# Impact of Cyclic Densification on Bending Strength and Modulus of Elasticity of Wood from Temperate and Tropical Zones

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Oak (*Quercus robur* L.), iroko (*Milicia excelsa* (Welw.) C.C. Berg), and tauari (*Couratari* spp.) wood were subjected to cyclic thermo-mechanical treatment (CTMT). The densification temperature amounted to 100 °C or 150 °C. The greatest changes in the modulus of rupture (MOR) value of the iroko wood, depending on the number of thermo-mechanical modification cycles, were noted. The MOR of the iroko wood, densified at 100 °C or 150 °C, after second thermo-mechanical modification cycle was twice as high as before the modification. No significant differences were observed between the modulus of elasticity (MOE) of oak wood before and after one modification cycle. Similar dependencies were noted in iroko wood. The thermo-mechanical modification performed over two cycles led to the highest increase, by about 56%, in MOE in oak wood densified at 150 °C. It was demonstrated that modification at 150 °C had a negative impact on iroko wood, which was manifested in the lower compression ratio of iroko at 150 °C than at 100 °C.

Keywords: European oak; Densification; Iroko; Tauari; Thermo-mechanical modification

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# INTRODUCTION

Wood can be subjected to various kinds of modification. One method of improving its physical and mechanical properties is through densification. Ülker *et al.* (2012) distinguished three densification methods: thermo-mechanical, thermo-hydro-mechanical, and viscoelastic thermally compression. The mechanical properties of wood are correlated with its physical properties. For this reason, increasing the wood's density by densification enhances its mechanical properties. Moreover, no chemicals are introduced into the wood's structure in the course of such modification, which is undoubtedly an advantage of this form of treatment.

Thermo-mechanical (TM) modification is a process involving compression by pressing heated wood. It is usually completed in a hydraulic press with heated plates (Kurowska *et al.* 2010; Laine *et al.* 2013; Bekhta *et al.* 2014a,b; Laine *et al.* 2016). Methods of continuous wood densification using rolls have also been developed (Gaff and Gašparík 2015). The technological process is usually divided into three stages. In addition to pressing, the wood is subjected to pre-treatment and final treatment. The purpose of pre-treatment is to plasticize the wood's structural components to facilitate the densification process (Fu *et al.* 2016). The final treatment aims to reduce post-densification internal strain to prevent deformations (Rautkari *et al.* 2013; Popescu *et al.* 2014; Zhan and Avramidis 2016). The TM modification is usually carried out in the temperature range 100 to 200 °C. The pressing pressure depends on many factors: kind of densification (surface,

volume), press type, wood species, wood moisture content, and sample thickness or assumed compression ratio (Rautkari *et al.* 2011; Ülker *et al.* 2012; Laskowska 2017). Therefore, the pressing pressure is in a very wide range from 1 MPa to 100 MPa. Densification is applied to both low- and high-density wood (Rautkari *et al.* 2010; Kutnar and Kamke 2012; Tu *et al.* 2014; Gašparík *et al.* 2016; Zhan and Avramidis 2016). Research has involved wood from different climatic zones. Tropical zone wood is characterized by a more complex anatomical and chemical structure than wood from the temperate zone. A typical feature of many wood species from the tropical zone is their irregular arrangement of fibres, which has a significant negative impact on the wood's mechanical properties. Moreover, tropical zone wood possesses more parenchyma (Richter and Dallwitz 2000; Wagenführ 2007), which may likewise significantly affect the course of TM modification.

The properties of densified wood are determined by a number of factors, among which the temperature of treatment is the most significant (Dilik and Hiziroglu 2012; Fang *et al.* 2012; Ülker *et al.* 2012; Laskowska 2017; Ülker and Hiziroglu 2017). Such dependence results from the fact that wood's structural components have different softening temperatures, which in turn largely depend on wood moisture content (Kutnar and Šernek 2007; Ülker *et al.* 2012). Another significant factor in the technological factors' group is the pressing pressure, which determines the wood's compression ratio, and consequently its density and the correlated mechanical properties (Gašparík *et al.* 2016). In the group of material-related factors, the wood's bending strength is principally affected by the anatomical structure, density, earlywood and latewood proportion, moisture content, wood's temperature, type and distribution of knots, and other defects (irregular fibres) or damage (Blomberg *et al.* 2005; Kretschmann 2010).

