

Effect of Tree Age and Active Alkali on Kraft Pulping of White Jabon¹

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ABSTRACT

White Jabon (*Anthocephalus cadamba* Miq.) is one of the fast growing species in Indonesia and has the potential as the raw material for pulp and paper. In this research, 3, 5, and 7 years old White Jabon woods were pulped under different active alkali charge of 15%, 18%, 21%, 24%, and 27%, and its effect on delignification degree, kappa number, pulp yield, pulp viscosity, brightness, unbeaten freeness, and delignification selectivity was investigated. The results showed that tree age and active alkali concentration influenced the quality of pulp and pulping properties, except for that of unbeaten freeness. Delignification degree increased with increasing active alkali charge, and this brought about the decrease of pulp kappa number. The pulping yield tended to decrease below the Klason lignin of approximately 4%. Even though the 3 years old wood resulted in the highest brightness and highest delignification selectivity, the highest pulp viscosity was obtained with the 5 years old wood. The dominant fiber length of all wood ages was in the range of 1.2 - 2.0 mm. The 3 years old wood was considered to be the most promising raw material for kraft pulping in the view point of pulping properties, pulp quality and harvesting rotation.

Keywords : White Jabon, kraft pulping, active alkali, tree age, pulping property

1. INTRODUCTION

Wood demand of Indonesia in 2013 was projected to about 58.87 million m³ and the production capability was only approximately of 41.39 million m³ (Department of Forestry, 2006). In this context, industrial plantation has been

actively endeavored as one of the promising ways to achieve the enough wood production and to satisfy wood demand. In particular, various fast growing species were expected to be capable of accelerating wood plantation productivity.

Among potential fast growing species, White

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Jabon (*Anthocephalus cadamba* Miq.), an endemic hardwood species of Indonesia, has recently attracted an interest and popularity. Under optimal silvicultural management, it can reach the height of 45 m and the diameter of 100 - 160 cm in 6 - 8 years (Johns *et al.*, 1993). It has been widely planted for reforestation, afforestation, and industrial plantation forest in Sumatera and Kalimantan (Krisnawati *et al.*, 2011). Even though White Jabon is suitable for the raw material of pulp production, the information on its pulping characteristics is lacking.

Kraft pulping method is the most widely applied for the production of chemical pulp (Dang and Nguyen, 2007; Hubbell and Ragauskas, 2010) due to its capability to pulp various species of wood, resulting in a high pulp quality, and retaining an established chemical and energy recovery systems (Gellerstedt *et al.*, 2004). Important variables concerned in kraft pulping include sulfidity, active alkali, L/W ratio, and H-factor (Smook, 1992). Chemical components composition of wood is also influential to the pulping properties.

The present work was aimed to determine the influence of tree age and active alkali concentration on the properties of Jabon wood kraft pulp. The results of the present research could give useful information to determine the most appropriate harvesting rotation of White Jabon wood to produce high quality kraft pulp.

2. MATERIALS AND METHODS

2.1. Raw Material Preparation

White Jabon woods of 3, 5, and 7 years old were chipped to 2 - 3 cm in length and 3 - 5 mm in thickness. The moisture content of chips was determined in accordance with the procedures of ASTM D4442-07 standard. The concentration of NaOH and Na₂S for pulping was set to be 309.94 g L⁻¹ and 184.27 g L⁻¹, respectively.

2.2. Chemical Composition Analysis

Extractives content (ethanol-benzene), holocellulose, α -cellulose, Klason lignin, and acid soluble lignin of wood were determined by following the standard procedures of TAPPI T 204 om-88, Browning (1967), TAPPI T 203 os-74, TAPPI 222 om-88, and TAPPI UM 250, respectively. Hemicelluloses content was calculated by subtracting the α -cellulose content from holocellulose content.

