

**CONTROLLING OF GREEN NANOCELLULOSE FIBER PROPERTIES
PRODUCED BY CHEMO-MECHANICAL TREATMENT PROCESS VIA SEM, TEM,
AFM AND IMAGE ANALYZER CHARACTERIZATION**

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ABSTRACT

Nanocellulose fibers were extracted from Oil Palm Empty Fruit Bunch (OPEFB) fibers by a chemo-mechanical treatment process. The aim of this study is to observe and investigate the morphological structure and fiber dimension characteristic of each stage in the nanocellulose production. The fiber structure characterization of the raw, purified pulps, extracted cellulose and nanocellulose fibers were controlled by observation and investigation under Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM), Atomic Force Microscopy (AFM) and Image Analyzer. The morphological observation illustrated that the isolation of nanocellulose fibers had greatly decreased from micron to nanoscale, up to 5-10 nm in diameter, and therefore suitable to be used as quality nano-reinforcement in the polymer matrix for a potential environmental green nanobiocomposites development.

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Keywords: nanocellulose fibers; morphological observation; fiber dimension; SEM; TEM.

1. INTRODUCTION

Oil palm is one of the most economical perennial oil crops and its plantation in Malaysia that generate a large amount of oil palm biomass around 40 million tons annually, representing an abundant, inexpensive and readily available source that will also cause arising serious environmental impacts [1-2]. Moreover, high cellulose content in oil palm empty fruit bunch (OPEFB) approximately around 44.4%, have attracted a great deal of research interest to isolation nanocellulose that can be utilized as a reinforcing material in the environmental green nanobiocomposites development [3].

Cellulose, a ubiquitous organic compound, is a main structural component that gives strength and stability in most of the plant's cell wall. The cellulose fiber is considered nearly inexhaustible source of raw material due to increasing demand for eco-friendly and green biocompatible products [4]. The excellent mechanical properties, remarkable reinforcing capability, low density, thermal stability and environmental benefits of cellulose have attracted scientists' interest in utilizing cellulosic fibers to develop environmentally friendly composite materials [5].

Nanocellulose term is referring to cellulosic materials that at least one dimension is in the nanometer scale, nano-size cellulose. Nanocellulose can be extracted from various lignocelluloses material including plant, agricultural/forest crops or residues and some bacteria. Recently, nanocellulose has attracted much attention during the past few years. The qualified characteristics for nanocellulose such as specific surface area and high aspect ratio, high strengthening and flexibility effect, good optical and thermal properties will find many applications in high technology and quality grade paper, nanocomposites, coating additives, food packaging, gas barriers and etc. [6].

Several processes were involved to isolate highly purified nanofibrils from cellulosic materials that lead to different size nanofibrillar materials dimension, depending on its cellulosic material, pretreatment and disintegration process [7]. Generally, pulping and bleaching with a wide range kinds of chemicals are frequently used as a starting material for the nanocellulose production. Thus, occasionally the isolation and defibrillation of nanocellulose need highly intensive mechanical treatment. The scale of cellulose in micro can be reduced to the nano-sized structure by various methods including chemical, mechanical

and chemo-mechanical treatment process [7-8].

A well-known mechanical instrument, High pressure homogenizer (HPH) and ultrasonication are usually used in this isolation and defibrillation process [7-11]. HPH can be considered as an efficient method for refining the fiber through high shear and pressure. There are many different pretreatments had been done before mechanically defibrillated the fibers such as acid hydrolysis, high shear mechanical disintegration, acetylation, carboxymethylation, TEMPO oxidation and enzyme treatment to reduce the fiber size and prevent from sticking the small orifice of HPH [7-8].

In the nanotechnology point of view, it is believed that nanocellulose have a high potential to be used as transparent and extremely strong films in many different areas. This could lead to environmentally compatible and high performance environmental green nanobiocomposite components. Many studies have been done using nano-reinforcement cellulosic sources in polymer matrices [5, 12]. Therefore, the main objective of the present study was to observe and demonstrate the morphological structure of each stage involved in the nanocellulose isolation process from OPEFB through various microscopic views. Furthermore, the fiber dimension characteristic of the treated nanocellulose compared to other raw and purified OPEFB fibers was also conducted.

2. RESULTS AND DISCUSSION

2.1. Morphology Observation of Raw, Pulp and Cellulose Microfibers

Along the process in nanocellulose production, it was observed that the morphological structure of the fibers differs with the treatment process. In the composite development, morphological structure and properties of the fiber are important to predict the interaction and gain better dispersion between composite material (fiber and matrix). SEM micrographs of the raw and treated OPEFB fibers at each process are presented in Fig. 1. The raw OPEFB fiber Fig. 1(a) is composed of bundles of continuous individual cells that are bound together by cemented components of lignin and hemicellulose. The surfaces seem uneven, tangled with lignin binder and irregular with cementing waxy materials. The diameters of the fibers were found to be around 50 μm .

