

CHEMICAL AND MECHANICAL CHARACTERIZATION OF THERMALLY MODIFIED *GMELINA ARBOREA* WOOD

KEMIJSKA IN MEHANSKA KARAKTERIZACIJA TERMIČNO MODIFICIRANEGA LESA VRSTE *GMELINA ARBOREA*

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Abstract / Izvleček

Abstract: *Gmelina arborea* (Roxb. ex. Sm.) wood samples were thermally modified at 180 °C, 200 °C and 220 °C for 3 h, by employing a process similar to ThermoWood®. The resulting effects on the basic chemical composition and mechanical properties were determined. The results were analyzed statistically with ANOVA, and Least Square Deviation was used to compare means. Generally, after the thermal modification (TM) process, the cellulose, hemicelluloses and extractives content decreased significantly. By contrast, lignin proportions increased significantly. Untreated wood and samples modified at 180 °C indicated comparable modulus of elasticity (MOE), modulus of rupture (MOR), degree of integrity (I), fine fraction (F) and resistance to impact milling (RIM). Noteworthy reductions however occurred at 200 °C and 220 °C. Significant increases in Brinell hardness (BH) took place at 180 °C, recording a high decrease at 220 °C. *Gmelina arborea* could be modified suitably at 180 °C for structural and other purposes. To take advantage of other improved properties, modification at 200 °C could be employed for non-structural uses.

Keywords: High-Energy Multiple Impact (HEMI), Resistance to Impact Milling (RIM), Thermal modification, Static Bending

Izvleček: Vzorce lesa vrste *Gmelina arborea* (Roxb. ex. Sm.) smo 3 ure termično modificirali pri 180 °C, 200 °C in 220 °C po postopku, ki je soroden ThermoWood® procesu. V nadaljevanju smo ocenili vpliv modifikacije na kemijsko sestavo in mehanske lastnosti. Rezultate smo testirali z analizo variance ANOVA, za primerjavo povprečij pa smo uporabili metodo najmanjših kvadratov. Na splošno se je delež celuloze, hemiceluloz in ekstraktivnih snovi v termično modificiranem lesu znatno zmanjšal. Nasprotno se je delež lignina v modificiranem lesu statistično značilno povečal. Neobdelani les, modificiran pri 180 °C, je imel primerljivo upogibno trdnost (MOR) in modul elastičnosti (MOE), stopnjo integritete (I), delež fine frakcije (F) in odpornost proti udarnemu mletju (RIM). Pri modifikaciji pri temperaturah 200 °C in 220 °C je prišlo do znatnega poslabšanja omenjenih lastnosti. Trdota po Brinellu (BH) se je znatno povečala pri 180 °C, pri 220 °C pa se je močno zmanjšala. Les vrste *G. arborea* bi bilo za konstrukcijske namene primerno modificirati pri 180 °C. Da bi izkoristili druge izboljšane lastnosti, bi za les za nekonstrukcijske namene lahko uporabili modifikacijo pri 200 °C.

Ključne besede: večkratni visokoenergijski udarci (HEMI), odpornost proti udarnemu mletju (RIM), termična modifikacija, statični upogib

1 INTRODUCTION

1 UVOD

Wood is a very important environmentally friendly, renewable and accessible material, which is widely preferred around the globe. However, the sustainable supply of tropical timber on the world market is increasingly threatened (Boonstra, 2008). For instance, deforestation in Ghana has assumed

alarming proportions, leading to massive reductions in the nation's primary forest cover (FAO, 2006). In its 2016 report, the Forestry Commission (FC) of Ghana indicated that about 80% of the country's forest resources under state management had been lost to illegal logging activities facilitated mainly by farming, the chainsaw operations which supply most of the local lumber demand, and mining (FC, 2016a;

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Hansen & Treue, 2008). Asiedu (2019) also recorded a 60% increase in loss of primary forest cover in 2018 compared to 2017 in Ghana, with clearing for cocoa farming being a leading cause of deforestation, while mining remains the biggest threat.

To reduce over-exploitation of primary forests and ensure sustainable supply of timber on the local and export markets, Ghana has embarked on the establishment of plantations, covering about 190,500 ha as of 2015. The FC further plans to cultivate 100,000 ha of plantations in the years 2016 – 2040. Among the many species to be cultivated is *Gmelina arborea*, which is an exotic species (FC, 2016b), indigenous to the Indo-Burma region of Southeast Asia (Nwoboshi, 1994). Over 5,000 ha of *Gmelina arborea* plantations have been established in Ghana, 2,000 ha of which was cultivated by the Subri Industrial Plantation Limited (SIPL), near Sekondi-Takoradi in the Western Region (Nwoboshi, 1994). *Gmelina arborea* has oven-dry density ranges of 390 – 599 kg/m³ and good machining properties (Mitchual et al., 2018). It is rated as naturally non-durable and thus requires preservative treatment to prolong its service life (Owoyemi et al., 2011). Kasmudjo (1990) indicated that suitable uses of *Gmelina arborea* wood are fuelwood, pulp and paper, plywood, furniture, construction and matches. The researcher further suggested harvesting cycles of five years for fuel wood, 15 years for plywood and boards, 20 years for construction and eight years for uses such as boxes, toothpicks and matches.

To complement the efforts of the FC there is the need to research the properties of these plantation species, in order to establish their suitability for furniture and other such applications. When thermally modified, *Gmelina arborea* could be used for applications exposed to the weather and humidity variations above ground, including cladding, garden furniture, windows and saunas, but could also be used for interior applications such as stairs, decorative panels, flooring, kitchen furniture, etc. (Sandberg & Kutnar, 2016). The current study aimed at redirecting the attention of wood industry stakeholders to plantation species like *Gmelina arborea* and encouraging its use.

Thermal modification (TM) of wood is one important method for improving wood properties, including dimensional stability, natural durability and hygroscopicity. Wood retains its environmen-

tally friendly nature after this process. TM of wood is less costly compared to wood preservation using biocidal treatment (Esteves et al., 2014). However, TM may cause a reduction in mechanical strength. The extent of the effect of TM on the properties of wood depends on species and process conditions (Esteves & Pereira, 2009). Differences in wood species' response to TM treatments are due to variations in chemical, anatomical and physical properties (Ranin, 2004). Výbohová et al. (2018) analysed the effect of TM intensity at the temperatures of 160 °C, 180 °C and 200 °C under atmospheric pressure in the presence of air for durations of 3, 6, 9, and 12 h on ash wood. The researchers reported that the extractives content initially increased up to 200 °C and 3 h, which decreased with extended treatment duration. Hemicellulose monosaccharide (D-xylose) degraded under all treatment conditions, which resulted in a decrease in holo-cellulose content. Lignin content also increased at temperatures of 180 °C and 200 °C. Bekhta and Niemz (2003) indicated that the MOR of spruce (*Picea abies* (L.) Karst.) decreased by 44% to 50% as the modification temperature was raised from 100 °C to 200 °C, while temperature had no effect on MOE. Korkut et al. (2008) reported that the surface hardness, MOE and MOR in the radial direction of Scots pine (*Pinus sylvestris* L.) decreased by 27%, 32% and 33%, respectively at 180 °C for 10 h.

