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Chemical, Thermal, and Morphological Properties of Sustainable Lignocellulosic Biomaterial

Kemijska, toplinska i morfološka svojstva održivoga lignoceluloznog biomaterijala

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ABSTRACT • *By the present paper, an experimental investigation was proposed to compare the morphological, chemical and mechanical properties of the Petiole Date Palm Wood (PDPW) collected from north and south of Algeria. Both regions have very distinctive climatic conditions – wet conditions in the north (near the Mediterranean Sea) and semi-arid conditions in the south Biskra. To achieve this aim, large quantities of waste from PDPW were collected, cleaned, and cut according to the normalization specific to each type of test. After that, scanning electron micrographs, infrared spectroscopy, dynamic mechanical thermal analysis (DMTA) and thermogravimetric analysis were made, and moisture content (MC) and water absorption (WA) were determined, to compare the effect of the different environmental conditions. From the results obtained, petiole date palm wood typical for the southern region had a high fiber content, attributed to its low porosity and excellent mechanical properties, which resulted in low water absorption. In contrast, the northern petiole wood reveals a higher glass transition temperature (T_g) and significant damping coefficient. At comparable relative density values, the specific properties were found to be comparable to those of balsa, foams and metallic honeycombs.*

KEYWORDS: *petiole date palm wood; mechanical properties; glass transition; porosity; damping*

SAŽETAK • *U radu je predstavljeno eksperimentalno istraživanje morfoloških, kemijskih i mehaničkih svojstava drva peteljki palme datulje (PDPW) prikupljenih na sjeveru i jugu Alžira. Te dvije regije imaju vrlo različite klimatske uvjete: na sjeveru su vlažni uvjeti (u blizini Sredozemnog mora), a na jugu Biskre klima je polusušna. Kako bi se postigao cilj istraživanja, skupljene su velike količine otpada PDPW-a, koje su očišćene i izrezane prema normi za svaku vrstu ispitivanja. Na pripremljenim uzorcima napravljena je pretražna elektronska mikrografija, infracrvena spektroskopija, dinamička mehanička toplinska analiza i termogravimetrijska analiza te je određen sadržaj vode (MC) i upijanje vode (WA) kako bi se odredio učinak različitih okolišnih uvjeta na svojstva PDPW-a. Rezultati istraživanja pokazali su da drvo peteljki palme datulje iz južne regije ima visok sadržaj vlakana, što se pripisuje njegovoj niskoj poroznosti i izvrsnim mehaničkim svojstvima, a rezultiralo je slabim upijanjem vode.*

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Nasuprot tome, drvo peteljke palme datulje iz sjeverne regije ima veću temperaturu stakljšta (T_g) i koeficijent prigušenja. U usporedbi s materijalima podjednake relativne gustoće, utvrđeno je da su specifična svojstva PDPW-a usporediva s onima balze, pjene i metalnog saća.

KLJUČNE RIJEČI: *drvo peteljke palme datulje; mehanička svojstva; staklište; poroznost; prigušenje*

1 INTRODUCTION

1. UVOD

Wood, a material composed of cellulose, hemicelluloses, lignin, and other components, exhibits viscoelastic properties. Its use in structural applications dates back to ancient times (Dave *et al*., 2018). Due to its renewability, affordability, specific strength, and ability to be tailored for various mechanical needs, wood is a promising ecological substitute for synthetic materials like carbon, glass fiber, and foams. These synthetic materials are commonly used in the construction of bridges, furniture, acoustic panels, and the interior structures of vehicles and aircraft. Consequently, researchers have increasingly focused on exploring wood, either independently or as an additive, in recent years (Candan *et al*., 2016). Atas *et al*. (2010) have used the balsa wood as a core of sandwich panels, demonstrating superior impact resistance compared to those using polyvinyl chloride (PVC) foam. Sofia *et al*. (2007) conducted a characterization study on cork oak wood, suggesting its potential application in solid wood structures due to its satisfactory physical properties. Hassanin *et al*. (2016) produced particleboards based on pine wood particles and polyester resins and observed significant increases of 19 % and 311.3 % in flexural strength and internal bending strength, respectively, compared to commercial wood particleboards.

Climate and environmental factors strongly influence the properties of biomass due to its hygroscopic nature (Makarona *et al*., 2017). Parameters such as density, wood species, temperature, water absorption (*WA*), and moisture content (*MC*) significantly impact wood performance (Hamdan *et al*., 2000). Hence, it is crucial to ascertain these factors to ensure the reliability of wood applications. Several studies have explored the relationship between moisture content and mechanical properties of wood. Oloyede *et al*. (2000) observed that drying wood in an oven at 50 °C increases tensile strength, while using a microwave at maximum power decreases tensile properties. However, the moisture content and damping properties of specimens were proportional during dynamic loading conditions. Benzidane *et al*. (2018) conducted experimental analyses on date palm wood under quasi-static and cyclic loading (fatigue), revealing that cutting wood in different directions significantly affects mechanical stiffness and dissipative energy. Srivaro *et al*. (2015) investigated the mechanical properties of oil palm wood in sandwich structures and observed that the longitudinal fiber direction exhibits greater rigidity compared to the perpendicular direction.

