

Eser Sözen<sup>\*1,2</sup>, Deniz Aydemir<sup>1</sup>, Gökhan Gündüz<sup>3</sup>, Sezgin Koray Gülsoy<sup>1</sup>

# Effects of DES and Chlorite Delignification Methods on Properties of Transparent Wood Materials

## Učinci dubokih eutektnih otapala i metoda delignifikacije kloritom na svojstva prozirnih drvenih materijala

### ORIGINAL SCIENTIFIC PAPER

#### Izvorni znanstveni rad

Received – prispjelo: 21. 11. 2023.

Accepted – prihvaćeno: 16. 4. 2024.

UDK: 630\*82

<https://doi.org/10.5552/drvind.2024.0163>

© 2024 by the author(s).

Licensee University of Zagreb Faculty of Forestry and Wood Technology.

This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license.

**ABSTRACT** • Wood has many uses as raw material and after being processed. Since the use of wood as raw material is limited, the interaction of physical and chemical processes with technological developments has led to new products and new areas of use. Transparent wood production, for example, has emerged to provide maximum efficiency from heat and light. In this study, sodium chlorite and deep eutectic solvents (DES) obtained from choline chloride and lactic acid were used. In the production of transparent wood, 1- and 2-mm thick beech wood veneers (*Fagus orientalis* L.) and two-component transparent epoxy resin were used as filler. As a result of delignification processes, the highest delignification rate was obtained with 59 % in wood samples treated with NaClO<sub>2</sub>. Transparent wood production was obtained from the samples where the most delignification took place. The adhesion of the epoxy used in the production of transparent wood in the wood cells was determined by SEM analysis. The tensile strength of the transparent wood samples increased by 31 % compared with the control samples. The study results contribute to the literature on the efficiency of different delignification methods.

**KEYWORDS:** deep eutectic solvent; delignification; transparent wood; beech wood

**SAŽETAK** • Drvo ima široku primjenu kao sirovina i nakon različitih procesa prerade. Budući da je uporaba drva kao sirovine ograničena, interakcija fizikalnih i kemijskih procesa s tehnološkim je razvojem dovela do pojave novih proizvoda i područja uporabe. Na primjer, proizvodnja prozirnog drva pojavila se kako bi se osigurala maksimalna učinkovitost topline i svjetlosti. U ovom su istraživanju korišteni natrijev klorit i duboka eutektna otapala (DES) dobivena od kolin klorida i mliječne kiseline. U izradi prozirnog drva korišteni su bukovi furniri (*Fagus orientalis* L.) debljine 1 i 2 mm i dvokomponentna prozirna epoksidna smola kao punilo. U procesu delignifikacije najveća je stopa delignifikacije od 59 % postignuta na uzorcima drva tretiranima s NaClO<sub>2</sub>, te su ti uzorci rabljeni za proizvodnju prozirnog drva. Adhezija epoksidne smole u stanicama drva, koja je upotrijebljena

\* Corresponding author

<sup>1</sup> Authors are researchers at Bartın University, Forest Industrial Engineering, Faculty of Forestry, Bartın, Türkiye. <https://orcid.org/0000-0003-4798-7124>; <https://orcid.org/0000-0002-7484-2126>; <https://orcid.org/0000-0002-3079-9015>

<sup>2</sup> Author is a researcher at Bartın University, Forest Products Research & Application Center, Bartın, Türkiye. <https://orcid.org/0000-0003-4798-7124>

<sup>3</sup> Author is a researcher at Iskenderun Technical University, Faculty of Engineering and Natural Sciences, Industrial Engineering, 31200, Iskenderun, Türkiye. <https://orcid.org/0000-0002-2602-2211>

u proizvodnji prozirnog drva, određena je SEM analizom. Vlačna čvrstoća prozirnih uzoraka drva povećala se za 31 % u usporedbi s kontrolnim uzorcima. Rezultati istraživanja pridonose literaturnim podatcima o učinkovitosti različitih metoda delignifikacije.

**KLJUČNE RIJEČI:** duboka eutektična otapala; delignifikacija; prozirno drvo; bukovina

## 1 INTRODUCTION

### 1. UVOD

Wood structures vary according to wood type (softwood and hardwood) and growing zones. Softwoods generally grow fast, and their cells are larger than hardwoods, whereas hardwoods generally grow slowly, and their cells are smaller; however, hardwood densities are higher than those of softwoods (Russel and Richard, 1984).

The lumens and cell wall, which are formed by the porous structure of wood, have an important place in the reflection of light. Lignin, which is one of the elements composing wood, has a complex structure and a brown color. Hemicellulose and cellulose, the other essential elements of wood, have a white color (Fink, 1992; Zhu *et al.*, 2014). Therefore, research on the optic properties cannot be conducted in the current form of wood, and lignin must be removed from inside the cell wall. The status requires pretreatments and chemical processes. After lignin is removed via several methods from the wood structure, cellulose and hemicellulose become individual forms. Li *et al.* (2016) treated wood with poly (methyl methacrylate) after delignification of wood under vacuum, obtaining 85 % transparent wood. Zhu *et al.* (2016a) modified a NaOH/Na<sub>2</sub>SO<sub>3</sub> solution to remove the lignin in the wood, and the wood without lignin was impregnated with polyvinylpyrrolidone (PVP) to obtain high transparency. The results showed wood obtained with high transparency of 90 %. Zhu *et al.* (2016b) modified the wood material with NaOH/Na<sub>2</sub>SO<sub>3</sub>, and later the modified wood was treated with hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>). The delignified wood was impregnated with epoxy under vacuum, and the obtained wood was found to have high transparency in the range of 75 – 90 %.

