

## Effect of Mechanical Restraint on Drying Defects Reduction in Heat-treated Okan Wood

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Mechanical restraint through the use of clamps was applied as an attempt to prevent drying defects during the heat treatment of high-density wood. Boards of okan (*Cylicodiscus gabunensis* (Taub.) Harms) with the initial moisture content of 8.99% and 9.68% for sapwood and heartwood, respectively, were prepared. The boards were heat-treated under an oxygen atmosphere at the peak temperatures of 160 °C, 180 °C, 200 °C, and 220 °C with a residence time of 2 h. The occurrence of drying defects as checks (*i.e.*, surface and end checks) and warps (*i.e.*, bow, cup, and twist) were evaluated. Heat treatment stimulated the occurrence of drying defects in okan wood. The results revealed that the surface checks, end checks, and twists increased linearly with increased temperature. The occurrence of warps, such as bow and cup, after heat treatment was relatively low. Heartwood showed a higher degree of drying defects compared to the sapwood. Application of mechanical restraint by clamping efficiently decreased the occurrence of drying defects of okan wood, particularly surface checks, end checks, and twists.

*Keywords:* Clamping; *Cylicodiscus gabunensis*; Drying defects; Heat treatment

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### INTRODUCTION

The heat treatment of wood has been considered an environmentally-friendly modification-technology because no toxic chemicals are used in the process (Esteves and Pereira 2009). Heat treatment of wood is principally similar to wood drying, *i.e.*, both technologies apply heat to remove moisture from wood. Conventional kiln drying of wood is generally performed at approximately 100 °C without any structural changes occurring in the wood (Pang *et al.* 1994; Johanson *et al.* 1997; Poncsak *et al.* 2006). However, the heat treatment is generally performed under an inert atmosphere at temperatures ranging from 160 °C to 260 °C, causing both the removal of water and structural modification (Hill 2006; Esteves and Pereira 2009). Heat treatment changes wood chemical composition by degrading cell wall compounds and extractives, resulting in a darker wood color, reduced equilibrium moisture content, reduced thermal conductivity, improved durability against decay, improved dimensional stability, and a reduced strength that is mainly affected by treatment temperature and duration (Bekhta

and Niemz 2003; Boonstra *et al.* 2007; Kocaefe *et al.* 2008; Cao *et al.* 2012; Dubey *et al.* 2012; Hidayat *et al.* 2016; 2017).

The exposure to heat during wood-drying or heat-treatment processes will cause moisture movement, shrinkage, and the development of stress, which will lead to the occurrence of drying defects if the drying process parameters are not carefully controlled (Hoadley 2000; Liu and Wang 2016). Kollmann and Sachs (1967) studied the change in wood anatomy after heat treatment at 220 °C and observed some drying defects due to development of high internal pressures caused by high temperature treatment. In addition, Awoyemi and Jones (2011) observed the destruction of tracheid walls, ray tissues, and pit deaspiration of red cedar due to heat treatment. However, wood species react differently during the wood-drying and heat-treatment process; for example high-density wood is more prone to checking than low-density wood (Morén 1989; Hoadley 2000).

Okan wood (*Cylicodiscus gabunensis* [Taub.] Harms) is a high-density wood that grows mainly in the rain forests of West Africa, from Sierra Leone to the Cameroons and Gabon (Chudnoff 1984; Kadiri *et al.* 2005). The wood was reported to have a high rate of shrinkage during drying (Louppe *et al.* 2008) and has a marked tendency to split and check (Ayensu and Bentum 1974). The use of heat-treated okan wood for decking and flooring in South Korea is increasing due to its naturally-modified dark color and exotic visual appearance (Hidayat *et al.*, 2016).

The authors' previous work reported an improvement in the dimensional stability of okan wood *via* heat-treatment at various temperatures and duration (Hidayat *et al.* 2015; Hidayat *et al.* 2016). This paper furthers the study of the previous results and reports on the heat-treatment of okan wood stacked with metal clamps as an attempt to prevent the occurrence of drying defects during heat-treatment at various temperatures. The information of drying defects that occur during the heat-treatment is of great importance for improving efficiency and quality control in the commercial production of new wood products.

## EXPERIMENTAL

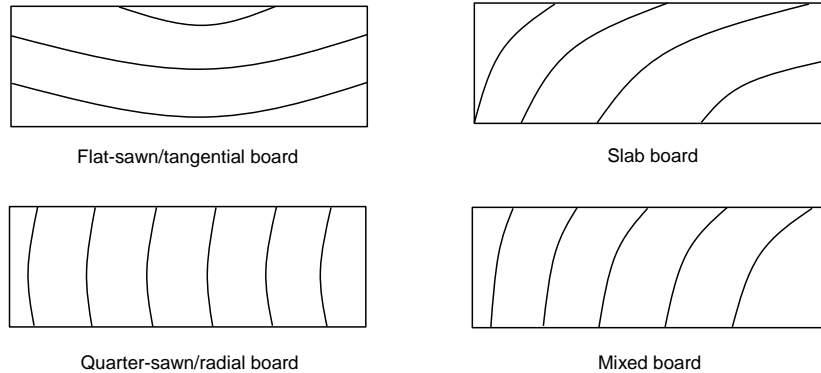
### Materials

Boards from sapwood and heartwood of okan (*Cylicodiscus gabunensis* (Taub.) Harms) were prepared for heat-treatment experiments. The boards were provided by TN WOOD in Chungju, South Korea. The dimensions of the specimens used were 300 mm × 90 mm × 20 mm (length × width × thickness). All boards were sorted according to their end-grain angles on cross-section, *i.e.*, flat-sawn or tangential board, slab board, quarter-sawn or radial board, and mixed board (Fig. 1).

Boards free of defects and with a small variation in density were selected. The boards were kept in a conditioning room under the relative humidity of 65% ± 3% and a temperature of 25 °C ± 2 °C for 2 weeks before heat-treatment. The initial moisture contents (MC) of sapwood and heartwood specimens were 8.99% ± 0.03% and 9.68% ± 0.14%, respectively.

The air-dry densities of the sapwood and heartwood specimens ranged from 0.77 g/cm<sup>3</sup> to 0.89 g/cm<sup>3</sup> and 1.16 g/cm<sup>3</sup> to 1.23 g/cm<sup>3</sup>, respectively. Six sapwood and six heartwood boards were stacked in a vertical clamps device. For comparison, another set of samples were stacked without the clamps. More detailed explanation on the sample-

stacking during heat-treatment can be found in previous papers (Hidayat *et al.* 2016).



**Fig. 1.** Illustration of the different selected wood samples submitted to heat treatment process

## Methods

### Heat treatment

Heat treatment was performed in the presence of air using an electric oven with a programmable controller (L-Series, JEIO TECH Ltd., Seoul, Korea). The heat treatment began at the initial temperature of  $25\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$  and then was raised to the target temperatures of  $160\text{ }^{\circ}\text{C}$ ,  $180\text{ }^{\circ}\text{C}$ ,  $200\text{ }^{\circ}\text{C}$ , and  $220\text{ }^{\circ}\text{C}$ , at a heating rate of  $2\text{ }^{\circ}\text{C}/\text{min}$ . The target temperatures were maintained for 2 h. In the final stage of heat treatment, the oven chamber was allowed to cool naturally until it reached  $30\text{ }^{\circ}\text{C}$ . Then, the boards were taken out and kept in a conditioned room under the relative humidity of  $65\% \pm 3\%$  and a temperature of  $25\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$  for 2 weeks until further testing.

