Kraft Pulp Yields and Paper Properties of Ten-year-old Clonal Acacia hybrid *(Acacia mangium × Acacia auriculiformis)* Wood

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Abstract. Clonal *Acacia* hybrid (*Acacia mangium* × *Acacia auriculiformis*) breeding in Indonesia aims to improve wood quality for the paper industry. This study investigated the effect of anthraquinone (AQ) addition on the quality of pulp and paper from ten-year-old clonal *Acacia* hybrid (015D) wood. Pulp making was carried out by kraft process with an active alkali concentration of 17%, sulfidity of 25%, and AQ concentrations of 0%, 0.05%, and 0.10% at a maximum temperature of 170ºC for two hours of cooking time. AQ addition increased the yield to 54.70%, 58.23%, and 59.14% and decreased the kappa number to 24.06, 20.30, and 16.87, respectively. In terms of physical and optical properties, the paper had a burst index ranging from 3.31 to 3.66 kPa·m^2/g, a tear index ranging from 1.72 to 2.40 mN·m^2/g, a tensile index ranging from 49.41 to 50.28 Nm/g, opacity ranging from 87.38% to 88.44%, and brightness degrees ranging from 24.18 to 25.78. Burst and tensile indices met the Indonesian National Standard for broadleaf wood. AQ at 0.05% to 0.10% significantly reduced kappa numbers but did not significantly affect yield, mechanical, or optical properties. Therefore, an AQ concentration of 0.05% is recommended for clonal *Acacia* hybrid wood

1 Introduction

Hybrid acacia is the crosses between *Acacia mangium* and *Acacia auriculiformis* characterized by fast-growing, good wood property, resistant to pests or diseases, able to grow on marginal land, and is easy to propagate [1]. Hybrid acacia has the potential as a raw material for making pulp and paper with the same quality as *Acacia mangium* by evaluating the characteristics of its wood fiber [2]. Since 1998 the Center for Research and Development of Biotechnology and Forest Plant Breeding has developed a hybrid acacia breeding strategy in order to support the pulp and paper industry [3]. Clonal propagation is one of the methods to obtain homogenous superior wood.

Clonal *Acacia* hybrid stands offer sufficient productivity to meet the raw material demand of the pulp industry in Indonesia. Since 2002, *Acacia* hybrid clones have been developed in Indonesia using artificial pollination and are currently in the clone test stage. The productivity of these clonal *Acacia* hybrid stands reached 48 m^3/ha/year, with a basic density of 342.9–536.0 kg/m^3, cellulose content of 56.15–59.94%, and kraft pulp yield of 50.09–55.14% [2].

Acacia hybrid trees cultivated from seeds produced better pulp and paper properties compared to their two parents—*Acacia mangium* and *Acacia auriculiformis* in terms of yield and handsheet mechanical properties [4]. These trees produced a pulp yield exceeding 45%, with less than 3% rejects and a Kappa number of 28.7. In contrast, three-year-old clonal *Acacia* hybrid wood produced a pulp yield greater than 50%, with less than 4% rejects, and produced handsheets with good burst index, tensile index, print opacity, and brightness levels [5].

Pulp quality is significantly influenced by the properties of the raw materials and the pulping process. Anthraquinone (AQ) can be added during pulping in both the kraft and soda processes to enhance delignification and reduce carbohydrate degradation, thereby increasing the pulp yield [6-7]. AQ addition at concentrations of 0.05% to 0.10% has been known to increase the delignification rate, pulp yield, and strength properties of pulp [8-9].

To the best of our knowledge, there has yet to be much research related to the properties of pulp and paper made from clonal *Acacia* hybrid wood. Therefore, this study investigated the properties of pulp and paper produced from ten-year-old clonal *Acacia* hybrid wood with AQ addition in the kraft process. The 015D wood clones were selected for this study due to their better properties compared to other clones in the same planting plot.

2 Materials and Method

2.1 Material preparation

This study used wood from three ten-year-old *Acacia* hybrid 015D clones, which were cross-bred between

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Acacia mangium and *Acacia auriculiformis*. These clones were propagated from shoot cuttings planted in KHDTK Wonogiri, Central Java, by the Forestry Instrument Standard Testing Centre (BBPSIK), These clones had heartwood percentages ranging from 15% to 25%, as illustrated in Figure 1, with tree diameters and heights detailed in Table 1.

Table 1. Tree diameter and height of clonal Acacia hybrid

Tree code	Diameter (cm)	Height (m)
P1	7	13
P2	8	15
P3	10	17

Fig. 1. Tree discs and wood chip of clonal Acacia hybrid

Wood samples were cut from the middle part of the tree and converted into chips measuring 2 × 2 × 0.2 cm. The middle part had less heartwood proportion than that of the base part as well as higher tree diameter than that of the top part in this study. Some of these chips were subsequently ground into sawdust with a 40–60 mesh size for chemical property testing.