2.3. Fiber Measurement

Fiber measurement was carried out in accordance with the procedures, which is applied for the fiber analyzer KaajaniFiberLabTM and explained in detail in Metso Automation (2001). The equipment consists of analyzer and sample unit. Two CCD cameras were used to capture fiber images. The length, width and thickness of the cell wall of the fiber were measured and

Table 1. Bleaching conditions of pulp

Stage	Chemical (% of OD pulp)			Temperature (°C)	Retention time (min)	Consistency (%)
	ClO ₂	NaOH	H ₂ O ₂			
D ₀	1.5	-	-	60-70	60	4
E _P	-	1.6	0.2	70-80	90	10
D ₁	1	-	-	80	120	10
D ₂	0.5	-	-	80	120	10

the curl index, coarseness, cross sectional area and volume index were calculated.

2.4. Kraft pulping process

Pulping was carried out in a static conventional digester at similar H-factor. The active alkali concentration of pulping liquor was varied at 15, 18, 21, 24, and 27% with equal sulfidity of 30%. Each batch of cooking was carried out with 150 g of oven dried chips in liquor to wood ratio (L/W) of 6. Impregnation time (time to maximum cooking temperature) was consisted of 3 stages, i.e. the first stage from 26 to 90°C for 26 min, the second stage from 90 to 140°C for 25 min and the third stage from 140 to 165°C for 25 min. Cooking at maximum temperature (165°C) was carried out for 65 min. Upon cooking stage, the resulting pulp was sieved through a flat screen and its cooking yield was then determined gravimetrically.

2.5. Evaluation of Pulping and Pulp Properties

Delignification degree (DD) of pulping was calculated following the formulae of:

$$DD (\%) = \frac{\text{total lignin of wood} - \text{residual lignin of pulp}}{\text{total lignin of wood}} \times 100\%$$

Kappa number of pulp, consumed alkali, and pulp viscosity were determined in accordance with the procedures of TAPPI T 236 cm-85, TAPPI T 625 cm-85, and TAPPI T 230 om-89 standards, respectively. Pulp brightness was expressed as % ISO determined in accordance with TAPPI T 452 om-98. Measurement of unbeaten pulp freeness was carried out with Canadian Standard Freeness (CSF) tester following the procedures of TAPPI T 227 om-94 standard. The delignification selectivity of pulping was calculated as:

$$\text{Selectivity} = \frac{\text{total lignin content of wood} - \text{residual lignin of pulp}}{\text{carbohydrate content of wood} - \text{carbohydrate content of pulp}}$$

The resulting data of the present results was statistically analyzed by the use of statistical package ad in Microsoft Excel 2010 for windows software.

2.6. Pulp Bleaching

The resulted pulp was bleached with an elementally chlorine free (ECF) bleaching method through D₀E_PD₁D₂ sequences. Table 1 indicates

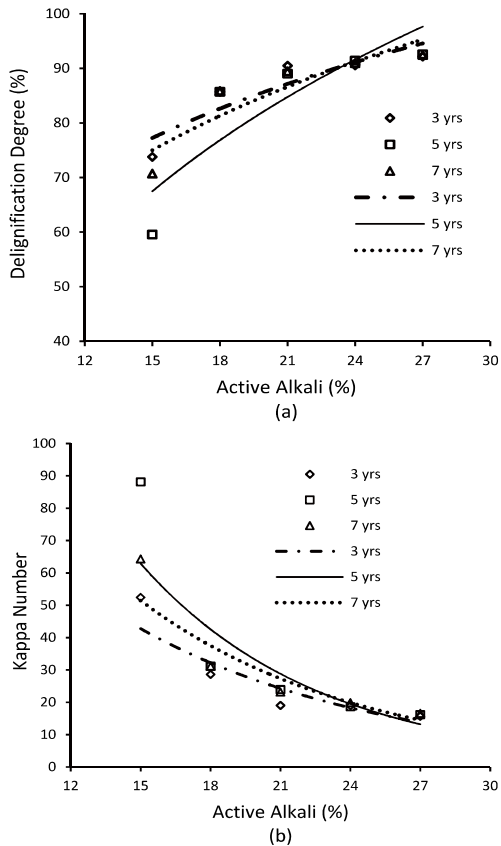


Fig. 1. Effect of tree age and active alkali amount on delignification degree and kappa number.

the conditions applied in bleaching process.

