Surface Chemistry and Adhesive Bonding of Tannin-rich Woods to a Poly(vinyl acetate) / Linseed Oil Coating: The Role of Thermal Modification

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GRAPHICAL ABSTRACT

Surface Chemistry and Adhesive Bonding of Tannin-rich Woods to a Poly(vinyl acetate) / Linseed Oil Coating: The Role of Thermal Modification

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Effects of thermal modification were investigated relative to the surface and adhesive bonding of coatings to tannin-rich oak (*Quercus petraea*) and chestnut (*Castanea sativa*) woods. Samples were heat-treated for 2 hours at different temperatures (160 °C, 180 °C, and 200 °C) and then coated sequencially with linseed oil followed by poly(vinyl acetate) (PVAc) varnish. Increasing heat treatment temperature led to a significant darkening of the wood and a decrease in gloss and surface roughness. While thermal modification alone caused a reduction in adhesion strength, the subsequent application of linseed oil was effective in mitigating this adverse effect. The oil created a more favorable bonding interface by reducing the negative influence of tannins and hydrophobicity on the adhesive. The combined thermal modification and linseed oil treatment achieved favorable adhesive bonding performance of tannin-rich wood species with the varnish. This study suggests that low heat treatment temperatures combined with a linseed oil coating can be recommended for applications where good adhesion strength is desired.

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Keywords: Heat treatment; Linseed oil; Oak; Chestnut; Adhesion strength; Surface properties

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INTRODUCTION

Wood material is widely used in fields such as construction and engineering due to its various positive features. Its mechanical properties, biological structure, physical properties, and chemical composition are the reasons why wood material is preferred. In addition, being a good insulation material and having high resistance values according to its density are the main reasons why it is a sought-after material (Bozkurt and Göker 1981; Yıldız *et al.* 2005). In addition to its many positive properties, wood also has disadvantageous properties that limit its areas of use or cause problems during use. Wood is not resistant to physical, chemical, and mechanical damage factors and biotic factors (fungi, insects, termites, *etc*.) in its natural state (Tomak *et al* 2014; Kılınç *et al* 2023). Natural weathering, which is a major cause of wood deterioration, has been linked to the effects of ultraviolet (UV) radiation and water (Feist and Hon 1984; Zhang and Kamdem 2000; Pelit *et al* 2019). The weathering process mainly affects the wood surface and significantly reduces the aesthetic appearance and performance of wood (Jirous-Rajkovic *et al*. 2004). Often the color undergoes a two-stage transition, initially acquiring a redder tone, and thereafter tending to become grayer and bluer (Kropat *et al.* 2020). Weathering results in color changes in wood, cracking, breaking, warping, mold formation, and splitting occur on wood surfaces (Williams 2005). In addition, due to weather conditions,

there are decreases in the physical, mechanical, and biological properties of wood and wood products (Grelier *et al*. 2000; Zhang and Kamdem 2000; Evans *et al*. 2005; Williams 2005; Özcan and Korkmaz 2018).

Among the various modification methods developed to overcome these difficulties, heat treatment, in which wood is thermally modified without the use of any chemicals, has attracted great attention in both academic and industrial fields in recent years as an environmentally friendly solution. Heat treatment application seems to be an economical and environmentally friendly option in terms of improving the quality of fast-growing and low-durability coniferous and broad-leaved tree species. Since 1990, Thermowood, which is the most widely used method, has taken its place in the forest products market as a commercial product. The industrial scale heat treatment process and the Thermowood trade name emerged from the joint work of Finnish industry with the Finnish Research Center, VTT (Yıldız *et al* 2005). All widely used heat-treatment methods for wood are designed to minimize the contact between the heated material and oxygen. This is essential, since exposure of wood to elevated temperatures (160 to 230 °C) in the presence of air leads to a high risk of fire, as well as uncontrolled oxidative degradation (Hill 2006; Esteves and Pereira 2009). In the case of the ThermoWood® process, oxygen contact is minimized by conducting the treatment in a steam atmosphere. The steam not only displaces oxygen and thereby prevents ignition, but also contributes to a more controlled thermal modification of the wood (ThermoWood Handbook 2003; Militz and Altgen 2014).

According to the ThermoWood Handbook (ThermoWood Association of Finland 2003), the process consists of stages: heating, drying, cooling, and conditioning. During the heating stage, the timber is heated and pre-dried. In addition, the temperature in the kiln is rapidly increased and a large amount of steam is produced. This produced steam is used to displace air from the kiln, creating an oxygen-free environment. This is crucial for preventing the wood from combusting at high temperatures. At the beginning of the drying stage, the temperature is continuously increased, and the timber is effectively dried. At a certain point during the drying stage, whilst the moisture content material of the wood reaches nearly 0%, the temperature is swiftly accelerated to various values of 185 ℃ to 215 ℃, relying at the programs of the processed products. The wood is kept at this temperature for 2 to 3 hours. During the cooling and conditioning stage, the temperature of the wood is decreased to the range 80 to 90 ℃ by the water spray method. Conditioning is completed to moisten the heat-treated wood and to adjust its moisture level to the range 4% to 7% (Finnish ThermoWood Association 2003). Heat-treated timber is preferred in the production of building exterior cladding, interior cladding, park and garden floorboards, park and garden furniture, children's playgrounds, windows and shutters, interior and exterior doors, saunas, interior furniture, and musical instruments. Heat-treated timber has a great potential for use in the construction industry. The final effects of heat treatment on wood properties vary significantly, depending on processing variables such as applied temperature and duration, as well as internal factors such as the anatomical structure of the wood, its density, and especially the amount of extractive substances (resin, tannin, oil, *etc*.) it contains. In this context, hardwoods such as sessile oak (*Quercus petraea*) and Anatolian chestnut (*Castanea sativa*), which have high commercial value in Europe and Türkiye, are of particular interest. These species have a chemical structure rich in hydrolyzable tannins. Although tannins are polyphenolic compounds that give wood its natural color, durability, and antimicrobial properties, they can cause serious problems during surface finishing processes, especially when they interact with water-based varnish systems. These compounds, which tend to migrate to the surface, can inhibit the curing

