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Research Article

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Transparent Wood Produced by Nitrate-Alkaline Delignification and its Properties

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ABSTRACT
Nowadays, emphasis is placed on the use of environmentally friendly materials both in terms of construction and the application of protective equipment. For this reason, dealing with wood products such as transparent wood is advisable, even though its production requires chemicals. Environmental friendliness is the main reason for producing transparent wood using the nitrate-alkali method. Transparent wood is mainly used indoors as a design element. This article deals with producing transparent wood from beech (Fagus sylvatica) and its impact on optical, mechanical properties, and fire resistance. Among the mechanical properties, the compressive strength was evaluated. The optical properties were very positive, especially regarding opacity, which had a zero value. Regarding fire resistance, maximum average rate of heat emission (MARHE) and limiting oxygen index (LOI) were evaluated, and from these values, the transparent wood does not reach the qualities of untreated wood. In the case of MARHE, the value for transparent wood was 2.6× higher than for beech wood, which was 208.38 kW·m-2. The LOI was reduced from 24.55% for beech to 18.75% for thorny transparent wood. This research's results serve to understand better the production of transparent wood, which can be used in its application.

Keywords: Beech Wood, Epoxy Resin, Mechanical Properties, Optical Properties, Fire Resistance

Introduction

Although the sample is de facto transparent in a partial sense, it is not a colorless material. The correct terminology is partially transparent wood. However, the term 'transparent wood' is used for ease of wording and communication. The wooden template must first be prepared by chemically isolating it with lignin. Delignification can be done with various chemicals, and then the material is impregnated with resin [1].

Transparent wood is becoming an increasingly researched material, mainly due to its properties, such as light transmittance (light transmittance is up to 40%). Currently, it is mainly used by construction and solar energy companies. For example, Li et al. used transparent wood as a material for the walls of a house [2]. The wall material is made using the H2O2 method. Thus, the delignification is done with hydrogen peroxide using its vapors, followed by impregnation with epoxy resin to let the outside light through more efficiently. Thus, this building becomes much more favorable for its occupants daily.

Other applications of this breakthrough material need to be explored, such as in the furniture industry. So far, all research has focused on literally transparent material, i.e., glass, colorless or textureless material. The material, therefore, lacks wood's visual and tactile natural properties. However, all the lignin, which carries the color component in wood, is not removed in the development of the material. In that case, some aesthetic elements proving a natural material with a semi-transparent texture can be retained. Such a material represents a high potential in the whole furniture sector. Specific parameters must be taken into account when choosing the wood species. Removing lignin from the material through chemical treatments weakens its structure, and low-density wood tends to break [3].

Researchers used hydrogen peroxide as a delignifying agent and epoxy as an additive to produce transparent wood [4,5]. In their work, they focused on the permeability, tensile strength and toughness of the transparent wood.

Similarly, Montanari et al. used sodium hydroxide for delignification, but polyacrylate was used instead of epoxy [6]. In this case, the authors used veneers of alder, birch, and

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beech, they aimed to determine the chemical composition of monosaccharides, the permeability of the transparent wood, and the thermal stability by thermogravimetric analysis and dynamic thermomechanical analysis.

The colorimetric properties for six wood species (birch, Chinese fir, linden, New Zealand pine, oguman, and black walnut) were investigated by Wu et al., who first used ethanol treatment followed by sodium hydroxide delignification and methyl-methacrylate as resin [3]. The colorimetric properties were determined as L*a*b* values and light transmittance, and in addition to these properties, the authors determined tensile strength or infrared spectra.

Sodium hydroxide combined with hydrogen peroxide, sodium silicate, magnesium sulphate, and DTPA (diethylene-triamine-penta-acetic acid) on balsa wood was also used by Samanta et al. and Chuturri et al, where first the wood was pretreated with acetone and ethanol [7,8]. These samples were also enriched with thermochromic or photochromic pigments as microcapsules. In the case of the thermochromic pigment, the pigment changed from black to colorless, and in the case of the photochromic pigment, it changed from white to purple shade when exposed to sunlight. The pigments reduced transmittance by 11–77% when exposed to UV light. The thermochromic pigments showed an increase in tensile modulus of 64% compared to when using the native thiolene polymer alone.