To reduce the focus on traditional primary timber species in order to curb deforestation in Ghana, it is necessary to promote lesser used species (LUS) like *Gmelina arborea* on both local and export markets. Successful adoption of *Gmelina arborea* for utilization will depend on its properties. This study therefore aimed to investigate the impact of the TM process applied to enhance the properties of *Gmelina arborea* wood from Ghana with regard to its basic chemical and mechanical properties.

2 MATERIALS AND METHODS

2 MATERIAL IN METODE

2.1 MATERIALS

2.1 MATERIAL

2.1.1 Source of Material and Preparation

2.1.1 Izvor materiala in priprava

Four *Gmelina arborea* trees with diameter at breast height (DBH, 1.3 m above ground) of 35 –

55 cm were obtained from a 40-year old plantation of the Centre for Scientific and Industrial Research – Forestry Research Institute of Ghana’s (CSIR – FORIG) research plot at Abofour, Offinso District in the Ashanti Region of Ghana. Abofour is located within the moist Semi-Deciduous Forest Zone with average annual rainfall of 1,400 mm. It lies 7°8’0” N and 1°45’0” W and it is about 60 km from the Ashanti regional capital, Kumasi.

Trees were bucked into 2.5 m length bolts with a chainsaw, which were further processed using a portable Wood-Mizer sawmill (LT 30) into boards of 25 mm thickness at the CSIR – FORIG wood workshop at Fumesua, Kumasi. The boards were air-dried for at least 12 months until a moisture content (MC) below 20% was achieved. Boards were randomly selected from within 15 cm radius of the pith to assure the use of heartwood. These boards were further processed into test slats of dimensions 20 x 50 x 650 mm³ (radial (r) × tangential (t) × longitudinal (l)) for the TM process. The slats were sorted according to weight and those in the range of 300 to 400 g were used for the study. This pre-sorting was necessary for the homogenization of the lot and to minimize the effect of the initial density on the results.

2.2 METHODS

2.2 METODE

2.2.1 Thermal Modification

2.2.1 Termična modifikacija

The open system TM similar to the ThermoWood® process (Mayes & Oksanen, 2002) was employed, using a 65 L capacity laboratory scale reactor. Firstly, the temperature in the vessel was raised at 12 °C/h to 100 °C and then 4 °C/h to 130 °C to allow high temperature drying of slats to nearly 0% MC. Secondly, the temperature was again increased at 12 °C/h until reaching the peak temperatures of 180, 200 and 220 °C. Each peak temperature was held for 3 h. Finally, the temperature was decreased at 20 °C/h until reaching 65 °C, at which the vessel was opened and the slats removed. Eventually, the thermally modified wood samples at the three peak temperatures resulted in three treatments with the untreated wood as control. The wood samples were assessed for selected chemical and mechanical properties.

2.2.2 Mass loss

2.2.2 Izguba mase

The mass loss of each slat after the thermal modification process was calculated as described by Metsä-Kortelainen et al. (2006). Each air-dried slat was weighed before and after the thermal modification process. Moisture content was then determined for the slats before and after the thermal modification process, in order to determine the mass loss.

Mass loss% (ML%), was thus calculated as in Equation 1.

$$ML = \frac{(m_1 - m_2) \times 100}{m_1} [\%] \quad (1)$$

Where m_1 is the dry mass of the sample before the thermal modification, in g; m_2 is the dry mass of the sample after the thermal modification, in g.

Dry mass (g), m_{dry} , was calculated as follows (Equation 2).

$$m_{dry} = \frac{100 \times m_u}{u + 100} [g] \quad (2)$$

where m_u is the mass of the sample at moisture content $u\%$, in g.

2.2.3 Chemical Analysis

2.2.3 Kemijska analiza

Slats were randomly selected per treatment for the analysis. Two samples of the dimensions 20 × 20 × 20 mm³ (r × t × l) were cut from both ends of each slat to make up six parts. The parts of the slats were reduced manually and ground in a cutting mill (Retsch, model SM 2000, Haan, Germany) with a sieve of 1 mm diameter, resulting in a compound sample per treatment. The powder was screened in a vibratory sieve shaker (Retsch, model KS 1000, Haan, Germany), operating at 200 rotations min⁻¹ for four minutes. The chemical analysis was done on the fraction remaining on the 40-mesh sieve. All chemical analyses were carried out in triplicate for each TM treatment.

The extractives content was determined in accordance with the hot water solubility method, de-

scribed in the standard T 207 cm-99 (TAPPI, 1999b). The insoluble lignin was determined using 72% sulfuric acid, following the method used by Sluiter et al. (2008). Holocellulose was prepared using a solution of sodium chlorite and acetic acid according to the method of Wise et al. (1946). The filtered fraction from the holocellulose analysis was used in the α -cellulose analysis, which was carried out with a solution of sodium hydroxide (17.5%), according to the standard T-203 cm-99 method (TAPPI, 1999a). The amount of hemicelluloses was calculated by difference [holocellulose - α -cellulose].

