

# Enhancement of technical value of oil palm (*Elaeis guineensis* Jacq.) waste trunk through modification with 1,3-dimethylol-4,5-dihydroxyethyleneurea (DMDHEU)

Mariusz Mamiński<sup>1</sup> · Paweł Kozakiewicz<sup>1</sup> · Waldemar Jaskółowski<sup>2</sup> ·  
Kit Ling Chin<sup>3</sup> · Paik San H'ng<sup>3</sup> · Renata Toczyłowska-Mamińska<sup>1</sup>

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**Abstract** Malaysia is the biggest producer of palm oil in the world. The production generates over 70 metric tons of waste trunks per hectare during replantation. Such an abundant feedstock should be considered a valuable raw material rather than an agro-waste. An approach for enhancement of low density trunks through the treatment with 1,3-dimethylol-4,5-dihydroxyethyleneurea (DMDHEU) was investigated. The treatment resulted in a great improvement in the properties of the material: 45 % density gain, water absorption and thickness swelling reduced by 48 and 43 %, respectively, 2.3-fold increase in hardness as well as 3.8-fold and 3.6-fold increase in bending strength and modulus of elasticity, respectively, were observed. Thus, enhancement in the physical and mechanical performance of the material as well as the increased aesthetic value due to the color changes proved the approach to be effective for the conversion of waste biomass to new products.

## 1 Introduction

Today wood-based composites industry suffers from the increasing deficiency of raw materials which results in price fluctuations and difficulties in long-term logistic

planning. In order to ease that inconvenience, it is rationale to undertake research aimed at the development of new types of materials based on alternative, non-classic lignocellulosic resources—preferably waste biomass from agriculture or fast growing species.

The underutilized raw material which cannot be neglected as a raw material is agricultural waste from oil palm (*Elaeis guineensis*) plantations. In 2012, total area of oil palm plantations located in Malaysia and Indonesia exceeded 10 million hectares. Since during harvesting each hectare yields over 70 tones of dry waste trunk biomass (Chin et al. 2012), the resource seems to be cheap, abundant and promising prospective raw material for chemical processing to sugars, alcohols and other derivatives (Akmar and Kennedy 2001; Tay et al. 2013; Chin et al. 2010; Wanrosli et al. 2004). Alternatively, palm trunk can be used as a source of lignocellulosic material for wood technology in particleboard (Baskaran et al. 2013), plywood (Hoong et al. 2013), furniture (Suhaily et al. 2012) or laminated veneer lumber (Sarmin et al. 2013).

To date wide industrial utilization of the resource is still not common, mainly, because of the serious drawbacks palm trunk exhibits. Investigations by Erwinsyah (2008) revealed extremely high variations in physical and mechanical properties: variable density (140–600 kg m<sup>-3</sup> within one trunk), while modulus of elasticity ranged from about 922–6860 MPa and modulus of rupture between 7.5 and 5.4 MPa depending on the trunk height and trunk cross-sectional zone. The variations in the properties result from palm morphology—i.e., primary vascular bundles embedded within the mechanically weak matrix of parenchyma cells. This also affects dimensional stability of the material in moist environments which markedly limits its applications.

In order to overcome the above mentioned drawbacks, it is necessary to improve biomechanical performance of

✉ Mariusz Mamiński  
mariusz\_maminski@sggw.pl

<sup>1</sup> Faculty of Wood Technology, Warsaw University of Life Sciences-SGGW, 159 Nowoursynowska St., 02-776 Warsaw, Poland

<sup>2</sup> The Main School of Fire Service, 52/56 Słowackiego St., 01-629 Warsaw, Poland

<sup>3</sup> Institute of Tropical Forestry and Forest Products (INTROP), Universiti Putra Malaysia, 43400 UPM Serdang, Selangor, Malaysia

palm trunk, so that a value-added material of uniformed and enhanced properties and thus, of increased technical value can be obtained. One of the approaches which significantly improve strength, hardness and dimensional stability is modification by impregnation. The treatments can be performed using various chemicals: reactive thermosetting resins, alcohols (Dungani et al. 2013; H'ng et al. 2013; Szymona et al. 2014; Abdul Khalil et al. 2010) or natural rubber and polypropylene (Sibirian et al. 2005). Recently, Szymona et al. (2014) reported on palm trunk impregnation with bio-based furfuryl alcohol which can be obtained from the renewable resources. The novel material exhibited significantly enhanced mechanical and physiochemical properties.

