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# INVESTIGATION ON PRE-TREATMENT PROCESS IN MICROCRYSTALLINE CELLULOSE (MCC) FROM OIL PALM EMPTY FRUIT BUNCH (EFB)

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## ABSTRACT

In this study, microcrystalline cellulose (MCC) was extracted from empty fruit bunch (EFB) with alkaline treatment, bleaching and acid hydrolysis treatment and its properties were compared with commercial MCC. Two conditions were optimized in this study which are fiber consistency and sodium hydroxide (NaOH) concentration in alkaline pre-treatment. The obtained optimum consistency and alkaline concentration are 5% and 20% respectively. The physicochemical and morphological characteristics, elemental composition and size distribution of the obtained alpha cellulose and MCC were analyzed in this work. Fourier transform infrared spectroscopy (FTIR) analysis provided clear evidence that the characteristic peak of lignin and hemicellulose were absent in the spectrum of the alpha cellulose and MCC. The difference in surface morphology and aggregation between alpha cellulose, MCC and commercialized MCC were observed by scanning electron microscopy (SEM). The mean length of approximately 251.3 µm, 41.4 µm and 138.6 µm were measured for alpha cellulose, MCC and commercialized MCC respectively. Thus, the isolated MCC from EFB has a good potential to be utilized as reinforcing agent in green composites and may be a precursor for future roselle derived nanocellulose, and thus a promising subject in nanocomposite research.

*Keywords*: Empty fruit bunch; microcrystalline cellulose; alpha cellulose; alkaline treatment; .

## 1.0 INTRODUCTION

Malaysia is the world's second-largest palm oil producer and the largest palm oil exporter which consisting of 88% of the market's palm oil in 2011 (Aghamohammadi et al., 2016). Currently, the largest solid biomass generated in Malaysia originated from oil palm plantations (Farhana Harun et al., 2013). Annually, a minimum of 168 million tonnes of biomass waste is generated in Malaysia. In general, palm oil waste accounts for 94% of biomass feedstock while the remaining contributors are agricultural and forestry by-products, such as wood residues (4%), rice (1%), and sugarcane industry wastes (1%) ("Malaysia's biomass potential," 2012). This biomass will assist to solve the waste management but also can create value-added products. Lignocellulosic biomass produced from the oil palm industries including oil palm trunks, oil palm fronds, empty fruit bunch,

palm pressed fibers, palm shells and palm oil mill effluent (Abdullah & Sulaiman, 2013).

Most of the agricultural lignocellulosic biomass is comprised of lignin, hemicellulose, and cellulose. Cellulose is a highly stable polymer consisting of glucose and it is majorly composed of (1,4)-D-glucopyranose units (Anwar, Gulfraz, & Irshad, 2014). Cellulose structure is aligned parallel to each other in fibrils, which are surrounded by a matrix of lignin and hemicellulose. Microcrystalline cellulose (MCC) consist of crystalline structure extract from cellulose. MCC has generated much attention and interest during these few last decades in both academic and industrial fields due to its good properties such as low density, high aspect ratio, stiffness and high strength (Jonoobi, Mathew, & Oksman, 2012) MCC gained major interest in various applications, such as stabilizer, fat replacer and texturing agent in food industry (Singh, Kanawjia, Giri, & Khetra, 2015), binder and water retainer in pharmaceutical industry (Johansson & Alderborn, 2001), and reinforcing agent in plastic industry (Wittaya, 2009).

Currently, the main raw material of cellulose derivative is from wood and cotton linter. However, deforestation and acceleration of greenhouse affect gradually grown interested in agriculture products and by-products as alternative for cellulose resources (Bono et al., 2009). EFB as an oil palm biomass product attracted interest among researcher due to high cellulose content (24-64%) which has been reported by Chang in 2014. A few years back, EFB was a valueless biomass produced from the oil palm mill and it will casually be thrown back to the oil palm plantation or burnt in the incinerators for it ash. Furthermore, under this route of disposal, no energy is recovered. According to Abdullah and Sulaim, EFB is a resource which has huge potential to be used for cellulose production which currently not being utilized. In Malaysia, there is no industry that produces MCC. Therefore, the consumer in Malaysia has to import the MCC from outside Malaysia such as China. The demand of MCC had increased from years to years ("Global Microcrystalline Cellulose (MCC) Market: Demand from Cosmetics and Pharmaceuticals Industries to Fuel Demand, says TMR," 2016) .