reactions of the varnish film or create a weak interface between the wood and the coating layer, critically reducing adhesion performance. Gündüz *et al.* (2011), in their study on tannin-rich wood species such as chestnut, reported that heat treatment improves the physical properties of wood by removing these compounds. The darkening of wood color caused by heat treatment is also associated with the degradation of tannins and other extractive substances (Gašparík and Barcík 2015). This process alters the chemical structure of the wood surface, increasing its hydrophobicity (water repellency) and reducing its surface energy. This can negatively affect the spreading ability and adhesion performance of polar adhesives. Similarly, Chen *et al.* (2012) linked the decrease in pH values during heat treatment to the formation of organic acids, emphasizing that this is an indication of thermal degradation of the chemical components.

In addition, in order to protect the wood material, coating processes are also carried out with some surface materials in addition to heat treatment. In the literature, coating processes with varnishes are generally carried out after heat treatment in order to protect the wood. For example, it was observed that the identity of the wood species did not have a large influence relative to surface roughness measurements for single and double component water-based varnish types, but Scots pine and chestnut wood types gave similar surface roughness results. It was reported that the reason for this was that the wood material surface was very well smoothed during the surface treatment preparation phase due to the heterogeneous structure of the ringed large-vessel chestnut wood and the vein gaps were completely filled by the filling varnish (Çakıcıer 2007). The gloss results on the ringed veined wood surfaces were found to be less than other species; the reason for this was explained as the fact that the vein gaps that were not completely filled. The increased scattering of light at the numerous air-solid transitions resulted in reduced specular reflection and increased diffuse reflection (Budakçı and Sönmez 2011). In the studies of Özalp *et al*. (2009), Scots pine (*Pinus sylvestris* L.) and chestnut (*Castanea sativa* M.) were subjected to heat treatment at 100, 150, and 200 °C for 2, 4, and 6 h before varnishing with water-based varnish. The hardness and gloss were found to decrease as the heat treatment temperature and time increased. Karamanoğlu and Kaymakçı (2018) determined the color and hardness changes of heat-treated chestnut wood (*Castanea sativa* Mill.) subjected to hygrothermal aging. The test samples were exposed to heat in an inert (nitrogen) environment under atmospheric pressure at three different temperatures (130, 180, and 230 °C) and two different times (2 and 8 h). According to the results, the color of the wood darkened in proportion to the increasing temperature and time after heat treatment. Compared to the control groups, the best color stability was obtained in samples heat-treated at 180 °C for 8 h. In terms of hardness, when natural control samples were compared with heat-treated samples, the least amount of hardness decrease occurred in samples heat-treated at 180 °C for 8 h.

In addition, many vegetable oils that are environmentally friendly and harmless to human health have been used in the field of wood coating, especially recently. Linseed oil (LO), which is preferred in this study, has been used for various applications in combination with wood since ancient times, but its areas of use and concentrations have changed over time. Nowadays, the risk of depletion of fossil resources and environmental concerns in renewable materials are becoming more and more important. For example, linseed oil, together with other natural oils, is increasingly in demand as a raw material for future applications, with its cost advantage, competitive performance, and ability to replace fossil-based products (Mahendran *et al*. 2012; Chang *et al*. 2018). LO is a triglyceride containing unconjugated polyunsaturated fatty acids produced by pressing the seeds of the

flax plant (*Linum usitatissimum*). Most LO fatty acids consist of C18 chains and contain unsaturated bonds. The major LO fatty acids are linolenic acid (35% to 60%), which has three unconjugated double bonds; linoleic acid (17% to 24%), which has two unconjugated double bonds; and oleic acid (12% to 34%), which has one double bond. Linseed oil also contains small amounts of saturated fatty acids compared to unsaturated fatty acids (Poth 2001). Addis *et al*. (2020) studied the strength of a LO-based adhesive on *Fraxinus americana* (white ash), *Liriodendron tulipifera* (yellow poplar), and *Pseudotsuga menziesii* (Douglas fir) woods. They stated that the adhesive they obtained could be used in timber applications as it showed positive effects.

These protective methods were employed due to the high tannin content of the wood species used in this study. When wood is processed or comes into contact with moisture, the tannin substance in its structure mixes with water and can cause stains and color changes on the surface. Such stains can be highly visible on surface coatings such as paint and varnish, causing the application to fail. In addition, wood species with high tannin content can cause corrosion and darkening in metal by chemically reacting with metal fasteners (screws, nails, *etc*.). This leads to weakening of the metal over time and a decrease in the durability of the connection. Finally, synthetic glues used in the production of wood-based panels can react with tannins, reduce the bonding strength, and negatively affect the durability of the product (Altun and Esmer 2017). Thermal treatment restructures the surface chemistry and physics of these tannin-rich woods in a complex way. Under high temperatures, thermal degradation of hemicelluloses and cross-linking reactions of the lignin polymer make the chemical structure of the wood more stable (Tjeerdsma and Militz 2005; Pétrissans *et al*. 2014). At the same time, extractive substances, including tannins, are partially degraded, polymerized or removed from the surface. As a result of these chemical transformations, the wood surface darkens (Bekhta and Niemz 2003), acquires a hydrophobic (water-repellent) character (Hakkou *et al*. 2005), and its surface energy decreases (Gérardin *et al.* 2007).

Another crucial effect of heat treatment is the modification of the hydrophobic/hydrophilic balance of the wood surface. The degradation of hemicelluloses during heating reduces the number of accessible hydroxyl (–OH) groups, thereby lowering the surface polarity and free surface energy (Hakkou *et al.* 2005). As a result, the surface becomes increasingly hydrophobic with rising treatment temperature, although the magnitude of this effect depends strongly on the species and process parameters. Hydrophobic wood surfaces generally show poor wetting by polar adhesives, leading to insufficient spreading and weaker interfacial bonding (Nejad and Cooper 2011). Since the present work evaluates adhesion performance after thermal modification, this phenomenon is of central importance to the interpretation of the results.

