

CHARACTERISTIC FEATURES OF THE OIL-HEAT TREATED WOODS FROM TROPICAL FAST GROWING WOOD SPECIES

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ABSTRACT

This study aimed to evaluate the effect of oil-heat treatment on the anatomical, physical, and chemical properties of the tropical fast-growing wood species as gmelina (*Gmelina arborea*) and mindi (*Melia azedarach*) wood. Vessel lumen area and diameter in radial and tangential direction of both species increased with increasing temperature. The fiber lumen areas in both woods were remarkably decreased by oil-heat treatment, and the fiber wall area increased considerably with increasing temperature. Both woods tended to gain weight after heat treatment at 180°C and 200°C, and then lose weight after heat treatment at 220°C. The density of mindi increased greatly at 180°C and 200°C and slightly decreased at 220°C. The dimension of the specimens in tangential direction increased with heat treatment, but the rate decreased with increasing temperature. The relative crystallinity and crystallite width of the heat-treated woods were greater than those of the untreated wood. In the Fourier transform infrared analyses, the peaks from the carbohydrates were changed after oil-heat treatment, mainly due to the degradation of hemicellulose. Consequently, it was revealed that the heat treatment affected various properties of gmelina and mindi woods. Differing characteristics between the species were also noted.

KEYWORDS: Anatomical properties, chemical composition, *Gmelina arborea*, *Melia azedarach*, oil-heat treatment, physical properties.

INTRODUCTION

The potential supply and utilization of wood from plantation forests continue to increase. The wood harvested from plantation forests in Indonesia mostly belongs to fast-growing wood species as well as gmelina (*Gmelina arborea*) and mindi (*Melia azedarach*). Gmelina has raised interest because of its rapid growth and rapid return on investment that can be used for many products ranging from pulp to furniture (Dvorak 2004). Mindi is considered as a fast-growth and drought-resistant species and widely used for construction, furniture, or interior decoration because it is quite durable and termite resistant (Bui et al. 2019). However, wood from most fast-growing species is generally low in quality, and its properties need to be improved, due to inferior quality with low density, low durability, and low mechanical properties, rendering them not suitable as structural materials (Febrianto et al. 2015).

Modification by heat treatment is one of the recent technologies used to improve some properties of woods (Biziks et al. 2013, Hidayat et al. 2016). Heat treatment of wood can be applied to achieve the desired improvement in dimensional stability and hydrophobicity (Tjeerdsmā and Militz 2006), and biological durability (Welzbacher et al. 2008). Welzbacher et al. (2011) also reported that thermal treatment leads to substantial loss of the strength of heat-treated woods. Heat-treated wood also had lower equilibrium moisture content than untreated wood (Hidayat et al. 2015) and increased in hydrophobicity with increasing contact angle on wettability (Kocafe et al. 2008). Meanwhile, heat treatment affects the anatomical structure of wood, as evidenced by the destruction of tracheid walls and ray tissues, as well as pit deaspersion (Awoyemi and Jones 2011), but this depends on the wood species, heat treatment process, and the conditions used (Boonstra et al. 2006).

With regard to cost, environmental-friendliness, and sustainability of chemistry, oil-heat treatment is considered one of the most practical approaches to eliminating the inferior features of wooden materials (Tang et al. 2019). Another related study on oil-heat treatment also explained that oil uptake also contributes to the dimensional stability and hydrophobicity (Wang and Cooper 2005) as well as fungal resistance and darkening (Dubey et al. 2011) of wood. Lee et al. (2018) demonstrated that oil-heat-treated wood showed superior dimensional stability to that of wood treated in hot air and nitrogen. Oil heat treatment can also be used to upgrade wood for outdoor uses and uniformly color its surface (Sailer et al. 2000).

