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OPTICAL PROPERTIES OF TRANSPARENT WOOD PREPARED FROM BALSA

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ABSTRACT

In this study, transparent wood was prepared by deactivating chromophore groups in raw balsa wood, followed by solvent free dehydration and a subsequent vacuum-assisted polymer infiltration. Thermal degradation of transparent wood takes place in two main steps. Optical properties (colorimetry, transmittance) of transparent wood made from balsa wood and acrylic polymers were studied. Highest values of transmittance in the visible part of spectra were achieved by 0.7 mm thick samples at approx. 77%. The dependence of thickness on transmittance showed a linear decrease with increasing thickness of transparent wood. The created material also exhibits absorbing properties in visible and UV spectra.

KEYWORDS: Balsa wood, colorimetry, optical properties, transmittance, transparent wood, wood composite.

INTRODUCTION

Wood has a distinct natural hierarchical structure that is advantageous since some nanotechnology applications necessitate it but it is challenging to replicate artificially (Berglund and Burgert 2018). The broad inner surface of wood and its structure provide a variety of possible uses for wood in functionalized materials. It is a renewable structural material and due to its processing ease, ability to regenerate, and capacity for biodegradation, wood has recently become a prominent sustainable building material (Chen et al. 2020). According to research, wood's inherent structure and composition support new alterations that can manage and govern the material's surface characteristics, molecular structure, porosity, and composition, all of which have the potential to greatly increase the material's use. For instance, wood has a hierarchically aligned porous structure that facilitates efficient ion and water transport as well as load-bearing capability and is principally composed of three key components: hemicellulose, cellulose, and

lignin (Beaumont et al. 2021, Berglund and Burgert 2018, Völkel et al. 2022a,b). It is possible to create novel engineered wood materials that retain many of the benefits of wood by utilizing these natural characteristics and enhancing them through physical or chemical modification (delignification, polymer/nanoparticle infiltration, densification, functional group grafting, chelation, and carbonization) (He et al. 2023). Due to its significant light absorption and dispersion, wood is not transparent in and of itself. The chemical components, in this example lignin, and other chromophores in the case of wood, have a significant impact on light absorption (Zhu et al. 2016). Wood's porous nature and the uneven distribution of its constituent parts cause light dispersion. As a result, the first stage in producing transparent wood is the removal or decolorization of lignin, which produces a white substance that is opaque due to the differing refractive indices of carbohydrates and air (Zelinka et al. 2022). An innovative material known as translucent wood has exceptional qualities such as low heat conductivity, high modulus, high strength, and transparent optical characteristics. Construction, light-emitting, photovoltaic, magnetic, and energy-storage materials all stand to benefit greatly from these benefits. Numerous academics from various nations have been studying the ecologically friendly, quick, and widespread preparation of very translucent wood and its use in certain technologies in recent years (Wan et al. 2021, Wu et al. 2021, Zhu et al. 2016). To create luminous wood, for instance, transparent wood can be coupled with other functionalizing agents including nanoparticles, quantum dots, and lasing dyes. Lasers, electroluminescent devices, and LED illumination are examples of potential application areas (Fu et al. 2020). The phrase "energy-efficient building" refers to plans intended to use less energy for heating or cooling, lighting, and ventilation (Pacheco et al. 2012). A promising construction material for energy-efficient structures is transparent wood. Further modifications may be made to it to accomplish various functionality, including improved light dispersion (Li et al. 2016), thermoregulation (Zhang et al. 2020), and formaldehyde gas detection (Liu et al. 2020). For instance Montanari et al. (2021), Zhu et al. (2016) and Li et al. (2016) have produced transparent wood bio-composites with a good mechanical performance and an exceptional optical transmittance of 90% that have the potential to replace glass.

Therefore, this work demonstrates an easy and efficient procedure to prepare transparent wood using a solvent-free dehydration with good optical and mechanical properties applicable in many fields. The main goal of this research was to determine the dependence of transmittance on the thickness of this composite material. Moreover, the evaluation of visible light filtering by transparent wood was successfully measured by colorimetry.

