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EFFECT OF EXTRACTIVE REMOVAL ON KRAFT PULP PROPERTIES OF *ACACIA AULACOCARPA* WOOD

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Acacia aulacocarpa A.Cunn. ex Benth is considered a potential source of fiber for the pulping and paper-making industries. This study aimed to evaluate the properties of *Acacia aulacocarpa* kraft pulp in relation to the presence of extractive. A separate assessment was conducted on the chemical composition and pulp properties of the sapwood and heartwood. Extractive removal was achieved with various organic solvents including n-hexane, ethanol, and hot water in successive steps. The results showed that heartwood and sapwood had different chemical characteristics affecting pulping outcomes. The total extractive content of heartwood (12.45%) was substantially higher than in sapwood (3.62%) with ethanol soluble being the major fraction ranging from 69% to 86%. Without extractive removal, only slight differences were found in the screened yield, kappa number, viscosity, as well as redness and yellowness values of pulps. Heartwood pulps had more hexeneuronic acid content and were darker than sapwood pulps. After extractive removal, substantial differences were found in screened yield, hexeneuronic acid content, and brightness. The sapwood part showed a considerable increase in screened yield (38.21% to 45.93%) than heartwood (37.21% to 40.92%). This suggested that components of sapwood extractive might inhibit delignification during pulping.

Keywords: Micropulping, pulp wood, heartwood, kraft, kappa number

INTRODUCTION

Acacia and eucalyptus trees are the most important raw materials for the pulp and paper industry in Indonesia. *Acacia mangium* and *Eucalyptus pellita* species have been massively planted in Industrial Forest Plantation. However, paper consumption is continuously increasing worldwide including Indonesia, causing several problems related to the shortage of fibrous raw material in the last decade. To address this issue, various efforts have been carried out to find alternative fibrous resources from locally grown acacia species such as *Acacia auriculiformis, Acacia hybrid* (Yahya et al. 2010), *Acacia nilotica* (Lukmandaru et al. 2019), and *Acacia crassicarpa* (Sugesty et al. 2015, Martins 2020).

A fast-growing species could offer a rational solution as a source of cellulosic material to address the increasing shortage of wood resources. *A. aulacocarpa* A.Cunn. ex Benth.is considered a potential source of fiber for the pulp and paper-making industries, producing one of the strongest bleached kraft pulps among acacias (Deng et al. 2006). This species is naturally found in Indonesia as well as in Australia, and Papua New Guinea (Nielsen 1992). The timber is utilized as construction timber, for furniture and cabinetwork, flooring, etc. This species also have been developed for Industrial Forest Plantations (IFP) in Indonesia (Suprapti & Krisdianto 2006). However, there are no available reports describing the chemical composition and pulping of this species in Indonesia.

Acacia aulacocarpa has dark-coloured heartwood which is assumed to contain high extractive content. Wood extractive poses significant challenges for the pulp and paper industry, negatively affecting the quality and causing a reduction in yield, increased chemical consumption, as well as longer duration in pulping (Lehr et al. 2021). This effect can be attributed to the formation of sticky deposits (Jansson et al. 2009). This extractive is mainly removed through chemical pulping but this leads to increased consumption of cooking chemicals due to their competition with lignin, hindering its removal (Gardner & Hillis 1962). Despite these challenges, there have been limited studies to explore the effect of extractive removal. Therefore, this study aimed to evaluate extractive removal and its impact on the pulp properties.

MATERIAL AND METHODS

Wood material

A total of three *A. aulacocarpa* trees were felled at the age of 27 years in the Wanagama Educational Forest, located in Gunungkidul Regency, Jogjakarta Province, Indonesia. Wood discs were taken at different stem heights. The heartwood was clearly distinguished on the cross-section by its dark brown colour (Figure 1). The sapwood and heartwood fractions were separated on each wood disc by drilling. The wood samples were ground into meals, and sieved, with 40–60 mesh fractions used for chemical analysis and the 20–40 mesh for micropulping (Lourenço et al. 2008). Each heartwood and sapwood from different trees were mixed to produce homogenous samples.