Cellulose can be extracted from biomass sources via a few pretreatment process which are chemical, biological fermentation, mechanical, physical or mixed processes. Alkali pre-treatment is the most common and important method for delignification of lignocellulosic materials. The main purpose of the pre-treatment is to break down the complex lignocellulosic structure of the biomass by solubilizing the lignin and hemicellulose; increase the porosity and surface area of cellulose and reduce the crystallinity of cellulose (Harmsen et al., 2010). But with the existing research, highly concentrated alkaline solution (40-50%) was being used. Thus, finding the optimum concentration will greatly save the treatment cost and eventually save the environment from great harm.

Other than that, fiber consistency also plays an important role in the extraction of cellulose. Consistency can be defined as the percentage of weight bone dry fibrous material in any combination of fiber and water. Fiber consistency will affect both the swelling time and swelling degree of the fiber in which both of them will greatly increase the fiber flexibility and strength (Joutsimo & Asikainen, 2013).

The previous work shows EFB is a potential raw material to produce MCC that have an almost similar characteristic with the commercial MCC which commonly produced from

wood and cotton (Nasution et al., 2017). Thus, the study on the operable condition in synthetizing the MCC take place by understanding the effect of consistency and NaOH concentration in the alkaline pre-treatment for the production of MCC from EFB. In this work, the empty fruit bunch are pre-treated using hot water that proceeded by with alkaline pre-treatment using NaOH and lastly, the MCC were produced by using hydrolysis process using hydrochloric acid (HCl). The alkaline pre-treatment was optimized by using different concentration of NaOH solution and the operable condition for the synthesis of MCC was determined by the consistency of the slurry.

## 2.0 METHODS AND MATERIALS

### 2.1 Materials and Chemical

EFB were obtained from LKPP Corporation Sdn. Bhd. palm oil mill, Lepar. Sodium hypochlorite (NaOCL) with purity 10%, sodium hydroxide (NaOH) pallet with purity 98%, hydrochloric acid (HCl) 37%, sulphuric acid ($H_2SO_4$) with purity 98% and commercial MCC were purchased from Sigma Aldrich. All the chemicals used were of analytical grade.

### 2.2 Sample preparation

EFB were dried at room temperature for 1 week. Then the EFB was ground using the SIMA grinder model FG 400×200 equipped with 200 mm mesh.

### 2.3 Synthesis of MCC

#### *2.3.1 Hot water treatment*

Ground EFB were weight accordingly (10g, 20g, 50g, 75g, and 100g) to obtain different consistency in 1000 ml water. Then the water was heated at 80˚C for 3 hours and stirred. Hot water treated fibers were washed and dried in oven at 80 ˚C overnight.

#### *2.3.2 Alkaline pre- treatment*

5g of treated EFB was treated with 10% concentration of sodium hydroxide at 80˚C for 60 minutes and stirred. The produced alpha cellulose was washed thoroughly with water until it neutralized and dried in oven at 80˚C overnight. The steps were repeated with 20% and 40% NaOH concentration.

#### *2.3.3 Bleaching*

Alpha cellulose was bleached at 80˚C using 100 ml of 2% sodium hypochlorite (NaOCl) solution for 1 hour. Bleached alpha cellulose was washed and dried in the oven at 80˚C overnight.

#### *2.3.4 Acid hydrolysis*

Alpha cellulose was hydrolyzed with 2.5N hydrochloric acid at 80˚C for 15 minutes. The MCC produced was washed thoroughly with water until neutral and dried in oven at 80˚C overnight.

## 2.4 Characterization

### *2.4.1 Determination of fiber consistency*

Fibers were dried in the oven at 105˚C for 2 hours before being weight using analytical balance. Formula was used to calculate the consistency of fiber for the synthesis using equation 1.

$$\frac{oven\ dry\ weight\ of\ pulp}{weigh\ \ of\ pulp+water} \times 100\% \quad (1)$$

### *2.4.2 Weight loss*

Formula was used to calculate the weight loss using equation 2.

$$\frac{W_1-W_2}{W_1} \times 100\% \quad (2)$$

Where $W_1$ is the dry weight before alkaline pre-treatment and $W_2$ is the dry weight after alkaline pre-treatment.

### *2.4.3 Fourier transform infrared (FTIR).*

The Infrared (IR) spectroscopy were performed on Perkin–Elmer equipment between band 500 to 4000 $cm^{-1}$ . The tests were carried out on the sample of alpha cellulose and MCC.