### Board evaluation

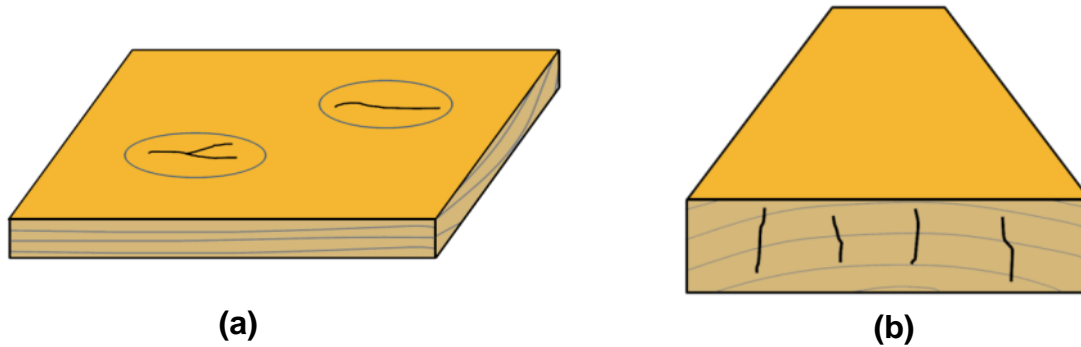
The drying defects evaluated in the samples were drying checks and warps. The drying checks included surface and end checks, while the warps included bow, cup, and twist. Six samples of sapwood and heartwood boards were stacked with and without clamps and were observed for the evaluation of drying defects at various temperatures.

Surface checks are failures that occur on the surfaces of the board, while end checks occur on the end-grain surfaces of the board (Fig. 2). The surface and end checks on each sample board with a minimum width of 0.1 mm were marked using a white highlighter, because as stated by Hanhijarvi *et al.* (2003) the width limit for the visibility of cracks by the naked eye is 0.1 mm.

The images of the surfaces and end-grain surfaces of the boards were captured using an 18-megapixel digital single-lens reflex camera (Canon EOS 100 D, Tokyo, Japan). The same acquisition conditions were set including a fixed distance between the camera and sample, fixed zoom, and fixed manual setting (*i.e.*, shutter speed 1/200, aperture f/11, focal length 55 mm, and ISO speed of 400). The images were processed using an open source image analysis software (ImageJ Version 1.50, Bethesda, Maryland, USA) to measure the length of each, surface and end checks. The average lengths of surface and end checks (C) were calculated using Eq. 1,

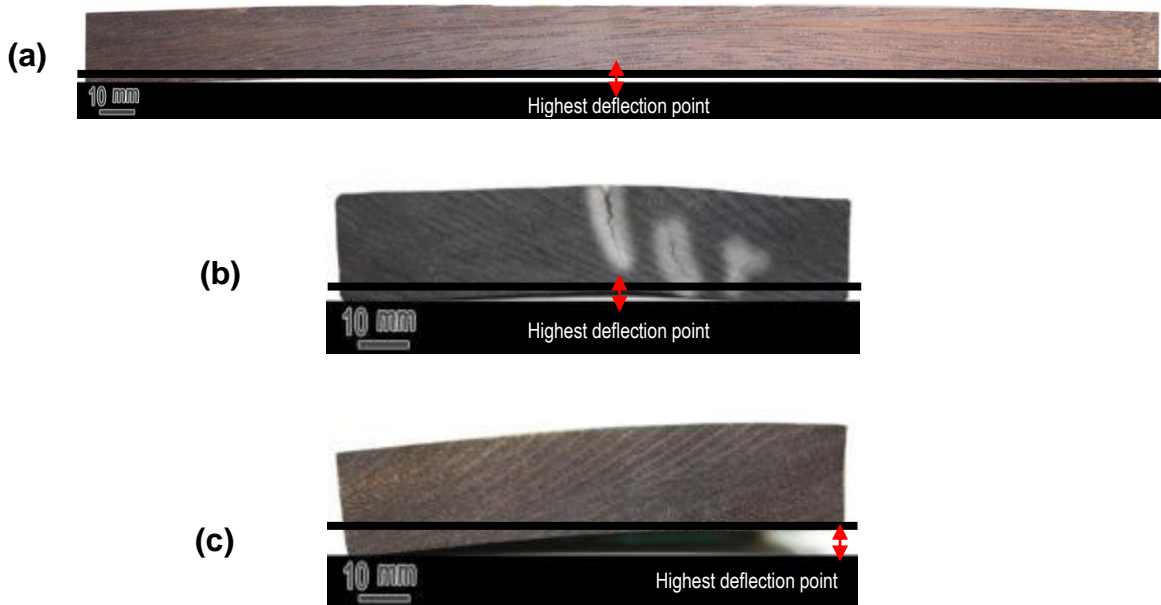
$$C = \frac{\Sigma L_1 + \Sigma L_2 + \Sigma L_3 + \Sigma L_4 + \Sigma L_5 + \Sigma L_6}{6} \quad (1)$$

where  $\Sigma L$  is the total length of surface or end check on each board sample (mm).



**Fig. 2.** Drying defects on the boards: (a) surface checks and (b) end checks

Bow and cup are the terms used for the deviation from the flatness of a board characterized by a roughly cylindrical or spherical curvature. The board with a bow defect is a board that rocks from end to end when laid on one face, while cup is a board that rocks from edge to edge when laid on one face. The board with twist defect is a board sample that rests on opposite diagonal corners when laid on one face. The defects bow, cup, and twist were evaluated by measuring the highest point of deflection (Fig. 3) in the samples.



