Studies on Properties of Thermomechanical Pulps from Sugarcane Bagasse and Oil Palm Empty Fruit Bunch

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Abbreviation

Abbreviation	**Description**
TMP	Themomechanical pulp
CTMP	Chemi-thermomechanical pulp
SB	Sugarcane bagasse
EFB	Empty fruit bunch
MDF	Medium density fiberboard
MLH	Mixed light hardwood
CSF	Canadian Standard Freeness
MOR	Modulus of rupture
MOE	Modulus of elasticity
IB	Internal bonding
TS	Thickness swelling
WA	Water absorption

Chapter 1 Introduction

1.1 The prospect of sugarcane bagasse and oil palm empty fruit bunch

Continued population growth will increase paper consumption and lead to an increased demand for fibers. Uncertainty regarding wood supply in some regions, due to restrictions on logging and inadequate forest resources, has caused increasing concerns over future fiber supplies (Wan Daud and Law, 2011). Given the shortage of wood resources, the Green World Campaign was initiated to decrease the use of trees as a source for pulp products, and places emphasis on the use of non-wood materials as new pulping resources (Reddy and Yang, 2005). Moreover, non-wood plant sources grow rapidly and are abundantly available in many regions (Shalatov and Pereio, 2006), and non-wood pulping capacities have increased two to three times faster than wood pulping capacities, worldwide (Rodriguez et al., 2008; Hedjazi et al., 2009; Jimenez et al., 2009; Moehieldin, 2014).

In Indonesia, sugarcane bagasse (SB) and oil palm empty fruit bunch (EFB) are abundantly available as residues from sugar and palm oil mills. SB is the residue remaining after the crushing of sugarcane for juice extraction, and the ratio of bagasse to stalk is around 30% by mass (Bilba and Arsene, 2008; Lee and Mariatti, 2008; Wirawan, et al., 2010). Dry bagasse comprises an upper layer consisting of a hard fibrous substance and an inner portion consisting of a soft material content called pith (Aggarwal, 1995); the upper layer accounts for 65% of the dry bagasse and the pith 35%. While EFB is the main product of oil palm fruit in oil palm industries, on average it represents around 22–24% of the total weight of fresh fruit bunches (Harsono et al., 2015). SB and oil palm EFB are non-wood resource fibers that will replace wood in pulping products in the future.

Many attempts have been made to convert SB and EFB into useful materials such as paper, composites, or fiberboards. SB particles have been evaluated as cement-composites (Aggarwal, 1995; Bilba and Arsene, 2008) and SB unrefined fibers have been used in the production of composites or particleboards (Cao et al., 2006; Luz et al, 2007; Xu et al, 2009; Filho et al, 2011; Tabarsa et al., 2011; Carvalho 2015; Fiorelli et al., 2015; Naguib et al., 2015). SB refined fibers combined with other fibrous materials as well as additional chemicals for pretreatment h ave also been used for manufacturing medium density fiberboard (MDF) (Lee et al., 2004; Ashori, 2009; Zare-Hosseinabadi et al., 2008; Adam et al., 2012). Moreover, the use of EFB as cellulosic pulp (Rodrigues et al., 2008; Jimenez et al., 2009; Rosnah et al., 2010; Wan Daud et al, 2013), and unrefined and refined EFB fibers in composites and fiberboards has also been investigated (Rozman, et al., 2001; Khalil, et al, 2010). Despite these previous studies, the details regarding the conditions for producing SB and EFB fibers suitable for paper and fiberboard production have not been clarified.

1.2 Thermomechanical pulping process

Mechanical pulping has two stages of processing. In the first stage, the fiber separation stage, chips are reduced to small wood particles, and in the second stage, the fiber development stage, a papermaking pulp is produced (Karnis, 1994; Gorski et al, 2010). Among the mechanical processes, thermomechanical pulping (TMP) is the most important process in the production of newsprint (Wu et al, 2004), increasingly in higher grade paper (Li et al., 2010), and also the critical step in the manufacturing of MDF (Hua et al., 2012), even though the mechanical pulp fibers retain almost all lignin from wood, which contributes to their high yield and high bulk properties (Li et al, 2010). The main effects of TMP or refining were found to be internal fibrillation, external fibrillation, and fiber shortening. Internal fibrillation increases fiber flexibility and collapsibility, and improves fiber-to-fiber contact in the paper sheet (Rusu et al, 2011). With the primary wall of the fiber partially removed, the secondary wall is in direct contact with water and subsequent swelling consequently improves the flexibility of the fiber (Samariha, 2011). The refining process, equipped with refining pressure, is a very important determinant of the physical and mechanical properties of refined fibers (Xing et al, 2008). In this process, the preheating retention time significantly affects bending strength (Xing et al., 2007). However, the strength properties of pulps produced via mechanical pulping are generally inferior to those of pulps produced via soda pulping (Hosseinpour, 2014; Zhai and Zhou, 2014).

1.3 TMP process for non-wood materials

TMP processes have been used for various non-wood materials including SB, oil palm EFB, bamboo, kenaf, canola, wheat, and rice straw. A mechanical process with a 0.10 mm refiner gap under atmospheric conditions used for oil palm EFB pretreated with 2% NaOH at 121°C for 2 h exhibited maximum tensile and tear indices strengths (Harsono et al., 2015). Oil palm EFB treated with caustic soda was refined using four stages under atmospheric refining to obtained suitable fibers and results indicated that the higher caustic concentration gave better properties (Wan Daud et al., 2013). Khalil et al. (2010) investigated the combination of EFB and rubberwood fibers for producing MDF using various pressures. This investigation indicated that the combination of EFB:rubberbood at a ratio of 20:80 showed higher mechanical and physical properties than other ratios. Oil palm trunk has also been investigated for fabricating MDF at a number of pressures and preheating times, and it was shown that the mechanical property value increased significantly with increased refining pressure and preheating time (Ibrahim et al., 2013). The use of wet- and dry-stored bagasse to produce MDF using an atmospheric refiner and subsequently a pressurized refiner at 0.8 MPa for 4–6 sec pre-heating time have been investigated. From these studies, it was found that dry-stored bagasse had a slightly higher performance compared with that of wet-stored bagasse (Adam et al. 2012). Additionally, wet- and dry-stored bagasse used in the production MDF using the pressurized refiner at 0.6 and 0.8 MPa revealed that wet bagasse had superior mechanical properties than dry-stored bagasse (Zare-Hosseinabadi et al, 2008). The combination of bagasse, bamboo, and tallow tree for MDF using an atmospheric condition refiner with a 0.13 mm disk gap has also been examined (Lee, et al., 2004; 2006). The kenaf bast and core fibers produced from the refiner with a 0.36 mm plate gap and a rotating speed of 4000 rpm with various pre-heating times and pressures for MDF panels gave results indicating that the kenaf core MDF panels had significantly lower permeability (Nayeri et al., 2014). Fibers for MDF were also produced from kenaf bast and kenaf core fibers by a pressurized refiner with three levels of pressures and for 3 and 5 min. It was exhibited that the higher pressure and pre-steaming temperature resulted in ideal mechanical and physical properties (Aisyah et al., 2013). The TMP process employing a 0.508 mm refiner gap and presteaming at 0.62 MPa for 5 and 10 min for rice straw pretreated with oxalic acid resulted in a product with good mechanical strength (Li et al., 2013). The MDF produced from the combination of bamboo and rice straw which was defibrated using a laboratory type defibrator with pressurized steam at 0.75 MPa and 165°C for 2.0 min revealed that no significant difference was found between the roughness values of panels made from the two individual types of raw materials (Hiziroglu et al., 2008). Yousefi (2009) produced MDF from canola straw as a bio-waste resource using a pressurized refiner at 0.8 MPa and 170°C, and exhibited that steaming time had a significant effect on mechanical properties.

1.4 Objective of this study

The utilization of pressurized thermomechanical pulp for preparing paperboard and MDF at optimum conditions have been examined. Many parameters must be considered to produce wet, half-dried, or dried fibers, which are suitable for paperboard and MDF.

The first objective of the study (Chapter 2) is to improve fiber properties through chemical pretreatment and pressurized refining using a thermomechanical refiner for the fabrication of SB and EFB paperboard and MDF, and to determine the conditions for obtaining suitable fibers.

The second objective of the study (Chapter 3) is to modify the operation of a laboratory pressurized TMP refiner to obtain dried fibers from SB and EFB for the fabrication of MDF under the suitable conditions.

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Chapter 2 Properties of Thermomechanical Pulps from Sugarcane Bagasse and Empty Fruit Bunch for Paperboard and Medium Density Fiberboard

2.1 Introduction

In 2014, Indonesia produced 2.8 million tons of cane sugar from 36 million tons of sugarcane (*Saccharum officinarum*) in 0.47 million ha of harvested area (Statistic Indonesia Bureau, 2014). The production of sugar from sugarcane yields the fibrous by-product sugarcane bagasse (SB), which is typically combusted in furnaces to produce steam for power generation. Because of this, SB is one of the most useful agricultural biomasses in that region. Indonesia is also the largest producer of palm oil in the world. In 2013, approximately 26 million tons of crude palm oil was produced, and about 30 million tons of empty fruit bunches (EFB) are produced each year from the oil palm (*Elaeis guineensis*) (Harsono et al., 2015, 2016).

Researchers have recently studied SB and EFB for their efficient use in various applications such as bio-ethanol, particleboards, and plant media. These studies were partially motivated by the fact that SB and EFB are fully biodegradable, light, abundant and renewable raw materials in high supply (Cardona et al., 2010; Das et al., 2014; Singh et al., 2013). Both unbleached and bleached SB chemical pulps obtained by soda, soda anthraquinone (AQ) and alkali sulfite-AQ (AS-AQ) cooking had tensile and tear indices greater than 50.0 N.m/g and 5.5 $mN.m^2/g$, respectively (Khristova et al., 2006; Banavath et al., 2011; Agnihotri et al., 2010). Of these, the highest tensile index was obtained by AS-AQ cooking (Hedjazi et al., 2008), and the quality of corrugated board materials made using a neutral sulfite semi-chemical SB pulp was investigated (Khakifirooz et al., 2013).

While there have been some reports on properties of fiber particles and mechanical pulps prepared under atmospheric pressure from non-wood fibrous materials, the properties of thermomechanical pulp (TMP) obtained specifically from SB and EFB of oil palm have not been clarified. Xavier et al, (2013) studied the bending stiffness component of medium density fibeboard (MDF) panels from mixtures of SB particles (unrefined fibers) and eucalyptus refined fibers. From these, they made particleboard by dry process but did not clarify the fiber length fractionation. Belini et al, (2011) studied panels made from mixture of SB particles (unrefined fibers) and eucalyptus refined fibers, using panels dry process to make the panels. Hoareau et al, (2006) studied fiberboard based on SB lignin and fibers, focusing on the SB fibers impregnated with some chemical and fiberboard using SB lignin to substitute of phenol, but they did not detail the fabricating process of the fiberboard.

Despite these previous studies, the conditions for producing SB and EFB fibers suitable for MDF are not known. Fibers for MDF are typically prepared by using a disk-refiner under pressurized steaming conditions at 160–175 ºC, which is similar to how paper-grade TMP is made. However, MDF fibers are directly dried immediately after refining followed by blending with adhesives. The main purpose of TMP processing with wood is to separate the fibers to make them usable for papermaking (Illikainen, 2008; Kang et al., 2006). In the TMP process, pressurized steam is applied before and during refining to raise the wood temperature, softening the lignin in the process. When the temperature is high enough to softens the lignin, but not so high that the lignin spreads over the fiber surface, the fibers can be torn from the matrix in such a way that the secondary cell wall layers (S1 and S2 layers) are exposed. In the production of chemi-thermomechanical pulps (CTMP), lignin is softened by chemical treatment and increased temperatures.

Fibers prepared from reed and wheat straws, and the MDFs obtained from such fibers, have been previously studied using a pressurized refiner (Han et al., 2001). In these experiments however, the refiner plate gap was 0.37 mm. The wide refiner gap implies that fibers with the desired properties cannot be produced. The properties of wheat straw and soybean straw fibers, and their resulting MDFs, have been previously investigated using an atmospheric refiner (Ye et al., 2007). Here, the fibers were prepared at atmospheric pressure and then collected by a wet blowing method. Properties of MDF produced from rice straw fibers obtained using a pressurized refiner (plate gap: 0.508 mm) and a wet pulp blowing method have been studied elsewhere (Li et al., 2013), but fibers with the desired properties cannot be produced in this instance. Elsewhere, wheat straw fibers were studied using a fully equipped pilot plant to produce fibers by a pressurized refiner, with a continuously drying method and a continuous mixing of resins, and the properties of these fibers and prepared MDFs were investigated (Halvarsson et al., 2008). When clarifying the properties of fibers used as paperboard and MDF materials, it is important to have a refiner plate gap on the scale of a single fiber width, appropriate pressure and temperature conditions for refining, and to select a dry blowing method that provides low-moisture fibers for MDF material. From this viewpoint, the previous study using wheat straw (Halvarsson et al., 2008) is important; however, there are no studies examining the use of SB and EFB in this manner.