Soda pulping and alkaline peroxide bleaching processes separated the fiber bundles into individual fibers with a significant decrease in fiber diameter. The surface of soda pulping

treated fibers, Fig. 1(b) was smooth and clear compared to the raw fibers due to the removal of the hemicelluloses and impurities. This alkaline treatment process improved the fiber bundles defibrillation and opening, thereby, the fiber diameter further reduced to 23 μm . As seen in Fig. 1(c), the bleached fibers using alkali hydrogen peroxide defibrillated the cellulose fiber with the diameter of 16 μm . This is attributed to the removal of lignin due to the bleaching process of soda pulping fibers and thereby decreased the fiber diameter. The bleaching treatment effectively modified the surface of the fibers, which appear smoother than the raw and unbleached pulp fibers.

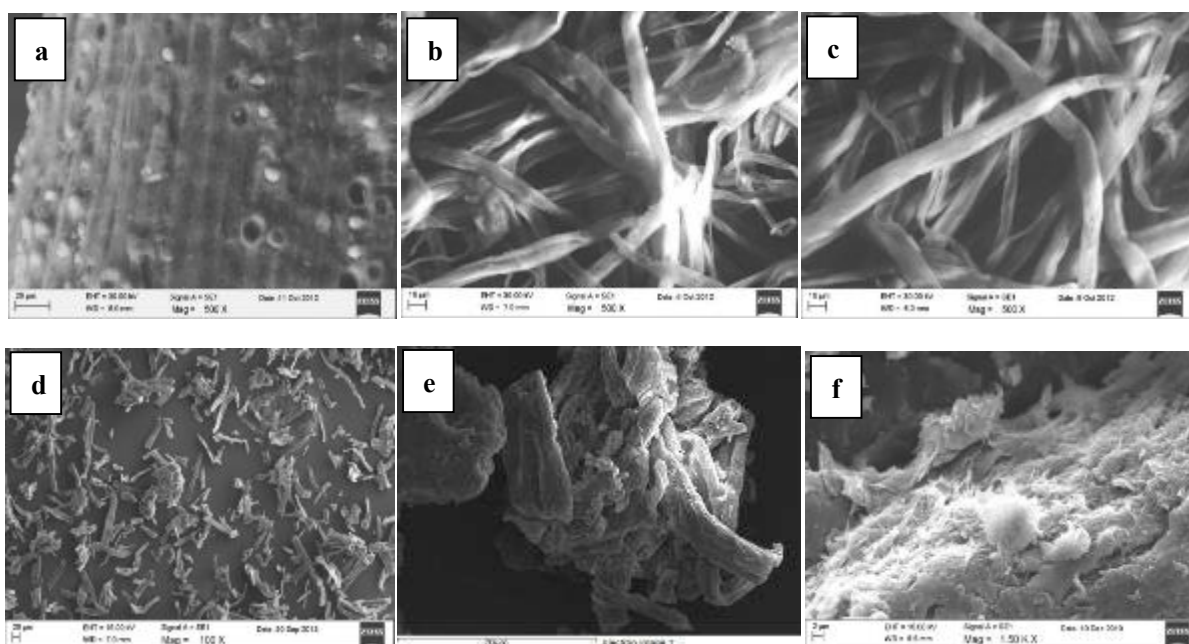


Fig.1. SEM micrographs of the raw and treated OPEFB fibers at each process. (a) Raw (b) unbleached pulp, (c) bleached pulp, (d) acid treated cellulose, (e) mechanical disintegration treated cellulose and (f) nanocellulose fibers

After the alkaline treatment, bleached slurry further undergoes acid treatment process using sulfuric acid at low concentration to decrease the fiber diameter and reduce the length. The sulfuric acid hydrolysis using 30% H_2SO_4 solution at 60 $^\circ\text{C}$ and an acid to fiber ratio of 17.5:1 for 2h had revealed that the diameter size of the acid-treated cellulose (A-cellulose) fibers slightly decreased to 9 μm , along with the shortened length of fibers (Fig. 1(d)). However, in [14] isolated nanofibrillated OPEFB fiber (100 nm-2 μm) using 64 wt% H_2SO_4 solutions at 45 $^\circ\text{C}$ and acid to fiber ratio of 10.5:1 for 15-90 min. Thus, formation and size of sulfuric acid hydrolysis treated fiber strongly depend on the acid concentration and acid to fiber ratio.

