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Mechanical properties and water resistance of Vietnamese acacia and rubberwood after thermo-hygro-mechanical modification

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Abstract

Low density and poor mechanical performance often limit utilisation of sawn wood from fast-growing plantation forests. Thermo-hygro-mechanical modification (THM) of timber is one innovation for improving the properties of light-weight wood species. The objective of this study was to determine the effects of THM and subsequent thermal treatment on dry density, modulus of elasticity (MOE), compression strength, Brinell hardness, and swelling behaviour in immersion tests on two fast-growing Vietnamese species, acacia (Acacia mangium) and rubberwood (Hevea brasiliensis). Test boards were modified in an industrial kiln, in which a tangential thickness compression of 14% and 12% were aimed for acacia and rubberwood, respectively, either with or without subsequent thermal treatment at 210 °C. Dry density, MOE, Brinell hardness, compression strength, and dimensional changes in water immersion tests of specimens were measured from the modified and unmodified reference materials, the latter ones being kiln dried at 50 °C. The results showed that the responses of the mechanical properties were more evident for rubberwood than for acacia. In rubberwood, the MOE and compression strength of wood thermo-hygro-mechanically modified with or without thermal treatment were higher than those of kiln-dried reference specimens throughout the thickness profile. In case of acacia, similar differences between the modified and reference specimens were observed only in the surface layer. Density and Brinell hardness of thermo-hygro-mechanically modified rubberwood were higher than those of reference specimens, but after thermal treatment they did not differ from (acacia) or were lower (rubberwood) than those of THM specimens. Post-compression thermal treatment increased the hydrophobicity of THM specimens.

1 Introduction

Fast-growing acacia (*Acacia mangium*) and rubber tree (*Hevea brasiliensis*) plantations have been successfully commercialised in Southeast Asia thanks to their adaptability to various climatic and soil conditions and short rotation period (e.g., Nguyen 2013; Hai et al. 2015). Their applications in wood product industries are still limited by the relatively low density and poor dimensional stability of wood (Mohammed Raphy et al. 2011; Teoh et al. 2011; Nambiar et al.

Veikko Möttönen veikko.mottonen@luke.fi 2014; Shukla and Sharma 2018). Consequently, their timber is mostly used by kraft pulping industries or as firewood.

Mechanical properties of wood are highly dependent on wood density (Pelit et al. 2018). Densification or compression in transverse direction is considered as an effective and environmentally friendly method to increase the density of wood. Densification reduces the void volume of lumens and reshapes the morphology of cells without loss of lignin, resulting in improvement of mechanical properties such as modulus of elasticity, compression strength, tensile strength, hardness, and abrasion resistance of low-density wood species (Anshari et al. 2011; Sandberg et al. 2013). However, once the atmospheric humidity and heat in the compression process terminate, elastic energy stored in microfibrils of compressed wood will be released, resulting in springing back towards its original shape, which is defined as "shape memory" or "set recovery" (Navi and Sandberg 2012). This phenomenon is considered as the biggest problem of the process, regardless of the mechanical property elevation of compressed wood (e.g., Möttönen et al. 2015).

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It is crucial to improve the dimensional stability of densified wood in the direction of compression to ensure further processing and usability of end products. Although the shape memory cannot be totally eliminated, it can be restricted by adjusting the heat, compressive force, and pressing time during the densification process (e.g., Navi and Girardet 2000; Kúdela et al. 2018). Depending on the combination of relative humidity and temperature used during the process, densification can be categorised into two types, thermo-hygro-mechanical modification (THM) and thermo-mechanical modification (TM). In THM process, atmospheric temperature and humidity are well-controlled and the compressive forces are subjected to wood in radial or tangential directions (Dogu et al. 2010; Sandberg and Kutnar 2016). In TM process, the compression takes place in an open system without detailed control of relative humidity, being also widely applied in the industries (Kúdela et al. 2018). If the conditions are well-controlled in THM, higher values of density and stronger surface layers would be achieved, resulting in surfaces well applicable to flooring, furniture, and decorative uses (Gao et al. 2016).