The background of this study seeks to expand the usage of SB and EFB, waste materials generated in the sugar and palm oil industries, by identifying the conditions needed to make fibers suitable for use in products such as paperboard and MDF.

This study was conducted to improve the fiber properties through chemical pretreatment and pressurized refining with a thermomechanical refiner for fabricating SB and EFB paperboard and MDF, and the conditions for obtaining suitable fibers were determined.

2.2 Materials and methods

2.2.1 Materials preparation

The SB and EFB used in this study were obtained from PT. Madukismo in Yogyakarta, Indonesia and PT. Perkebunan Nusantara VIII in Bogor, West Java, Indonesia, respectively. SB was washed twice manually and dried in direct sunlight to a moisture level of 8–10%. Dried SB was cut into 0.5–2.0 cm by a shredding machine, while EFB was cut into 0.5–4.0 cm by a laboratory disk mill. Based on original shapes of SB and EFB, materials-cut dimensions are not specific other than for length. Wood chips of mixed light hardwoods (MLH) for MDF provided by Hokushin Co., Ltd. Kishiwada, Osaka, Japan, and a softwood (Larch: *Larix leptolepis*) obtained from the Agricultural and Forestry Research Center of University of Tsukuba, Yatsugatake Forestry, Kawakami, Nagano, Japan were used for comparison, with dimensions of 2 cm × 2 cm × 0.4 cm (length × width × thickness).

2.2.2 Chemical pretreatment

Materials (300 g as oven-dried weight) were soaked with water containing 0–4 wt% Na_2SO_3 or 2 wt% NaOH (based on oven-dried weight), using an autoclave at 121 ºC for 2 h. Due to the bulkiness of the fibers, the liquid to fiber ratio was 7 L/kg, while the liquid to wood ratio was 4 L/kg. After pretreatment, the yield loss and pH of the liquid were determined.

2.2.3 Thermomechanical pulping

The pretreated materials were treated under pressurized steaming conditions using a laboratory pressurized single disk refiner with a refiner plate (Type J) 305 mm in diameter (model BRP45-300SS manufactured by Kumagai Riki Kogyo Co., LTD., Nerima, Tokyo), equipped with a steam boiler (model SU-200 supplied by MIURA Co., LTD., Matsuyama, Ehime). The temperature of the refiner was 140 or 165 ºC, with supplied steam pressures of 0.5 and 0.7 MPa respectively. The temperature was increased over a 10 min span, and the refiner was held at the maximum temperature for 2 min. Refinement was conducted at a rotation speed of 3000 rpm. The disk clearance (plate gap) of the refiner was adjusted to 0.10 mm based on previous studies (Harsono et al., 2015). A smaller gap in the grinder produces more external fibrillation in the fibers of non-woody materials, which could benefit the physical properties of the pulp (Kang et al., 2006). The second refining (beating) was conducted using a PFI mills (ISO 5264-2:2011) at 10000 revolutions to make fibers suitable for fiberboard based on conditions used previously (Harsono et al., 2015).

As fibrous materials, SB and EFB have certain difficulties when used as supply materials for feeding processes, compared to wood chips. To improve their feeding efficiency in screw conveyors, pressurized steam was blown in a controlled switching manner to push these materials into the disk region. Two types of pulps were obtained depending on the blowing method used, labeled as "half-dried pulp" with 32–55% solid contents (SC) and "wet pulp" with SC < 10%. For the half-dried pulp, the refined fibers were vented from the refining chamber through a blow line into a blow box consisting of four sides with 60 mesh wire (250 µm opening), while for the wet pulp the fibers were discharged into a blow tank.

2.2.4 Evaluation of pulp properties

Classification of the refined fibers was conducted according to a pulp test method with a Bauer McNett Fiber Classifier No. 2593 using 1180, 600, 300, 150 and 75 µm opening screens (14, 28, 48, 100, and 200 mesh, respectively) at the School of Light Industry, Zhejiang University of Science and Technology. The refined pulp was further treated with a PFI mill at 5000 to 12500 revolutions according to ISO 5264-2:2011. Forming hand sheets for physical tests of pulp followed the ISO 5269-1:2005 standard. The physical properties, as well as the tensile and tear indices of the handsheets, were determined according to ISO 1924-2:2008, 1974:2012, and 5270:2012 standards. The length and width of the pulps were also determined using a Lorentzen-Wettre fiber tester CODE 912. These experimental design are summarized in **Table 2.1**.

Table 2.1 Experimental design

Material	Pretreatment				Refining [a]				PFI (rev.)
	Chemical	Dosage (%)	Temp. (ºC)	Time (h)	Clearance (mm)	Temp. at press. (ºC at MPa)	Time (min)	Blowing method	
SB	Na_2SO_3	0	121	0	0.10	40 at 0	2	Half-dried	5000
EFB	NaOH	2		2		140 at 0.5		Wet	7500
MLH		4		3		165 at 0.7			10000
Larch									12500

[a] Rotation speed: 3000 rpm

2.2.5 Chemical analysis of materials

Materials were milled with a Wiley mill and sieved to retain particles 40–80 mesh in size. These were stored at room temperature and air-dried, and Soxhlet-extracted with acetone. The contents of the acid-insoluble lignin (Klason lignin), acid-soluble lignin and ash were determined using TAPPI Test Methods T 222 om-15 and T 211 om-02, and the amounts of glucose and xylose in the acid hydrolysate were determined using ion chromatography according to previously published procedures (Harsono et al., 2016).

2.2.6 MDF fabrication from EFB and the properties

EFB TMP with approximately 92% solid content was prepared according to a modified method reported in Chapter 3 of the thesis, and MDF was fabricated with 5% methylene diphenyl diisocyanate, 0.8% polyol, and 0.5% wax. The mixture was cold-pressed and hot-pressed at 180 ºC and 3 MPa and at 0.5 MPa for degassing. A piece of MDF in dimensions of 350 × 350 × 2.7 mm^3 (after polishing) with a target density of 780 kg/m^3 was conditioned at 20 ºC and 65% RH for one week, after which the density, modulus of rupture (MOR), modulus of elasticity (MOE), and internal bonding (IB) were determined. Thickness swelling (TS) and water absorption (WA) after immersion in water at 20 ºC for 24 h were determined.

2.3 Results and discussion

2.3.1 Chemical compositions of SB and EFB raw materials

The lignin and glucan (cellulose) contents of the raw materials were 25.0% and 41.2% for SB, and 29.6% and 35.1% for EFB, respectively (**Table 2.2**).

2.3.2 Effect of pretreatment on yield loss

We expect softening and swelling effects on cell walls of these materials after chemical pretreatment. Most of the lignin and cellulose do not react under these mild conditions, except for a small extention of lignin sulfonation with Na_2SO_3 pretreatment, because the conditions of these chemical pretreatments are not so strong compared to those of chemical cooking.

As shown in **Table 2.3**, increasing the Na_2SO_3 dosage causes yield loss. Increased alkali dosage also caused decreased chemi-mechanical pulp yield of bagasse and canola straw (Khakifirooz et al., 2013; Fatehi et al., 2011). This yield loss is caused by reduced amounts of hemicellulose, organic acid and ash (Harsono et al., 2015a). Soaking these materials in a 2% NaOH solution significantly affected the properties of tropical bamboo mechanical pulp (Ashaari et al., 2010).

2.3.3 Properties of chemical pretreated thermomechanical pulp for paperboard

2.3.3.1 Effect of chemical pretreatment on pulp properties

The addition of 2% Na_2SO_3 or 2% NaOH to pretreatment had a positive effect on the properties of EFB refined pulp, but had a negative impact on the properties of the SB pulp (**Table 2.4**). When pretreated with 2% NaOH, the tensile and tear indices of the EFB pulp improved to 23.1 N·m/g and 5.46 mN·m^2/g, respectively. It has been previously reported that soaking wood with certain chemicals can adversely affect its properties (Hillis, 1984). The SB pulp with a chemical-free pretreatment had the highest tensile and tear indices with values of 22.0 N·m/g and 3.82 mN·m^2/g, respectively. SB with a chemical-free pretreatment might be sufficient to soften the lignin in fibers. On the other hand, under the same pretreatment and refining conditions, the EFB pulp had tensile and tear indices of 14.6 N·m/g and 3.61 $mN \cdot m^2/g$, respectively. TMP made from SB exhibited higher tensile and tear indices compared to EFB pulp with a chemical-free pretreatment.

The disadvantage of extremely low strength properties was not observed for SB and EFB pulps compared to the MLH and Larch wood pulp control samples (**Fig. 2.1**). In addition, the ISO brightness values of the SB and EFB pulps were similar to those of the Larch wood and MLH pulps, respectively.

Table 2.2 Chemical compositions of SB and EFB raw materials

	Acid-insoluble (soluble) lignin (%)	Glucan [a] (%)	Xylan (%)	Other sugars [b] (%)	Extractive [c] (%)	Ash (%)	Other organics [d] (%)
SB	23.9 (1.1)	41.2	18.5	2.4	1.3	1.6	10.0
EFB	26.7 (2.9)	35.1	19.8	1.9	5.4	5.9	2.3

[a] As polymer of glucose

[b] As polymer of arabinose, galactose and mannose

[c] Acetone extractives

[d] Organic acids and other extractives

Table 2.3 Effects of chemical pretreatment on liquid pH and yield loss

Material	Chemical	Dosage (%)	After pretreatment at 121 ºC for 2 h	
			Liquid pH	Yield (%) [a]
SB	-	0	4.13	96.2
	Na_2SO_3	2	5.27	95.7
	Na_2SO_3	4	5.45	94.2
	NaOH	2	6.07	95.9
EFB	-	0	6.40	94.9
	Na_2SO_3	2	6.60	94.8
	Na_2SO_3	4	6.90	93.5
	NaOH	2	8.12	94.7

[a] Yield after pretreatment, based on raw materials

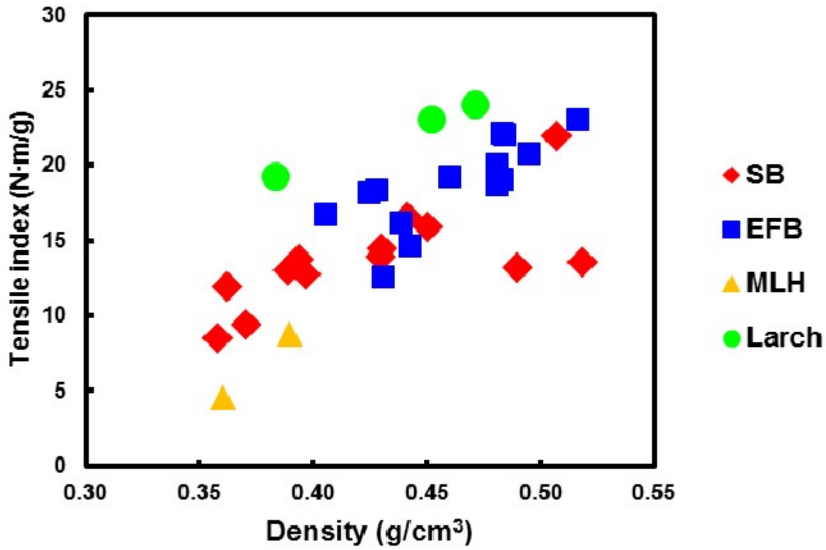

Fig. 2.1 Correlation between density and tensile index of paper boards

2.3.3.2 Effect of post-refinement blowing methods on pulp properties

Handsheets of the half-dried SB and EFB pulps had slightly higher tensile and tear indices compared to their respective wet pulps (**Table 2.4**). The results of fiber fractionation (**Table 2.5**) show that SB and EFB half-dried pulps had smaller ratios of the largest fiber fractions (> 1180 μm opening) than the wet pulps. Increasing the contents of the smallest fine (150 μm opening >) fractions is expected to increase the density and tensile strength of SB and EFB pulp sheets. Fines from chemical pulps were previously demonstrated to increase the strength of fiber-to-fiber bonds, enhancing the density as well as the tensile strength of pulp sheets (Marie et al., 2008). This fiber fractionation can be explained by effects from the pretreatment and refining conditions. SB or EFB pulps with no chemical pretreatment had a larger ratio of the smallest (150 μm opening >) fraction compared to other SB or EFB pulps. The addition of these chemicals softens the fibers and suppresses the formation of the finer structures. Unfortunately, we found that the refining temperature sometimes decreased to approximately 120 ºC during refining due to the repeated pressurized steam flow used to push the materials into the disks.

