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PREPARATION AND CHARACTERIZATION OF MICROCRYSTALLINE CELLULOSE FROM OIL PALM FROND BY CHEMICAL AND MECHANICAL TREATMENT

Randis [2)]✉, Djarot B. Darmadi [1)], Femiana Gapsari [1)], Achmad As'Ad Sonief [1)], Tatag Yufitra Rus [3)]

[1)] **Mechanical Engineering Department** Universitas Brawijaya Malang, Indonesia randis@student.ub.ac.id

[2)] **Mechanical Engineering Department** Balikpapan State Polytechnic Balikpapan, Indonesia randis@poltekaba.ac.id

[3)] **Civil Engineering Department National** Taiwan University Taipei City, taiwan tatag.yufitra@poltekba.ac.id

Abstract

Oil palm fronds are currently an underutilised by product of plantations. The micro and nano cellulose can be utilised to create new goods, such as being used as a filler in environmentally friendly degradable composites. This study examined the process of extracting oil palm frond fiber (OPFF) by using a series of chemical and mechanical treatments to get pure microcrystalline cellulose (MCC). The MCC obtained by OPFF was subsequently analysed in further detail. The identification of morphological analysis, presence of functional groups, and crystallinity index were conducted using Scanning Electron Microscopy (SEM), Fourier Transform InfraRed (FTIR) and X-Ray Difraction (XRD) techniques. The findings revealed the presence of microcrystalline structures in OPF fibers, having a mean measurement of width 3.2 ± 0.42 µm. The application of FTIR verified that the elimination of noncellulosic constituents from the treated OPF fibers had achieved its utmost level. Simultaneously, X-ray diffraction research revealed that the utilization of chemical procedures resulted the formation of a characteristic cellulose crystal structure and an augmentation in the crystallinity index. However, the mechanical treatment resulted in a minor drop in the crystalline index. This can be ascribed to the disturbance of cellulose chains and the crystal structure within the cellulose fibers. The cellulose obtained from raw fibers and subsequent processing exists in a very pure form, specifically in the cellulose Iβ structure. This enables its utilization as reinforcement in eco-friendly MCC-based green composites, offering numerous benefits.

Keywords: *Oil Palm Frond Fibers, Microcrystalline Cellulose, Chemical Treatment, Mechanical Treatment, Ball mill, Characterization*

1. INTRODUCTION

Indonesia has been widely acknowledged as one of the top oil palm producers globally. Large palm oil production supports the national economy and significantly impacts the global palm oil market [1]. According to recent statistics, palm oil production in Indonesia continues to increase, carving a niche as one of the leaders in the industry. However, this success of the palm oil industry also brings significant challenges related to managing the waste generated [2]. Besides producing valuable palm oil, the palm oil industry also creates a significant volume of waste. One of the primary sources of this waste is palm fronds. Oil palm fronds generated by the industry account for nearly 70% of the overall solid waste generated [3]. The

Corresponding Author:
✉ **Randis**
randis@student.ub.ac.id
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presence of this waste is not only an environmental problem but also an opportunity that has yet to be fully utilized.

This oil palm frond waste has enormous potential to be processed into innovative microcellulose-based products. So far, palm frond waste is often left to rot in plantations or burned, which produces harmful emissions and adversely affects the environmental ecosystem [4], [5]. In this context, using palm frond waste in microcellulose-based products can be a significant step in reducing the negative environmental consequences of the palm oil company [6]–[11]. The microcellulose that can be produced from palm fronds is a revolutionary material and has potential in various industrial applications, including adsorption of methylene blue [7], immobilized adsorbent [8], filler in polymer electrolyte [9], [10], and raw material in renewable energy production [11].