MATERIAL AND METHODS

JAF Holz Slovakia s. r. o. provided longitudinally cut balsa wood (*Ochroma pyramidale*, L.) with dimensions of 1000 x 10 x (0.7 – 3.0) mm of density 0.15 – 0.17 g.cm^{-3}. Two samples for each thickness (0.7 mm – 3.0 mm) were prepared (in total 16 samples). CentralChem s.r.o. supplied the sodium silicate, sodium hydroxide, magnesium sulfate, DTPA, and H_2O_2 (35%). Direct preparation of deionized water took place in the lab. Hexan-1,6-diyl

(2-hydroxyethyl)-methacylate, dodecyl-methacrylate-diacrylate were produced in the lab. and 2,2´-azobis(2-methylpropionitrile) was purchased from Sigma-Aldrich.

Lignin modification

Li et al. (2017) first suggested the lignin modification process. Balsa wood samples are immersed in a solution that modifies lignin at a temperature of 80°C. Deionized water, sodium silicate (3.0 wt%), sodium hydroxide solution (3.0 wt%), magnesium sulphate (0.1 wt%), DTPA (0.1 wt%), and finally H_2O_2 (4.0 wt%) are mixed to create the solution. Depending on the thickness of the samples, H_2O_2 (35% vol) is gradually added to the solution until the samples turned entirely white after a few hours. To allow the remaining chemical to disperse and leach away, the samples are then put back in a deionized water bath for 24 h.

Transparent wood preparation

Transparent wood composites were produced in too steps: dehydration without solvents and infiltration and curing of polymers: *(1)* Wood samples were dried without employing a solvent exchange phase prior to polymer infiltration. A quick and efficient procedure was designed to preserve the wood microstructure of the samples. The samples were initially taken out of the deionized water and put in a freezer (-25°C). The samples were loaded with a metal cube and placed in a vacuum chamber once they had completely frozen. This prevented the samples from twisting. Fresh silica gel was then completely placed over the setup, it was sealed, and a vacuum of 0.3 Pa was applied until the samples were completely dried (this might take anywhere from several hours to 24 h, depending on the thickness of the samples). *(2)* Hexan-1,6-diyl-diacrylate (60:30:10 wt%), 2 -hydroxyethyl-methacylate, and dodecyl-methacrylate were infiltrated without first polymerizing them. The acrylates were combined with the activator (2,2'-azobis(2-methylpropionitrile, 0.2 wt%) and allowed to dissolve for one hour. Following complete vacuum infiltration, the samples were sandwiched between two glass slides, wrapped in aluminium foil, and subjected to a 2 h polymerization process in a hot oven set to 90°C. Individual stages of transparent wood sample preparation are shown in the *Fig. 1*.

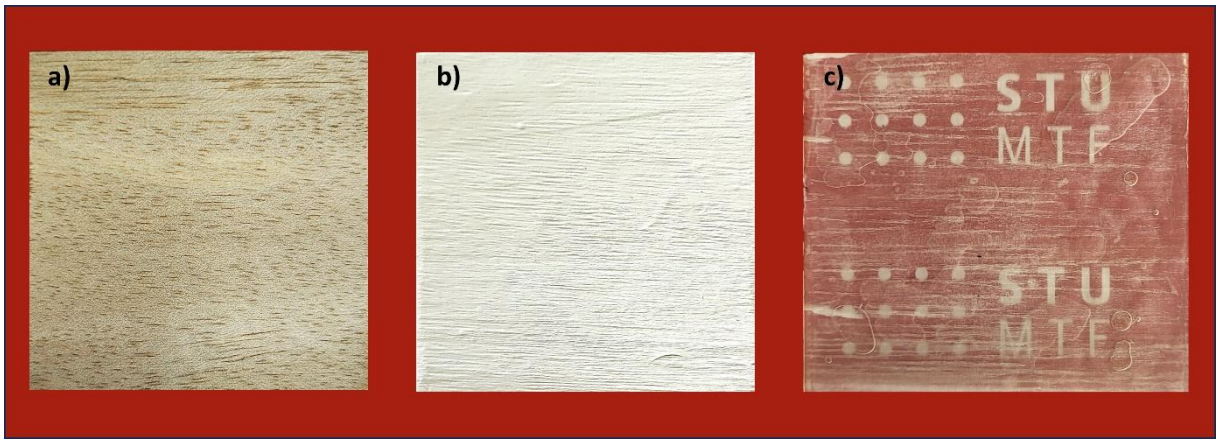

Fig. 1: Different stages of transparent wood sample (1.1 mm) preparation: a) raw balsa wood; b) lignin-modified wood; c) transparent wood.