In this work the modification of oil palm trunk with 1,3-dimethylol-4,5-dihydroxyethyleneurea (DMDHEU), unlike furfuryl alcohol, provides light-colored product of increased technical value.

1,3-dimethylol-4,5-dihydroxyethyleneurea molecule contains two *N*-methylol and two secondary hydroxyl groups which are reactive toward hydroxyls present in cotton (cellulosic) fiber (Ibrahim et al. 2008; Yang et al. 2009).

Although an industrial process for wood modification based on DMDHEU has been developed and patented under the tradename Belmadur® by BASF, investigations on the use of DMDHEU resins in wood technology for wood and veneers modifications are still being carried out (Dieste et al. 2009; Pfeffer et al. 2011; Xie et al. 2005). The treatment with DMDHEU was found to be effective at preventing the degradation of the wood during weathering (Xie et al. 2005) and fungal penetration into the wood tissue (Pfeffer et al. 2012). Modification of veneers resulted in increased dimensional stability and hardness as well as in a positive effect on veneer bending strength (Dieste et al. 2008).

The objective of this work was to investigate the effect of DMDHEU modification on the physical and mechanical properties of modified palm trunk, so that the technical value of waste biomass was increased and—subsequently—the area of its utilization was extended.

According to the authors' knowledge this is the first paper on the novel type of material rendered by the treatment of oil palm trunk with DMDHEU.

## 2 Materials and methods

Oil palm trunk (*Elaeis guineensis*) of average density  $400 \pm 20 \text{ kg m}^{-3}$  was used in the studies. DMDHEU resin (pH 4.3; solids 34 %; viscosity 27 mPas manufactured by BASF Chemicals, Germany) was used in the experiments.

Modification of oil palm trunk was based on the modified procedure previously described by Szymona et al. (2014). Prior to modification, specimens ( $50 \times 50 \times 30 \text{ mm}^3$  or  $20 \times 20 \times 250 \text{ mm}^3$ ) were subjected to 24-h oven-drying at  $103 \text{ }^\circ\text{C}$ , then weights and dimensions of the specimens were recorded. Vacuum impregnations (0.01 MPa, 45 min) were performed in 34 % (34 %–DMDHEU series) or diluted 17 % (17 %–DMDHEU series) aqueous solutions of 1,3-dimethylol-4,5-dihydroxyethyleneurea containing 5 %wt *p*-toluenesulfonic acid (*p*-TSA) as the catalyst. Then samples were dried for 24 h at  $160 \text{ }^\circ\text{C}$ , afterwards the final weights and dimensions of the specimens were recorded. In order to assess the effect of the temperature, a series of specimens (thermal reference) was subjected to thermal treatment at  $160 \text{ }^\circ\text{C}$  for 24 h.

Thickness swelling (*TS*) was measured according to EN 317 (1999) standard. Water absorption (*WA*) calculations were based on the following formula (1):

$$WA = (m_n - m_s) / m_s \times 100 \% \quad (1)$$

where: *WA*—water absorption (%),  $m_n$ —weight of a specimen after soaking (g),  $m_s$ —weight of a specimen before soaking (g). 24-h soaking was performed in deionised water.

