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Microfibril Angles and Crystalline Properties of Reaction Woods in Agathis and Sumatran Pine Woods

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ABSTRACT

The microfibril angles (MFAs), relative crystallinity index (RC), and crystallite width (CrW) of compression wood (CPW), lateral wood (LTW), and opposite wood (OPW) in Agathis (*Agathis loranthifolia*) and Sumatran Pine (*Pinus merkusii*) stem wood were observed and compared to obtain valuable information on wood quality for effective utilization. The iodine staining method and optical microscopy were applied to measure the MFA in the tangential section. RC and CrW were analyzed with an X-ray diffraction technique. CPW had the largest MFA and the smallest RC in both species. In Agathis, LTW and OPW had comparable MFA, RC, and CrW, whereas the CrW of CPW was the smallest. In Sumatran pine, there was a significant difference in MFA and RC between LTW and OPW. CPW, LTW, and OPW showed comparable CrW. The MFA decreased and RC increased from near the pith to bark in both species. The CrW increased from near the pith to the bark of Sumatran pine, whereas it was constant in Agathis. In conclusion, MFA and RC could be used to identify CPW, LTW, and OPW in both species. There were distinctive MFA and RC properties between reaction wood in both species.

1. Introduction

Tropical trees are primary resources to supply eco-friendly wood materials, such as agathis (*Agathis loranthifolia*), mangium (*Acacia mangium*), red meranti (*Shorea parvifolia*), sengon (*Paraserianthes falcataria*), Sumatran pine (*Pinus merkusii*), puspa (*Schima wallichii*), and Afrika wood (*Maesopsis eminii*) (Aisyah et al. 2023; Baskara et al. 2023; Hidayat et al. 2017; Savero et al. 2022). Especially, Agathis (*A. loranthifolia*) and Sumatran Pine (*P. merkusii*) are common raw material for furniture, paneling, molding, packaging, furniture, pulp and paper, and musical instruments (Darmawan et al. 2018; Trisatya et al. 2019). Both wood species grow naturally in mountain areas of Indonesia and are widely planted in Indonesian plantation forests.

Compression wood (CPW) is frequently detected in both species and causes troubles in wood industries. CPW is reaction wood produced by trees to respond to the mechanical stress or gravitational forces on a tree, commonly formed in the lower part of crooked stems and branches of conifers (Gardiner et al. 2014). CPW was easily recognized with the brown to dark reddish color on the cross-section of stem and branch wood, and also, there were opposite wood (OPW) and lateral wood (LTW) in the leaning stems and branches. CPW showed distinctively different anatomical characteristics compared to LTW and OPW, such as circular shape tracheid with helical cavities on the tracheid wall, numbers of intercellular spaces, highly lignified S_2 layer, thicker cell wall, distorted bordered pits and cross-field pits, greater microfibril angles (MFAs), shorter tracheid length, smaller radial tracheid diameter, lower relative crystallinity index (RC), and smaller crystallite width (CrW) (Park et al. 1979, 1980; Purusatama and Kim 2018, 2020). CPW also showed distinct chemical, physical, and mechanical properties with normal wood, which could be less desirable than normal wood for commercial utilization in wood industries (Wimmer and Johansson 2014).

A few studies have reported MFA and crystalline properties as key indices for CPW, LTW, and OPW identification using various temperate softwoods. Parham (1971) suggested that the relative crystallinity in mature wood of *Pinus taeda* was smaller in CPW than in normal wood. In contrast, the crystallite width between normal wood and CPW was comparable. Marton et al. (1972) reported that CPW in *Picea abies* had smaller relative crystallinity than OPW, whereas the CPW had thinner and much smaller crystallite than OPW. Tanaka et al. (1981) mentioned that the RC of CPW, OPW, and normal wood was 45–50%, 50–60%, and 50%, respectively. In addition, the crystallite width of CPW, normal wood, and OPW was comparable, while the crystallite length of CPW was shorter than those of normal wood and OPW. As mentioned by Park et al. (1979, 1980), the compression side of *Pinus densiflora* branch wood showed typical CPW characteristics, such as a higher latewood portion, helical cavities, thicker cell wall, and higher microfibril angle than the lateral and opposite sides. In addition, the lateral and opposite sides showed similar anatomical characteristics except for the thickness of the S_3 layer in the latewood. Li et al. (2014) reported that the tracheid of CPW in radiata pine branches showed thicker walls and greater MFA than OPW. Shirai et al. (2016) revealed that compression wood-like in *Gingko biloba* stem wood showed a higher MFA, air dry density, lignin content, and lower cellulose content than the tissue in the upper and lateral sides.