3. RESULTS AND DISCUSSION

3.1. Delignification Degree and Kappa Number of Pulp

The degree of delignification and the kappa number of pulp were significantly influenced by the age of tree, active alkali, and the combined effect between the tree ages and active alkali. Fig. 1 shows the effect of tree age and

active alkali amount on delignification degree and kappa number of the resulted pulp. Delignification degree increased with decreasing age of trees and increasing active alkali amount. Consequently, kappa number of pulp decreased with decreasing age of trees and increasing active alkali amount. At active alkali amount of 27%, the delignification degree in all ages reached about 92%. The concentration of OH^- and HS^- in cooking liquor significantly influences delignification degree (Nguyen and Dang, 2006). It has been well understood that HS^- improves cell wall swelling and facilitates delignification. A relatively low kappa number of 16 - 17 (at active alkali of 27%) was achieved under H-factor of 803-815. It seems that White Jabon wood is more readily to be pulped, compared to *Pinus pinaster* wood, because its kraft pulping requires H-factor of 5009 to reach pulp kappa number of 22.3 (Baptista *et al.*, 2008).

3.2. Pulping Yield

The increase of active alkali decreased the screened pulp yield. Polynomial regression between active alkali and pulping yield are indicated in Fig. 2. Tree age, active alkali, and their interaction significantly influenced the yield of pulping ($P < 0.05$). MacLeod (2007) quantified the influence of increasing alkali charge to the decreasing of pulping yield. He concluded that every 1% increase of the active alkali decrease the pulping yield of 0.15%, and worse in the case of hardwood due to its high

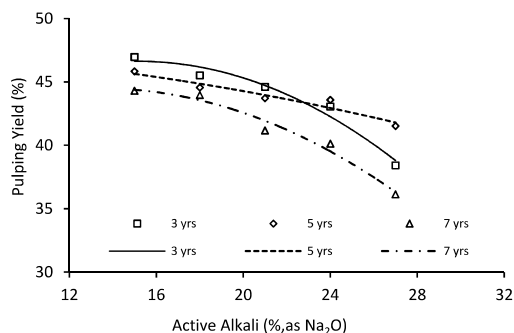


Fig. 2. Effect of tree age and active alkali amount on pulping yield.

hemicellulose content susceptible to alkali. Statistical evaluation indicated that the pulping yield of the 3 and 5 years old woods was not significantly different but higher than that of the 7 years old wood.

Dang and Nguyen (2007) reported that there is a strong relationship between pulping yield and kappa number dependent on pulping conditions in the pulping of *Eucalyptus nitens*. Fig. 3 shows the relationship between pulping yield and kappa number. With increasing Kappa number to about 40, pulping yield was increase and then became stable. The maximum yield was achieved at the kappa number of about 40. That maximum yield of pulping was achieved at active alkali between 15% and 18%. At 15% of active alkali, delignification degree was relatively low compared to those of using higher active alkali. Lower delignification degree resulted in more uncooked chips (rejects), thus brought about lower screened pulping yield. The increase of active alkali increases the delignification degree (Copur and Tozluoglu, 2008) and should increase the screened pulping

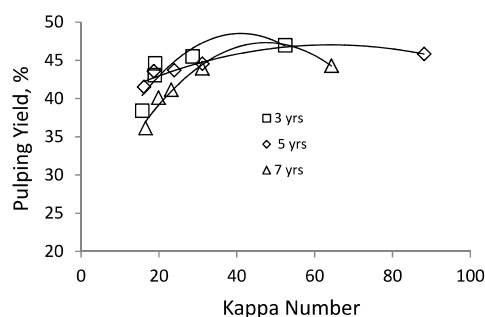


Fig. 3. The relationship between the kappa number of pulp and the yield of pulping.

yield. However, the increasing of delignification degree at higher active alkali will be also accompanied by the increasing degree of carbohydrate degradation, and consequently the screened yield of pulping would be also decreasing.

