Chemical Components of Boiling-Treated Sengon (*Paraserianthes falcataria* (L.) I. C. Nielsen) Wood

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Abstract

Sengon is a fast-growing wood species widely distributed on Java Island and commonly utilised for plywood production. It is frequently subjected to boiling treatment to reduce veneer defects, making it important to understand how this treatment affects its properties. This study investigated the effect of boiling treatment on the properties of sengon wood, involving three seven-year-old trees which were subjected to a control and an experimental treatment at 80°C for ten hours. The wood specimens were divided into near-pith, middle, and near-bark sections. The specimens were powdered to a size of 40–60 mesh for successive extraction with n-hexane, methanol, and hot water. The total phenolic content was measured from the methanol-soluble extract, and the total polysaccharide content was measured from the hot-water-soluble extract. Chemical components such as holocellulose, alpha-cellulose, hemicellulose, lignin, ash, and silica contents, as well as pH values, were determined. T-test revealed that boiling treatment significantly reduced extractive levels (in ethanol-toluene and methanol solubles) but significantly increased total phenolic and polysaccharide contents. No significant effect was observed on cell wall chemical components. One-way ANOVA indicated that the radial direction significantly affected methanol-soluble extractive content, total phenolic content, and total polysaccharide content.

Keywords: Boiling treatment, radial direction, chemical properties, wood extractives, *Paraserianthes falcataria (*L.) I.C.Nielsen.

Introduction

Sengon (*Paraserianthes falcataria* (L.) I. C. Nielsen) is one of the most popular tree species, particularly prevalent in community forests on Java island. Known for its fast growth, it is capable of producing large volumes of wood under favorable conditions (Amelia *et al.* 2021). These fast growth and high yield make sengon wood the main raw material in the plywood industry (Alim and Suseno 2022).

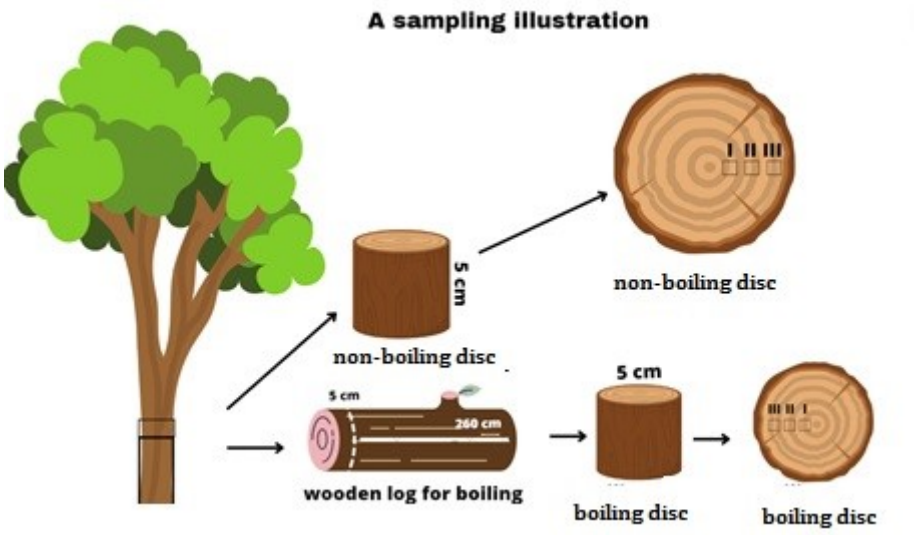
Sengon wood, used as a plywood material, is prone to have physical defects following felling due to the high growth stress experienced by standing trees. These defects reduce the price or potential added value of the wood (Marsoem *et al.* 2014). Additionally, cracks in the logs can compromise the yield during the wood peeling process. Therefore, a special treatment to minimise these risks is necessary. For example, soaking wood in hot water at 80°C for 10 hours has been reported to reduce wood damage during peeling (Mazela *et al.* 2004). Technically, wood boiling affects not only the wood's physical properties but also its chemical properties.