When using wood materials for industrial structures or as reinforcement for polymers, thermal stability becomes a crucial parameter to consider. Thermogravimetric analysis (TGA) stands out as the most employed technique for assessing the thermal decomposition of polymeric materials. Typically, the degradation of wood under the influence of temperature involves four main reactions: evaporation of water content, decomposition of hemicelluloses followed by cellulose, and ultimately, a gradual decomposition of lignin over a broader temperature range (Merzoug *et al*., 2020; Maache *et al*., 2017; Kamperidou, 2021).

The cross-linking of lignin and the orientation of wood grain significantly influence the viscoelastic properties of wood materials (Olsson *et al*., 1992; Placet *et al*., 2007). The harmonic test, dynamic thermal mechanical analysis (DMTA), is one of the high precision techniques to evaluate the viscoelastic properties and the glass transition of materials. Recently, there has been a growing interest among researchers in measuring the viscoelastic properties of wood materials along their longitudinal direction using DMTA (Backman *et al*., 2001; Li *et al.,* 2021; Li *et al.,* 2023). However, there remains a limited amount of research concerning the dynamic mechanical behavior of wood along different grain directions.

The date palm tree (*Phoenix dactylifera*), belonging to the palm tree family, is primarily grown for human consumption. These trees are widespread across the Middle East and Northern Africa, with significant potential. Globally, there are over 100 million date palm trees (Abdal-hay *et al*., 2012), and each harvest season results in approximately 2 million tons of date palm wood waste. Despite its lightweight nature (with an average density of 210 kg/m³), from an economic and environmental perspective, the utilization of date palm wood waste is a promising project. There appears to be a lack of investigation into the use of petiole date palm wood as a core for sandwich composites (Benzidane *et al*., 2022). However, no reference is found in the literature regarding the thermophysical, chemical, and dynamic mechanical properties of the petiole date palm wood. Knowledge of these properties enables the development of more effective industrial processes and materials. Thus, this research aims to assess the physical, thermal, and dynamic mechanical properties of petiole date palm wood coming from two distinct environmental regions. The results found suggest that the PDPW is suitable as a core for sandwich composite panels.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Materials
2.1. Materijali

Around 18 million date palm trees planted in Algeria cover an area of 164 695 hectares. The petiole wood part is considered a natural unidirectional (UD)-composite structure. It has an Eiffel's tower feet shape, with a symmetry plane (Benzidane *et al.*, 2018). PDPW used in this study was harvested in two different environmental growing regions: the first region was the oasis area in the south of Algeria (Biskra), where the climate is hot and dry. The second region is a humid region close to the Mediterranean (Sidi- bel abbes). The average age of PDP wood samples ranged from 70 to 80 years.

2.2 Methods
2.2. Metode

2.2.1 Morphological analysis
2.2.1. Morfološka analiza

Microscopic examinations of the PDPW specimens and their interfacial (fiber/matrix) characteristics were conducted using a Tescan Vega3 scanning electron microscope (SEM) at 10 kV and 15 kV. The specimens were coated with gold and mounted on aluminum holders using double-sided electrically conducting carbon adhesive tabs before analysis (Horiyama *et al.*, 2023).