Purusatama and Kim (2018) reported that CPW of *G. biloba* stem wood had higher ray height, lower ray number, and greater MFA than LTW and OPW, while LTW and OPW showed comparable characteristics. CPW had the lowest RC among parts, while LTW had the highest. Moreover, CPW, LTW, and OPW showed a comparable RC in the fifth growth ring. In addition, the crystal width of CPW, LTW, and OPW showed no significant difference. Purusatama et al. (2020) mentioned that CPW had the greatest MFA in the *P. densiflora* stem wood, while OPW had the smallest MFA. Furthermore, CPW had the lowest RC and the smallest CrW, and OPW had the greatest RC and CrW. The MFA, RC, and CrW of LTW were the intermediate among the parts.

Up to now, there has been no study on the MFA and crystalline properties and their radial variation in CPW, LTW, and OPW of tropical softwoods, such as Agathis (*A. loranthifolia*) and Sumatran Pine (*P. merkusii*). Therefore, this study observed and compared the MFA and crystalline characteristics of CPW, LTW, and OPW in Agathis and Sumatran Pine stem wood to understand the wood quality for effective utilization.

2. Materials and Methods

2.1. Materials

In the present study, a tree each of 65 years old Agathis (*A. loranthifolia*) and 49 years old Sumatran Pine (*P. merkusii*) was harvested from Gunung Walat University Forest, Sukabumi, West Java, Indonesia (6.882937°N, 106.818511°E). Both trees have a tilt of the stem axis close to 45° (**Fig. 1**). In addition, **Table 1** shows the detailed information of the sample trees. We extracted a wood disc with a diameter of approximately 40 cm from 4 m above ground. In addition, the MFA and crystalline properties of CPW, LTW, and OPW were observed in the three zones: near the pith (NP), middle zone, and near the bark (NB). The NP, middle zone, and NB in CPW were 5 cm, 20 cm, and 35 cm from the pith, respectively.

Table 1. Basic information of the sample trees

No.	Common name	Species	Age (year)	DBH (cm)
1	Agathis	*Agathis loranthifolia*	65	89
2	Sumatran Pine	*Pinus merkusii*	49	40

Note: DBH = diameter at breast height.

Fig. 1. Sample trees of (a) Agathis and (b) Sumatran pine (The yellow arrows indicate the position of the wood discs obtained).

2.2. Microfibril Angel Measurement

To measure MFA, the tangential sections of CPW, LTW, and OPW with 20–30 μm in thickness were prepared, and the slices were briefly dried in the oven at 103 ± 2°C and placed in 50% ethyl alcohol, and the microfibril slope was revealed by using the iodine staining method (Donaldson and Frankland 2004). The stained tangential sections were observed with an optical microscope (Eclipse E600; Nikon, Tokyo, Japan) connected to an i-Solution Lite software (IMT i-Solution Inc., Burnaby, BC, Canada). MFA was calculated from 20 tracheids from each part.