Chemical composition

The total extractive content was determined successively using *n*-hexane (6 h), ethanol (6 h), and hot water (2 h). Ethanol and *n*-hexane (technical grades) extraction was conducted in a Soxhlett apparatus with extraction thimbles while hot water extraction was performed in a water bath by refluxing. The extractive content was measured gravimetrically after each solvent extraction. Holocellulose and insoluble lignin were determined in the extractive-free material according to the modified chlorite method (Browning 1967) and ASTM D1106-84, respectively. Cellulose content was assessed by the 17.5% NaOH treatment (Rowell et al. 2005), while hemicellulose was determined by subtracting holocellulose from the cellulose content. Ash content and solubility values in 1% NaOH were determined by ASTM D1102-1984 and ASTM D1109-84, respectively. All analyses were made in duplicate and average results were reported as the percentage of initial mass.

Micropulping

The heartwood and sapwood meal samples were separated into two parts, one to be pulped directly, and the other to be pulped after extraction (successively with *n*-hexane, ethanol, and hot water). Pulping was carried out in 125-mL stainless reactors immersed in an oil bath at a maximum temperature of 170 °C, using 10 g (eq. oven dry weight) of air-dried wood meals (Neiva et al. 2015). The pulping conditions were as follows: active alkali of 25% (as Na_2O), sulfidity of 30%, liquor-to-wood ratio of 6:1, and duration of 2 hours at maximum temperature. The calculated H-factor in heat-up time was 88 and in cooking was 1838 (in total: 1926). After the reaction, the reactors were cooled in an ice bath. The pulps were subsequently filtered (200-mesh sieve), thoroughly washed with water, and

Figure 1 A cross section of *Acacia aulacocarpa* tree (base part)

air-dried at room temperature. Screened yields were determined based on the oven-dry mass of wood meal charged to the reactor, and all pulping experiments were replicated (duplo).

Pulp characterisation

The *A. aulacocarpa* pulps were extracted by ethanol/toluene (1:2, v/v) using a Soxhlet extraction system to remove residuals of extractable materials. After that, the pulps were evaluated for kappa number method (TAPPI standard T2 14m-50), and viscosity (TAPPI T230 om-08). Hexeneuronic acid content was measured using the spectrophotometric method (Chai et al. 2001).

Colour measurement

The colourimetric parameters of the air-dried wood meal and pulp sheet were determined by the CIE-L*a*b* system through a portable spectrophotometer NF333 spectrocolourimeter (Nippon Denshoku Ind. Co Ltd.). The parameters of color determination were as follows: an opening diameter of 6 mm, standard illuminant D65, a 10° observation angle, and a tungsten halogen light source was used. A total of three measurements were made for each sample and the value L* described psychometric lightness (0 = black to 100 = white). The value a* indicated the redness from the axis red (+) to green (–). Meanwhile, the value b* described the yellowness from the Y axis yellow (+) to blue (–).

RESULTS AND DISCUSSION

Chemical composition of wood

The chemical analysis of sapwood and heartwood from *A. aulacocarpa* is summarised in Table 1. The total extractive content of sapwood (3.62%) and heartwood (12.45%) differed significantly. The ethanol-soluble extractive was the major fraction, accounting for more than 86% of the total extractive (69% in sapwood). This high concentration might be attributed to the age of the tree (27 years). According to a previous study, the darker colour of heartwood is due to secondary metabolites such as the extractive (Kampe & Magel 2013). Experiments by Lukmandaru (2012) in *A. mangium* and Arisandi et al. (2020) in *E. pellita* showed that heartwood was found to contain significantly more extractive than the surrounding sapwood.

The heartwood had slightly higher levels of lignin (29.68%) but lower levels of holocellulose (73.94%) and cellulose (45.71%). On the other hand, sapwood had more 1% NaOH solubility (17.84%) and ash (0.81%) contents. For the same species (17 years) grown in Thailand, high cellulose (65%), low lignin (18–20%), and ash contents (0.02–0.04%) were observed by Atiwannapat (2009). Furthermore, there are several published references on the chemical composition of other Acacia species grown in Indonesia. Yahya et al. (2010) reported 5.38% alcohol-benzene extractive, 31.3% Klason lignin, and 45.1% cellulose for *Acacia mangium* (7 years) as well as 5.96% alcohol-benzene extractive,

Table 1 Chemical properties of sapwood and heartwood of *Acacia aulacocarpa*

Chemical properties	Sapwood	Heartwood
Extractives		
- *n*-hexane	0.46	0.63
- ethanol	2.50	10.74
- hot-water	0.65	1.07
Holocellulose	76.92	73.94
α-Cellulose	47.83	45.71
Hemicellulose	29.09	22.23
Lignin	27.40	29.68
Ash	0.81	0.43
Solubility in 1% NaOH	12.61	17.84