Mi et al. used sodium chlorite as a delignifying agent for Douglas fir. However, in this case, the delignification was carried out on the entire wood block, followed by epoxy treatment [9]. The authors then investigated how the microscopic aspect changed compared to a reference untreated wood sample. They also obtained a tensile strength of 91.95 MPa for the transparent wood, a significant increase over the untreated Douglas fir sample, which only reached 21.56 MPa. The authors followed previous research using PVA (polyvinyl alcohol) instead of epoxy, where mainly toughness was investigated. This research shows that transparent wood with polyvinyl alcohol has higher toughness and tensile strength than transparent wood made with epoxy [10].

Sodium chlorite in combination with polymethyl-methacrylate on birch veneer was used by Jungstedt et al. who looked at the chemical composition, which decreased from 19.8% to 2.8% in the case of lignin [11].

Based on these findings, the nitrate-alkali process was applied to beech wood to obtain fiber for transparent wood. Subsequently, epoxy resin was applied, and the transparent wood was compared for its optical and mechanical properties, and fire resistance.

Materials and Methods

Materials

The primary raw material for delignification was beech wood (Fagus sylvatica). The main properties of beech wood include high strength and hardness, high-quality durability and workability, and esthetic appearance. Beech wood is, however, susceptible to fungal and wood-boring insect attacks [12]. Beech wood is suitable for producing stairs, parquet, wall and ceiling cladding, wooden toys, kitchen utensils, and sports equipment [13]. This material was chosen because of its high availability and the ratio of its purchase price to its high strength and durability. The density of the wood used was approximately 700 kg·m-3.

The chemicals required for delignification were nitric acid (CAS 7697-37-2) at a concentration of 6% and sodium hydroxide at a concentration of 5% (CAS 1310-73-2) both from Penta Chemicals (Czech Republic).

Epoxy resin was used for production, which has several advantages, especially its fascinating transparency and protection against degradation by sunlight. The resin used for this research was Veropal UV PLUS 100, manufactured by Synpo a.s. (Czech Republic) consisting of component A (4,4′-isopropylidene-dicyclohexanol, oligomeric reaction products with 1-chloro-2,3-epoxypropane and 1,4-bis(2,3-epoxypropoxy) butane) and hardener B (propoxylated propylene-trimethanol and reaction products with ammonia). The processing time is 40 to 50 minutes, and the curing time at laboratory temperature is 24 to 36 hours. The viscosity of the resin was 700–800 mPa·s, and that of the hardener B was 250–300 mPa·s. The mixing ratio of the substances was A to B 100:66.

Delignification

Delignification removes lignin from lignocellulosic materials, which is necessary to produce transparent wood. This study used the nitrate-alkali method because of its efficiency and versatility on different wood species [14].

The delignification of beech chip wood was carried out in several steps:

- Cook beech wood in 6% nitric acid for 45 minutes,
- washing with water,
- extraction in 5% sodium hydroxide for 8 hours,
- washing with water and removal of black liquor,
- pulping for 3 minutes.

Transparent Wood Production

After the first stage of preparing the material to produce transparent wood, we obtained the necessary amount of pulp. This material was then mixed adequately with epoxy resin. First, the two components of epoxy resin were mixed in a 100:66 weight ratio. The resin, hardener, and pulp were mixed for 15 minutes (3000 rpm) using a WITEG shaft mixer (Wertheim, Germany).

After mixing the pulp with epoxy resin and hardener, it was necessary to obtain the desired shape for further investigation of this material. Using a 3D printer, forms of the required dimensions were produced. They were 10×10×74 mm for oxygen index, 30×20×20 mm for the compressive strength test, 100×100×10 mm for the fire resistance test, and 195×290×15 mm for optical properties measurement.

For optimum consistency and quality results, all handling of the mixed resin and pulp was at ambient and bulk temperatures of approximately 20 °C. This is because low temperatures can negatively affect the result.