Several publications have researched the impact of technological parameters of thermo-mechanical modification, chiefly on the physical properties of wood. The most frequently described wood properties are set recovery, density profile, and surface properties (Kutnar *et al.* 2009; Candan *et al.* 2010; Rautkari *et al.* 2010; İmirzi *et al.* 2014; Laine *et al.* 2016). Among the mechanical properties of thermo-mechanically modified wood, the most frequently analysed is hardness (Fu *et al.* 2016; Gašparík *et al.* 2016; Laine *et al.* 2016). Data on the bending strength and the modulus of elasticity of thermo-mechanically modified work of such building elements as floors or beams, it is important to analyse these issues. Thermo-mechanical modification is dedicated to such materials.

Cyclic thermo-mechanical treatment (CTMT) is an innovative solution developed to improve the properties of wood to a higher degree than that achieved by 'standard' thermo-mechanical modification (Laskowska *et al.* 2018). Consequently, the main objective of this study was to determine the impact of CTMT on the modulus of rupture (MOR) and the modulus of elasticity (MOE) of wood species from the temperate climate zone (oak) and tropical zone (iroko, tauari). The wood species selected for research are widely used in manufacturing floors, furniture, and interior furnishings. The following issues were subject to analysis: significance and degree of impact of technological factors (densification temperature, number of treatment cycles) and material-related factors (wood species) on the thickness, density, MOR, and MOE of wood. Interrelations were determined between these factors and the tested wood properties.

## EXPERIMENTAL

### Materials

The wood samples used in the tests were made of European oak (*Quercus robur* L.), iroko (*Milicia exelsa* (Welw.) C.C. Berg), and tauari (*Couratari spp.*). Their dimensions were: 130 mm (longitudinal), 80 mm (tangential), and 8.50 mm (radial). Each variant of thermo-mechanical modification was tested on 20 samples. The European oak wood was obtained from central Poland, iroko wood from Nigeria, and tauari wood from Brazil. The surface of the wood samples was finished by planing. After the samples had been brought to an air-dry condition in normal climate (temperature 20 °C  $\pm$  2 °C, relative humidity 65%  $\pm$  5%), the wood moisture content was determined in accordance with ISO 13061-1 (2014). The moisture content of wood amounted to 7.92% ( $\pm$  0.68%). The wood density was determined using the stereometric method in accordance with the requirements of ISO 13061-2 (2014).

Latin Name	English Trade Name of Wood (and Code) According to EN-13556 (2003)	Origin	Structure of Wood	Wood Anatomical Features
Couratari spp.	Tauari (CIXX)	South America	Deciduous diffuse- porous	Axial parenchyma banded, apotracheal axial parenchyma diffuse-in-aggregates (in short, wavy lines), paratracheal axial parenchyma scanty
<i>Milicia excelsa</i> (Welw.) C.C. Berg	Iroko (MIXX)	Africa	Deciduous diffuse- porous	Irregular fibres arrangement, axial parenchyma banded, paratracheal axial parenchyma vasicentric, aliform, and confluent
Quercus robur L.	European oak (QCXE)	Europe	Deciduous ring-porous	Broad rays

## Table 1. Data on the Wood Species Tested

## Methods

#### *Cyclic thermo-mechanical treatment*

Oak, iroko, and tauari wood were subjected to CTMT. One treatment cycle consisted of three stages: i) heating the wood in a hydraulic press (platens dimension 650 mm × 650 mm, maximum manometric pressure 25 MPa) in laboratory conditions; ii) wood densification; and iii) cooling the wood samples in an unheated hydraulic press without exerting pressure. The thermo-mechanical modification was completed in one or in two cycles. Both the heating time and wood densification time amounted to 120 s, whereas the time of cooling the wood in a hydraulic press was 240 s. The treatment temperature amounted to 100 °C or 150 °C, and the unit pressure to 45 N/mm<sup>2</sup>. The cooling took place in a normal climate (temperature 20 °C  $\pm$  2 °C, relative humidity 65%  $\pm$  5%). After the treatment, the wood samples were conditioned in a normal climate for 7 days.