According to the literature review, chemicals generally provide high transparency of wood; however, the chemicals are petroleum-based materials and can cause environmental pollution. With increased interest in the environment, the use of chemicals is often decreased in many applications due to legal regulations. For the delignification of wood materials, green chemicals such as deep eutectic solvents (DES) have started to be used in the same applications. Deep eutectic solvents are a new class of solvents that have gained significant attention in recent years. Their unique properties make them attractive for a wide range of applications in fields such as chemistry, materials science, and

biotechnology. These solvents are formed from the complexation of a hydrogen bond donor (such as a quaternary ammonium salt) and a hydrogen bond acceptor (such as a metal salt or organic compound). They exhibit a eutectic behavior, which means they have a lower melting point compared with the individual components. This property allows them to be used as low-temperature solvents, making them suitable for processes that require low temperature conditions. Some of the advantages of deep eutectic solvents include their biodegradability, non-toxicity, and low volatility (El Achkar *et al.*, 2021).

Traditional delignification methods have been used for many years in various industries, such as pulp and paper production and biofuel production. These methods involve the use of chemicals, such as sodium hydroxide, to break down and remove lignin from cellulose fibers. This process is known as “delignification” and is essential for separating lignin from cellulose to obtain high-quality cellulose fibers for different applications, such as papermaking or producing biofuels (Sivasubramanian *et al.*, 2008).

Previous studies showed that DES can provide a good delignification in wood materials as compared with conventional chemicals and it generally does not affect cellulosic materials (Francisco *et al.*, 2012; Lynam *et al.*, 2017). The extraction mechanism of lignin from wood with DES occurs with the breaking of phenyl propane units in a lignin structure as occurring in the conventional chemicals (Alvarez-Vasco *et al.*, 2016).

The building sector (electrical devices, heating, air conditioning, and hot or cold-water vb.) generally accounts for the highest energy consumption in the world (about 30 – 40 %) (Rotzetter *et al.*, 2012). Therefore, world demand for sun energy, i.e., a clean, low-cost, and sustainable resource, has been increased to meet the increase of energy consumption amounts in accordance with the economic development. The demands for artificial light sources can be reduced via natural light sources with transparent buildings (He *et al.*, 2014). Further, transparent wood can be used as transparent construction materials, and sun battery staffs due to its optic properties and high mechanical strength (Yaddanapudi *et al.*, 2017). The aims and objectives of this study are to determine the results of delignification with DES, i.e., to determine transparency by obtaining transparent wood as well as to determine the morphological and mechanical properties of transparent wood.

## 2 MATERIALS AND METHODS

### 2. MATERIJALI I METODE

#### 2.1 Materials

##### 2.1.1. Materijali

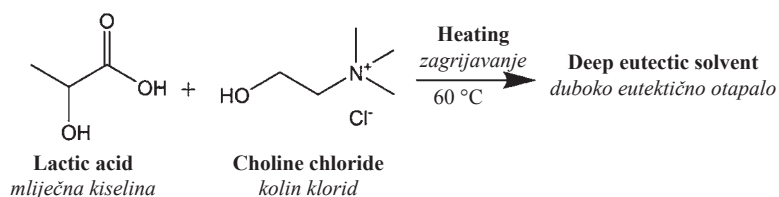
In the study, beech wood veneers with 1- and 2-mm thickness were used. The veneers have dimensions of 5 x 5 cm and moisture content of 12 %. The hybrid methods, including DES, sodium chlorite ( $\text{NaClO}_2$ ), and their mixes (50/50 wt.), were used for the delignification of the veneers. Choline chlorite ( $\text{ChCl}$ ) with a molecular weight of 139.62 g/mol and lactic acid (LA) with a molecular weight of 90.8 g/mol were used to obtain the DES. After the delignification with 1.7 mol/L  $\text{NaClO}_2$ , 30 %  $\text{H}_2\text{O}_2$  was used to provide higher transparency in the wood. The variations and material properties for the delignification of the wood are given in Table 1.

#### 2.2 Preparation of DES

##### 2.2.1. Priprema dubokih eutektičnih otapala

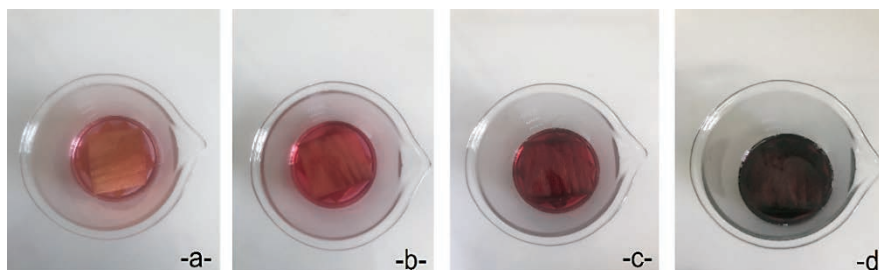
DES was prepared by mixing  $\text{ChCl}$  with LA at 1:10 (mol: mol). The blends were made at a temperature of 60 °C under a magnetic mixer until the blends were homogenous and the final blends were transparent. The production mechanism of the DES is given in Figure 1.

To determine the optimum DES activity, the veneers were treated with ten-fold DES at 120 °C for 1, 2, 3, and 4 h.; images of the resulting solvents after the treatment of the veneers are given in Figure 2. In treatments of more than 4 h (5-6 h), it was determined that the dissolved lignin was reabsorbed by the wood. In this case, the main purpose of the delignification process is lost.