Multiple accounts exist regarding the isolation of MCC from multiple plants with different isolation techniques, such as cellulose from soybean hulls using extrusion [12], Gluconacetobacter xylinus and kenaf fibers using centrifugation method [13], OPEFB fiber using maceration and ultrasonic treatment [14] and sugarcane bagasse using chemical and grinding process [15]. Meanwhile, oil palm fronds processed to obtain MCC generally use chemical treatment [6]–[11],[16], [17]. However, to our knowledge, studies on MCC sourced from oil palm fronds using chemical and mechanical (ball milling) treatments have yet to be found in the literature.

The current work employs chemical and mechanical (ball milling) techniques to produce MMC from OPFF. Extensive investigations have been conducted on MCC's chemical composition and surface morphology. The investigations were conducted using various analytical techniques, including chemical, scanning electron microscope, FTIR and XRD analysis.

2. MATERIALS AND METHODS

2.1. Materials

The primary material used this present research is the fibers from Oil Palm Frond (OPF), that were procured from agricultural waste of oil palm plantations in Kuaro District, Paser Regency, located in East Kalimantan Province, Indonesia. The gathered OPF diminished to a lesser extent of 30 cm and subsequently purged of thorns and outer peel. The selected OPF sections were derived from trees that were at least 20 years old. The optimal age range for this portion is between 18 and 19 years old [18], [19]. Subsequently, the flowing water purifies the fibers by removing contaminants. Subsequently, the fibers are individually extracted and subjected to a drying process in an oven in a temperature range of 70^{o}C until a water concentration at 9-10 % is achieved. The fibers were reduced to smaller dimensions of 1-2 cm to achieve consistent fiber diameters. Subsequently, they were pulverised using a blender until they formed particles that passed through a 60-mesh screen (figure 1. a-b). The chemical treatment method involved the use of analytical-grade distilled water, sodium hydroxide (NaOH), and hydrogen peroxide (H_2O_2) as reagents.

2.2. Method

2.2.1. Delignification and Bleaching Treatment

The process of treating fibers chemically involves the use of maceration and bleaching process to eliminate lignin and hemicellulose from the raw fibers [20]. The alkaline treatment process combines finely crushed raw OPFF with a 9% sodium hydroxide (NaOH) solution,

followed by heating and stirring the mixture for 1 hour within a temperature scope of 70°C. The byproduct of the alkali process is utilised in the bleaching procedure, wherein a mixture of 25% H2O2 and macerated powder is heated for 60 minutes. The process is iterated twice to acquire a residue of pure white cellulose. The residues are washed with pure water until they reach a pH level of neutrality and then subjected via a drying process in a 105°C oven to a water concentration at 9-10 % after each treatment [21].

Figure. 1. OPFF chopped (a), Raw OPFF (b), OPFF after chemical treatment (c), OPFF after mechanical treatment (d).

2.2.2. Mechanical Treatment Using Ball Mill

In the investigation, 5 grammes of chemically treated dry pulp samples were ground using a ball mill in a 300 ml cylinder. The mill was operated when spinning at a rate of 200 RPM, at atmospheric pressure, at a temperature reaching 30°C during a period of 2 hours [22]. The outcomes will yield MMC, which will be characterized using SEM, FTIR, and XRD analyses. The process of obtaining MCC from raw OPFF is illustrated in Figure 1.

2.3. Characterization Methods

2.3.1. SEM Analysis

The SEM model FEI inspect S-50 was utilized for analyzing the surface structure of OPF microcrystalline cellulose fibers. The fibers were uniformly spread onto conductive tape and subjected to a 60-second gold spraying process to disperse static charge. The scanning electron microscope (SEM) captured images using a 20 kilovolt (kV) accelerating voltage.

2.4. FTIR Analysis

FTIR spectra are employed to discern the chemical and physical characteristics of materials, as well as the molecular vibrations [23]. This work utilised a Shimadzu FTIR analyzer to analyse the structural changes that occur during the transformation of raw fibers into microcrystalline cellulose fibers. The specimens were mixed with potassium bromide, and FTIR spectra were acquired using the transmittance mode within the 500-4000 cm-1 range.