The morphological surface of A-cellulose fibers was not affected by the acid hydrolysis, but it exhibits in a smaller particle dimension with microfibrils formation [15-16]. Formation of several kinks, terraces and steps were also observed on the surface of the fibrils at higher magnification after minimal hydrolysis. This can be attributed to the removal of reactive amorphous cellulose on the surface [17].

Never-dried cellulose fibers produced from acid treatment initially had been subjected to the homogenization process directly, but unfortunately it resulted in an undesired condition where the blockage still occurred as well as difficulties in washing and cleaning the HPH machine. Mainly this happens due to the treated fiber microfibril entanglement delamination and the effects related to fiber dimension. Alternatively, a mechanical disintegration that also acts as dispersion was introduced as a solution for this matter. This mechanical disintegration process was coordinated to make sure that the treated cellulose fibers are able to flow smoothly in the HPH and at the same time minimize plugging. It also helps in easing the cleaning process after the homogenized material had gone through several passes and achieved the finishing target.

The mechanical disintegration was performed using Ultra Turrax T-18 machine for 20 minutes or less. This appropriate machine not only facilitates homogenization process by exposing more accessible defibrillation through high shear forces and cutting action but also disperses and liberates the treated fiber [18]. The dispersion represents the treated fibers to individualize and avoid the fiber that is usually in distilled water to settle down and delaminate fast in the HPH machine. This giving appropriate time for the water to flow well through a small orifice with less entanglement around/covered the surface that led to machine blockage especially. As a result of this overall process, the clogging problem is largely overcome.

SEM micrographs of mechanical disintegration treated cellulose (M-cellulose) fibers had demonstrated that the fibril diameter reduced significantly from 9 μm to 0.7 μm as compared to the earlier reported solely A-cellulose fibers. The M-cellulose fibers in Fig. 1(e) had succeeded in greater reduction were gradually converted to smaller diameter size in nanometer scale dimension within the range of 600-800 nm. They also were liberated to form individualized, finer and less aggregated fibril compared to the A-cellulose fiber. Therefore,

M-cellulose fiber exhibited the lower dimension (diameter and length) with the high specific surface area was assumed to result in highest aspect ratio compared to A-cellulose fibers.

The dimension of the M-cellulose fiber was determined and measured by SEM micrographs and an image analyzer. As the fibrils form an entangled fine structure, the exact fibril length was difficult to be measured. Even though it is not possible to precisely measure the length of the M-cellulose fiber, but from the micrographs observation, they are shortened around less than 9 μm . In [17] finding also reported that in their research some part of the microfibrils become agglomerated while the rest remain separated. Agglomeration in the corresponding microfibrils will allow incorrect and inaccurate dimension measurement of the diameter and length. Thus, the microfibrils changes in diameter and length will not be able to measure statistically when the agglomeration occurred after the hydrolysis process.

In addition, from the nanocellulose observation in Fig. 1(f) and measurement obtained in this study, it was found that the result was similar to the reports on nanofibrillation process done by other researchers [19-21]. This clearly indicated that our approach on applying high shear mechanical disintegration, ultra turrax is considered more effective and practical. Moreover, refining through the homogenization is a process to reduce the energy consumption of the isolated cellulose nanofiber (CNF) where it facilitates in loosens and softens the cellulose fibers structure. This process will generate external fibrillation by increasing fine fibers on the primary and secondary, S1 surface layers via abrasive action as well as allows internal fibrillation by breaking the bonds between CNF [21-22].

2.2. Morphology Observation of Cellulose Nanofibers

In the first stage of nanocellulose individualization process, treated ultra turrax cellulose fibers was subjected to numerous passing times in the HPH machine. The effect of passing time was examined through Transmission Electron Microscope (TEM) micrographs to evaluate the optimum passing time. The aim was to achieve the smallest fibril size dimension of homogeneous individual nanocellulose that requires less time and energy consumption. This optimum passing time parameter, lead to better properties related to the high-end nanobiocomposite production. Fig. 2(a)-(c) illustrated the TEM micrograph of N-10p, N-20p and N-30p which indicated the treated nanocellulose passing time at 10, 20 and 30 cycles in

the HPH machine respectively.