2.2.4 Static Bending

2.2.4 Statični upogib

A three-point bending test based on the DIN 52186 (1978) test norm was performed on a universal testing machine (Zwick Roell Z010, Zwick, Ulm, Germany) using the central loading method and 30 samples per treatment, measuring $10 \times 10 \times 180 \text{ mm}^3$ ($r \times t \times l$). For each TM treatment, three slats were used with each providing 10 samples. All the samples were conditioned at 20 °C and 65% relative humidity for seven days until constant weight. This ensured that the moisture content and temperature, which affect the strength of wood, were maintained to enhance comparability of the results. The loading heads moved at constant speeds of 3 mm/min for untreated wood, 3 mm/min, 2 mm/min and 1 mm/min for samples thermally modified at 180 °C, 200 °C and 220 °C, respectively. Loading head speed was varied for each modification to be able to cause the failure of the samples within 90 ± 30 s. The test samples were supported at the ends by rotating supports with the diameter of 15 mm. The distance between the supports was 160 mm. At the point of failure the modulus of elasticity (MOE) and modulus of rupture (MOR) generated by the universal testing machine and displayed on a monitor were recorded. MOE and MOR were determined according to Equations 3 and 4, respectively:

$$MOE = \frac{l3}{4 * b * h^3} * \frac{\Delta F}{\Delta f} \quad [\text{N/mm}^2] \quad (3)$$

Where ΔF is the force difference in N (Newton) in the elastic deformation range of the specimen; Δf is the deflection, corresponding to the force differ-

ence ΔF , in the middle of the specimen in mm; l is distance between the supports (span) in mm; b is width of the sample in mm; and h is height of the sample in mm.

$$MOR = \frac{1.5 * P * l}{b * h^2} \quad [\text{N/mm}^2] \quad (4)$$

Where P is the breaking (maximum) load in N; l and h are the span and height of the samples, respectively.

2.2.5 High Energy Multiple Impact (HEMI-test)

2.2.5 Visokoenergijski večkratni udar (HEMI-test)

The HEMI-test was carried out according to the procedure presented by Brischke (2017). For each treatment, the test was replicated five times. Each replicate was made up of 20 oven-dry samples of $10 \times 10 \times 10 \text{ mm}^3$ ($r \times t \times l$). In all, a total of 100 samples from four slats were assessed for each TM treatment. Each of the 20 samples was placed into a bowl of 140 mm inner diameter of a heavy impact ball mill (Herzog HSM 100, Osnabrück, Germany), together with one steel ball of 35 mm diameter, three steel balls of 12 mm diameter, and three steel balls of 6 mm diameter. The bowl was shaken for 60 s with a rotary frequency of 23.3 s^{-1} and a stroke of 12 mm. The fragments of the 20 samples were fractionated on a slit sieve with a slit width of 1 mm using an orbital shaker at an amplitude of 25 mm and a rotary frequency of 200 min^{-1} for 1 min. The following values were calculated using Equation 5, Equation 6, and Equation 7:

$$I = \left(\frac{m_{20}}{m_{all}} \right) \times 100 \quad [\%] \quad (5)$$

Where the degree of integrity (I) is the ratio of the mass of the 20 biggest fragments (m_{20}) to the mass of all fractions (m_{all}) after the crushing process.

$$F = \left(\frac{m_{<1mm}}{m_{all}} \right) \times 100 \quad [\%] \quad (6)$$

Where the fine fraction (F) is the ratio of the mass that is sieved and has a diameter of less than 1 mm ($m_{<1mm}$) to the mass of all fractions (m_{all}).

$$RIM = \frac{(I - 3 * F + 300)}{4} \quad [\%] \quad (7)$$

Where RIM is the resistance to impact milling and represents the value of the measure for the structural integrity of the material.

2.2.6 Brinell hardness

2.2.6 Trdota po Brinellu

The Brinell hardness (static hardness) perpendicular to the grain on the radial face was measured according to DIN EN 1534 (2011) with a universal testing machine (Zwick Roell Z010, Zwick, Ulm, Germany). Ten samples obtained from five slats (i.e. two samples per slat) were used per treatment. The samples were conditioned at 20 °C/ 65% RH for 14 days until constant weight. A maximum force of 500 N was exerted using a steel ball with a diameter of 10 mm applied for 25 seconds on the samples with dimensions of 20 × 50 × 200 mm³ (r × t × l). The diameters of the residual impressions at three points on a face of each sample, with any two points being at least 50 mm apart, were automatically determined by the testing machine. The Brinell hardness was then calculated according to Equation 8:

$$BH = 2.F / \pi . D \left[D - \sqrt{D^2 - d^2} \right] \quad [N/mm^2] \quad (8)$$

Where BH is the Brinell hardness (N/mm²), F is the maximum force used (N), D is the diameter of the steel ball (mm) and d is the diameter of the imprint on the sample (mm).

2.2.7 Data Analysis

2.2.7 Obdelava podatkov

Descriptive statistics comprising means with standard deviations were presented for each treatment and test. A comparison of the results from the treatments was made using Analysis of Variance (ANOVA). The Least Square Deviation test was used to compare means at $\alpha = 0.05$, when ANOVA revealed significant differences. The Statistical Package for Social Sciences (IBM Statistics) version 26 was used for the analyses.

3 RESULTS AND DISCUSSIONS

3 REZULTATI IN DISKUSIJA

3.1 MASS LOSS

3.1 IZGUBA MASE

Table 1 presents the mass loss, proportions of cellulose, hemicelluloses, lignin and extractives in the thermally-modified *Gmelina arborea* wood. Most properties of thermally modified wood depend to a large extent on the mass loss (Bal, 2013). This indicates that high mass loss results in more

Table 1. Mass loss, proportions of cellulose, lignin, hemicelluloses and extractives per treatment.

Preglednica 1. Rezultati izgube mase in deleži celuloze, lignina, hemiceluloz in ekstraktivov glede na uporabljen postopek termične modifikacije

| Treatment | Mass Loss [%] | α -Cellulose [%] | Hemicelluloses [%] | Lignin [%] | Extractives [%] |
|-----------|------------------------------|-------------------------------|------------------------------|-------------------------------|-----------------------------|
| Untreated | 0.00 (0.00) | 55.93 ^a (0.98) | 19.16 ^a (1.13) | 34.86 ^a (1.09) | 7.87 ^a (0.20) |
| 180 °C | 5.44 ^a (0.86) | 51.50 ^b (0.55) | 18.08 ^a (0.43) | 39.62 ^b (0.98) | 6.35 ^b (0.34) |
| 200 °C | 10.08 ^b (0.95) | 53.20 ^{ab} (0.14) | 10.56 ^b (0.88) | 40.50 ^{bc} (0.64) | 9.11 ^a (0.23) |
| 220 °C | 15.13 ^c (2.17) | 52.45 ^b (0.52) | 6.63 ^c (0.23) | 44.16 ^c (0.62) | 6.35 ^b (0.40) |

Means followed by the same letter are not significantly different at $\alpha = 0.05$
Standard error values are shown in parentheses

pronounced changes in wood properties, including chemical and mechanical properties (Militz, 2002; Hill, 2006; Esteves & Pereira, 2009). Mass loss increased with the modification temperature, from 5.44% at 180 °C to 15.17% at 220 °C. Esteves and Pereira (2007) reported that mass loss is primarily due to thermal degradation of hemicelluloses and volatilization of extractives. This is evidenced in Table 1, where the hemicelluloses and extractives contents decreased from 19.16% and 7.87% in untreated wood to 6.63% and 6.35% at 220 °C, respectively. The influence of the reduced hemicelluloses content on mass loss is more pronounced ($R^2 = 0.9197$) than that of the extractives content ($R^2 = 0.0369$) (Fig. 1).