2.5. XRD Analysis

The study employed XRD to examine the chemical composition of a substance using an X'Pert PRO PANalytical device [23][24]. A copper target was used as the X-ray source, and the specimens were surveyed at an angle ranging from 2 to 60 degrees (2θ). The Segal approach was employed to quantify the crystallinity index (Ic) by analysing the diffraction intensity data [25]. The index of crystallinity was determined by employing Equation 1. The XRD technique yields precise and detailed data on both the quantitative and qualitative aspects of a material's chemical makeup.

$$\text{CrI (\%)} = \frac{I002 - Iam*}{I002} \text{ x } 100 \qquad (1)$$

3. RESULTS AND DISCUSSION

3.1. SEM Analysis

Figure 2 illustrates the alteration in surface morphology of the unprocessed fibers subsequent to each treatment. Raw OPFF appears to be still in bundled form, covered with various impurities, as shown in Figure 2(a). Raw OPFF without treatment have a diameter of 69.86 ± 1.24 μm. Changes in morphology and fiber diameter are clearly visible after the chemical and mechanical treatment. Figure 2(b) and (c) clearly illustrates the attendance of hemicellulose and lignin, along with other extractives, have been eroded, resulting in clean and smooth-looking fibers, as well as a significant reduction in diameter, the fiber diameter being 36.32 ± 1.56 μm indicating that chemical treatments such as alkali and bleaching can remove lignin content and partial removal of impurities and reduction in fiber diameter as has been reported in previous studies [26]–[28].

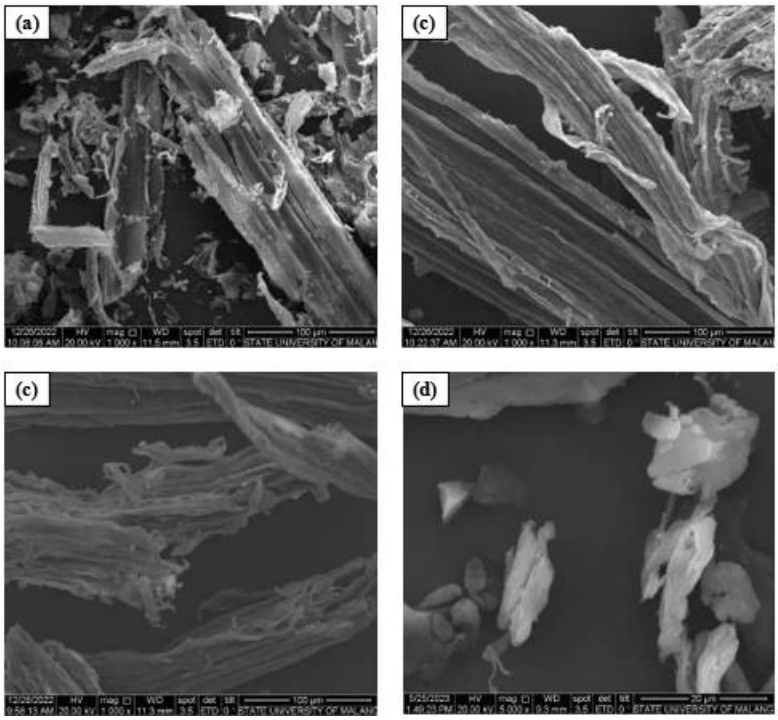

Figure 2. SEM micrograph of raw OPF fibers (a); chemical treat (b-c); dan mechanical treat (d).

After mechanical treatment in the form of a ball mill, cellulose fibers undergo a reduction in length and fiber diameter [29], [30]. The fibers are transformed into irregularly shaped microcrystalline cellulose (MCC) (Figure 2(d). This may be due to mechanical forces in the form of impact forces, shear forces, and friction forces that produce high energy repeatedly during the ball mill process, which can grind cellulose fibers into smaller particles and form MCC or nanocellulose [31]–[33]. A reduction in fiber diameter occurred, with MCC particles obtained measuring ~3.2 ± 0.42 μm. Similar morphological changes were observed during chemical and mechanical (ball mill) extraction of cellulose from kenaf fibers, wheat straw fibers, and pineapple leaf fibers [30], [34].

