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*Article*

# Synthesis of Nano α-Cellulose-Based Bioplastic from Empty Fruit Bunches of Palm Oil

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**Abstract.** Cellulose, as a renewable resource, has recently emerged as a primary focus for researchers aiming to develop sustainable biomaterials. The extraction of α-cellulose from Empty Fruit Bunch (EFB) has been conducted through hydrolysis with 3.5% $HNO_3$, delignification with 2% $Na_2SO_3$, pulping with 17.5% NaOH, and bleaching with 10% $H_2O_2$. The yield obtained according to those processes was 22.97%. Then, hydrolysis using 45% $H_2SO_4$ and sonication at a frequency of 20 kHz were used to convert α-cellulose to nano α-cellulose. Moreover, the obtained products were characterized using Fourier Transform Infra-Red (FTIR), Scanning Electron Microscopy (SEM), Particle Size Analyser (PSA), and Differential Thermal Analysis (DTA). FTIR spectra show that there is no significant difference between them. Apparently, the significant absorption band of cellulose structure at 3300 $cm^{-1}$ (O-H stretching), 2900 $cm^{-1}$ (C-H stretching), and at 1022 $cm^{-1}$ (OCH-O-$CH_2$ cellulose framework). SEM images revealed that changing the surface morphology of the α-cellulose (fibril-shaped) to nano α-cellulose (rod-shaped) resulted in average fiber diameters of 3.07 µm and 2.80 µm, respectively. Particle size distribution of the suspended particles after acid and sonication treatment becomes 380.79 nm. Based on the DTA results, the obtained products have good thermal stability, which decomposes at a temperature 290 °C. Furthermore, nano α-cellulose was used as a starting material to generate biodegradable plastic with the addition of silica as a filler and glycerin as a plasticizer. Mechanical tests of bioplastics, such as tensile strength, elongation, and modulus of elasticity, show that silica can improve the properties of bioplastics. Finally, the elongation properties of bioplastics meet the Indonesian National Standard (SNI) requirements; however, the tensile strength and modulus of elasticity values require further experiments.

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## 1. Introduction

Palm oil is now a major commodity of world trade, and the oil palm is a leading source of vegetable oil. In the period 2025/2026, Indonesia's palm oil production experienced significant growth to 46.70 million metric tonnes [1], making Indonesia becomes 1st rank as palm oil producer in the world. Every year, an increase in plantation area, production, and productivity. Increased production of Crude Palm Oil (CPO) clearly led to an increase in waste production, too [2-3]. Besides that, palm oil plantations can produce Empty Fruit Bunches (EFB) as a side product around 25% (12.42 metric tonnes) and have only been used 10% [4], which have not been fully utilized.

The processing of Fresh Fruit Bunches (FFB) at a Palm Oil Mill (POM) produces the main products such as CPO and Crude Palm Kernel Oil (CPKO). However, it also produces by-products in the form of solid (EFB, shells, etc) and liquid waste (palm oil mill effluent, etc). In detail, EFB is the largest solid by-product produced from POM activity, which is about 17 - 23% of FFB [5]. EFB is one of the lignocellulosic biomass that is considered waste due to the abundance of its production. The waste produced by this industry can become a serious problem if there is not properly managed. In fact, almost 50% of EFB content is cellulose, which has not been used optimally. Therefore, a high percentage of cellulose shows the huge potential of EFB for cellulose-based products and high economic value [6-9].

However, the existing literature indicates that few studies have been reported on cellulose content in EFB and its applications. In fact, cellulose is the largest component in EFB [10] and is applied as the starting material for cellulose acetate [11-13], membrane [14], fashion [15-16], and biomaterials [17]. Some research have been performed on prosperous cellulose synthesis and its purification using eco-friendly reagents like formic acid, acetic acid, and alkaline. Their reports conclude that an alkaline reagent produces a higher cellulose yield than another. Also, it has been applied to bioplastic production. Many researchers reported that the synthesis of cellulose-based bioplastics has mechanical properties [18-19], but its value has yet to be explored for the required standards [20].

Cellulose is a biopolymer with great potential as a basic material for bioplastics due to its biodegradable properties. Cellulose can be used as the main material for plastic production, which positively impacts the environment compared to conventional plastic. However, conventional plastic has high chemical resistance, is easy to make in various shapes and sizes, has good elasticity, and is relatively cheap. On the other hand, bioplastics are considered to lack mechanical strength and durability. In order to increase the physical properties of bioplastics, the most common way is to add a filler such as starch or silica, and to add a plasticizer such as glycerin. Silica was chosen as a filler due to its non-toxic nature, having hydroxyl groups on its surface, and high surface area. Moreover, silica could be used as a filler to produce a fiber-reinforced material [21]. Their study showed that the addition of silica has highly improved the mechanical properties of the material. On the other hand, the effect of nano-silica filler on the mechanical characteristics of natural fiber polymer has also been investigated [22]. The results evidenced that the increment in nano-filler content proportionally improved the strength of the materials.