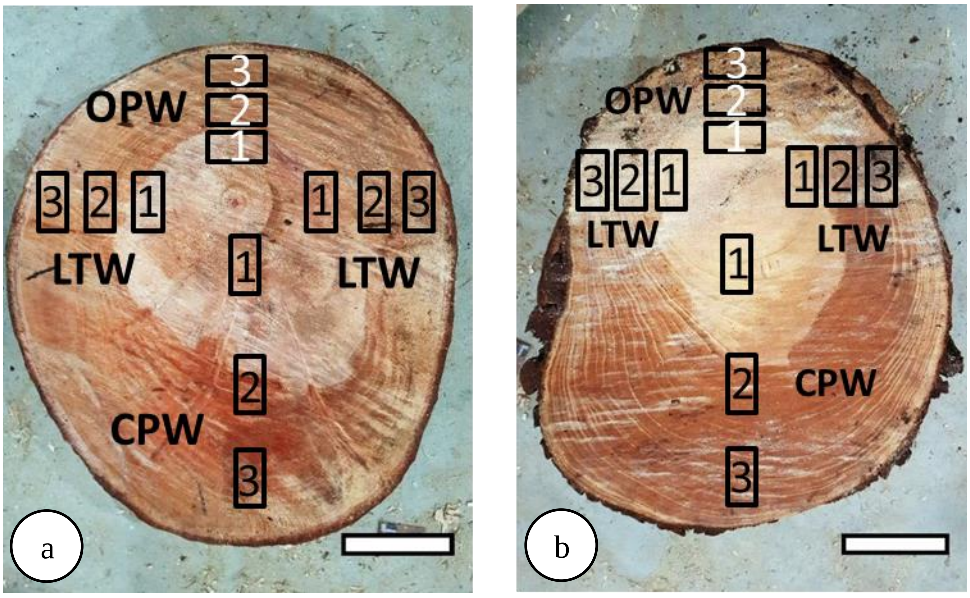

Fig. 2. Fresh-cut wood discs of (a) Agathis and (b) Sumatran pine. CPW, LTW, and OPW refer to compression, lateral, and opposite woods. NP, middle zone, and NB are indicated with 1, 2, and 3, respectively (Scale bars = 10 cm).

2.3. X-ray Diffraction Analysis

The crystalline properties of CPW, LTW, and OPW, such as relative RC and CrW, were measured with specimens of approximately 1 mm (radial) × 15 mm (tangential) × 15 mm (longitudinal). The crystalline properties were obtained with an X-ray diffractometer (DMAX2100V; Rigaku, Tokyo, Japan) equipped with a Cu target (λ = 0.1542 nm). The measurement was performed using three samples for each part. The RC was calculated using Segal's equation (Segal et al. 1959), as shown in Equation 1.

$$RC = \frac{I_{200} - I_{am}}{I_{200}} \times 100 \qquad (1)$$

where I_{200} is the peak intensity of the crystalline substance at 2θ (22.8°) and I_{am} refers to the amorphous substances at 2θ (18°).

The CrW was calculated using Scherrer's equation (Lee et al. 2023), as shown in Equation 2.

$$CrW\ (nm) = \frac{K \cdot \lambda}{\beta \cdot cos\theta} \qquad (2)$$

where K refers to Scherrer's constant (0.9), λ is the wavelength of the X-ray (λ=0.1542nm), and β belongs to half-width in radians.

2.4. Statistical Analysis

The statistical differences at a 5% significance level in the MFA, RC, and CrW between parts and between zones were analyzed with a one-way analysis of variance and post-hoc analysis with Duncan's multiple range tests (SPSS ver. 26, IBM Corp., Armonk, NY, USA).

3. Results and Discussion

3.1. Microfibril Angles (MFA)

The light micrographs of the iodine-stained tangential sections are presented in **Fig. 3**. The orientation of the microfibril became apparent due to the dark crystal formed by the iodine solution between microfibrils, which was in line with Senft and Bendtsen (1985). As the authors described, iodine solution and nitric acid produced crystals parallel with the microfibril orientation in the cell wall. In the present study, the CPW showed a greater slope of microfibril and a more distinct microfibril orientation than LTW and OPW.

