

Preparation and characterization of physical properties of molded pulp from empty fruit bunches of oil palm



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Abstract. This research aims to determine the impact of pretreatment on empty fruit bunches (EFB) of palm oil fibers before the pulping process on the quality of the molded pulp produced. The study includes EFB fiber pretreatment processes, pulp production, pulp molding, physical characteristic analysis, and molded pulp performance evaluation. The pulp production process involves acid-base hydrolysis reactions. The produced molded pulp is done manually. Characterization of the molded pulp includes measurements of paper grammage, thickness, density, and moisture content. The characteristics of pulp produced from pretreated EFB fibers are as follows: an average grammage value of 415.48 g/m², paper thickness of 1.53 mm, water absorption capacity of 13.33-33.33%, density of paper of $0,026 \times 10^6 - 0,027 \times 10^6$ g/m³, and a smooth surface morphology. Meanwhile, the characteristics of pulp produced from without pretreatment EFB fibers include an average grammage value of 372.18 g/m², paper thickness of 0.77 mm, water absorption capacity of 20.34-43.21%, density of paper of $0,004 \times 10^6 - 0,008 \times 10^6$ g/m³, and a rough surface morphology due to pores on the fiber surface.

Keywords: pulp, molded pulp, non-wood, empty palm oil bunches, packaging

INTRODUCTION

The palm oil processing industry is one of the important sectors in the Indonesian economy. However, the growth of this industry also results in an increase in the amount of waste, including Empty Fruit Bunches (EFB). EFB are one of the biomass wastes generated from the production process of Crude Palm Oil (CPO). EFB are organic waste in the form of lignocellulose containing a high amount of carbon elements, such as hemicellulose, cellulose, and lignin, around 25.83%. [1][2].

Indriati & Elyani (2018) stated that for every unit of Fresh Fruit Bunches (FFB) of oil palm processed, 25%-26% of Empty Fruit Bunches (EFB) are produced, and from each unit of FFBs processed, 20%-22% of EFB are produced, which is nearly equivalent to the yield of Crude Palm Oil (CPO) [3]. If so, based on data from the Indonesian Palm Oil Association (GAPKI) in 2023, which states that CPO production in October 2023 reached 4,523 thousand tons, an increase of 9.2% from September 2023 production of 4,143 thousand tons. Based on data obtained from the United States Department of Agriculture (USDA), Indonesia retains its title as the world's largest palm oil producer, producing approximately 45.5 million metric tons (MT) of CPO in the 2022/2023 period. The numbers indicate that EFB are

non-wood biomass with high value for utilization. Chemical analysis results show that EFB contain cellulose, hemicellulose, and lignin. The cellulose, hemicellulose, and lignin content in EFB indicate that they are non-wood biomass with potential as raw material for molded pulp production. Several studies suggest that molded pulp can be utilized as environmentally friendly food packaging compared to packaging materials made from Polypropylene (PP), Polystyrene (PS), and Polyethylene (PE). [3][4]

The International Molded Fiber Association (IMFA) defines molded pulp as a single-use packaging material made from natural fibers. Molded pulp is specifically designed to have a shape with a sturdy structure and pressure resistance. The use of molded pulp as packaging offers several advantages, including being environmentally friendly, biodegradable, design flexibility, and ease of recycling. [5][6][7]. The technical process of making Molded Pulp Packaging consists of several stages: (a) Stage 1 – Mixing; the raw fiber material is soaked in water and mixed until it forms a pulp slurry with the desired consistency. Subsequently, additives such as sizing agents or fillers are added to produce the required functions [8] [9] [10] (b) Stage 2 – Forming: the pulp slurry is placed into a special designed mold equipped with a vacuum. At this stage, the molded



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pulp is in a wet condition, thus requiring drying, (c) Stage 3 – Drying: conventional drying processes use open-air drying racks, but nowadays automatic drying machines with temperature, time, and pressure controls are used, (d) Stage 4 – Pressing: aimed at smoothing the surface of the molded pulp. The goals of pressing include enhancing the mechanical properties of molded pulp, facilitating more efficient stacking and nesting for storage and shipping, and aiding in the formation of stronger bonds between fibers. Additionally, pressing aims to remove excess steam and prevent issues like delamination, which can significantly reduce product strength, (e) Stage 5 – Trimming, and (f) Stage 6 – Quality Inspection: this final stage involves the quality inspection of the produced molded pulp [11] [12][13] [14].