To date, information on the improvement of the wood quality of fast-growing wood species using oil-heat treatment has been lacking. Therefore, in the present study, oil heat treatment was applied to improve the wood quality of two fast-growing wood species, gmelina and mindi, growing in Indonesia

MATERIAL AND METHODS

Materials

Gmelina (*Gmelina arborea*) and Mindi (*Melia azedarach*) woods were harvested from Perum Perhutani Kesatuan Pemangkuan Hutan (KPH) Bogor and KPH Purwakarta in Java, Indonesia. Logs of woods were converted into quarter-sawn boards with dimensions of 200 mm (L) x 90 mm (R) x 20 mm (T). Boards were selected that were free of natural defects and straight grain. Then the selected boards were air-dried and conditioned at 25°C ± 5°C under a relative humidity of 70–80% until they reached the equilibrium moisture content.

Methods

Oil-heat treatment

Boards were heat-treated in a lab-scale oil bath using commercial cooking palm oil. The heat treatment was started at an initial temperature of 25°C - 30°C. The temperature was then raised to the target of 180°C, 200°C, and 220°C at the rate of 2°C·min⁻¹. The target temperature was maintained over a period of 1 h. In the final stage of heat treatment, the oil bath was allowed to cool naturally until it reached ± 30°C. The boards were then taken out and stored in a room with the relative humidity of 70–80% and a temperature of 25 ± 5°C for two weeks until further testing.

Scanning electron microscopy

The smooth transversal section of the block with a dimension of 10 mm (L) x 10 mm (T) x 10 mm (R) was prepared with a microtome, and then oven-dried and a Cressington sputter coater (ULVAC G-50DA, Japan) used to coat the sample. Microscopic observation was performed with a scanning electron microscope (JSM-5510, JEOL, Japan, 15kV). The micrographs of each sample were analyzed for measuring vessel lumen area (μm²), diameter of vessel lumen in the radial (RD) and tangential (TD) direction (μm), fiber lumen area (μm²), and fiber wall area (μm²). The measurements for each parameter were examined 90 times. The change of cell lumen area in vessel and fiber was calculated using the following formula (Biziks et al. 2013):

$$SEM (\%) = \frac{SEM_1 - SEM_0}{SEM_0} \times 100 \quad (1)$$

where: SEM (%) is the change in the cell lumen area in the vessel and the fiber (%), SEM₁ is the cell lumen area after heat treatment, and SEM₀ is the cell lumen area before heat treatment. Measurements were performed using the freely-available ImageJ software package (University of Wisconsin, Madison, USA).

Measurements of physical properties

Weight change before and after the heat treatment was intended according to the following formula:

$$WC (\%) = \frac{m_1 - m_0}{m_0} \times 100 \quad (2)$$

where: m₀ is the weight of samples before oil-heat treatment (g) and m₁ is the weight of samples after oil-heat treatment (g). Density (D) of samples before and after heat treatment was intended according to the following formula:

$$D = \frac{m}{v} \quad (3)$$

where: m is the weight (g) and v is the volume of samples (cm³). Swelling in tangential direction of samples before and after heat treatment was intended according to the following formula:

$$S (\%) = \frac{S_1 - S_0}{S_0} \times 100 \quad (4)$$

where: S₀ is the swelling of samples before oil-heat treatment (mm) and S₁ is the swelling of samples after oil-heat treatment (mm). Three samples were measured for each physical property after each treatment and mean values were determined.

Crystalline characteristic analyses

An X-ray diffractometer (DMAX2100V, Rigaku, Tokyo, Japan) was used to measure crystalline characteristics as relative crystallinity and crystallite width. Segal's equation

(Segal et al. 1959) was used to calculate the relative crystallinity:

$$\text{Relative crystallinity} = \frac{I_{200} - I_{am}}{I_{200}} \times 100 \quad (5)$$

where: I_{200} is the diffraction intensity of (200) ($2\theta = 22.8^\circ$) and I_{am} is the diffraction intensity of the non-crystalline portion ($2\theta = 18^\circ$). The crystallite width was calculated using the Scherrer's equation (Burton et al. 2009):

$$L = \frac{K\lambda}{\beta \cos\theta} \quad (6)$$

where: L is the crystallite width, K is the Sherrer constant (0.9), λ is the X-ray wave length ($\lambda = 0.1542$ nm), and β is half-width in radians. Three samples were measured for each treatment and average values were determined.