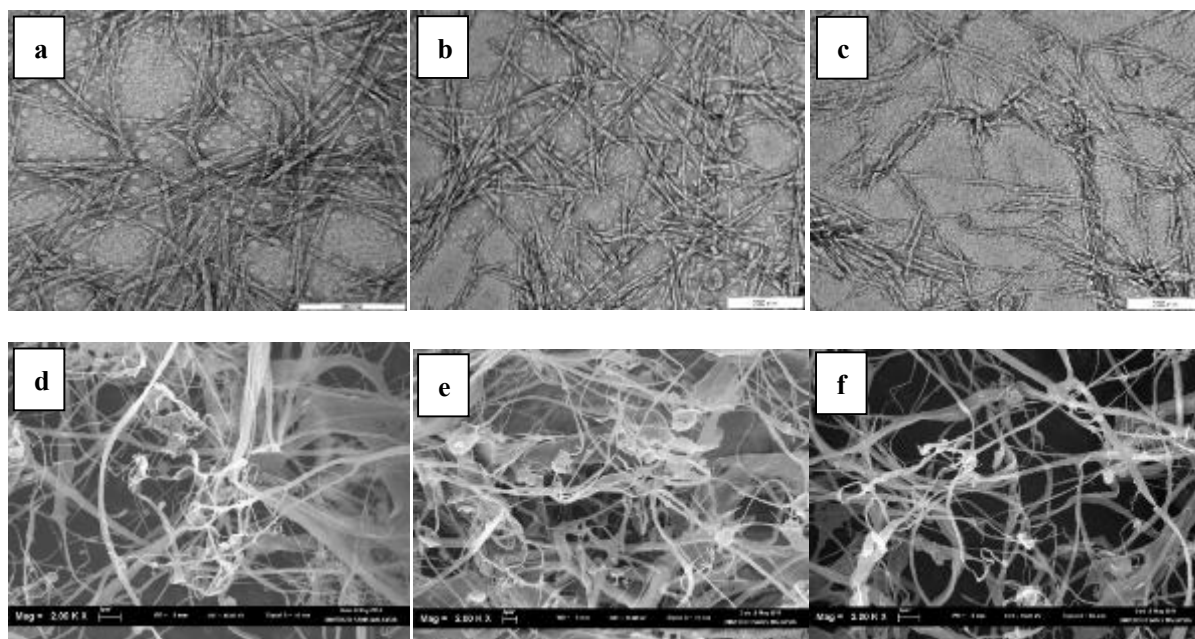


Fig.2. TEM and SEM micrograph of nanocellulose at different passing time.

TEM micrograph: (a) N-10p, (b) N-20p and (c) N-30p; SEM micrograph: (d) N-10p, (e) N-20p and (f) N-30p

The TEM micrograph demonstrates the efficiency of the combination of chemical and mechanical treatments, which illustrated the individual nanofibrils with little agglomeration. Basically, all of the nanocellulose had experienced similar changes upon homogenization in terms of the cell wall extensive deconstruction and the diameter size reduction. There was an only small significant difference among the treated nanocellulose at the different passing time. In addition, after ultra turrax and homogenization treatment, fibers were no longer visible and only small fibrils in nanometer dimension were observed. In Fig. 2(c), the N-30p nanofibrils range roughly from 5 to 10 nm in diameter which was the finest/smallest diameter size among two other different passing times, N-20p and N-10p. This performance indicates large potential as a reinforcing agent for environmental green nanobiocomposites [5].

In comparison with other previous studies on the isolation of cellulose nanofiber using different chemo-mechanical processes, the diameter of 5 to 40 nm was achieved for OPEFB cellulose nanofiber [11, 23] and 10 to 70 nm for kenaf nanofibers [24]. Almost similar nanofibril dimensions had also found by other researchers with a different combination of treatment and processes using different machine and techniques approached [9-10]. Thus, the

combination of sulfuric acid hydrolysis, mechanical disintegration and high-pressure homogenization processes is more highly efficient and effective in the isolation of nanocellulose fibers.

After the nanocellulose suspension was collected from HPH, it was subjected to vacuum freeze-drying method. Morphology structure from SEM micrograph images in Fig. 2(d)-(f) at the different passing time revealed that nanocellulose consisted of slender, narrow and long nanofibrils web-like network structure with high surface area. From the appearance, these strong skeleton structures of nanocellulose are very lightweight material and flexible similar as cotton structure due to expected low density and high porosity.

Nanocellulose suspension had been assembled to a dry state nanofibril structure within vacuum freeze drying process. During the vacuum freeze drying, nanocellulose experienced few changes in following three stages such as freezing to form separated ice crystals sublime stage, nonfreezing bound water immersed adsorption by vacuum and nanocellulose agglomeration in the final stage of freeze drying process. This particular method allows the sample to be frozen by liquid nitrogen resulting in ice sublimation, then this ice frozen state was excluded from the presence of vacuum water suction and leftover was cotton-like dried nanocellulose [25].

The morphological characterization indicated that the combination of acid treatment techniques with mechanical disintegration by ultra turrax and finally homogenization produced nanoscale with high aspect ratio fibrils from entangled strong network structures. Based on TEM and SEM observation as well as average fiber dimension measurement, the smallest nanocellulose dimension was obtained by N-30p and therefore 30 passes/cycles have been selected/detected as the optimum HPH passing time. Thus, these nanocellulose properties can be introduced and utilized as reinforcement in new applications of environmental green nanobiocomposites.