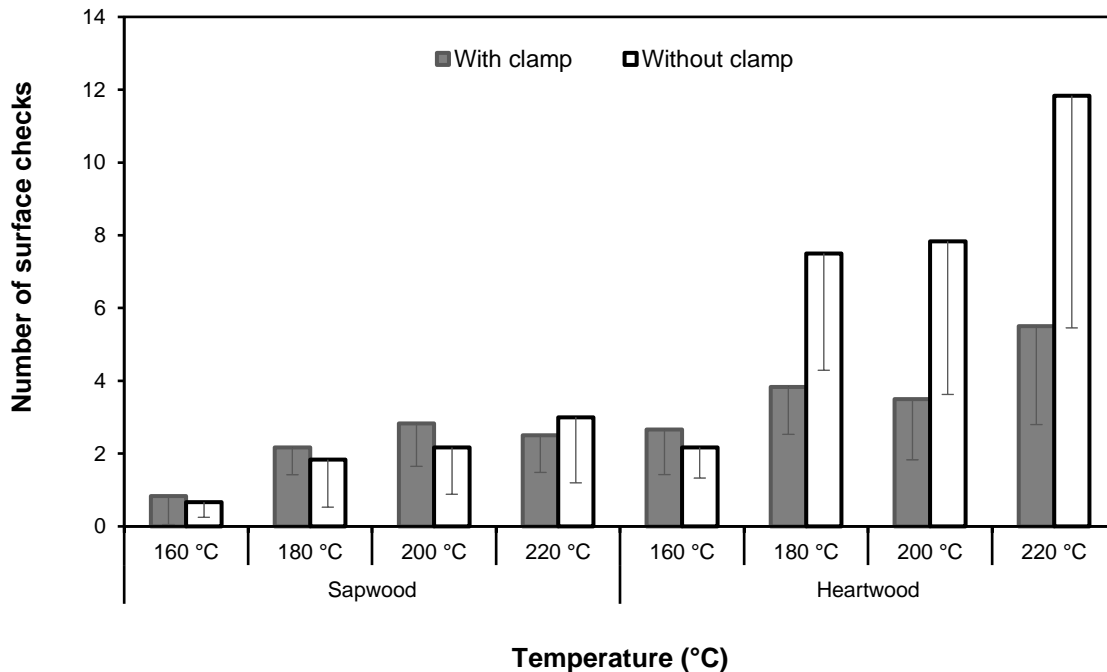
**Fig. 3.** Measurement of warps: (a) bow, (b) cup, and (c) twist

The cell morphology in the cross-section of wood before and after heat treatment were observed using a scanning electron microscope (SEM) (JEOL, JSM-5510, Tokyo, Japan, 5 kV to 20 kV). The control sample and heat-treated boards were cut into small pieces with the dimensions of 10 mm (R)  $\times$  10 mm (T)  $\times$  5 mm (L) using a small chisel. A sliding microtome (Leica RM 2155, Wetzlar, Germany) was then used to obtain a smooth surface in the cross-section. The samples were coated with approximately 10 nm of gold using an ion sputtering coater (Cressington Sputter Coater 108, Watford, England) prior to SEM observation.

## RESULTS AND DISCUSSION

### Surface Checks

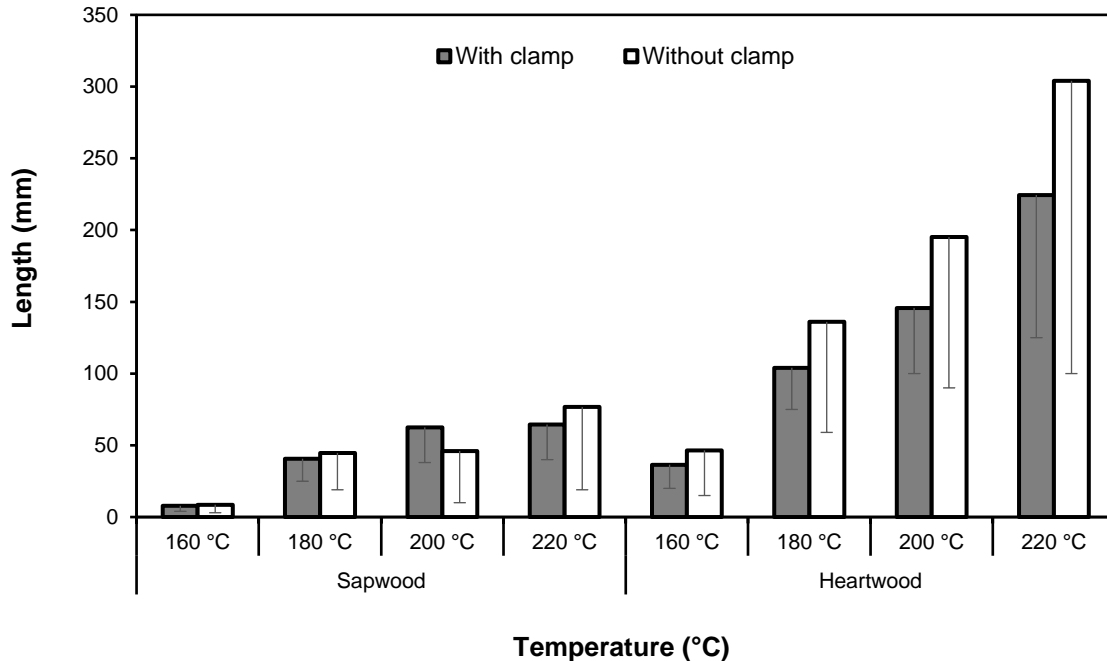
One of the negative outcomes of the heat treatment of wood is the occurrence of cracks or checks such as surface checks, end checks and splits, collapse, and honeycombs (Simpson 1991; Oltean *et al.* 2007). The results from this study have shown that the heat treatment of okan led to the occurrence of surface checks. The frequency of surface checks in heartwood was remarkably higher than in sapwood, particularly in the samples without the clamps (Fig. 4). This could have been attributed to the higher density of heartwood. Mugabi *et al.* (2010) also reported that the heartwood of *Eucalyptus grandis* is more susceptible to surface checking than the sapwood. A previous study by Morén (1989) noted that the high-density wood is more prone to checking than low-density wood. Hoadley (2000) stated that dense woods generally shrink and swell more, and usually pose greater problems in drying. However, low-density woods seem favorable to drying, apparently because of their ability to deform internally to relieve the shrinkage stress. Shrinkage stress during drying can be relieved by several methods such as: (1) by increasing the surface moisture content (MC) of the wood *via* steam conditioning (Keey *et al.* 2000); (2) by decreasing the internal MC of the wood; and (3) by increasing the MC of external wood and by decreasing the MC of internal wood simultaneously using microwave conditioning (Grinchik *et al.* 2015; He and Wang 2015).



**Fig. 4.** Average number of surface checks after heat treatment at different temperatures

Figure 5 shows that the length of surface checks increased linearly with increased treatment temperature both in sapwood and heartwood. These results were in agreement with okan wood behavior during the drying process, showing a marked tendency to split and check because of its typical interlocked grains (Ayensu and Bentum 1974). Similar to the wood drying method, water and moisture were also evaporated from wood during the heat treatment process, causing wood shrinkage.

Shrinkage is one of the most important physical properties of wood because it may develop various stresses and its magnitude directly affects the dimensional stability of wood, and it also contributes to the occurrence of surface and internal checks (Liu and Wang 2016).