Post-compression thermal treatment is known as another way to improve the dimensional stability of compressed wood, thereby offsetting the shape memory of THM modified wood (e.g., Gong et al. 2010; Möttönen et al. 2013; Yan and Morrell 2014; Möttönen et al. 2015; Sandberg and Kutnar 2016; Marttila et al. 2017; Sandberg et al. 2017). During the thermal treatment process, wood is exposed to high temperature (≥ 160 °C) (e.g., Millet and Gerhards 1972; Hillis 1975; Kocaefe et al. 2015). In this process, the structure of chemical components in wood will be altered to a specific extent, thereby affecting other properties of wood (e.g., hygroscopicity, equilibrium moisture content, dimensional stability, fungal and insect resistance, mechanical properties, colour, and odour) (e.g., Sandberg et al. 2017). Optimised parameters during the thermal treatment processes need to be designed for specific species, considering their inherent properties (Sandberg et al. 2013).

The objective of this study was to investigate the effect of thermo-hygro-mechanical modification and subsequent thermal treatment on selected mechanical properties, i.e., modulus of elasticity, compression strength, and Brinell hardness, and extreme-condition swelling behaviour of plantation-grown Vietnamese acacia and rubberwood.

2 Materials and methods

Frozen logs of two different tree species from Vietnam, acacia (*Acacia mangium*) and rubber tree (*Hevea brasiliensis*), were transported by sea freight to Juankoski, Finland, where they were melted, sawn into boards, and, finally, thermohygro-mechanically (THM) modified. The pilot modification kiln patented by KWS Timber Tech Ltd (Finland) allows wood drying, mechanical compression, and subsequent thermal treatment in one single kiln unit. Different combinations of various process phases can be achieved by adjusting parameters such as temperature, hydraulic pressure, relative humidity, steaming, and treatment time. The boards were divided into six groups: four groups for different modification treatments and two reference groups, which were dried at 50 °C without any modification (Table 1).

In the kiln, the boards were placed between perforated aluminium plates, where the compression takes place evenly on the boards and their moisture content can be indirectly controlled by the air circulation rate between the plates (Fig. 1). Drying and hydraulic compression proceeded simultaneously. The drying temperature was steadily increased up to 130 °C for acacia and 120 °C for rubberwood, and the nominal degrees of mechanical compression (thickness change/original thickness) for acacia and rubberwood were set to 14% and 12%, respectively. The different values of modification parameters were used due to difference in the initial basic density between the species. After the drying and mechanical compression, a three-hour thermal treatment at 210 °C was instantly applied to one group of the boards of both

 Table 1
 Treatments and number of boards in the six treatment groups

Code	Group	Treatment	Number of boards	Compres- sion degree (%)	Time and tem- perature in thermal treatment
A	Unmodified acacia	Reference, 50 °C oven-drying	14	-	-
AC	Thermo-hygro-mechanically modified acacia	Compression, kiln drying	10	14	-
ACT	Thermo-hygro-mechanically modified acacia with subsequent thermal treatment	Compression and thermal treatment	9	14	210 °C, 3 h
R	Unmodified rubberwood	Reference, 50 °C oven-drying	12	_	-
RC	Thermo-hygro-mechanically modified rubberwood	Compression, kiln drying	11	12	-
RCT	Thermo-hygro-mechanically modified rubberwood with subsequent thermal treatment	Compression and thermal treatment	10	12	210 °C, 3 h



Fig. 1 Above: A set of acacia boards $(60 \times 80 \times 1200 \text{ mm})$ after the compression process loaded in the THM treatment kiln. Below: Brinell hardness test specimens of reference and modified acacia and rubberwood. From the left: A, AC, ACT, R, RC, RCT

species (compressed and thermally treated acacia and rubberwood). During this process, certain amount of steam was applied in the kiln to protect the boards from oxidation reactions. After the thermal treatment process, the system was cooled down, and the compression was released.

Moisture content (MC, %) and dry density (ρ_b , kg/m³) of the specimens from all boards were determined by the gravimetric method. The dimensions of the specimens, which were sawn from one end of each modified and unmodified board, were 50×80×50 mm and 60×80×50 mm (thickness×width×length), respectively.