The main objective of the present research was to explore cellulose extraction from EFB and use it as a starting material for eco-friendly plastic with good mechanical properties. The alkaline reagent was used to extract the cellulose, which was then converted to nanocellulose using hydrolysis and sonication. Moreover, the effect of fillers on the bioplastic's mechanical behavior was also briefly investigated.

## 2. Experimental Section

### 2.1. Materials

EFB samples were collected from a mini factory in PTKI Medan, $HNO_3$, NaOH, $Na_2SO_3$, NaOCl, $H_2O_2$, $H_2SO_4$, $SiO_2$, Glycerin, and aquadest. All the reagents were used as received.

### 2.2 Pre-Treatment

EFB samples were soaked with detergent overnight and washed several times with water to remove oil, sand particles, and unwanted materials. The fibers were dried at room temperature and were cut into small pieces of about 2 cm, then mashed until fine fibers were obtained.

### 2.3 Extraction of Cellulose from EFB

75 g of fine fibers were hydrolyzed by adding 1L of $HNO_3$ 3.5% and then heated at 90 °C for 2 hours. The fibers were then delignified using a 750 mL mixture of NaOH 2% and $Na_2SO_3$ 2% (ratio of 1:1) at 50 °C for 1 hour. Delignified fibers were then collected after filtration and washed further with aquadest. Addition of 250 mL of NaOCl 1.75% for the bleaching process at 80 °C for 30 minutes and then washed using aquadest until neutral conditions were reached.

Purification steps were conducted by soaking the obtained material in 500 mL of NaOH 17.5% at 80 °C for 30 minutes, then soaking in $H_2O_2$ 10% at 60 °C for 15 minutes. The white insoluble solid was filtered, washed several times, and collected, and the yield (w/w) was calculated.

### 2.4 Synthesis of nano α-cellulose from α-cellulose

5 g of α-cellulose was dissolved in $H_2SO_4$ 45% and then heated at 45 °C for 45 minutes while stirred process was also applied. The white solid suspension was filtered and washed several times using aquadest. The final step was sonicated with a frequency of 20 kHz at room temperature for 1 hour.

### 2.5 Synthesis of Bioplastic

0.2 g of nano α-cellulose was dissolved in 1 mL NaOH 8%, 1% glycerin (of the sample weight) was added, and then silica concentration was varied (0, 10, 20, 30, 40, and 50%). All materials were stirred at 65 °C for 30 minutes. Homogeneous solutions were cast and then dried at 70 °C for 6 hours.

### 2.6 Characterization

Burker, German FTIR spectroscopy was used to elucidate the functional groups of the obtained cellulose. The wavelength was set in the range of 4000 - 500 $cm^{-1}$. The morphology of the cellulose was identified with SEM EVO MA 10 Carl Zeiss. Particle size distributions were analyzed using PSA from Corduan Technologies; the wavelength was set at 657 nm. The thermal decomposition of the obtained cellulose was characterized using DTA instrumentation. To determine the mechanical properties of bioplastics, a tensile strength instrument was used using ASTM-D638.