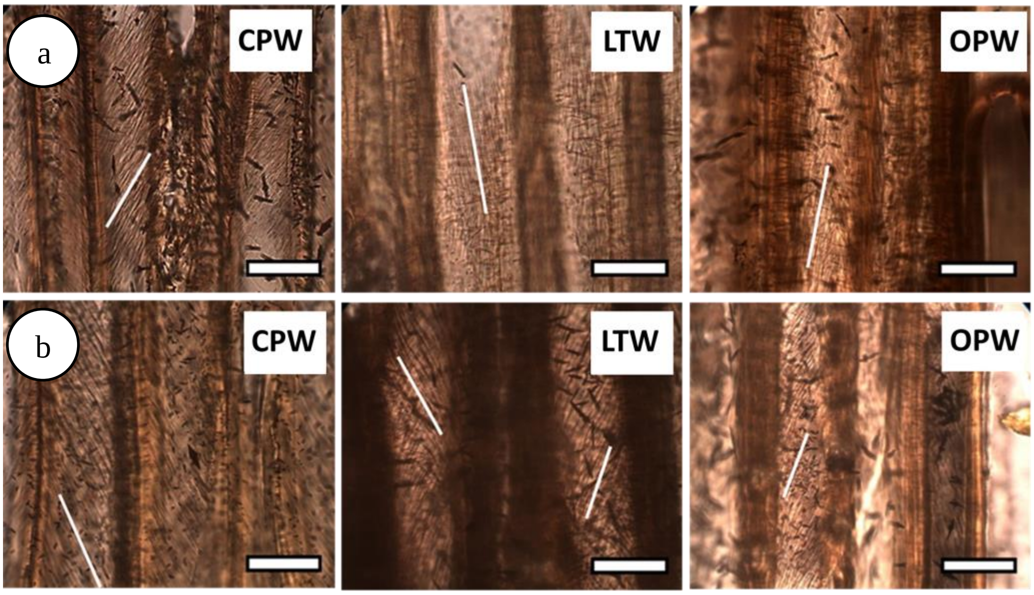

Fig. 3. Light micrographs of the iodine-stained tangential sections of CPW, LTW, and OPW in (a) Agathis wood and (b) Sumatran pine wood. White lines indicated the slope of microfibril (Scale bars: 25 µm).

The MFAs of CPW, LTW, and OPW from Agathis and Sumatran pine woods are presented in **Table 2**. CPW from Agathis and Sumatran pine yielded the largest MFA among the parts in each zone. In Agathis, the MFA of LTW and OPW was comparable in each zone. For the average values, CPW yielded the largest MFA among parts in both species, while LTW and OPW showed no significant differences. Near the pith of Sumatran pine, LTW had greater MFA than OPW, while LTW and OPW in the middle zone had comparable MFA. Near the bark, LTW was the smallest among all parts, and OPW was intermediate. The results in our study are in line with the previous studies on the MFA of CPW. Park et al. (1979) summarized that the compression side had higher MFA than the lateral and the opposite side in the branch wood of *P. densiflora*, while the lateral side had slightly higher MFA than the opposite side. In *P. abies* and *Abies alba*, the MFAs of CPW were greater than those of normal wood (Gorisek et al. 1999; Sahlberg et al. 1997). Donaldson et al. (2004) described that CPW of *Pinus radiata* showed higher MFA than OPW, while CPW and OPW had similar MFA in juvenile wood. Pandit and Rahayu (2007) reported that the CPW of *A. loranthifolia* showed distinctively greater MFA than normal wood. Purusatama and Kim (2018) mentioned that CPW showed the greatest MFA among the parts in *G. biloba* stem wood, whereas LTW and OPW had comparable MFA. Purusatama et al. (2020) revealed that CPW had the largest MFA in the stem wood of *P. densiflora*, while OPW had the smallest MFA.