Chemical composition analyses

To investigate the chemical composition of gmelina and mindi woods before and after oil-heat treatment, wood powders for each treatment were prepared. The spectra were measured using the attenuated total reflection (ATR) method in the range of 4000–400 cm^{-1} with Fourier transform infrared spectrometer (Perkin Elmer Inc., USA) set in the main laboratory of Kangwon National University.

Statistical analysis

All multiple comparisons were analyzed with multivariate analysis of variance. Significant ($\alpha \leq 0.05$) differences between values of the untreated and treated samples were determined using Duncan's multiple range tests. All statistics were performed using SPSS ver. 24, IBM Corp., New York, USA.

RESULTS AND DISCUSSION

Anatomical properties

As shown in Tab. 1, the vessel lumen area and diameter of vessel lumen in a radial (RD) and tangential (TD) direction increased with increasing temperature. Gmelina wood increased up to 5.6% in the vessel lumen area and 24.7% and 18.3% in the diameter of vessel lumen in radial and tangential directions, respectively, at 220°C . While in mindi wood, there was an increase of up to 59.9% in vessel lumen area, 37.4% in the radial diameter, and 17.1% in the tangential diameter of vessel lumen at 220°C . Comparing both species, it can be explained that mindi wood showed a greater change in the dimension of the vessel than in gmelina wood during heat treatment. Fig. 1 shows the vessels on the transverse section of the gmelina and mindi woods with the same magnification before and after oil-heat treatment; the vessel dimension in both the wood species increased with heat treatment. Batista et al. (2015) reported that the vessel diameter of *Eucalyptus grandis* wood increased after heat treatment at 140°C , but this increase was not significant. However, Biziks et al. (2013) reported opposite results, that in *Betula pendula*, lumen area and diameter of the vessels slightly decreased, and the wall area and wall thickness of the vessels considerably decreased after hydrothermal treatment. A significant change in fiber dimension was observed, as shown in Tab. 1. The fiber lumen area of gmelina wood decreased by 36.2%, fiber wall area increased by 60.1%, and fiber total area decreased by 1.37% at 220°C . The fiber lumen area in mindi greatly decreased by 61.9%, the fiber wall area increased by 61.1%, and fiber total area decreased by 11.58% at 220°C . The fiber lumen area and fiber total area decreased, while the fiber wall area increased with increasing temperature. This shows that the fiber wall swelled as a result of oil uptake during heat treatment and that the heat temperature affected the swelling of the cell wall. Scanning

electron micrographs of the fibers of gmelina and mindi before and after oil-heat treatment are shown in Fig.2. The fiber lumen area of the treated wood clearly decreased to less than that of the untreated wood, but the fiber wall area increased with increasing temperature. Ling et al. (2016) reported similar results in *Populus cathayana* sapwood, in which the fiber walls thickened and fiber lumen size shrank during and after heat treatment. Biziks et al. (2013) observed structural changes in *Betula pendula* wood: the fiber lumen area, fiber total area, and fiber wall area decreased during hydrothermal treatment. They described that the degradation of hemicelluloses in cell wall, which is known as the most unstable wood component, causes the change in cell dimension by heat treatment. Awoyemi and Jones (2011) also explained that changes in wood anatomy might be affected by wood properties with chemical degradation during the process of heat treatment. In this study, the oil-heat treatment led to anatomical changes in the wood cells. This phenomenon could be explained as follows: (1) The cell wall in the wood fiber and vessel might be softened and become more flexible following oil uptake, (2) The increased size of the vessel lumen can also be attributed to the tension force induced by the change of fiber dimension in the swollen state due to oil uptake, (3) The space created due to changes in fiber dimensions allowed the vessel dimension to increase in oil-heat-treated wood as compared to that of untreated wood. Bernabei and Salvatici (2016) demonstrated that the tracheid cell dimensions decreased, but in certain cases, it increased after treatment, because of the power given to the lumen by surrounding cells. Boonstra et al. (2006) also explained that the dimensions of vessels and rays were changed due to the contraction of the cell walls and the shrinkage of the surrounding cells, such as the wood fibers or tracheid.