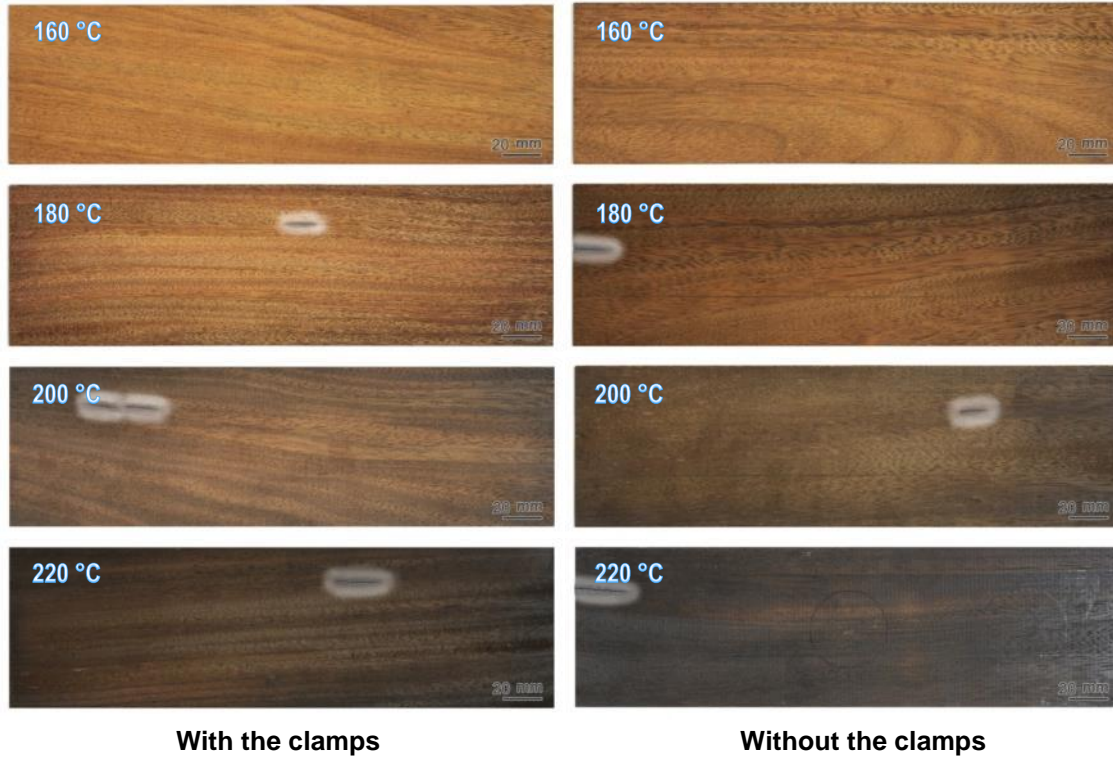
**Fig. 5.** Average length of surface checks after heat treatment at different temperatures

The results from this study showed a positive effect of clamping during heat treatment. The application of clamps effectively reduced the number and length of surface checks during heat treatment, particularly in heartwood (Figs. 4 and 5). However, in sapwood, the effect of clamping on the reduction of surface checks was not clearly seen. The visual appearances of surface checks that occurred on sapwood and heartwood of okan after heat treatment at various temperatures are shown in Figs. 6 and 7.

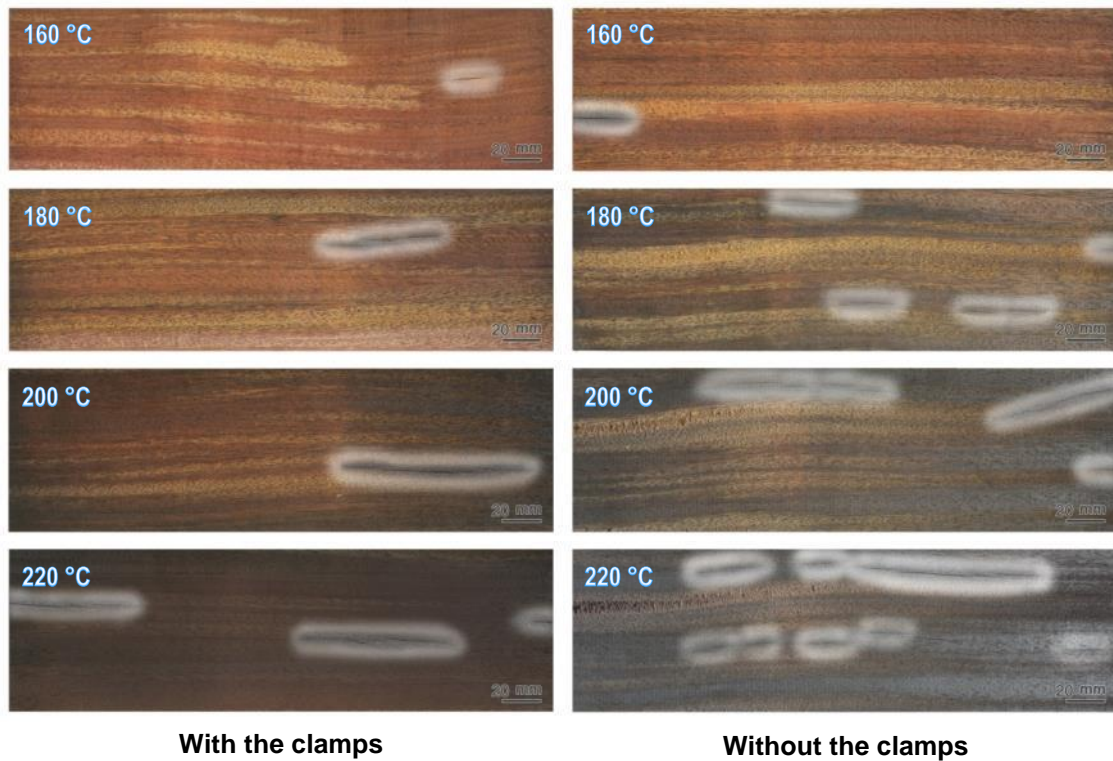
### End Checks

End checks showed similar results to surface checks, and displayed an increase in number and length of checks with increased temperature (Figs. 8 and 9). In addition, both sapwood and heartwood samples heat-treated using clamps revealed a lower degree of end checks compared to the samples heat-treated without the clamps. The occurrence of end checks in heartwood was remarkably higher than that in sapwood. Increased temperature resulted in wider end checks and in higher treatment temperatures such as 200 °C and 220 °C, the end checks developed into end splits that particularly occurred in heartwood (Figs. 10 and 11).

The results also showed that severe end checks frequently had a wavy appearance on the surface (*i.e.*, in heartwood samples heat-treated with and without the clamps at 220 °C), which is often associated with severe collapse, a deformation caused by the flattening or crushing of wood cells (Simpson 1991; Oltean *et al.* 2007).