Currently, research for the development of molded pulp is intensively growing both in Asia, including Indonesia, and in other parts of the world. Innovations in molded pulp are continuously being developed through fundamental and practical research. Many researchers are exploiting various potential plant fiber sources, for example: pomace fruit fibers [15], rice straw, pineapple leaves, banana stem fibers [16], [17], [18], sugarcane bagasse [19], bamboo [9], pineapple peel, orange peel, hemp [20], and Empty Fruit Bunches of oil palm (EFB) [21].

The production of pulp using EFB fibers as raw material for molded pulp generally similar to wood fiber pulp production. EFB are first chipped into smaller pieces. These chipped EFB fibers undergo cooking, bleaching, washing, drying, and molding processes. Chipped EFB fibers are placed in a digester vessel along with chemicals such as soda or sulfate. The purpose of the cooking process is to dissolve lignin that binds cellulose fibers, enabling their separation. Cooking methods include Soda Pulping using sodium hydroxide (NaOH) solution and Sulfate Pulping (Kraft) using a mixture of sodium hydroxide and sodium sulfide (Na₂S). Besides soda and sulfate methods, other methods have been

researched for EFB pulp production such as Soda Anthraquinone, Alkaline Peroxide, Acetosolve, Soda-AQ Prehydrolysis, and Chemi-Mechanical methods. [22], [21],[23]

Typically, studies conduct EFB fiber pulping processes without pretreatment involving separating the inner and outer parts of EFB fibers and drying them to achieve a moisture content of 7-10%. Despite extensive research on EFB, particularly in pulp production, there is limited research on separating the inner and outer parts of EFB fibers and drying them to achieve a moisture content of 7-10%. Investigating the separation of the inner and outer parts of EFB fibers and drying them aims to remove pitch and impurities present in the fibers. It is suspected that pitch and impurities in the fibers affect the quality of the molded pulp produced. Pitch from EFB fibers can hinder the separation of cellulose fibers, requiring longer cooking times and higher concentrations of cooking chemicals.[24], [25], [26], [23] The time and concentration of cooking chemicals during the pulping process can reduce the cellulose content of EFB fibers, thereby lowering the quality of the molded pulp produced, such as tensile strength, density of molded pulp, and grammage of molded pulp. Therefore, the objective of this study is to determine the impact of pretreatment on EFB fibers before the pulping process on the quality of the molded pulp produced.

METHODOLOGY

Samples of EFB were obtained from community oil palm plantations. The chemical materials used were in the form of pro analysis (pa) from Merck, consisting of nitric acid, HNO₃, sodium hydroxide, NaOH, sodium perchlorate, NaOCl, and hydrogen peroxide, H₂O₂.