Boiling treatment is believed to improve the quality of veneer by improving the gluing process through the reduction of extractive levels (Widyorini and Puspitasari 2009). Previous research on kamper and keruing woods showed that boiling treatment significantly reduced the extractive contents soluble in alcohol-benzene, hot water, and cold water (Wahjudi 1990). However, the effect of boiling treatment on sengon wood properties has yet to be widely investigated. Thus, this research aimed to find out how boiling treatment could alter the chemical components of sengon wood.

Materials and Methods

Materials and extraction

Three seven-year-old sengon trees were harvested from a community forest in Kedung Pomahan Kulon Hamlet, Kemiri District, Purworejo Regency, Central Java, by cutting at the bottom part. The logs were treated with soaking and boiling at 80°C for 10 hours (Fig. 1). The temperature applied followed previous temperature application to reduce the tree growth stress (patent number IDS000005180). After boiling, the logs were air-dried for approximately four hours and cut into 5 cm thick discs from the end parts (Fig. 2). The samples were then drilled near the bark, at the center, and near the pith at the radial direction and grounded to produce a 40–60 mesh powder size. A comparison was carried out between treated and untreated wood samples (control).



Note: (I): near the pith, (II): center, (III): near the bark.
Figure 1. Scheme of wood sampling.

Figure 2. The boiling process of the 7-year-old sengon log

Determination of wood chemical components

Two extraction methods were applied to the sengon wood samples. The first method involved successive extraction of 2 g of dry wood using *n*-hexane, methanol, and hot-water solvents. The *n*-hexane and methanol extractions used a Soxhlet apparatus for six hours, while the hot-water extraction used a water bath at 100°C for approximately three hours. The extract filtrates were evaporated to obtain dry extracts. The *n*-hexane extractive content (HEC), methanol extractive content (MEC), and hot-water extractive content (WEC) were summed up to obtain the total extractive content (TEC). The second method involved successive extraction of 2 g of wood powder using ethanol-toluene solvent (ASTM D1107-96 and hot water (ASTM D 1110-96).

The wood extracts obtained using the first extraction method were analysed for total phenolic and polysaccharide contents, while the wood residue obtained using the second method was subjected to cell wall component determination. The total phenolic content was determined using the Folin-Ciocalteu method, with absorbance read at 765 nm using a UV-Vis spectrophotometer (WPA 800) (Gao *et al.* .2007). Meanwhile, the total polysaccharide content was determined in the hot-water-soluble extract (1000 ppm) using the phenol-sulfuric acid method, with absorbance measured at 490 nm (Dubois *et al.* 1956).

The extractive-free wood powder residue from the second method was used for determining the holocellulose, cellulose, and lignin contents. Holocellulose content was determined using the acid chlorite method (Browning 1967).

Cellulose content was determined using 17.5% NaOH immersion (Rowell 2005). Hemicellulose content was calculated as the difference between holocellulose and α-cellulose contents (Lukmandaru *et al.* 2018). Lignin content was determined using the Klason method, as specified by SNI 0492: 2008, involving a treatment with 72% sulfuric acid.

Ash and silica contents were measured based on SNI 14-1031-1989. The pH value was measured by soaking 1 g of wood powder in distilled water for 48 hours. After filtering, the pH value of the filtrate was measured using a pH meter (in duplicate).

Statistical analysis

The effects of boiling treatment factors on chemical components and extractive contents were analysed using t-test, while radial variation was analysed using one-way ANOVA (analysis of variance). Tukey's Honestly Significant Difference (HSD) was used for post-hoc analysis (Steel *et al.* 1997)

Results and Discussion

Chemical properties

Sengon wood is primarily composed of carbohydrates, including holocellulose (which consists of cellulose and hemicellulose) and pectin, as well as lignin. The primary components of sengon wood in this study are detailed in Table 1. The holocellulose content was within the range observed for sengon wood from West Kalimantan (Putra *et al.* 2018), whereas the α-cellulose content was higher (47.99–48.91%) than that observed for five-year-old sengon wood from West Java (Pari 1996). Furthermore, the sengon wood in this study had higher lignin content (26.58–30.81%) compared to the value reported for sengon wood from plantations in West Java (Pari and Hartoyo 1990). Based on three individual trees, the values of lignin content is within the range of 5-year-old sengon wood (Pari 1996). However, the range of lignin in this study is smaller than that of Windeisen *et al*. (2003) on teak wood from Panama (33.3-38.3%).