Table 2 Pulp properties of sapwood and heartwood of *Acacia aulacocarpa* with extractives removal treatment by organic solvents

Pulp properties	Sapwood	Extracted sapwood	Heartwood	Extracted heartwood
Screened yield (%)	38.21	45.93	37.21	40.92
Kappa number	23.24	18.95	23.78	20.00
Viscosity (ml/g)	831	709	871	734
Hexeneuronic acid content (µmol/g)	2.54	14.39	4.89	21.72

34.1% Klason lignin, and 40.5% cellulose for *Acacia auriculiformis* (7 years). Martins et al. (2020) found 44.4% cellulose and 27% Klason lignin in *Acacia crassicarpa* (4 years).

Pulp characterisation without extractives removal

Table 2 summarises the extractive removal by organic solvents of the sapwood and heartwood fractions used for the pulping experiments. The results of the kraft pulping showed an overall yield of 37–45% and moderate residual lignin (mean kappa number of 19–24). Yield values of 46.95% with a kappa number of 18.62 were reported by Deng et al. (2006) for the same species. Similarly, pulp yield values of 31–46% with a kappa number of 12–26 were recorded by Mahdiyanti and Marsoem (2015) using sapwood and heartwood of *A. mangium* as the raw materials. Lourenço et al. (2008) found yield values of 56.2% (sapwood) and 52.9% (heartwood) with a kappa number of 7 (sapwood) and 11 (heartwood) for *A. melanoxylon* pulps.

There were minor variations between the pulping as summarised in Table 2, with heartwood producing 1% less pulp than sapwood. This suggested that, given the pulping conditions utilized, heartwood had no impact on the degree of delignification. Consequently, there was a minimal impact on cellulose, and pulp viscosity values were found to be comparable. The mean pulp viscosity in this study was higher than the values reported by Lourenço et al. (2008) for the *A. melanoxylon* at 666 ml g^{-1} and 756 ml g^{-1} and 666 ml $g^{-1}$for sapwood and heartwood, respectively. The content of hexeneuronic acid groups was higher in heartwood than in sapwood (4.8 µmol g^{-1} and 2.5µmol g^{-1}, respectively), but was below the values for *Acacia melanoxylon* pulps (11–45 µmol g^{-1}). The presence of hexeneuronic acids in pulp has detrimental effects on bleaching and influences the estimation of the kappa number (Sjḍstrḍm 2006). Nevertheless, this fact could be advantageous for the bleachability of pulps made from acacia wood, depending on the bleaching reagents and the sequence utilised.

The slight differences between heartwood and sapwood in yield, kappa number, and viscosity were unexpected. A previous report on *A. melonoxylon* showed that the heartwood part with higher extractive content had lower values of pulp yield, and brightness but higher kappa number, hexeneuronic acid content, and viscosity compared to sapwood (Lourenço et al. 2008). The difference between heartwood and sapwood pulp yields was larger (40.0% vs. 49.7%) but not on kappa number for *Pinus pinaster* pulps (Esteves et al. 2005). The kappa number indicates the residual lignin in pulp and estimates the degree of delignification. A significant effect of heartwood on kappa number was observed but not on screened yield for *A. mangium* pulps (Mahdiyanti & Marsoem 2015). When comparing the amounts of cellulose and ash, as well as the water retention value, sapwood pulp was significantly greater than heartwood pulp of *Leucaena leucocephala* (Pydimala et al. 2019). It might be because of the variations in their extractive and lignin compositions.

Based on the result, the higher extractive content in the heartwood samples (12.42% vs 3.6% in sapwood) did not affect pulp properties except for hexeneuronic acid content, indicating the influence of other factors. The higher levels of 1% NaOH solubility in the sapwood parts (17.84%) might be attributed to the variation of screened yield and kappa number levels. This solubility indicated the amount of dissolved low molecular weight sugars which could reduce the screened yield.

Effect of extractives removal

The negative influence of extractive in pulping was reviewed by Lehr et al. (2021). The objectives of wood extractive removal are to avoid problems during pulping and to improve pulp properties (Koch 2006, Silverio et al. 2011, Umezawa 2000). Extracted wood meals were assumed to be more susceptible to delignification. The increase in screened yield and decrease in kappa number were expected after extraction. Pulp yield for extracted heartwood increased slightly (37.21 vs 40.93%) while for sapwood, it increased significantly (38.21 vs 45.83%). Kappa number and viscosity decreased with extractive removal which the heartwood parts showed slightly higher levels than those of the sapwood parts.