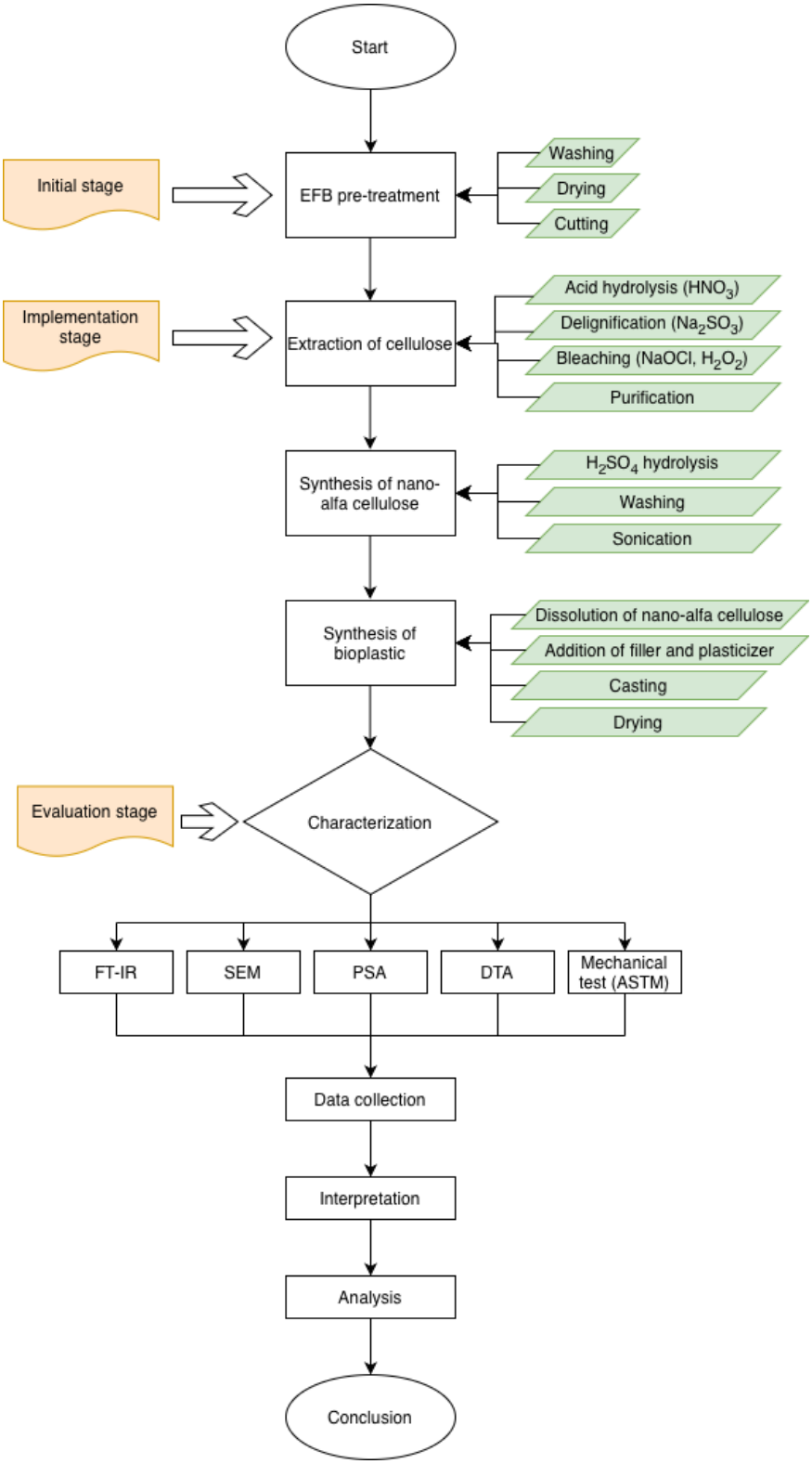


**Figure 1**. Flow chart of research

## 3. Results and Discussion

EFB is biomass from palm oil industries with a high content of lignocellulosic composition. Numerous studies have investigated the utilization of EFB to produce fiber, microcrystalline cellulose, and modified cellulose. Chemical treatment in cellulose extraction is very important in cellulose extraction. The combination of acid hydrolysis, alkaline delignification, and bleaching agents will enhance the quality and purity of extracted cellulose by removing the lignin content in EFB.

The effectiveness of the alkaline and bleaching treatments in extracting cellulose from EFB can be explained by the structural organization of cellulose within the EFB fibers. Cellulose fibers consist of two regions on its wall: crystalline and amorphous regions. It was observed that the NaOH concentration affected the yield of cellulose [23]. They reported that when EFB fiber is soaked in NaOH solution, cellulose becomes swollen, thus causing the outer layer and the amorphous region to burst out. NaOH solution breaks down the hydrogen bonding between the lignocellulosic components, which causes the lignin and lower molecular fractions to dissolve in an alkaline solution as a black liquor. However, not all the lignin was removed when using NaOH, thus NaOCl as a bleaching agent was also used to remove the remaining lignin after the alkali treatment. NaOCl enabled to break down the ether bond in the lignin structure due to its strong oxidant of hypochlorite natural behavior, as well as increasing the white brightness of the fibers.

However, increasing the NaOH concentration was found to decrease the yield because some of the cellulose chains would degrade during the treatment process. Finally, at the high NaOH concentration tend to interrupt some of crystallin region in cellulose (β- and γ-cellulose) and easily dissolve in the solution. Thus, this phenomenon will reduce the yield of cellulose.

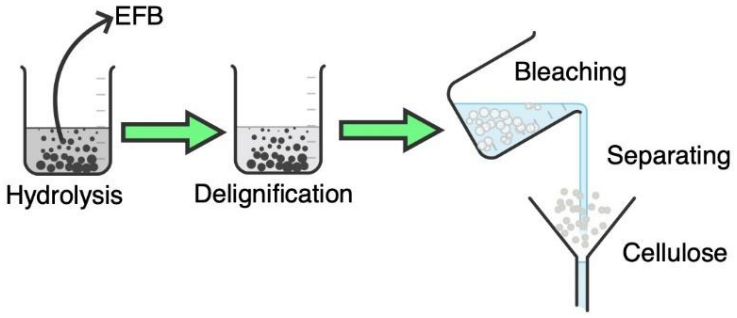


**Figure 2**. Schematic diagram of experimental procedure

Visual results from extracted cellulose were identified in white solid material and odorless. Cellulose was obtained through several stages, i.e., pre-treatment, hydrolysis, delignification, pulping, and bleaching. Those steps were applied to remove all lignin content adhered to EFB. In particular, the removal of lignin at the initial procedure played an important role in the quality of cellulose, such as attaining high purity and crystalline cellulose [24]. One of the indicators of successfully separating lignin from EFB fibers is the white color of cellulose (Figure. 3).