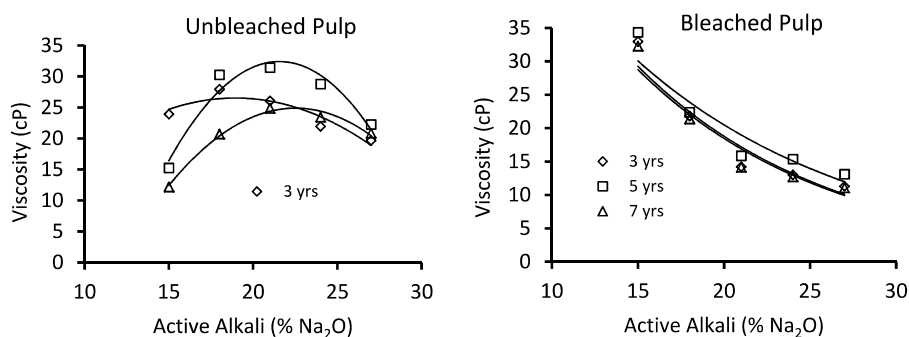
3.3. Pulp Viscosity and Brightness

The viscosity of unbleached pulp is commonly determined to be a basis of subsequent pulp processing. In oxygen delignification, for instance, the kappa number reduction of the unbleached pulp is limited to a level of insignificant pulp viscosity reduction. Brightness of unbleached pulp is useful indicator for the bleach ability of pulp. However, the viscosity of unbleached pulp is not a reliable indicator of pulp strength (Kubes *et al.*, 1981). When strength properties are not determined, bleached pulp viscosity is commonly used to predict pulp strengths.

The viscosity of pulp can represent the polymerization degree of cellulose in pulp. Bleached pulp viscosity can be also an indirect indication

Table 2. Cell wall components of White Jabon wood

Content (%)	The age of tree (yrs.)		
	3	5	7
Holocellulose	75.4	76.2	74.9
α -Cellulose	42.4	40.3	42.3
Hemicelluloses	33.0	36.0	32.6
Klason Lignin	25.2	27.2	27.6
Acid Soluble Lignin (ASL)	0.8	1.1	0.9
Total Lignin	26.0	28.3	28.5
Extractives (Ethanol-Benzene, %)	5.1	4.8	5.2

**Fig. 4.** The viscosity of unbleached and bleached pulp obtained by pulping of 3, 5, and 7 years old wood.

of pulp strength. It is known that pulp with high viscosity shows better mechanical properties (Khiari *et al.*, 2010). The tree age, active alkali, and their interaction significantly influenced the pulp viscosity ($p < 0.05$). As shown in Fig. 4, pulp produced from 5 years old wood showed a higher viscosity. Statistical evaluation indicated that the viscosity of pulp from the 3, 5, and 7 years old wood was different. It has been reported that the presence of hemicelluloses decreases pulp viscosity due to its lower degree of polymerization, compared to that of cellulose (Vu *et al.*, 2004; Copur and Tozluoglu, 2008; Gullichsen and Paulapuro, 2000). However, the present result indicated

that the pulp from 5 years old wood showed higher viscosity than those of 3 and 7 years old wood, despite the hemicellulose content was not statistically different in all woods as summarized in Table 2.