**Fig. 6.** Surface checks in sapwood of okan after heat treatment at different temperatures



**Fig. 7.** Surface checks in heartwood of okan after heat treatment at different temperatures

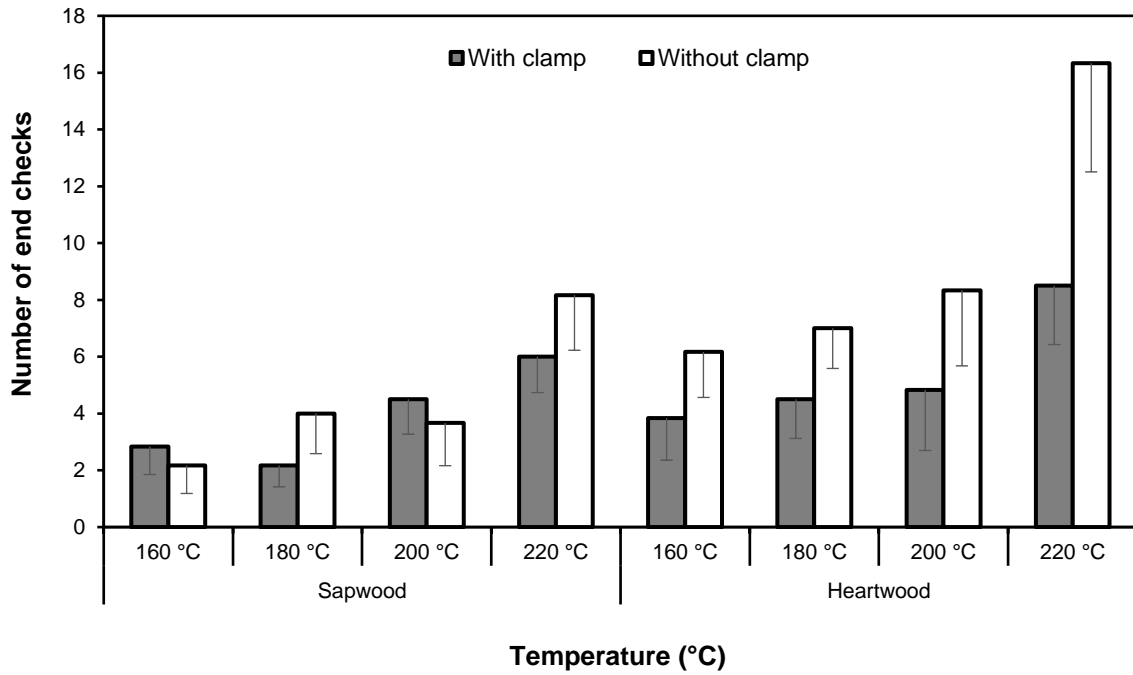


Fig. 8. Average number of end checks after heat treatment at different temperatures

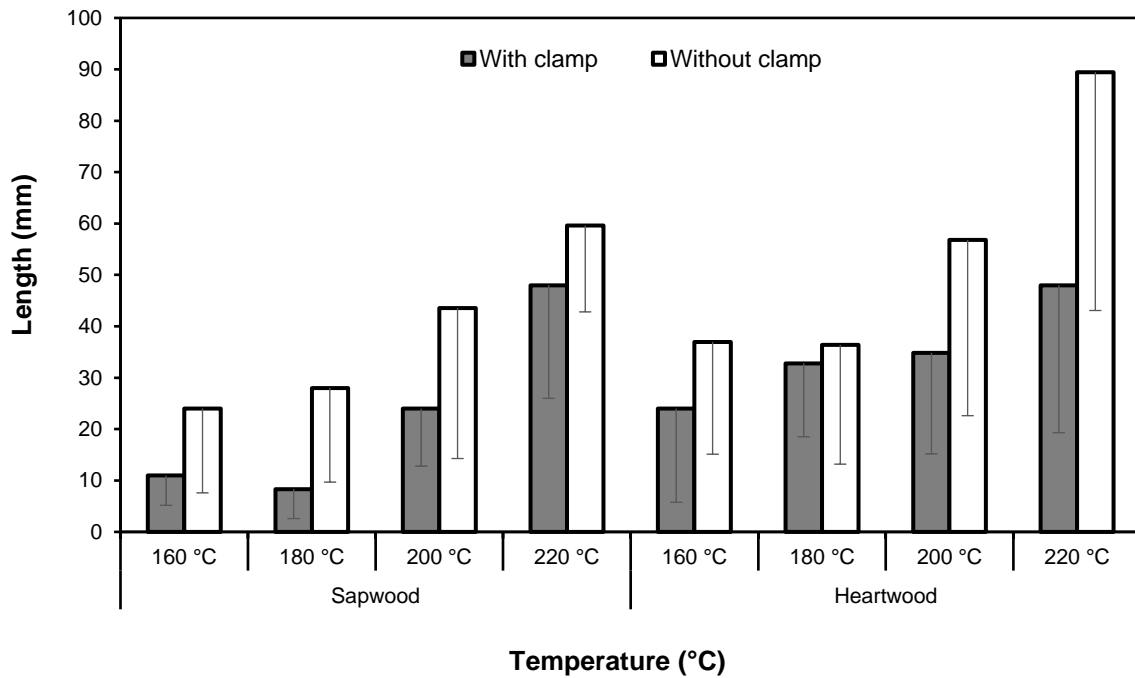
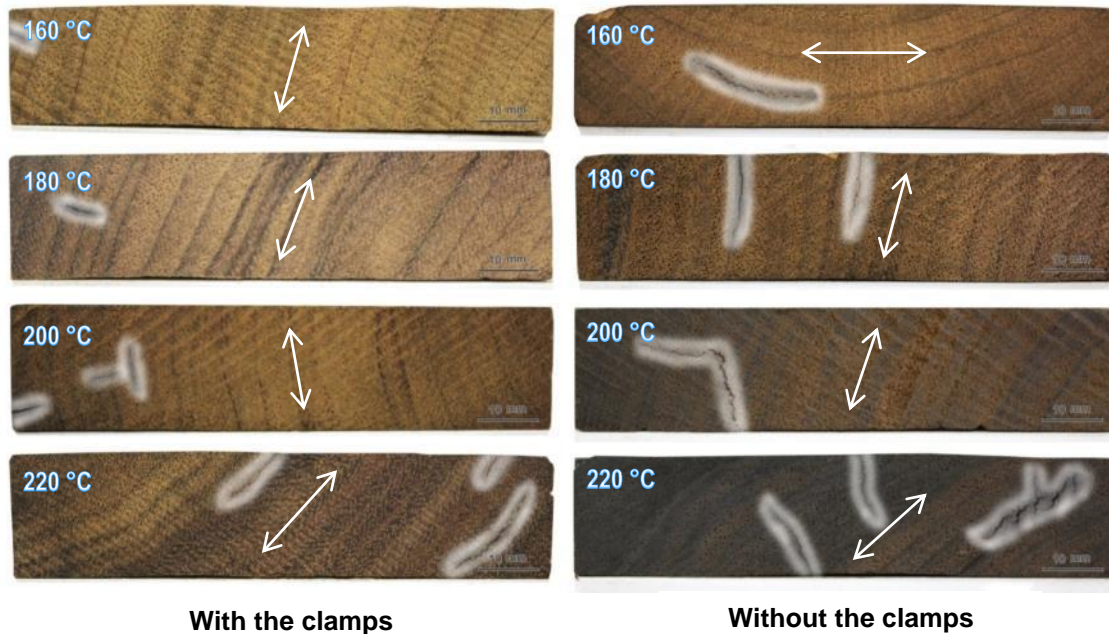
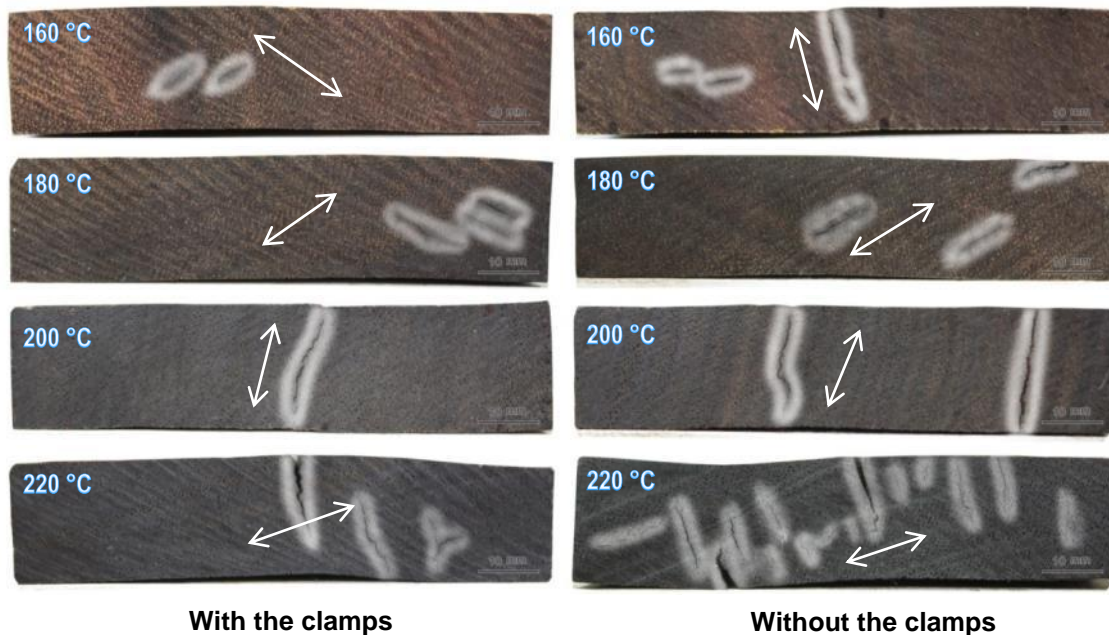