### *2.4.4 Lignin content*

Acid-insoluble lignin content was measured in accordance with TAPPI standard T 222 om-98 “Acid-insoluble Lignin in Wood and Pulp.” The moisture content of the sample is determined for air-dried wood/pulp and was used to weigh a known weight of wood/pulp. Concentrated sulphuric acid was utilized to hydrolyze and solubilize the carbohydrates in wood and pulp samples. The acid-insoluble lignin was filtered, dried, and weighed.

### *2.4.5 Surface morphology*

Scanning electronic microscopy (SEM) was carried on a Hitachi Tabletop TM3030 Plus under 1-15-kV acceleration voltage was employed to analyze the morphology of alpha cellulose and MCC. Samples were mounted on aluminium stubs with a carbon tape. The average length and diameter of fiber were calculated using Image J software.

# 3.0 RESULTS AND DISCUSSIONS

## 3.1 Effect of consistency in the pre-treatment process

The optimization of fiber consistency was done using the Daihan Scientific (MSH-20D) 600W hotplate stirrer. Figure 1 shows the graph between consistency and energy (rpm) needed to achieve a homogeneous slurry solution. The consistency has been varied at 1%, 2%, 5%, 7.5%, and 10%. From the figure, it can be seen that as the energy (rpm) needed to achieve homogeneous proportional to the consistency. But when the consistency of the fiber reaches 10%, the solution cannot achieve a homogeneous solution. It also can be concluded that when the rpm increases the amount of energy needed also increases. From

the result, that most feasible and applicable consistency is 5% which showed good agreement with the study reported by Lindsay et.al which mentioned in their study that the best consistency is below than 6%. Thus by using the optimum consistency, a great deal of energy can be saved and 5% consistency was opted to continue the study.

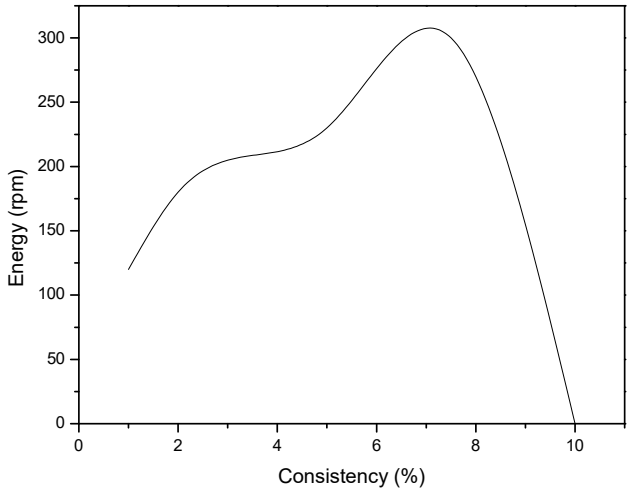


**Figure 1:** Effect of consistency (%) on the rpm needed to achieve a homogeneous slurry

### 3.2 Alkaline pre-treatment

#### *3.2.1 Effect of NaOH concentration*

Figure 2 shows the effect of NaOH concentration used in the alkaline pre-treatment expressed by percent of weight loss. It is shown that significant weight loss increases gradually from 10% until 20% of NaOH and then decrease until 40% of NaOH. This indicates that the amount of hemicellulose and lignin removal increases gradually with increasing of NaOH concentration. The extent of hemicellulose and lignin removal reaches the highest value at 20% NaOH concentration with 50% loss in weight and the weight loss percent began to decrease until 40% NaOH. It can be seen that when the NaOH concentration is 20% the weight loss is 50% and the typical empty fruit bunch hemicellulose and lignin are 35-65%. Thus, it can conclude that almost all of the hemicellulose and lignin were removed in that condition.

TAPPI standard T 222 om-98 “Acid-insoluble Lignin in Wood and Pulp” testing was conducted for the 20% NaOH fiber to measure the acid-insoluble lignin content. The result shows that the lignin content in the treated fiber using 20% NaOH solution is only 6.5%. The typical lignin content in an empty fruit bunch (EFB) is between 19%-20%. It can conclude that 13% of the lignin has been removed during the alkaline treatment using 20% NaOH solution. From both of the result it can be concluded 20% NaOH is the best condition of the alkaline pre-treatment with the maximum amount of hemicellulose and

lignin removal. At the same time to reduce the treatment cost and the impact of the treatment on the environment due to solvent usage.