Although the effects of heat treatment on the basic properties of various wood species have been extensively investigated in the literature, there has been a need for studies systematically investigating the synergistic interaction of the surface properties of woods with high tannin content such as oak and chestnut on the adhesion mechanisms of LO and coating systems after heat treatment are quite limited. The reason for preferring LO in this study is that it is plant-based, harmless to the environment and human health, and relatively more economical compared to other vegetable-based oils. In summary, this study aimed to systematically investigate the changes in the surface properties of oak and chestnut woods with high tannin content as a result of heat treatments at different temperatures (160, 180, and 200 °C) and their effects on PVAc varnish adhesion performance after coating with LO.

The innovative aspect of this work lies in combining thermal modification with linseed oil treatment specifically for tannin-rich hardwoods, and in evaluating both surface characteristics (color, gloss, roughness) and the strength of adhesion to a coating prepared using linseed oil as a primer coat and then applying poly(vinyl acetate) varnish. While previous research has mostly addressed these factors separately, the integrated approach presented here provides new insights and practical recommendations for industrial applications.

EXPERIMENTAL

Materials

Preparation of wood samples

Sessile oak (*Quercus petraea* (Matt.) Liebl.) and Anatolian chestnut (*Castanea sativa* Mill.) wood materials were used in this study due to their richness in tannin and industrial importance. All wood materials used in the experiments were selected from trunk woods of trees with similar age and morphological characteristics obtained from the trial fields of the Western Black Sea Forestry Research Institute in Türkiye. The timber was taken from smooth-fibreed sections, free from defects such as knots, cracks, rot and abnormal coloration. Final test samples were prepared with dimensions of 150 mm (longitudinal) × 75 mm (radial) × 20 mm (tangential) according to the TS ISO 3129 (2019) standard. For the color, gloss, and roughness test group, a total of 40 oak and 40 chestnut test samples were prepared, 10 from each species, for heat treatment purposes only. Since heat treatment + coating was applied in the adhesion test, a total of 40 oak and 40 chestnut test samples were prepared, 10 from each species. All samples were conditioned in a climate chamber at 20 ± 2 °C and 65 ± 5% relative humidity, at approximately 12% equilibrium humidity, before any processing and before the tests, until their masses stabilized (TS ISO 13061-1 2021).

Thermal modification

In the study, only heat treatment was applied in color, gloss, and surface roughness tests. Contrary to commercial heat treatment methods carried out in protective environments to prevent oxidation, fire hazards, and excessive mass loss, the primary objective of this work was to investigate the fundamental effects of thermal modification on the surface properties of tannin-rich woods in a controlled laboratory environment. The heat treatment process was therefore carried out in a programmable Nabertherm N11/HR model laboratory furnace operating under atmospheric pressure, which made it possible to systematically study the direct relationship between temperature and the resulting changes in wood surface properties. The experimental design was based on a control group (untreated) and three different heat treatment temperatures (160, 180, and 200 °C). The furnace temperature was ramped from ambient temperature to the target temperature at an average ramp rate of 5 °C/min. The samples were kept at this temperature for a net period of 2 h after the oven reached the desired temperature. At the end of the process, the oven power was cut off and the samples were allowed to cool slowly to room temperature in the oven without opening the oven door in order to prevent thermal shock and cracks.

Coating

In the study, the coating process was used only after heat treatment on adhesion test samples. Linseed oil used for the experiments was obtained from *Linum usitatissimum* (flax plant) seeds by cold- or hot-pressing method. It is a natural, biodegradable and renewable product. It is used as a binder in wood preservatives, oil paints, varnishes, antique furniture restoration, and traditional oil-based coatings (European Pharmacopoeia and Codex Alimentarius 2025). Before the application, the wood surfaces were cleaned and sanded. The coating process was carried out using a brush and was done in a single layer at a rate of 150 g/m^2 ± 10 g/m². The second layer was applied 24 h after the first layer. During the application process, care was taken to keep the ambient temperature and relative humidity constant; the processes were carried out at approximately 20 ± 2 °C and 50 ± 5% relative humidity. Samples with two coats were placed horizontally in the same environmental conditions and kept for 7 days in order to ensure complete curing. During this process, a controlled environment was preferred to prevent dust accumulation and surface deterioration that may occur on the surface of the samples. Poly(vinyl acetate) (PVAc) adhesive was applied to the surfaces of both thermally modified and thermally modified samples treated with linseed oil. It is important to note that the linseed oil application was a pretreatment step, not the final adhesive. The linseed oil primer coat was applied to improve the final bond strength by enhancing adhesive wetting and penetration (Hubbe and Laleicke 2025). All samples were then bonded together, and their bond strengths were measured according to the specified standard. Polyvinyl acetate (PVAc), used as the final coating in our study, is a colorless, non-toxic, water-dispersible thermoplastic adhesive. Commonly known as wood glue, school glue, and carpenter's glue, it is used as a water-based emulsion. When dry, PVA forms a hard, transparent, and hydrophobic film that is resistant to moisture, oil, and petroleum fuels. With its high initial tack, virtually invisible bond line, and low cost, PVA is also used in hot-melt adhesives, sealants, fabric finishes, and plastics.

The chemical properties of the LO used in the study are presented in Table 1.

Table 1. Some Chemical Properties of Linseed Oil

Properties	Explanation
Density (25 °C)	0.930 to 0.935 g/cm³
Refractive index	1.479 to 1.485
Iodine number	170 to 204 (shows a high degree of unsaturation)
Drying days	2 to 8 days
Acid value	4 to 7 mg KOH/g
Type of protective oil	It hardens by oxidation when in contact with air.