Fig. 4 indicates that the increase of active alkali charge to approximately 20% increase the pulp viscosity. The degradation of hemicellulose might be the cause of this, since hemicellulose is very susceptible to alkaline hydrolysis and has a lower degree of polymerization (DP) compared to cellulose (Gullichsen and Paulapuro, 2000). Removal of low DP hemicellulose could result in a higher pulp viscosity. The decrease of unbleached pulp viscosity at

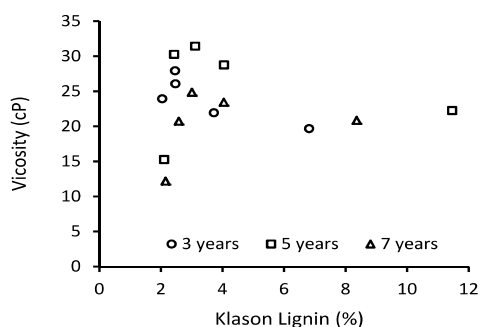


Fig. 5. The influence of lignin content on the viscosity of the unbleached pulp.

active alkali charge of more than 20% can be caused by the increase of OH^- concentration in the pulping liquor. OH^- is not just degrades lignin, but at higher concentration is also degrading cellulose. Degradation of cellulose by alkali can proceed through peeling reaction and alkaline hydrolysis that causes the loss of pulp viscosity (Sharma and Shukla, 2013). The decreasing tendency of bleached pulp viscosity (Fig. 4) can be a clear indication of cellulose degradation with increasing alkali charge. Area *et al.* (2010) stated that the increase of the OH^- concentration accelerates the degradation of cellulose (Area *et al.*, 2010).

The presence of lignin, an amorphous tridimensional polymeric substance, is also reported to influence pulp viscosity. Feng and Alén (2001a, b) reported that the decreasing amount of lignin in pulp indicated by the decreasing of kappa number, can increase pulp viscosity. Fig. 5 indicates that maximum viscosity of unbleached pulp was obtained at Klason lignin content slightly lower than 4%, and the viscosity tended to decrease when lignin content

was further reduced. Cellulose can be depolymerized at high degree of delignification (Abrantes *et al.*, 2007). Table 2 indicates that the α -cellulose, hemicellulose, and lignin content of the investigated woods were in the range of 40-42%, 33-36%, and 26-29%, respectively. Furthermore, Fig. 3 indicates that the pulping yield below the lignin content of approximately 4% (kappa number of 20-30) was below 45%. These data ascertained that cellulose degradation was occurred. Cellulose degradation is also indicative from Fig. 4 that shows the viscosity of bleached pulp decreased with increasing active alkali charge. The degradation of oxidized cellulose in alkaline treatment can decrease the viscosity of bleached pulp (Camarero *et al.*, 2004).

Brightness of pulp indicates the amount of light reflected by pulp sample relative to that of a standard material (titanium oxide). As shown in Fig. 6, the age of trees, active alkali, and their interaction influenced on the brightness of pulp ($p < 0.05$). The brightness of pulp increased with increasing active alkali and all level of active alkali statistically resulted in a different brightness of pulp. Pulp produced from wood of 3, 5, and 7 years old showed different level of brightness and the highest brightness was shown in the 3 years old wood. Different lignin content might cause different brightness. The lignin content of the 3 years old wood was lowest as shown in Table 2.

The increase of brightness will be mainly due to lignin removal during pulping and bleaching processes. This can be seen from the presence

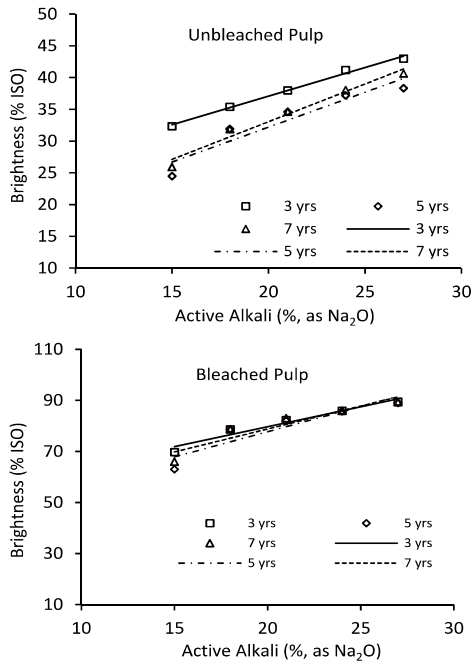


Fig. 6. Unbleached and bleached pulp brightness at various active alkalis.

of negative correlation between the kappa number of pulp and the brightness of pulp (Fig. 7). Chlorine dioxide is not only efficiently dissolves residual lignin fragments, but also reduce the chromophoric groups in pulp, and reduce the capacity of lignin in pulp to absorb light (Gullichsen and Fogelholm, 1999). High coefficient of determination (Fig. 7) indicates that the presence of lignin (measured as kappa number) highly influenced on the brightness of pulp.