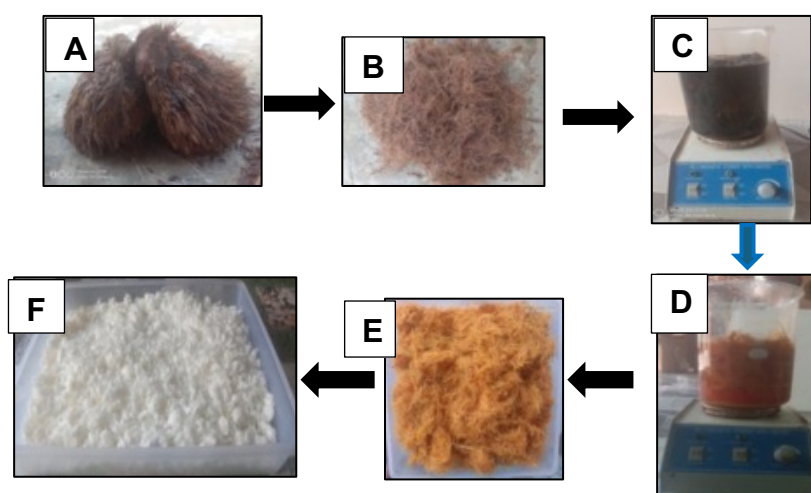


Figure 1. Schematic diagram of the pulping process from EFB without pre-treatment: A. EFB, B. EFB fibers, C. Acid Hydrolysis Process of EFB, D. Alkaline Hydrolysis Process of EFB, E. Fibers resulting from Alkaline Hydrolysis, F. Bleached EFB pulp



Figure 2. A set of equipment for molding pulp into sheets

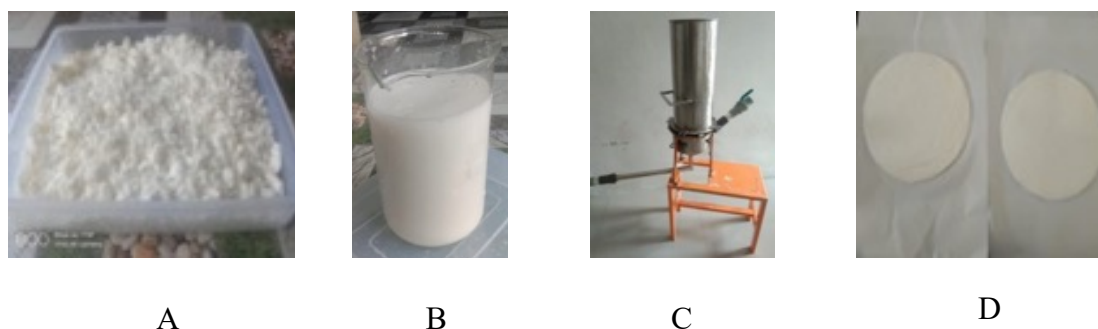


Figure 3. Schematic diagram of molded pulp preparation: A. pulp, B. slurry pulp, C. Handsheet, and D. molded pulp

Pretreatment of EFB

EFB are chipped into fibers approximately 1 - 2 cm in length. The chipped EFB fibers are dried by sun-drying directly under sunlight. Drying is carried out until the EFB fibers reach a moisture content of 7-10%. The dried fibers are then ground into powder with particle size of 300 mesh.

Pulp preparation

The preparation of EFB pulp was conducted using the same method as our previous research and has been modified [27]. 1000 grams of fibers were added to 5 L of 2.5% concentrated nitric acid solution (HNO_3 p.a) into an autoclave reactor. The steam process was carried out for 30 minutes (the temperature indicator on the device will show 126°C and pressure 0.15 MPa. The heating switch was turned off, and the cooling process continued until the temperature indicator reached zero, indicating the end of the steam process). The resulting product was then washed with distilled water until reaching pH 7. The fibers that had undergone autoclaving with nitric acid solution (HNO_3 p.a) were then added to 5 L of 2% sodium hydroxide solution, NaOH, and refluxed at a temperature of $70\text{-}80^\circ\text{C}$ for 30 minutes. The refluxed product was bleached with 5 L of 1.75% sodium perchlorate solution, NaOCl, at a heating temperature of $70\text{-}80^\circ\text{C}$ for 30 minutes. Washing was conducted with distilled water until reaching pH 7. After washing to pH 7, bleaching was performed again using 5 L of 10% hydrogen peroxide solution and heating at a temperature of $70\text{-}80^\circ\text{C}$ for 30 minutes. Washing was carried out using distilled water until the hydrogen peroxide odor

disappeared. Figure 1 depicts Schematic diagram of the pulping process from EFB.