2.3. Atomic Force Microscopy Observation

The dimensions and homogeneity of selected nanocellulose material (N-30p) were also studied using Atomic Force Microscopy (AFM). AFM is another instrument used to observe the nanostructure material other than TEM. Both microscopic observations, AFM and TEM

revealed that the nanofibrils have a high aspect ratio, form entangled network structures and moderately agglomerated even though at very low concentration of nanocellulose suspension in the HPH. The AFM imaging and properties of nanocellulose substrate were frequently performed to characterize the surface morphology and structure. Surface roughness characteristics of the nanocellulose that also contribute to the microscopic measurement were determined through surface AFM imaging. Representative phase contrast, topography and height images of the surface roughness 3D profile of AFM images were presented in Fig. 3(a)-(c) respectively.

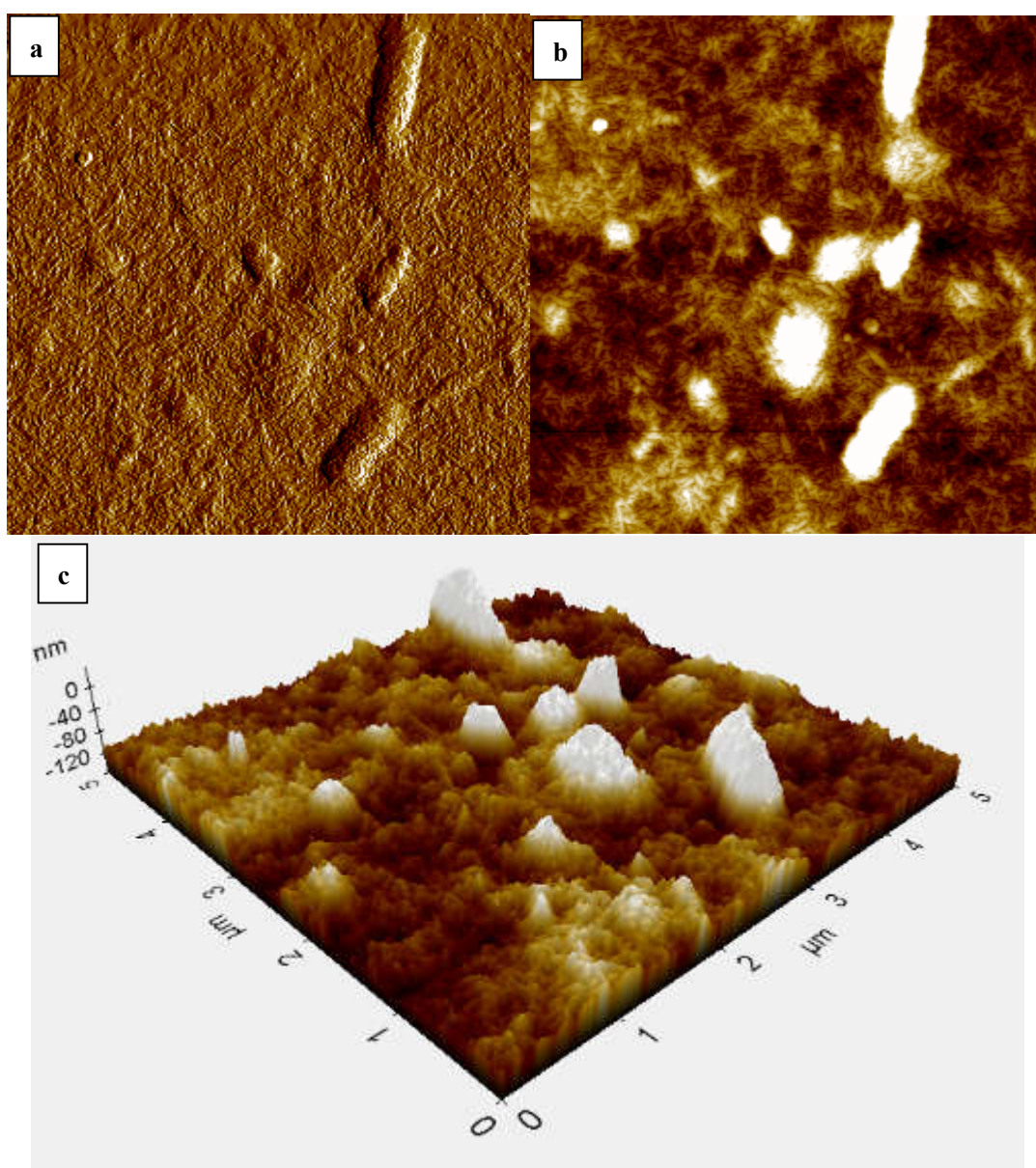


Fig.3. AFM images of nanocellulose, N-30p. (a) Phase contrast, (b) Topographical image and (c) Height image surface roughness 3D profiles