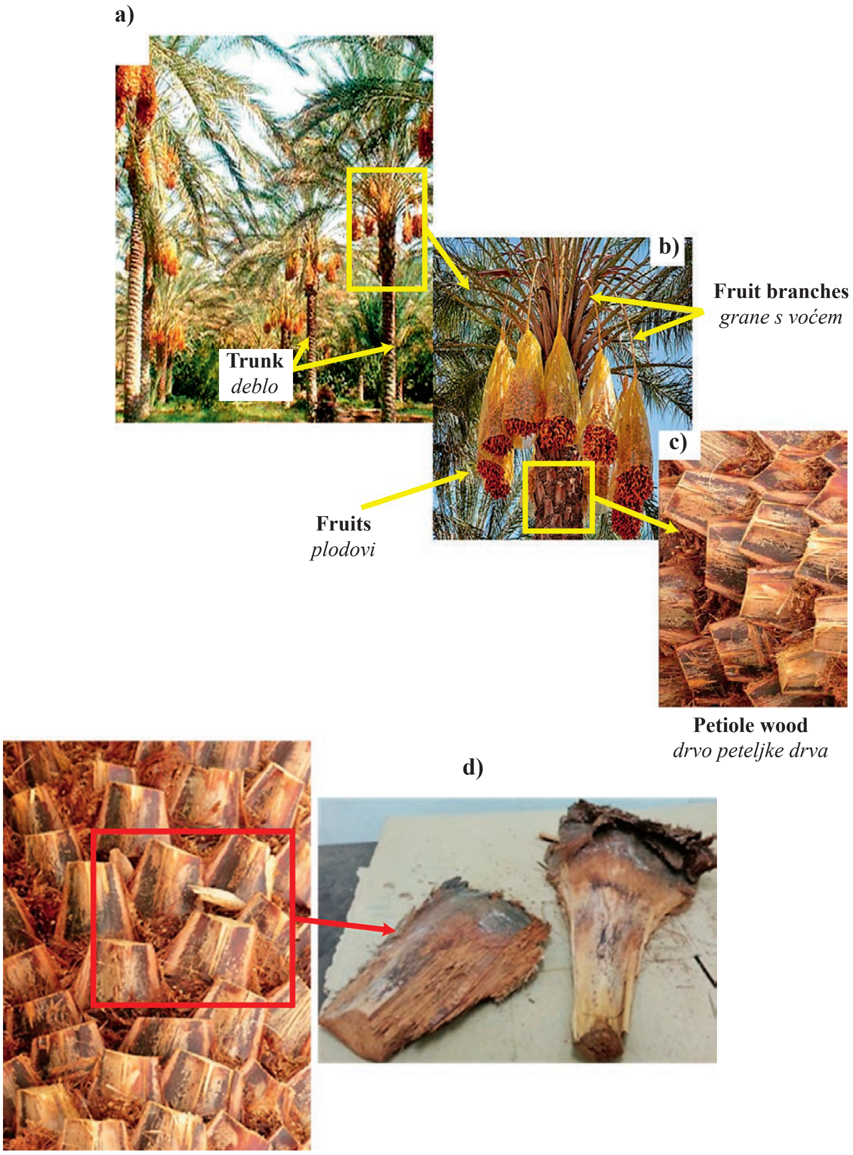


Figure 1 Date palm tree morphology: a) Date palm trees, b) Zoom on top parts, c) Petiole wood, d) Petiole wood collected
Slika 1. Morfologija stabla palme datulje: a) stablo palme datulje, b) povećani gornji dijelovi, c) drvo peteljke, d) skupljeno drvo peteljke

a) b) c)

Figure 2 Measurement steps of *MC*: a) Normalized specimens, b) Balance, c) Oven drying at 103 °C
Slika 2. Koraci pri mjerenju sadržaja vode: a) pripremljeni uzorci, b) vaganje, c) sušenje u sušioniku na 103 °C

2.2.2 Fourier transfer infrared spectroscopy (ATR-FTIR)

2.2.2. Infracrvena spektroskopija s Fourierovom transformacijom (ATR-FTIR)

The ATR-FTIR spectra of the PDPW (Northern / Southern) were carried out using a Thermo Scientific Nicolet iS10 type device with its own quantitative analysis software. The spectrum was obtained with a scanning speed of 32 acquisitions between 500 and 4000 cm^{-1} with a resolution of 2 cm^{-1}.

2.2.3 Physical characterization

2.2.3. Određivanje fizičkih svojstava

Moisture content

The European standard (EN 322) was followed. The volume (*V*) was assessed through the water displacement method and the weights were measured both in wet and dry conditions (dried at 103 ± 2 °C). PDPW specimens were created with dimension 20 mm × 20 mm × 20 mm. The moisture content of both PDPW regions (Northern/ Southern) was evaluated using the following formula.

$$MC = \frac{M_h - M_o}{M_h} \cdot 100 \tag{1}$$

Where, M_h (g) and M_o (g) represent the wet and dry masses, respectively.

Water absorption

The water uptake was carried out for PDPW specimens for both regions based on ASTM D1037-99 Standard. First, the wood was dried in oven at (103±2) °C until the weight was constant. Next, the weight was measured, and it was considered as the initial weight when using a numerical balance. Then, the specimens were immersed in distilled water (20 °C) and weighed at a specific time (24, 48 and 72h). The gain weight difference was used for the estimation of water uptake of the specimens. The formula below represents the water uptake equation used.

$$WA = \frac{W_t - W_i}{W_t} \cdot 100 \tag{2}$$

Where, W_t (g) represents the wet weight specimen and W_i (g) represents the oven-dried specimen at *t=0* time interval.

2.2.4 Thermogravimetric analysis (TGA/ DTA)

2.2.4. Termogravimetrijska analiza (TGA/DTA)

Thermogravimetric analysis was conducted using a Mettler Toledo TGA/DSC 3+ star system (located at the University 8 Mai 1954, Guelma Algeria) under a dynamic nitrogen atmosphere heating at room temperature from (20 °C) to 600 °C at a heating rate of 10 °C/ min. Specimens weighing approximately 6 mg were used, and their weight changes were measured throughout the process (Ma *et al*., 2018).