Tab. 1: Dimensional changes in vessels and fibers before and after oil-heat treatment.

Wood species	Cell type	Vessel			Fiber		
	Measurements	Lumen area (μm ²)	RD (μm)	TD (μm)	Lumen area (μm ²)	Wall area (μm ²)	Total area of fiber (μm ²)
Gmelina	Initial sizes	11455 (1524)	112.1 (1.53)	92.2 (3.30)	97.6 (1.74)	59.4 (4.74)	158 (2.45)
Mindi	Initial sizes	24126 (7008)	164.6 (3.72)	143.5 (1.52)	87.7 (1.18)	61.4 (1.59)	149 (1.47)
	Temperature (°C)	Changes (%)					
Gmelina	180	2.3 (2.28)	14.4 (3.82)	4.7 (2.70)	-2.2 (1.32)	1.6 (3.24)	-0.84 (1.73)
	200	4.9 (2.35)	23.2 (3.33)	10.1 (5.24)	-23.0 (0.55)	37.0 (1.52)	-0.86 (1.15)
	220	5.6 (2.39)	24.7 (2.10)	18.3 (2.51)	-36.2 (0.65)	60.1 (3.94)	-1.37 (3.74)
Mindi	180	19.0 (7.53)	19.9 (4.05)	1.2 (3.72)	-12.9 (1.16)	12.5 (1.11)	-2.39 (1.98)
	200	30.7 (3.81)	24.6 (1.52)	13.7 (6.08)	-34.5 (2.58)	42.5 (1.76)	-2.75 (2.02)
	220	59.9 (6.92)	37.4 (4.65)	17.1 (2.64)	-61.9 (6.14)	61.1 (2.75)	-11.58 (2.65)

Notes: The numbers in parenthesis are the standard deviations. RD: diameter of vessel in radial direction, TD: diameter of vessel in tangential direction.

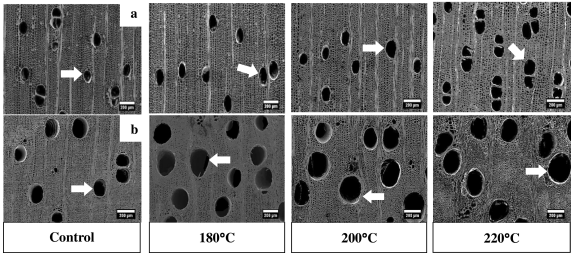


Fig.1: Vessels on the transverse sections of gmelina (a) and mindi (b) before and after oil-heat treatment. White arrows indicate the change in vessel lumen size before and after oil-heat treatment. Scale bars: 200μm.

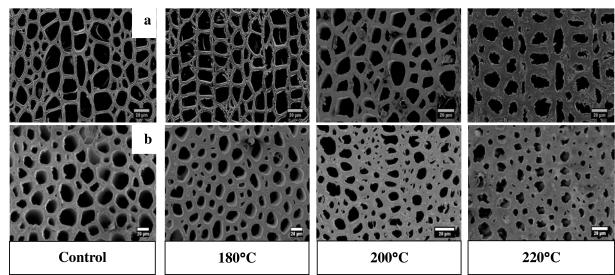


Fig. 2: Fibers on the transverse sections of gmelina (a) and mindi (b) before and after oil-heat treatment. Scale bars: 20μm.