Characterization of the samples

The transparent wood samples preserved up to 90 wt% of lignin, according to research by Li et al. (2017), making them stronger than the delignified alternative. They reported an average weight loss of the Klason lignin (TAPPI method TAPPI T 222 om-02) caused by the chromophore modification to be 21.3 ± 1.1%.

After polymer infiltration, a high-lignin content transparent wood was produced. Proportion of the wood in the samples was 28 ± 1 wt%, by comparing weight of raw balsa wood and prepared transparent wood.

The colorimetry of the samples was performed using a Colorimeter NR200 Precision (Threenh Technology Co., Ltd.; Shenzhen, China) with the following characterizations: Measuring aperture F8 mm, Colour space CIE L*a*b* and Light Source D65. Change of the colour passing through the samples was calculated from the Eq. 1:

$$\Delta E_{ab}^{*} = \sqrt{(L_2^{*} - L_1^{*})^2 + (a_2^{*} - a_1^{*})^2 + (b_2^{*} - b_1^{*})^2} \quad (1)$$

where: L_2^{*}, a_2^{*} and b_2^{*} are the colour parameters of light passing through the sample and L_1^{*}, a_1^{*} and b_1^{*} are the colour parameters of the background base paper.

By comparing these two sets of parameters we could found the transparency of the samples for the chosen colours. Total transmittance was measured using a modified photometer RMG2.1 (Heil Metalle GmbH, Mülheim, a. d. Ruhr, Germany). UV-VIS spectroscopy was realized by Cary 60 – Agilent UV-VIS spectrophotometer.

Thermogravimetric analysis was performed on a Netzsch STA 449 F5 Jupiter device. The weight of the samples was approximately 10 mg, and the measurements took place in nitrogen atmosphere at a flow rate of 100 ml.min^{-1}. The heating rate was 10 K.min^{-1}.

RESULTS AND DISCUSSION

Thermogravimetric analysis

During thermogravimetric analysis (Fig. 2), balsa wood had a stable weight up to approximately 200°C, which is typical for all wood specimens (Sun et al. 2020). Subsequently, it decreased in one significant step between the temperatures of 201.6°C and 373.2°C by 71.74% with the maximum rate at 348°C. The rest of the curve shows a gradual slow decline.

The thermogravimetric curve (Fig. 2) of the pure resin in the first phase slowly decreases to a temperature of 190°C. Subsequently, decomposition occurs in two phases. The first of them is more pronounced and occurs between temperatures of 190°C and 350.6°C. The weight loss achieved 74.8% and the maximum rate of weight loss was recorded at a temperature of 279.8°C. The second phase directly follows the first and continues to approximately 455°C, with a weight loss of 22.7%.

The course of the curve of transparent wood (*Fig. 2*) is more complex compared to the previous cases. The influence of both components, as well as delignification is reflected in it. Similar to the case of the balsa wood sample, the weight loss up to 200°C is practically negligible.

At 207°C, the first phase begins, characterized by a relatively slow decrease with the maximum rate of mass loss at 235.3°C. Since this decrease does not occur in the wood sample, it is probably the decomposition of the polymerized resin. The beginning of the second phase can be determined at 298°C. There is a significant acceleration of weight loss in it. It lasts up to a temperature of 366.1°C and reaches its highest speed at 338.9°C. This area is very similar to the main wood decomposition area. It is therefore possible to assume that the decomposition of cellulose and hemicellulose plays a more significant role in the mentioned temperature range. During the last phase, which takes place between temperatures of 366.1°C and 427.1°C, the rate of weight loss is slightly reduced (maximum at 400.7°C). It can be attributed to the last phase of resin decomposition.