**Figure 1** Production of lactic acid-based DES (Li *et al.*, 2019)

**Slika 1.** Proizvodnja dubokih eutektičnih otapala na bazi mliječne kiseline (Li *et al.*, 2019.)



**Figure 2** Resulting solvents after treatment of veneers (a=1 hour, b=2 hours, c=3 hours, d=4 hours)

**Slika 2.** Dobivena otapala nakon tretiranja furnira (a=1 sat, b=2 sata, c=3 sata, d=4 sata)

### 2.3 Delignification of wood materials

#### 2.3.1. Delignifikacija drvnih materijala

In this study, the delignification process with DES was conducted on the four wood samples having a moisture content of 12 %. The ratio of sample to solvent was 1:10 (w/w). The delignification was applied to the wood samples at 120 °C for 4 h; after the treatment, the samples were dried with distilled water. The washing process was continued for 5 min under running water. The washed samples were kept in distilled water for 12 h. The delignification process with sodium chlorite ( $\text{NaClO}_2$ ) is a commonly used method in the pulp and paper industry. This study followed the same method, and the samples with the dimensions of 5 mm x 5 mm were put into a beaker containing 1.7 mol/L  $\text{NaClO}_2$  with the ten-fold weight of each wood sample. The beaker was kept in the water bath at the temperature of 80 °C under an air cabinet, and 0.5 mL acetic acid ( $\text{CH}_3\text{COOH}$ ) and 1.5 g  $\text{NaClO}_2$  were added to the beaker for each 160 mL  $\text{NaClO}_2$  solvent per hour (total 6 h). After the delignification, the whitened samples were cleaned for 6 h with distilled water. After the delignification of the samples, the whitening process with 30 % hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) was applied to the samples in the water bath at 80 °C for 30 min. Four types of the delignification process were applied, including DES,  $\text{NaClO}_2$ ,  $\text{NaClO}_2+\text{H}_2\text{O}_2$ ,  $\text{DES}+\text{NaClO}_2+\text{H}_2\text{O}_2$ . After the delignification, all samples were put in a beaker containing ethyl alcohol for 12 h; further, some of the delignified samples were dried and then ground for determining residue lignin. Table 1 shows the delignification type and formulations of the wood samples.

**Table 1** Delignification type and formulations**Tablica 1.** Vrste i formulacije delignifikacije

Variations / Varijacije	Delignification process / Proces delignifikacije	
DES	1 <sup>st</sup> stage	1:10 DES at 120 °C for 4 h
NaClO <sub>2</sub>	1 <sup>st</sup> stage	1.7 mol/L NaClO <sub>2</sub> at 80 °C for 6 h
	2 <sup>nd</sup> stage	30% H <sub>2</sub> O <sub>2</sub> at 80 °C for 2 h
DES+NaClO <sub>2</sub>	1 <sup>st</sup> stage	1:10 DES at 120 °C for 4 h
	2 <sup>nd</sup> stage	1.7 mol/L NaClO <sub>2</sub> at 80 °C for 6 h
	3 <sup>rd</sup> stage	30% H <sub>2</sub> O <sub>2</sub> at 80 °C for 2 h
Control / Kontrola	No action taken / nije poduzet nikakav postupak	

## 2.4 Determination of residue lignin percentages

### 2.4. Određivanje postotnog sadržaja lignina nakon delignifikacije

The Klason method, according to the TAPPI T222 om-66 standard, was used to determine the amount of residue lignin in wood veneers with 1-mm thickness. Lignin amount was calculated according to the following Eq. 1:

$$\text{Lignin amount (\%)} = \frac{A}{W} \cdot 100 \quad (1)$$

where  $A$  is residue lignin amount (g), and  $W$  is oven-dried wood weight (g).

## 2.5 Transparent wood production

### 2.5. Proizvodnja prozirnog drva

Wood veneers with dimensions of 5 cm × 5 cm were impregnated via immersion into epoxy resin with two components (2:1 wt. %, resin/hardener) in a silicon plate under a vacuum cabinet, after the delignification and H<sub>2</sub>O<sub>2</sub> treatment. A stainless-steel web was covered on the top of the silicon plate during the immersion to prevent the movement of the wood veneers in the vacuum cabinet. The vacuum process was applied at 0.1 mbar for 40 min. After the vacuum process, the immersed wood veneers were put inside two silicon mats for 24 h to obtain a smooth surface.

## 2.6 Methods

### 2.6. Metode

#### 2.6.1 Scanning electron microscope (SEM) analysis

##### 2.6.1. Analiza skenirajućim elektronskim mikroskopom (SEM)

Morphology and fractured sections of the transparent woods were examined with a Tescan MAIA3 XMU-SEM at various magnitudes. For SEM analysis, sections were taken from the samples at equilibrium moisture (12 %). The samples were coated with a mixture of palladium/gold particles to enhance the flow of electrons.

#### 2.6.2 Fourier transform infrared (FTIR) analysis

##### 2.6.2. Analiza Fourierovom infracrvenom spektroskopijom (FTIR)

The transparent wood was scanned with a Shimadzu IRAffinity-1 FTIR (Kyoto, Japan). The spectra

of the samples were recorded by 16 scans from 800 to 3800 cm<sup>-1</sup> with a resolution of 4 cm<sup>-1</sup>. This analysis was carried out to determine whether the treatments caused changes in the chemical structure of the samples.

#### 2.6.3 X-Ray diffraction (XRD) analysis

##### 2.6.3. Analiza difrakcijom X-zraka (XRD)

The XRD pattern was obtained with a Philips PANalytical Empyrean X-ray diffractometer using Ni-filtered Cu K $\alpha$  (1.540562 Å) radiation at scales from 10° to 80° 2  $\theta$  range, and the crystallinity was determined using the Segal and curve-fitting methods. XRD analysis was used to determine the differences in the crystal structure of the samples.

#### 2.6.4 Tensile strength tests

##### 2.6.4. Ispitivanja vlačne čvrstoće

A U-test mechanical tester was used to characterize the mechanical properties of the transparent wood. The tensile test was conducted according to ASTM D 638-03 Type I at 5 mm/min of test speed. Elongation during the test was measured with an extensometer.