The total color change ( $\Delta E$ ) of a material can be described in the CIEL\*a\*b coordinates system defined in ISO 7724–3 (2003) (2)

$$\Delta E = \sqrt{(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2} \quad (2)$$

where  $\Delta L$  sample lightening/darkening,  $\Delta a$  red/green shift,  $\Delta b$  yellow/blue shift. An SP-60 (X-Rite) spherical spectrophotometer was used for color analysis. Untreated oil palm was used as the reference.

Weight percent gain (*WPG*) was an estimator of the uptake of the DMDHEU by the specimens. *WPG* values were calculated from the relation (3):

$$WPG = (m_1 - m_0) / m_0 \times 100 \% \quad (3)$$

where:  $m_0$ —initial oven-dry weight of a specimen (g),  $m_1$ —oven-dry weight of a specimen after treatment.

Hardness of the DMDHEU-modified palm was determined on the tangential cross-section using a Brinell digital hardness tester CV-3000LDB (Bowers Metrology, UK) and calculated according to the following formula (4):

$$HBN = \frac{2P}{\pi D(D - \sqrt{D^2 - d^2})} \quad (4)$$

where: *P*—loading (N), *D*—ball diameter 10 mm and *d*—indentation diameter (mm). Low intrinsic hardness of the tested materials imposed a modification of the procedure, so that a loading of 100 N was applied by a steel ball indenter onto a specimen for 30 s.

Bending properties were examined in a three-point static bending test according to DIN 52186 (1978). Tests were performed on a universal testing machine Instron 3369 (Instron, USA). Specimens of dimensions 20 × 20 × 250 mm<sup>3</sup> (radial × tangential × longitudinal) were subjected to bending at 1 mm min<sup>-1</sup> rate.

The specimens of dimensions 50 × 50 × 30 mm<sup>3</sup> were subjected to density profile measurements on an X-ray density analyzer DA-X (GreCon, Germany). Measurements were carried out at a scanning speed of 0.21 mm s<sup>-1</sup>.

Light microscopy analyses were performed on 30-μm thick microtomed slices of the material using an Olympus BX41 (Olympus Deutschland GmbH, Germany) microscope. Prior to microtoming, specimens were soaked in a mixture of water/glycerol/ethanol (1/1/1, volumetric ratio). Quantitative flammability tests of the palm trunk were run on a cone calorimeter (Fire Testing Technology, UK) according to ISO 5660-1 (2002) standard. Specimens of dimensions 100 × 100 × 10 mm<sup>3</sup> wrapped in aluminum foil were exposed horizontally to external heat flux of 50 Kw m<sup>-2</sup>. The following parameters were determined: heat release rate (HRR), total heat release (THR), total smoke release (TSR), effective heat of combustion (EHC), yield CO and CO<sub>2</sub>, and maximum average rate of heat emission (MARHE). Additionally, estimators of fire development—i.e., HRR after 60, 180 and 300 s from ignition were determined.

### 3 Results and discussion

In general, chemical modification of a woody material results in an alternation in the density due to fixing of the modifying agent within wood tissue. The retention of an impregnating agent within the material subjected to modification can be expressed by the weight percentage gain (WPG). Relevant WPG values presented in Table 1 show that the density and WPG depended on the concentration of DMDHEU solution which remains in accordance with the report by Dieste et al. (2008). So that 36 ± 4 and 22 ± 6 % WPGs were observed for 34- and 17 %-DMDHEU series, respectively, which resulted in apparent

45 % and 22 % increase in the density from initial 400 kg m<sup>-3</sup>. The values remain several times lower than those previously reported for modifications with furfuryl alcohol (Szymona et al. 2014). The phenomenon is apparently correlated with the concentration of the resin in the impregnating solution—i.e., 34 or 17 % for DMDHEU versus 90 or 45 % for furfuryl. However, the obtained WPG values remain comparable to those found for other types of modification—ca. 20 % (Gabrielli and Kamke 2010; Papadopoulos 2005).