Several extractives affected the sulfonation reactions, competing with lignin and increasing cooking liquor consumption (Gardner & Willis 1962). It was assumed that the competitive reaction with lignin was less pronounced to produce more intense delignification after removal. Another cause might be the outcome of the solvent extraction and the treatment temperature (100 °C for water extraction) which serve as a pre-treatment to remove lignin in the subsequent pulping (Esteves et al. 2005). With a severe treatment condition, the effect of hot-water extraction using a digester (145–160 °C) on soda pulping of aspen woodchips showed part of the hemicellulose and lignin from virgin woodchips was dissolved in the extraction liquor (Lu et al. 2012). Furthermore, compared with the control sample, the overall pulp yield, rejects, and kappa number for extracted woodchips decreased while viscosity of the pulp increased. Based on the result, the sapwood with lower extractive content had a more considerable effect on screened yield than that of heartwood after extraction. This could be due to the type of extractive in the sapwood, particularly from the major fraction namely ethanol, having a more profound effect on accelerating delignification.

Viscosity in various pulping methods is a significant predictor of pulp damage. Positive correlations were observed between viscosity and several paper strengths in kenaf pulp Umezawa (2000). The lower values after extraction in the both parts were presumably due to the extractive in the pulp having a protective effect on viscosity. Ohtani et al. (2001) suggested that hot-water soluble extractives consumed a significant amount of alkali during cooking, but acted as a protector of hemicellulose of kenaf cores. On the other hand, there was a significant increase (4–5 times) after extraction in the content of hexeneuronic acid groups, with heartwood (21.72 μmol g^{-1}) showing higher values than sapwood (14.39 μmol g^{-1}). The extractive removal might accelerate delignification and intensify the modifications in the methyl-glucuronic acids present in xylans. It is assumed that hexeneuronic acid contributes to the decreased brightness stabilities of bleached pulps (Sixta et al. 2006). This results in pulp and paper losing their whiteness and requiring more chemical reagents during the process.

Colour properties

The visual distinction between sapwood and heartwood meals was quantified with colour parameters (Figure 2). Sapwood had a higher brightness index (L*) than heartwood (65 and 49), but lower redness (a*) (7.5 and 17.8), and yellowness (b*) (21.2 and 29.9). After wood meal extraction, only slight differences were found in brightness and yellowness for sapwood. Considerable differences were observed in all parameters for heartwood and redness in sapwood. The redness in sapwood and brightness levels in heartwood were higher but redness and yellowness levels in heartwood parts of extracted powders were lower compared to original wood meals. This indicated the presence of soluble colouring substances in both samples of *A. aulacocarpa*. However, no study has been conducted to explore the phenolic content and its effect on colour properties. Several flavonoids have been isolated in *Acacia mangium*, and *A. auriculiformis* (Mihara et al. 2005) as well as *A. confusa* heartwood (Chang & Chang 2019).

The dark colour of unbleached kraft pulp is mostly from the residual lignin (Shin et al. 2019) but the brightness can also be affected by extractives (Jansson & Nilvebrant 2009). Based on the result, the brightness of heartwood pulps averaged 52, considerably below that of sapwood namely 67 (Figure 2). The redness and yellowness indices between both samples showed only a slight difference. For wood meals converted into pulp without extractive removal, brightness slightly increased, while redness and yellowness