## 3 RESULTS AND DISCUSSION

### 3. REZULTATI I RASPRAVA

#### 3.1 Delignification results

##### 3.1. Rezultati delignifikacije

The delignification process of lignin from structural components of the wood was conducted prior to transparent wood production. In this study, various delignification methods including DES, NaClO<sub>2</sub>, and DES+ NaClO<sub>2</sub>, were used. Delignification processes determined by the ultrasound method according to TAPPI T122-om-66 were compared with control samples. Table 2 shows 19.5 % of lignin for the control beech wood in the delignification with the Klason method. The delignification with DES was provided 13 % of lignin in beech wood. The minimum amount of lignin of 8 % wt. was found in the delignification with NaClO<sub>2</sub>. With the chlorite method, 59 % of the existing lignin (19.5 %) in the control sample was removed, leaving 8 % lignin. After combining DES and NaClO<sub>2</sub>, the amount of lignin was found to increase from 8 % to 12 %. On the other hand, the use of DES in combina-



**Table 2** Delignification rate and lignin amount obtained with different methods**Tablica 2.** Stopa delignifikacije i količina lignina dobiveni različitim metodama

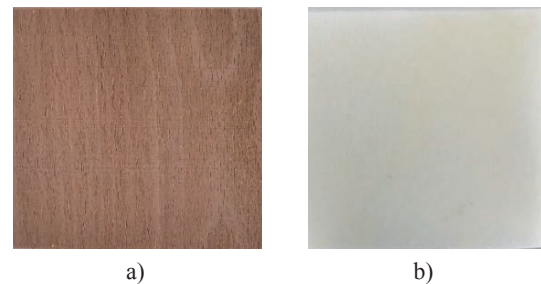
Methods / Metode	Amount of lignin before delignification, wt. % Količina lignina prije delignifikacije, tež. %	Amount of lignin after delignification, wt. % Količina lignina nakon delignifikacije, tež. %	Delignification rate, % Stupanj delignifikacije, %
Control			-
DES (in veneers)	19.5	13	33.3
NaClO <sub>2</sub> (in veneers)		8	59.0
DES+NaClO <sub>2</sub> (in veneers)		12	38.5

tion with sodium chloride did not cause a significant change in the delignification process. As a result, it has been seen that using delignification methods alone will be more efficient and economical.

In a study, Yaşar and Tanrıverdi (2008) grounded the beech wood and researched the lignin amount in the wood after delignification conducted with DES; the lignin amount was determined as 26.1 % wt. In this study, the lignin amount was lower due to the wood veneers, according to Yaşar and Tanrıverdi (2008), because of lower contact surface of the solvents. The reason might be that the veneers were produced with the pretreatments of hot water or by water steaming, and the methods might have been affected by the lignin percentages in wood (Cristescu and Karlsson, 2013). Yang *et al.* (2020) studied with DES the effect of ChCl/oxalic acid (1:1) on the delignification of balsa wood, and the delignification rate was determined as 60 %; further, Hou *et al.* (2018) found the delignification rate as 84.7 % via similar DES (2:1) with ChCl at higher percentages. In this study, the delignification rate was generally lower than found in the literature, i.e., the studies in the literature review were generally conducted on grounded wood; therefore, the contact surface and efficiency of the solvents were higher. However, this study was conducted on wood veneers, and both the contact surface and efficiency of the DES were lower than them.

Therefore, in this study, the lignin amount was lower than that of the studies in the literature review. Although the minimum lignin amount was obtained with the DES delignification, the maximum delignification rate was determined as 59 % for the NaClO<sub>2</sub>. In another study, Yang *et al.* (2020) studied the lignin amount and the delignification rate of balsa wood with DES; the delignification rate was determined to be 77 %. The rate was found to change according to the wood anatomical structure and solvent contact surface. However, the data obtained and the literature have differences, as the solvents were applied to the coating in the study; therefore, the solvent could not penetrate into the inner parts of the wood sufficiently.

On the other hand, in the literature studies where high delignification was achieved, solvents were used after grinding the wood, which resulted in a more effi-

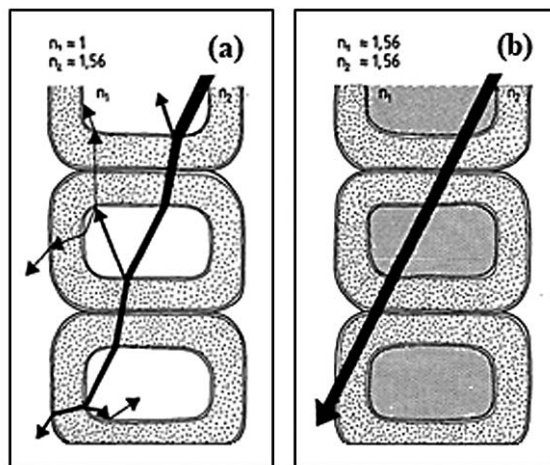


**Figure 3** 1-mm thick wood veneers delignified with NaClO<sub>2</sub>. a) veneers before and b) veneers after delignification  
**Slika 3.** Furniri drva debljine 1 mm delignificirani s NaClO<sub>2</sub>: a) furniri prije delignifikacije, b) furniri nakon delignifikacije

cient delignification process. Figure 3 shows 1-mm thick wood veneers delignified with NaClO<sub>2</sub> - (a) veneers before and (b) veneers after delignification. Generally, appearances of the samples after all delignification processes were similar to each other.

The delignification with DES+NaClO<sub>2</sub> was provided at 12 % of the lignin amount and 38.5 % of the delignification rate. As a result, DES efficiency on the delignification of the veneers was higher than the efficiency of NaClO<sub>2</sub>, and it can be said that only DES treatment is a good method for the lignin delignification. As shown in Figure 2, the treatment time was an important factor in the delignification with DES, and it can be said that the best delignification parameters were determined at 120 °C for 4 h due to the black color of DES after the delignification. After the delignification with DES, the veneers were softened, and the dimensional integrity of the veneers was broken down. This status might be caused by the removal of lignin, as a filling material in the wood.

In another study, Fu *et al.* (2018) provided a high lignin-removing rate during the delignification with DES. Weakening of bonds between wood cells and then loosening of the cytoskeleton of veneers were found to occur with too high lignin delignification, which decreased the mechanical properties with the dimensional integrity of the veneers. Consequently, it can be said that treatment time and temperatures were important factors in the delignification rate. Although the minimum lignin amount was determined with the delignification with NaClO<sub>2</sub>, the best delignification rate was obtained with the delignification with NaClO<sub>2</sub>; therefore, this study



**Figure 4** Light reflection and direction in (a) empty lumen and (b) lumen filled with epoxy (Fink, 1992)

**Slika 4.** Refleksija i smjer svjetlosti u praznom lumenu (a) i u lumenu ispunjenom epoksidom (b) (Fink, 1992.)

was conducted on the properties of the 1- and 2-mm samples delignified with  $\text{NaClO}_2$ .