2.2.5 Dynamic Mechanical Thermal Analysis (DMTA)

2.2.5. Dinamička mehanička toplinska analiza (DMTA)

Dynamic Mechanical Thermal Analyzer (Hitachi, Japan) located in the "Thermal Analysis Laboratory" and "Nanotechnology Laboratory" at Istanbul University-Cerrahpasa was used to evaluate viscoelas-

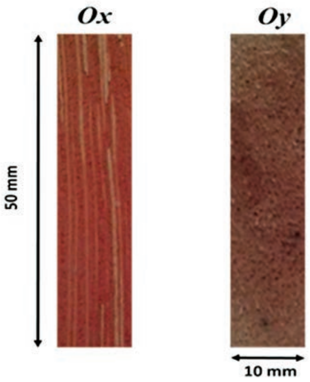


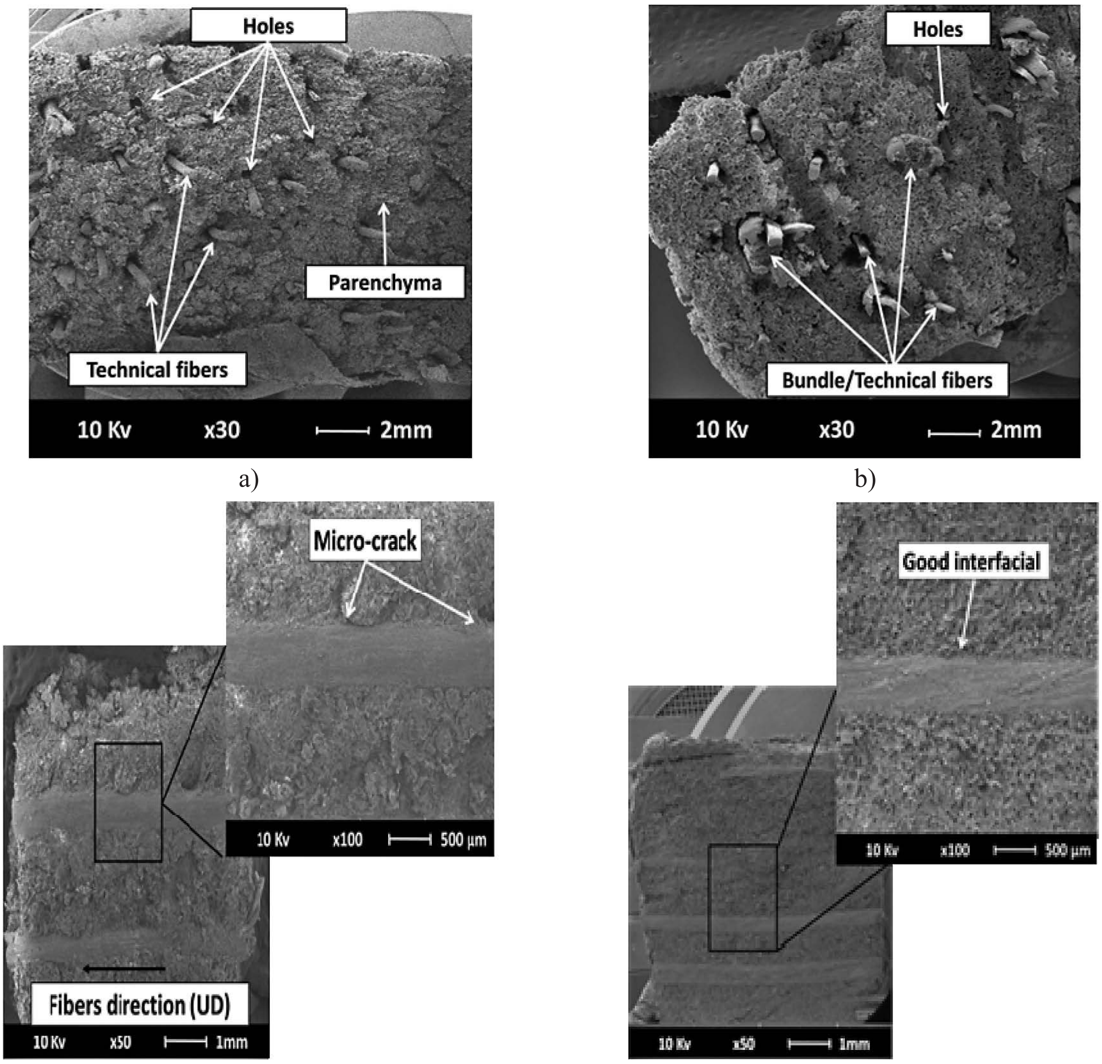
Figure 3 DMTA specimens
Slika 3. Uzorci za DMTA analizu

tic performance properties of PDPW (North/South) specimens. Dimensions of the samples were 50 mm by 10 mm by 2 mm in both fiber directions (Figure 3). All analyses were conducted in dual cantilever mode (3-point bending) at a frequency of 1 Hz, with temperatures ranging from 30 °C to 150 °C and an increased heating rate of 5 °C/min. Three runs were performed for each group (Toubia *et al*., 2019).