It has been reported that the differences between sheet properties of hardwood and softwood pulps arise not only from the average fiber length but also from the fine fiber characteristics (Kamijo et al., 2015); the densities and tensile indices of handsheets could be improved by addition of fine fractions to the pulps (Kamaluddin et al., 2012). Compared with the results of fiber characterization from mill MLH pulps, SB and EFB pulps having smaller ratios of long fiber fractions (> 1180 μm) and containing shives were obtained.

Table 2.4 Effects of chemical pretreatment on physical properties of thermomechanical pulps

		Pretreatment [a]	Refining [b]	Pulp properties [c]					
	Sample Name	Chemical dosage	Blowing method	CSF (mL)	Fiber length [d] (μm)	Density (g/cm^3)	Tensile index (N·m/g)	Tear index ($mN·m^2/g$)	Brightness (% ISO)
SB	S1	None	Half-dried [e]	215	550	0.507	22.0	3.82	32.3
	S2	2% Na_2SO_3	Half-dried	388	449	0.429	14.0	2.77	37.6
	S3	2% NaOH	Half-dried	415	542	0.394	13.7	3.00	36.6
	S4	2% NaOH	Wet	219	517	0.519	13.6	2.73	35.2
EFB	E1	None	Half-dried	239	410	0.443	14.6	3.61	28.4
	E2	2% Na_2SO_3	Half-dried	284	424	0.483	19.1	4.31	29.1
	E3	2% NaOH	Half-dried	231	467	0.516	23.1	5.46	26.3
	E4	2% NaOH	Wet	345	565	0.483	22.2	4.37	29.0
MLH	-	2% NaOH	Half-dried	302	478	0.389	8.79	2.42	29.7
Larch	-	2% Na_2SO_3	Half-dried	262	688	0.523	18.9	6.46	36.2

[a] Pretreatment conditions: 121 ºC, 2 h

[b] Refining conditions: 165 ºC, 0.7 MPa, 10 min and 0.10 mm disk clearance

[c] PFI mill beating: 10000 revolution

[d] Determined using a Lorentzen-Wettre fiber tester CODE 912

[e] Solid content: 55%

Table 2.5 Effects of blowing methods on characterization of thermomechanical pulps using screens

	Sample name	Refining [a,] Blowing method	CSF (mL)	Classification using screens [b] >1180 µm (> 14 mesh) (%)	600–1180 µm (28-14 mesh) (%)	300–600 µm (48-24 mesh) (%)	150–300 µm (100-48 mesh) (%)	150 µm > (100 mesh >) (%)
SB	S1	Half-dried [c]	812	17 ±0.4	15 ±0.2	22 ±0.2	21 ±1.0	25 ±0.5
	S2	Half-dried	785	25 ±0.4	17 ±0.5	22 ±0.4	18 ±0.2	18 ±0.4
	S3	Half-dried	788	15 ±0.2	19 ±0.2	26 ±0.4	20 ±0.3	20 ±0.3
	S4	Wet	778	34 ±0.9	17 ±0.9	21 ±0.2	14 ±1.8	14 ±1.4
EFB	E1	Half-dried	768	4 ±0.4	15 ±0.4	27 ±1.0	24 ±1.1	30 ±2.4
	E2	Half-dried	782	6 ±0.4	16 ±0.3	26 ±0.5	22 ±1.5	30 ±0.6
	E3	Half-dried	782	10 ±0.4	22 ±1.2	27 ±0.3	19 ±0.7	22 ±0.4
	E4	Wet	780	30 ±0.5	22 ±0.3	20 ±0.1	14 ±0.1	14 ±0.4
Mill MLH	-	Dried	-	21 ±0.5	23 ±1.5	23 ±1.3	25 ±0.4	8 ±0.1

[a] Pretreatment conditions: 121 ºC, 2 h; Refining conditions: 165 ºC, 0.7 MPa, 10 min and 0.10 mm disk clearance

[b] Determined using a Bauer McNett Fiber Classifier No. 2593

[c] Solid content: 55%

2.3.4 Effect of refining temperature on pulp physical properties

Refining can improve the bonding ability of fibers, forming strong and smooth paper with good printing properties (Lei et al., 2010). Refining can not only separate fiber walls, but also create finer fibers through cutting and shortening, as well as external and internal fibrillation. This is caused by the partial removal of the primary wall, bringing the secondary wall into direct contact with water; water absorption promotes swelling which improves the fiber flexibility. Flexibility governs many physical and optical properties of pulp and paper, including paper formation (Fernando et al., 2011), and therefore control over the flexibility is of vital importance. In the refining process, these fibers are typically exposed to compression and shear forces, which alter the fibers (Gharehkhani et al., 2015).

As shown in **Table 2.6**, the refining process of half-dried SB and EFB pulps at 165 ºC resulted in stronger pulp sheets compared to the 140 ºC process. The strength of wet EFB pulp at 165 ºC was similar to that at 140 ºC, while the strength of the wet SB pulp at 140 ºC was higher than that at 165 ºC. Using a half-dried blowing method, increasing the temperature improved the physical properties of paperboard. It has been reported that the temperature and moisture present during refining are key factors affecting fibers separation and development, due to the softening and viscoelastic behavior provided to the wood material (Illikainen, 2008). High temperatures before and during refining contribute to softening of the fibers, making them easier to refine and improving their physical properties (Salmen, 1984; Fernando et al., 2011; Muhic, 2010). Increased temperatures also enhance fibrillation, leading to better single fiber properties (Aisyah et al., 2013). High temperatures could lead to very low strength poorly bonding pulps when it would be so high that the lignin spreads over fiber surfaces.

In the preparation of SB pulps, a chemical-free pretreatment, thermomechanical refining at 165 ºC and a half-dried blowing method provided the best paper strength properties of the resulting pulp sheets. In the preparation of EFB pulps, pretreatment with 2% NaOH, thermomechanical refining at 165 ºC and a half-dried blowing method provided the best paper strength properties of the resulting pulp sheets.

2.3.5 Properties of MDF fabricated from EFB TMP

As shown in **Table 2.7**, MOR, MOE and IB of the EFB MDF were 18.5, 1649 and 1.4 N/mm^2, respectively. The MOR and MOE were suitable for actual use, but still lower than those of commercial MDFs industrially produced from MLH. The IB and TS were suitable to the minimum requirement of JIS A 5905:2003 for fiberboards.

Table 2.6 Effect of refining temperature on pulp physical properties

	Sample name	Pretreatment [a] Chemical dosage	Refining Temp. (ºC)	Blowing method	Pulp properties [b] CSF (mL)	Fiber length [c] (µm)	Density (g/cm^3)	Tensile index (N·m/g)	Tear index (mN·m^2/g)
SB	S5	2% Na_2SO_3	140	Half-dried [d]	197	470	0.358	8.5	2.37
SB	S2	2% Na_2SO_3	165	Half-dried	388	449	0.429	14.0	2.77
EFB	E5	2% Na_2SO_3	140	Half-dried	390	452	0.439	16.2	4.22
EFB	E2	2% Na_2SO_3	165	Half-dried	284	424	0.483	19.1	4.31
SB	S6	2% NaOH	140	Wet	168	531	0.450	16.0	3.16
SB	S4	2% NaOH	165	Wet	219	517	0.519	13.6	2.73
EFB	E6	2% NaOH	140	Wet	308	535	0.484	22.1	4.47
EFB	E2	2% NaOH	165	Wet	345	565	0.483	22.2	4.37

[a] Pretreatment conditions: 121 ºC, 2 h

[b] PFI mill 10000 revolutions

[c] Determined using a Lorentzen-Wettre fiber tester CODE 912

[d] Solid content: 55%

Table 2.7 Effect of refining temperature on pulp physical properties

	Density (kg/m^3)	MOR [a] (N/mm^2)	MOE [b] (N/mm^2)	IB [c] (N/mm^2)	Immersion in water 20ºC for 24 h	
					TS [d] (%)	WA [e] (%)
EFB	769	18.5 ±1.6	1649 ±147	1.4	11.7	32.1
MLH	777	30.8 ±2.8	3006 ± 304	1.5	8.3	24.2

[a] MOR: modulus of rupture in static bending

[b] MOE: modulus of elasticity in static bending

[c] IB: internal bond test

[d] TS: thickness swelling

[e] WA: water absorption

2.4 Conclusions

The optimum thermomechanical pulping conditions for EFB involved pretreatment with 2% NaOH at 121 ºC for 2 h and thermomechanical refining at 165 ºC, 0.7 MPa and a 0.10 mm disc clearance with a half-dried blowing method. Meanwhile, with a chemical-free pretreatment under the same thermomechanical refining and blowing conditions, the resulting SB pulp had its highest tensile and tear indices. The tensile indices of SB and EFB pulps obtained under these conditions were 22.0 and 23.1 N·m/g. The relationship between the fiber properties and the solid content of the pulp fibers blown into the atmosphere just after pressurized refinement were examined. The strength properties of SB and EFB half-dried pulps having 55% solid contents were slightly higher than those of the SB and EFB wet pulps. These results clearly suggest that SB and EFB can be fabricated into promising pulp fibers for raw materials of paperboard and MDF. MDF was successfully made from dried EFB refined fibers with 92% solid content by dry process, and the suitable properties for actual use were obtained.

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Table 2.A1 Experimental results