**Figure 3**. White solid cellulose extracted from EFB

To obtain cellulose with a higher degree of purity, the extracted cellulose was treated with 17.5% NaOH solution, resulting in an α-cellulose yield of 22.97%. The use of concentrated NaOH is a well-established method for cellulose fractionation because it selectively dissolves the alkali-soluble cellulose fractions, namely β- and γ-cellulose, while leaving the alkali-insoluble α-cellulose largely unaffected.

The α-cellulose yield of 22.97% obtained in this study indicates that the extraction and purification processes were effective in isolating the highly stable cellulose fraction from EFB. The preservation of α-cellulose during treatment suggests that the applied NaOH concentration was sufficient to remove undesirable cellulose fractions without causing significant degradation of the crystalline cellulose structure. Consequently, a cellulose product with relatively high purity was obtained, making it a promising precursor for the production of value-added materials such as nanocellulose, biocomposites, and biodegradable plastics.

**Table 1.** Physical properties of extracted α-cellulose from EFB

| No | Parameter | Annotation |
|---|---|---|
| 1 | Visual appearance | White |
| 2 | Phase | Solid/ Powder |
| 3 | Yield | 22.97% |

### 3.1 Fourier Transform Infra-Red (FTIR) spectroscopy

The chemical structure of the extracted cellulose was investigated using FTIR spectroscopy, a widely employed analytical technique for the identification of functional groups and structural changes in lignocellulosic materials. FTIR analysis is based on the interaction between infrared radiation and molecular bonds, where each functional group absorbs infrared energy at characteristic frequencies, producing a unique spectral fingerprint. The different compounds will also produce specific spectra compared to others [25-26].

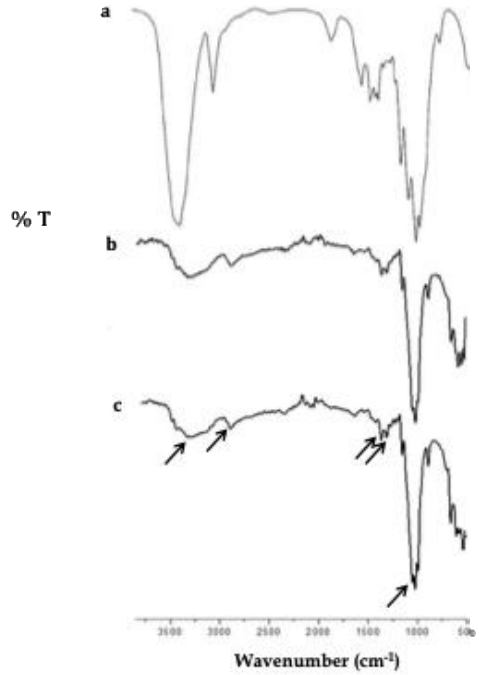


**Figure 4.** FTIR spectra of: a) cellulose standard, b) α-cellulose, and c) nano α-cellulose

The FTIR spectra show the transmittance broadening peaks at 3328 $cm^{-1}$, attributed to cellulose O-H group stretching vibration that associated with the extensive intra- and intermolecular hydrogen-bonding network within cellulose fibrils. The significant peak at 2800 – 2900 $cm^{-1}$ was associated with the stretching vibration of C-H methyl group from aliphatic groups. Another evidence is the bands at 1366 and 1312 $cm^{-1}$ indicate the bending vibration of -$CH_2$ and C-H of cellulose, respectively. These peaks are related to bending, deformation, and asymmetric stretching of β-(1-4)-glycosidic linkages. However, the peak at 1022 $cm^{-1}$ with high intensity was attributed to C-O stretching from the OCH-O-$CH_2$ cellulose framework, in particular vibration of the pyranose ring structure, which is a characteristic feature of cellulose [27]. These absorption bands serve as important indicators for confirming the successful extraction of cellulose from EFB. Refers to the data above, the FTIR spectrum among standard, α-cellulose, and nano α-cellulose is no different, which leads to the successful extraction of cellulose from EFB.