Physical properties

The physical properties of gmelina and mindi woods before and after oil-heat treatment are shown in Tab. 2. A higher degree of weight change was observed in mindi, showing a great increase in the weight after heat treatment at 180°C and 200°C and a decrease in the weight at 220°C. Gmelina wood gained weight slightly at 180°C and 200°C and rapidly lost weight at 220°C. This result agreed with the study conducted by Dubey et al. (2011) that observed an increase in specimen weight due to oil absorption during heat treatment and a weight decrease with increasing temperature and time. Sailer et al. (2000) also reported that the weight of *Pinussylvestris* and *Piceaabies* increased by about 50 ± 70% after oil-heat treatment. Dubey et al. (2011) also stated that the weight decrease after heat treatment indicated the change of chemical composition in wood, particularly, the degradation of the hemicellulose content up to 70%. Hidayat et al. (2015) and Mburu et al. (2007) also explained that the weight loss of wood was due to the degradation of hemicelluloses during heat treatment.

Tab. 2: Weight change, density, and swelling after heat treatment.

Wood species	Temperature (°C)	Weight change* (%)	Density (gcm ⁻³)	Swelling in tangential direction (%)
Gmelina	Control	-	0.43 (0.02)	-
	180	1.98 (0.02)	0.45 (0.01)	3.46 (0.65)
	200	2.50 (0.06)	0.43 (0.01)	2.32 (0.58)
	220	-5.73 (1.04)	0.42 (0.02)	2.09 (0.29)
Mindi	Control	-	0.51 (0.03)	-
	180	24.61 (4.38)	0.66 (0.02)	5.77 (1.36)
	200	15.03 (0.98)	0.59 (0.02)	4.20 (1.02)
	220	-7.39 (0.74)	0.48 (0.01)	0.66 (1.06)

Note: *minus (-) values mean weight loss and vice versa. The numbers in parenthesis are the standard deviations.

The density of mindi wood after heat treatment was changed as shown in Tab. 2. The density increased greatly at 180°C and 200°C and slightly decreased at 220°C. However, the density of gmelina wood before and after treatment was scarcely changed. Wang et al. (2014) observed similar result as the density of *Eucalyptus pellita* wood decreased slowly with increasing the temperature at 200°C and 240°C. They stated the two possible factors for the decrease in density during heat treatment as the degradation of hemicellulose and volatile components.

The specimen's thickness in tangential direction increased with heat treatment as shown in Tab. 2. The degree of swelling decreased with increasing temperature. The woods treated at 220°C appeared more stable than those treated at 180°C and 200°C. This result agreed with the study of Bal (2015) that the swelling values of wood treated with oil-heat treatment decreased with increasing temperature. Lee et al. (2018) stated that oil absorption and oil deposits in the cell walls of wood also act as factors, which contribute to the improvement of the dimensional stability of wood.

Crystalline characteristics

The relative crystallinity of the heat-treated woods was slightly larger than that of the untreated woods, as shown in Tab. 3. It could be suggested that the relative crystallinity increased with heat treatment. Kim et al. (2018) reported that heat treatment increased the relative crystallinity of Paulownia wood. Yun et al. (2015) also reported that the relative crystallinity of *Eucalyptus urophylla* and *E. camaldulensis* increased as the temperature increased. The increase in crystallinity can probably be attributed to the degradation of hemicelluloses and the realignment of cellulose (Tang et al. 2019).

Tab 3: Crystalline properties of *gmelina* and *mind*i woods before and after oil-heat treatment.

Wood species	Temperature (°C)	Relative crystallinity (%)	Crystallite width (nm)
Gmelina	Control	57.3 ^a (1.5)	2.69 ^a (0.14)
	180	58.2 ^a (3.4)	2.84 ^a (0.16)
	200	60.9 ^b (1.7)	2.97 ^a (0.14)
	220	69.3 ^c (0.5)	3.22 ^b (0.11)
Mind	Control	57.8 ^a (2.8)	2.85 ^a (0.07)
	180	58.4 ^a (2.0)	2.91 ^a (0.14)
	200	61.4 ^b (2.0)	3.03 ^a (0.28)
	220	68.5 ^c (2.3)	3.45 ^b (0.38)

Note: The numbers in parenthesis are the standard deviations. The different letters beside the mean value show the significance at 5% level between untreated and treated wood using Duncan's multiple range tests.