3.2. FTIR Analysis

The infrared spectrum of the OPFF before and after treatment are illustrated in Figure 3, while the Fourier transform infrared wave number peak data of the OPFF are presented in Table 1. The absorption peaks observed at 3342.63-3346.49 cm-1 in the OPFF spectrum correspond to the stretching vibrations of OH- groups and hydrogen bonds in the hydroxyl groups. These peaks serve as an indicator of the existence of cellulose within the fiber sample, as reported in references [21], [26], [35]. The wave values ranging from 2899.01 to 2912.51 cm-1 demonstrate the stretching of the CH- group [36], indicating the presence of the cellulose component in all samples.

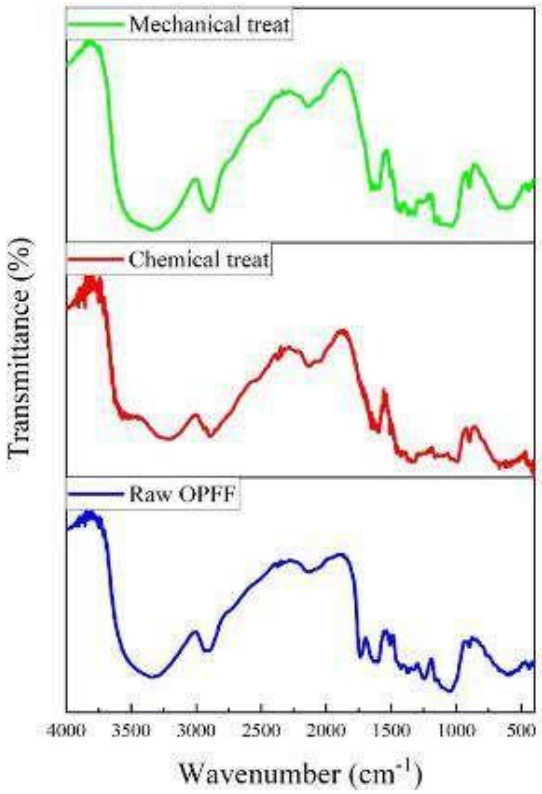

Figure 3. FTIR spectrum of raw OPFF, chemical treat, and mechanical treat

Peaks corresponding pertaining to the stretching vibration of C=O in hemicellulose were observed at wavenumbers of 1735.93 and 1246.01 cm-1 [31], [37]. Additionally, peaks corresponding to the stretching vibration of C-C in aromatic hydrocarbons of the lignin ring were observed at a wavenumber of 1508.33 cm-1. These three peaks were exclusively observed in the unprocessed OPF fiber samples. The nonattendance of hemicellulose and lignin in the post-treatment samples suggests that these components were eliminated through chemical and mechanical processes, resulting in a microcrystalline material with a high cellulose concentration.

The absorbance bands observed at 1030, 1316, 1370, and 1424 cm-1 are caused by the asymmetric valence vibrations of C-O-C, stretching vibrations of C-H_2, and deformation vibrations of C-H_2. These peaks are identified as carbohydrate forms of cellulose [38]. The presence of C-H functional groups may be observed at wave numbers 896.89-898.82 cm-1, which suggests a characteristic cellulose structure associated with (β-glycosidic cellulose) [39], [40] in both the raw and treated fibers.