The overview on surface roughness from AFM images of nanocellulose, Fig. 3(c) showed a nanoscale roughness features that appears to be fine and small which similar to the TEM micrograph results obtained. However, the images showed no evidence of individual fibrils such as those clearly viewed in the TEM image. This effect and dense appearance were believed to originate from the aggregation of the nanocellulose. A cross-sectional AFM image for the height of the nanocellulose substrate occurred from the tip that moves along the sample glass slip. The AFM height profile in the middle of the image showed structured nanofibrils that form uniform coverage network with few aggregated fibril bundles. The height of the fibrils was determined to be about 5 nm, which supports and correlates with the TEM measurements discussed earlier.

The fibrils dimension results from AFM height image and profile indicated that the widths were approximately 8-13 nm and the length estimated in the range of $\sim 1 \mu\text{m}$. For instance, the width from AFM result was larger than as compared to TEM result analysis. This referred case can be due to the fibrils dimension being affected by aggregation or flattening that may occur during the drying process in the sample preparation stage. Furthermore, the AFM tip used to detect the aggregated fibrils will not be able to reveal the actual size on account of small error being caused in the fibrils diameter measurement. Mainly, both appearance and fibril dimension correspond to similarly prepared fiber surfaces by [26].

2.4. Image Analyzer Observation

Morphology characterization of the fibers at each stage, before and after homogenization was displayed and presented in this study. In addition, Fig. 4 illustrated the overall OPEFB morphology and appearance photos captured using image analyzer camera in dried form at each different stage that took place in the nanocellulose isolation process from micro to nanoscale fibers (raw, unbleached pulp, bleached pulp, acid treated cellulose, mechanically treated cellulose and nanocellulose). This soft white cotton-like fibrous structure dried nanocellulose in the final stage will be later used as reinforcement material in environmental green nanobiocomposite development.

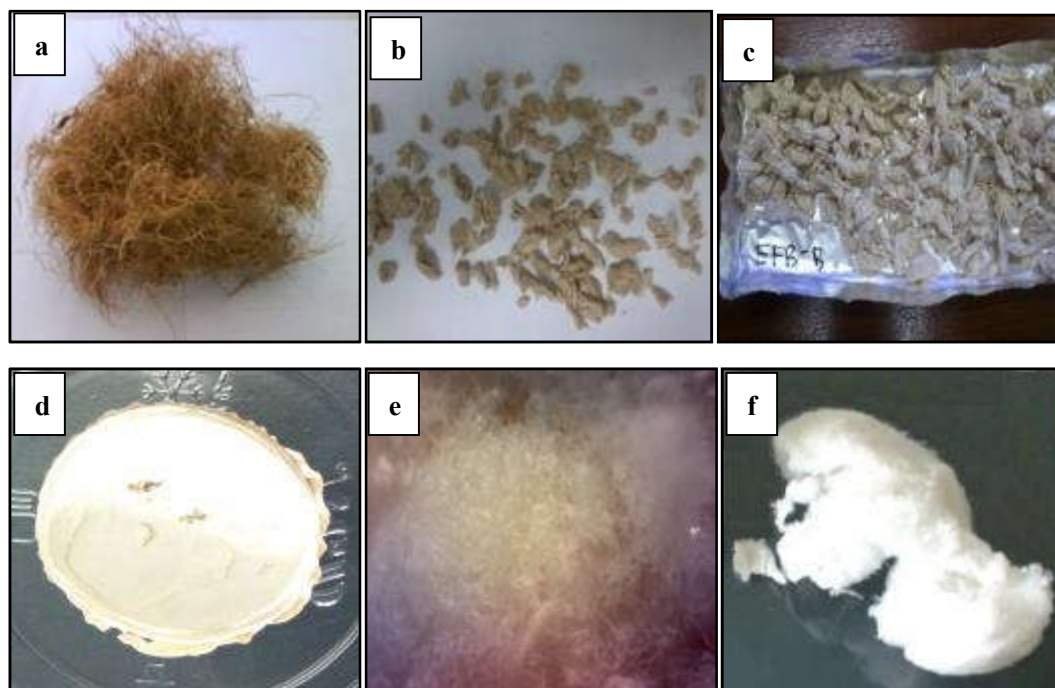


Fig.4. Image analyzer photos of OPEFB fibers at different stages of nanocellulose isolation process in dried form (from micro to nano scale); (a) raw, (b) unbleached pulp, (c) bleached pulp, (d) acid treated cellulose, (e) mechanically disintegrated treated cellulose and (f) nanocellulose fibers