Molded pulp preparation

The procedure for sheet preparation followed the steps in Fig. 3. The EFB pulp is dispersed with distilled water at room temperature until a pulp slurry is formed. Subsequently, the pulp slurry is poured into molds (Fig. 2) and dried in an oven at temperatures of $60\text{-}80^\circ\text{C}$.

Measurement of paper grammage

Test The Paper Grammage Test is conducted by weighing the material's weight and dividing it by the material's area (g/m^2). Each sample has the same area, which is 10×10 cm, and is weighed using an analytical balance. The Grammage Testing method refers to the SNI 140440:2006 and ISO 536-2010 standards for paper and cardboard grammage quality standards. The Grammage value is calculated using Equation 1 as follows:

$$\text{Grammage (G)} = (m / A) \times 10,000 \quad (1)$$

G : paper grammage (g/m^2)
m : mass of the test sample expressed in grams
A : area of the test sample (length x width)

Murement of paper thickness

The thickness of molded pulp is measured using the SNI 14-0435-1989 method: Paper and Cardboard Thickness Test. Samples are measured using a screw micrometer at five different points. The measured thickness values, still

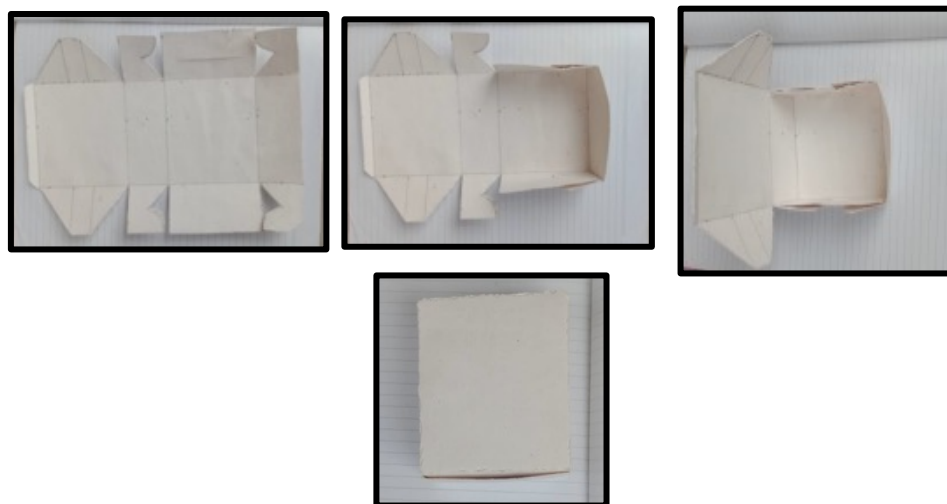


Figure 4. Molded Pulp of EFB

in millimeters, are divided by 10 for conversion to obtain thickness values in centimeters.

Sheet Density Determination

The sheet density value is calculated by dividing the paper grammage value by the paper thickness. Paper thickness measurement is conducted using a digital Vernier caliper. The measurements are performed under standard conditions (50% relative humidity and room temperature of 25°C).

Water uptake Capability

Molded pulp from EFB were first weighed and (m_0) soaked in distilled water for different time point at 1, 2, 3, 4, 5, 6, and 7 h. Upon taking out the sample pieces from the solution, the clean tissue paper was used to remove excessive water on the surface of sample and the second weight was recorded (m_1). The swelling ratio of each sample was calculated according to the Eq. (2).

$$\text{Swelling ratio (\%)} = [(m_1 - m_0) / m_0] \times 100\% \quad (2)$$

Sheet Morphology

The surface morphology characterization of Molded pulp from EFB were determined using scanning electron microscope (SEM) using JEOL EO JSM-6510LA Version 1.0

RESULTS AND DISCUSSION

Pulp from EFB

Pulp or paper pulp is a raw material in the papermaking process and its derivatives. Pulp or paper pulp mainly consists of cellulose. Pulp can be formed from wood or non-wood materials. Non-wood materials can be organic waste from plantations, such as EFB, bagasse, rice straw, corn cobs, and *etc* [16] [28] [1][29].[20]. The pulping process consists of acid hydrolysis, alkaline hydrolysis, and bleaching processes. Acid hydrolysis is carried out using 2.5% nitric acid. The purpose of the acid hydrolysis process is to separate hemicellulose, starch, wax, and fats found in the material. Alkaline hydrolysis uses a sodium

hydroxide solution, NaOH, to separate lignin (delignification) [30][21]. [30][31]. The bleaching process uses Sodium Perchlorate (NaOCl) and Hydrogen Peroxide (H₂O₂) bleach solutions. The stages of the EFB pulping process are shown in Figure 1.