Matertest model FMT-MEC 100 kN (Finland) material testing device was used to carry out the modulus of elasticity (MOE) test and to record the MOE value of each board. Prior to the MOE test, the boards of groups A (acacia) and R (rubberwood) were dried in an oven at a temperature of 50 °C for two weeks, while those of AC (compressed acacia), ACT (compressed and thermally treated acacia), RC (compressed rubberwood), RCT (compressed and thermally treated rubberwood) were conditioned indoors for two weeks in order to achieve the adequate moisture level $(12 \pm 5\%)$ required by the testing standards.

MOE (E_w , MPa) was determined using static three-point bending test according to the standard ISO 13061-4, where the applied forces caused the deflection in the mid-span of each board:

$$E_W = \frac{Pl^3}{4bh^3 f} \tag{1}$$

where *P* is the load equal to the difference between the upper and lower limits of loading (N), *l* is the span (mm), *b* is the width of the test specimens (mm), *h* is the height of the test specimens (mm), and *f* is the deflection between the upper and lower limits of loading (mm). MOE values were determined from the full-length boards using the span of 750 mm and 840 mm for modified and reference specimens, respectively. Different spans were used to maintain equal height-to-length ratio of 1:14 between the untreated and treated specimens. After this, specimens were utilised in measurements of density, compression strength, swelling behaviour, and Brinell hardness.

Compression strength parallel to grain was determined using Zwick Z050 (Germany) material testing machine according to ISO 13061-12. All the samples were stored in the conditioning chamber at 20 °C and 65% relative humidity until they reached equilibrium moisture content (EMC), i.e., the mass did not change anymore. The ultimate strength in compression (σ , MPa) was determined according to the standard ISO 13061-17 using gradually increasing load in parallel to the grain direction:

$$\sigma = \frac{F_{\text{max}}}{ab} \tag{2}$$

where F_{max} is the maximum load force (N), *a* and *b* are the cross-sectional dimensions of the test specimen (mm). Nominal dimensions of the compression test specimens were $20 \times 20 \times 60$ mm (radial × tangential × longitudinal).

The MOE in bending and the ultimate compression strength were computationally adjusted to 12% moisture content using the following formulae (ISO 13061-4), which are valid for moisture content of $12 \pm 5\%$:

$$E_{12} = \frac{E_W}{1 - \alpha(W - 12)}$$
(3)

$$\sigma_{12} = \sigma(1 + \alpha(W - 12)) \tag{4}$$

where E_{12} is the calibrated MOE value at the moisture content of 12% (MPa), σ_{12} is the calibrated compression strength at the moisture content of 12% (MPa), α is the correction factor (0.02) for the moisture content, *W* is the moisture content of wood during the test, determined according to ISO 13061-1.

Prior to the immersion test, all the test specimens were stabilised in the normal climate chamber at 20 °C and 65% relative humidity until their EMC was reached. After that, the initial length, width, and thickness of the specimens were measured using calliper. Next, the specimens were soaked in buckets filled with tap water and stored at room temperature for 14 days. The dimensions were measured from the same positions once again after the 14 days of soaking. The dimensional changes were determined according to the formula:

$$\alpha = \frac{L_1 - L_0}{L_0} \times 100\%$$
(5)

where α is the swelling rate in length, width, and thickness (%), L_0 is the initial dimension (length, width, and thickness, mm), L_1 is the dimension (length, width, and thickness, mm) after the 14-day immersion.

Brinell hardness (*HB*) (kg/mm²) of the test specimens was determined according to the standard EN 1534 (2010):

$$HB = \frac{2F}{g\pi D \left(D - \sqrt{D^2 - d^2} \right)} \tag{6}$$

where *F* is the maximum load applied (1000 N), *g* is the acceleration of gravity (9.81 m/s²), *D* is the diameter of the indenter (10 mm), *d* is the average value of the diameter $[(d_1+d_2)/2]$ on specimen surface (mm), d_1 and d_2 being the diameters of the residual indentation along the grain and across the grain, measured with calliper.