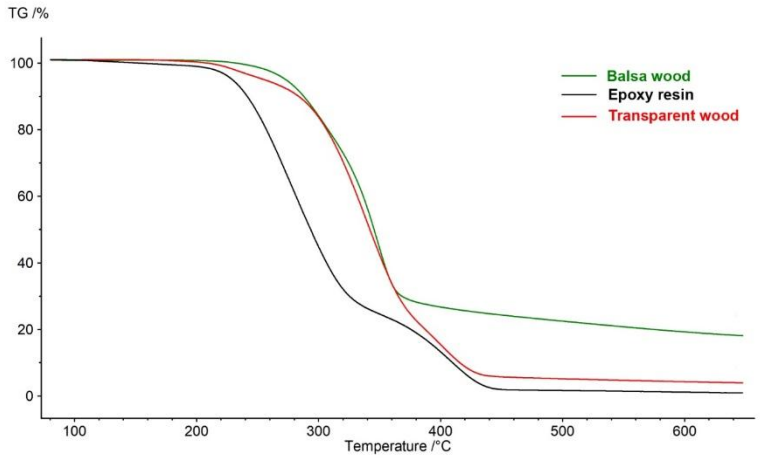

Fig. 2: Thermogravimetric curves of balsa wood, epoxy resin and transparent wood.

Colorimetric analysis

*CIE L*a*b* colour difference*

Colour change analysis was performed by comparing the colour of the reference background with the colour transmitted by a 1.1 mm thick transparent wood sample. Three reference base papers were chosen. Due to the heterogeneity of the transparent wood, the colour of the background passing through it was measured at five points of the sample. The measured values of individual parameters (L*, a*, b*) are recorded in *Tab. 1*.

Tab. 1: Colour parameters for four different background colours.

	L*	a*	b*
White background			
Reference base	**91.102** ± 0.10	**0.493** ± 0.01	**-2.115** ± 0.04
Sample	**77.416** ± 0.47	**2.506** ± 0.27	**3.477** ± 0.2
Red background			
Reference base	**43.746** ± 0.18	**46.079** ± 0.13	**23.124** ± 0.13
Sample	**59.107** ± 1.47	**13.701** ± 1.38	**3.700** ± 0.33
Green background			
Reference base	**68.615** ± 0.07	**-30.337** ± 0.09	**37.413** ± 0.19
Sample	**65.150** ± 0.70	**-8.640** ± 0.86	**11.786** ± 0.70
Blue background			
Reference base	**46.682** ± 0.30	**-9.228** ± 0.12	**-26.401** ± 0.11
Sample	**59.143** ± 1.78	**-1.679** ± 0.27	**-5.850** ± 1.34

From the displayed results, a decrease in the value of L* for white (-13.7) and green (-3,46) backgrounds is visible. In the case of red (+15,36) and blue (+12,46) background, the lightness parameter L* increase. Comparison of the colours and the calculated ΔE value (from Eq. 1) can be seen in Fig. 3.