2.5. Fiber Dimensions Characterization

Table 1 displayed overall fiber diameter size for the raw (R), unbleached pulp (P), bleached pulp (B), acid-treated cellulose (A), mechanical disintegration treated cellulose (M), nanocellulose (N) fibers obtained from image analyzer measurement. From the table, as observed along the process, fiber diameter size of the treated cellulose fibers had successfully reduced from micro to nanoscale at each stage/process throughout the whole nanocellulose production. Furthermore, it also indicated that N-30p exhibited the smallest dimensions with expected highest aspect ratio and specific surface area as compared to other nanocellulose samples at a different passing time. From the microscopic measurements, the length of the nanofibrils was difficult to determine due to the earlier mentioned entangled network structure. Consequently, the fibril length was estimated to be up to several micrometers.

Table 1. Fiber dimension of acid and ultra turrax treated fibers

Treated Fibers	R	P	B	A	M	N-10p	N-20p	N-30p
Diameter (μm)	50 μm	23 μm	16 μm	9 μm	0.7 μm	13 nm	10 nm	6 nm

*R = raw, P = unbleached pulp, B = bleached pulp, A = acid treated cellulose, M = mechanical disintegration treated cellulose, N = nanocellulose at 10 passes, 20 passes and 30 passes

3. METHODOLOGY

3.1. Materials

Raw oil palm empty fruit bunch (OPEFB) fibers were obtained from the Malaysian Palm Oil Board (MPOB, Kajang, Selangor, Malaysia). Analytical grade chemicals used for chemical treatment process were: sodium hydroxide (NaOH), hydrogen peroxide (H_2O_2), sodium silicate (NaSiO_3) and magnesium sulfate (MgSO_4). Sulfuric acid, H_2SO_4 (95%) was used in acid hydrolysis pre-treatment process. The entire analytical grade chemicals were supplied by Chembumi Sdn. Bhd., Penang, Malaysia.

3.2. Preparation of Cellulosic and Nanocellulosic Fiber

The preparation of cellulosic and nanocellulosic fiber extracted from OPEFB was conducted according to [8] experimental method and condition. The raw OPEFB fibers undergone soda pulping and alkaline peroxide bleaching process in order to efficiently remove the non-cellulosic materials. These purified OPEFB pulps were then treated with acid and followed by ultra turrax treatment to produce, liberate soften and reduce diameter micron size of the treated cellulosic fibers.

At the end of the prolonged process, treated micron cellulosic fibers were subjected to homogenization process to obtained nanocellulosic fiber. The morphology and fiber dimension of the OPEFB fibers at each stage of the process were observed recorded and named as raw (R), unbleached pulp (P), bleached pulp (B), acid-treated cellulose (A), mechanical disintegration treated cellulose (M), nanocellulose (N). Overall process was demonstrated in the flowchart in Fig. 5.

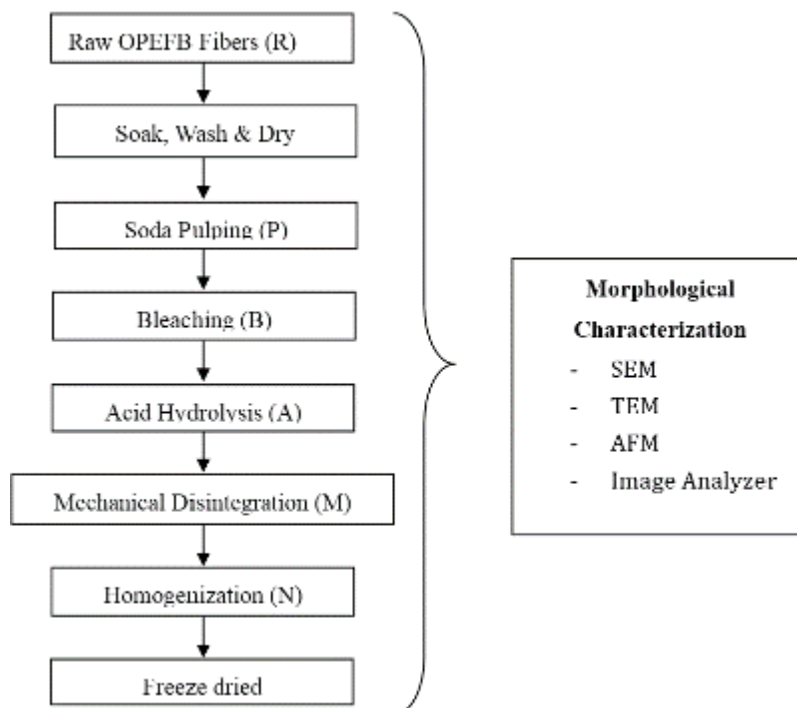


Fig.5. Flowchart of overall nanocellulose isolation process and its morphological characterizations. Raw (R), unbleached pulp (P), bleached pulp (B), acid treated cellulose (A), mechanical disintegration treated cellulose (M) and nanocellulose (N) fibers