3 Results and discussion

3.1 Dry density

Significant differences were found between R and RC, as well as RC and RCT, suggesting that THM had a positive effect on dry density of rubberwood (increase by 7.3%), while subsequent thermal treatment decreased the dry density from RC to RCT by 7.5% (Tables 2, 3). This might indicate that the THM changed the structure of rubberwood tissue to contain less void space and more solid cell wall per unit volume, thereby increasing the dry density (e.g., Kutnar et al. 2008; Fang et al. 2012). The post-compression thermal treatment caused degradation of holocellulose, in particular hemicellulose, and evaporation of most extractives and volatile organic compounds (VOCs) resulting in mass loss of cell walls (Sandberg and Kutnar 2016; Severo et al. 2016; Shukla and Sharma 2014).

No significant differences were observed between the experimental groups of acacia. High initial moisture content or inappropriate compression temperature, pressure, or time might lead to the failure of compression, in which the whole thickness profile of acacia specimens was not totally subjected to the compressive pressure (see: Kúdela et al. 2018). Another potential reason is that the strong springback effect in AC specimens counterbalanced the positive effect of compression on the density of acacia (Pelit et al. 2018). The sea freight of frozen logs at green state may

Treatment	MC (%)		Dry density (kg/m ³)		MOE (MPa)		HB (kg/ mm ²)		CS-C (MPa)		CS-S (MPa)	
	\overline{x}	σ	$\overline{\overline{x}}$	σ	\overline{x}	σ	\bar{x}	σ	\overline{x}	σ	\overline{x}	σ
Untreated (A)	22.6	11.6	571	65	10.6	2.3	2.6	0.9	49.6	2.0	49.6	2.0
Compression (AC)	7.1	2.2	590	79	9.8	1.2	2.8	1.1	56.5	2.8	61.2	1.9
Compression and thermal (ACT)	3.1	0.4	553	70	10.6	1.0	1.9	0.6	58.8	1.9	61.0	3.1
Untreated (R)	7.9	1.3	620	40	8.1	0.8	2.5	0.5	49.7	1.4	49.7	1.4
Compression (RC)	6.0	0.3	665	40	11.3	1.4	3.9	0.9	59.4	1.9	59.8	2.4
Compression and thermal (RCT)	4.2	0.2	615	38	11.1	0.8	2.3	0.4	60.8	1.7	63.5	1.2

HB

0.538

0.008*

0.001*

0.000*

0.578

0.000*

CS-C

0.176

0.124

0.974

0.007*

0.002*

0.981

CS-S

0.004*

0.021*

1.000

0.005*

0.000*

0.597

Swelling T

0.000*

0.012*

0.085

0.000*

0.000*

0.002*

Table 3Significance levels ofthe differences in dry density,MOE, Brinell hardness (HB),compression strength in the core(CS-C) and surface layer (CS-S), and swelling (T) between thedifferent treatment groups

Table 2 Mean values (x) and standard deviations (σ) of physical and mechanical

properties

*Significancy at $p \le 0.05$

Pair

A-AC

A-ACT

AC-ACT

R-RC

R-RCT

RC-RCT

Dry density

0.786

0.826

0.493

0.027*

0.959

0.019*

MOE

0.478

0.998

0.584

0.000*

0.000*

0.806

also have had adverse effects on physical and mechanical properties of wood, for example, by causing frost cracks (Kubler 1988). The higher moisture content of green acacia logs (65.8%) compared to green rubber tree logs (47.9%) might have resulted in the observed differences between the species.

3.2 Modulus of elasticity (MOE)

Compared to the reference material, THM and THM with subsequent thermal treatment resulted in an increase in the MOE of rubberwood of 39.5% and 35.9%, respectively, while no increase was observed in acacia (Tables 2, 3). The differences between species might partly be caused by the differences in the chemical composition of wood. The chemical and mechanical properties have been found to correlate and to be dependent on the height and clone of rubber trees (Riyaphan et al. 2015).

Several studies have shown that densification is an effective way to improve the mechanical properties of wood (e.g., Kutnar et al. 2008; Anshari et al. 2011; Fang et al. 2012; Möttönen et al. 2015; Gao et al. 2016). Generally, the bending stiffness of wood increases proportionally to density increment as a result of compression (Kutnar 2012). Increasing temperature up to 150 °C during compression also has a positive effect on MOE value of wood as it reduces the spring-back effect of compressed wood, although the increase in MOE caused by high temperature is not as obvious as that caused by high compression ratio (Tabarsa and Chui 1997; Lamason and Gong 2007).