### 3.2 Transparency results

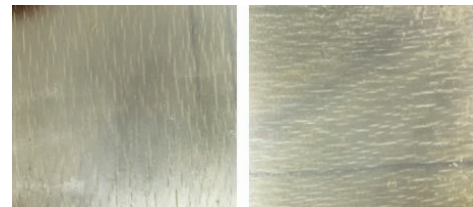
#### 3.2. Rezultati prozirnosti

After filling the wood cells with epoxy, the appearance of the delignified wood changed from white to transparent; however, the transparency of the samples may generally not be enough due to low filling rate with the epoxy as given in Figure 4a; further, the good transparency can be provided with good filling rate in the wood cells, as seen in Figure 4b. This status might be caused due to the areas with different densities and the different light reflections in the wood inside (Fink, 1992).

Refractive index is the ratio of the speed of light in a vacuum to its speed in a given medium. The speed of light in a medium depends on the properties of the medium. The reflective material (epoxy) and lumen refractive index ( $n$ ) play an important role in the light reflection. Increasing the difference between the lumen refractive index ( $n_1 = 1$ ) and the wood cell refractive index ( $n_2 = 1.56$ ) causes the scattering of the light (a decrease in the light intensity), as seen in Fig. 4 (a). If the optical properties of the filling material and wood cells were similar to each other, the scattering of the light would not occur, and the materials would have transparency properties due to good light reflection, as seen in Figure 4b. The transparent wood with 1-mm (a) and 2-mm (b) thickness is shown in Figure 5. For providing transparency, the differences between refractive indexes must be little or similar, as presented by Fink (1992). For example, neat cellulose is white in color and is not transparent. When cellulose is saturated with water ( $n_c = 1.3$ ), wet cellulose exhibits transparency properties, as opposed to dried cellulose. In this study, the delignified



a)



b)

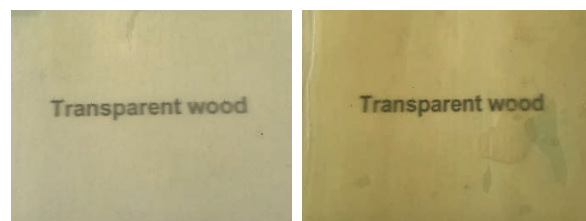
**Figure 5** Transparent veneers with 1-mm (a) and 2-mm (b) thickness before impregnation with epoxy

**Slika 5.** Prozirni furniri debljine 1 mm (a) i debljine 2 mm (b) prije impregnacije epoksidom

wood was impregnated with epoxy, including two components, and epoxy filling under vacuum exhibited the transparency properties in the veneers due to similarity in the refractive indexes of epoxy and delignified samples as seen in Figure 5 and 6.

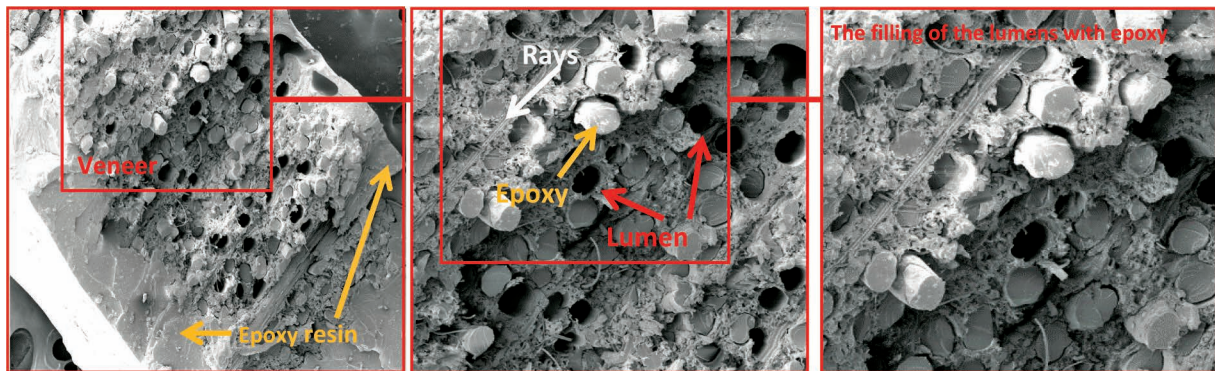
After the delignification process of the veneers, lightening was found to occur in the colors of the rays, and their visibility did not disappear, as seen in Figure 5. It can be said that this situation provides a natural appearance to transparent woods. On the other hand, if the desired look is not achieved, various softwoods or tree species, including high sapwood, can be selected to produce transparent wood. According to the SEM images in Figure 7, the epoxy resin filled many wood cell lumens; therefore, transparency was ensured. In the empty lumens, transparency remained at a certain level due to high light diffraction. Less dense epoxy or stronger vacuum can be applied to fill the empty lumens.

According to SEM images, it was determined that the epoxy generally filled the wood cells very well. Furthermore, SEM images show that the cell filling ability of epoxy is quite high (60-70 %). The empty lumens are presumably latewood trachea (thick cell walls, narrow lumens).



**Figure 6** Transparent samples with 1-mm (a) and 2-mm (b) thickness after impregnation with epoxy

**Slika 6.** Prozirni uzorci debljine 1 mm (a) i debljine 2 mm (b) nakon impregnacije epoksidom



**Figure 7** SEM images of transparent veneers prepared with epoxy resin  
**Slika 7.** SEM slike prozirnih furnira s epoksidnom smolom

### 3.3 FTIR results

#### 3.3. FTIR rezultati

After the delignification, most of the lignin in the veneers is removed with the delignification solvents. This turns the veneers into a cellulosic-structured material. The changes in the structure of the cellulosic material were investigated as given in Figure 8. FTIR analysis was conducted from 800 to 4600  $\text{cm}^{-1}$ . FTIR results generally exhibited the wavenumbers belonging to holocellulose, and hemicelluloses were detected at the wavenumber of 1734  $\text{cm}^{-1}$  (Cheng *et al.*, 2016). However, the aromatic rings of the lignin and C = C bonds were found at the wavenumbers of 1595 and 1603  $\text{cm}^{-1}$ , respectively. The vibration of the aromatic rings was detected at 1509  $\text{cm}^{-1}$  (Popescu *et al.*, 2007; Zhou *et al.*, 2015), and C–O bonds of the guaiacyl and syringyl lignin were detected at 1240 and 1670  $\text{cm}^{-1}$  (Popescu *et al.*, 2007; Chen