Table 2. Microfibril angle in CPW, LTW, and OPW of Agathis and Sumatran pine woods

Species	Reaction wood	Microfibril angle (°)			Average
		Near the pith	Middle	Near the bark	
Agathis	CPW	31.1 (3.6)[bC]	28.1(1.7)[bB]	25.8 (4.2)[bA]	28.3 (2.7)[b]
	LTW	17.2 (4.9)[aB]	9.7 (2.3)[aA]	9.4 (1.5)[aA]	12.1 (4.4)[a]
	OPW	19.9 (4.4)[aB]	10.7 (3.6)[aA]	10.8 (2.6)[aA]	13.8 (5.3)[a]
Sumatran pine	CPW	31.1 (5.1)[cB]	30.5 (5.3)[bB]	23.7 (2.8)[cA]	28.4 (4.1)[b]
	LTW	26.4 (3.4)[bC]	16.4 (3.5)[aB]	12.5 (2.9)[aA]	18.4 (7.4)[a]
	OPW	21.3 (2.2)[aB]	16.6 (1.7)[aA]	15.5 (1.7)[bA]	17.8 (3.1)[a]

Notes: Standard deviations are numbers in parentheses. The superscript letters in the same columns and capital letters in the same line next to the mean values represent insignificant results at the 5% significance level for comparisons between parts and among zones after the post-hoc test, respectively.

The variation of MFA in CPW, LTW, and OPW of Agathis and Sumatran pine woods is shown in **Fig. 4**. In both species, the MFA of CPW gradually decreased from near the pith to the bark. The MFA of LTW and OPW decreased rapidly from near the pith to the middle zone, and it was almost constant from the middle zone to near the bark.

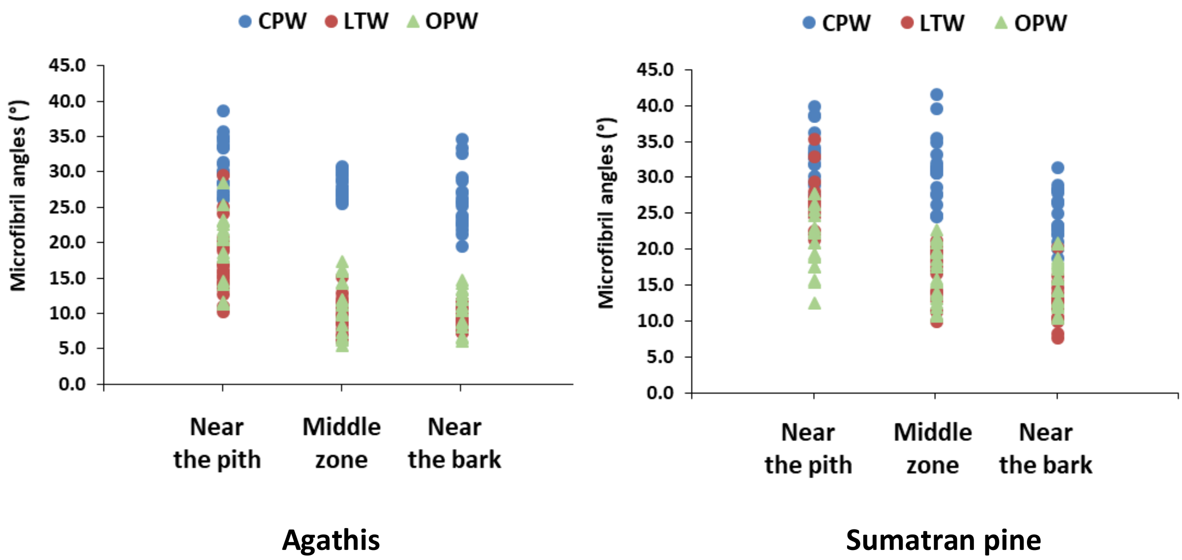

Fig. 4. Variation of MFA in each zone of CPW, LTW, and OPW of Agathis and Sumatran pine woods.