The effect of thermal treatment on MOE is dependent on wood species, temperature, initial moisture content, surrounding atmosphere, and reaction time (Mitchell 1988). Therefore, the effects can be either positive (Fang et al. 2012; Shi et al. 2007; Heräjärvi 2009) or negative (Gong et al. 2010; Johansson and Morén 2006), depending on the parameters applied during the process. Thermo-hygro-mechanical modification followed by thermal treatment increased the MOE of green European aspen (Populus tremula) by 18% and that of Silver birch (Betula pendula) by 6% with densification degrees of 30% and 10%, respectively (Marttila et al. 2016). High temperature and long processing time may decrease the MOE due to the deterioration of wood components, particularly hemicellulose (Yan and Morrell 2014; Korkut and Aytin 2015). In addition, since the proportion of amorphous cellulose decreases with increasing temperature (e.g., Sivonen et al. 2002; Yildiz and Gümüşkaya 2007; Cai et al. 2018), an increase in crystallinity of cellulose may cause an increase in the MOE. However, increase in MOE is associated with the reduced moisture content in the modified wood (Xie et al. 2013). Usually moisture content is negatively correlated with MOE below the FSP (Kretschmann 2010). The dependence of MOE or compression strength on moisture content may differ between modified and unmodified wood. Therefore, the computational adjustment of MOE and compression strength values to 12% moisture content may not be proportional between treatments. Detailed characterization of chemical changes and crystallisation would require techniques such as Fourier transform infrared spectroscopy (FTIR) or differential scanning calorimetry (DSC). Diouf et al. (2011) found cleavage of hemicellulose and removal of high carbon compounds and extractives using Fourier transform infrared spectroscopy (ATR-FTIR) and X-ray photoelectron spectroscopy (XPS), respectively.

Decrease in MOR values after thermal treatment of wood has been noticed in acacia (Wahab et al. 2012) rubberwood (Zhou et al. 2019) and in several other species (e.g., Millett and Gerhards 1972; Heräjärvi 2009; Widmann et al. 2012). Marttila et al. (2016) observed that subsequent thermal treatment at 190 °C after thermo-hygro-mechanical modification of green timber reduced MOR values of European aspen and Silver birch by 10% and 16%, respectively. The decrease in bending strength might be due to the hemicellulose degradation (Kass et al. 1970; LeVan et al. 1990; Kocaefe et al. 2008). Bending strength was not measured in this study.

3.3 Compression strength

Both THM and THM with subsequent thermal treatment increased the compression strength of surface specimens of acacia by 23%, while the change in compression strength of core specimens was insignificant. In case of rubberwood, the increments in compression strength of surface and core specimens were 20% and 25%, respectively (Tables 2, 3).

There are several explanations for the increment of compression strength achieved by thermal treatment. Firstly, increase in crystallinity increases the stiffness of wood in its longitudinal direction (Anderson et al. 2005; Yildiz et al. 2006). Secondly, increased cross-linking of lignin polymer network can better connect and stiffen cellulose fibrils and prevent them from bending or crashing when they are subjected to compressive forces, thereby increasing the longitudinal compression strength of wood (Boonstra et al. 2007).

3.4 Swelling behaviour in immersion test

The results showed that modified specimens of both species swell more in the direction of compression than the untreated references: 385%, 218%, 172%, and 114% higher dimensional changes were detected in AC, ACT, RC, and RCT, respectively, compared to their references (Tables 3, 4). It seems that the irreversible set recovery phenomenon explained the swelling in the modified specimens, in addition to the reversible hygroscopicity of wood itself (Fang et al. 2012).