		Pretreatment 121°C			Refining condition			PFI	CSF (ml)		Fiber						Pulp physical properties			
Material	Symbol	Chemical	Dosage (%)	Time (h)	Pressure (MPa)	Temp. (°C)	Retention time (min)	Revolution	Before beating	After beating	Length (mm)	Length distribution (%) 0.2-0.8 mm	0.8-1.4 mm	1.4-2.0 mm	2.0-7.5 mm	Width (µm)	Density (g/cm^3)	Tensile index (N.m/g)	Tear index ($mN.m^2/g$)	Brigthness (% ISO)
EFB pellet	A	-	-	-	0.7	165	10	10,000	838	205	0.368					28.5	0.486	11.30	2.33	24.0
EFB	B	-	-	-	0.7	165	10	10,000	801	359	0.417					25.9	0.431	12.59	3.00	29.6
EFB	C	-	-	2	0.7	165	10	10,000	768	239	0.410	95.3	4.1	0.2	0.4	26.1	0.443	14.64	3.61	28.4
EFB	D	Na_2SO_3	2	2	0.5	140	10	10,000	793	390	0.452	92.7	4.2	1.2	1.9	26.4	0.439	16.22	4.22	30.8
EFB	E	Na_2SO_3	2	2	0.7	165	10	7,500	782	322	0.418	95.1	4.4	0.4	0.1	24.5	0.481	18.72	4.14	29.9
EFB	F	Na_2SO_3	2	2	0.7	165	10	10,000	782	284	0.424	94.8	4.4	0.2	0.6	25.0	0.483	19.11	4.31	29.1
EFB	G	Na_2SO_3	2	2	0.7	165	10	12,500	782	245	0.444	93.4	6.0	0.6	0.0	25.5	0.481	20.11	4.47	29.8
EFB	H	Na_2SO_3	4	2	0.7	165	10	10,000	760	262	0.433	93.9	5.7	0.4	0.0	25.5	0.495	20.76	4.54	32.5
EFB	I	NaOH	2	2	0.7	165	10	10,000	782	231	0.467	90.7	8.1	0.6	0.6	26.0	0.516	23.05	5.46	26.3
SB	J	-	-	2	0.7	165	10	10,000	812	215	0.550	85.0	12.7	2.3	0.0	32.3	0.507	21.98	3.82	32.3
SB	K	Na_2SO_3	2	2	0.5	140	10	10,000	793	197	0.470	92.3	6.9	0.8	0.0	32.1	0.358	8.48	2.37	43.0
SB	L	Na_2SO_3	2	2	0.7	165	10	7,500	785	416	0.492	90.8	8.4	0.6	0.2	34.5	0.430	14.51	2.55	38.4
SB	M	Na_2SO_3	2	2	0.7	165	10	10,000	785	388	0.449	93.4	6.1	0.4	0.1	32.3	0.429	13.95	2.77	37.6
SB	N	Na_2SO_3	2	2	0.7	165	10	12,500	785	339	0.522	87.7	11.0	1.0	0.3	34.0	0.396	12.71	2.63	37.6
SB	O	Na_2SO_3	4	2	0.7	165	10	10,000	786	364	0.514	89.1	9.5	1.2	0.2	33.9	0.441	16.55	3.09	38.9
SB	P	NaOH	2	2	0.7	165	10	10,000	788	415	0.542	86.8	10.2	1.7	1.3	33.0	0.394	13.71	3.00	36.6
MLH	Q	Na_2SO_3	2	2	0.7	165	10	10,000	818	409	0.457	90.5	8.6	0.8	0.1	34.1	0.360	4.59	0.60	31.3
MLH	R	NaOH	2	2	0.7	165	10	10,000	805	302	0.478					32.7	0.389	8.79	2.42	29.7
Larch	S	Na_2SO_3	2	2	0.7	165	10	10,000	788	262	0.688	71.4	21.6	5.3	1.7	44.8	0.523	18.92	6.46	36.2
Larch	T	NaOH	2	3	-	40	-	5,000	-	310	0.817					42.2	0.383	19.25	6.31	35.7
Larch	U	NaOH	2	3	-	40	-	10,000	-	210	0.721					44.3	0.471	24.07	5.69	36.7
Larch	W	-	-	3	-	40	-	5,000	-	190	0.897					43.9	0.452	23.09	7.65	42.0
SB	S-1	NaOH	2	2	0.7	165	10	5,000	756	548	0.583					30.8	0.493	13.18	3.43	36.0
SB	S-2	NaOH	2	2	0.5	140	10	5,000	763	473	0.578					32.5	0.370	9.41	3.09	35.4
SB	S-3	NaOH	2	2	-	40	-	5,000	516	220	0.545					33.2	0.362	11.96	3.26	43.3
EFB	E-1	NaOH	2	2	0.7	165	10	5,000	759	404	0.594					28.2	0.428	18.38	4.57	29.9
EFB	E-2	NaOH	2	2	0.5	140	10	5,000	725	312	0.562					26.9	0.425	18.13	4.80	32.6
EFB	E-3	NaOH	2	2	-	40	-	5,000	539	279	0.584					31.9	0.406	16.76	3.97	31.8
SB	X	NaOH	2	2	0.7	165	10	10,000	778	219	0.517					35.0	0.519	13.58	2.73	35.2
SB	B-2	NaOH	2	2	0.5	140	10	10,000	702	168	0.531					32.2	0.450	15.95	3.16	35.3
SB	B-3	NaOH	2	2	-	40	-	10,000	589	140	0.499					34.2	0.389	12.98	2.51	43.2
EFB	Y	NaOH	2	2	0.7	165	10	10,000	780	345	0.565					34.0	0.483	22.16	4.37	29.0
EFB	F-2	NaOH	2	2	0.5	140	10	10,000	753	308	0.535					27.8	0.484	22.05	4.47	32.0
EFB	F-3	NaOH	2	2	-	40	-	10,000	610	172	0.532					28.2	0.462	19.26	4.86	28.6

TMP disk clearance 0.1 mm

Table 2.A2 Fibers fractionation

	Symbol		14 mesh on >1180 µm opening Average (%)	SD	14 to 28 mesh 600-1180 µm opening Average (%)	SD	28 to 48 mesh 300-600 µm opening Average (%)	SD	48 to 100 mesh 150-300 µm opening Average (%)	SD	100 to 200 mesh 75-150 µm opening Average (%)	SD
EFB pellet	A	165	11	± 0.4	20	± 1.4	34	± 0.7	16	± 1.0	19	± 0.4
EFB	B	165	14	± 0.6	20	± 1.2	26	± 0.4	20	± 0.6	20	± 1.7
EFB	C	165	4	± 0.4	15	± 0.4	27	± 1.0	24	± 1.1	30	± 2.4
EFB	D	140	28	± 2.7	24	± 0.3	21	± 0.7	14	± 1.0	13	± 1.1
EFB	EFG	165	6	± 0.4	16	± 0.3	26	± 0.5	22	± 1.5	30	± 0.6
EFB	H	165	2	± 0.4	10	± 1.3	25	± 1.2	24	± 1.2	38	± 1.4
EFB	I	165	10	± 0.4	22	± 1.2	27	± 0.3	19	± 0.7	22	± 0.4
SB	J	165	17	± 0.4	15	± 0.2	22	± 0.2	21	± 1.0	25	± 0.5
SB	K	140	35	± 1.9	18	± 0.5	20	± 0.6	14	± 0.7	14	± 0.3
SB	LMN	165	25	± 0.4	17	± 0.5	22	± 0.4	18	± 0.2	18	± 0.4
SB	O	165	28	± 1.4	15	± 0.2	21	± 0.1	18	± 0.5	18	± 1.3
SB	P	165	15	± 0.2	19	± 0.2	26	± 0.4	20	± 0.3	20	± 0.3
MLH	Q	165	0	± 0.0	4	± 0.3	22	± 0.1	26	± 1.5	49	± 1.2
MLH	R	165	1	± 0.3	10	± 0.4	28	± 0.2	25	± 0.1	37	± 1.0
Larch	S	165	1	± 0.3	14	± 0.6	24	± 1.6	22	± 0.6	39	± 1.5
Larch	TUW	40	12	± 2.1	35	± 0.6	29	± 0.9	14	± 0.8	10	± 1.0
SB	X	165	34	± 0.9	17	± 0.9	21	± 0.2	13	± 1.8	14	± 1.4
SB	SB-2	140	15	± 0.9	20	± 0.7	23	± 0.8	21	± 0.5	21	± 2.4
SB	SB-3	40	10	± 0.4	17	± 0.6	23	± 0.8	23	± 0.7	27	± 2.3
EFB	Y	165	31	± 0.5	22	± 0.3	20	± 0.0	14	± 0.0	14	± 0.4
EFB	EF-2	140	16	± 1.0	22	± 0.4	25	± 0.3	19	± 0.4	19	± 0.4
EFB	EF-3	40	15	± 0.6	27	± 0.5	23	± 0.4	15	± 1.0	20	± 1.6
Mill MLH	Z	-	21	± 0.7	23	± 1.5	23	± 1.3	25	± 0.4	8	± 0.1

Table 2.A3 Comparison of dry fiber fractionation between EFB and MLH

Material	Dry fiber fractionation (%)				
	2.8 mm opening	2.0 mm opening	1.0 mm opening	0.5 mm opening	pass to 0.5 mm opening
EFB	6.2	1.7	7.8	19.1	67.4
MLH	1.1	1.3	5.9	19.2	69.1

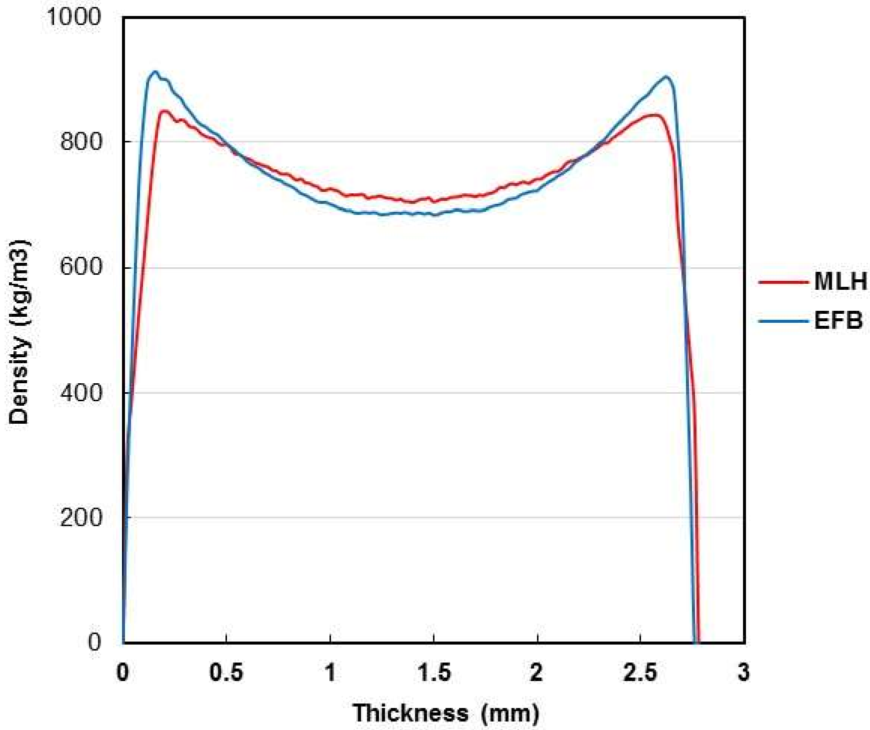

Fig. 2.A1 Dendity profile of EFB and MLH medium density fiberboard

Photo 2.A1 MDF produced from EFB dried fibers

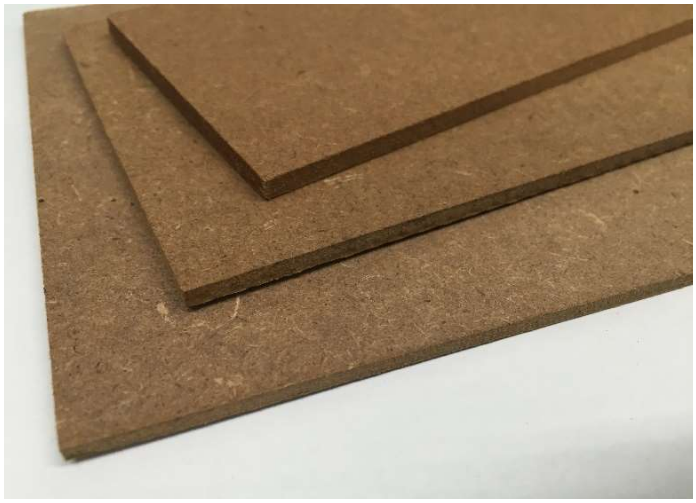

Photo 2.A2 MDF produced from MLH dried fibers

Chapter 3 Operation Method of a Laboratory Thermomechanical Refiner for Obtaining Sugarcane Bagasse and Empty Fruit Bunch Dried Fibers of Medium Density Fiberboard

3.1. Introduction

The pulp yield of approximately 90% obtained in the mechanical refining processs is one of the advantages of using empty fruit bunch (EFB) of oil palm (*Elaeis guineensis*) as a raw material in the pulp and paper industry (Harsono et al., 2015). In the refining process of thermomechanical pulp (TMP), individual fibers are separated by mechanical forces and then substantially developed to meet their papermaking properties (Gorski et al., 2010). Usually, higher temperatures can have a better softening effect on fibers, leading to easier initial fiber separation and fibrillation (Li et al., 2011), which leads to better single fiber properties (Muhic, 2010). The ideal refining process removes the middle lamella and outer layers from the single fiber (cell) wall to produce fibers and fines with good bonding potential (Li et al., 2011). It is possible to preserve the fiber length with softening by increasing the temperature (Kure et al., 1999). Refining pressure had a significant effect on the mechanical properties of medium density fiberboard (MDF board) (Xing et al., 2006; Aisyah et al., 2012). The increment of steam pressure in refining significantly improved the mechanical properties of MDF board fabricated from oil palm trunk (Ibrahim et al., 2013).

In Japan, one of the refining equipment used for TMP is a laboratory pressurized refiner (model BRP45-300SS) manufactured by Kumagai Riki Kogyo Co., LTD. (Nerima, Tokyo), and it mainly comprises three parts: machine, processing and outlet. The machine of TMP refiner includes a main electric motor that has a maximum disk rotation of 3000 rpm. The processing part consists of a pressurized hopper (material input and a heater), a screw conveyor (material feeder), a gearbox (for the speed reduction ratio), a disk-clearance (plate gap) adjustment, a belt-pulley (for transmission), and a single disk refiner (consisting of a rotor and a stator of 300 mm diameter) with a certain pattern (Disk pattern J in this study). The outlet part includes a pulp container (blow tank) (**Fig. 3.1**) (Harsono et al., 2015; Han el al., 2001; Kamijo et al., 2015).

In this study, the TMP refiner was equipped with a steam boiler (model SU-200) supplied by MIURA Co., LTD. (Matsuyama, Ehime) that provides a maximum steam pressure of 0.98 MPa (**Photo 3.1**). Additionally, the pulp blow tank can be connected to a conical bottom joint, an on-off blow valve, a blowing pipe, and a pulp blow box (**Photo 3.1**) to fabricate dried fibers for use in the manufacturing of MDF board. The pulp blow box had a length of 72 cm, width of 60 cm and height of 58 cm with four 60 mesh (250 μm opening) stainless steel wire sides.