Fig. 9. Average length of end checks after heat treatment at different temperatures





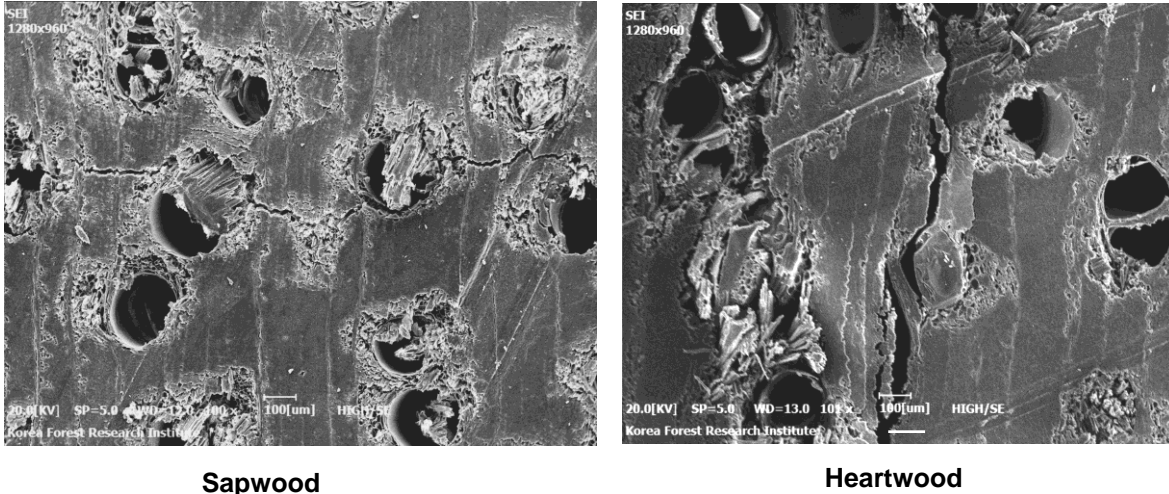
**Fig. 10.** End checks in sapwood of okan after heat treatment at different temperatures (arrows show tangential direction)



**Fig. 11.** End checks in heartwood of okan after heat treatment at different temperatures (arrows show tangential direction)

In sapwood, the end checks occurred frequently across the ray. In contrast, ring failure occurred due to the heat treatment. In heartwood the end checks occurred across and along with the rays, while the heat treatment also caused ray separation in heartwood.

Micrographs taken from the cross-sections of okan wood showed that micro-cracks occurred in sapwood and heartwood with similar results to the macro-cracks observation, which showed ring failure in sapwood and ray separation in heartwood with more severe damage (Fig. 12).



**Fig. 12.** SEM images showing end checks in sapwood and heartwood of okan after heat treatment.

### Warps

Warping, such as bow, cup, and twist, is another problem that occurs in the okan wood during its drying (Ayensu and Bentum 1974) process. A similar trend also arose after the heat treatment of okan. These results showed that the degree of bow and cup resulting from the heat treatment of okan wood was relatively low, which showed the highest deflection points of lower than 2 mm in all temperatures. Overall, the results showed that the bows and cups increased with increased temperature (Figs. 13 and 14). The sapwood tended to have lower deflections of bows and cups after heat treatment at 160 °C and 180 °C, but higher deflections occurred after heat treatment at higher temperatures of 200 °C and 220 °C. In addition, tangential and slab boards tended to have a higher degree of cup and twist than the other type of boards.

However, the occurrence of bows and twists was not always constantly increased by increased temperature. For example, Fig. 13 shows that in heartwood heat-treated with the clamps, the bows that occurred at 180 °C were higher than that at 200 °C and 220 °C. Similar results were reported by Thiam *et al.* (2002), who found that western hemlock and fir timber showed a decrease of warps, particularly bows and crooks, by applying a higher drying temperature compared to a lower drying temperature.

In general, the occurrence of bows and cups in samples with the clamps was lower than that without the clamps, particularly for the heat treatment at higher temperature such as 200 °C and 220 °C.

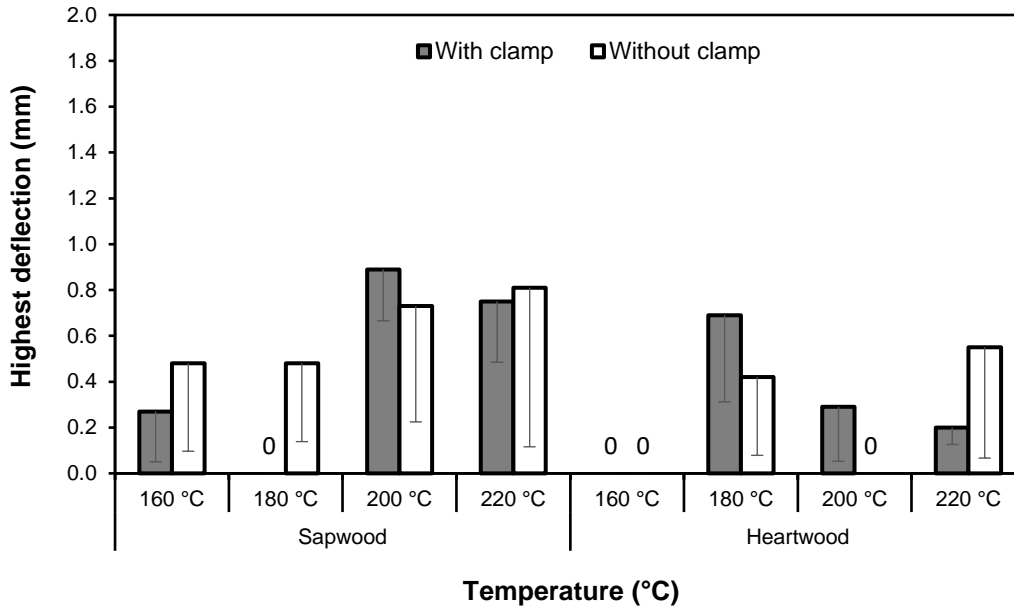


Fig. 13. Average highest deflection of bows after heat treatment at different temperatures