3.2 CELLULOSE AND HEMICELULOSES CONTENT

3.2 VSEBNOST CELULOZE IN HEMICELULOZ

Variations in the cellulose concentrations at 180 °C and 220 °C were not significantly different, with both differing significantly from untreated wood. Yildiz et al. (2006) and Esteves et al. (2008) noted that cellulose is less affected by TM in comparison to hemicelluloses in an atmosphere without oxygen. Degradation of amorphous cellulose is principally what takes place, making the resulting cellulose more crystalline and causing a reduction of cellulose content in TM woods (Boonstra & Tjeerdsma, 2006). The hemicelluloses content decreased significantly from 19.16% when untreated

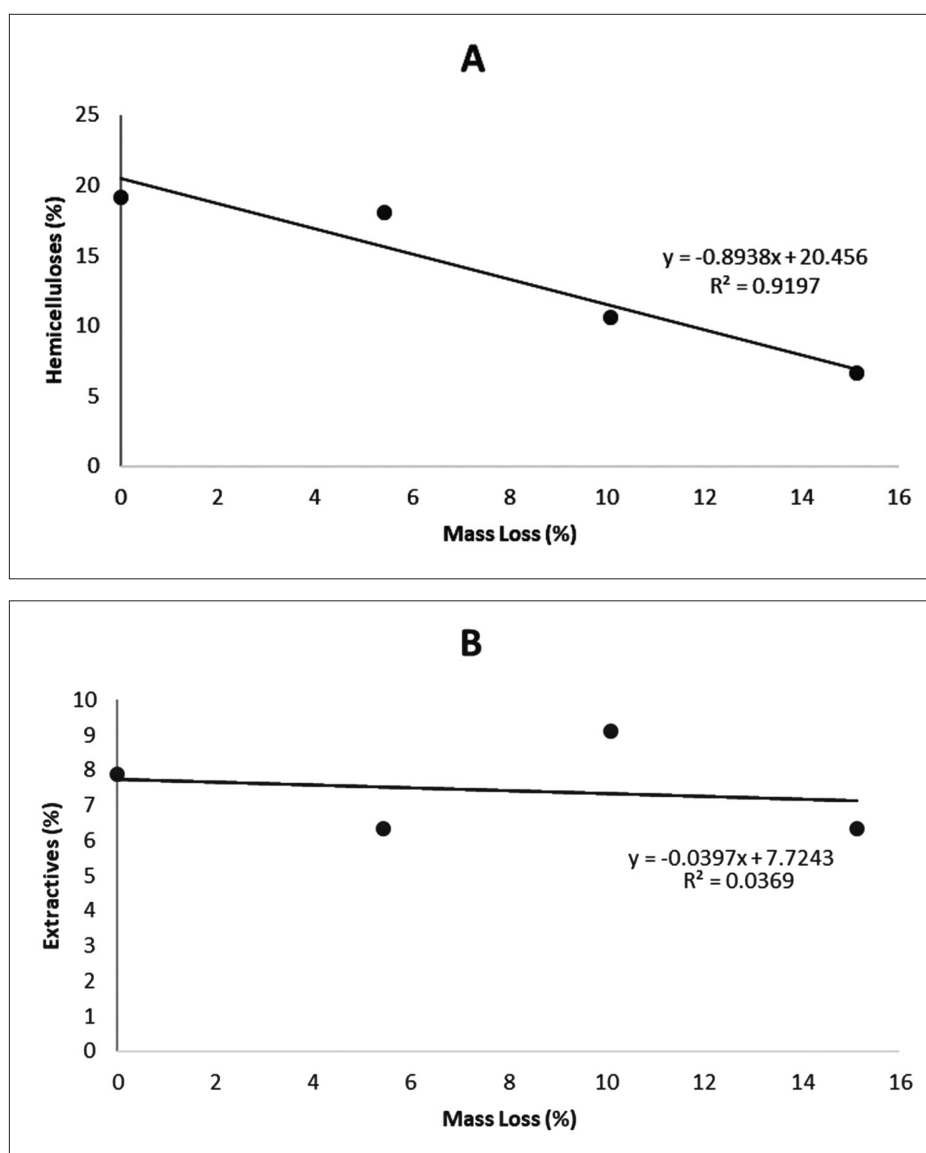


Figure 1. Relationship between mass loss and hemicelluloses (A) and extractives (B) contents [%].

Slika 1. Zveza med izgubo mase in vsebnostjo hemiceluloz (A) in ekstraktivnih snovi (B) [%].

to 6.63% at 220 °C modification temperature (Table 1). According to Sivonen et al. (2002) and Nuoponen et al. (2004), hemicelluloses are the first wood structural component to be affected during TM. They pointed out that deacetylation and the released acetic acid act as a depolymerization catalyst that further facilitates polysaccharide degradation, in line with the observations made in this study. The hemicelluloses content decreased with increased modification temperature, showing the highest reduction of 65.40% at 220 °C. On the other hand, cellulose decreased by a maximum of 8.60% at 180 °C. As such, cellulose is less degraded than hemicelluloses when wood is thermally modified.

3.3 LIGNIN CONTENT

3.3 DELEŽ LIGNINA

Table 1 shows that the lignin content increased significantly and consistently from 34.86% for untreated wood to 44.16% for a modification temperature of 220 °C. Similar results were obtained by Zaman et al., (2000) in their study of Scots pine (*Pinus sylvestris* L.) and birch (*Betula pendula* Roth Tent. fl. Germ.) thermally modified at 205 °C and 230 °C with respective holding times of 4 and 8 hours. They recorded increased lignin content from 24.5% to 38.7% and from 21.8% to 35.8%, respectively. Several researchers have suggested that an increase in lignin content after TM could be due to polycondensation reactions of lignin with other cell wall components (Tjeerdsma & Militz, 2005; Boonstra & Tjeerdsma, 2006). This results in cross-linking of lignin, leading to an increase in lignin content. Other reports have also indicated that lignin degrades at the beginning of the modification process, although the rate of degradation is slower than that seen with polysaccharide (hemicellulose) degradation (Windeisen et al., 2007). Therefore, the higher decrease in polysaccharide content gives the apparent observed increase in lignin content.