#### Measurement methods

The wood samples were prepared in accordance with ISO 3129 (2012). The compression ratio (CR) was calculated according to Eq. 1, where  $t_0$  is the original thickness (mm) and  $t_d$  is the thickness of wood after densification (mm):

$$CR(\%) = \frac{t_0 - t_d}{t_0}$$
 (1)

The wood's MOR and MOE tests were conducted in accordance with the methodology specified in the ISO 13061-3 (2014) and ISO 13061-4 (2014) standards, with minor alterations. They resulted from the size of the wood samples used for thermomechanical modification. The samples used for testing the MOR and MOE of wood were 130 mm long and 10 mm wide. The thickness of the control samples was 8.66 mm ( $\pm$  0.12 mm) and that of the modified samples corresponded to the thickness of samples after modification. In accordance with the requirements of the ISO 13061-3 (2014) and ISO 13061-4 (2014) standards, the support spacing depended on the thickness of samples and equalled twelve times the sample thickness. The MOR and MOE research was carried out using a computer program coupled with an Instron<sup>®</sup> testing machine, model 3369 (Instron<sup>®</sup>, Norwood, MA, USA). The wood properties were determined for 20 control samples and for each variant of thermo-mechanical modification of oak, iroko, and tauari.

#### Statistical analysis

Statistical analyses were performed using STATISTICA (version 12) software from StatSoft, Inc. (Tulsa, OK, USA). The statistical analysis of the results was performed at a significance level of 0.05.

## **RESULTS AND DISCUSSION**

A detailed analysis (based on analysis of variance (ANOVA), Fischer's F-test) of the significance and degree of impact of temperature and number of thermo-mechanical modification cycles on thickness, density, MOR, and MOE, depending on the species of wood modified, are presented in Table 2. The impact of modification temperature on the properties of densified wood was greatest in tauari wood, ranging from 39% to 57% depending on the wood property tested, whereas the smallest impact of temperature was observed in densified iroko wood. The modification temperature affected the thickness and density of densified iroko wood by 38% and 28%, respectively, but it had no significant impact on MOR or MOE. The number of thermo-mechanical modification cycles had the greatest impact on the mechanical properties of iroko wood, and the smallest on the tauari wood. The impact of the number of cycles on MOR and MOE of iroko wood amounted to 78% and 61%, respectively, and 6% and 11% for the MOR and MOE of tauari wood, respectively. For oak wood, the number of cycles was responsible for 33% and 72% changes, respectively, of MOR and MOE variation. The impact of the number of thermomechanical modification cycles on wood thickness and density was greatest in oak, 65% and 61%, respectively. The number of cycles had a comparable impact, ranging from 25% to 37%, on the thickness and density of iroko and tauari wood, respectively. A significant impact of the interaction between temperature and number of modification cycles on the thickness and density of oak and iroko wood (impact of 18 to 26%) was also observed.

Table 2.	. Statistical Evaluation	of the Factors	Influencing the	Parameters of	Oak,
Iroko, ar	nd Tauari Wood				

Wood	Factor	Properties					
Species		Thickness		Density			
-		Fisher's	Sig. Level	Influence	Fisher's	Sig. Level	Influence
		F-test	-	of Factors	F-test	-	of Factors
				(%)			(%)
		F	р	X	F	p	X
Oak	Temp. (1)	21.45	0.000161	9	17.93	0.000406	8
	Cycle (2)	158.95	0.000000	65	139.56	0.000000	61
	$(1) \times (2)$	42.81	0.000002	18	49.48	0.000001	22
	Error	-	-	8	-	-	9
			MOR			MOE	
		F	р	Х	F	р	Х
	1	19.202	0.000288	32	27.397	0.000040	13
	2	19.941	0.000237	33	156.729	0.000000	72
	1 × 2	0.949	0.341574	2	14.576	0.001077	7
	Error	-	-	33	-	-	9
Iroko			Thickness	_	Density		
		F	р	Х	F	р	Х
	1	93.00	0.000000	38	59.15	0.000000	28
	2	72.04	0.000000	30	78.39	0.000000	37
	1 x 2	57.53	0.000000	24	56.21	0.000000	26
	Error	-	-	8	-	-	9
			MOR			MOE	
		F	р	Х	F	р	Х
	1	0.076	0.785209	0	0.819	0.376337	2
	2	69.526	0.000000	78	32.817	0.000013	61
	1 × 2	0.107	0.746563	0	0.098	0.758072	0
	Error	-	-	22	-	-	37
Tauari	Tauari Thickness		1	Density			
		F	р	Х	F	р	Х
	1	29.09	0.000028	39	44.87	0.000002	49
	2	25.41	0.000063	34	23.03	0.000109	25
	1 × 2	0.01	0.934693	0	4.11	0.056172	4
	Error	-	-	27	-	-	22
		MOR			MOE		
		F	р	Х	F	р	Х
	1	36.617	0.000006	57	19.801	0.000246	42
	2	3.933	0.061247	6	5.149	0.034472	11
	1 × 2	3.152	0.091068	5	2.483	0.130753	5
	Error	-	-	31	-	-	42