Some studies were in line with our results. Park et al. (1979) reported that the MFA of *P. densiflora* branch wood gradually decreased with increasing growth rings. Purusatama et al. (2020) reported that the MFAs of CPW, LTW, and OPW in Korean red pine decreased from the 5th to the 30th growth ring and then became constant. MFAs in the normal wood of *Pinus rigida*, *Pinus koraiensis*, and *P. densiflora* decreased from the pith to approximately the 15th to 20th growth ring, and then the MFAs were constant (Eun and Kim 2008). Ishiguri et al. (2010) mentioned that the MFA of normal wood in *Agathis* sp. and *Pinus insularis* rapidly decreased up to 2 cm from the pith and almost constantly to the bark. Darmawan et al. (2018) mentioned that the MFA of *P. merkusii* steeply decreased from pith to 9 cm from the pith and then gradually decreased towards the bark.

3.2. Crystalline Properties

The X-ray diffractograms and RC of CPW, LTW, and OPW in Agathis and Sumatran pine woods are in **Fig. 5** and **Table 3**, respectively. In Agathis, CPW had the smallest RC in each zone, and LTW and OPW had a similar RC. In Sumatran pine, CPW, LTW, and OPW had similar RC near the pith, while CPW showed the smallest RC in the middle zone and near the bark. The RC of LTW was the highest among all parts in the middle zone and near the bark. Furthermore, CPW yielded the lowest average relative crystallinity, LTW had the highest average RC, and OPW was intermediate. Purusatama and Kim (2018) mentioned that CPW showed the lowest RC in *G. biloba* stem wood, while LTW was the highest. Moreover, Purusatama et al. (2020) reported that CPW in Korean red pine had the lowest RC, OPW had the largest RC, and LTW was intermediate. Lee (1961) and Parham (1971) mentioned that CPW in Douglas-fir and loblolly pine had lower RC than normal wood, likely due to higher lignin and lower cellulose content in CPW than normal wood.