Molded Pulp of EFB

The pulp is formed into sheets before being molded. The resulting molded pulp falls into the category of "Thermoformed" molded pulp, as the printing process involves simultaneous pressure and heat treatment. The production of molded pulp is carried out without the use of adhesive additives because the lignin in the pulp has been previously known to function as a thermoset binder. As seen in Fig. 4, the molded pulp of empty palm oil fruit bunches produced has a relatively smooth surface on both sides because lignocellulosic-based products molded at high temperatures will result in smooth-surfaced products.

Paper Grammage

Paper grammage is the measure of paper weight per unit area, usually expressed in grams per square meter (gsm). Grammage is one of the important parameters that determine the properties and quality of paper, including thickness, strength, and flexibility. The results of the paper grammage measurement for molded pulp from EFB are shown in Table 1.

The measurement results of grammage for molded pulp from EFB show significant differences between molded pulp produced from EFB with pretreatment compared to molded pulp produced from EFB without pretreatment. Paper produced from EFB with pretreatment tends to have a more consistent grammage and can be controlled as needed. This consistent grammage is achieved because the fibers used have been processed to be easier to form and more homogeneous. Referring to SNI 8218:2015 on Standard quality requirements for paper and cardboard packaging for food (table 2), pulp produced from EFB with pretreatment falls under the category of high grammage paper, ranging from 416 to 500 g/m².

Table 1. Examines the impact of pretreatment of EFB fibers on paper grammage

Treatment	Measurement	Mass (g)	Area (cm ²)	Grammage (g/m ²)	Average (g/m ²)
Pretreatment	1	4,1076	100	410,76	415.48
	2	4,1205	100	412,05	
	3	4,2363	100	423,63	
Without Pretreatment	1	3,8673		386,73	372.18
	2	3,7562	100	375,62	
	3	3,5421	100	354,21	

Table 2. Quality Requirements for Paper and Cardboard Packaging

No	Parameters	Unit	Quality Requirements			
			Low grammage	High grammage		
1	Grammage	g/m ²	26 – 210	225 – 300	312 - 400	416 – 500
2	Stiffness	mN.m	-	min 7	min 11	min 24
3	Bonding strength between sheets	J/m ²	-		min 100	
4	Tensile strength, AM	kN/m	min 1,6		-	
5	Water absorption capacity (Cobb ₆₀)	g/m ²			max. 50	
6	Heavy metal content	mg/kg				
	- Hg				max. 0,3	
	- Pb				max. 3	
	- Cd				max. 0,5	
	- Cr ^(VI)				Negative	
7	Formaldehyde content	mg/dm ²			max.1	
8	Pentachlorophenol content	mg/cm ²			Negative	
9	Total migration	mg/cm ²			Max.0,078	
10	Phthalate compound migration	mg/kg	-			
	- DBP (Dibutyl phthalate)					max.0,3
	- DEHP (Di(2-ethylhexyl) phthalate)					max.1,5
	- total (DIDP + DINP)					max. 9

NOTE 1 Tolerance for grammage values according to SNI 0440

NOTE 2 The symbol "-" indicates not required.