Testing of Transparent Wood

Mechanical properties

The test samples underwent an acclimatization process in laboratory conditions before testing. This acclimatisation lasted long enough for the moisture content of all samples to become uniform. A TIRA test machine 2850 (Schalkau, Germany), designed for strength testing of materials, was used to measure mechanical properties under laboratory conditions. The compressive strength of the material was tested in accordance with CSN 49 0112 [15].

Optical Properties
The optical properties of the transparent wood were evaluated using a spectrophotometer FRANK PTI CM-3830 (Birkenau, Germany). For each sample, the coordinates were determined repeatedly at different sample locations. The CIE L*a*b* space at daylight illumination D65 and an observer angle of 10° were used to describe the optical properties. From these samples, the chroma C, which represents the saturation of the color, was calculated according to ISO 11664-4 [16]:

$$C = \sqrt{\Delta a^2 + \Delta b^2} \qquad (1)$$

where: C – chroma, Δa – difference of color coordinates, Δb – difference of color coordinates (ISO, 2008).

The hue of a color h can be expressed as the hue angle in degrees, defined as:

$$h = arc\,tg\left(\frac{\Delta b^2}{\Delta a^2}\right) \qquad (1)$$

Where: h – hue of a color, Δa – difference of color coordinates, Δb – difference of color coordinates. The hue angle starts on the + a axis and is therefore equal to 0°, 90°, 180° and 270° for red, yellow, green and blue, respectively [17].

In addition to the L*a*b* color space, whiteness was measured using the Whiteness CIE and Whiteness CIE-UV instrument. According to ISO, the brightness was determined as ISO Brightness and ISO Brightness -UV. The last value measured by the spectrophotometer was the opacity.

Fire Resistance
Measurements were performed using a conical calorimeter ISO 5660-1 by standard (ISO, 2015) [18]. The calorimeter was used to determine the following parameters: maximum average rate of heat emission (MARHE), peak heat release rate (PHRR), total oxygen consumption, which correlates with the total heat released and the energy balance of the combustion, the final amount of CO_2 smoke produced, which poses a risk of visibility impairment and toxicity, and loss of weight of the material. The measurements were carried out under defined conditions of thermal radiation load, standard radiator temperature of 850 °C, and heat flux value of 50 kW·m-2.

Another parameter was the limiting oxygen index (LOI). For this parameter, 5 beech wood and 5 transparent wood samples were used all in accordance with BS 2782-0 and ISO 4589-2 by equipment FTT0077 01 (Fire Testing Technology, United Kingdom). The samples were conditioned at 23°C and 50% relative humidity before determination [19,20].

Results

Mechanical Properties
Figure 1 shows the values for determining the material's compressive strength.

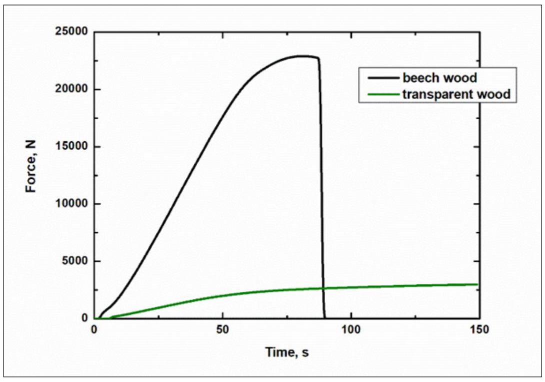

Figure 1: The material's compressive strength

Figure 1 shows that each material shows an entirely different trend when measured. In the case of timber, when pressure is applied in the direction of the grain, there is a deformation which results in a shortening of the length of the body. However, in the case of transparent wood, there is no deformation, only compression of the elastomer. The results of the compression tests are given in Table 1.

Table 1: The material's Compressive Strength

Materials	Beech wood	Transparent wood (20% fiber)
Compression tests, MPa	51.82±3.41	7.29±0.47

Optical Properties
Table 2 below shows the optical parameters for transparent wood with 10, 20, and 40% fiber content. Figure 2 shows the surfaces of the transparent wood samples used for optical property measurements.

