellulose nanocrystals (CNC) of fibre (BTF) obtained were analyzed by using PAN analytical X'PERT PRO MPD x-ray diffraction analysis, performed at room temperature with a monochromatic CuKα radiation source of λ=0.154060 at the scanning rate of 0.5 /min.

3. RESULTS AND DISCUSSION

As the result, a corresponding crystallinity index and crystallite size values are listed in the Table 1 and

Table 2 that were calculated by using Segel's [8] and Scherer [9] equation, respectively. From the XRD data, it was found that the optimum condition of the hydrolyzed banana trunk fiber is at 90 ˚C for 30 minutes (CNC4). For this condition, the crystallinity index is much higher than the other conditions of the hydrolyzed banana trunk fiber. George and Sabapathi [6] mentioned that, the longer the hydrolysis time, the less amorphous region was remained which in turns, increases the crystallinity of the cellulose nanocrystal. Conversely, if the hydrolysis time is insufficient, thus more amorphous region left and the crystallinity index become lower. However, according to sample CNC6, the intensity peak was not detected. If both time and temperature of hydrolysis is high, there is no crystallinity peak observed due to the degradation of cellulose crystalline region by acid [7].

Table 1 The crystallinity index of treated acid hydrolyzed banana trunk fiber (BTF)

Sample	Cellulose Intensity Peak (2ϴ)	Crystallinity Index (%)
CNC1 (30°C 30min)	-	-
CNC2 (30°C 60min)	22.74	30.17
CNC3 (30°C 90min)	-	-
CNC4 (90°C 30min)	22.63	31.73
CNC5 (90°C 60min)	22.68	28.04
CNC6 (90°C 90min)	-	-

For the sample that were fixed at 30 ˚C conditions, only one sample that shows the crystalline peak at XRD result. The crystallinity index of CNC2 is 30.17 % lowered that the CNC4. This may due to the inadequate temperature for the acid to hydrolyze the banana fiber. This resulted to the low crystallinity index. As for the CNC3, the hydrolysis time is much longer than the CNC2. Thus, the acid not only attacked the amorphous region but also the crystalline region as well. This resulted to non-exist crystalline peak. For the CNC1, the crystalline peak is not existed due to the low of hydrolysis temperature and time, thus no possible hydrolysis reaction occurred at this condition.

The crystallite size of the cellulose nanocrystals (CNC) from banana trunk fibre (BTF) were determined by using Scherer equation. From the calculation, it can be concluded that, the higher the temperature, the smaller the crystallite size of the cellulose nanocrystals. This is due to the higher reaction temperature, will leads to the effectiveness in obtaining shorten CNC [6].

Table 2 The crystallite size of treated acid hydrolyzed banana trunk fiber (BTF)

Sample	Cellulose Intensity Peak (2ϴ)	Crystallite Size (nm)
CNC1 (30°C 30min)	-	-
CNC2 (30°C 60min)	22.74	13.47
CNC3 (30°C 90min)	-	-
CNC4 (90°C 30min)	22.63	11.99
CNC5 (90°C 60min)	22.68	5.47
CNC6 (90°C 90min)	-	-

4. CONCLUSIONS

As the conclusion, the cellulose nanocrystals (CNC) derived from banana trunk fiber (BTF) has successfully prepared by using 40 wt % sulphuric acid (H_2SO_4) acid concentration with 90 ^oC and 60 min as the optimum condition for the hydrolysis process. Based on XRD analysis, the CNC of BTF give result with crystallinity index ranging 28.04 to 31.73 % and crystallite size ranging 5.47 to 13.47 nm.

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