The results of the research showed that oak wood was the most susceptible to densification among the species tested. After the densification process, the oak wood had the smallest thickness (Fig. 1a, c), *i.e.*, the highest compression ratio (CR). Regardless of the levels of factors tested, the CR of oak wood amounted to *ca.* 40%. Iroko wood was the least susceptible to densification, with a CR of *ca.* 30%. Iroko wood has an irregular fibre arrangement, a defect that enriches the wood's figure. In contrast, this characteristic of renders its densification more difficult. The areas of irregular fibres are less susceptible to densification than those with straight fibres. Moreover, unlike oak and tauari wood, iroko possesses expanded axial parenchyma (Richter and Dallwitz 2000), which also hinders densification. It is generally assumed that wood with a lower initial density is more influenced by densification than wood with a higher initial density (Gašparík *et al.* 2016).

Although it had lower density than oak and tauari, iroko wood was least susceptible to densification because of the other features mentioned. A higher modification temperature determined the thickness of iroko and tauari wood to a higher degree than that of oak wood. Moreover, it was demonstrated that a high modification temperature, at 150 °C, had a negative impact on iroko wood. This was manifested in the lower compression ratio of iroko wood at 150 °C than at 100 °C. The CR of the wood species tested was greater after the second cycle of thermo-mechanical modification than after the first cycle, and the differences were statistically significant (p < 0.05). After the second cycle, the CR of tauari, oak, and iroko wood was 5, 7, and 9 percentage points higher, respectively, than after the first cycle of modification.



Fig. 1. Thickness (a, c) and density (b, d) of thermo-mechanically modified wood species

The modification temperature had the greatest impact on the density of tauari wood and the smallest on the density of oak wood (Table 2). The density of tauari wood densified at 100 °C was 47% higher than that of non-densified tauari wood, whereas the tauari densified at 150 °C showed a 61% higher density than non-densified tauari wood (Fig. 1b). The density of oak wood densified at 100 °C and 150 °C was higher 62% and 64%, respectively, than that of non-densified oak wood. No significant differences were observed between the density of oak wood modified at 100 °C and 150 °C (p > 0.05). After the second modification cycle, the density of oak wood was 6% higher than after the first

cycle. A similar percentage of density increase was observed in tauari wood (Fig. 1d). However, the density of the tauari wood after the modification was greater. The lowest increase in density after the second cycle was observed in iroko (4%), and the differences were statistically significant (p < 0.05). After the first cycle, the tauari wood density amounted to 1036 kg/m<sup>3</sup> (± 70 kg/m<sup>3</sup>), whereas after the second cycle, the density was 1105 kg/m<sup>3</sup> (± 53 kg/m<sup>3</sup>). The differences were statistically significant (p < 0.05). Laine *et al.* (2016) densified pine wood (*Pinus sylvestris* L.) at 150 °C for 1 h. At 39% densification ratio, the authors obtained a density of 757 kg/m<sup>3</sup> ± 2.6 kg/m<sup>3</sup> for the pine wood.