**3.2 Scanning Electron Microscopy (SEM)**

SEM images revealed that the extracted α-cellulose possessed a relatively compact and densely packed fibrillar structure with a fiber diameter of approximately 3.07 μm. This morphology indicates the successful removal of a substantial portion of non-cellulosic components, such as lignin and hemicellulose, during the delignification and bleaching stages, thereby exposing the cellulose microfibrils and promoting closer fibril aggregation. Similar observations have been reported for cellulose extracted from EFB, where chemical purification resulted in smoother fiber surfaces and more organized fibrillar arrangements due to the elimination of the lignocellulosic matrix surrounding the cellulose framework [28].

Following acid hydrolysis, a pronounced alteration in the fibrillar morphology was observed. The SEM images demonstrated partial rupture and fragmentation of the original cellulose framework, indicating that the acid treatment effectively disrupted the less ordered region of the cellulose structure. Acid hydrolysis preferentially attacks the amorphous domains of cellulose, which are more susceptible to chemical degradation than the highly ordered crystalline regions. Consequently, cleavage of glycosidic bonds within these amorphous regions leads to fibril shortening, defibrillation, and a reduction in fiber dimensions. The breakdown of the fibrillar network observed in the present study is consistent with previous reports describing the conversion of α-cellulose into nano α-cellulose through acid-mediated hydrolysis, where the removal of amorphous cellulose results in the extraction of smaller and more crystalline particles [29].

As a result of the hydrolysis process, the average diameter of the nano α-cellulose decreased from 3.07 μm to approximately 2.80 μm, confirming the effectiveness of the acid treatment in reducing fiber size. The reduction in diameter can be attributed to the progressive disintegration of cellulose bundles and the removal of interfibrillar amorphous material, which facilitates the separation of individual fibrils. Furthermore, the decrease in particle dimensions suggests an increase in the specific surface area of the cellulose, a characteristic that is highly desirable for applications involving adsorption, reinforcement, and advanced biocomposite materials. Previous studies on EFB-derived nanocellulose have similarly reported a significant reduction in fiber dimension following acid hydrolysis, accompanied by improved crystallinity and more compact fibrillar structures due to the preferential removal of amorphous cellulose fractions [29-30].

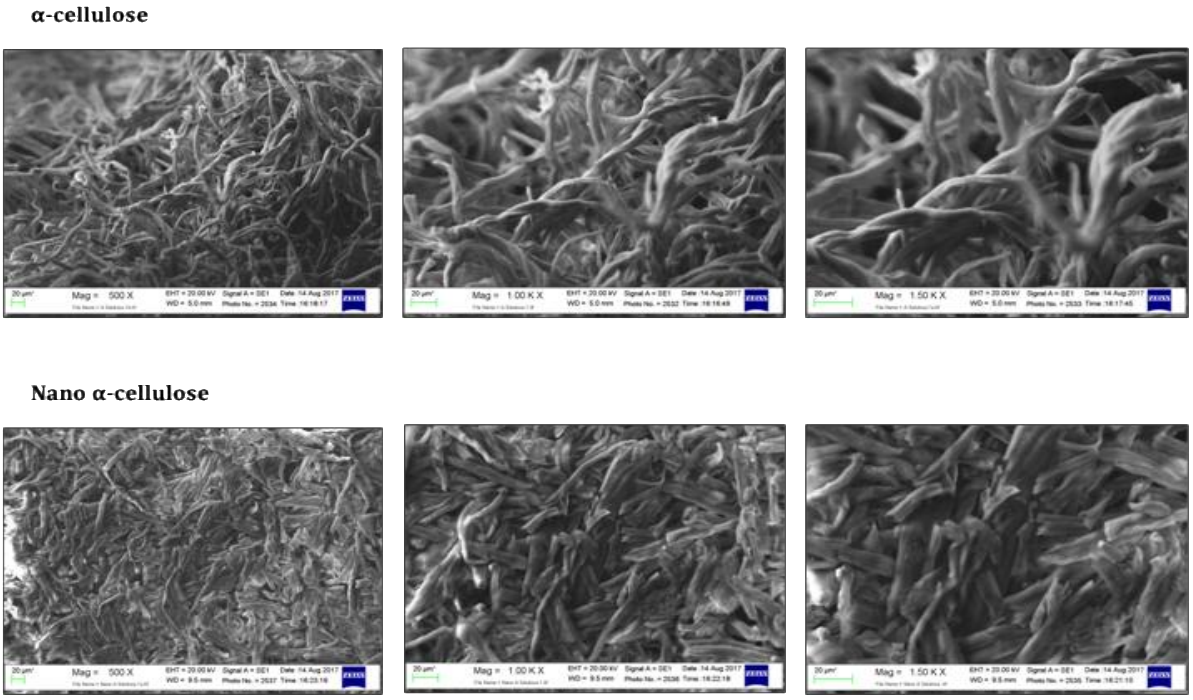


**Figure 5.** SEM images of α-cellulose and nano α-cellulose with varying magnification (500x, 1000x, and 1500x)

### 3.3 Particle Size Analysis (PSA)

PSA evaluates the dimension, size range, and distribution of particles in powders, liquids, or granular materials [31-32]. The PSA operates based on the principle of Dynamic Light Scattering (DLS). This technique enables the measurement of particle size and distribution across a wide range of solution concentrations. The measurement system assesses the Brownian motion of particles in solution using the DLS principle. Brownian motion refers to the random movement of particles in a solution resulting from collisions between particles [33-35].