Table 1. Peak data of the fourier transform indfrared wave number of OPFF

Raw OPF fibers	Chemical Treat	Mechanical Treat	Functional group	Corresponding chemical constitution
3342,63	3342,63	3346,49	-OH streaching	Cellulose
2912,51	2899,01	2899,01	CH- streaching	Cellulose
1735,93	-	-	C=O stretching	Hemi-cellulose
1508,33	-	-	C-C stretching	Lignin
1246,01	-	-	C=O stretching	Hemi-cellulose
1049,84	1041,16	1041,61	C-O Streaching	Cellulose
898,82	896,89	898,82	C-H vibration	Cellulose

3.3. 3.3XRD Analysis

The XRD spectra of untreated raw fiber and chemically and mechanically treated fiber obtained from OPF fiber indicate crystallinity indices of 58.75%, 73.09%, and 66.02%, respectively (Figure 4). The occurrence of prominent diffraction peaks at angles 2θ = 18◦, 22.6◦ and 34.5◦ signifies the presence of structure of cellulose-I [29], [41]–[43]. This suggests that all fiber samples exhibit identical diffraction patterns, suggesting that the cellulose fiber structure remains undamaged following chemical and mechanical treatment.

The results indicate that the CrI value will increase following the chemical treatment in comparison to the raw fiber. However, the crystallinity index (CrI) dropped following mechanical treatment (ball mill) compared to the CrI after chemical treatment, although it remained greater than the initial CrI of the raw fiber. The decrease in CrI may be attributed to the impairment of the cellulose chain and crystal structure caused by the mechanical treatment [44]–[46]. Consequently, the mechanically treated fiber exhibited an 8% reduction in CrI value when compared to the chemically treated fiber. The crystallinity index (CrI) of Microcrystalin obtained in this study was 70.02%, which is greater than the CrI of MCC obtained from earlier palm frond microcrystalin extractions with different procedures (62.5%) [17].

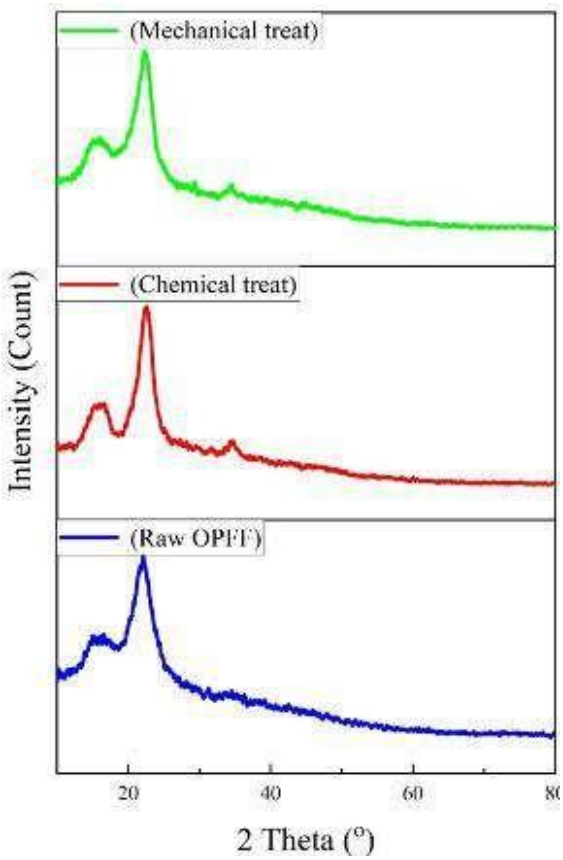

Figure 4. XRD curves of raw OPFF, chemical treatment, and mechanical treatment.

4. CONCLUSION

From The investigation effectively isolated OPFF using chemical and mechanical processes. Consequently, MCC was generated exhibiting an average dimension of 3.2 ± 0.42 μm. The

FTIR spectroscopy verified the most extensive Elimination of noncellulosic components from the treated OPFF. The chemical-mechanical treatment resulted in a greater crystallinity index of 73.09% compared to the untreated fibers, which had a crystallinity index of 58.75%. The optimal approach for achieving cellulose purity and synthesising MCC is regarded as perfect, rendering it a feasible choice for producing lightweight, eco-friendly green biocomposites.

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