The depth of DMDHEU penetration into the material was investigated by X-ray profiling. The profiles shown in Fig. 1 indicate that the low-molecular impregnating agent penetrated the whole volume of the specimens, so that material density was affected even in the most internal zone. The gravimetrically determined density remained in accordance with observations from the X-ray measurements and revealed changes from initial 400 ± 20–490 ± 25 and 580 ± 30 kg m<sup>-3</sup> for 17 %- and 34 %-DMDHEU series, respectively while the average DMDHEU retentions manifested in WPG values were 22 and 36 % (Table 1).

A throughout-penetration must have resulted in the uniformed and improved mechanical properties of the palm. Indeed, Brinell hardness tests revealed a relative

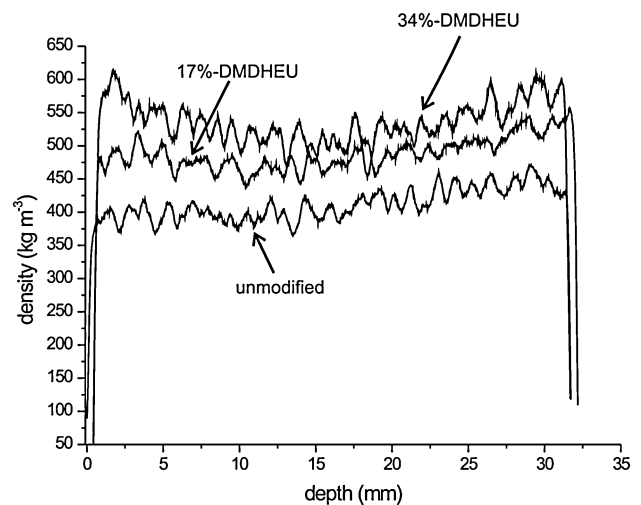


Fig. 1 Density profiles from X-ray scanning

**Table 1** Selected properties of DMDHEU-modified oil palm trunk

Series	WPG %	Density kg m <sup>-3</sup>	Water absorption %	Thickness swelling %	BHN
Untreated	–	400 (20)	36.5 (4.7)	2.3 (0.8)	1.3 (0.3)
17 %-DMDHEU	22	490 (25)	27.2 (3.5)	2.5 (1.0)	2.1 (0.3)
34 %-DMDHEU	36	580 (30)	19.0 (3.2)	1.3 (0.6)	3.0 (0.2)
Thermal reference	–	395 (33)	32.1 (4.9)	2.6 (0.8)	1.3 (0.4)

Values in parentheses are standard deviations (10 replicates)

WPG weight percentage gain, BHN Brinell hardness number

increase in Brinell hardness number (BHN) 2.3- and 1.6-fold for 34 %- and 17 %-DMDHEU, respectively, while hardness of the thermal references remained unchanged (Table 1). The results exactly correspond to those reported by Dieste et al. (2008) for *Betula sp.* and *Fagus sylvatica*. The obtained increase in hardness was even higher than that reported for pine in the Belmadur<sup>®</sup> process—i.e., 35 % ([http://www.muenchinger-holz.de/pdfs/Belmadur\\_handout\\_1005d.pdf](http://www.muenchinger-holz.de/pdfs/Belmadur_handout_1005d.pdf), Accessed 30 June 2015) and was close to those found for the beech wood modified with vinyl monomers (BHN 3–5) (Şolpan and Güven 1999). The phenomenon can be explained by the polymerization of DMDHEU within the tissue of palm trunk resulting in hardening and consolidation of soft parenchymatous matrix.