The raw OPEFB fiber (R) was pulverized using soda pulping with the optimized pulping condition: NaOH concentration 26.1%; temperature 161 °C and time 100 min [13]. Approximately, 200 g of raw OPEFB fiber was taken in a digester at ratio of cooking liquor to fibers, 10:1 and treated at 161°C for 100 min. Wherein, the heating time was found to be 60 min (the time duration required to reach to target temperature) and the exposure time was 40 min. Subsequently, the pulps were washed with distilled water and then mechanically disintegrated in a three-bladed mixer for 1 min at a 2.0% of pulp consistency and screened on a 0.15 mm slits flat-plate screen.

The alkaline hydrogen peroxide bleaching process was carried out in order to purify and removes residual lignin from the OPEFB soda pulps (P). The bleaching process was conducted using 5% pulp consistency with gradual series of chemical: 3% H₂O₂, 0.5% MgSO₄ and 3% NaOH at 50°C for 90 min. After bleaching, the bleached slurry was washed to a neutral pH and then oven dried at 60°C for 24 h.

In the acid treatment process, the bleached slurry (B), 10g was hydrolyzed in 30% H₂SO₄ solution at 60°C and acid to fiber ratio of 17.5:1 for 2 h. The hydrolyzation process was

terminated by adding cold distilled water. The never dried slurry after acid treatment (A) was dispersed in distilled water at the concentration between 0.5-1.2 %. The diluted slurry was then mechanically treated using high shear ultra turrax (T-18 basic, IKA Company, Königswinter, Germany) for 20 min or less and repeated three times.

Subsequently, the suspension (M) was passed through a high pressure homogenizer (HPH) (Model: HPH 2000/4, IKA company, Germany) 30 times at a pressure of 50 MPa. Finally, nanocellulose suspension (N) was washed and freeze-dried in a freeze dryer (FDU-1200) at temperature -54°C for 24 hours. White cotton-like nanocellulose produced from the freeze dryer was collected prior to morphology observation and ready to be used as reinforcing the material in the nanobiocomposite development.

3.3. Morphology Observation

The morphology of cellulose fibers was observed by Scanning Electron Microscopy (SEM, EVO MA10, Carl- ZEISS SMT, Germany). The fiber surfaces were mounted on aluminum stubs and sputter coated with a thin layer of palladium to avoid electrostatic charging during the examination. The nanocellulosic fibers were studied under Transmission Electron Microscope (TEM, Philips CM12 instrument, Amsterdam, The Netherlands). One drop of nanocellulosic suspension was put on the carbon-coated grid at room temperature for 24 hours. Then, the samples were stained with 2% uranyl acetate and lead citrate. Image of nanocellulosic was captured at acceleration voltage 120 kV. The diameter and average mean size distribution value of cellulose and nanocellulose fibers were measured and recorded by image analyzer program.

The AFM (XE-70 Park System) was utilized to measure the size and study the surface roughness of nanocellulosic fibers. One drop of diluted nanocellulosic suspension was placed on freshly cleaved mica and the analysis was done using 300 kHz resonance frequency.

4. CONCLUSION

In this study, purified cellulose fibers were effectively isolated from OPEFB fiber by soda pulping and alkaline peroxide bleaching process. Acid hydrolysis and mechanical disintegration treatment had facilitated the HPH machine by defibrillation and size dimension

reduction of the nanocellulose fibers. The SEM study showed that the morphological surface of treated fibers was not affected by the acid hydrolysis, but it exhibits in a smaller particle dimension. The diameter of nanocellulose produced from the HPH machine is in between 5-10 nm as was confirmed by TEM. On the overall morphology and fiber dimension observation, along with the process, the treated cellulose fibers had successfully reduced the size from micro to nanoscale at each stage throughout the whole nanocellulose production as were confirmed and evident by various microscope instruments. Therefore, based on the morphological observation, it was evident that the isolation of nanocellulose fibers prepared by the combination of chemical and mechanical treatment is suitable and effective to be used as quality nano-reinforcement in the polymer matrix for a potential environmental green nanobiocomposites production in various wide high-end applications.

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