In this work, the wet method was employed for particle size analysis, in which a dispersing medium is utilized to disperse the particles. This method was selected due to its higher accuracy compared to the dry method, particularly for samples in the submicron and nanometer range that tend to agglomerate. Dispersing the particles in a medium prevents agglomeration, ensuring that the measured particle size reflects individual particles. Furthermore, the results are presented as a particle size distribution, which is assumed to represent the overall condition of the sample. Accordingly, the particles were dispersed in aqueous solution to form suspended particles.

In Figure 6. shows that sharp peaks with symmetrical patterns indicate that the particle size distribution is even in all parts. The peak appears in the area with an average particle size of 380.79 nm. This finding is totally different from the SEM result (in micro-scale) due to the distinct measurement principles of the two techniques. SEM provides direct measurements of the physical dimensions of dry particles, whereas PSA estimates an equivalent spherical diameter based on particle scattering behaviour in a dispersed medium. Furthermore, differences in aggregation state and sample preparation contribute to the observed discrepancy. For irregularly shaped cellulose particles, PSA results represent the hydrodynamic or equivalent spherical diameter rather than the actual geometric dimensions observed in SEM micrographs [36-38].

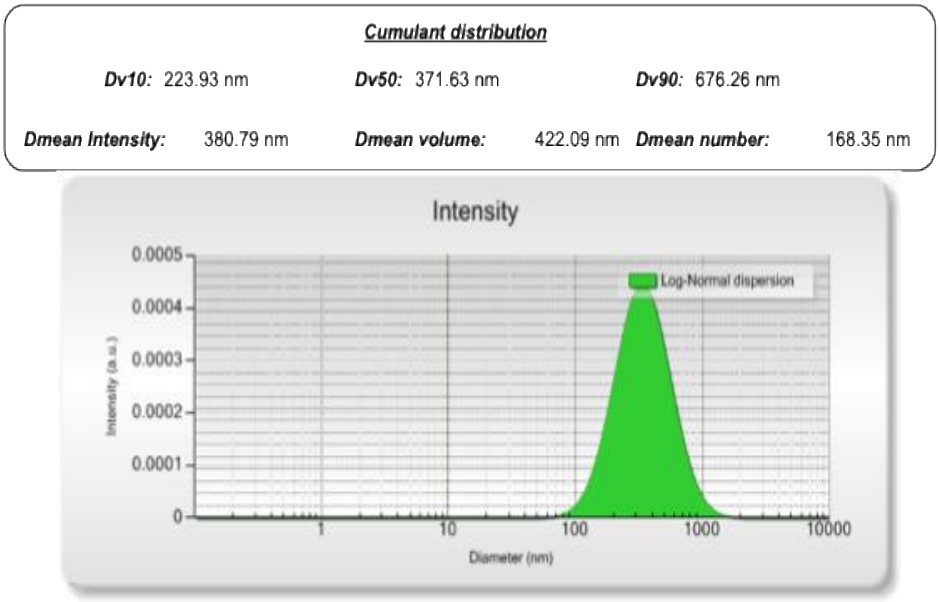


**Figure 6.** Particle size distribution of nano α-cellulose

### 3.4 Differential Thermal Analysis (DTA)

DTA is used to evaluate the thermal behavior of the materials by measuring the temperature difference between the sample and an inner reference during controlling heating. DTA enables the identification of thermal events and determines whether a process is endothermic, involving heat absorption (such as dehydration, melting, or bond dissociation), or exothermic, involving heat release (such as combustion or thermo-oxidative degradation). The resulting thermogram provides valuable information on thermal stability and decomposition of the material [39-40].