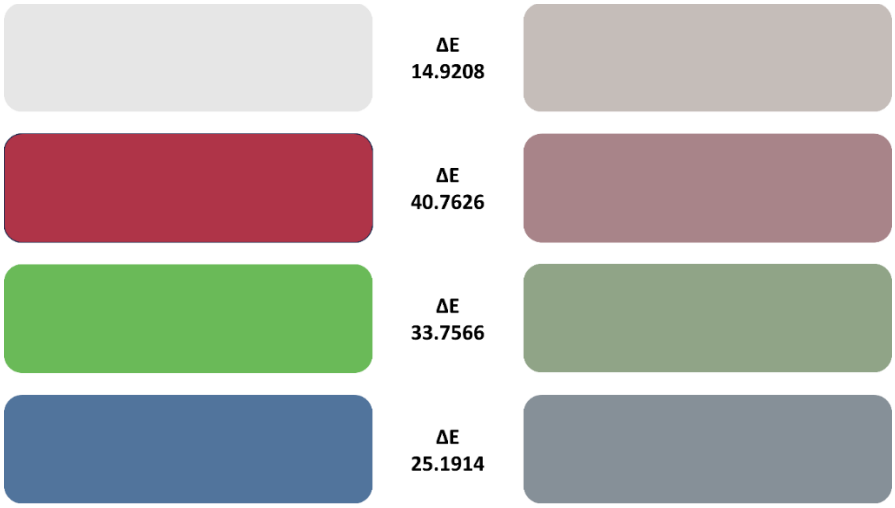

Fig. 3: Background colours (left) and the corresponding colours filtered by the transparent wood sample (right) with a calculated colour difference.

Transmittance of the 0.9 mm thick transparent wood samples, in the UV and visible range is shown in Fig. 4. Highest values were reached in the near infra-red part of the spectra (70.4% - 800 nm), with linearly declining course in the visible spectra (36.8% - 350 nm). This material also shows high absorbance rate in the UV past of the spectra. The result proves that the transparent wood sample absorbs all measured wavelengths of light to a certain extent. Similar results were obtained by Yang et al. (2021) (1.5 mm, 72% transmittance), Wang et al. (2021) (2.0 mm, 60% transmittance), Fu et al. (2020) (1.0 mm, 81% transmittance) or Qiu et al. (2020) (1.5 mm, 74.9% transmittance), Al-Qahtani et al. (2021) (0.2 mm, 65% transmittance).

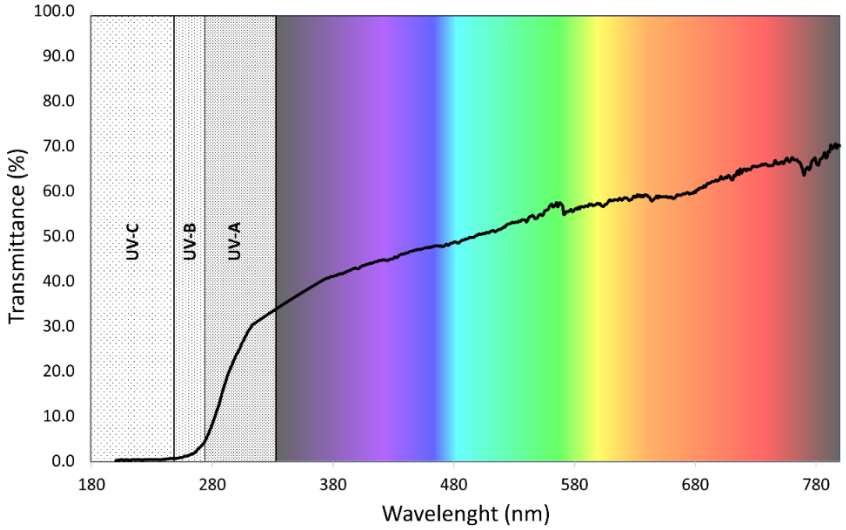

Fig. 4: Transmittance of the 0.9 mm thick transparent wood sample in the UV-VIS spectra.

Wood fibres are irregular, almost cylindrical tubular cells that have a few microns of cell wall thickness and tens of microns of empty centre lumen space. The cell wall itself has a hierarchical wood structure and is formed of aligned cellulose nanofibrils in a lignin/hemicellulose matrix (Nilsson and Rowell 2012). In transparent wood bio-composites, a refractive index-matching polymer is introduced to cover the wood substrate's micro- and nanoscale pores. Consequently, a material that is optically homogenous is created, reducing light scattering. Depending on how the wood and polymer interact, the interface between the cell wall and lumen polymer will be different. At the cell wall-lumen interface, air pockets at the micro- and nanoscale can be seen when the wood-polymer adhesion is poor (Li et al. 2018).