In this study, total extractive content was calculated by summing the extractive levels derived from successive extractions using *n*-hexane, methanol, and hot water. As presented in Table 3, the total extractive content in this study ranged from 5.23% to 11.57%, which was higher than those reported in other studies (Pari 1996; Martawijaya *et al.* 1989). The total phenolic content in the methanol-soluble extract in this study was higher than that of six- and eight-year-old Jati Unggul Nusantara wood, while the total polysaccharide content in the hot-water-soluble extract was lower (Rahman and Lukmandaru 2022).

Ash is an inorganic residue derived from various salts deposited in the cell wall and lumen (Sjostrom 1995). Silica, a major constituent of ash, is more varied in quantity in tropical broadleaf species (Shmulsky and Jones, 2011). In this research, the ash content ranged between 0.78% and 1.04%, which was lower than the 0.64% ash content found in five-year-old sengon wood and fell within the range of 0.37–0.50% for sengon wood from West Java plantations (Pari 1996; Pari and Hartoyo 1990). Ash content can be affected by the age of the wood, with higher ash content found in older wood (Pari and Saepuloh 2000). Additionally, silica content higher than 0.3% can accelerate saw blade dulling (Shmulsky and Jones 2011). In other words, the seven-year-old sengon wood in this study might negatively affect sawing tools as most samples had silica levels above 0.3%. Thus, a special treatment is needed to reduce the silica content.

The pH value of sengon wood in this study ranged from 5.83 to 6.06, which fell within the acidic range. This range is comparable to that found for mahogany wood (5.20–5.94) (Lukmandaru *et al.* 2018). Technically, a low pH value will accelerate the curing of UF adhesive in the gluing process during hot pressing for particleboard (Maloney 1993).

Boiling treatment

The t-test results provided in Table 2 showed that boiling had no significant effect on any of the wood cell wall components. It can be assumed that the carbohydrate components of the sengon wood in this study were not degraded, likely due to the low temperature used in the experiment. In contrast, Ates *et al.* (2009) demonstrated that heat treatment at 130°C for 2–8 hours had a significant effect on the degradation of wood carbohydrates in *Pinus brutia*. The finding of this study is in line with that of Ismanto and Saputro (2014), who found almost no change in cellulose content after boiling treatment of rubber wood. Similarly, lignin content did not indicate any significant difference after the boiling treatment due to the same reason.

Wood extractive contents were determined by two extraction methods. The t-test results showed that boiling treatment had no significant effect on the extractive contents soluble in hot water and *n*-hexane, and neither did it on the total extractives (Table 3). Although the extractive content soluble in *n*-hexane increased from 1.39% to 1.63% after boiling, the increase was not considered statistically significant. As *n*-hexane naturally dissolves terpene and fat groups, the lack of significant change in the extractive content in *n*-hexane indicates that these groups are not sensitive to boiling treatment at 80°C for 10 hours. In addition, although the average hot-water-soluble extractive content and total extractives decreased after boiling, these decreases were not significant. This is in contrast to an earlier study by Wahjudi (1990), which reported decreases in both hot-water-soluble and total extractive contents of kamper and keruing woods after boiling at 100°C.

Boiling significantly reduced the extractive contents soluble in ethanol-toluene and methanol, while it significantly increased the total phenolic and polysaccharide contents. After boiling, the extractive content soluble in ethanol-toluene decreased to 3.5%, compared to 4.36% in control. This pattern was also observed by Lukmandaru *et al.* (2018), who investigated the alcohol-benzene extractive content of

mahogany wood using the steaming method at temperatures of 90°C, 120°C, and 150°C for two hours. The observed reduction in extractive content might be due to the loss of volatile substances during heat treatment. Previous research reported that volatile substances were lost from *Pinus pinaster* wood as other components were degraded after heat treatment at 260°C (Bourgeois and Guyonnet 1988). Ates *et al.* (2009) also observed that extractives, mostly volatile compounds, were lost during heat treatment. Methanol extractive content decreased from 3.97% to 2.99% after boiling, a more pronounced change compared to that reported for hot water, which is a relatively more polar solvent than methanol. Methanol technically dissolves various compounds in wood, such as flavonoids, tannins, carotenoids, and other phenol compounds. This suggests that boiling treatment has a more significant impact on wood phenolic components with lower molecular weight.