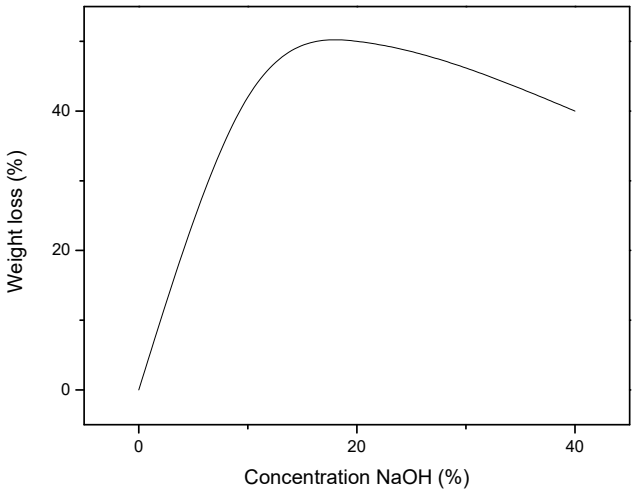


**Figure 2**: Effect of sodium hydroxide concentration on percent loss in weight

### 3.3 Characterization of MCC

#### *3.3.1 Surface morphology analysis*

The alpha cellulose and MCC were obtained after bleaching and acid hydrolysis process respectively. Figure 3 shows the morphology of alpha cellulose and MCC under 500X and 2kX magnification. The SEM micrographs for the alpha cellulose (Figure 3 (A and B), showed a long smooth fibril with the average length 251.3 µm and diameter 9.1 µm. On the other hand, the MCC image shows a crystal-like structure and individualized fiber (Figure 3, C and D) with the average length 41.4 µm and diameter 8.3 µm. Acid hydrolysis led to changes in morphological structure from the fibrillar structure of alpha cellulose to irregular fiber fragments. During the acid hydrolysis, the amorphous structure of the alpha cellulose will be dissolved in the acid leaving the crystalline structure of the alpha cellulose. According to Elanthikkal, Gopalakrishnapanicker, Varghese, & Guthrie (2010), hydrolysis process penetrates the amorphous regions of alpha cellulose, and cleave the β-1,4-linkage between the cellulose repeating units, where the alpha cellulose fibers were broken into shorter cellulose, MCC.

For comparison purpose, Figure 3 (E) shows the microstructure of commercial MCC obtained from Sigma Aldrich which extracted from wood. The Figure 3 shows the morphology of commercial MCC is long narrow fibrils strand which is different from the synthesized MCC. The morphology of the synthesized MCC was shorter and less fibril than the commercial MCC that have an average length of 138.6 µm and diameter 13.2 µm. This indicates that the characteristics of the extracted MCC such as particle sizes and aggregation were influenced by the raw materials and the conditions used during the hydrolysis process (Das et al., 2009)