Source: SNI 8218:2015 on Quality Requirements for Paper and Cardboard Packaging

The grammage value of paper is influenced by raw materials from pulp and the addition of adhesive additives used. A study by [32] reported that the grammage value of corn stalk molded pulp without adhesive additives was 320 g/m². Meanwhile, research by [33] shows that the grammage value of bagasse molded pulp using PVA (polyvinyl alcohol) adhesive varies depending on the concentration used, namely 153.33 g/m² for 1% PVA and 300 g/m² for 5% PVA

Paper Thickness

The measurement of paper thickness utilizes the method specified in SNI 14-0435-1989 to test the thickness of pulp sheets, paper, and cardboard. The thickness of molded pulp is measured at five measurement points

(right-top, right-bottom, center, left-top, and left-bottom). The molded pulp is sized at 10 cm x 10 cm. The thickness of molded pulp from three measurements is indicated in Table 3.

Based on Table 1, it shows that the measured thickness values at five measurement points (right-top, right-bottom, center, left-top, and left-bottom) yielded different values. This indicates that the distribution of pulp dispersion during printing is not uniform on all sides. The uneven distribution during printing is caused by the manual mold used in making this molded pulp, thus the distribution of pulp slurry cannot be evenly spread on all sides.

Table 3. Measurement Results of Thickness Values for Molded Pulp of EFB

Treatment	Measurement	Thickness Values					
		Right – Top (mm)	Right-bottom (mm)	Left – Top (cm)	Left – bottom (mm)	Center (mm)	Average (mm)
Pretreatment	1	1,42	1,55	1,60	1,56	1,40	1,51
	2	1,85	1,52	1,60	1,20	1,50	1,53
	3	1,75	1,51	1,60	1,34	1,52	1,54
Without Pretreatment	1	0,86	0,78	0,75	0,96	0,67	0,80
	2	0,89	0,67	0,70	0,87	0,63	0,75
	3	0,80	0,79	0,74	0,82	0,60	0,75

Table 4. Grammage, Thickness, and Paper Density

Treatment	Measurement	Grammage (g/m ²)	Thickness (cm)	Paper Density (g/m ³)
Pretreatment	1	410,76	1,51	0,027 x 10 ⁶
	2	412,05	1,53	0,026 x 10 ⁶
	3	423,63	1,54	0,027 x 10 ⁶
Without Pretreatment	1	386,73	0,80	0,008 x 10 ⁶
	2	375,62	0,75	0,005 x 10 ⁶
	3	354,21	0,75	0,004 x 10 ⁶

The thickness value of the paper for molded pulp from EFB with pretreatment is higher compared to molded pulp from EFB without pretreatment. This difference is due to the varying grammage values of the EFB molded pulp. The resulting grammage has an impact on the paper's thickness; if the grammage is high, the paper will also be thicker. [23]

Paper Density

The density of paper is influenced by the grammage and thickness values. Paper density is obtained by dividing the grammage of the sample material by the thickness of the material. Technically, mass density is closely related to the bonding strength between fibers and the degree of fiber fibrillation in pulp, which will subsequently affect the printing process (print opacity). Generally, the higher the paper density, the thicker and stronger the paper tends to be. Paper density becomes an important factor in selecting the type of paper for specific applications, such as printing, packaging, or other industrial uses. The density values of molded pulp from Empty Fruit Bunches (EFB), both with pretreatment and without pretreatment, are shown in Table 4.

Water uptake capability

The degree of swelling or the ability of fibers to absorb water is highly influenced by the hydrophilic nature of the material immersed in a solution due to the differences in osmotic pressure between the interior and the surroundings of the solution [34]. In addition to the hydrophilic nature of the material, absorption is also influenced by porosity. High porosity in a material enhances its water absorption ability. Molded pulp is a paper material formed from cellulose fibers. Cellulose fibers with numerous OH groups can form hydrogen bonds [35]. The hydrogen bonds formed contribute to the hydrophilic nature of cellulose molecules [36]. The

measurement results indicate that the water absorption capability of molded pulp produced from Empty Fruit Bunches (EFB) with pretreatment is lower compared to molded pulp from EFB without pretreatment, as shown in Table 5. Additionally, the density and grammage of the paper produced from EFB molded pulp can also influence its water absorption capability. Generally, paper with higher density or grammage tends to have lower porosity and lower water absorption capacity. Testing results for paper density and grammage show that molded pulp from EFB without pretreatment has lower paper density and grammage compared to molded pulp from EFB with pretreatment.