The heat-treated woods showed slightly larger crystallite width than that of the untreated one, as shown in Tab. 3. It seems that the crystallite width could be increased after oil-heat treatment. Kubojima et al. (1998) also reported that the crystallite length of the heat-treated Sitka spruce seemed to be increased. The same tendency was also observed by Andersson et al. (2005), stating that the increase in the size of cellulose crystallites was due to the changes in porosity of the cell wall by the degradation of the amorphous part of wood during heat treatment.

Chemical composition analyses

Fig.3 shows the FTIR spectra of *gmelina* and *mind*i before and after oil-heat-treatment, and the appropriate peak assignment is summarized in Tab. 4. The spectral differences before and after oil-heat treatment were more considerable for *mind*i wood than for *gmelina* wood. The heat-treated wood showed the decreased characteristics of carbohydrates as compared to the untreated wood. The peak at 1740 cm⁻¹ corresponding C=O stretch decreased greatly in both *gmelina* and *mind*i with increasing temperature. Lin et al. (2018) reported that the peaks at 1730–1740 cm⁻¹ of poplar and fir wood decreased with increasing temperature. They explained that the decrease was caused by the cleavage of acetyl side chains of hemicellulose

after heat treatment. Ling et al. (2016) also reported that the peak at 1740 cm^{-1} on heat-treated *Populus cathayana* wood decreased. Similarly, Cheng et al. (2016) reported that the peak at 1734 cm^{-1} of Chinese fir wood changed slightly at 190°C and considerably decreased at 230°C. Esteves et al. (2013) also reported that the peaks at 1740 cm^{-1} assigned to the division of acetyl or acetoxy groups in xylan of eucalypt wood decreased, showing the degradation of hemicelluloses. The peak at the range of 1105 and 1050 cm^{-1} , corresponding to C-O-C stretching in cellulose and hemicellulose, in mindi also slightly decreased at 220°C. Mburu et al. (2007) reported that the peaks at 1058 cm^{-1} in heat-treated *Grevillea robusta* wood slightly decreased as temperature increased. They also stated that the decrease of the band confirmed the degradation of hemicelluloses.

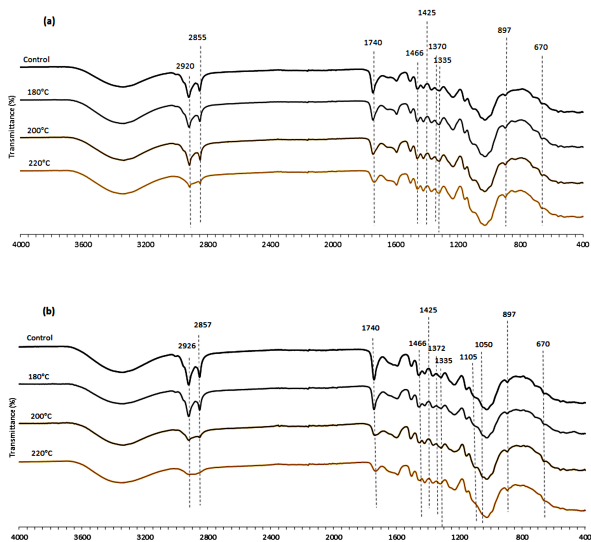


Fig. 3: FTIR spectra of gmelina (a) and mindi (b) wood before and after oil-heat treatment.

Tab. 4: Peak assignment for FTIR spectra of gmelina and mindi before and after oil-heat treatment.