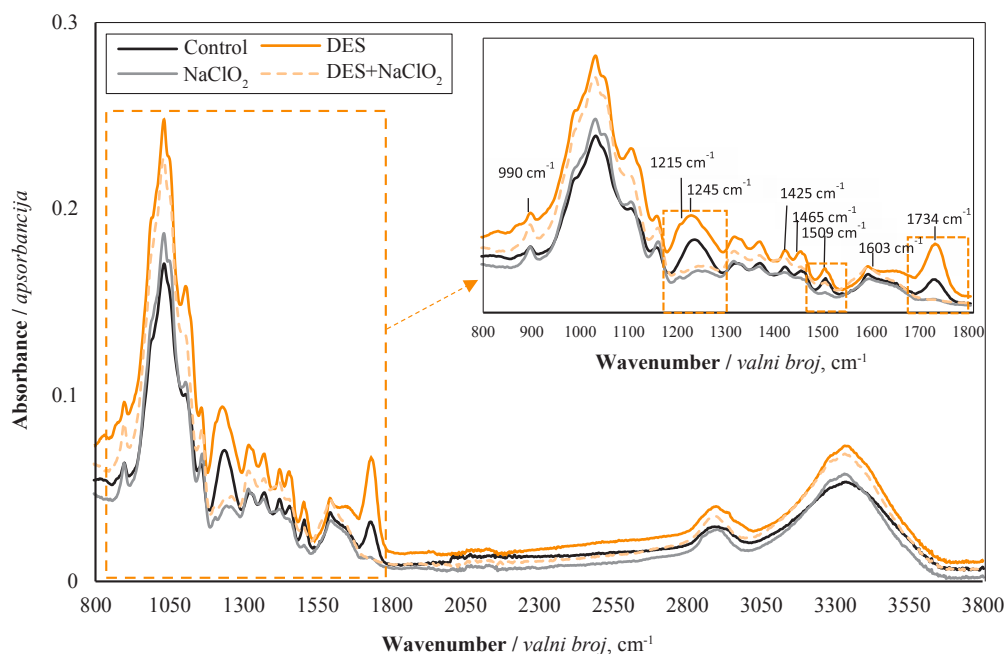
*et al.*, 2010; Traoré *et al.*, 2018) and 1215  $\text{cm}^{-1}$  (Zhou *et al.*, 2015).

As seen in Figure 8, especially in the veneers treated with  $\text{NaClO}_2$  and  $\text{DES}+\text{NaClO}_2$ , the bonds at 1245 and 1215  $\text{cm}^{-1}$  for guaiacyl and syringyl units, and the bonds at 1425 and 1465  $\text{cm}^{-1}$  for C–H asymmetrical deformation of methoxyl/aromatic skeletal vibrations were determined to be of low intensity (Kaur *et al.*, 2023). However, the lignin content in the veneers treated with DES was found to be high. Consequently, it can be said that the lignin delignification rate conducted with  $\text{NaClO}_2$  and  $\text{DES}+\text{NaClO}_2$  was found to be higher than the delignification made with DES. These results are supported by the data in Table 2.

### 3.4 XRD results

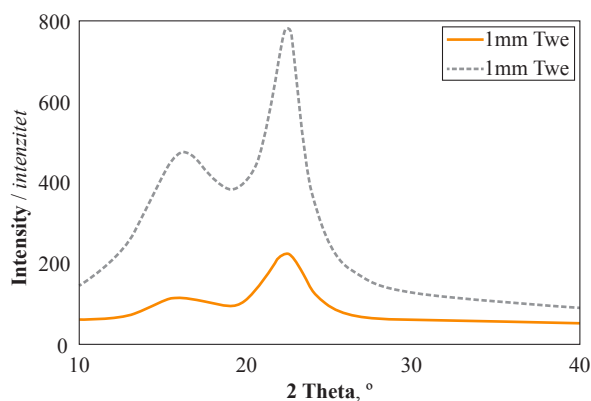
#### 3.4. XRD rezultati

XRD was used to determine the structural analysis and crystallinity of the veneers at two thickness (1-



**Figure 8** FTIR spectra of control and delignified woods  
**Slika 8.** FTIR spektri kontrolnoga i delignificiranog drva





**Figure 9** XRD patterns of transparent veneers with 1- and 2-mm thickness

**Slika 9.** XRD dijagrami prozirnih furnira debljine 1 i 2 mm

**Table 3** Peak points and crystallinity of transparent veneers with 1- and 2-mm thickness

**Tablica 3.** Vršne točke i kristaliničnost prozirnih furnira debljine 1 i 2 mm

Samples Uzorci	2 Theta, °	Crystallinity, % Kristaliničnost, %
1-mm Tve	15.8, 22.4	45
2-mm Tve	16.4, 22.6	65

and 2-mm), as given in Figure 9. As seen in the XRD patterns, the highest peak was detected at 16.4° and 22.6° for the transparent veneers with 2-mm thickness. For the transparent veneers with 1-mm thickness, two peaks were determined at 15.8° and 22.4°, but the peak at 15.8° was found to be weaker than the peak at 22.4°. The crystallinity index was measured according to the Gauss method and calculated at 65 % and 45 % for the transparent veneers 2-mm and 1-mm thick, respectively, as given in Table 3. Epoxy has an amorphous peak at 20°-23° (Alhumade *et al.*, 2019), and it can be seen that the veneers 1-mm thick absorbed the epoxy resin better than the veneers 2-mm thick (2-mm Tve). This status means that the veneers 1-mm thick (1mm Tve) had better transparency.