Methods

Color changes

The color parameters L^*, a^*, and b^* were determined by the CIEL*a*b* method. The L^* axis determines the lightness, whereas a^* and b^* are the chromaticity coordinates. The $+a^*$ and $-a^*$ parameters show the colors red and green, respectively. The $+b^*$ parameter represents yellow, whereas $-b^*$ represents blue. The L^* value can vary from 100 (white) to zero (black) (Zhang 2003). The color difference, (ΔE^*) was measured according to ASTM D1536-58T (1964). The color changes were determined using Eqs. 1 through 4.

$$\Delta a^* = a_f^* - a_i^* \quad (1)$$

$$\Delta b^* = b_f^* - b_i^* \quad (2)$$

$$\Delta L^* = L_f^* - L_i^* \quad (3)$$

$$\Delta E^* = ((\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2)^{1/2} \quad (4)$$

The color coordinates (L^*, a^*, b^*) on each sample surface were measured under the D65 standard illumination source and 10° observer angle. The average of the measurements taken from five different points for each sample was accepted as the L^*, a^*, b^* values of that sample. Total color change was performed with an X-Rite brand color measurement device.

Gloss

The gloss of the samples was determined using the Micro-TRI-Gloss device in accordance with the ASTM D523 (2018) standard. A 60° incidence angle was used in the measurements. A total of six measurements were made on each sample surface, three in parallel and perpendicular directions to the fibers, and the arithmetic average of these values was recorded as the final gloss value.

Surface roughness

Surface roughness measurements were made using the Mitutoyo Surftest SJ-301 profilometer device with a sensitivity of 0.01 µm in accordance with the TS EN ISO 21920-2 (2022) standard. The average surface roughness (R_a) value was taken as the basis as the evaluation parameter. Measurements were made perpendicular to the wood fibers, using a 5 µm radius device needle, a cut-off length of 2.5 mm, and a total evaluation length of 12.5 mm. The final R_a value of each sample was determined as the arithmetic average of measurements taken from three different regions.

Adhesion strength

After heat treatment, the adhesion performance of the plant-based coating was determined by the pull-off method according to ASTM D4541-22 (2022). The tests were carried out with a constant loading rate of 0.2 MPa/s. The maximum stress value (MPa) at the moment when the test head was separated from the surface was recorded as the adhesion strength of the surface. Before the test, the samples were sanded and cleaned of dirt and marks.

Statistical analysis

With the help of SPSS 26.0 computer program, Duncan tests and variance analysis were applied at 95% confidence interval after the results obtained from the tests. Statistical studies were carried out on homogeneity groups (HG) where various letters indicate statistical significance.

RESULTS AND DISCUSSION

Color Changes

The effect of heat treatment on the surface color of oak (*Quercus petraea*) and chestnut (*Castanea sativa*) woods was investigated through color changes values. The results regarding color changes by wood type and heat treatment temperature are presented in Table 2.

Table 2. Color Coordinates (CIE L*a*b*) of Control and Treated Wood Samples

Samples	Temperature (°C)	Time (h)	Color coordinates			Total color changes		
			*L**	*a**	*b**	*E**	Std. Dev.	H.G.
Control (Oak)	-	-	60.4	0.2	0.1	60.4	0.30	D
Oak	160	2	59.1	8.0	11.4	60.8	0.40	D
Oak	180	2	58.4	13.6	14.8	61.8	0.45	C
Oak	200	2	57.9	18.4	14.2	62.4	0.55	B
Control(Chestnut)	-	-	60.0	0.8	0.2	60.0	1.13	E
Chestnut	160	2	59.2	14.1	12.8	62.1	0.75	B
Chestnut	180	2	58.5	14.1	15.8	62.2	1.48	B
Chestnut	200	2	57.8	16.8	18.8	63.0	1.75	A
*Note: Std. Dev.: Standard deviation; and H.G.; Homogeneity group.								

In this study, the values presented for the control group are the directly measured CIE Lab coordinates of untreated wood, not the Δ values. Therefore, they are not expected to be zero. The color changes of the treated samples were interpreted according to these control coordinates. According to the color change results, the *L** values of the heat-treated samples decreased compared to the control samples. These decreases became larger as the heat treatment temperature increased. A positive increase in *a** values occurred in all samples. This indicates that the wood color shifted to red with heat treatment. This reddish coloration became more pronounced, particularly as the temperature increased. Similarly, the *b** values increased positively. This indicated that the wood color shifted to yellow after heat treatment. In this study, the lowest total color change value was obtained in the control (chestnut) samples, while the highest total color change value was determined in the chestnut wood samples heat treated at 200 °C (63.01). In both oak and chestnut woods, the total color changes increased gradually as the heat treatment temperatures increased. In the literature, a low total color change value is a sought-after feature. From this perspective, samples with low heat treatment temperatures showed more positive color change values.

The color of the wood material typically darkens during the heat treatment phase. The degree of color change depends particularly on the heat treatment time and temperature conditions (Chen *et al*. 2012; Mitani and Barboutis 2013; Lovrić *et al*. 2014). Color change is primarily due to the formation of chromophoric (color-giving) groups during the thermal decomposition of hemicelluloses in the wood structure and the restructuring of the lignin polymer. High temperatures trigger the emergence of new chemical structures such as carbonyls and quinones, darkening the color of the wood and increasing its yellow/red tones. These results are in full agreement with numerous recent studies examining the effects of heat treatment on wood color (Pétrissans *et al.* 2014; Candelier *et al.* 2016).