Referring to SNI 8218:2015 on Standard quality requirements for paper and cardboard packaging for food (table 2), molded pulp produced from EFB, both with pretreatment and without pretreatment, meets the standard quality requirement of having water absorption capacity below 50%.

Surface Morphology

Figures 5A and 5B show the morphological imaging of molded pulp from EFB fibers at 1000x magnification, respectively for samples with pre-treatment and without pre-treatment. In Figure 5A, the molded pulp from EFB fibers with pre-treatment has a smoother morphology. Conversely, Figure 5B shows the molded pulp from without pre-treatment EFB fibers, which has many small holes on the fiber surface. These small holes indicate the

Table 5. Water Absorption Capacity of Molded pulp EFB

Treatment	Soaking time (h)	Water Absorption Capacity (%)
Pre-treatment	1	13,33
	2	23,07
	3	23,08
	4	25,0
	5	28,57
	6	33,33
	7	33,89
Without Pre-treatment	1	20,34
	2	23,44
	3	25,89
	4	30,67
	5	33,76
	6	38,87
	7	43,21

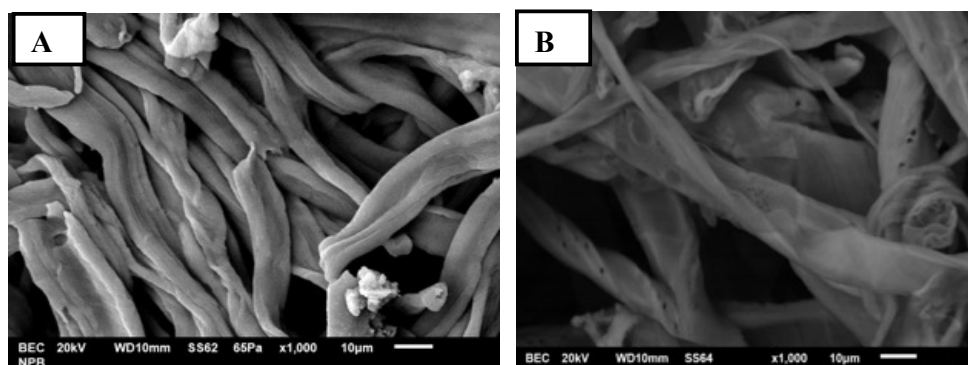


Figure 5. Scanning Electron Micrograph at 1,000x magnification of molded pulp from EFB: A. with pre-treatment, and B. without pre-treatment

presence of pores, which serve as water absorption media on the fiber surface. The more pores present on the fiber surface, the higher the fiber's water absorption capacity.

CONCLUSION

The research findings indicate that pretreatment of EFB before the pulp formation process can enhance the quality of the resulting pulp. This pretreatment includes the separation of fibers from the empty palm oil bunch nodes, drying the fibers to achieve a moisture content of 7-10%, and grinding the EFB. Such pretreatment maximizes the separation of cellulose from hemicellulose and lignin in the fiber during the pulping process. As a result, the pulp yield obtained reaches 40-50%, whereas the yield from untreated empty palm oil bunch fibers is only about 30-35%.

The characteristics of pulp produced from pretreated EFB fibers are as follows: an average grammage value of 415.48 g/m², paper thickness of 1.53 mm, water absorption capacity of 13.33-33.33%, density of paper of 0,026 x 10⁶ - 0,027 x 10⁶ g/m³, and a smooth surface morphology. Meanwhile, the characteristics of pulp produced from without pretreatment EFB fibers include an average grammage value of 372.18 g/m², paper thickness of 0.77 mm, water absorption capacity of 20.34-43.21%, density of paper of 0,004 x 10⁶ - 0,008 x

10⁶ g/m³, and a rough surface morphology due to pores on the fiber surface.

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