Table 2: Optical Parameters of Transparent Wood

Optical Parameter	Chroma	Hue of a color	Whiteness		Brightness		Opacity
			CIE	CIE-UV	ISO	ISO-UV	
10% fibre content	14.31 (0.01)	1.57 (0.00)	-3.59 (0.08)	-4.58 (0.46)	53.61 (0.06)	53.41 (0.03)	0.00
20% fibre content	15.87 (0.02)	1.57 (0.00)	-16.82 (0.49)	-17.30 (0.21)	50.64 (0.15)	50.53 (0.11)	0.00
40% fibre content	14.57 (0.01)	1.57 (0.00)	4.61 (0.17)	3.75 (0.11)	59.05 (0.03)	58.87 (0.02)	0.00

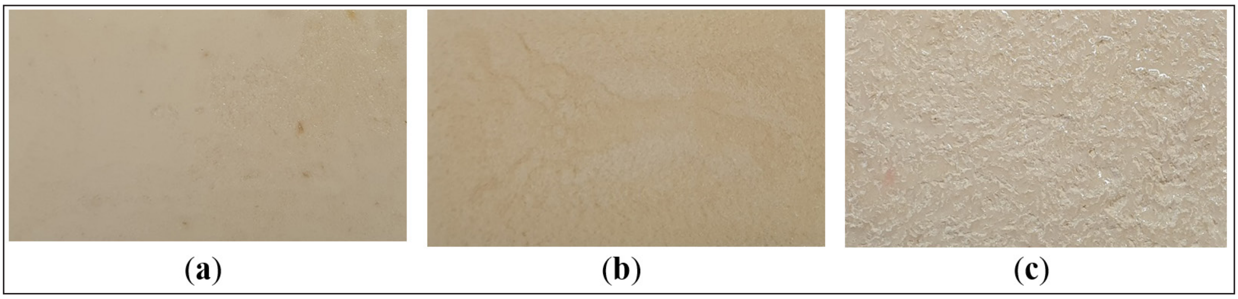

Figure 2: Surfaces of transparent wood: (a) – 10% fiber content, (b) – 20% fiber content, (c) – 40% fiber content

Fire Resistance

Table 3 below shows the variables (MARHE, PHRR, total heat, heat of combustion, and LOI) for the transparent wood variant with 20% fiber. The progression of the selected average variables (PHRR, O2 concentration, CO2 concentration – amount of smoke, loss of weight of the material) is shown in Figure 3.