In this study, oak control samples and oak samples heat-treated at 160 °C were in the same homogeneity group, and no statistical difference was observed. No statistical

difference was detected between oak wood samples heat-treated at 160 °C and chestnut wood samples heat-treated at 160 and 180 °C. Significant differences were detected between the other samples ($p < 0.05$). The main reason for this difference is that the amount of tannin naturally found in the chemical structure of chestnut wood is higher than that of oak. Polyphenolic extractive substances such as tannins are quite sensitive to thermal degradation and easily oxidize or polymerize during heat treatment to form dark colored compounds. This explains why chestnut wood undergoes a more intense color change at the same temperature conditions (Salca and Hiziroglu 2014). This means that the effect of heat treatment temperature on color varies according to the wood type. In other words, while both species darken as the temperature increases, the extent of darkening in chestnut is higher than in oak. It was concluded that the total color difference (ΔE^*) values increased with the increase in heat treatment time in the heat treatment application applied to the wood samples. It has been reported that the ΔE^* increased with the increase in heat treatment time in the ThermoWood method (Ayata *et al* 2017a,b; Gürleyen *et al* 2017a,b; Şahin and Ayata 2017). The results obtained from the literature are consistent with this study. The graph showing the effect of heat treatment on ΔE^* is given in Fig. 1.

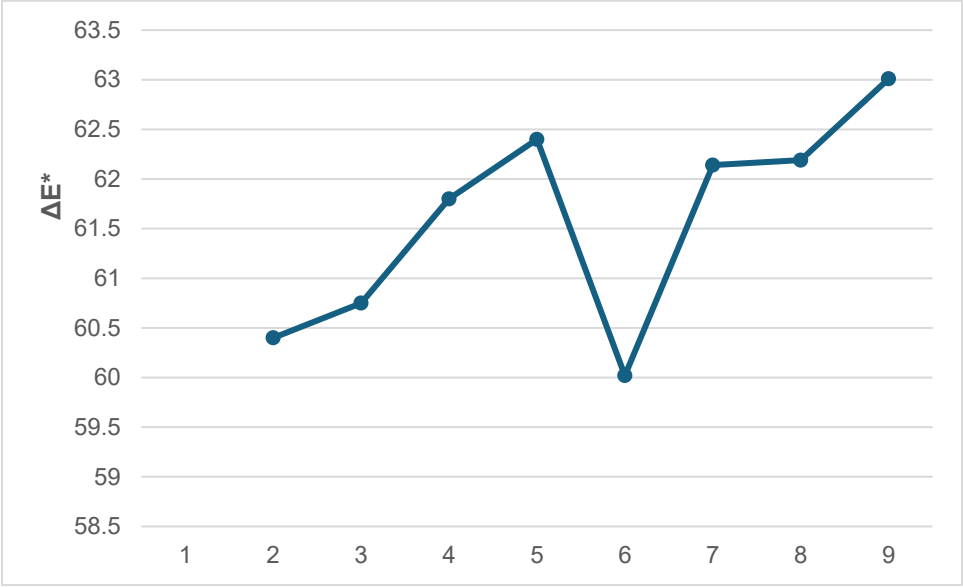


Fig. 1. Effect of heat treatment on color change (ΔE^*)

Gloss

The results regarding the gloss changes of oak (*Quercus petraea*) and chestnut (*Castanea sativa*) woods due to heat treatment are presented in Table 3. The gloss value in the oak control sample was 32.4, while that in the chestnut control sample was 30.1. As the heat treatment temperature increased, a periodic decrease in gloss was observed in both wood species. The main physical reason for this decrease in gloss value is the increase in surface roughness as a result of heat treatment. Heat treatment causes the degradation of sensitive components such as hemicellulose on the surface and generate micro-collapses in the cell structure, changing the topography of the surface. The rougher surface scatters the incident light in different directions in a scattered manner (diffuse) instead of reflecting it in a single direction (specular). This decrease in the ability of the surface to reflect light properly is directly recorded as a lower gloss value by the gloss measurement device. These

results are in line with literature studies reporting that heat treatment reduces gloss by increasing surface roughness (Korkut and Aytin 2015).

Table 3. Results of the Gloss Changes Based on Wood Type and Heat Treatment Temperature

Samples	Temperature (°C)	Time (h)	Gloss (Mean)	Std. Dev.	H.G.
Control (Oak)	-	-	32.4	0.30	A
Oak	160	2	28.3	0.40	BC
Oak	180	2	25.7	0.45	CD
Oak	200	2	22.1	0.55	E
Control (Chestnut)	-	-	30.1	0.13	AB
Chestnut	160	2	27.5	0.75	C
Chestnut	180	2	24.9	0.48	D
Chestnut	200	2	21.3	0.75	E
*Note: Std. Dev.: Standard deviation; and H.G.: Homogeneity group.					

Oak and chestnut woods that were heat treated at 200 °C were in the same homogeneity group and no statistically significant difference was found between them. However, the greatest loss in gloss was determined in chestnut wood samples at 200 °C. This suggests that the surface structure of chestnut wood may be more sensitive to thermal degradation at high temperatures than oak. The different anatomical structure and chemical composition of chestnut wood may cause faster deterioration of surface integrity and therefore more diffuse reflection of light, resulting in greater reduction in gloss. Sikora *et al*. (2018) examined of gloss changes in spruce (*Picea abies* L.) and oak wood (*Quercus robur* F.) that occur due to thermal treatment.

The thermal modification was performed at 160, 180, and 210 °C according to Thermowood process. Acccording to their results thermally modified samples of both wood species showed a very significant drop in values due to the increasing temperature of the thermal modification. The results obtained in this study were similar to those obtained by Sikora *et al*. (2018), as the gloss of the samples decreased as the heat treatment temperature increased. Čabalová *et al*. (2018) observed that changes in some properties of pedunculate oak (*Quercus robur* L.) wood samples were assessed after thermal treatment. Heat treatment was performed at 160, 180, and 200 °C in an oxidizing atmosphere. Their results showed that the color lightness of pedunculate Oak decreased as temperature increased. The results obtained in this work were similar to the findings obtained by Čabalová *et al*. (2018), as the gloss of the samples decreased as the heat treatment temperature increased.