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

3.1 Morphological analysis
3.1. Morfološka analiza

Scanning electron micrographs of petiole date palm wood (north and south) are shown in Figure 4. The transversal surface images of PDPW (northern/southern) showed quasi-unidirectional natural composite materials: technical and bundle fibers reinforced tissue parenchyma matrices. The surface of PDPW from the northern region exhibits a higher presence of holes compared to that from the southern region. However, the amount of fibers in PDPW from the southern region is notably higher than that in PDPW from the northern region, which significantly affects physical properties, especially density. Figure 4 (c and d) depicts the longitudinal surface of PDP woods, clearly showing the direction of fibers and their interface with the matrix. It is evident that PDPWs exhibits better adhesion between its components compared to PDPWn from the northern region. Micro-cracks are observed in the inflated parenchyma matrix of PDPW from the northern region due to environmental factors, a phenomenon also reported by Benzidane *et al*. (2018).



a) b) c) d)

Figure 4 SEM images of PDPW from north and south in both grain directions: a) *Ox* direction PDPW North, b) *Ox* direction PDPW South, c) *Oy* direction PDPW North, d) *Oy* direction PDPW South
Slika 4. SEM fotografije PDPW-a iz sjeverne i južne regije u oba smjera vlakanaca: a) *Ox* smjer PDPW-a iz sjeverne regije, b) *Ox* smjer PDPW-a iz južne regije, c) *Oy* smjer PDPW-a iz sjeverne regije, d) *Oy* smjer PDPW-a iz južne regije

3.2 Chemical analysis (FTIR)
3.2. Kemijska analiza (FTIR)

The infrared spectroscopy was used to define the chemical composition of PDPW of north and south regions as shown in Figure5. Similar peaks of both spectra correspond to northern and southern PDPW without deviation (1034, 1240, 1595, 2848, 2925 and 3351), but with different intensities. The band groups presented on the spectra are associated with hemicelluloses, α-cellulose and lignin. For instance, the strong peak at 1034 cm^{-1} is associated with the O-H and C-O stretching mode of polysaccharides in cellulose groups (Romanzini *et al*., 2012). The band at 1240 cm^{-1} is related to C-O ether, phenol and ester vibration groups, this being attributed to the presence of waxes, lignin and xylan units in the wood surface (Esteves *et al*., 2013). Further, both peaks 2925 and 2848 cm^{-1} “C-H$_2$ and C-H bands” defined the acetyl group from hemicelluloses and cellulose components. A broad and intense peak was observed at 3351 cm^{-1} associated with hydroxyl groups O-H present in the cellulose, water, and lignin structure (Gonultas *et al*., 2018). The transmittance intensity of the southern PDPW is the lowest observed, which means a lower amount of hydroxyl groups and lignin in this wood region compared to the north region.

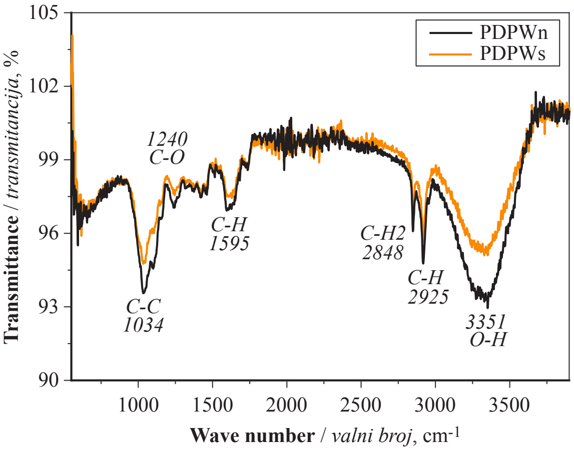


Figure 5 Fourier transform infrared spectroscopy (FTIR) spectrogram of PDPW (North/South)
Slika 5. Spektar PDPW-a (sjever/jug) dobiven infracrvenom spektroskopijom s Fourierovom transformacijom (FTIR)

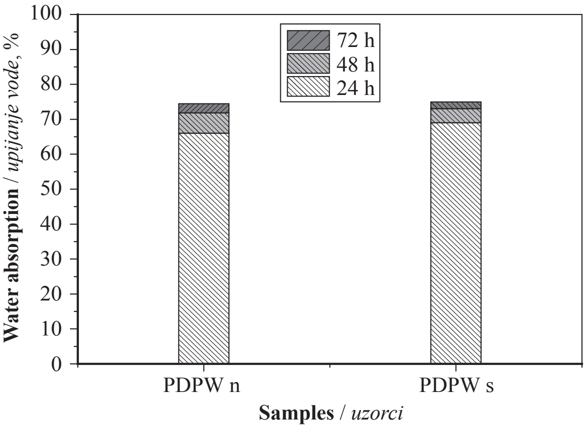


Figure 6 Water absorption evaluation of PDPW specimens
Slika 6. Rezultati upijanja vode PDPW uzoraka