3.4 EXTRACTIVES CONTENT

3.4 DELEŽ EKSTRAKTIVOV

Generally the extractives content (EC) was significantly reduced after TM. It decreased from the initial content of 7.87% for untreated wood to 6.35% for modification temperatures of 180 °C and 220 °C (Table 1). A spike in EC of 9.11% was, however, recorded at a modification temperature

of 200 °C. Similarly, Esteves et al. (2008) reported a substantial increase in EC with increased modification temperature followed by a decrease with a further increase in temperature. Esteves and Pereira (2009) explained that although EC generally decreases with increased modification temperatures, new extractable compounds released at certain temperatures from polysaccharide degradation, such as water and ethanol, may cause it to increase. Esteves et al. (2008) indicated that the composition of the extractives changed as the original extractives disappeared and new compounds were formed in their place. In the present study, part of the inherent extractives may have been removed at 180 °C resulting in the observed decrease in EC. In contrast, the increased EC at 200 °C could have been contributed by degradation products of polysaccharides or hemicelluloses at that temperature. A further increase in temperature to 220 °C resulted in increased removal of extractable materials leading to the decreased EC and the significant mass loss observed. Consequently, the composition of the extractives may also vary from the original.

3.5 MODULUS OF ELASTICITY IN STATIC BENDING

3.5 MODUL ELASTIČNOSTI IZ STATIČNEGA UPOGIBA

Table 2 presents results of mechanical tests of thermally modified *Gmelina arborea* wood. Generally, a decreasing trend was observed in the Modulus of Elasticity (MOE) of the thermally modified wood with an increase in temperature. At 220 °C a significant reduction of 25.71% in MOE was observed compared to the untreated wood. Despite the marginal increase of 6.89% at 180 °C and a decrease of 3.50% at 200 °C compared to the untreated wood's MOE, the differences were not statistically significant. The initial marginal increase or relatively stable MOE at 180 °C (mass loss of 5.44% (Table 1)) was mainly due to crystallization of cellulose and condensation of lignin resulting from cross-linking reactions with furfurals produced from the thermal degradation of hemicelluloses (Bal & Bektas, 2013; Wang et al., 2018). However, it is noteworthy that comparing the MOE's for the various modification temperatures shows that there were significant reductions with an increase in temperature (Table 2). A similar observation has been made by other researchers. Esteves and Pereira (2007) and Inoue

et al. (1993), for example, observed an increase in MOE at about 4.0% mass loss, which decreased subsequently at higher mass losses or modification temperatures. Xu et al. (2019) reported an initial increase in MOE from 9230 N/mm² in the control samples to 10840 N/mm² of TM white oak (*Quercus alba* L.) at 160 °C which decreased to 7640 N/mm² at TM temperature of 200 °C and 9 h holding times. Hidayat et al., (2016) also obtained significant reductions in the MOE for thermally modified wood of *Cylicodiscus gabunensis* (Harms).

3.6 MODULUS OF RUPTURE IN STATIC BENDING 3.6 TRDNOST PRI STATIČNEM UPOGIBU

Modulus of rupture (MOR) is one of the mechanical properties most affected by wood TM, and it decreases with increasing modification temperature (Esteves & Pereira, 2009). The MOR of *Gmelina arborea* wood was significantly reduced after TM, from 85.85 N/mm² when untreated to 38.76 N/mm² at 220 °C (Table 2). Decreased MOR could be caused by degradation of wood structural components, specifically cellulose and hemicelluloses. The hemicelluloses content gets degraded much more than cellulose (Table 1). When amorphous hemicelluloses are degraded, the remaining and dominant cellulose becomes more crystalline (Boonstra &

Tjeerdsma, 2006). Wood then becomes increasingly brittle with increased modification temperature, lowering its MOR. One of the functions of hemicelluloses is to absorb stress transferred in wood by reinforcing cellulose microfibrils in the wood cell wall. Removal of hemicelluloses thus leads to the distribution of stress over less cell wall material which is brittle, resulting in failure with minimal stress (Winandy & Lebow, 2001).

3.7 DEGREE OF INTEGRITY (I), FINE FRACTION (F) AND RESISTANCE TO IMPACT MILLING (RIM) 3.7 STOPNJA INTEGRITETE (I), DROBNA FRAKCIJA (F) IN ODPORNOST NA UDARNO MLETJE (RIM)

The degree of integrity of *Gmelina arborea* decreased significantly from 55.30% for untreated wood to the lowest value of 42.92% at 220 °C modification temperature. The fine fraction, which shows greater discrimination between untreated and modified wood, generally increased significantly when wood was modified, from 1.32% in untreated wood to 8.17% at 220 °C (Table 2). Overall, a significant reduction in RIM was recorded between untreated and modified wood. Untreated wood recorded RIM of 87.84% reaching a lowest value of 79.60% at 220 °C (Table 2). The reduction in RIM as a result of increased modification temperature

Table 2. Results of static bending, HEMI-test and Brinell hardness per treatment.

Preglednica 2. Rezultati statičnega upogiba, HEMI-testa in trdote po Brinellu glede na postopek obdelave.

| Treatment | MOE [N/mm ²] | MOR [N/mm ²] | I [%] | F [%] | RIM [%] | BH [N/mm ²] |
|-----------|-----------------------------------|------------------------------|------------------------------|-----------------------------|------------------------------|-------------------------------|
| Untreated | 9562.73 ^{ab} (125.60) | 85.85 ^a (2.00) | 55.30 ^a (0.78) | 1.32 ^a (0.08) | 87.84 ^a (0.16) | 17.39 ^{ab} (0.49) |
| 180 °C | 10221.82 ^a (250.97) | 82.56 ^a (2.11) | 53.85 ^a (0.67) | 1.19 ^a (0.11) | 87.57 ^a (0.22) | 20.65 ^c (0.57) |
| 200 °C | 9227.88 ^b (177.32) | 56.45 ^b (1.76) | 47.19 ^b (0.72) | 3.66 ^b (0.17) | 84.05 ^b (0.26) | 19.18 ^{ac} (0.52) |
| 220 °C | 7103.94 ^c (178.34) | 38.76 ^c (1.13) | 42.92 ^c (2.01) | 8.17 ^c (0.35) | 79.60 ^c (0.45) | 15.90 ^{bd} (0.53) |

Means followed by the same letter are not significantly different at $\alpha = 0.05$

Standard error values are shown in parentheses

MOE: Modulus of Elasticity, MOR: Modulus of Rupture, I: Integrity, F: Fine fraction, RIM: Resistance to Impact Milling, BH: Brinell Hardness.

could be ascribed to the reduced microstructural integrity which underlies the recorded increase in fragmentation and decrease in fragment size (Rapp et al., 2006; Welzbacher, et al., 2011). The decline in microstructural integrity occurred as a result of reductions in hemicelluloses content (Table 1) and increased cellulose crystallinity at higher modification temperatures leading to increased wood brittleness.