Table 1. Comparison of chemical components of sengon wood with previous studies.

Chemical properties	This research	Previous research results		
		Putra *et al.* (2018)	Pari & Hartoyo (1990)	Pari (1996)
Holocellulose (%)	74.86-76.21	69.16-88.33	59.41	70.9
α-cellulose (%)	47.99-48.91	57.11-74.21	46.31	46.62
Hemicellulose (%)	26.84-27.30	-	-	-
Lignin (%)	26.58-30.81	16.69-23.77	25.14	29.1
Ash (%)	0.78-1.04	-	0.81	0.64
Silica (%)	0.37-0.50	-	0.13	0.5
pH	5.83-6.01	-	-	-
Total extractive content (%)	5.23-6.01	-	5.39[1]	2.06-4.34[1]

Note: (a) Putra *et al.* (2018) (sengon wood from West Kalimantan with a diameter of 33 cm), (b) Pari & Hartoyo (1990) (sengon wood from West Java plantations), (c) Pari (1996) (5 year old sengon wood from West Java), (1) : Ethanol-benzene extractives.

Table 2. Average value of chemical components of sengon wood

Chemical properties	Control	Boiling
Holocellulose (%)	76.07	74.73^{NS}
α-cellulose (%)	48.72	48.06^{NS}
Hemicellulose (%)	27.35	26.67^{NS}
Lignin (%)	29.63	28.19^{NS}
Ash (%)	1.04	0.75^{NS}
Silica (%)	0.61	0.26^{NS}

Note: NS: Not significant in t-test.

Table 3. Average value of extractive content and pH value of sengon wood

Chemical properties	Obtained values		Treatment	
	Minimum	Maximum	Control	Boiling
Ethanol-toluene content (%)	2.68	4.92	13.09	10.5*
Hot water extractive content (%)	2.51	2.8	8.79	7.2^{NS}
n-heksane extractive content (%)	0.25	2.84	4.17	4.89^{NS}
Methanol extractive content (%)	2.1	8.59	11.92	8.97*
Hot water extractive content (%)	1.71	4.25	11.92	7.06^{NS}
Total extractive content (%)	5.23	11.57	23.2	20.9^{NS}
Total phenolic content (gallic acid equ./g sample)	16.01	277.05	259.34	421.52*
Total polysaccharide content (glucose equ./g sample)	43.96	155.16	149.988	337.76*
pH values	5.83	6.01	5.97	5.88^{NS}

Note: NS: Not significant in t-test, (*): highly significant in t-test at the 5% test level

The amount of total phenolics content increased from 86.45 (GAE/g sample) to 140.51 (GAE/g sample) after boiling. It is assumed that lignin compounds are degraded in the boiling process. However, no significant effect of boiling was found in lignin content (Table 2). It is assumed that the boiling factor with a temperature of 80 ℃ did not cause lignin degradation. Hemicellulose degradation starts at 200-260℃ while cellulose at 240-350 ℃ and lignin at a wider temperature range (280-500℃) (Nawawi *et al.* 2018). The increase of the total phenolic content might be due to the degradation of phenol constituents in the form of phenolic acids contained in the hydrophilic part of wood extractives.

This trend was also observed on reeds boiling (Dhyanaputri *et al.* 2022). Another work (Tan *et al.,* 2014) showed that water can be used effectively to extract phenolic compounds in bitter melon where the optimal condition for water extraction by a single extraction at 80 ℃ for 5 minutes. Phenolic compounds are known to be sensitive to heating even though in a short time treatment (Ismail 2004),. The heat-sensitive nature could have a different effect, both increasing and decreasing the total phenolic content.