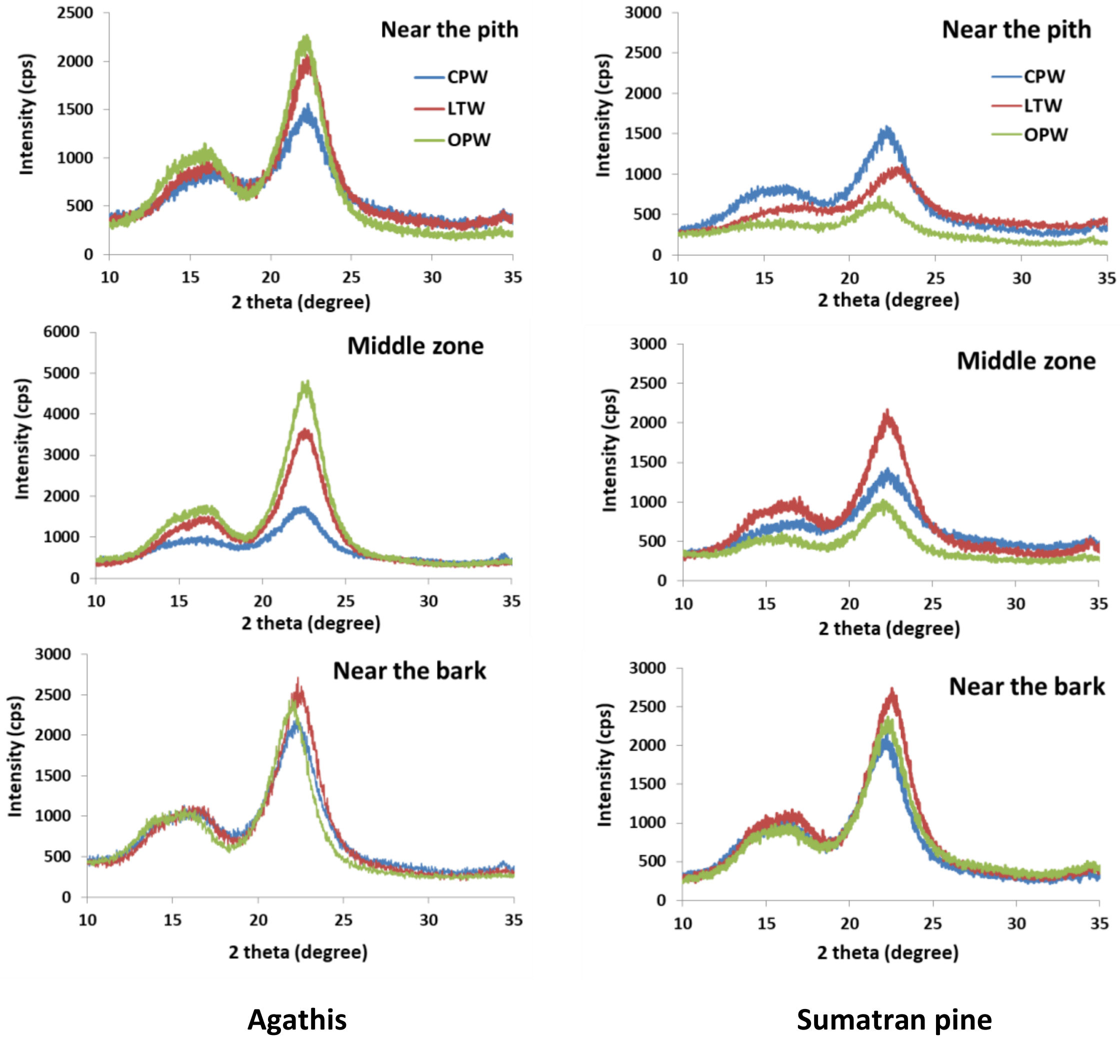

Fig. 5. The X-ray diffractograms of CPW, LTW, and OPW in Agathis and Sumatran pine woods.

The RC of CPW, LTW, and OPW in both species increased from near pith toward near bark. In other conifer species, the RC of CPW, LTW, and OPW increased as growth ring numbers increased in *G. biloba* (Purusatama and Kim 2018) and *P. densiflora* (Purusatama et al. 2020) stem woods. Besides, radial variation of RC in CPW, LTW, and OPW was comparable with normal wood in conifer species, such as *P. densifora*, *P. koraiensis*, *P. rigida* (Eun et al. 2008), and *Chamaecyparis obutsa* (Kim and Lee 1998).

Table 3. Relative crystallinity in CPW, LTW, and OPW of Agathis and Sumatran pine woods

Species	Reaction wood	Relative crystallinity (%)			Average
		Near the pith	Middle	Near the bark	
Agathis	CPW	55.9 (2.3)^{aA}	54.7 (3.2)^{aA}	63.5 (4.5)^{aB}	58.1 (4.8)^{a}
	LTW	65.6 (1.3)^{bA}	72.4 (0.4)^{bB}	75.8 (0.3)^{bC}	71.3 (5.2)^{b}
	OPW	63.9 (2.5)^{bA}	74.7 (3.1)^{bB}	74.1(1.7)^{bB}	70.9 (6.1)^{b}
Sumatran pine	CPW	53.2(1.7)^{aA}	60.1(1.9)^{aB}	65.7 (0.8)^{aC}	59.7 (6.3)^{a}
	LTW	54.1(1.0)^{aA}	70.7 (1.8)^{cB}	75.9 (1.1)^{cC}	66.9 (11.4)^{a}
	OPW	54.1(3.4)^{aA}	64.9 (1.2)^{bB}	71.6 (1.8)^{bC}	62.8 (8.8)^{a}

Notes: Standard deviations are numbers in parentheses. The superscript letters in the same columns and capital letters in the same line next to the mean values represent insignificant results at the 5% significance level for comparisons between parts and among zones after the post-hoc test, respectively.