A process that produces TMP from non-wood fibrous materials such as sugarcane (*Saccharum officinarum*) bagasse (SB) and EFB of oil palm has not yet been industrialized. On the other hand, there is a requirement for the manufacture of dried TMP from these non-wood fibers for the preparation of MDF board, which can be an alternative to wood, and a possible method of treatment of agricultural wastes. Dried fibers are required to produce high quality MDF board. SB and oil palm EFB are non-wood fibrous materials that are easily available in Indonesia, and have the potential to be developed as fibers for use in MDF board.

This research was aimed at modifying the operation of a laboratory pressurized TMP refiner to obtain dried fibers from SB and oil palm EFB for fabricating MDF materials under suitable conditions.

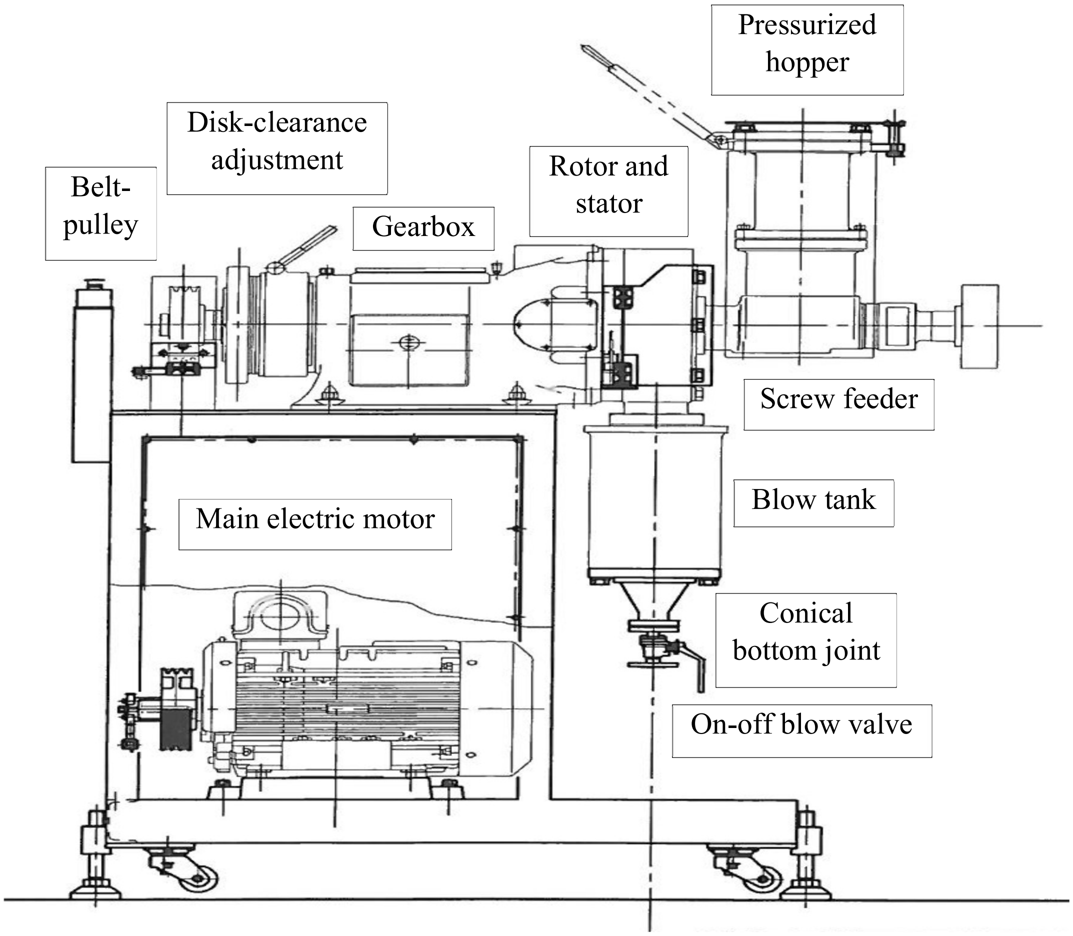

Fig. 3.1 Technical drawing of a TMP refiner

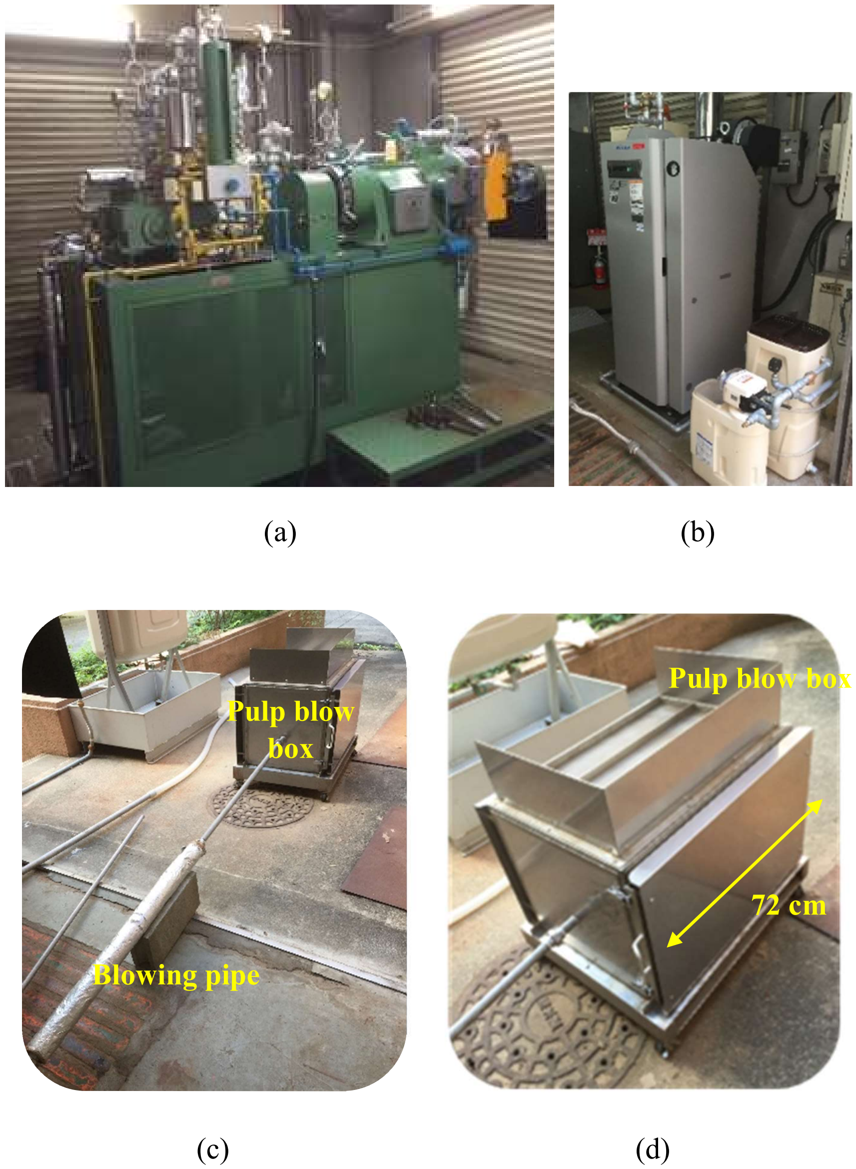

Photo 3.1 Pressurized refiner model BRP45-300SS (a), steam boiler SU-200 (b), blowing pipe (c), and pulp blow box (d)

3.2. Materials and methods

3.2.1 Material preparation

In this study, SB and oil palm EFB were obtained from PT. Madukismo in Yogyakarta, Indonesia and PT. Perkebunan Nusantara VIII in Bogor, West Java, Indonesia, respectively. SB was manually washed twice and dried in direct sunlight. The moisture level after drying was approximately of 8–10%. The dried SB was cut into 0.5–2.0 cm size pieces using a shredding machine, while the oil palm EFB was cut into 0.5–4.0 cm size pieces by a laboratory disk mill (Harsono et al, 2015). (**Photo 3.2**).

Wood chips with dimenssions of 2 cm x 2 cm x 0.4 cm (length × width × thickness) of mixed light hardwoods (MLH) and MLH mill fibers provided by Hokushin Co., Ltd. Kishiwada, Osaka, Japan were used for comparison, Prior to pressurized refining, the materials were prepared as follows:

(1) SB (200 g oven-dried weight; approximately 10% moisture content) and oil palm EFB (300 g oven-dried weight; approximately 10% moisture content) were weighed and put into the pressurized hopper of a refiner (without water condition). These amounts are suitable for SB and oil palm EFB, as the final moisture contents of the refined fibers (blow pulp) can reach approximately 80%.

(2) Four times deionized water (800 and 1200 mL) was weighted and mixed with the SB and oil palm EFB (200 and 300 g oven-dried weight) to soften the material prior to refining (with water condition). A chemical (6 g of NaOH: 2% dosage based on materials) was added to water and the solution was then mixed with the oil palm EFB. The advantage of using the chemical for oil palm EFB has been explained in a previous study (Mulyantara et al., 2016).

Photo 3.2 Sugarcane bagasse (SB) (left) and oil palm empty fruit bunch (EFB) (right)

3.2.2 Preparation of a TMP refiner

Before using a TMP refiner, the operation is prepared according to procedures in **Table 3.A1** and **Photos 3.A1-3.A2** in the **Appendix**.

3.2.3 Control of steam pressure and detection of disk temperature

In this experiment, there are two line-valves for adjusting the pressure of the steam for the refiner, and the upper valve was used to obtain a pressure of 0.5–1.25 MPa and the other valve was used to obtaine a pressure of 0.035–0.5 MPa (**Photo 3A2-(9)).** A thermometer is put onto a stator disk for measuring the temperature during pre-heating and refining (**Photo 3.3**).

3.2.4 Operating procedure of TMP refiner

The TMP refiner was operated using the following procedure.

(1) After switching on the button of the TMP refiner to rotate a rotor disk, the refiner was left on for 20 min to warm up the rotor, rotor shaft, and a stator. A steam boiler was switched on and left on for 8 min until the pressure of supplied steam was approximately 0.8-0.9 MPa.

(2) A steam valve to the outlet pipe (**A** in **Photo 3.A1-(5)**) was closed (after opening it to allow the water inside the steam pipe system to leak out), and the other steam valve to the refiner (**B** in **Photo 3.A1-(5)**) was opened. Meanwhile, it was confirmed that the yellow alarm light was turning on automatically to indicate the flow of the pressurized steam inside the refiner.

(3) The TMP refiner was kept under pressurized conditions, where the pressure of the steam was adjusted to 0.7 MPa at 165°C.

(4) The pressurized TMP refiner (a rotating disk) was switched off after 20 min of warming up, and the refiner disk clearance was then set at 0.0 mm. This setting was confirmed by

audibly detecting the sound of the disk friction when the disk was rotated manually while the pressurized steam continued to flow in the refiner.

(5) The disk clearance was set at 0.10 mm, which was recommended in a previous study (Harsono et al, 2015) where the oil palm EFB was refined at 0.10-0.20 mm clearance, and the obtained fibers weere characterized by fractionation.

(6) The pressurized TMP refiner was then switched on again, and the steam valve to the refiner (**B**) was closed while the valve to the outlet pipe (**A**) was opened. To charge the materials, the steam valve outlet on top the pressurized hopper (**C** in **Photo 3.A1-(5)**) was opened to allow the steam to escape untill the internal pressure reached atmospheric pressure (0.0 MPa). Meanwhile, it was confirmed that the yellow alarm light had turned off automatically.

(7) The cover of the pressurized hopper was carefully opened and removed as all pressurized hopper equipment were still hot at 120-140°C. Then the material was charged inside the hopper after ensuring that the material was below the pressurized steam nozzle (**Photo 3.4**).

(8) The cover of the pressurized hopper was tightened after installing the steam valve outlet pipe, and the steam-release valve (**C**) was closed. The pressurized steam valve to the outlet pipe (**A**) was closed, and the pressurized steam valve to the refiner (**B**) was opened again.

(9) A little steam was allowed to escape to adjust the pressure by opening a small valve below the pulp blow tank (**D** in **Photo 3.A1-(5)**), and the TMP refiner was kept pressurized until the desired pressure 0.7 MPa was attained and stable conditions were reached. Then, the pressure was retained in the hopper for 20 min, which included the time (10 min) for raising the temperature to 165°C.