3.3 Physical analysis
3.3. Analiza fizičkih svojstava

3.3.1 Water absorption
3.3.1. Upijanje vode

Figure 6 presents the water absorption patterns for 24, 48, and 72 hours of PDPWn and PDPWs. The PDPWs shows a maximum water absorption rate of 74 %, which is 3 % higher than that of PDPWn. It could be explained by the filling up of capillaries, diffusion phenomena, and the cellular wall of wood. The high-water absorption phase was observed after 24h. The PDPWs absorbed 69.02 % of water, which was higher by 13 % than PDPWn due to the high fiber ratio and the presence of fewer holes. The free movement of water due to large holes and porosities in PDPWn resulted in a slow water uptake. Next, the immersion times of 48 hours and 72 hours showed a small amount of water absorption, where the PDPWs saw a slight increase of 5 % and 2 %, respectively, while PDPWn increased by 16 % and 4 %. The process of saturation occurs at a slow pace until the wood grain is completely saturated, including its fibers.

3.3.2 Moisture content
3.3.2. Sadržaj vode

Moisture content plays a crucial role in the thermal and mechanical properties of polymeric materials, especially biomass. Figure 7 presents the evaluation of PDPW moisture content of both regions Northern and Southern dried at 102 °C for 24 h. Similar curves are shown with different values. After one hour of drying, a high quantity of water was evaporated from both PDPW. In particular, the PDPWn (lost around 12 % of its initial weight) due the concentration of water in this wood region and the easier movement of water in large

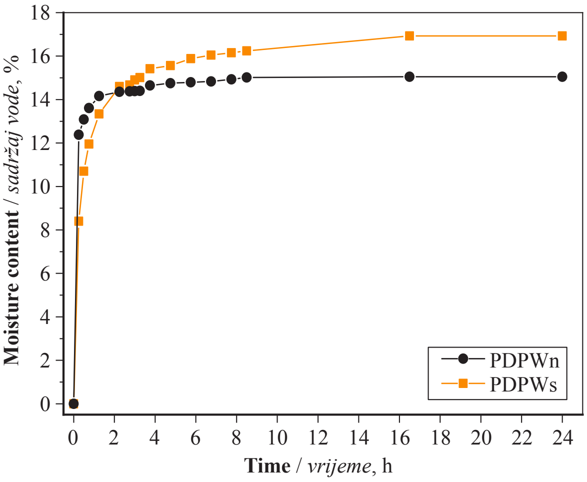


Figure 7 Moisture content evaluation of PDPWood specimens
Slika 7. Evaluacija sadržaja vode PDPW uzoraka

holes of PDPWn. In comparison to PDPWs, the smaller hole dimensions, good adhesion between fibers and matrix, and higher fiber ratio result in less water evaporation. Subsequently, the moisture content gradually stabilizes for PDPW of both regions. The moisture content of PDPWs was around 17 %, which is 3 % higher than that of PDPWn. The morphologies and environmental factors of PDPW significantly influence its moisture content properties.

3.4 TGA/DTG
3.4. TGA/DTG

In order to study the thermal stability behavior of PDPW (Northern/ Southern), all specimens were subjected to heating in the Mettler Toledo TGA/DSC 3+ device. Figure 8 (a, b) displays the TGA curve and its DTG derivative obtained for the PDPW. Despite showing a similar trend, there were variations in weight loss percentages, with PDPWn and PDPWs losing 89.97 % and 92.43 % of their initial weight, respectively. During the initial heating of specimens, the water evaporation started at around 40 °C and was completed at roughly 128 °C. The same was observed in the case of balsa and oak wood (Tranvan *et al.*, 2017; Ceylan *et al.*, 2014). The first decomposition stage was attributed to water evaporation, accounting for 12.78 % and 11 % of weight loss for PDPW from the Northern and Southern regions, respectively. Subsequently, an active decomposition stage was evident, comprising three domains (Figure 8): 1) hemicellulose decomposition, 2) cellulose decomposition, and 3) lignin decomposition occurring between 405 and 500 °C (Todaro *et al.*, 2017). The decompositions of hemicelluloses and cellulose presented the major weight loss that occurred in the temperature range of 175-405 °C, where PDPWn gradually lost 54.87 % of its weight. Regarding the PDPWs, a dramatic weight loss of 61.57 % was noted. Ceylan *et al.* (2014) explained that decomposition in the temperature range of 180-350 °C is attributed to the depolymerization of hemicelluloses and the random cleavage of glycosidic linkages of cellulose. In addition, passive decomposition stages displayed quasi-constant values associated with lignin residues or char, accounting for 32.35 % PDPWn and 27.43 % of PDPWs. The products of cellulose degradation contain non-degraded fillers and carbon residues (Maache *et al.*, 2017). The petiole date palm wood exhibited a similar thermal stability compared to that of balsa wood (Hellmeister *et al.*, 2021; Marwanto *et al.*, 2021).