→ Non-densified → Densified

Fig. 2. MOR (a) and MOE (b) of the tested wood species

The mean MOR value for non-densified oak wood was 123 MPa (± 22 MPa), whereas the MOR for non-densified iroko and tauari wood originating from the tropical zone amounted to 74 MPa (± 26 MPa) and 155 MPa (± 28 MPa), respectively (Fig. 2a). The oak and iroko MOR values were close to the data found in literature. Wagenführ (2007) stated that the MOR of oak wood ranges from 74 MPa to 105 MPa, and the MOR of iroko ranges from 70 MPa to 158 MPa. According to data gathered by CIRAD (2012), the mean MOR of tauari wood at a density of 620 kg/m<sup>3</sup> amounts to 87 MPa ( $\pm$  9 MPa). The higher MOR values for tauari resulted from the higher density of the wood used in the tests (694  $kg/m^3 \pm 41 kg/m^3$ ). The density of non-densified iroko wood was lower than that of oak and tauari 13% and 19%, respectively. Moreover, iroko wood had an irregular arrangement of fibres. This natural characteristic of the species made its wood more difficult to densify. The MOR of densified oak and tauari wood were higher 64% and 84%, respectively, than the MOR of these wood species before the densification. The MOR of densified iroko wood was 78% higher than before the densification. Changes to a lesser degree were observed in the wood's MOE (Fig. 2b). The MOE of oak and iroko wood was 20% higher than the MOE of these species before the densification. As a result of thermo-mechanical modification, the MOE of tauari wood was 30% higher than that of non-densified tauari wood.

The greatest changes in MOR affected by temperature were observed in tauari wood. The MOR of tauari wood modified at 100 °C exceeded the MOR of non-densified tauari wood by 65%. After treatment at 150 °C, the MOR of the tauari wood was twice as high as the MOR of the non-densified tauari wood. The MOR of the oak wood modified at 100 °C and 150 °C was higher than its MOR before densification 52% and 77%,

respectively. No significant differences were noted between the MOR of iroko wood modified at 100 °C and 150 °C (Fig. 3a). Similar dependences were observed by Ülker *et al.* (2012) in densifying Scots pine wood (*Pinus sylvestris* L.). The authors pointed out that the densification process increased the bending strength, but an increase in the temperature level (from 140 °C to 160 °C) caused a decrease in bending strength.



Fig. 3. MOR (a, c) and MOE (b, d) of thermo-mechanically modified wood species

The greatest changes in the MOR values that were dependent on the number of thermo-mechanical modification cycles were noted in iroko wood (Fig. 3c). The MOR of iroko wood after one treatment cycle increased 45%; after two cycles it was twice as high as before the modification. This meant a 36% difference between its MOR after one and after two treatment cycles. The MOR of tauari wood after one and two treatment cycles increased 78% and 90% from its baseline, respectively. The MOR of oak wood after one treatment cycle was higher 51%, and after two cycles 77% in comparison to its value before the modification. Ülker *et al.* (2012), after densifying pine (*Pinus sylvestris* L.) wood at 120 °C, noted an increase in density of 83% and that the highest increase in bending strength was 42% (an increase from 69.13 N/mm<sup>2</sup> to 98.55 N/mm<sup>2</sup>). The MOE of pine wood modified at 120 °C for a higher MOR.

As a result of higher modification temperature, the change in the MOE was greater in tauari wood than in oak or iroko wood (Fig. 3b). The MOE of tauari wood modified at 100 °C and 150 °C increased 20% and 46%, respectively, with regard to the wood's MOE before the densification. Smaller differences were noted in oak wood. The MOE of oak wood modified at 100 °C and 150 °C was, respectively, 11% and 29% greater than before the densification. The MOE of iroko wood densified at 100 °C and 150 °C was at a similar level (MOE increased *ca.* 20%).

Performing the second thermo-mechanical modification cycle resulted in a considerable improvement in the MOE of the tested wood species (Fig. 3d). The MOE values of non-densified oak and tauari wood (Table 4) were close to literature data. According to literature, the MOE of oak wood amounts to 10000 MPa to 13200 MPa, and the MOE of tauari wood is 14500 MPa (Wagenführ 2007; CIRAD 2012). The MOE of the tested iroko wood was lower than 9400 MPa to 13000 MPa, which is the range established by previous studies (Wagenführ 2007). This resulted from its irregular fibres arrangement, a typical defect of iroko wood, which significantly contributed to the lower MOE values. After one modification cycle, the MOE of oak and iroko wood were nearly identical to the MOE of non-densified wood. However, after two thermo-mechanical modification cycles, the MOE of oak and iroko wood after one modification cycle was 26% higher, and after two modification cycles it was 40% higher than the MOE measured prior to densification (Fig. 3d).