The CrW of CPW, LTW, and OPW in Agathis and Sumatran pine woods are shown in **Table 4**. There was no significant difference within the CrW of CPW, LTW, and OPW in Sumatran pine. In Agathis, CPW had the smallest CrW, while LTW and OPW showed no significant differences. Purusatama and Kim (2018) summarized that the CPW in *G. biloba* stem wood had the smallest CrW among CPW, LTW, and OpW. As Purusatama et al. (2020) revealed, CPW in *P. densiflora* stem wood had the smallest CrW, while OPW had the largest CrW, and LTW was intermediate. Tanaka et al. (1981) mentioned that the cellulose crystallite dimension of CPW in *P. densiflora* was slightly smaller than that of OPW, whereas that of OPW was the biggest. Andersson et al. (2000) reported that the CPW had smaller CrW than normal wood of *P. abies*.

Table 4. Crystallite width in CPW, LTW, and OPW of Agathis and Sumatran pine woods

Species	Reaction wood	Crystallite width (nm)			Average
		Near the pith	Middle	Near the bark	
Agathis	CPW	2.7 (0.0)^{aA}	2.7 (0.1)^{aA}	2.8 (0.0)^{aA}	2.8 (0.1)^{a}
	LTW	2.9 (0.2)^{abA}	3.2 (0.2)^{bA}	3.2 (0.2)^{abA}	3.1 (0.3)^{b}
	OPW	3.1 (0.1)^{bA}	3.3 (0.1)^{bA}	3.1 (0.1)^{bA}	3.2 (0.2)^{b}
Sumatran pine	CPW	2.7 (0.1)^{aA}	2.6 (0.0)^{aA}	2.9 (0.0)^{aB}	2.7 (0.1)^{a}
	LTW	2.8 (0.0)^{aA}	2.8 (0.0)^{bA}	2.9 (0.0)^{aB}	2.8 (0.1)^{a}
	OPW	2.7 (0.2)^{aA}	2.9 (0.1)^{cB}	2.9 (0.1)^{aB}	2.9 (0.1)^{a}

Notes: Standard deviations are numbers in parentheses. The superscript letters in the same columns and capital letters in the same line next to the mean values represent insignificant results at the 5% significance level for comparisons between parts and among zones after the post-hoc test, respectively.

In Sumatran pine, the CrW near the bark was significantly larger than that near the pith of CPW, LTW, and OPW. The CrW near the pith of CPW and LTW showed no significant differences from that in the middle zone. In OPW, the near the bark showed a similar CrW with the middle zone. In Agathis, there was no significant difference in the CrW near the pith, the middle zone, and near the bark of CPW, LTW, and OPW. As mentioned in previous studies, the CrW of CPW, LTW, and OPW in *G. biloba* and *P. densiflora* stem wood showed no specific pattern in the radial direction (Purusatama and Kim 2018; Purusatama et al. 2020). In the normal wood, the CrW showed no variation in the radial direction of the stem in *Chamaecyparis obutsa*, *P. densifora*, *P. koraiensis*, *P. rigida*, *Larix gmelinii*, and *Larix kaempferi* (Eun et al. 2008; Kim et al. 2022; Kim and Lee 1998).

4. Conclusions

MFA and crystalline properties of CPW, LTW, and OPW of Agathis and Sumatran pine woods were examined. CPW of both species had the largest MFA, while LTW and OPW showed a similar value. The CPW of both species showed the smallest RC among parts. In Sumatran pine, the LTW had the greatest RC among all parts, and OPW showed a smaller RC than LTW. All parts showed a comparable CrW. In Agathis, LTW and OPW showed a similar RC. CPW had a smaller CrW than LTW and OPW, while the CrW of LTW and OPW showed no significant difference. In conclusion, CPW of both species showed distinctive MFA and crystalline properties compared to LTW and OPW, whereas LTW and OPW mostly had comparable MFA and crystalline properties. There were differences in the MFA and crystalline properties of the reaction wood between Sumatran pine and Agathis. The results of the present study could be used to identify reaction wood in both species for effective utilization of the wood.

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