Although no microscopic analysis was conducted in this study, previous research has demonstrated that the anatomical structure of chestnut wood, which contains larger vessels and a more heterogeneous distribution of fibers, can lead to faster deterioration of surface integrity during thermal degradation (Čabalová *et al.* 2018; Sikora *et al.* 2018). This structural sensitivity may explain why chestnut exhibited a greater loss of gloss compared to oak at 200 °C. Future studies employing microscopic techniques such as SEM could provide direct evidence of these anatomical differences and validate the interpretation presented here. A graph showing the effect of heat treatment on the gloss change is given in Fig. 2.

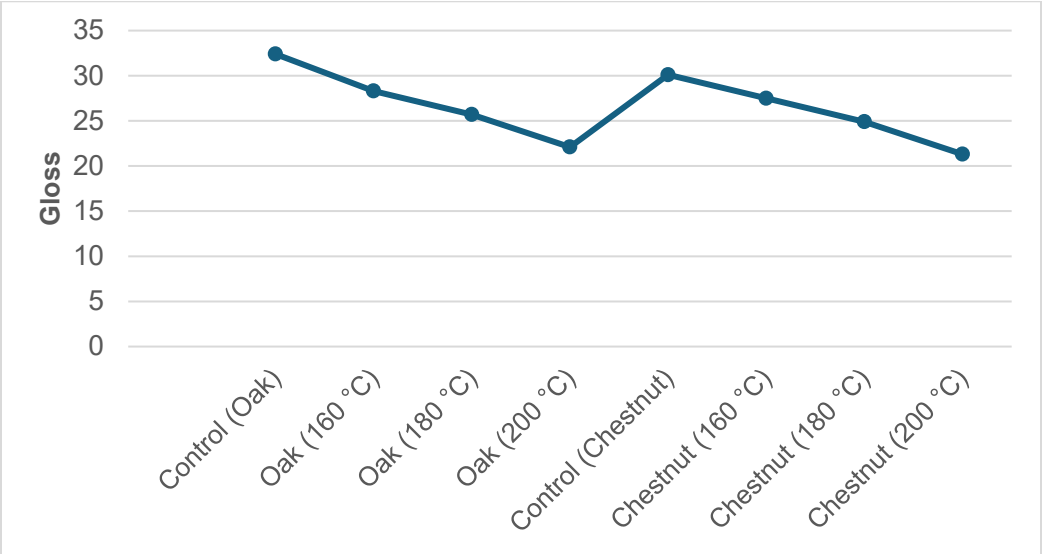


Fig. 2. Effect of heat treatment on gloss

Surface Roughness

The results regarding the changes in surface roughness (R_a) of oak (*Quercus petraea*) and chestnut (*Castanea sativa*) wood species after heat treatment are presented in Table 4.

Table 4. Results of the Changes in Surface Roughness Based-on Wood Type and Heat Treatment Temperature

Samples	Temperature (°C)	Time (h)	R_a (Mean)	Std. Dev.	H.G.
Control (Oak)	-	-	4.12	0.18	A
Oak	160	2	4.65	0.21	B
Oak	180	2	5.38	0.25	C
Oak	200	2	6.14	0.32	D
Control (Chestnut)	-	-	3.85	0.16	A
Chestnut	160	2	4.41	0.19	B
Chestnut	180	2	5.15	0.24	C
Chestnut	200	2	5.98	0.29	D
*Note: Std. Dev.: Standard deviation; and H.G.: Homogeneity group.					

Wood surface quality depends mainly on the wood structure and the implementation of wood mechanical processing procedures, such as cutting, sanding, finishing, painting, curving, applying preservation methods, adhesives, coatings, other substance layers, *etc*. (Magoss 2008; Vassiliou *et al*. 2016; Li *et al*. 2018; Kamperidou *et al.* 2020). According to the results obtained in this work, it was determined that the heat treatment temperature has a statistically significant effect on the surface roughness (R_a). The lowest roughness values, as expected, were measured in the untreated control samples (4.12 µm for Oak, 3.85 µm for Chestnut). On the other hand, the highest roughness values were determined in the samples heat treated at 200 °C (6.14 µm for oak, 5.98 µm for chestnut).

In both oak and chestnut woods, it was observed that the surface roughness values

increase gradually and significantly as the heat treatment temperature increases. Duncan test results also showed that this increase creates statistically different groups (A, B, C, D) at each temperature level.

The main reason for this increase in surface roughness is the chemical and physical degradations that occur on the wood surface during heat treatment. In particular, the breakdown of hemicellulose polymers, which are the most thermally unstable, leads to the weakening of the cell wall structure and the formation of micro-cracks on the surface. In addition, partial collapses in the cell structure and loosened surface fibers are directly measured by the profilometer as higher roughness values.

These results are in full agreement with many studies reporting that heat treatment increases surface roughness (Hidayat *et al*. 2017). On the other hand, the fragility of wood often leads to greater unwanted plucking of the fibres (torn fibres), which causes an increase in roughness after processing thermally modified wood (Gurau *et al*. 2017). In this study, for oak and chestnut, the increases in roughness were consistent with the results obtained by Bembenek and Pawlik (2021). In terms of the total amount of irregularity, the values of the R_z parameter also increased in a statistically significant manner (Lo Monaco *et al*. 2020). The present findings are consistent with those of Lo Monaco *et al*. (2020).

Among the wood species, it was observed that oak wood was naturally slightly rougher than chestnut wood in the control groups, but both species responded to heat treatment in a similar manner with increasing surface roughness. Although the present study did not directly measure the relative contributions of physical and chemical mechanisms, the literature suggests that hemicellulose degradation leads to surface weakening (chemical effects), while cell wall collapse and fiber relaxation (physical effects) become more pronounced at higher processing temperatures. Therefore, both mechanisms act synergistically, with chemical degradation facilitating subsequent physical deformation.

The graph showing the effect of heat treatment on surface roughness change is given in Fig. 3.