2.2 Density and Fiber Dimension

The basic density was determined by measuring the oven-dry weight of wood chips and dividing it by their wet volume. For defibrillation, chip samples from each tree were cut into matchstick-like pieces measuring 2 × 0.2 × 0.2 cm. These samples were then soaked and heated in a test tube containing a mixture of glacial acetic acid solution and hydrogen peroxide in a ratio of 1:20 until they swelled and turned white. After washing the samples with water to remove any residual solution, they were stained with safranin and left for 24 hours. Specimen preparations were made to be photographed using an Olympus digital microscope.

Fibre dimensions, including the fibre length, cell diameter, lumen diameter, and cell wall thickness, were measured using Image-Pro Plus software based on the images taken using the Olympus digital microscope. Fifty fibres from each tree sample were assessed for these measurements. The values obtained were then used to calculate the Runkel ratio, slenderness ratio, coefficient of flexibility, and coefficient of rigidity using the formulas provided below [10-11]:

$$\text{Runkel ratio} = \frac{Double\ fibre\ cell\ wall\ thickness}{Fibre\ lumen\ diameter} \quad (1)$$

$$\text{Slenderness ratio} = \frac{Fibre\ length}{Fibre\ diameter} \quad (2)$$

$$\text{Coefficient of flexibility} = \frac{Fibre\ lumen\ diameter}{Fibre\ diameter} \quad (3)$$

$$\text{Coefficient of rigidity} = \frac{Fibre\ wall\ thickness}{Fibre\ diameter} \quad (4)$$

2.3 Chemical Properties of Wood

The extractive content soluble in ethanol-toluene was measured following ASTM D1107-96, while the extractive content soluble in hot water was measured following ASTM D1110-84 [12-13]. Holocellulose content was determined using the chlorite acid method, alpha-cellulose content was determined by soaking in a 17.5% NaOH solution, and lignin content was assessed following SNI 0492:2008 [14-16].

2.4 Kraft Pulping

Wood chips (250 g eq. oven-dry weight) were cooked in an electric rotary digester (RKK, 5 l), with an active alkali concentration of 17%, sulfidity of 25%, and AQ concentrations of 0%, 0.05%, and 0.1%. The cooking process was conducted with a liquor-to-wood ratio of 4:1, initially heated to the maximum temperature of 170°C for 45 minutes and then maintained at that temperature for another 120 minutes. After cooking, the pulp was washed to stop the delignification process and remove the remaining cooking solution. The washed pulp was then filtered using a flat screen with a sieve size of 100 mesh to separate the cooked pulp from dirt and uncooked chip residues. The pulp retained on the flat screen was called the residual yield (reject), while the pulp passing through the screen was called the screened yield.

The total weight of the screened pulp was recorded, and a sub-sample was dried to a constant weight at 105°C to determine the moisture content and calculate the screened yield, based on oven-dried chips. The pulp samples were then analysed for Kappa number to indicate the lignin content [17].

2.5 Papermaking Properties

Pulp beating was performed using a Niagara beater to flatten the fibres. The beating degree was measured using the Canadian Standard Freeness Tester [18], targeting a beating degree of 200–300 mL CSF. Laboratory handsheets, 80 g/m^2 in weight and 15.9 cm in diameter, were prepared [19]. The burst, tear, and tensile indices were determined in accordance with SNI ISO 2758:2011, SNI 0436-1989, and SNI 14-0437-1989-A, respectively [20-22].

ISO brightness was measured using a WSB-3 whiteness meter, which operates at an effective wavelength of 457 nm with an half-bandwidth of 44 nm and includes UV content in the irradiation that hits the test sample, in accordance with the CIE standard D56 illuminant [24]. Printing opacity was measured in accordance with SNI ISO 2470-2:2014 [25].

2.6 Data Analysis

Randomised Completely Block Design (RCBD) was used, with the tree diameter serving as the blocking factor. Each treatment was repeated three times. The effects of the treatments were analysed with one-way analysis of variance (ANOVA), with a significance level of 0.05. Post-hoc analysis was carried out using Tukey's Honestly Significant Difference (HSD) test. All statistical calculations were conducted using SPSS Version10.0.