(10) After retaining for 20 min in the pressurized hopper, a screw feeder was started and at the same time the pulp blow valve (**E** in **Photo A1-(5)**) with a conical bottom joint was

immediately opened. Refining, which included the finishing of all mateials was completed in 3 min, the screw feeder was then stopped, and the blow valve (**E**) was closed.

(11) The disk temperature was recorded at 1 min intervals.

(12) The steam valve to the refiner (**B**) was closed while the valves (**A**, **C**) were slightly opened to allow the steam to gradually escape from the refiner. The disk was then unlocked and the clearance was at 5.0 mm of before stopping the rotation of the TMP refiner.

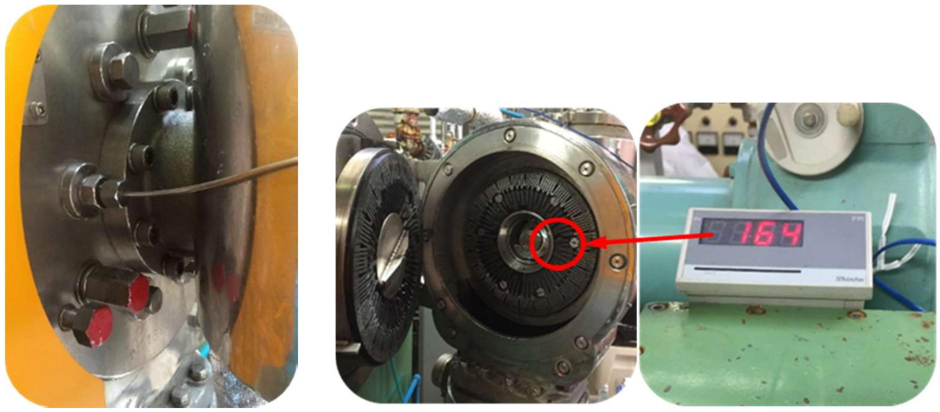

Photo 3.3 Thermometer (left) and indication of temperature of stator disk (right)

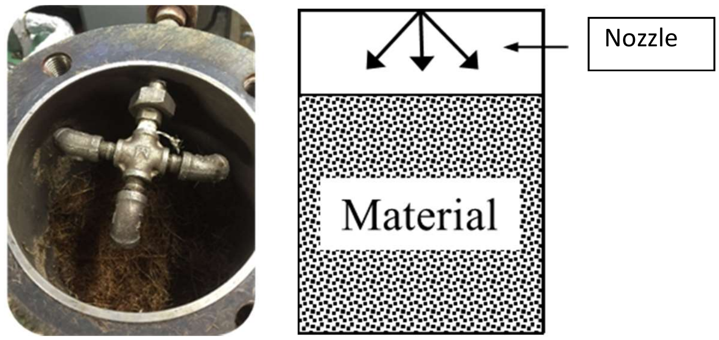

Photo 3.4 Charging the material into hopper

3.2.5 Final drying of obtained pulp

Finally, the obtained pulp was dried by blowing a warm air into the pulp blow box from its bottom wire side for 180 min for oil palm EFB and 360 min for SB using a commercial warm-air drier (Mitsubishi Electric AD-X80). The flow rate and temperature of the drier were approximately 20 L/min and 60-65°C, respectively. After separating the pulp into two parts in the pulp blow box, the solid contents of pulp was determined. The drying duration was confirmed as the solid content of the pulp in one part had reached 90-92%.

3.2.6 Evaluation of fibers properties

The properties of the fibers can be classified by the fractionation methods using dried fibers and wet fibers (Benthien et al., 2014). In this study, fiber fractionation was done according to the condition of the dried fibers. Three screens with 850, 355, and 180 µm opening (20, 45, and 80 mesh, respectively) were used for this method. Ten grams (oven-dried weight) of refined fibers was charged into the upper screen (850 µm opening) with 30 stainless steel balls (12.5 mm diameter) to disperse the fibers and to send them smoothly to the subsequent screens during shaking for 2 min (**Photo 3.5**). The four fractions were named as "on 850 µm opening", '355-850 µm opening", "180-355 µm opening", and "pass 180 µm opening". The refined fibers of SB and oil palm EFB were compared to the refined fibers of MLH obtained from the industrial process of Hokushin Co., Ltd.

Additionally, the three fractions except the longest fraction ("on 850 µm opening") dispersed in water, and the fiber length and width of each fraction was determined by a Lorentzen-Wettre fiber tester CODE 912.

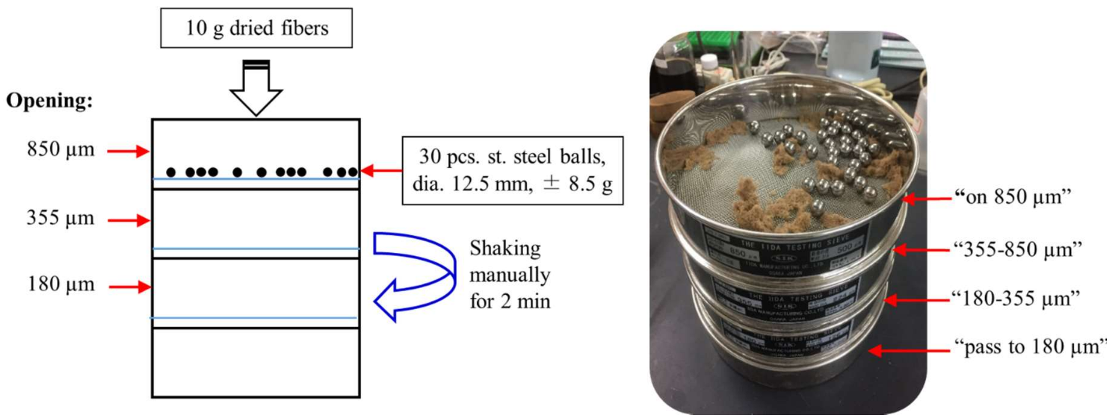

Photo 3.5 The fibers classified by the dry fiber fractionation method

3.3. Results and discussion

3.3.1 Profile of disk temperature during pre-heating and refining of materials

The disk temperature increased from 160°C to 165°C in 20 min while the material was heated to 165°C in a pressurized hopper. However, it decreased immediately to 120°C during refinement when a screw feeder started pushing the material into the refiner disk because of the opening of the pulp blow valve (**E**) (**Fig. 3.2**). The blow valve must be opened immediately to allow the fibrous material to flow into the disk part and to move the refined fibers to the pulp blow box through the pulp blowing pipe.

3.3.2 Solid content of obtained pulp

The SB and oil palm EFB fibers obtained in the pulp blow box were divided into two parts depending on the difference between average solid contents, and named as first grade pulps (higher solid contents) and second grade pulps (lower solid contents) (**Fig. 3.3**)

The separation should occur as follows. After the pulp blow valve was opened and the pressurized steam blew the refined fibers through the pulp blow pipe to the blow box, the flocks of refined fibers hit the stainless steel wall of the pulp blow box, which was on the opposite side of the pulp blow pipe. Then, the flocks of fibers that separated from the steam turned toward to the wall at the other side of the pulp blow box to become individually separated fibers, which resulted in sufficiently dried fibers.

The first and second grade oil palm EFB fibers had a solid content of 81.4-86.8% and 46.6-53.4%, respectively (**Table 3.1**). The first and second grade SB fibers had a solid content of 55.6% and 50.2%, respectively. These fibers can be used for MDF board preparation after obtaining a solid content of more than 90%.

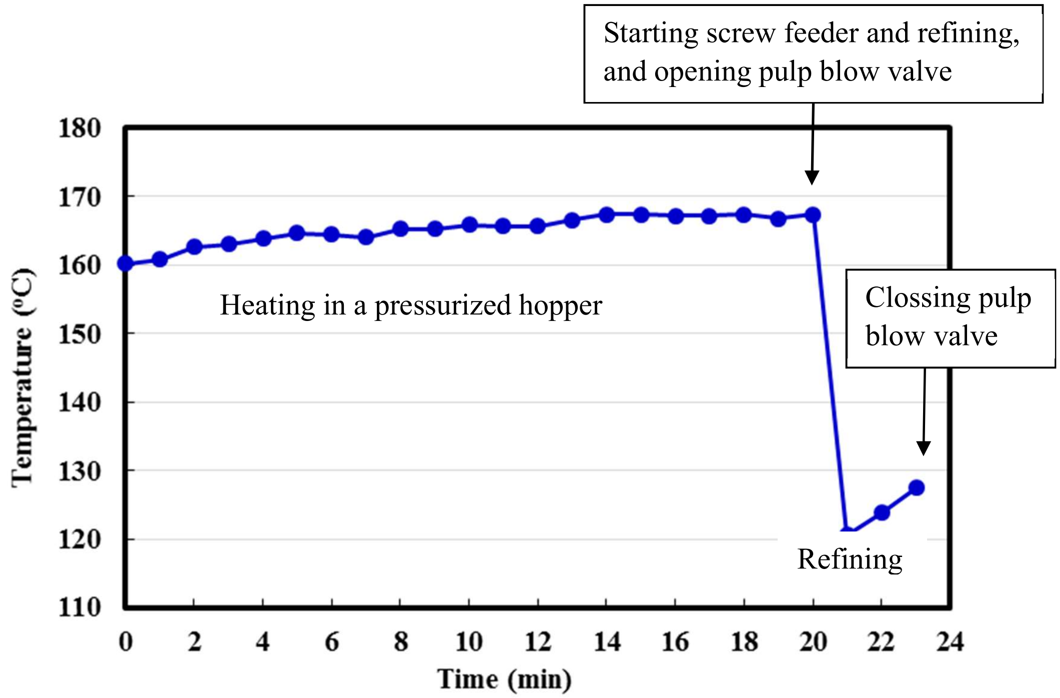

Fig. 3.2 Profile of disk temperature during pre-heating and refining

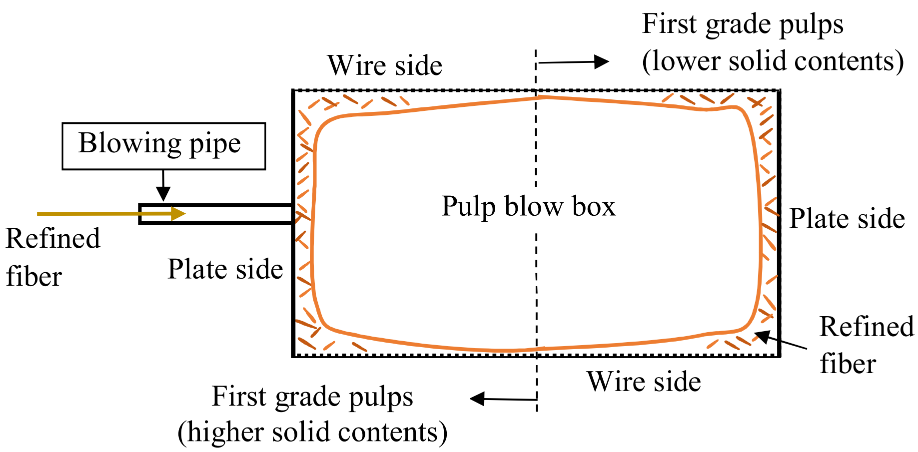

Fig. 3.3 Dried pulp distribution in a pulp blow box

Table 3.1 Solid content and weight ratios of the first and second grades fibers

No.	Materials	Liquid to materials ratio	Solid content (%)		Weight (%)	
			1st grade [a]	2nd grade [b]	1st grade	2nd grade
1	EFB	0.1	86.8	53.4	69.6	30.4
2	EFB	4.0	81.4	49.9	59.7	40.3
3	EFB-2% NaOH	4.0	81.9	46.6	73.2	26.8
4	SB	0.1	55.6	50.2	67.4	32.6

[a] 1st grade: higher solid content

[b] 2nd grade: lower solid content

3.3.3 Characterization of fiber fractionation

The results of the fractionation of the dried fibers showed that the ratios of the "355-850 µm opening" fractions for oil palm EFB and SB refined fibers were almost the same as that of MLH fibers. (**Fig. 3.4**). The ratio of the coarse "on 850 µm opening" fraction for oil palm EFB fibers without water was slightly higher than those of other oil palm EFB and SB conditions. The reason for this observation could be that impregnation with water softens the lignin prior to refining (Back and Salmen, 1982, Illikanen, 2008). The result of fiber fractionation shows that oil palm EFB fibers impregnated with water prior to refining had the lowest ratio of the coarse fraction.