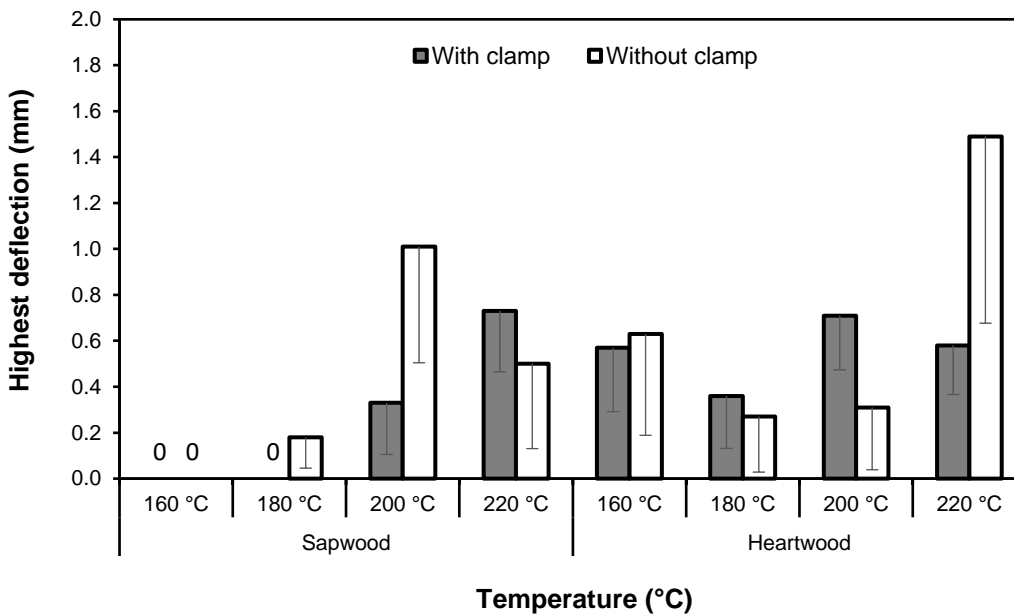


Fig. 14. Average highest deflection of cups after heat treatment at different temperatures

The degree of twists during heat treatment of okan wood was remarkably higher compared to the occurrence of bows and cups, which showed the highest deflection points of 3.87 mm and 6.54 mm in sapwood and heartwood, respectively (Fig. 15). The results also showed the increase of twists occurrence with increased temperature. The application of clamps effectively reduced the occurrence of twists, particularly in sapwood. In heartwood, during heat treatment at lower temperatures of 180 °C and 200 °C, the application of clamps slightly reduced twists. A remarkable reduction of twists by clamping can be seen during the heat treatment at a higher temperature of 220 °C.

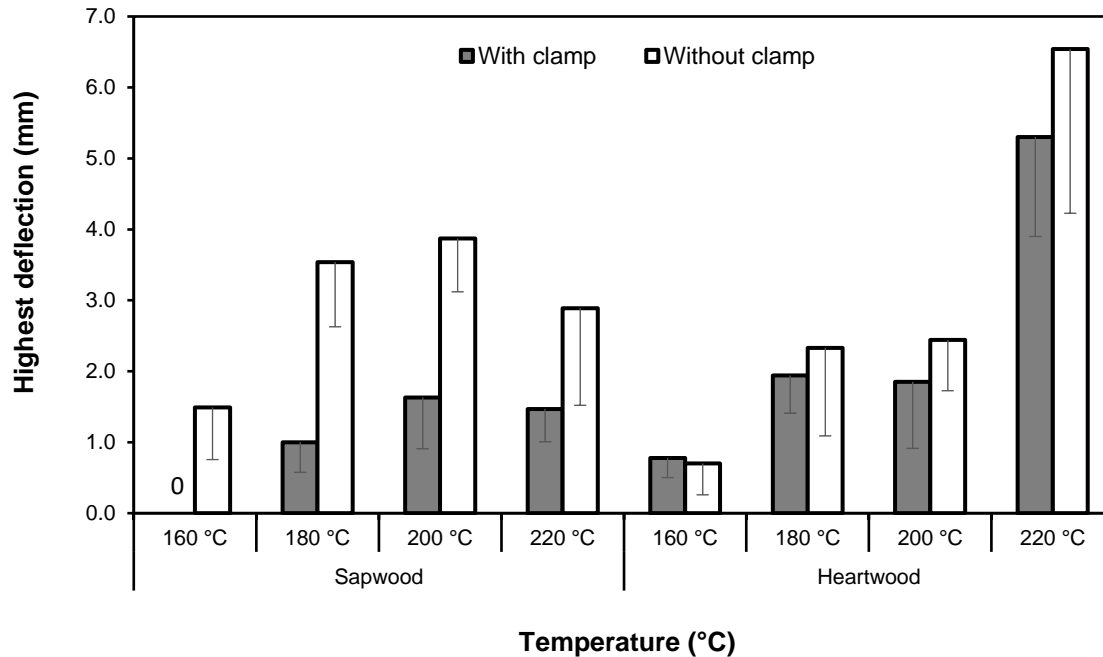


Fig. 15. Average highest deflection of twists after heat treatment at different temperatures

## CONCLUSIONS

1. A heat treatment stimulated the occurrence of drying checks and warps in okan wood.
2. The occurrence of surface checks, end checks, and twists increased with increased treatment temperature with a higher degree that occurred in heartwood compared to sapwood.
3. End checks in sapwood happened frequently across the ray, while in heartwood it occurred mainly along the ray with more severe damage.
4. The bows and cups occurrence during heat treatment was low and not considerably affected by the increase of temperature.
5. Application of mechanical restraint by clamping efficiently decreased the occurrence of drying defects during the heat treatment of okan wood, particularly surface checks, end checks, and twists.