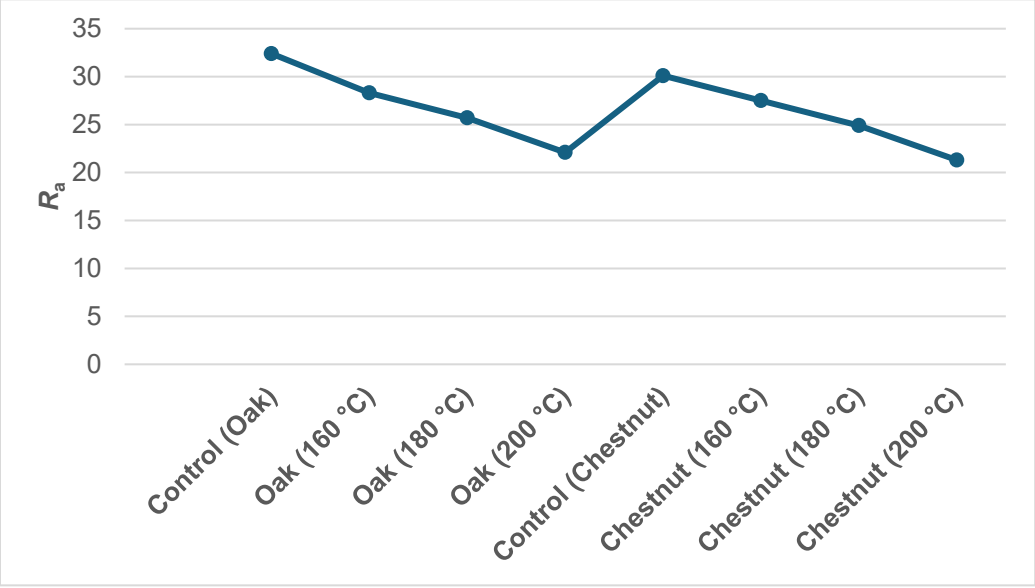


Fig. 3. Effect of heat treatment on roughness

Images of the samples after heat treatment are given in Fig. 4.

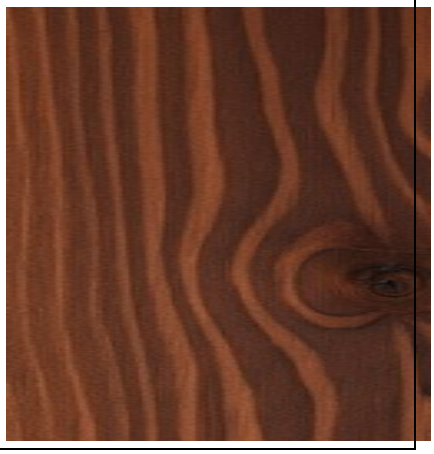
Temperature	Oak	Chestnut
160 °C		
180 °C		
200 °C		

Fig. 4. Images of samples after heat treatment

Adhesion Strength

The findings regarding the change in cohesion (MPa) of oak (*Quercus petraea*) and chestnut (*Castanea sativa*) woods after heat treatment and vegetable oil coating process are presented in Table 5.

The Duncan test results given in Table 5 show the effects of heat treatment and wood type on the adhesion strength with LO. In the study, the highest adhesion strength was measured in oak samples heat treated at 160 °C for 2 h with 6.8 MPa and coated with LO and PVAc varnish. As the temperature increased, the adhesion strength decreased gradually in both wood types. Furthermore, chestnut wood gave higher values than oak wood at similar temperature levels in both wood species. Oak woods heat treated and coated at 160 °C and 180 °C were in the (homogeneity A) group and did not show a statistically significant difference. There was a 45.6% decrease in oak wood samples treated at 200 °C compared to oak wood samples treated at 160 °C. In addition, there was a 37.0% decrease in chestnut wood samples treated at 200 °C compared to chestnut wood samples treated at 160 °C. Chestnut wood samples heat treated and coated at 180 and 200 °C were in the same homogeneity group and did not show a statistically significant difference.

Table 5. Results of the Adhesion Changes of Wood Type and Heat Treatment Temperature

Samples	Temperature (°C)	Time (h)	Coating	MPa (Mean)	Std. Dev.	H.G.
Oak	160	2	LO+ PVAc	6.8	0,09	A
Oak	180	2	LO+ PVAc	5.4	1.12	A
Oak	200	2	LO+ PVAc	3.7	0,08	B
Chestnut	160	2	LO+ PVAc	7.3	1.45	B
Chestnut	180	2	LO+ PVAc	5.8	1.75	C
Chestnut	200	2	LO+ PVAc	4.6	0,06	C
*Note: LO: Linseed Oil; Std. Dev.: Standard deviation; and H.G.: Homogeneity group						

The main reason for this significant decrease in adhesion strength is that heat treatment fundamentally changes the chemistry and energy of the wood surface. During heat treatment, hemicelluloses, the most hydrophilic (water-loving) component of wood, are degraded. This degradation significantly reduces the number of polar hydroxyl (-OH) groups required for the adhesion of water-based varnish. As the polarity of the surface decreases, it becomes more hydrophobic (water-repellent). Water-based varnish, which has a polar structure, may not be able to sufficiently wet this hydrophobic surface and cannot spread on the surface. It is well known that the composition of linseed oil changes gradually over time. The multiple unsaturated carbon-carbon bonds in the alkenyl chains can be oxidized and cross-linked. A consequence of oxidation, especially after the linseed oil has fully cured, is somewhat higher wettability (Hubbe and Laleicke 2025). Insufficient wetting results in a weak interfacial bond between the varnish and the wood, resulting in poor adhesion performance. These findings are in line with studies indicating that heat treatment negatively affects coating performance by changing the chemical structure of the surface (Gérardin *et al*. 2007; Nejad and Cooper 2011). Furthermore, if the moisture content of the wood is very high, the varnish layer may not adhere sufficiently to the wood surface (Budakçı and Sönmez 2011). There are very limited studies in the literature on the adhesion performance of wood material coated with vegetable oils after heat treatment. In general, there are studies in the literature on the adhesion performance of wood coated with varnish after heat treatment. In the study of thermally modified Türkiye oak wood finished with LO, a decrease in adhesion was observed with an increasing wood thermal modification temperature (Vidholdová *et al.* 2021). This decrease in adhesion performance