3.8 BRINELL HARDNESS

3.8 TRDOTA PO BRINELLU

Table 2 shows that Brinell hardness parallel to the grain increased from 17.39 N/mm² up to 20.65 N/mm² and 19.18 N/mm² as *Gmelina arborea* wood was thermally modified at 180 °C and 200 °C, respectively. It reduced to a minimum of 15.90 N/mm² at 220 °C modification temperature. Wood hardness increased with modification temperature in the instance of 180 °C and 200 °C, due to increased cellulose crystallinity as the modification temperature increased. However, the significant decrease observed at 220 °C could be due to enhanced degradation of hemicelluloses (Table 1), which reduced the wood's ability to withstand stresses (Winandy & Lebow, 2001). A similar trend was recorded in ash (*Fraxinus spp.* L.) and tali (*Erythrophleum ivorense* A. Chev.), each thermally modified at 180 °C for 1.5 h and 210 °C for 2 h. The Brinell hardness increased from 6.79 N/mm² and 4.51 N/mm² in untreated wood to 7.01 N/mm² and 10.81 N/mm² at 180 °C and subsequently reduced to 6.95 N/mm² and 9.19 N/mm² at 210 °C (Sivrikaya et al., 2015).

4 CONCLUSIONS

4 ZAKLJUČKI

The research focused on the effects of thermal modification at temperatures of 180 °C, 200 °C and 220 °C on chemical and mechanical changes in *Gmelina arborea* wood. Generally, the cellulose and hemicelluloses content decreased significantly after TM, with only lignin recording a significant increase with TM temperature. Additionally, MOE, MOR, I, and RIM decreased with increased modification temperature, recording the highest decreases of 26.13%, 58.30%, 83.69%, 22.37%, and 9.38%, respectively, at 220 °C. The

fine fraction and Brinell hardness increased up to 518.94% at 220 °C and 18.75% at 180 °C, respectively. The closely comparable strength properties between untreated wood and that modified at 180 °C makes this particular temperature acceptable for modification of this species, especially when used for structural purposes and other such applications, where the strength properties are critical. However, for purposes other than structural ones a modification temperature of 200 °C could be adopted to offer the added advantage of other improved wood properties. The results obtained in this study are generally useful as a reference for applications of thermally-modified *Gmelina arborea* wood.

5 SUMMARY

5 POVZETEK

Wood is a very important environmentally friendly, renewable and accessible material, which is widely preferred around the globe. However, the sustainable supply of tropical timber on the world market is increasingly threatened (Boonstra, 2008). In its 2016 report, the Forestry Commission (FC) of Ghana indicated that about 80% of the country's forest resources under state management had been lost to illegal logging activities (FC, 2016a). To reduce over-exploitation of primary forests and ensure a sustainable supply of timber on local and export markets, Ghana has embarked on the establishment of plantations covering about 190,000 ha as of 2015. The FC further plans to cultivate 100,000 ha of plantations from 2016 – 2040. Among the many species to be cultivated is *Gmelina arborea*, which is an exotic species (FC, 2016b). To complement the effort of the FC to curb deforestation in Ghana and reduce the focus on traditional primary timber species, there is a need to research the properties of the plantation species to establish their suitability for furniture and other such applications. Successful adoption of plantation species such as *G. arborea* for utilization on both local and export markets will depend on their properties. This study therefore investigated the impact of TM applied to enhance the properties of *G. arborea* wood from Ghana with regard to its basic chemical and mechanical properties.

Four *Gmelina arborea* trees were bucked into 2.5 m length bolts with a chainsaw, which were further processed using a portable Wood-Mizer sawmill (LT 30) into boards of 25 mm thickness. The boards were air-dried until moisture content (MC) below 20% was achieved. Boards were selected from within 15 cm radius of the pith to ensure the use of heartwood. These boards were further processed into slats of dimension 20 × 50 × 650 mm³ for the TM process. The slats were sorted according to weight and those in the range of 300 – 400 g were used for the study. The pre-sorting was necessary to homogenize the lot and minimize the effect of the initial density on the results.

An open TM system similar to the ThermoWood® process (Mayes & Oksanen, 2002) was employed. Peak temperatures of 180, 200, and 220 °C were adopted. Each peak temperature was held for 3 h. The mass loss of the wood slats after the TM process was determined according to Metsä-Kortelainen et al. (2006). The chemical composition of the wood was also conducted based on TAPPI and Wise et al. (1946). Static bending (MOE and MOR) was determined in accordance with DIN 52186 (1978). High Energy Multiple Impact (HEMI) testing was performed using methods outlined by Brischke (2017). The degree of integrity (I), fine fraction (F) and resistance to impact milling (RIM) were determined. Brinell (static) hardness were also carried out according to DIN EN 1534 (2011). Descriptive statistics comprising means with standard deviations were presented for each treatment and test. Comparison of the results from the treatments was made using Analysis of Variance (ANOVA). Least Square Deviation was used to compare means at $\alpha = 0.05$ when ANOVA revealed significant differences. The Statistical Package for Social Sciences (IBM Statistics) version 26 was used for the analysis.

Table 1 presents the mass loss, proportions of cellulose, hemicelluloses, lignin and extractives in the thermally modified *Gmelina arborea* wood. Mass loss increased along with the TM temperature from 5.44% at 180 °C to 15.17% at 220 °C. According to Esteves and Pereira (2007), mass loss is primarily due to thermal degradation of hemicelluloses and volatilization of extractives (Fig. 1). Variations in the cellulose concentrations at 180 °C (51.50%) and 220 °C (52.45%) were not significantly different, with both differing significantly from

untreated wood (55.93%). Hemicelluloses content decreased significantly from 19.16% when untreated to 6.33% at 220 °C (Table 1). Esteves et al. (2008) noted that cellulose is less affected by TM in comparison to hemicelluloses. According to Sivonen et al. (2002) and Nuopponen et al. (2004), hemicelluloses are the first wood structural component to be affected during TM. The highest reductions in cellulose and hemicelluloses were 8.60% at 180 °C and 65.40% at 220 °C respectively. Lignin content increased consistently from 34.86% for untreated wood to 44.16% for a modification temperature of 220 °C. Windeisen et al. (2007) reported that lignin degrades at the beginning of the modification process, but the rate of degradation is slower than polysaccharide (hemicelluloses) degradation, giving the apparent observed increase in lignin content. Generally, the extractive content (EC) was significantly reduced after TM. It decreased from the initial content of 7.87% for untreated wood to 6.35% at 180 °C and 220 °C (Table 1).