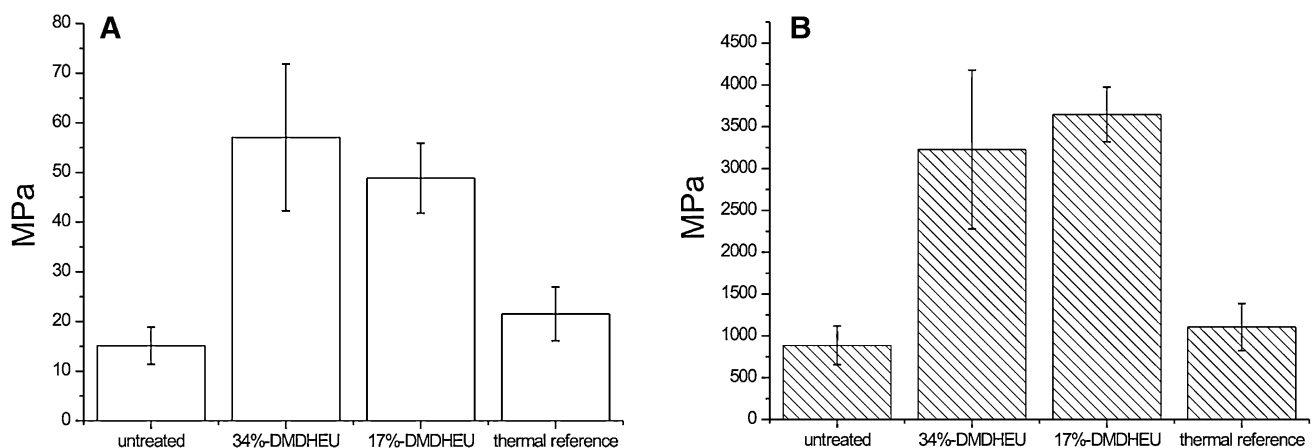
Thus, it was demonstrated that DMDHEU modification of a soft and weak material such as oil palm trunk may become an approach to upgrade its hardness to the level close to wood species like pine and maple. Subsequently, it is possible to increase palm technical value, so that its application as an interior design material may be considered—for example class-2 flooring (EN 13226 2002). The use of more concentrated impregnating solutions would probably provide a material of even higher hardness.

Another important feature which determines the field of application of a material is bending strength. The performed bending tests revealed a significantly positive effect of the treatment on the modulus of rupture (MOR) and elasticity (MOE) as shown in Fig. 2. It is clear that the modification resulted in 3.8- and 3.2-fold increase in MOR, as well as 3.6- and 4.11-fold increase in MOE for 34 %- and 17 %-DMDHEU series, respectively when compared to the unmodified references. The observed improvement in bending properties was significantly higher than that reported for the acrylate-modified beech or pine where a few percent were a typical gain (Şolpan and Güven 1999; Epmeier et al. 2004). It is worth noting that the approach allowed obtaining MOR values comparable to those of low

density wood species—for example poplar (53 MPa) which undoubtedly is a significant improvement. As mentioned in the literature, wood modification with DMDHEU, due to the use of magnesium chloride as a catalyst, is recognized as a process which degrades cellulose in the cell wall resulting in a substantial decrease in wood bending strength (Nicholas and Williams 1987; Xie et al. 2005, 2007) while the present studies rendered an opposite effect. The phenomenon might possibly come from the use of *p*-toluenesulfonic acid instead of magnesium chloride which is converted in situ to hydrochloric acid. Since the diluted *p*-toluenesulfonic acid is a weaker acid ( $pK_a = -2.8$ ) than HCl ( $pK_a = -8.0$ ), the degradation of cellulose is lesser.

Although the benefit in bending strength is significant, the approach is not effective enough to upgrade material properties to construction-grade. However, non load-bearing applications of DMDHEU-treated oil palm trunk are very likely and furniture or interior decorations may become potential fields of its utilization.

In general, modifications are aimed, among others, at the improvement of wood dimensional stability upon sorption-desorption in highly moist environment. The results from thickness swelling (TS) and water absorption (WA) after 24-hr soaking (Table 1) revealed that impregnation with 34 %-DMDHEU reduced both TS and WA by 43 and 48 %, respectively while impregnation with 17 %-DMDHEU reduced only water absorption by 25 %. Resultant thickness swelling was 2–3 times lower than that reported in the literature for beech or birch impregnated with 2.3 M DMDHEU (Dieste et al. 2008) and slightly higher than that previously reported for furfural-modified palm (Szymona et al. 2014). On the other hand, water absorption after 24-h soaking was lower than that found for furfural-treated palm. Thus, the phenomena can be explained by DMDHEU penetration into the material (Fig. 1) and curing which provided an enhanced dimensional stability and reduced interactions with water.