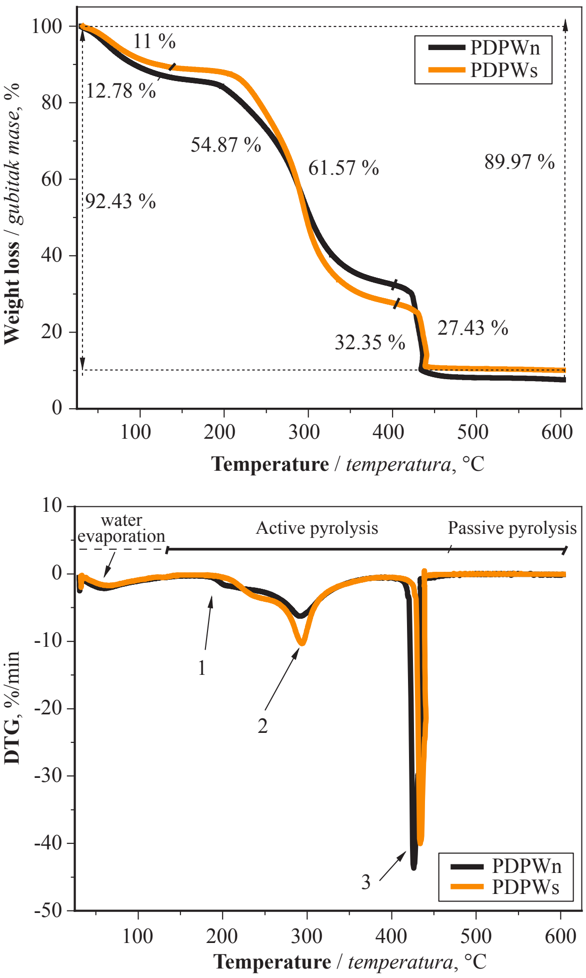


Figure 8 Thermogravimetric analysis test results of PDPW (north/ south)

Slika 8. Rezultati termogravimetrijske analize PDPW-a (sjever/jug)

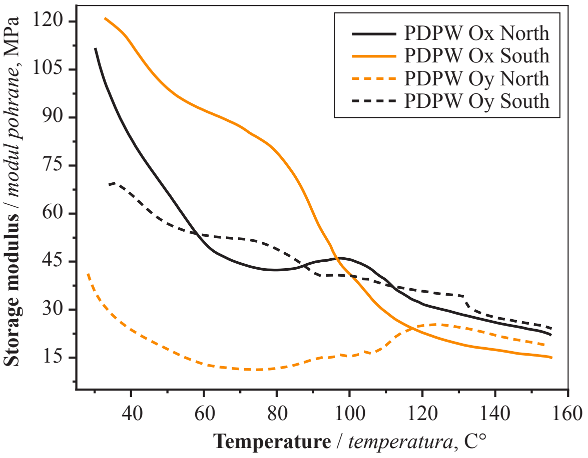


Figure 9 Storage modulus of PDPW (north/south) for both directions (*Ox* and *Oy*)

Slika 9. Modul pohrane PDPW-a (sjever/jug) za oba smjera (*Ox* i *Oy*)

3.5 Dynamic Mechanical Thermal Analysis
3.5. Dinamička mehanička toplinska analiza

Stiffness, damping and glass transition of PDP Wood specimens were studied using dynamic mechanical thermal analysis (DMTA). The grain direction and environment region of specimens were considered as relevant parameters for the investigation. The results are plotted as storage modulus (*E*'), loss modulus (*E*") and loss factor (tan δ).

3.5.1 Storage modulus (E')
3.5.1. Modul pohrane (*E*')

The variation of storage modulus as the function of temperature for PDP wood specimens at the frequency of 1 Hz is given in Figure 9. The results have shown similar curves with different values for the same groups (regions). Values of the specimens of perpendicular fiber direction (*Ox*) were significantly higher than those of the corresponding parallel specimens, where the higher value of 120 MPa was noted for southern wood. Fiber rate, fiber performance and its length played a significant role for this property. In the case of perpendicular fiber direction (*Oy*), *E*' of PDPWs was considerably more important than that of PDPWn (higher by 40 %). This could be due to holes effect, poor interfacial adhesion (fiber/ matrix) and stat of parenchyma. The storage modulus of all specimens was unproportioned to the temperature, especially PDPWn wood (both directions), where its *E*' decreased dramatically until 70 °C. This diminution is due to the micro-Brownian movement of polymer chains as the polymer approaches the glass transition (Ornaghi *et al.*, 2011). Plus, due to the augmentation of fully separated fibers, fibers are not susceptible to bonding again. It is known that PDPWs undergo a gradual diminution of E' before glass transition due to less holes, high fiber rate and strong interfacial adhesion (Sreekala *et al.*, 2005). Below T_g, northern wood *E*' was increased slightly due to the final evaporation of water from fibers. However, the southern wood continued to diminish *E*' because the evaporation of water was the same for both fiber and parenchyma matrix.