3.4. The Freeness of the Unbeaten Pulp

Pulp freeness is a criterion for the drain ability of pulp suspension. It is also related to the pulp surface characteristics and fiber swelling. Other characteristics such as charge ratio, drain-

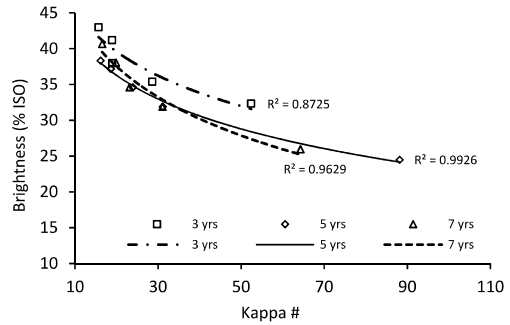


Fig. 7. The relationship between the kappa number of pulp and the brightness of pulp.

age time, specific surface area, water retention value, tensile index, and burst index are known to have linearly correlation with pulp freeness (Banavath *et al.*, 2011). It is also influential in the pulp flow and wet end process of papermaking.

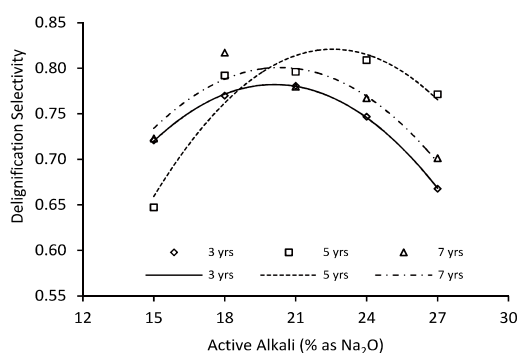
The present results indicated that the freeness of unbeaten kraft pulp were in the range of 646-722 ml CSF. Measurement of unbeaten pulp freeness is important to the subsequent processes of pulp (such as pulp beating) for papermaking. It was found that the pulping yield did not influence the freeness of unbeaten pulp. The present finding was in the contrary with the finding of Tran (2006), where he found that unbeaten pulp freeness increased with increasing yield of pulping.

3.5. Delignification Selectivity

During pulping processes, delignification is accompanied with various degree of carbohydrate degradation. Delignification selectivity is the ratio between lignin and carbohydrate, which is removed from chips within a certain

Tabel 3. Fiber measurement of White Jabon wood

Parameter	The age of Tree (yrs.)		
	3	5	7
Fiber length (mm)	1.23	1.41	1.28
Fiber diameter (μm)	27.15	28.68	29.31
Cell wall thickness (μm)	4.65	6.46	6.52
Lumen cell diameter (μm)	18.52	15.77	16.27
Curl (%)	8.38	5.98	6.28
Coarseness (mg/mm)	0.08	0.12	0.12

**Fig. 8.** Delignification selectivity of pulping 3, 5, and 7 years old White Jabon wood.

pulping time or predetermined delignification degree. High delignification selectivity indicates high degree of delignification with low cellulose degradation.