Table 2 presents the results of mechanical tests of thermally modified *Gmelina arborea* wood. Generally, a decreasing trend was observed in the modulus of elasticity (MOE) with an increase in temperature. At 220 °C, a significant reduction of 25.71% in MOE was observed compared to untreated wood. In spite of the marginal increase of 6.86% at 180 °C and decrease of 3.50% at 200 °C compared to the untreated wood's MOE, the differences were not statistically significant. The modulus of rupture (MOR) is one of the mechanical properties that are most affected by wood TM and decreases with increasing modification temperature (Esteves & Pereira, 2009). The MOR of *Gmelina arborea* wood was significantly reduced after TM, from 85.85 N/mm² when untreated to 38.76 N/mm² at 220 °C (Table 2). When amorphous hemicelluloses are degraded, the remaining and dominant cellulose become crystalline (Boonstra & Tjeerdsma, 2006). Removal of hemicelluloses thus leads to the distribution of stress over less cell wall material which is brittle, resulting in failure with minimal stress (Winandy & Lebow, 2011). The degree of integrity (I) of *Gmelina arborea* decreased significantly from 55.30% for untreated wood to a low of 42.92% at 220 °C modification temperature. The fine fraction, which shows greater discrimination

between untreated and modified wood, generally increased significantly when wood was modified, from 1.32% in untreated wood to 8.17% at 220 °C (Table 2). Overall, a significant reduction in RIM was recorded between untreated and modified wood. Untreated wood recorded RIM of 87.84% reaching a lowest value of 79.60% at 220 °C (Table 2). The reduction in RIM as a result of increased modification temperature could be ascribed to the reduced microstructural integrity which underlies the recorded increase in fragmentation and decrease in fragment size (Rapp et al., 2006; Welzbacher et al., 2011). Brinell hardness parallel to the grain increased from 17.39 up to 20.65 and 19.18 N/mm² as *Gmelina arborea* wood was thermally modified at 180 °C and 200 °C, respectively. It fell to a minimum of 15.90 N/mm² at 220 °C.

Generally, the cellulose and hemicelluloses contents decreased significantly after TM, with only lignin recording a significant increase with TM temperature. Additionally, MOE, MOR, I, and RIM decreased with increased modification temperature, recording the highest decreases of 26.13%, 58.30%, 83.69%, 22.37%, and 9.38%, respectively, at 220 °C. The fine fraction and Brinell hardness saw increases of up to 518.94% at 220 °C and 18.75% at 180 °C, respectively. Closely comparable strength properties between untreated wood and those modified at 180 °C, makes this particular temperature acceptable for modification of this species, especially for structural purposes and other such applications, where the strength properties are critical. However, for purposes other than structural ones a modification temperature of 200 °C could be adopted to offer the added advantage of other improved wood properties. The results obtained in this study are generally useful as a reference for applications of thermally modified *Gmelina arborea* wood.

Les je pomemben okolju prijazen, obnovljiv, dostopen in priljubljen material po vsem svetu. Trajnostna ponudba tropskega lesa je na svetovnem trgu vse bolj ogrožena (Boonstra, 2008). V svojem poročilu za leto 2016 je gozdarska komisija (FC) iz Gane navedla, da je bilo okoli 80 % državnih gozdnih virov izgubljenih zaradi nezakonite sečnje (FC, 2016a). Da bi zmanjšala prekomerno izkoriščanje primarnih gozdov in zagotovila trajnostno oskrbo z

lesom za porabo doma in za izvoz, je Gana v letu 2015 zasnovala nasade v obsegu približno 190.000 ha. FC nadalje načrtuje gojenje lesa na 100.000 ha nasadov v obdobju 2016–2040. Med številnimi vrstami, primernimi za gojenje, je *Gmelina arborea*, ki na območju predstavlja eksotično vrsto (FC, 2016b). Da bi dopolnili prizadevanja FC, omejili krčenje gozdov v Gani in zmanjšali prekomerno izkoriščanje tradicionalnih primarnih lesnih vrst, želijo raziskati lastnosti lesa plantažnih vrst in njihovo primernost za pohištvo in podobne namene. Uspešno uvajanje plantažnih vrst, kot je *G. arborea*, za uporabo doma in za izvoz, bo odvisno od lastnosti lesa. Ta študija zato vključuje raziskave vpliva postopka termičnega modificiranja (TM), ki se uporablja za izboljšanje relevantnih lastnosti lesa *G. arborea* iz Gane, in vpliva na njegove osnovne kemijske in mehanske lastnosti. Štiri drevesa vrste *Gmelina arborea* so bila z motorno žago razžagana na hlode dolžine 2,5 m, ki so bili nato s prenosno žago Wood-Mizer (LT 30) razžagani v deske debeline 25 mm. Deske smo osušili na zraku, do lesne vlažnosti (MC) pod 20 %. V nadaljevanju smo izbrali deske znotraj polmera 15 cm od stržena na območju jedrovine in jih razžagali na letve dimenzij 20 mm × 50 mm × 650 mm za postopek TM. Letve smo sortirali glede na maso lesa in za raziskavo uporabili tiste z maso od 300 do 400 g. Predhodno razvrščanje je bilo potrebno za homogenizacijo vzorca in zmanjšanje učinka gostote lesa na rezultate. Uporabili smo odprti sistem termične modifikacije, podoben postopku ThermoWood® (Mayes & Oksanen, 2002). Najvišje temperature tretiranja so bile 180, 200 in 220 °C, trajanje delovanja vsake od navedenih temperatur pa je bilo 3 ure. Izguba mase lesa je bila po TM določena v skladu z Metsa-Kortelainen et al. (2006). Kemijsko sestavo lesa smo določili na podlagi TAPPI in Wise et al. (1946). Statično upogibno trdnost in modul elastičnosti (MOR in MOE) smo določili v skladu z DIN 52186 (1978). Preskus večkratnih visokoenergijskih udarcev (HEMI) je bil izveden z uporabo metod, ki jih je opisal Brischke (2017). Določena je bila stopnja integritete (I), drobne frakcije (F) in odpornost na udarno mletje (RIM). (Statična) trdota po Brinellu je bila določena v skladu z DIN EN 1534 (2011). Za rezultate posamičnega postopka in testiranja smo izračunali osnovno statistiko, srednje vrednosti s standardnim odklonom. Primerjavo rezultatov po postopkih smo ocenili z analizo variance

(ANOVA). LSD test mnogoterih primerjav smo uporabili za primerjavo povprečij pri stopnji zaupanja $\alpha = 0,05$, ko je ANOVA pokazala statistično značilne razlike. Za analize smo uporabili Statistični paket za družbene vede (IBM Statistics) različice 26.