Table 4 Mean values (\bar{x}) , standard deviations (σ) , and significance levels (p) (compared with untreated wood) of dimensional stability in tangential (T), longitudinal (L) and radial (W) directions

Treatment	Swelling T	Swelling L	Swelling W
A			
\overline{x}	1.1%	0.2%	0.7%
σ	0.7%	0.1%	0.5%
р	0.001*	0.666	0.202
AC			
\overline{x}	5.4%	0.1%	0.9%
σ	1.5%	0.3%	0.6%
р	0.000*	0.978	0.616
ACT			
\overline{x}	3.5%	0.1%	1.3%
σ	2.9%	0.1%	0.7%
р	0.045*	0.996	0.022*
R			
\overline{x}	2.7%	0.1%	2.3%
σ	0.8%	0.3%	1.1%
р	0.000*	0.958	0.001*
RC			
\overline{x}	7.4%	-0.1%	1.3%
σ	1.1%	0.1%	0.4%
р	0.000*	0.987	0.231
RCT			
\overline{x}	5.8%	-0.0%	1.8%
σ	0.9%	0.1%	0.4%
р	0.000*	1.000	0.001*

*Significancy at $p \le 0.05$

As much as 21% lower thickness swelling was detected in RCT specimens compared with RC specimens. Thus, the set-recovery of compressed rubberwood was partially eliminated by thermal treatment (Table 4). This might be explained by the following mechanisms: firstly, thermal treatment above 200 °C temperature can increase the cross-linking network in lignin, which increases the hydrophobicity of wood and reduces its swelling (Santos 2000). Secondly, the stored elastic stresses are released because of degradation of hemicellulose at high temperature (Navi and Sandberg 2012). Elimination of some hygroscopic hydroxyl groups due to the depolymerisation of carbohydrates explains the improved hydrophobicity in thermally treated wood (Xie et al. 2013). In addition, in cellulose components, amorphous regions in cellulose are degraded due to acid catalysis at high temperature, which increases the crystallinity and thereby decreases the accessibility of water molecules in hydroxyl groups (e.g., Cai et al. 2018; Kúdela et al. 2018).

3.5 Brinell hardness

An average increment of 58% in Brinell hardness was detected in RC specimens, whereas no difference was observed between A and AC (Tables 2, 3). Flattening of fibre lumens and vessels in THM may increase the Brinell hardness of wood (Fang et al. 2012). However, a slight loss of Brinell hardness takes place due to increased temperature during the thermo-hygro-mechanical modification (Fang et al. 2012). Typically, higher degree of compression leads to higher Brinell hardness value, although Rautkari et al. (2013) argued that Brinell hardness is mainly influenced by the density and hardness of the surface layer. In addition to varying densities among wood species, other factors such as modification temperature, final density, moisture content, techniques of measurement, and measuring conditions (e.g., load level, loading time) also influence the results of surface hardness (e.g., Holmberg 2000; Gašparík et al. 2016).

On the other hand, compared to AC and RC, subsequent thermal treatment caused the reduction in Brinell hardness in ACT and RCT by 33% and 41%, respectively, which was probably due to the deterioration of cell wall structures (Pelit et al. 2015). High temperature and long processing time typically result in greater reduction in Brinell hardness because of the degradation of hemicellulose and lignin at high temperatures (Fang et al. 2012; Salca and Hiziroglu 2014). In addition, reduction in hardness due to heat treatment was found to be highest for wood species with high density (Kesik et al. 2014; Salca and Hiziroglu 2014). Junkkonen and Heräjärvi (2006) also pointed out that thermal treatment alters the character of hardness: because of the loss in elasticity, the indentation on the thermally modified wood surface remains, and unlike in unmodified wood, virtually no springback of the indentation takes place in the course of time. Therefore, even if the measured Brinell hardness value may be identical, the end use related effects of any indentation are far more critical in thermally modified wood.

4 Conclusion

Thermo-hygro-mechanical modification is a potential technique to improve some properties of rubberwood (density, stiffness, compression strength, Brinell hardness) and acacia (surface compression strength). However, modified wood swells more than non-modified wood under extreme ambient conditions. Post-compression thermal treatment improves the dimensional stability under such conditions, but reduces the hardness of the surface.

The unfavourable effects of any single modification method can be partially compensated by applying combinations of modification methods and appropriate process parameters. Further experiments are needed to investigate the influence of treatment time, compressive force, and modification temperature on acacia and rubberwood properties. Detailed analysis on the economics of thermo-hygromechanical modification process would provide a basis for planning wood modification investments in Vietnam.

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Compliance with ethical standards

Conflict of interest On behalf of all authors, the corresponding author states that there is no conflict of interest.

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