Wave number (cm^{-1})	Peak assignment
2926	C-H stretching in cellulose (Esteves et al. 2013, Schwanninger et al. 2004)
2857	
1740	C=O stretch (unconjugated) in esters, ketones, aldehydes and acids (Esteves et al. 2013, Schwanninger et al. 2004, Lin et al. 2018)
1466	C-H deformations; asymmetric in $-\text{CH}_3$ and $-\text{CH}_2-$ (Huang et al. 2012, Schwanninger et al. 2004)
1425	Aromatic skeletal vibrations (lignin) and C H deformation in plane (cellulose) (Schwanninger et al. 2004)
1372	C H deformation in cellulose and hemicellulose (Schwanninger et al. 2004)
1105	C-O-C stretching in cellulose and hemicellulose (Traoré et al. 2018)
1050	C-O stretching of secondary alcohols (Mburu et al. 2007, Traoré et al. 2018)
897	C H deformation in cellulose (Schwanninger et al. 2004)
670	C-OH out-of-plane bending mode (Schwanninger et al. 2004)

In addition to the characteristics of carbohydrates, heat-treated wood and untreated wood were also pronounced well by the peak of lignin at 1466 cm^{-1} . The peak in gmelina and mindi decreased scarcely as the temperature increased. It indicates that lignin is also degraded by heat treatment, but the rate of degradation is much less than that of the hemicelluloses. Huang et al. (2012) reported that the peak at 1465 cm^{-1} in jack pine wood decreased after heat treatment and weathering. Cheng et al. (2013) also reported that the peak at 1462 cm^{-1} in moso bamboo decreased after being heat-treated with three aqueous solutions.

The cellulose crystallinity was also determined using different IR crystallinity ratios. There are two methods to determine the crystallinity, i.e., the first is the ratio of peak areas A_{1370}/A_{670} , and the second is the ratio of peak heights at H_{1429}/H_{897} and at H_{1372}/H_{2900} (Åkerholm et al. 2004). Tab. 5 summarizes the result of the measurements.

Tab. 5: Cellulose crystallinity of gmelina and mindi using different IR peak ratios.

Wood species	Temperature (°C)	IR peak ratio		
		A_{1370}/A_{670}	H_{1429}/H_{897}	H_{1372}/H_{2900}
Gmelina	Control	7.50	1.67	0.14
	180	8.33	1.75	0.19
	200	8.80	2.00	0.20
	220	8.93	1.60	0.55
Mindi	Control	8.05	1.25	0.12
	180	8.25	1.00	0.14
	200	8.46	1.67	0.58
	220	8.48	1.50	0.60

The ratio of the peak areas (A_{1370}/A_{670}) at 1370 and 670 cm^{-1} increased with increasing temperature in gmelina wood whereas the rate increased slightly in mindi wood. Then the values of H_{1429}/H_{897} and H_{1372}/H_{2900} showed a tendency to increase with increasing temperature in both species. These results indicate that the crystallinity of both woods increased as temperature increased. It is well matched with the result of X-ray diffraction analyses. Akgül et al. (2016) reported that the crystallinity in *Pinussylvestris* and *Abiesnordmanniana* wood increased after heat treatment. They explained that the increase in crystallinity might be due to the re-crystallization of cellulose molecules in quasi-crystalline amorphous region. Yıldız and Gümüşkaya (2007) reported that all of the IR peak ratios in spruce and beech woods increased with increasing temperature from 150 to 200°C .

CONCLUSIONS

The oil-heat treatment process of gmelina and mindi wood were investigated to clarify their characteristics before and after treatment. The vessel lumen area and diameter in radial and tangential direction increased with increasing temperature. The fiber lumen area and the fiber total area decreased and the fiber wall area increased with increasing temperature. Oil heat-treatment at 180°C and 200°C led to considerable weight gain for mindi wood but only slight weight gain for gmelina wood. Both wood species lost weight after treatment at 220°C . The dimensions of specimens in tangential direction were increased by the oil-heat treatment. The degree of swelling decreased with increasing temperature. The relative crystallinity of the heat-treated woods was measured by the XRD and FTIR methods increased with

increasing temperature. In FTIR analyses, the peaks from carbohydrate changed after the oil-heat treatment, mainly due to the degradation of hemicellulose. In this study, there were considerable changes in the anatomical, physical, and chemical properties of both wood species after the oil-heat treatment. Mindi wood showed greater changes in the properties than that of gmelina. The results suggest that in the process of heat treatment, species characteristics may be important for the evaluation of the final products.

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