3 Results and Discussion

3.1 Density and fibre dimension

Fibre dimensions are key wood properties that predict the properties of pulp. Three clonal *Acacia* hybrid trees with different diameters were observed for their fibre dimensions, as detailed in Table 2. Compared to the three-year-old clonal *Acacia* hybrid wood obtained from orchards in Wonogiri, Central Java, the clones in this study had lower fibre lengths, fibre diameters, lumen diameters, and flexibility ratios, but they had higher fibre cell wall thickness values, Runkel ratios, slenderness ratios, coefficients of rigidity, and specific gravity values [5]. These differences were probably due to variations in tree age and growing conditions.

Table 2. Fibre dimension, derived fiber values, and basic density of clonal Acacia hybrid wood (10 years)

Physical properties	Tree diameter (cm)			Average	Hybrid 3 years [5]
	7	8	10		
Fiber length (mm)	0.82	0.77	0.90	0.83	0.87
Fiber diameter (μm)	16.04	12.67	13.80	14.17	17.50
Cell wall thickness (μm)	3.60	3.56	3.72	3.63	2.26
Lumen diameter (μm)	8.84	5.54	6.36	6.91	12.99
Runkel ratio	0.93	1.65	1.54	1.37	0.37
Slenderness ratio	52.77	64.10	67.74	61.53	51.10
Flexibility ratio	0.54	0.42	0.44	0.47	0.74
Coefficient of rigidity	0.23	0.29	0.28	0.27	0.13
Basic density	0.43	0.46	0.49	0.46	0.42

The fibres measured from the samples were still relatively short, which would affect the physical properties of the paper produced. The longer the wood fibre, the higher the tear strength of the paper. A Runkel ratio of less than one is recommended for pulp and paper; a Runkel ratio greater than one, as the 1.37 ratio found in this study, will produce paper sheets with low tensile and burst strengths. For the slenderness ratio, the recommended range for pulp raw materials is between 70 and 80 [26]. The samples in this study fell outside this range, which may produce paper sheets with low tear strength. Furthermore, the low flexibility ratio and high coefficient of rigidity observed in this study are expected to produce weaker paper sheets.

3.2 Chemical components

The chemical properties of clonal *Acacia* hybrid wood measured in this study are summarised in Table 3. While the samples in this study had lower extractive content than that observed in the eight-year-old *Acacia* hybrid trees from Gazipur Forest Station, Bangladesh, they had higher holocellulose, α-cellulose, and lignin contents [4]. When compared to three-year-old clonal *Acacia* hybrid wood, these samples had lower α-cellulose and holocellulose contents, while the lignin content was higher [5]. Furthermore, the holocellulose content in this study was lower than that reported for the seven-year-old *Acacia* hybrid trees from South Sumatra, whereas the lignin content was higher [27].

Table 3. Chemical components of hybrid acacia of wood clone (10 years)

Content (%)	Tree diameter (cm)			Average	Hybrid 3 years [5]
	7	8	10		
Ethanol-toluene extractive content	3.90	3.24	4.04	3.73	3.6
Solubility in hot-water	1.26	1.33	0.81	1.14	3.2
Holocellulose	65.57	74.94	71.93	70.82	80.4
α-cellulose	42.16	47.68	45.88	45.24	47.4
Lignin	28.44	30.19	29.00	29.21	26.3

High holocellulose and α-cellulose contents indicate excellent pulp quality and yield [28]. The results further showed low extractive content soluble in hot water and high lignin content, which will affect the brightness degree of the handsheets as well as the pulp yield. High lignin content will require more chemicals in the pulping process [29]. The higher the lignin content, the higher the alkali consumption and Kappa number. In addition, it can also produce darker pulp and paper [30].

3.3 Yield and Kappa number

The screened pulp yield of clonal *Acacia* hybrid wood in this study ranged from 52.74% to 62.08%, whereas the rejects ranged from 0.45% to 1.07%. In addition, the average Kappa number ranged from 15.35 to 26.03. In contrast, the previously studied three-year-old *Acacia* hybrid, using an active alkali concentration of 17% and sulfidity of 25%, had a screened yield ranging from 51% to 52%, a Kappa number ranging from 22 and 26, and rejects ranging from 2.0% to 2.4% [5]. This suggests that the cooking process in this experiment was more effective than in the previous study, likely due to differences in the use of active alkali and the chemical properties of the raw materials used. Specifically, the previous study repported higher holocellulose content (80.40%) and lower lignin content (26.60%) than this study.