V preglednici 1 so predstavljeni deleži celuloze, hemiceluloz, lignina, ekstraktivnih snovi in izgube mase v termično modificiranem lesu vrste *G. arborea*. Razlike v deležih celuloze pri 180 °C (51,50 %) in 220 °C (52,45 %) niso bile statistično značilne, v obeh primerih pa smo zabeležili bistvene zmanjšanje deleža celuloze v primerjavi z neobdelanim lesom (55,93 %). Vsebnost hemiceluloz se je znatno zmanjšala z 19,16 % (neobdelan les), na 6,33 % pri 220 °C (preglednica 1). Esteves et al. (2008) so ugotovili, da TM bolj zmanjšuje delež hemiceluloz kot celuloze. Sivonen et al. (2002) in Nuopponen et al. (2004) so objavili, da TM najbolj vpliva na deleže hemiceluloz. Najvišje znižanje deležev celuloze in hemiceluloz je bilo 8,60 % pri 180 °C in 65,40 % pri 220 °C. Vsebnost lignina se je sistematično povečala s 34,86 % pri neobdelanem lesu na 44,16 % pri lesu, obdelanem pri temperaturi 220 °C. Windeisen et al. (2007) so poročali, da se lignin razgrajuje na začetku postopka modifikacije, vendar je stopnja razgradnje počasnejša od razgradnje polisaharidov (hemiceluloze), kar ima za posledico znatno povečanje vsebnosti lignina. Na splošno se je vsebnost ekstraktivnih snovi (EC) po TM znatno zmanjšala. Z začetne vsebnosti 7,87 % v neobdelanem lesu se je zmanjšala na 6,35 % pri 180 °C in 220 °C (preglednica 1). Izguba mase se je povečala s povečanjem temperature TM za 5,44 % pri 180 °C do 15,17 % pri 220 °C. Esteves & Pereira (2007) poročata, da je izguba mase predvsem posledica toplotne razgradnje hemiceluloz in hlapenja ekstraktivnih snovi (slika 1).

V preglednici 2 so predstavljeni rezultati mehanskih preskusov toplotno modificiranega lesa vrste *G. arborea*. Na splošno smo opazili trend zniževanja modula elastičnosti (MOE) z višanjem temperature. Pri 220 °C smo opazili znatno zmanjšanje MOE za 25,71 % v primerjavi z neobdelanim lesom. Kljub mejnemu povečanju 6,86 % pri 180 °C in 3,50 % pri 200 °C v primerjavi z MOE neobdelanega lesa razlike niso bile statistično pomembne. Upogibna trdnost (MOR) je ena od mehanskih lastnosti, ki se najbolj zmanjša pri TM v odvisnosti od naraščanja temperature (Esteves & Pereira, 2009).

Upogibna trdnost lesa vrste *G. arborea* se je po TM znatno zmanjšala, in sicer s 85,85 N/mm² pri neobdelanem, na 38,76 N/mm² pri lesu, obdelanem pri temperaturi 220 °C (preglednica 2). Ko se amorfne hemiceluloze razgradijo, postane preostala celuloza bolj kristalinična (Boonstra & Tjeerdsma, 2006). Odstranjevanje hemiceluloz vodi do porazdelitve stresa po manjši količini materiala celične stene, ki je krhek, kar povzroči porušitev že pri nizki napeptosti (Winandy & Lebow, 2011). Stopnja integritete (I) lesa *G. arborea* se je znatno zmanjšala s 55,30 % pri neobdelanih do 42,92 % pri 220 °C. Drobna frakcija, ki kaže večje razlike med neobdelanim in modificiranim lesom, se je na splošno znatno povečala z modifikacijo, od 1,32 % v neobdelanem lesu na 8,17 % pri 220 °C (preglednica 2). V glavnem je bilo zabeleženo znatno zmanjšanje RIM med neobdelanim in modificiranim lesom. Neobdelani les je imel RIM 87,84 %, ki je pri 220 °C padel na 79,60 % (preglednica 2). Zmanjšanje RIM zaradi povečane temperature modifikacije bi lahko pripisali zmanjšani mikrostrukturni celovitosti, ki je podlaga za povečanje fragmentacije in zmanjšanje velikosti delcev (Rapp et al., 2006; Welzbacher et al., 2011). Trdota po Brinellu vzporedno s potekom aksialnih elementov se je povečala s 17,39 na 20,65 oz. 19,18 N/mm², po toplotni modifikaciji pri 180 °C oziroma 200 °C. Trdota se je znižala na 15,90 N/mm² pri 220 °C. Na splošno se je vsebnost celuloze in hemiceluloz po TM znatno zmanjšala, le delež lignina se je znatno povečal s temperaturo TM. Poleg tega so se MOE, MOR, I in RIM znižali s povišano temperaturo modifikacije, pri čemer smo zabeležili njihova najvišja znižanja za 26,13 %, 58,30 %, 83,69 %, 22,37 % in 9,38 % pri 220 °C. Drobna frakcija in trdota po Brinellu sta se povečali do 518,94 % pri 220 °C in 18,75 % pri 180 °C. Majhna izguba trdnosti po obdelavi pri 180 °C nakazuje, da je ta temperatura sprejemljiva za modifikacijo lesa proučene vrste, zlasti kadar jo želimo porabiti za konstrukcijske in podobne namene, kjer so trdnostne lastnosti zelo pomembne. Za uporabo lesa za nekonstrukcijske namene pa bi bila lahko sprejemljiva tudi temperatura modifikacije 200 °C, da bi dosegli dodatno izboljšanje drugih lastnosti lesa. Predstavljeni rezultati te študije bodo po pričakovanju splošno uporabni kot referenca za uporabo toplotno modificiranega lesa pogoste plantažne vrste *Gmelina arborea*.

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