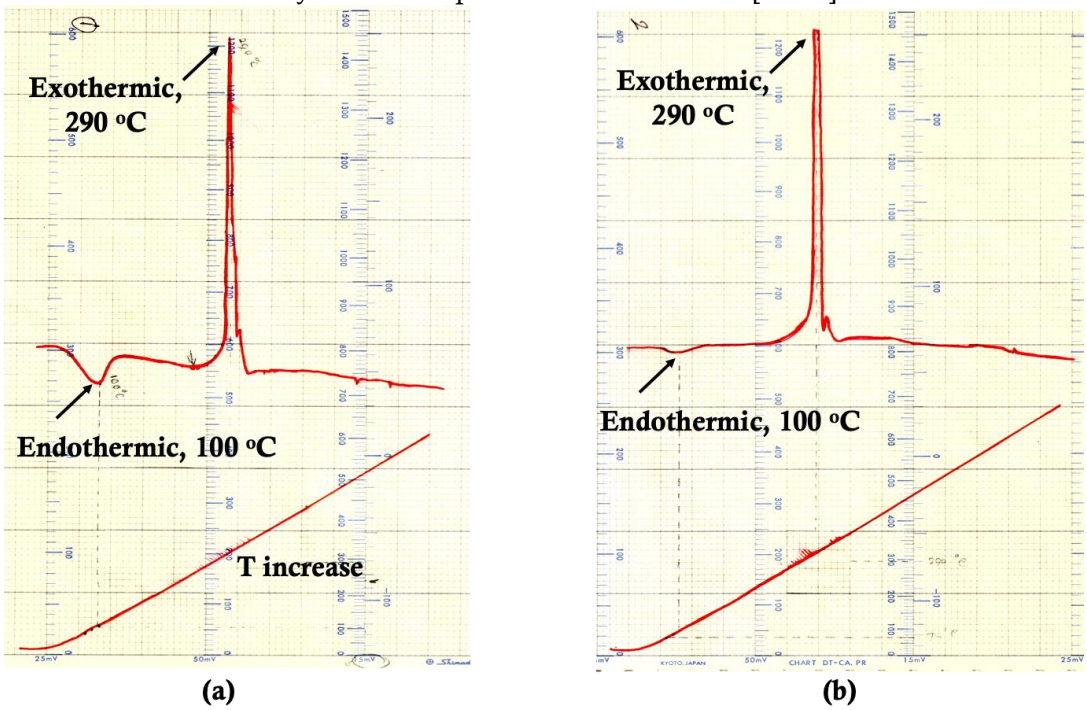


**Figure 7.** Thermograms of: a) α-cellulose and b) nano α-cellulose

Figure 7. shows the thermogram leading to a downward peak at 100 °C, indicating an endothermic process. This phenomenon can be explained as a decrease in temperature due to the materials absorbing heat during the vaporization of water molecules that are entangled inside the cellulose framework. As the temperature increases, the thermogram generates a significant upward peak at 290 °C, indicating that an exothermic process occurs. At this point, the material releases some heat due to the decomposition of the molecular structure of the cellulose. Thus, it can be concluded that both α-cellulose and nano α-cellulose have good thermal stability, which starts to decompose at high temperature. These findings are to be inline with review work [41]. They reported that the moisture content and other volatile compounds tend to evaporate at the lower temperature (<105 °C), whereas cellulose decomposes at higher temperatures (>220 °C).

### 3.5 Mechanical Test

Three kinds of mechanical tests were conducted to examine bioplastic properties: tensile strength (Figure. 8), elongation at break (Figure. 9), and modulus of elasticity (Figure. 10). Tensile strength measurement was used to determine the ability of bioplastics to withstand heavy load. Meanwhile, the elongation at break is an indicator of the flexibility of the bioplastic and is determined by the strain poin on the film when it breaks. On the other side, the modulus elasticity is the resistance value of the material when it is elastically deformed when some of force is applied.