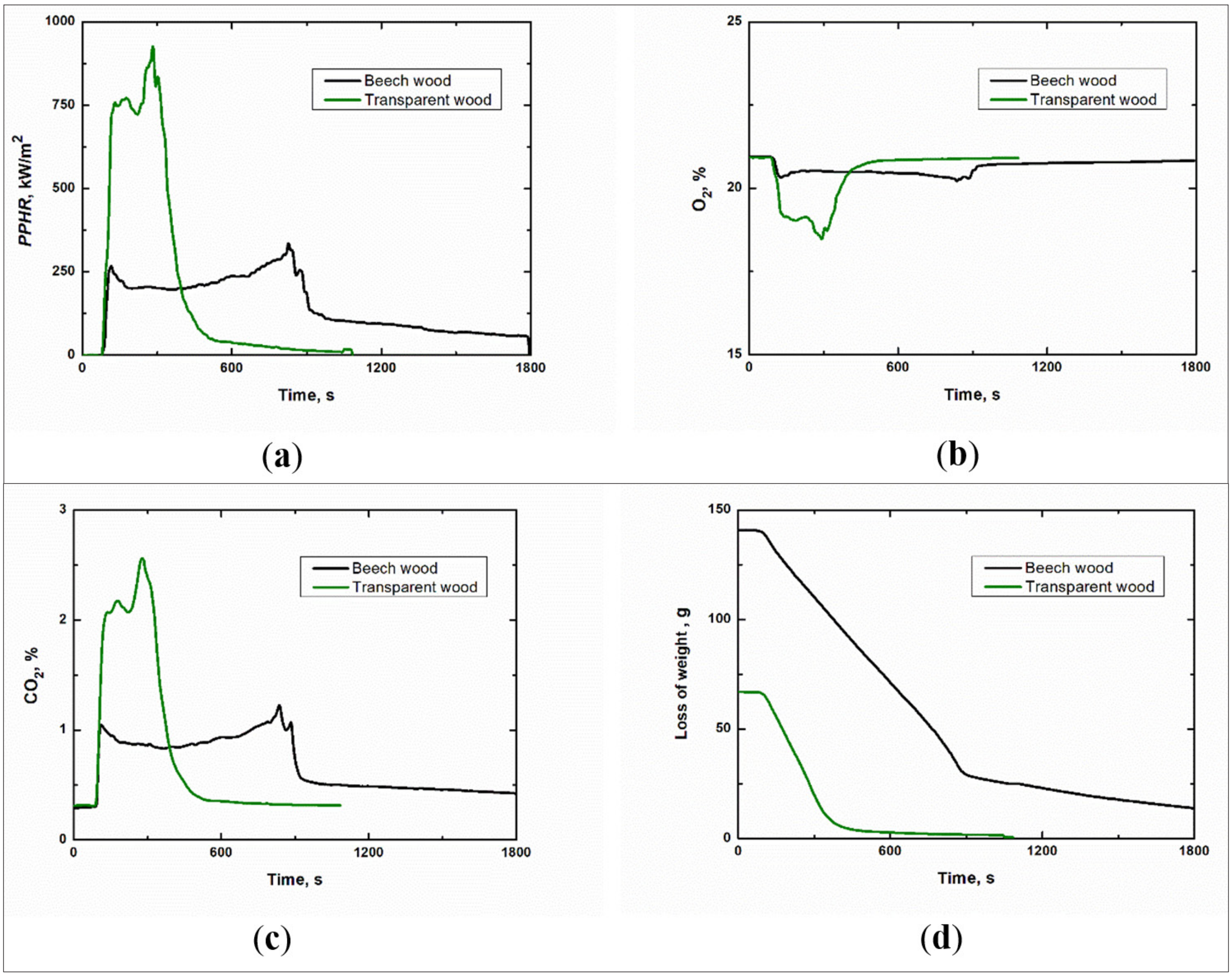

Figure 3: Fire resistance parameters of transparent wood: (a) – PHRR, (b) – O2 concentration, (c) – amount of smoke, (d) – loss of weight of the material.

Table 3: Fire resistance parameters of transparent wood

Fire resistance parameter	MARHE	PHRR	Total heat	Heat of combustion	LOI
	kW·m-2	kW·m-2	MJ·m-2	MJ·kg-1	%
Beech wood	208.38±15.50	403.32±64.93	257.80±19.94	18.57±2.23	24.55±0.55
Transparent wood (20% fibre)	550.75±13.16	981.81±16.18	227.22±13.71	30.86±0.78	18.75±0.24

Discussion

From the results measured using the TIRA test machine 2850, it can be seen that transparent wood has a significantly lower compressive strength compared to beech wood. In contrast to beech wood, where the strength limit was reached in all measurement cases, transparent wood did not show this phenomenon under the same measurement conditions. Since the jaw feed rate was set to a constant 10 mm/min, the force required for this feed was up to 10 times less for the transparent wood. The results show that the compressive strength limit of beech wood is 18-23 kN. However, the limit point for the transparent wood was not found even after 20 minutes of constant increasing force applied to the specimen due to the high elasticity. However, the material deformed (compressed) by about 66.6% under the same force at which the strength limit for beech wood was reached. Therefore, the compressive strength limit for transparent wood cannot be determined from this measurement, but it can be said that the compressive strength of this material is significantly lower. The compressive strength was determined to be 51.8 MPa for beech wood and more than 7 times lower at 7.3 MPa for transparent wood. Nevertheless, some authors could determine the compressive strength limit using a resin containing 56.1–74.6 MPa nanoparticles for variously treated transparent wood composites [21]. In the case of chroma (saturation), the so-called saturation, the highest values were achieved with the 40% suspension of beech pulp, which would be expected. In comparison with the literature, Wachter et al. who used hydroxyethyl-methacrylate and methyl methacrylate, achieved color variation equal to that of nitrate-alkali pulp after about 4 days of exposure of the samples to UV light. Among other optical properties, both CIE and ISO whiteness/brightness for nitrate-alkali delignification are good at 40% fibre representation. A very positive finding is the opacity value. Opacity is the ratio of light incident to light transmitted. The lower the value, the more transparent the material [22,23]. In our case, therefore, it is a transparent material according to the measured values.