### 3.5 Mechanical results

#### 3.5. Rezultati mehaničkih ispitivanja

The tensile strength of the transparent veneers with 2-mm thickness was compared with beech wood veneers of the same dimension. Five specimens were tested for both veneers, and the obtained results are given in Table 4.

After the tensile test, the mean deformation and maximum load were found as 1.2 and 1.5 mm and 606 and 795 N for the control wood and the transparent wood, respectively. Tensile strength was calculated as 95 N/mm<sup>2</sup> for the control wood and 125 N/mm<sup>2</sup> for the transparent wood. The tensile strength of transparent wood was found to be higher (31 %) than the control

**Table 4** Tensile strength values of transparent and control veneers

**Tablica 4.** Vrijednosti vlačne čvrstoće prozirnih i kontrolnih furnira

Values / Vrijednosti	Control wood Kontrolno drvo	Transparent wood Prozirno drvo
Force, N sila, N	606	795
Tensile strength, N/mm <sup>2</sup> vlačna čvrstoća, N/mm <sup>2</sup>	95	125
Deformation, mm deformacija, mm	1.2	1.5

wood. The tensile strength of beech wood in the literature varies between 90-120 N/mm (Efe and Kasal, 2007; Çetin and Gündüz, 2017). As a result, the transparent veneers were found to have better mechanical performance than those presented in the literature.

Based on the above, the performance of the epoxy used as a filler has an important effect on the increase of the tensile strength of the transparent veneers. The literature reported that the used filling materials increase the mechanical properties depending on their structure (Li *et al.*, 2016; Zhu *et al.*, 2016b). From these results, it can be said that transparent wood can be an alternative material to wood in decorative structural applications for which high tensile strength is demanded.

## 4 CONCLUSIONS

### 4. ZAKLJUČAK

The sustainable, natural, and widespread availability of wood makes it a preference in many applications. This situation continues today, as in the past, with scientific studies and technological advances. The production and use of energy have strategic importance. Therefore, the interest in natural and renewable energy sources such as heat, light, and wind is quite high. The increase in delignification in the production of transparent wood obtained within the scope of this study naturally increased the transparency. It was observed that the deep eutectic solvent provided delignification at a lower rate than the chlorite method.

The structure of wood cells was also found to be important in the production of transparent wood. Large lumens are easier to fill with epoxy. More favorable results are obtained in materials with refractive indices close to each other. It has been determined that the voids in the material reduce the transmittance by reducing the intensity of light.

It was observed that the applied epoxy was effective in the higher mechanical resistance of the transparent wood samples compared with the control samples. Since it is possible to affect many stages in the production of transparent wood, luminous materials and decorative products and materials can be obtained during



the production phase. Larger surface transparent wood materials can be preferred especially in smart buildings to obtain maximum benefit from light. Transparent wood applications can be carried out in the division of office and private areas and in areas where privacy is required. Again, with different disciplines, their potential to be used as a panel for the production of electricity from solar energy and the storage of the produced electricity can be increased.

### Acknowledgements – Zahvala

The authors gratefully acknowledge the support of the Scientific and Technological Research Council of Türkiye (TÜBİTAK), (Project No: 120O337).