Total polysaccharide content increased from 50 GAE/g to 112.59 GAE/g sample after boiling treatment. In this study, cell wall components such as cellulose, hemicellulose, holocellulose, and lignin did not give significant differences after boiling treatment (Table 2). This suggests that the increase in total polysaccharide content after boiling is not caused by the degradation of wood cell wall components. Theoretically, softwood consists of galactoglucomannan (about 20%), arabinoglucoronoxylan (5%-10%), arabinogalactan, and other small amounts of polysaccharides (pectin and starch compounds) (Sjostrom 1998). Therefore, the increase in total polysaccharide content is probably due to the degradation of other components of small polysaccharides such as pectin and starch compounds. Starch is a polysaccharide in the form of a polymer of α-D-glucose (Erika 2010). Starch consists of two fractions that can be separated with hot water. The soluble fraction is called amylopectin and the insoluble fraction is called amylose (Hee-Young 2005). Generally, starch contains 15-30% amylose, 70-85% amylopectin, and 5-10% intermediate materials (Zulaidah 2012). The starch fractions dissolved in this hot water during boiling caused the high total polysaccharide content.

Radial direction

One-way ANOVA results showed that radial direction had no significant effect on any of the wood cell wall components, including alpha-cellulose, holocellulose, hemicellulose, lignin, silica, and ash contents. This finding contrasts with that of Jahan *et al.* (2019), who investigated the effect of radial direction on ten-year-old *A. auriculiformis*. In addition, the pH value was found to be related to ash content, particularly in terms of the alkaline properties of its main components: Ca, Mg, and K (Panshin and de Zeeuw 1981). However, pH value does not fluctuate along radial direction. The low pH value near the pith or heartwood was generally due to the presence of phenolic compounds, which have weak acidic properties (Krilov and Lasander 1998). The pH value observation revealed that ash and phenolics may not be the only factors influencing pH value.

These one-way ANOVA results further indicated that radial direction had no significant effect on the extractive contents soluble in hot water, *n*-hexane, and methanol, as well as the total extractives. This suggests that sengon wood has a relatively uniform extractive composition along the radial direction. This absence of a significant effect might also be attributable to the unclear division between the heartwood and sapwood. In general, sengon wood has white heartwood and white sapwood, which makes it difficult to visually distinguish them (Martawijaya *et al.* 1989).

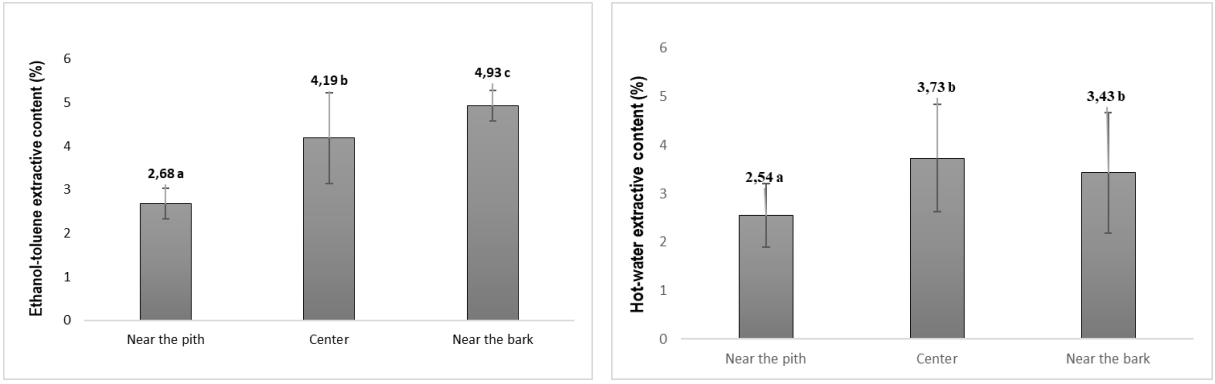


Figure 3. Radial direction of ethanol-toluene extractive content and hot-water extractive content in sengon wood. The same letters on the same graphic are not statistically different at $p < 0.05$ by Tukey's test.