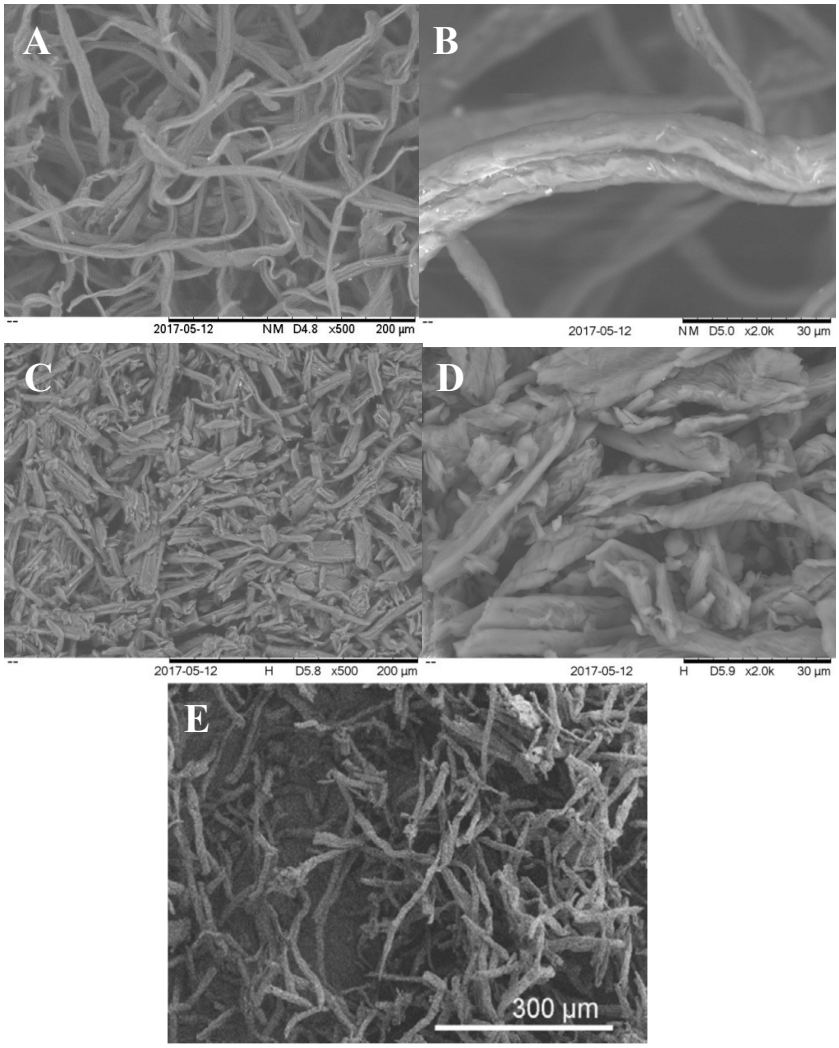


**Figure 3:** SEM micrographs of alpha cellulose (A & B) and MCC (C & D) at a magnification of 500X and 2kX; and (E) commercial MCC at magnification 500x (Xiang et al., 2016)

### *3.3.2 Fourier transforms infrared*

FTIR spectra of alpha cellulose and MCC were recorded in the range of 4000–500 $cm^{-1}$. Figure 4 shows the stretching vibration of OH group (3200 to 3400 $cm^{-1}$), C–H group (2886 $cm^{-1}$), O-H bending (1657 $cm^{-1}$), C–O-C stretching (1029 $cm^{-1}$) and the β-glucosidic (896 cm-1) (Khalil et al., 2001; Nacos et al., 2006). The dif transmittance for hydroxyl (-OH) group is represents by wavenumber around 3300 $cm^{-1}$ which existed for MCC and alpha cellulose.The broadening was attributed to the presence of amorphous fraction of the cellulose. This was due to the degradation of the hydrogen bond between the cellulosic chains during the hydrolysis process (Nasution et al., 2017) and indicating that the hydrolysis process weakened the hydrogen bonding of cellulose (Zhang, Ren, & Li, 2013). However, the peak of MCC is slightly broader, than alpha cellulose due to the increase of hydroxyl group on the surface of MCC after acid hydrolysis on cellulose. The infrared spectroscopy shows that the acid hydrolysis reaction performed to obtain MCC

does not affect the main chemical structure of the cellulosic fragments. It means that acid hydrolysis of alpha cellulose did not affect the cellulosic components.

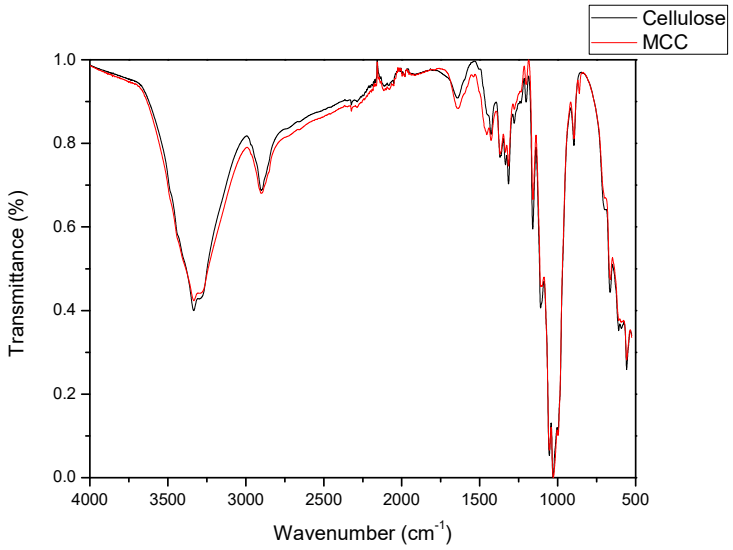


**Figure 4:** Fourier Transform Infrared (FTIR) spectra of the obtained alpha cellulose and microcrystalline cellulose (MCC)

## 4.0 CONCLUSION

MCC has been successfully synthesized from EFB. The most suitable fiber pulp consistency for the synthesis of microcrystalline cellulose was 5%. This study showed that alkaline pre-treatment at 20% of NaOH concentration gave the best result. MCC was prepared by hydrolysis acid using hydrochloric acid (2.5N). The results obtained from FTIR analysis confirmed that chemical structure of the cellulosic fragments is not influenced by the acid hydrolysis and SEM shows that the MCC has a rough and compact structure, similar to the commercial MCC, although it exhibits much smaller fragments.

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