Next, the fiber length of each classified fraction was determined by a Lorentzen-Wettre fiber tester. **Table 3.2** shows that the fiber length of the "355-850 µm opening" fraction for oil palm EFB fibers impregnated with water was longer than those of the oil palm EFB fibers without water and oil palm EFB fibers with alkaline solution (2% NaOH). Additionally, the fiber lengths of these fractions of all oil palm EFB fibers were shorter than that of MLH refined fibers.

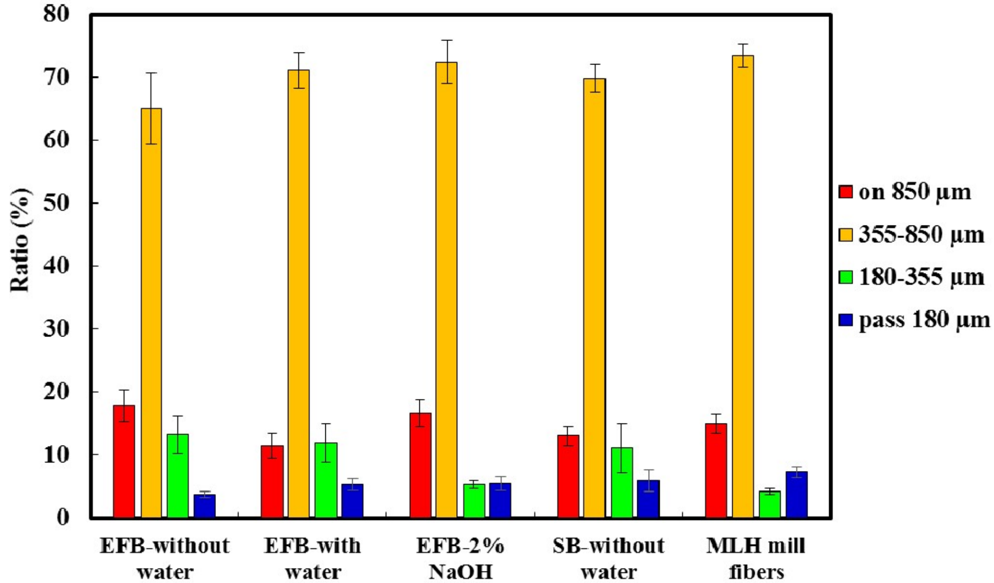

Fig. 3.4 The dried fiber fractionation of resulting TMP fibers

Table 3.2 Comparison of mean fiber length and width of EFB and SB laboratory refined fibers and MLH mill fibers

No.	Materials	Opening 355-850 µm		Opening 180-355 µm		Opening 180 µm pass	
		Length (µm)	Width (µm)	Length (µm)	Width (µm)	Length (µm)	Width (µm)
1	EFB-without water	767	31.9	493	28.9	425	29.2
2	EFB-with water	893	30.5	537	26.9	368	26.6
3	EFB-2% NaOH	826	31.5	694	29.5	462	28.2
4	SB-without water	1055	39.0	591	38.8	473	37.0
5	MLH mill fibers	1134	28.6	788	28.5	596	30.8

[a] Determined with a Lorentzen-Wettre fiber tester CODE 912.

3.4. Conclusions

This research is aimed at modifying the operation of a laboratory pressurized TMP refiner to obtain dried fibers from non-wood materials such as SB and oil palm EFB for fabricating MDF materials under suitable conditions. A solid content of approximately 80% of oil palm EFB dried fibers and approximately 55% of SB dried fibers were obtained by this modified method for the production of MDF materials. These fibers could be fully dried to a solid content of 90-92% for fabricating MDF materials. According to the results from fiber fractionation and the measurement of the fiber length of these dried fibers, the SB and oil palm EFB dried fibers were comparable to the mixed light hardwoods fibers produced in an industrial MDF board process.

Appendix

A manual of additional operation procedures is shown in **Table 3.A1,** with demonstrations in **Photos 3.A1-A2**. EFB, SB and MLH dried fiber with “on 850 µm opening”, “355-850 µm opening”, “180-355 µm opening”, and “pass 180 µm opening” presented at **Photo 3.A3-A5,** respectively.

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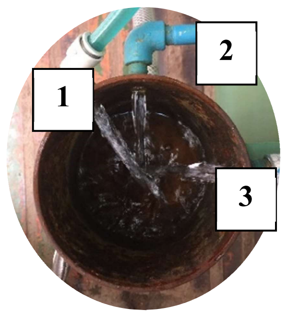
Table 3.A1 Procedure before using a TMP refiner

	Procedure	Reference
(1)	Use gloves, a safety glass and long-sleeved clothes. Check the cleanness and dryness of the refiner disks (rotor and stator disks), pressurized hopper, screw feeder, blow tank (pulp container) and pulp blow box.	**Photo 3.A1-(1)**
(2)	Check that the oil level in the gearbox with SAE 32 oil is should be above the minimum line level. Apply a small amount (several drops) of oil to an upper bearing and a disk clearance adjustment with SAE 46 oil.	**Photo 3.A1-(2)**
(3)	Open the tap water for cooling and check water balance at three points at the outlet of the coolig system.	**Photo 3.A1-(3)**
(4)	Ensure that all the bolts of a disk cover are tightened. The left bolt should be tightened first.	**Photo 3.A1-(4)**
(5)	Ensure all the valves (5 valves) of the refiner are closed. The valve for the outlet pipe (A), the valve for the refiner steam pressure (B), the valve on top of the pressurized hopper (C) for leaking steam, the valve below the pulp container for leaking the steam pressure during refining (D) and the valve for pulp outlet (E).	**Photo 3.A1-(5)**
(6)	Ensure that the steam leak pipe on top of the pressurized hopper (C) is directed to an area outside the laboratory.	**Photo 3.A2-(6)**
(7)	Ensure that the cover of the pressurized hopper is closed and the four bolts of the cover have been tightened. Ensure that three safety censors (belt cover, disk holder and disk cover censor) and one censor to measure the temperature inside the disks refiner are located at suitable positions. Direct the blowing pipe to the pulp outlet and connect to the pulp blow box.	**Photo 3.A2-(7)**
(8)	Adjust the disk clearance at 5.0 mm and then lock for machine safety. First, loosen the fastener, adjust the disk clearance to 5.0 mm, and then lock the fastener.	**Photo 3.A2-(8)**

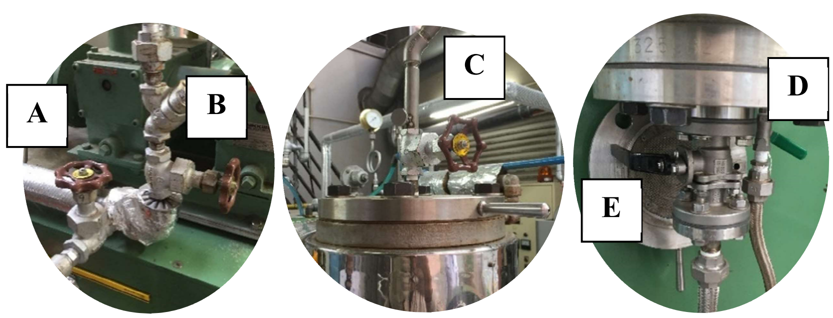
(1) Oil level for gearbox

(2) Upper bearing and disk clearance adjustment



(3) Cooling system

(4) Disk cover

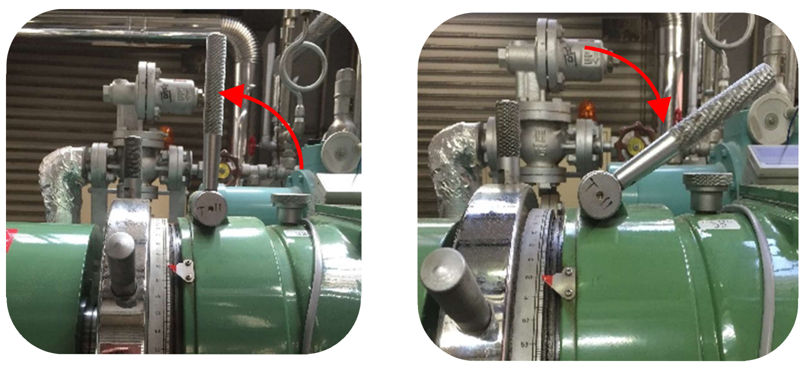


(5) Five valves of a refiner

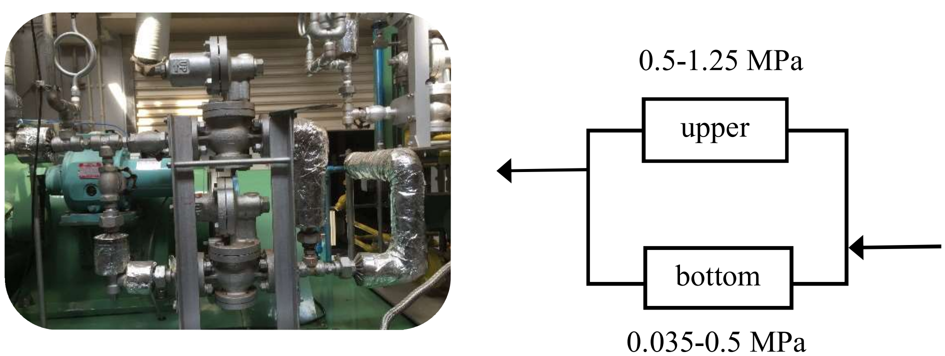
Photo 3.A1 Appearance of procedure to be followed before using a TMP refiner (1)-(5)

(6) Steam leak pipe on top of the pressurized hopper

(7) Cover of pressurized hopper



(8) Adjusting disk clearance

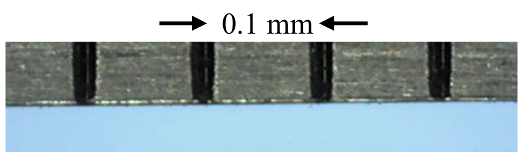


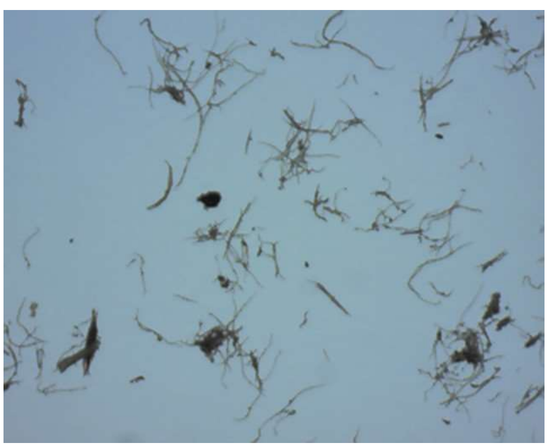
(9) Adjusting steam pressure

Photo 3.A2 Appearance of procedure to be followed before using a TMP refiner (6)-(9)

(1) "on 850 µm opening" EFB dried fibers

(2) "355-850 µm opening" EFB dried fibers





(3) "180-355 µm opening" EFB dried fibers

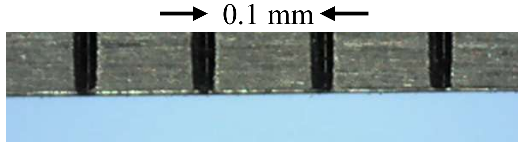
(4) "pass 180 µm opening" EFB dried fibers

Photo 3.A3 EFB dried fiber with "on 850 µm opening", "355-850 µm opening", "180-355 µm opening", and "pass 180 µm opening" (1)-(4)

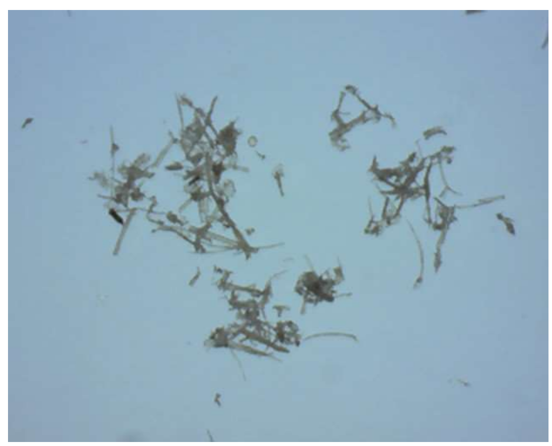
(5) “on 850 µm opening” SB dried fibers

(6) “355-850 µm opening” SB dried fibers

→ 0.1 mm ←



(7) “180-355 µm opening” SB dried fibers

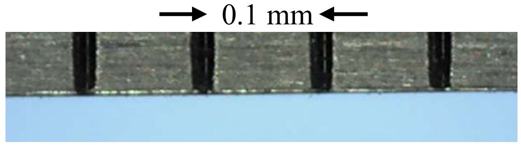


(8) “pass 180 µm opening” SB dried fibers

Photo 3.A4 SB dried fiber with “on 850 µm opening”, “355-850 µm opening”, “180-355 µm opening”, and “pass 180 µm opening” (5)-(8)