## 5 REFERENCES

### 5. LITERATURA

- Alhumade, H.; Rezk, H.; Nassef, A. M.; Al-Dhaifallah, M., 2019: Fuzzy logic based-modeling and parameter optimization for improving the corrosion protection of stainless steel 304 by epoxy-graphene composite. *IEEE Access*, 7, 100899-100909.
- Alvarez-Vasco, C.; Ma, R.; Quintero, M.; Guo, M.; Geleynse, S.; Ramasamy, K. K.; Wolcott, M.; Zhang, X., 2016: Unique low-molecular-weight lignin with high purity extracted from wood by deep eutectic solvents (DES): a source of lignin for valorization. *Green Chemistry*, 18 (19): 5133-5141. <https://doi.org/10.1039/C6GC01007E>
- Çetin, F.; Gündüz, G., 2017. Evaluation of research studies about mechanical properties of some wood species in Turkey. *Journal of Bartın Faculty of Forestry*, 19 (1): 161-181. <https://doi.org/10.24011/barofd.306723>
- Chen, H.; Ferrari, C.; Angiuli, M.; Yao, J.; Raspi, C.; Bramanti, E., 2010: Qualitative and quantitative analysis of wood samples by Fourier transform infrared spectroscopy and multivariate analysis. *Carbohydrate Polymers*, 82: 772-778. <https://doi.org/10.1016/j.carbpol.2010.05.052>
- Cheng, S.; Huang, A.; Wang, S.; Zhang, Q., 2016: Effect of different heat treatment temperatures on the chemical composition and structure of Chinese fir wood. *BioResources*, 11 (2): 4006-4016. <https://doi.org/10.15376/biores.11.2.4006-4016>
- Cristescu, C.; Karlsson, O., 2013: Changes in content of furfurals and phenols in self-bonded laminated boards. *BioResources*, 8 (3): 4056-4071.
- Efe, H.; Kasal, A., 2007: Determination of some physical and mechanical properties of various wood and wood composite materials. *Journal of Polytechnic*, 10 (3): 303-311.
- El Achkar, T.; Greige-Gerges, H.; Fourmentin, S., 2021: Basics and properties of deep eutectic solvents: a review. *Environmental Chemistry Letters*, 19: 3397-3408. <https://doi.org/10.1007/s10311-021-01225-8>
- Fink, S., 1992: Transparent wood: A new approach in the functional study of wood structure. *Holzforschung*, 46 (5): 403-408. <https://doi.org/10.1515/hfsg.1992.46.5.403>
- Francisco, M.; Bruinhorst, A. V. D.; Kroon, M. C., 2012: New natural and renewable low transition temperature mixtures (LTTMs): screening as solvents for lignocellulosic biomass processing. *Green Chemistry*, 14 (8): 2153-2157. <https://doi.org/10.1039/C2GC35660K>
- Fu, Q.; Ansari, F.; Zhou, Q.; Berglund, L. A., 2018: Wood nanotechnology for strong, mesoporous and hydrophobic biocomposites for selective separation of oil/water mixtures. *ACS nano*, 12 (3): 2222-2230. <https://doi.org/10.1021/acsnano.8b00005>
- He, Z.; Wu, H.; Cao, Y., 2014: Recent advances in polymer solar cells: Realization of high device performance by incorporating water/alcohol-soluble conjugated polymers as electrode buffer layer. *Advanced Materials*, 26 (7): 1006-1024. <https://doi.org/10.1002/adma.201303391>
- Hou, X. D.; Li, A. L.; Lin, K. P.; Wang, Y. Y.; Kuang, Z. Y.; Cao, S. L., 2018: Insight into the structure-function relationships of deep eutectic solvents during rice straw pretreatment. *Bioresource Technology*, 249: 261-267. <https://doi.org/10.1016/j.biortech.2017.10.019>
- Kaur, J.; Mankoo, R. K.; Chahal, G. K., 2023: Synthesis of rice straw biopolymers based hydrogels and their use as media for growth of monocot (wheat) and dicot (moong bean) plants. *Chemical Papers*, 77 (5): 2539-2555. <https://doi.org/10.1007/s11696-022-02644-9>
- Li, Y.; Fu, Q.; Yu, S.; Yan, M.; Berglund, L., 2016: Optically transparent wood from a nanoporous cellulosic template: combining functional and structural performance. *Biomacromolecules*, 17 (4): 1358-1364. <https://doi.org/10.1021/acs.biomac.6b00145>
- Li, L.; Yu, L.; Wu, Z.; Hu, Y., 2019: Delignification of poplar wood with lactic acid-based deep eutectic solvents. *Wood Research*, 64 (3): 499-514.
- Lynam, J. G.; Kumar, N.; Wong, M. J., 2017: Deep eutectic solvents' ability to solubilize lignin, cellulose, and hemicellulose; thermal stability; and density. *Bioresource Technology*, 238: 684-689. <https://doi.org/10.1016/j.biortech.2017.04.079>
- Mankar, A. R.; Pandey, A.; Modak, A.; Pant, K. K., 2021: Pretreatment of lignocellulosic biomass: A review on recent advances. *Bioresource Technology*, 334: 125235. <https://doi.org/10.1016/j.biortech.2021.125235>
- Popescu, C. M.; Popescu, M. C.; Singurel, G.; Vasile, C.; Argyropoulos, D. S.; Willfor, S., 2007: Spectral characterization of eucalyptus wood. *Applied Spectroscopy*, 61: 1168-1177.
- Rotzetter, A. C.; Schumacher, C. M.; Bubenhofer, S. B.; Grass, R. N.; Gerber, L. C.; Zeltner, M.; Stark, W. J., 2012: Thermoresponsive polymer induced sweating surfaces as an efficient way to passively cool buildings. *Advanced Materials*, 24 (39): 5352-5356. <https://doi.org/10.1002/adma.201202574>
- Russel, A. P.; Richard, L. G., 1984: Formation and structure of wood. In: *The chemistry of solid wood*, Advances in Chemistry. American Chemical Society, Washington, Chapter 1, pp. 3-56. <https://doi.org/10.1021/ba-1984-0207.ch001>
- Sivasubramanian, S.; Manohar, B. M.; Rajaram, A.; Puvanakrishnan, R., 2008: Ecofriendly lime and sulfide free enzymatic dehairing of skins and hides using a bacterial alkaline protease. *Chemosphere*, 70 (6): 1015-1024. <https://doi.org/10.1016/j.chemosphere.2007.09.036>
- Traoré, M.; Kaal, J.; Cortizas, A. M., 2018: Differentiation between pine woods according to species and growing location using FTIR-ATR. *Wood Science and Technology*, 52 (2): 487-504. <https://doi.org/10.1007/s00226-017-0967-9>
- Yaddanapudi, H. S.; Hickerson, N.; Saini, S.; Tiwari, A., 2017: Fabrication and characterization of transparent wood for next generation smart building applications. *Vacuum*, 146: 649-654. <https://doi.org/10.1016/j.vacuum.2017.01.016>

25. Yang, R.; Cao, Q.; Liang, Y.; Hong, S.; Xia, C.; Wu, Y.; Lam, S. S., 2020: High capacity oil absorbent wood prepared through eco-friendly deep eutectic solvent delignification. *Chemical Engineering Journal*, 401: 126150. <https://doi.org/10.1016/j.cej.2020.126150>
26. Yaşar, S.; Tanriverdi, H., 2008: Effect of residual lignin amount on color properties of holocellulose after delignification process. *Turkish Journal of Forestry*, 9 (2): 170-176.
27. Zhou, C.; Jiang, W.; Cheng, Q.; Via, B. K., 2015: Multivariate calibration and model integrity for wood chemistry using Fourier transform infrared spectroscopy. *Journal of Analytical Methods in Chemistry*, 2015: 429846. <https://doi.org/10.1155/2015/429846>
28. Zhu, H.; Fang, Z.; Preston, C.; Li, Y.; Hu, L., 2014: Transparent paper: fabrications, properties and device applications. *Energy & Environmental Science*, 7 (1): 269-287. <https://doi.org/10.1039/C3EE43024C>
29. Zhu, M.; Li, T.; Davis, C. S.; Yao, Y.; Dai, J.; Wang, Y.; Hu, L. 2016a: Transparent and haze wood composites for highly efficient broadband light management in solar cells. *Nano Energy*, 26: 332-339. <https://doi.org/10.1016/j.nanoen.2016.05.020>
30. Zhu, M.; Song, J.; Li, T.; Gong, A.; Wang, Y.; Dai, J.; Hu, L., 2016b. Highly anisotropic, highly transparent wood composites. *Advanced Materials*, 28 (26): 5181-5187. <https://doi.org/10.1002/adma.201600427>

**Corresponding address:**

**Assoc. Prof. Dr. ESER SÖZEN**

Bartın University, Faculty of Forestry, Ağdacı Street, 74100, Bartın, TÜRKİYE, e-mail: esozen@bartin.edu.tr