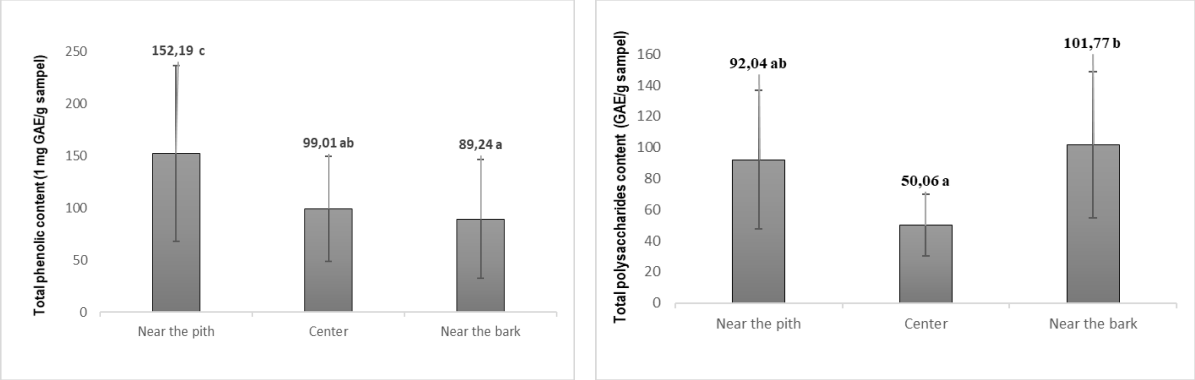


Figure 4. Radial direction of total phenolic and total polysaccharide contents in sengon wood. The same letters on the same graphic are not statistically different at $p < 0.05$ by Tukey's test.

Radial direction had significant effects on the extractive contents soluble in toluene-ethanol and hot water, as well as the total phenolic and total polysaccharide contents. Tukey's test showed the highest ethanol-toluene-soluble extractive content near the bark (Fig. 3), which is at odds with the finding of Shmulsky and Jones (2011). This result indicates a higher concentration of non-polar components in this part of the wood. It is likely that heartwood in seven-year-old sengon wood is not present near the pith. Heartwood formation is characterized by the conversion of polysaccharides from the sapwood into phenolics (Nobuchi *et al.* 2005). In addition, the lowest level of hot-water-soluble extractives was observed near the heartwood. This pattern is in agreement that substances soluble in water such as starch and pectin are more abundant in the sapwood near the bark than in the heartwood (Fengel and Wegener 1984). The highest average value of total phenolic content was found near the pith (152.19 GAE/g sample) (Fig. 4), which might be related to heartwood formation. Earlier work by Datta and Kumar (1987) on teak wood showed that the highest amounts of phenolics were found in the heartwood, which decreased towards the bark and were completely depleted in the outer sapwood. Similarly, Asdar *et al.* (2016) observed the highest ethanol-toluene-soluble extractive content in the heartwood of ebony wood, which tended to decrease towards the sapwood. This decrease was due to the accumulation of secondary metabolites, such as phenolics, in the cell wall and lumen of the heartwood.

The highest total polysaccharide content of 101.77 GAE/g was obtained near the bark. Tukey's HSD showed a significant difference between the center and the part near the bark. Similarly, the highest concentrations of hot-water-soluble extractives were found in the same region. The sapwood is generally richer in nutrients such as sucrose and wood glycosides, while the heartwood and bark are usually richer in secondary metabolites (Gao *et al.* 2007).

Conclusions

Boiling treatment had significant effects on methanol-soluble extractive content, total phenolic content, and total polysaccharide content, but it did not have any significant effect on cell wall components. These trends may be attributable to the application of low temperatures in the experiment. Radial direction had significant effects on hot-water-soluble extractive content, total phenolic content, and total polysaccharide content at the 5% significance level. As with boiling treatment, radial direction also lacked a significant effect on cell wall components. The variation in extractive contents might be related to heartwood formation, which necessitates a further study to confirm this hypothesis.

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