(9) "on 850 μm opening" MLH dried fibers **(10)** "355-850 μm opening" MLH dried fibers

→ 0.1 mm ←



(11) "180-355 μm opening" MLH dried fibers **(12)** "pass 180 μm opening" MLH dried fibers

Photo 3.A5 MLH dried fiber with "on 850 μm opening", "355-850 μm opening", "180-355 μm opening", and "pass 180 μm opening" (9)-(12)

Chapter 4 General Conclusions

A process for the production of thermomechanical pulp (TMP) from non-wood fibrous materials such as sugarcane (*Saccharum officinarum*) bagasse (SB) and empty fruit bunch (EFB) of oil palm (*Elaeis guineensis*) have not been industrialized. On the other hand, there is a requirement for the production of dried TMP from non-wood fibers for the preparation of medium density fiberboard (MDF) as an alternative to wood and as a possible method of treatment of agricultural wastes. Dried fibers are required to produce high quality MDF. SB and oil palm EFB are non-wood fibrous materials that are easily available in Indonesia, and have the potential to be developed as fibers for use in materials of MDF. This research is aimed at modifying the operation of a laboratory pressurized TMP refiner to obtain dried fibers from both these non-wood fiber sources for fabricating MDF materials under suitable conditions. An approximate solid content of 80% of oil palm EFB dried fibers and an around 55% solid content of SB dried fibers were obtained by this modified method. These fibers could then be fully dried to obtain a solid content of 90–92%. On observing the results from fiber fractionation and the length of these dried fibers, it was found that the SB and oil palm EFB dried fibers were comparable to mixed light hardwoods (MLH) fibers produced in an industrial MDF process.

Before that, the suitable conditions to fabricate pulp fibers for paperboards and MDF from the SB and the EFB fibers have examined. First, the effects of chemical pretreatments at 121 ºC and pressurized refining under steaming conditions were studied in regards to the thermomechanical pulps. The optimal conditions to achieve the highest paper strength properties of the EFB pulp involved pretreatment with 2% NaOH for 2 h and refining at 0.7 MPa of pressure at 165 ºC. The sugarcane bagasse (SB) pulp properties could not be improved by chemical pretreatment, while the EFB pulp properties clearly benefited from such pretreatment. Second, the effects of dry and wet blowing methods after pressurized refining on the fiber properties were examined. The strength properties of the half-dried SB and EFB pulps with 55% solid content were slightly higher than those of their wet counterparts. These results clearly suggest that SB and EFB can be promising refined fibers for paperboard and MDF preparation. Third, MDF was successfully made from dried EFB refined fibers with 92% solid content by dry processing. The modulus of rupture (MOR), modulus of elasticity (MOE) and internal bond (IB) of the EFB MDF were 18.5, 1649 and 1.4 N/mm^2, respectively. The MOR and MOE were suitable for actual use, but still lower than those of commercial MDFs industrially produced from MLH. The IB and TS were suitable to the minimum requirement of JIS A 5905:2003 for fiberboards.

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Lilik Tri MULYANTARA

November 2016

Appendix

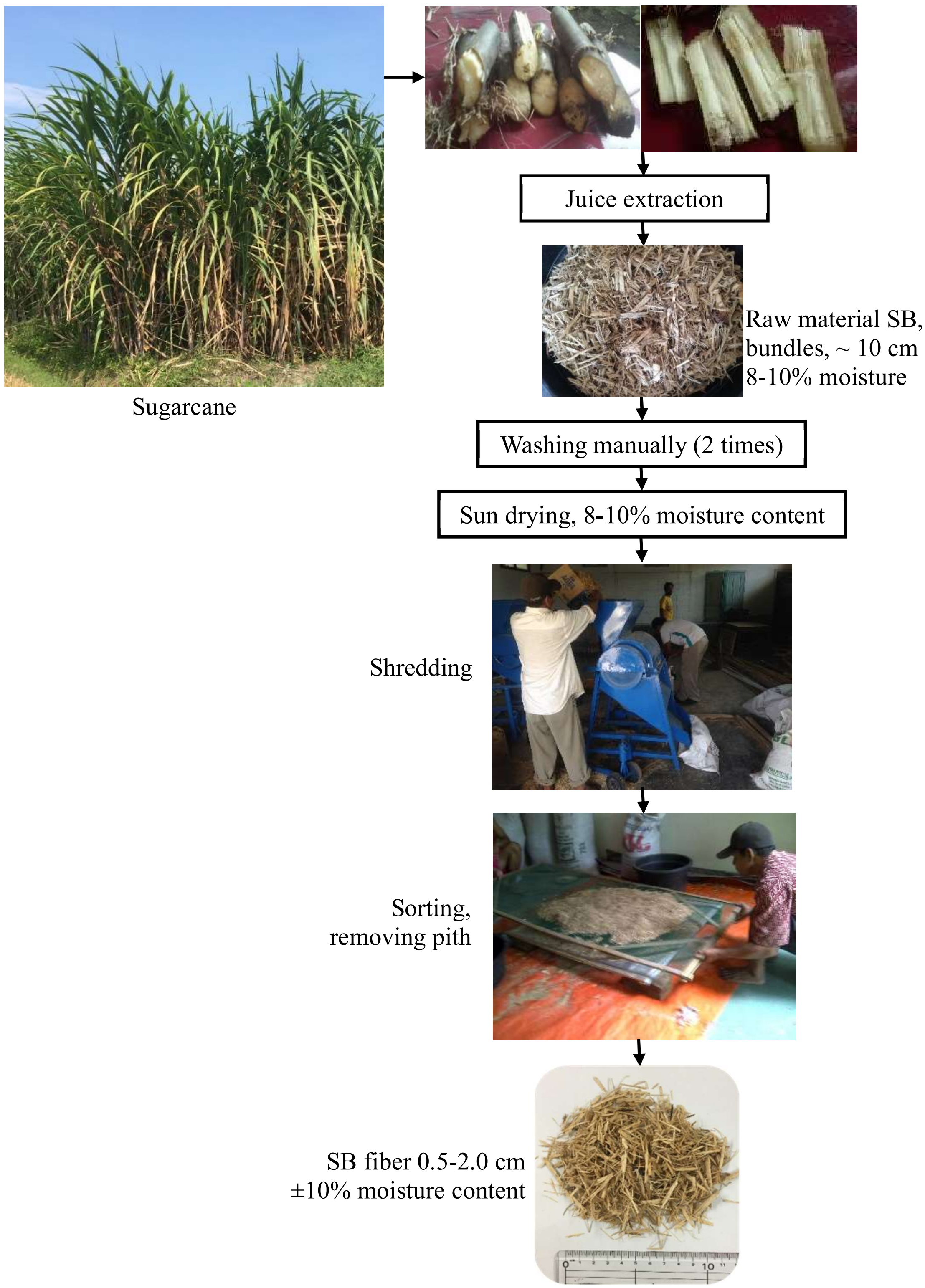


Photo Ap.1 Sugarcane bagasse(SB) preparation

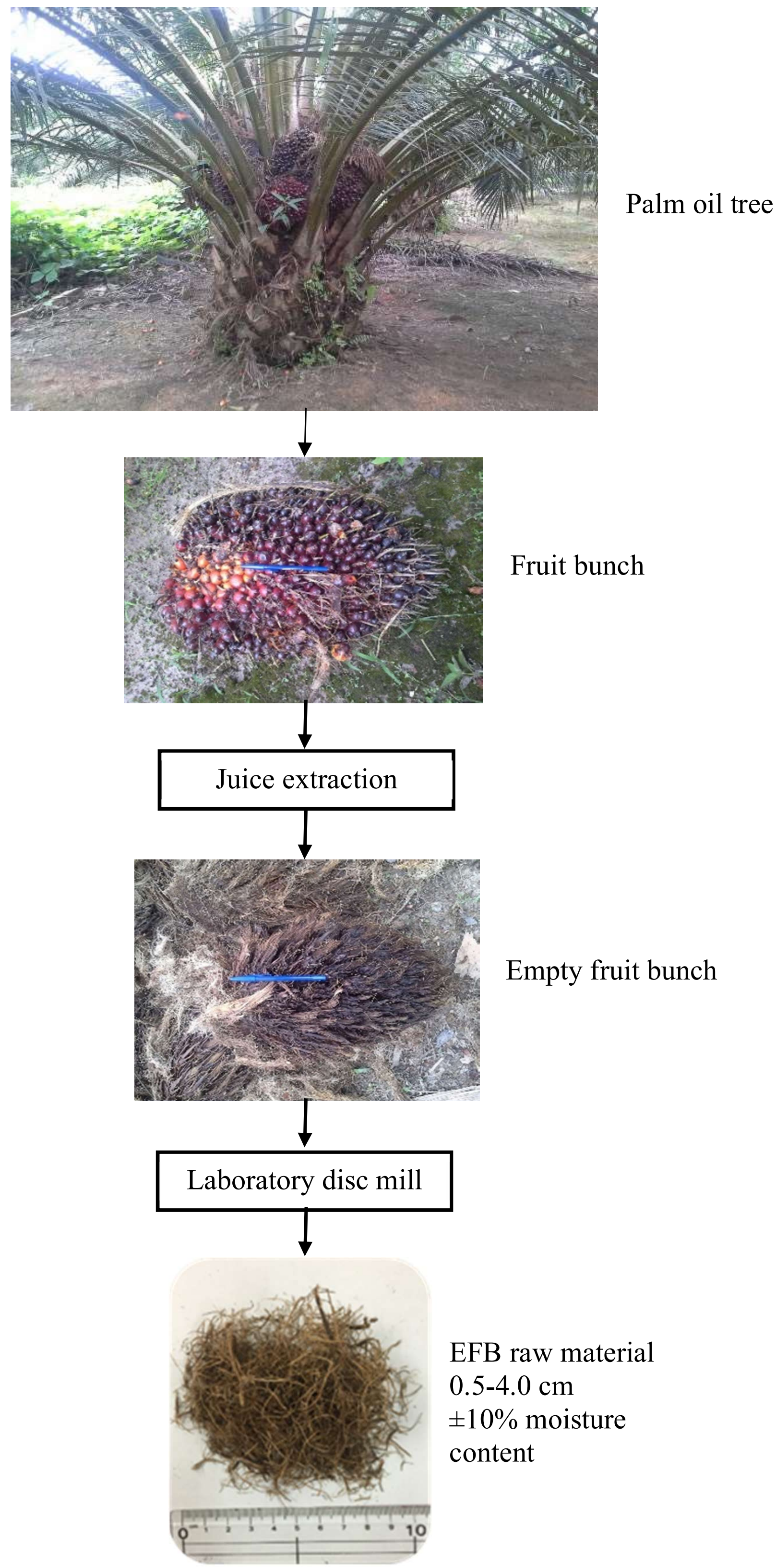
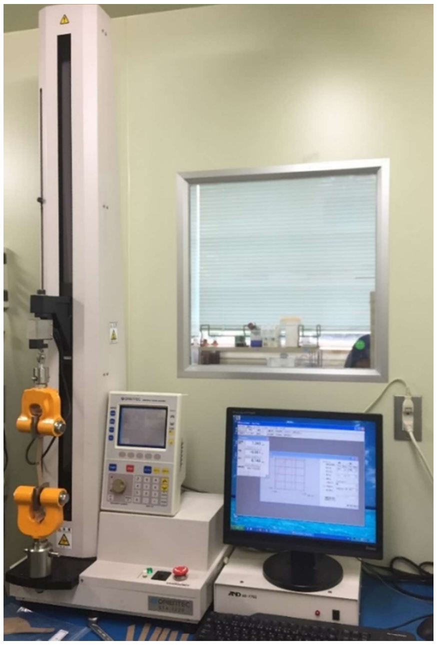


Photo Ap.2 Oil palm empty fruit bunch (EFB) preparation

(a)

(b)



(c)

(d)

Photo Ap.3 Bauer-McNett Fiber Classifier No. 2593 (a), Lorentzen-Wettre Fiber Tester CODE 912 (b), Orientec STA-1225 Universal Testing Machine (c), and Elmendorf Tearing Tester (d)