Previously, Haque et al. reported that, with an active alkali concentration of 16% and sulfidity of 25%, eight-year-old *Acacia* hybrid wood from Bangladesh

produced screened yield, rejects, and Kappa number of 49.2%, 2.4%, and 28.7, respectively [4]. Applying a different treatment with an active alkali concentration of 18% and sulfidity of 25%, the same raw materials resulted in a total yield and screened yield of 45.9% and a Kappa number of 27.6, without any rejects. These results indicated lower screened yield values and higher Kappa numbers than reported in this study. Variations in active alkali use were thought to significantly affect the yield, rejects, and Kappa number of the pulp. Excessively high concentrations of active alkali can cause overcooking of the pulp, which compromises its properties. On the other hand, too low a concentration of active alkali can result in undercooking [31].

One-way ANOVA revealed that different AQ concentrations did not significantly affect rejects (p = 0.72), but their effects on the screened yield (p = 0.02) and Kappa number ($p < 0.01$) were significant. Tukey's HSD test results, as presented in Figures 2 and 3, add insights into these effects. Increasing AQ concentration from 0% to 0.05% resulted in a 3% increase in the screened yield, and increasing it from 0% to 0.1% produced a 4% increase. Purba et al. [32], previously studying nine-year-old *Acacia mangium* from Suban Jeriji, South Sumatra, with an active alkali concentration of 16% and AQ concentrations of 0.10% and 0.15%, also observed increases in yield and decreases in Kappa number [32]. It is assumed that AQ addition in the pulping process will increase delignification, reduce carbohydrate degradation, and maintain carbohydrate stability, thus increasing the pulp yield [6-7]. However, no significant effect of AQ addition was found in rejects or undercooked chips. The low reject levels, ranging from 0.65% to 0.71%, indicate an effective delignification process. The comparatively high yield of *A.* hybrid suggested that the *A.* hybrid would produces the highest biomass yield at the lowest possible cost to strengthen the forest sustainability.

Kappa number indicates lignin content in the pulp. AQ addition decreases the Kappa number due to the increasing delignification rate and maintained stability of polysaccharides during the pulping process [33-34]. A lower Kappa number indicates more removal of lignin, but it also suggests potential damage to cellulose in the pulp, which can negatively affect the quality of the pulp. For instance, delignification may degrade the pulp strength and complicate the pulp bleaching process [30]. However, the use of AQ at a concentration of 0.1% can reduce the need for bleaching chemicals in *Acacia* hybrid pulp.

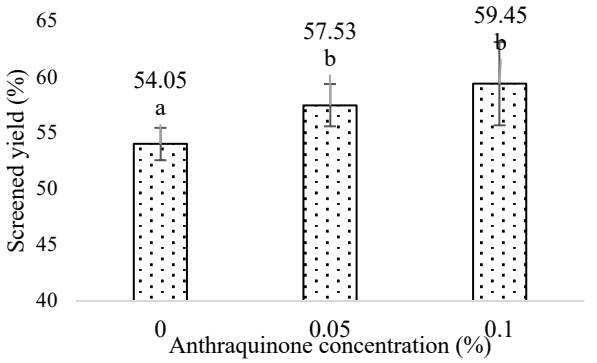

Figure 2. Screened yield pulps of hybrid Acacia wood clone (10 years). The same letters on the same graphic are not statistically different at $p < 0.05$ by Tukey's test.

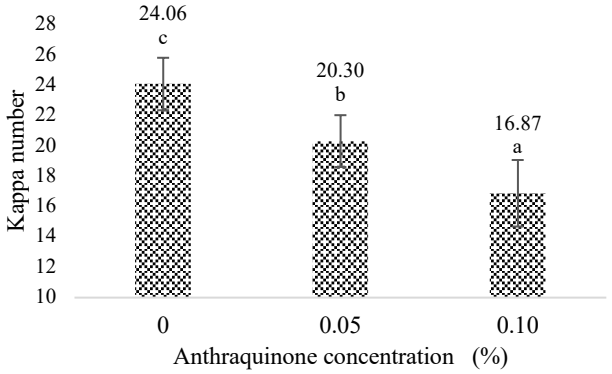

Figure 3. Kappa number of pulps of hybrid acacia wood clone (10 years). The same letters on the same graphic are not statistically different at $p < 0.05$ by Tukey's test.

Increasing AQ concentration from 0.05% to 0.10% significantly improved delignification, thereby reducing the Kappa number by four units. However, it did not significantly increase the screened yield. It is possible that the process of sugar degradation has begun to occur, but this experiment did not measure the pulp viscosity, which is necessary to determine the level of sugar degradation.

3.4 Physical properties of handsheets

The mechanical and optical properties of the handsheets in this study are summarised in Table 4. The burst and tensile indices of the samples met the SNI 6107:2015 standard for broadleaf wood, although the tear index did not [35]. Compared to those of three-year clonal *Acacia* hybrid wood [5], the burst and tensile indices of the handsheets in this study were higher, but the tear index was lower. Similarly, Purba et al. [32] observed the mechanical properties of handsheets from nine-year-old *Acacia* hybrid trees and reported lower burst, tear, and tensile indices compared to those observed in this study [32].