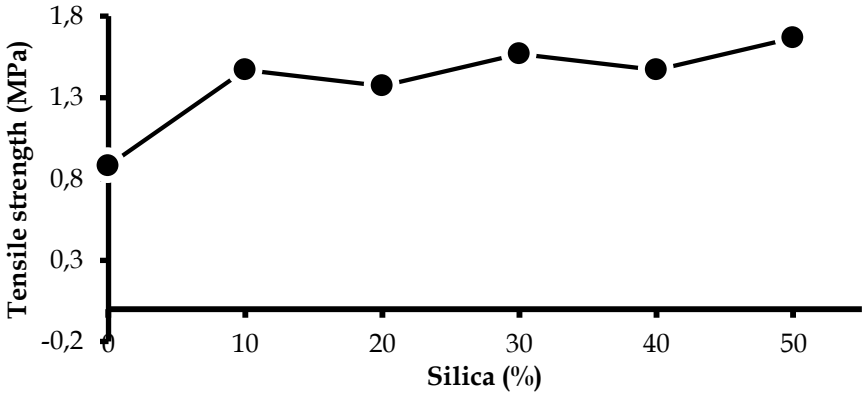


**Figure 8.** Effect of silica on tensile strength of bioplastic

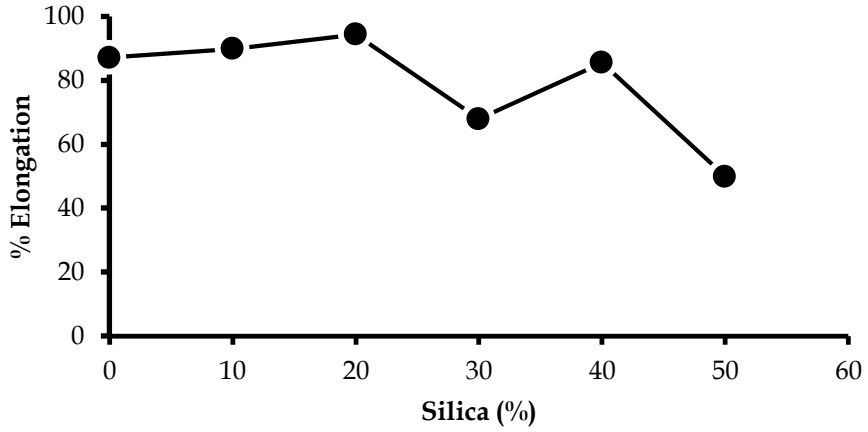


**Figure 9.** Effect of silica on elongation of bioplastic

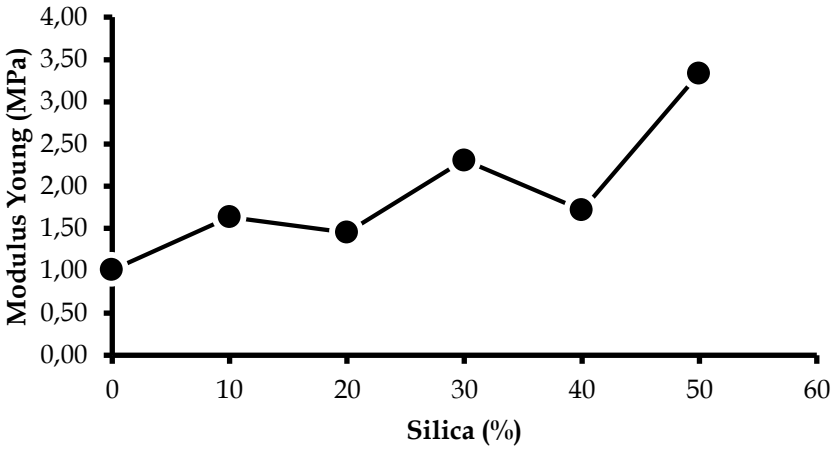


**Figure 10.** Effect of silica on modulus of elasticity of bioplastic

Moreover, the mechanical properties of bioplastics highly depend on their constituent, such as plasticizer agents or reinforcement compounds. The composition of bioplastics has an impact on its mechanical properties due to the physical interaction between constituent compounds. The addition of silica and glycerin to the bioplastic tends to increase the mechanical properties of the bioplastic.

ASTM-D638 was used to identify the tensile strength ability of bioplastic. We noted that the tensile value increases with increasing silica concentration. However, these tensile values are unsatisfied with the Indonesian National Standard (SNI) requirement, namely 24.7 - 302 MPa.

The addition of glycerin to the bioplastic formula could improve the elasticity of the materials. This phenomenon could be described due to the nature of glycerin's chemical behaviour, which can stretch the bond between molecules [18]. At 20%, silica holds the highest elongation value, indicating that bioplastics have good stretchability behavior with average elongation values above 80%. These values meet the Indonesian National Standard requirement of 21 - 220%, so it's assumed the obtained bioplastic can be used as a substitute for commercial plastic. To be in line with that, the Modulus Young values were increased by increasing silica concentration as a filler material for bioplastic production.

## 4. Conclusion

This study successfully demonstrated the extraction of α-cellulose from oil palm Empty Fruit Bunches (EFB) through a series of processes, including pre-treatment, hydrolysis, delignification, pulping, and bleaching, resulting in an α-cellulose yield of 22.97%. The purity of the extracted cellulose was further enhanced through treatment with 17.5% NaOH, which effectively removed β- and γ-cellulose fractions while preserving the α-cellulose structure. Subsequently, hydrolysis combined with sonication treatment was employed to reduce the cellulose fiber size and produce nano α-cellulose with improved physicochemical characteristics.

The extracted cellulose showed significant potential as a renewable raw material for bioplastic production. The incorporation of silica as a reinforcing filler and glycerin as a plasticizer contributed to the modification of the bioplastic properties and improved its flexibility and overall performance. Mechanical testing revealed that the elongation at break of the produced bioplastic met the requirements of the Indonesian National Standard (SNI), indicating satisfactory elasticity However, the tensile strength values remained below the commercial standard, suggesting that further optimization of the formulation and processing conditions is necessary.

Overall, the findings highlight the potential of EFB-derived α-cellulose as a sustainable and value-added biomaterial for bioplastic applications. Future studies should focus on improving interfacial compatibility among cellulose, silica, and the polymer matrix, as well as optimizing filler loading and

processing parameters to enhance tensile strength and broaden the commercial applicability of the resulting bioplastic materials.

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