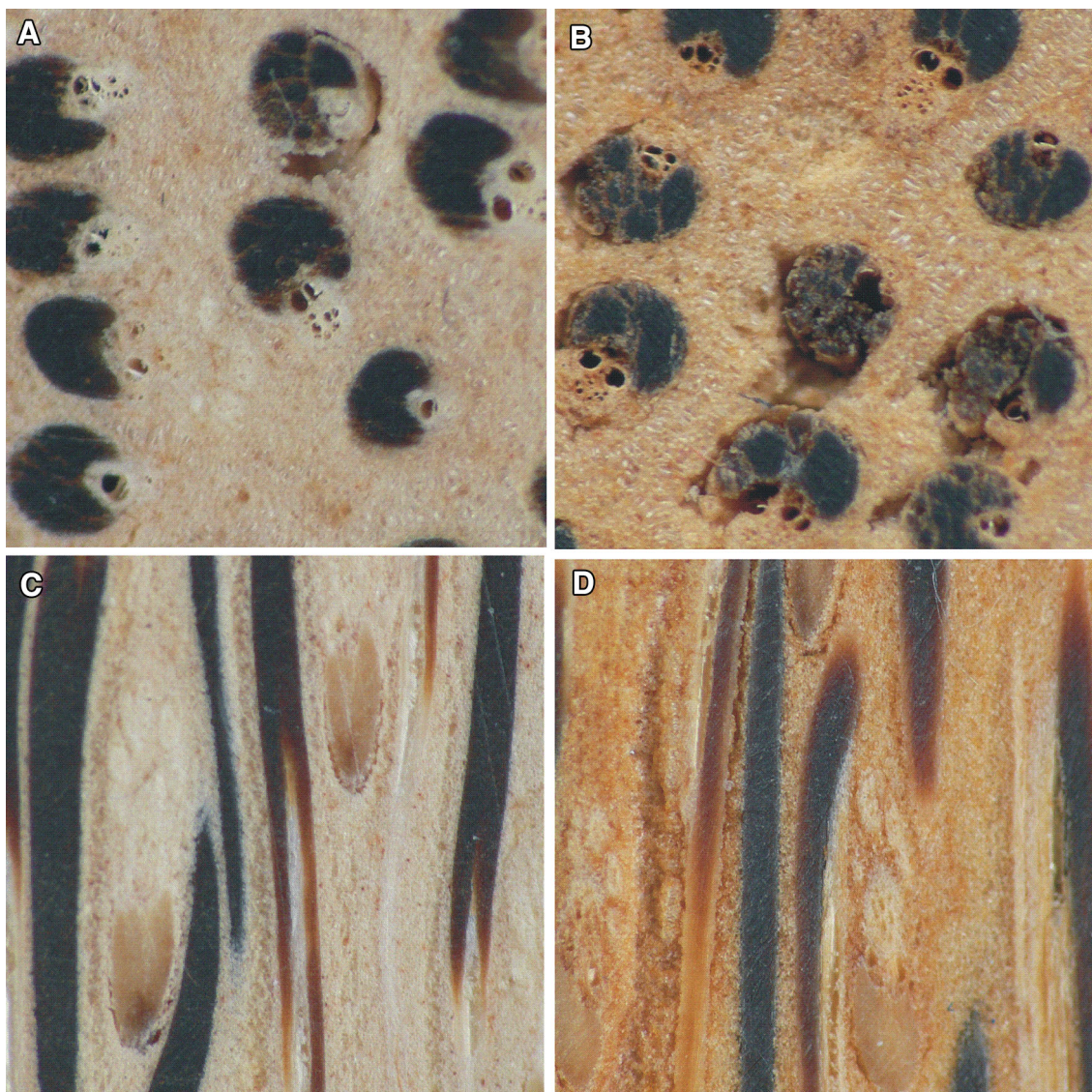


**Fig. 2** a Modulus of rupture (MOR), b Modulus of elasticity (MOE) of the DMDHEU-modified oil palm trunk

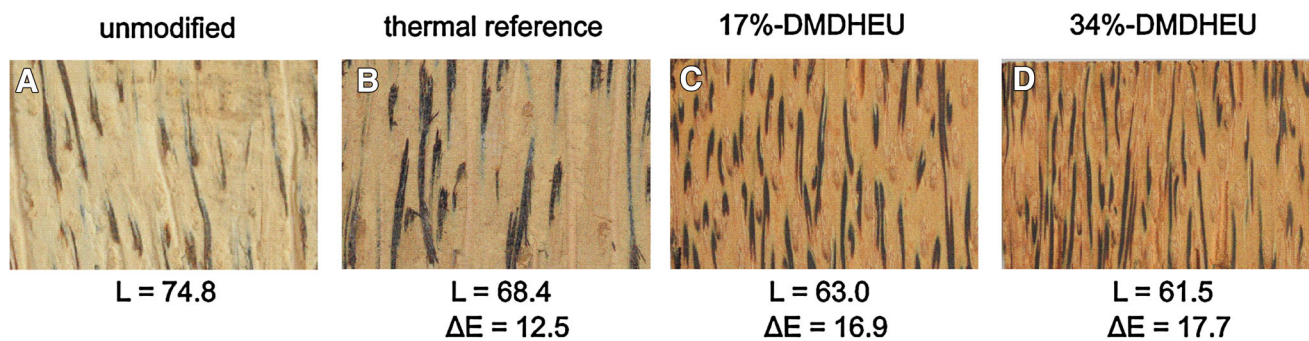


Obviously, such retention of DMDHEU must be accompanied by visible changes in morphology of the material (Fig. 3a–d). Light microscopy showed DMDHEU deposition in the form of thin film onto lumen walls without filling the whole volume. Higher concentration of the cured-DMDHEU is apparent in the thin-walled porous parenchyma cells as well as in the vessels and sieve tubes, while lower concentration was found in phloem and sclerenchyma cells. Such distribution resulted from the abundance of tracheids and sieve tubes in the parenchyma which made solution penetration easy. On the other hand, penetration into thick-walled fibers was limited. Wall thickness was not affected and remained comparable to that for the unmodified palm.

From the aesthetic point of view the color and hue of a material is the key factor, therefore the color change of the material resulting from the DMDHEU treatments was analyzed by means of spectrophotometry. In Fig. 4, resultant color changes and respective  $L$  and  $\Delta E$  values are presented. One can see that—unlike for the furfurylation where dark brown to black color was obtained (Szymona et al. 2014)—the treatment with DMDHEU does not bring such a severe effect. The most intense darkening observed for 34 %-DMDHEU manifested in  $\Delta L = 13.3$  and  $\Delta E = 17.7$ . The values are much lower than those for furfurylated palm where  $\Delta L$  was ca. 44. It can also be noticed that the color change due to the thermal treatment was weak and overwhelmed by the effect of DMDHEU



**Fig. 3** Microscopic structure of *Elaeis guineensis* Jacq.: **a** natural, transverse section; **b** modified (34 %), transverse section; **c** natural, longitudinal section; **d** modified (34 %), longitudinal section