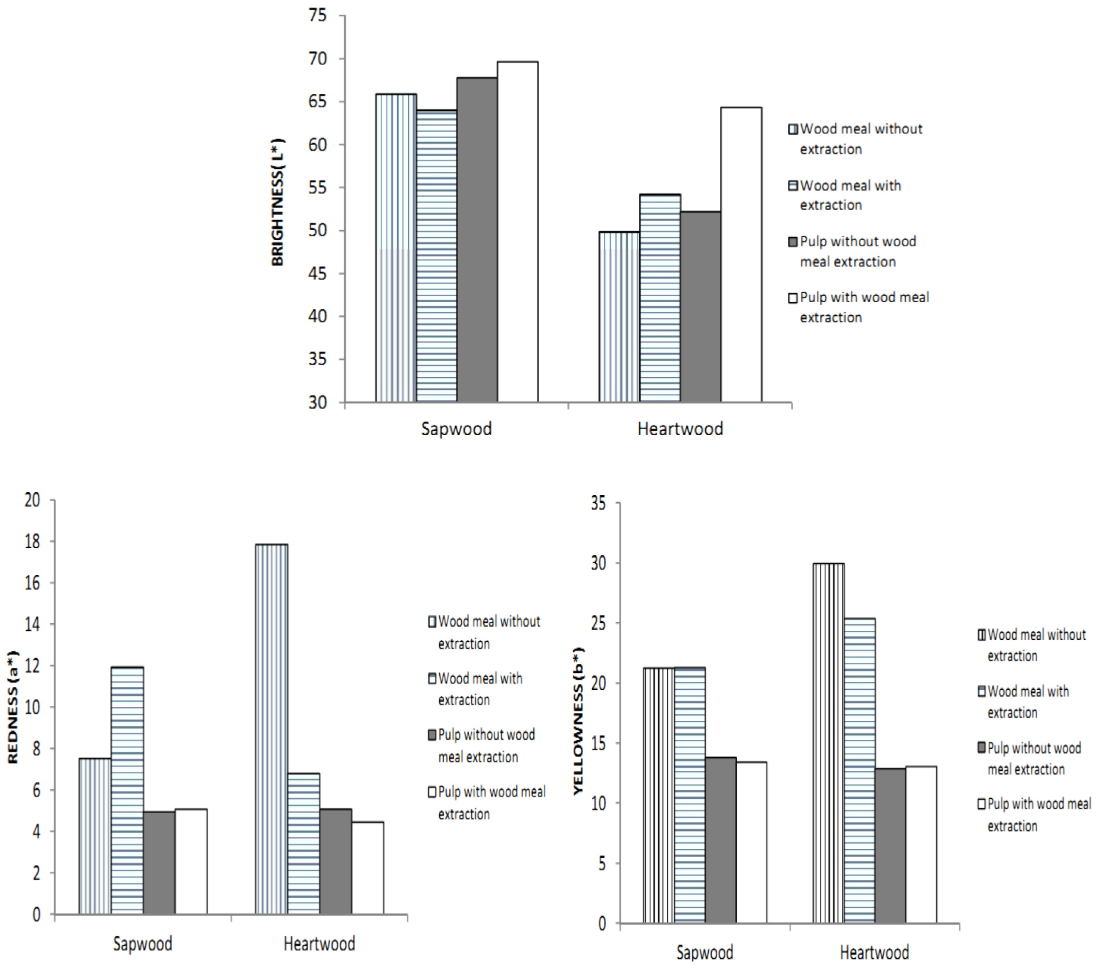

Figure 2 Colour properties of sapwood and heartwood of *Acacia aulacocarpa* with extractives removal treatment by organic solvents

considerably decreased in both heartwood and sapwood parts. Except for brightness, a similar pattern was observed for wood meals converted into pulp with extractive removal. In the context of brightness, significant differences were observed after extractive removal.

In an earlier study, the chromophore groups of aspen kraft pulp were reduced significantly by extracting with organic solvents to remove residual extractive (Shin et al. 2009). Compared to the original wood, the pulps of *Acacia melanoxylon* showed a more uniform colour with a reduction in *a** and *b** parameters and an increase in *L** (Lourenço et al. 2008). There huge differences between heartwood and sapwood pulps were found mostly by a lower brightness of heartwood pulps. In this study, comparison between pulps from the wood meal, with and without extractive removal generally showed no substantial differences in sapwood. A significant increase in brightness and a slight decrease in redness were found for the heartwood parts implying the extractive affecting brightness were largely removed during the extraction. Therefore, these soluble extractives should be analysed in future studies. The higher brightness of heartwood pulps has a positive impact as it results in lower consumption of bleaching chemicals. During bleaching, improved bleachability and less chemical consumption to reach the same brightness levels were achieved by extracting wood with organic solvents even before kraft cooking (Baptista et al. 2006).

CONCLUSIONS

In conclusion, when extractive was not removed, only a slight difference occurred in pulp properties between heartwood and sapwood in relation to screened yield, kappa number, viscosity, redness, and yellowness. The removal of extractive from wood meals resulted in higher yield, especially in the sapwood part, and also intensified delignification indicated by the decrease in kappa number. However, the decrease in viscosity and increase in hexeneuronic acid contents indicated that extraction might have negative effects on paper strength and bleaching. For colour properties, extractive removal almost showed no effect in sapwood pulp but significantly increased brightness in heartwood. Therefore, further studies are needed to investigate the extractive, contributing to delignification and colour.

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