From the comparison of the results of the tested samples, it can be said that the average MARHE value of the beech samples is 208.4 kW·m-2 and PHRR is 403.3 kW·m-2, but for the transparent wood samples, these values were 550.7 kW·m-2 for MARHE and 292 kW·m-2 for PHRR value. These results suggest that the maximum average heat release rate of the transparent wood samples is more than twice as large as that of the beech wood samples. It can also be said from this test that the material flash rate is very similar for both samples. While the beech wood burned with a sustained flame after (on average) 87.7 seconds, the transparent wood burned after 86.5 seconds. It can also be seen from the graphs that the oxygen concentration of the beech wood did not fall below 19.6% during the measurements, whereas the lowest value for the transparent wood was measured between 18.0–18.5%. The maximum carbon dioxide concentration was measured between 1.6–1.8% for the beech samples and 2.5–3.0% for the transparent wood samples.

Regarding material loss, beech wood decreased weight by 82–89%. The weight loss of transparent wood at the end of testing was 92–98%. From these data, the fire performance of transparent wood is imperative to be further investigated and optimized for the development of materials that meet fire safety requirements.

Transparent wood and epoxy resin, as such, do not have a specific EURO fire resistance class, i.e., they are not included in a specific fire classification. However, according to CSN EN 13501-1 classification, transparent wood can be classified from the measured values between groups D (easily flammable, total ignition will occur) and F (extremely flammable, unclassified products) [24].

The LOI measurement data obtained indicate that transparent wood is characterized by a lower oxygen demand in the surroundings, thus the material ignition is correspondingly easier. This may be due to the combination of epoxy resin and pulp, or the higher density of transparent wood compared to beech wood, but also to the change in structure by delignification or lower lignin content.

From the LOI results, the lower oxygen demand in the surroundings makes it easier for transparent wood to ignite, and this can lead to faster flame spread. Further research on transparent wood is imperative for a deeper understanding of fire safety properties and mechanisms and the subsequent optimization of these properties.

Militky described all materials with an LOI higher than 26% as less flammable and those with an LOI lower than 21% as materials that burn easily [25]. Therefore, based on this classification and the results measured for transparent wood, this material should be classified as a readily combustible material, specifically plastic sheeting, which has similar results under the exact measurement according to BS 2782-0 and ISO 4589 [19,20].

Conclusions

This research involved a comprehensive investigation of the properties of transparent wood produced by the nitrate-alkali method to assess its potential use in architecture, design, and other industries.

Based on the measured values and results, it is not possible to say unequivocally whether transparent wood is in all respects a better or more desirable material than conventional wood in the future. As with other materials, each has its use for specific situations. For example, based on the results of this work, transparent wood would not be viable in building components due to its fire performance. Conversely, although wood is a highly desirable material in the design industry, transparent wood is unique. Its advantage is primarily its versatility in various possible shapes and sizes. This opens up new possibilities for use in interior design and decorative objects. The optical properties of this material are fascinating, so much so that, from the measured values, transparent wood can be considered a de-facto transparent material, allowing energy companies to apply transparent wood in connection with solar panels.

However, the properties of transparent wood need to be further investigated, both in terms of the long-term sustainability of the material and in terms of recycling and biotic and abiotic factors.

Author Contributions: For research articles with several authors, a short paragraph specifying their individual contributions must be provided. The following statements should be used "Conceptualization, K.H. and T.H., methodology, K.H., validation, K.H. and T.H., formal analysis, K.H. and T.H., investigation, K.H., writing—original draft preparation, K.H., writing—review and editing, K.H.., visualization, K.H.., supervision, K.H., project administration, K.H.., funding acquisition, K.H. All authors have read and agreed to the published version of the manuscript.

Please turn to the CRediT taxonomy for the term explanation. Authorship must be limited to those who have contributed substantially to the work presented.

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Data availability Statement: The datasets generated and/or analyzed during the current study are not publicly available but are available from the authors upon reasonable request.

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