**Fig. 4** Aesthetic effect of the modification on the color of palm and respective  $L$  and  $\Delta E$  values

**Table 2** Fire properties of modified oil palm

	Unmodified reference	34 %-DMDHEU	17 %-DMDHEU	Thermal reference
Mean HRR ( $\text{kW m}^{-2}$ )	88.6 (3.4)	136.8 (4.2)	94.6 (3.4)	104.2 (3.6)
Mean EHC ( $\text{MJ kg}^{-1}$ )	11.8 (0.4)	19.4 (0.7)	12.1 (0.5)	12.6 (0.5)
Mean COY ( $\text{kg kg}^{-1}$ )	0.02 (0)	0.02 (0)	0.02 (0)	0.02 (0)
Mean CO2Y ( $\text{kg kg}^{-1}$ )	1.1 (0)	1.1 (0)	1.2 (0)	1.1 (0)
THR ( $\text{MJ m}^{-2}$ )	40.9 (1.8)	106.2 (2.9)	72.1 (2.6)	72.4 (2.8)
TSR ( $\text{m m}^{-1}$ )	93.7 (2.8)	90.2 (2.6)	94.9 (2.8)	88.4 (2.5)
MARHE ( $\text{kW m}^{-2}$ )	96.4 (3.6)	143.6 (4.3)	111.9 (3.6)	116.3 (3.8)
HRR (60) ( $\text{kW m}^{-2}$ )	105.3 (5.7)	104.4 (6.3)	135.9 (8.0)	140.2 (6.7)
HRR (180) ( $\text{kW m}^{-2}$ )	105.4 (4.7)	120.8 (5.9)	103.7 (5.0)	107.9 (6.1)
HRR (300) ( $\text{kW m}^{-2}$ )	106.9 (4.4)	128.7 (6.0)	97.3 (4.6)	100.9 (3.9)

Standard deviations are given in parentheses

*HRR* heat release rate, *THR* total heat release, *TSR* total smoke release, *EHC* effective heat of combustion, *MARHE* maximum average rate of heat emission

(Fig. 4). The observed effect of heat treatment on the color of palm was much weaker than that reported by Esteves et al. (2008) for pine and eucalypt wood. Thus, it indicates a lower palm susceptibility to formation of colored degradation products from hemicelluloses and extractives when compared to typical wood species (McDonald et al. 1997; Sundqvist 2004). The developed mild “gold tan-effect” allowed to retain and expose natural material structure and texture.

Thermokinetic properties of oil palm under fire conditions were also characterized. It was found that its behavior was typical for wood and wood based composites. The values shown in Table 2 indicate an increase in mean heat release (HRR), total heat release (THR), effective heat of combustion (EHC) and maximum average rate of heat emission (MARHE) when compared to the unmodified reference.

It is not surprising since a significant amount of the flammable resin was incorporated into the porous woody material. The results are coherent with those found for DMDHEU-treated oak by Xie et al. (2014). The observed total release of smoke, CO and CO<sub>2</sub> remained constant for

all types of materials, so that it is clear that DMDHEU had no effect on that parameter.

Since fire development is closely related to the heat release in time, it is apparent that DMDHEU-incorporated into the porous structure of palm should have affected its flammability. When HRR after 60, 180 and 300 s from the ignition point is analyzed, one can see that HRR value was constant in time for the reference series only, while HRR for 34 %-DMDHEU-modified series increased with time. The time-dependent decrease in HRR was observed for thermal reference and 17 %-DMDHEU modified series which indicates that the amount of the resin deposited in the latter material was not sufficient to sustain fire.

## 4 Conclusion

The obtained results showed that DMDHEU treatment of oil palm trunk was a convenient and effective approach to significantly increase its technical value. The modification provides improvement in the key physical and mechanical properties of this naturally weak and low-value agro-waste.



The present studies revealed bulk consolidation of the material, improved bending strength and hardness, as well as, enhanced dimensional stability and reduced interactions with water. Additionally, the observed color changes increased palm aesthetic value. It was demonstrated that the DMDHEU-modified palm may be considered as a new, waste biomass-derived material for furniture, flooring or interior decorative purposes. Further studies on the machining and processing of this new material are in progress.

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