Fig. 5 shows data for total transmittance of transparent wood at various thicknesses. With increasing thickness, transparent wood exhibits a linear reduction in transmittance in the visible light spectrum. The highest total light transmittance was achieved by 0.7 mm thick transparent wood (77.4%) and the lowest was achieved by 3 mm thick sample (38.7%). The light scattering inside transparent wood, which contributes to the reduction in transmittance, can be explained by the fact that light enters the cell lumen space, scattering occurs at the interface between the polymer and the wood cell wall, and the photon can travel in any direction, as proposed by Chen et al. (2019). Similar results were obtained by Pang et al. (2021) who concluded that with increasing transparent wood thickness, the total transmittance of the material steadily decreases as a result of increased light scattering during light transmission through the material. According to Chen et al. (2022) further investigation is necessary since it is challenging to discriminate between forward scattering caused by minor wood-polymer refraction index mismatch at interfaces and random scattering contributions from nano-porosity in the cell wall.

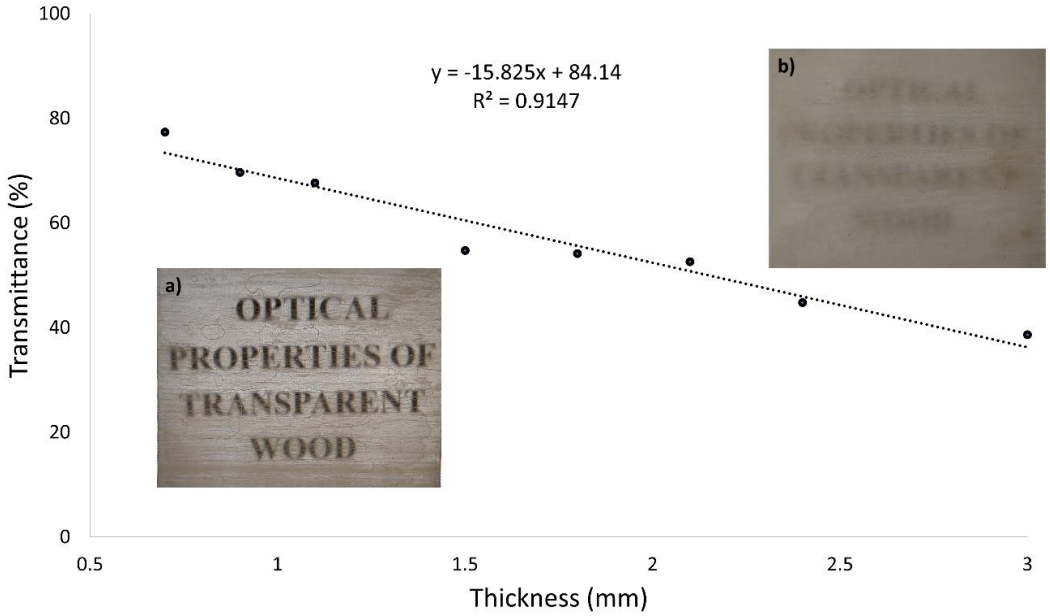

Fig. 5: Dependance of transmittance on the thickness of the transparent wood (TW) sample; a) TW sample of thickness of 0.7 mm; b) TW sample of thickness of 3 mm.

CONCLUSIONS

This paper deals with the investigation of the impact of transparent wood (based on lignin modification and acrylate polymer infiltration). Obtained data proved that: *(1)* Forward scattering from the cell wall and the polymer's mismatched refractive indices is the major cause of light scattering in transparent wood. *(2)* Transparent wood shows two phases during thermogravimetric analysis. First up to approx. 360°C which is similar to pristine wood used for transparent wood production, and second between 360 up to 650°C which is similar to acrylate resin. *(3)* Transparent wood (0.9 mm thick) is virtually opaque to UV-C radiation and transmits only a negligible portion of UV-B radiation (below 5%, transmission rate with wavelength decreases exponentially), and a significant part of UV-A radiation (5% up to 35% in strongly dependent on wavelength). Transmission of the visible part of the spectrum is also dependent on the wavelength from approx. 35% (violet) to approx. 70% (red). *(4)* Transmittance of acrylate transparent wood can be estimated from its thickness as follows: Transmittance = 84.14 – 15.825 * sample thickness (valid for 0.7 to 3 mm thickness).

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