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## REFERENCES CITED

- Awoyemi, L., and Jones, I. P. (2011). "Anatomical explanations for the changes in properties of western red cedar (*Thuja plicata*) wood during heat treatment," *Wood Science and Technology* 45(2), 261-267. DOI: 10.1007/s00226-010-0315-9
- Ayensu, E. S., and Bentum, A. (1974). *Commercial Timbers of West Africa*, Smithsonian Contributions to Botany Number 14, Smithsonian Institution Press, Washington, DC.
- Bekhta, P., and Niemz, P. (2003). "Effect of high temperature on the change in colour, dimensional stability and mechanical properties of spruce wood," *Holzforchung* 57(5), 539-546. DOI: 10.1515/HF.2003.080
- Boonstra, M. J., Van Acker, J., Kegel, E., and Stevens, M. (2007). "Optimisation of a two-stage heat treatment process: Durability aspects," *Wood Sci. Technol.* 41(1), 31-57. DOI: 10.1007/s00226-006-0087-4
- Cao, Y., Jiang, J., Lu, J., Huang, R., Jiang, J., and Wu, Y. (2012). "Color change of Chinese fir through steam-heat treatment," *BioResources* 7(3), 2809-2819. DOI: 10.15376/biores.7.3.2809-2819
- Chudnoff, M. (1984). *Tropical Timbers of the World*, Agriculture Handbook Number 607, US Department of Agriculture, Forest Service, Washington, DC.
- Dubey, M. J., Pang, S., and Walker, J. (2012). "Changes in chemistry, color, dimensional stability and fungal resistance of *Pinus radiata* D. Don wood with oil heat-treatment," *Holzforchung* 66(1), 49-57. DOI: 10.1515/HF.2011.117
- Esteves, B. M., and Pereira, H. (2009). "Wood modification by heat treatment: A review," *BioResources* 4(1), 370-404. DOI: 10.15376/biores.4.1.340-404
- Grinchik, N. N., Adamovich, A. L., Kizina, O. A., and Kharma, U. M. (2015). "Modeling of heat and moisture transfer in wood in finish drying by the energy of a microwave field," *J. Eng. Phys. Thermophy.* 88(1), 35-41. DOI: 10.1007/s10891-015-1165-y
- Hanhijarvi, A., Wahl, P., Rasanen, J., and Silvennoinen. (2003). "Observation of development of microcracks on wood surface caused by drying stresses," *Holzforchung* 57(5), 561-565. DOI: 10.1515/HF.2003.083
- He, Q., and Wang, X. (2015). "Drying stress relaxation of wood subjected to microwave radiation," *BioResources* 10(3), 4441-4452. DOI: 10.15376/biores.10.3.4441-4452
- Hidayat, W., Jang, J. H., Park, S. H., Qi, Y., Febrianto, F., Lee, S. H., and Kim, N. H. (2015). "Effect of temperature and clamping during heat treatment on physical and mechanical properties of okan (*Cylicodiscus gabunensis* [Taub.] Harms) wood," *BioResources* 10(4), 6961-6974. DOI: 10.15376/biores.10.4.6961-6974
- Hidayat, W., Qi, Y., Jang, J. H., Febrianto, F., Lee, S. H., and Kim, N. H. (2016). "Effect of treatment duration and clamping on the properties of heat-treated okan wood," *BioResources* 11(4), 10070-10086. DOI: 10.15376/biores.11.4.10070-10086
- Hidayat, W., Qi, Y., Jang, J. H., Park, B. H., Banuwa, I. S. B., Febrianto, F., and Kim, N. H. (2017). "Color change and consumer preferences towards color of heat-treated Korean white pine and royal paulownia woods," *Journal of the Korean Wood Science and Technology* 45(2), 213-222. DOI: 10.5658/WOOD.2017.45.2.213
- Hill, C. A. S. (2006). *Wood Modification: Chemical, Thermal and Other Processes*, Wiley Series in Renewable Resources, John Wiley & Sons, Ltd., West Sussex, England. DOI: 10.1002/0470021748
- Hoadley, R. B. (2000). *Understanding Wood: A Craftsman's Guide to Wood Technology*, The Taunton Press, Newtown, Connecticut.

- Johanson, A., Fhyr, C., and Rasmuson, A. (1997). "High temperature convective drying of wood chips with air and superheated steam," *Int. J. Heat Mass Transf.* 40(12), 2843-2858. DOI: 10.1016/S0017-9310(96)00341-9
- Kadiri, A. B., Olowokudejo, J. D., and Ogundipe, O. T. (2005). "Some aspects of foliar epidermal morphology of *Cylicodiscus gabunensis* (Taub.) Harms (Mimosaceae)," *J. Sci. Res. Dev.* 10, 33-38.
- Keey, R. B., Langrish, T. A. G., and Walker, J. C. F. (2000). "Kiln-drying of lumber," Springer Berlin, Germany. DOI: 10.1007/978-3-642-59653-7
- Kocaefe, D., Shi, J. L., Yang, D. Q., and Bouazara, M. (2008). "Mechanical properties, dimensional stability, and mold resistance of heat-treated jack pine and aspen," *Forest Prod. J.* 58(6), 88-93.
- Kollmann, F.F.P., and Sachs, I.B. (1967). "The effects of elevated temperature on certain wood cells," *Wood Science and Technology.* 1(1): 14-25. DOI: 10.1007/BF00592253
- Liu, J. X., and Wang, X. M. (2016). "Effect of drying temperature and relative humidity on contraction stress in wood," *BioResources* 11(3), 6625-6638. DOI: 10.15376/biores.11.3.6625-6638
- Louppe, D., Oteng-Amoako, A. A., and Brink, M. (Eds). (2008). *Plant Resources of Tropical Africa 7(1): Timber 1*, PROTA Foundation, Backhuys Publishers, Wageningen, Netherlands.
- Morén, T. J. (1989). "Check formation during low temperature drying on Scots pine: Theoretical considerations and some experimental results," in: *Proceedings of the IUFRO International Wood Drying Symposium*, Seattle, Washington, USA.
- Mugabi, P., Rypstra, T., Vermaas, H. F., and Nel, D. G. (2010). "Relationships between drying defect parameters and some growth characteristics in kiln-dried South African grown *Eucalyptus grandis* poles," *J. Eur. J. Wood Prod.* 68(3), 329-340. DOI: 10.1007/s00107-009-0375-4
- Oltean, L., Teischinger, A., and Hansmann, C. (2007). "Influence of temperature on cracking and mechanical properties of wood during wood drying –A review," *BioResources* 2(4), 789-811. DOI: 10.15376/biores.2.4.789-811
- Pang, S., Langrish, T. A. G., and Keey, R. B. (1994). "Moisture movement in softwood timber at elevated temperatures," *Dry Technol.* 12(8), 1897-1914. DOI: 10.1080/07373939408962212
- Poncsak, S., Kocaefe, D., Bouazara, M., and Pichette, A. (2006). "Effect of high temperature treatment on the mechanical properties of birch (*Betula papyrifera*)," *Wood Sci. Technol.* 40(8), 647-663. DOI: 10.1007/s00226-006-0082-9
- Simpson, W. T. (1991). *Dry Kiln Operator's Manual, Chapter 2. Kiln Types and Features*, Forest Products Laboratory, Madison, WI, 198, pp. 43-73.
- Thiam, M., Milota, M. R., and Leichti, R. (2002). "Effect of high-temperature drying on bending and shear strengths of western hemlock lumber," *Forest Prod. J.* 52(4): 64-68.

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