In the present results, the age of trees, active alkali, and their interaction influenced the delignification selectivity ($P < 0.05$). Further statistical analysis indicated that pulping with alkali charge of 18%, 21%, and 24% retained equal delignification selectivity and were higher than these of pulping with active alkali of 15% and 27%. Delignification selectivity of pulping 5 and 7 years old wood were the same and were higher than that of pulping 3 years old wood. Differences in chemical composition of cell

walls among the three different age of wood could be the origin of the differences of delignification selectivity. Fig. 8 indicates that better delignification selectivity of pulping 3, 5, and 7 years old wood were achieved with active alkali of 21%, 24%, and 18%, respectively. Increasing active alkali above these percentages tended to decrease delignification selectivity. Baptista *et al.* (2008) has previously indicated that increasing delignification decreased delignification selectivity.

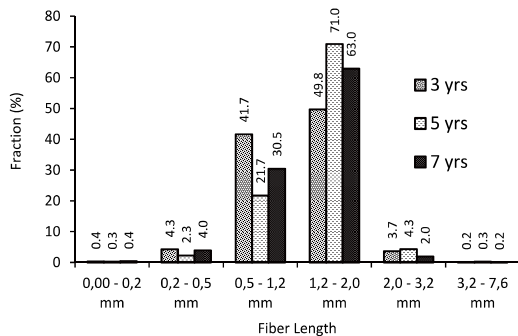
3.6. Fiber Analysis

3.6.1. Fiber Dimension

Fiber dimension is an important parameter determining the suitability of wood as pulp and paper material. Fiber measurement was carried out with fiber analyzer kajaaniFiberLabTM. to measure fiber length, fiber diameter, cell wall thickness, lumen cell diameter, curl, and coarseness. The age of tree influenced fiber length, fiber diameter, lumen cell diameter, cell wall thickness, curl, and coarseness ($p < 0.05$). Table 3 indicates the fiber parameters of the White Jabon wood of 3, 5, and 7 years old.

Table 4. Fiber derivative values of White Jabon wood

Parameter	The age of Wood (yrs.)		
	3	5	7
Runkle Ratio	0.318	0.451	0.445
Felting Power	45.275	49.278	43.828
Muhlstep Ratio	53.376	69.576	69.135
Coefficient of Rigidity	0.234	0.419	0.404
Flexibility Ratio	0.682	0.549	0.555

**Fig. 9.** Length weighted fraction of the 3, 5, and 7 years old White Jabon wood.

It can be seen from Fig. 9 that in all wood samples, fiber length of 1.2-2.0 mm retained the highest proportion, i.e. 49.8%, 71.0% and 63.0% of all measured fibers for the 3, 5, and 7 years old wood, respectively. A similar fiber length of White and Red Jabon wood (in the range of 900-1600 μm) was reported by Kim *et al.* (2013). According to the criteria of Indonesian wood fiber quality, Jabon wood of 3, 5, and 7 years old are classified as the 2nd fiber length quality (Rahman and Siagian, 1976). Therefore, from the view point of its fiber length, White Jabon wood is considered suitable for the raw material of pulp and paper.

In many cases, fiber derivative value is a better parameter to predict the suitability of a

wood for the raw material of pulp and paper. Fiber derivative values include Runkle ratio, Muhlstep ratio, Flexibility ratio, Felting power, and Coefficient of Rigidity. Table 4 indicates the values of these parameters.

Statistical analysis indicated that the 5 years old wood retained the highest Runkle ratio, felting power, Muhlstep ratio, and coefficient of rigidity. Total scoring value of all fiber derivative values indicated that White Jabon wood pulp was classified as the 2nd grade quality class for the raw material of pulp and paper (Rahman and Siagian, 1976).

4. CONCLUSION

The age of tree and active alkali influenced the quality of the resulted pulp. The present results indicated that White Jabon wood of 3 years old resulted in a better pulp quality than these of the 5 and 7 years old wood. Due to its lower lignin content, the wood was easier to be delignified at lower alkali consumption. Lower kappa number of pulp should result in a lower requirement of bleaching chemical to achieve certain pulp brightness. The yield of pulping of the 3 and 5 years old wood was equal, but both

were higher than that of the 7 years old wood. The present result indicates that for the purpose of pulp and paper raw material, technically White Jabon wood can be harvested at 3 years rotation.

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