Wood	Temperature	Cycle	Thickness	Density	MOR	MOE
Species/Variant	(°C)	-	(mm)	(kg/m <sup>3</sup> )	(MPa)	(MPa)
Oak Non-densified	-	-	8.67 (0.35)	651 (35)	123 (22)	11850 (920)
Oak Densified	100	1	5.93 (0.09)	1015 (18)	168 (10)	11410 (480)
		2	5.05 (0.08)	1079 (32)	206 (10)	14890 (580)
	150	1	5.36 (0.20)	1028 (27)	205 (21)	11980 (960)
		2	4.96 (0.09)	1116 (24)	230 (23)	18510 (910)
Iroko Non-densified	-	-	8.69 (0.32)	564 (29)	74 (26)	6840 (900)
Iroko Densified	100	1	5.72 (0.07)	769 (15)	108 (12)	7030 (860)
		2	5.59 (0.14)	784 (6)	148 (15)	9340 (320)
	150	1	5.92 (0.11)	732 (33)	121 (14)	7400 (860)
		2	5.83 (0.43)	758 (49)	132 (12)	8760 (670)
Tauari Non-densified	-	-	8.62 (0.43)	694 (41)	155 (28)	13830 (920)
Tauari Densified	100	1	6.01 (0.10)	974 (26)	238 (24)	15000 (950)
		2	5.60 (0.21)	1071 (39)	274 (18)	18140 (1280)
	150	1	5.57 (0.19)	1098 (27)	314 (23)	19920 (1270)
		2	5.17 (0.26)	1138 (44)	316 (29)	20490 (2900)

Table 3. Mean Values of the	e Tested Parameters
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Values in parenthesis are standard deviation

Detailed data on the tested wood properties (for each variant of CTMT) are presented in Table 4. The smallest thickness (4.96 mm  $\pm$  0.09 mm) was observed in oak wood after two modification cycles, densified at a temperature of 150 °C. The oak wood densified in these conditions was characterized by a *ca*. 43% compression ratio and a 70% increase in density over non-densified oak wood, which was the greatest change in density. The highest density (1138 kg/m<sup>3</sup>  $\pm$  44 kg/m<sup>3</sup>) was noted for tauari wood after two modification cycles, densified at 150 °C. The tauari wood modified in this manner had the greatest MOR (316 MPa  $\pm$  29 MPa) and MOE (20490 MPa  $\pm$  2900 MPa). The thermomechanical modification performed in two cycles doubled the MOR of iroko wood, which was the greatest increase in MOR. This was regarding the iroko wood modified at temperatures 100 °C and 150 °C. The second treatment cycle neutralized the negative impact of the irregular fibres on the properties of thermo-mechanically modified iroko wood. A similar increase in the MOR value was observed in the tauari wood modified at 150 °C. The biggest differences in the MOE after densification were noted in oak wood. The MOE of oak wood densified at 150 °C after two modification cycles was 56% greater than the MOE of non-densified oak wood.

# CONCLUSIONS

- 1. The thermo-mechanical modification had significant impact on the thickness, density, MOR, and MOE of oak, iroko, and tauari wood.
- 2. The impact of modification temperature was greatest on the properties of densified tauari wood and smallest on the properties of densified iroko wood.
- 3. The second cycle of thermo-mechanical modification had a significant impact on the MOR and MOE of oak, iroko, and tauari wood densified at 100 °C and the MOE of oak and iroko wood densified at 150 °C.
- 4. A negative impact of high modification temperature, at 150 °C, on iroko wood was demonstrated. It was manifested in the lower compression ratio of iroko wood at 150 °C than at 100 °C.
- 5. The thermo-mechanical modification performed over two cycles doubled the MOR of iroko wood (densified at 100 °C or 150 °C) and led to the highest increase, by about 56%, in MOE in oak wood (densified at 150 °C).

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