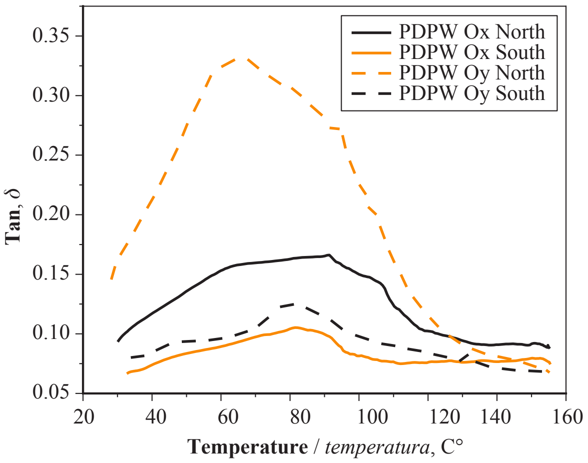


Figure 10 Loss factor vs temperature of PDPW (north/ south) for both directions (*Ox* and *Oy*)
Slika 10. Faktor gubitka s obzirom na temperaturu PDPW-a (sjever/jug) za oba smjera (*Ox* i *Oy*)

Table 1 Glass transition temperature values at 1 Hz and damping of PDPW
Tablica 1. Vrijednosti temperature stakliša PDPW-a pri 1 Hz i prigušenje PDPW-a

PDPW	**Fiber direction** *Smjer vlakana*	**tan δ**	**Temperature, °C** *Temperatura, °C*
Northern *sjeverna regija*	*Ox*	0.15	67.7
	Oy	0.33	65.43
Southern *južna regija*	*Ox*	0.10	81.44
	Oy	0.12	79.55

3.5.2 Loss factor (tan δ)
3.5.2. Faktor gubitka (tan δ)

Loss factor variation of northern and southern PDPW for two different fiber directions as the function of temperature is shown in Figure 10. Tan δ is explained by the report of E''/E' and it can be called damping. It was very high in the parallel direction of PDPWn (0.33), due to the diminution of *E*' when the temperature increased. In addition, lower fiber rate and the presence of porosities led to free mobility of polymer molecules. Besides, it was observed that PDPWn in the perpendicular fibers was damper (higher by 20 %) than PDPWs in parallel direction even though its storage modulus was less than that of PDPWn (*Ox*). Furthermore, no considerable change between the fiber direction properties of southern PDPWood was observed. The glass transition of PDPW was similar in the case of the same regions (Table 1). In other words, the T_g of PDPW was dependent on the environment more than on the fiber direction.

4 CONCLUSIONS
4. ZAKLJUČAK

Due to their lightweight nature and mechanical performance, foams, honeycomb structures, and balsa wood are widely used in mechanical technology as core materials for sandwich structures. However, their prices remain one of the major disadvantages. In this study, an experimental investigation on the physical, thermal and dissipative properties of new bio-mass potential, PDPW "Petiole Date Palm Wood", from two different Algerian regions was conducted. The Northern region (Sidi Bel Abbes), located near the Mediterranean Sea, is characterized by a humid and rainy climate, while the Southern region (Biskra) is a semi-arid area with hot and dry weather. The results of this study can be summarized as follows:

SEM images revealed that the morphology of PDPW resembles unidirectional composite materials. The wood from the southern region exhibited a higher fiber content (technical and bundle fibers) compared to that from the northern region. Additionally, the presence of voids was significant in the wood from the northern region.

The chemical composition of both PDPW was similar but with varying intensity.

There was a small difference (3 %) in moisture content between PDPW from both regions, despite the different climate conditions. The good interfacial adhesion and high fiber content of PDPW from the southern region led to gradual water evaporation compared to that from the northern region. For the latter, the presence of porosities facilitated easier water evaporation. Additionally, both PDPW types exhibited significant initial water absorption, especially in the case of PDPWs, due to their hydrophilic nature. Subsequently, fiber water absorption occurred.

The dissipative test was carried out on PDPW specimens for two different fiber directions (*Ox* and *Oy*) using DMTA. Due to the morphology of PDPWs (good interfacial adhesion, less voids and high fiber rate) and fiber properties, the storage modulus in perpendicular direction (*Ox*) was higher than in parallel (*Oy*) direction by 41 % for PDPWs and by 65 % for PDPWn. From a loss factor point of view, the fiber directions (perpendicular and parallel) were more affected by damping than by environmental regions.

The results of this study suggest that PDPW has the potential to be used as a sandwich core material in automotive, furniture, and boat interior structures.

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