The low tear index might be due to the low slenderness ratio of the wood fibres, ranging from 52.77 to 67.74 (Table 2). Meanwhile, the burst index might be affected by fibre length, kappa number, fibre quality, fibre bonding, and beating time [36-39]. In this study,

the burst index might be partially influenced by the fibre length of clonal *Acacia* hybrid wood, which ranged from 0.77 to 0.90 mm. In addition, the tensile index, which met the Indonesian National Standard, was affected by fibre length, interfibre bonding, interfibre strength, cell wall diameter, and cell wall thickness [40]. The flexibility ratio, or the ratio of the lumen diameter to the fibre diameter, is also thought to play a role. A higher flexibility ratio allows for more contact between fibre surfaces, generating more fibre bonding and consequently stronger paper sheets.

Table 4. Physical properties of handsheets of hybrid acacia wood clone (10 years)

Physical properties	Tree diameter (cm)	Anthraquinone concentration (%) 0	0.05	0.10	Hybrid 3 years [5]
Burst index ($kPa.m^2/g$)	7	4.07	3.67	3.82	
	8	2.99	3.65	3.62	
	10	3.15	3.67	2.50	
	Average	3.40	3.66	3.31	2.94
Tear index ($mN.m^2/g$)	7	2.09	2.51	1.49	
	8	2.90	2.53	1.56	
	10	1.83	2.14	2.12	
	Average	2.27	2.40	1.72	4.82
Tensile index (N.m/g)	7	52.21	51.92	50.02	
	8	50.16	52.75	54.07	
	10	45.86	44.54	46.74	
	Average	49.41	49.73	50.28	43.79
ISO Brightness (%)	7	24.57	25.72	24.83	
	8	23.64	25.23	26,92	
	10	24.34	25.40	25.60	
	Average	24.18	25.45	25.78	24.18
Opacity (%)	7	88.42	87.23	87.70	
	8	86.56	88.53	90.04	
	10	87.16	87.50	87.58	
	Average	87.38	87.76	88.44	94.18

The brightness value in this experiment was almost similar to that of three-year-old *Acacia* hybrid wood pulp, while the opacity was lower [5]. Brightness levels are negatively affected by the presence of residual lignin and extractives in the pulp [41-42]. Additionally, brightness levels are negatively related to Kappa number [43]. In this study, the printing opacity of the handsheets from clonal *Acacia* hybrid wood met the Indonesian National Standard. This was likely due to the lower kappa number or less residual lignin content in the pulp, as lower lignin content typically results in higher print opacity [44].

AQ addition can both increase the tensile index, print opacity, and brightness and decrease the burst and tear indices. However, one-way ANOVA did not demonstrate any significant differences in these properties in this study. It is assumed that the mechanical properties of the handsheets were affected more by physical properties, such as fibre length, fibre size, and fibre direction, than by chemical properties. Similar results were reported by Purba et al. [32], who used AQ concentrations of 0.10% and 0.15% in their study of nine-year-old *Acacia mangium* [32]. In other words, kappa number, which is a chemical property, has no significant effect on the handsheets.

Furthermore, AQ addition did not significantly affect the optical properties of the handsheets, such as print opacity and brightness, either. Despite the significant reduction in lignin content, as indicated by the kappa number, this study suggests that the chemical structure of lignin may be more influential than its content. Previous research noted that lignin content decreased as opacity decreased and brightness increased [45]. Theoretically, the lower the lignin content in the pulp, the higher the brightness of the paper produced [44]. In addition, brightness is also influenced by wood extractives; lower pulp extractive content typically leads to higher brightness of the paper [42].

4 Conclusion

Clonal *Acacia* hybrid wood produced a comparatively high screened pulp yield ranging from 52.74% to 62.08%, with low reject values between 0.45% and 1.07%. Additionally, the average kappa number ranged from 15.35 to 26.03. Furthermore, the burst and tensile indices of the samples in this study met the SNI 6107:2015 standard for broadleaf wood [35]. Increasing AQ concentration from 0% to 0.05% increased the screened yield from 54.05% to 57.53% and decreased the kappa number from 24.06 to 20.30. However, no significant effect of AQ addition on the physical properties of the handsheets was observed. Meanwhile, increasing AQ concentration from 0.0.5% to 0.10% did not significantly affect the screened yield. Based on these results, it is concluded that the AQ concentration of 0.05% is recommended for kraft pulping.
white, and this should be considered when preparing them.

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