with increasing heat treatment temperature is consistent with the results obtained from our study. While the present study focused primarily on macroscopic adhesion results, we recognize that additional microscopic and chemical evidence could further support these interpretations. In particular, microscopic imaging (*e.g.*, SEM) can reveal fiber pullout or interface fracture patterns, while wettability measurements (contact angle) and spectroscopic analyses (*e.g.*, FTIR) can provide direct confirmation of heat-induced chemical changes that influence adhesion. These analyses are beyond the scope of our current study but offer valuable directions for future studies to deepen the mechanistic understanding of adhesion in thermally modified tannin-rich woods. In another study, Kesik and Ayyıldız (2015) investigated that effects of heat treatment on the adhesion strength of water based wood varnish were studied using four tree species woods (Anatolian black pine (*Pinus nigra* J.F. Arnold subsp. *nigra* var. *caramenica* (Loudon) Rehder), Calabrian pine (*Pinus brutia* Ten.), sessile oak (*Quercus petraea* Liebl.), and sweet chestnut (*Castanea sativa* Mill.)) and two components water based varnish (one-component semi-matte and two-component glossy water-based varnishes). Under atmospheric pressure, the wood samples were subjected to three different temperatures (130, 180, and 230 °C) at two different time intervals (2 and 8 h). After that, the wood samples were coated with two components water-based varnish. Their results showed that the adhesion strength of all wood types decreased with increasing temperature and time. In our study, the findings obtained are consistent with the findings obtained by Kesik and Ayyıldız (2015) as the adhesion strength of the samples decreased as the temperature increased. Özalp *et al*. (2009) found that the lowest varnishes' surface adhesion strength obtained higher heat treatment time and temperature. They also suggest that the heating process should not be applied where the sticking resistance is important. The graph showing the effect of the vegetable-based coating process on the adhesion change after heat treatment is given in Fig. 5.

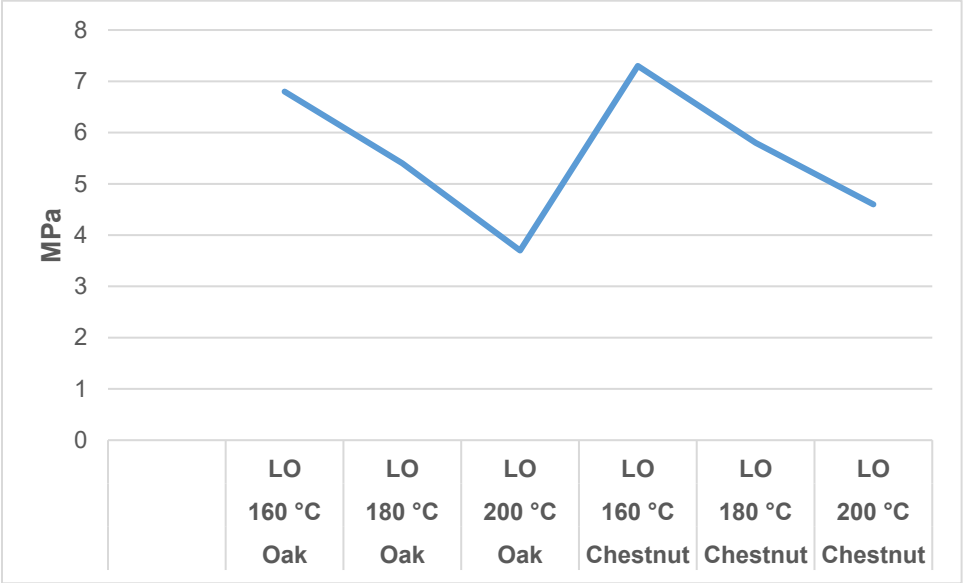


Fig. 5. Effect of heat treatment and coating on adhesion strength

The adhesion strength of thermally modified wood is directly related to the reduction of tannins on the wood surface. This is a key finding that addresses the central question of this study.

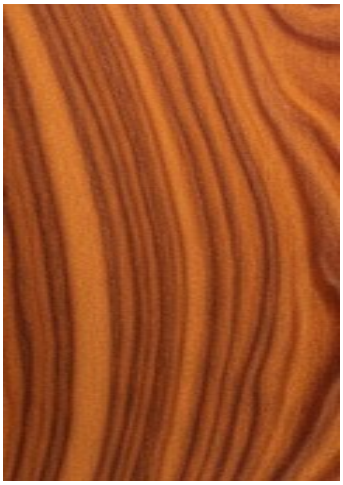
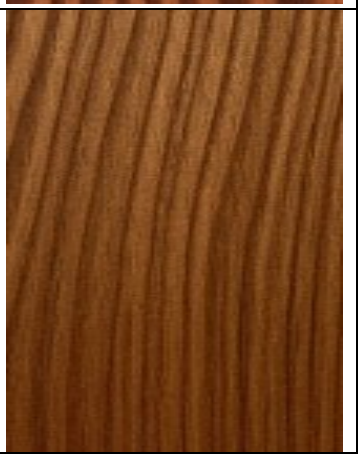
Temperature	Varnish	Oak	Chestnut
160 °C	LO+PVAc		
180 °C	LO+PVAc		
200 °C	LO+PVAc		

Fig. 6. Images of samples heat treated and then coated with linseed oil

The results demonstrated that as heat treatment temperature increased, the amount of extractives, including tannins, on the wood surface decreased significantly. This reduction mitigates the negative effects of tannins on adhesive performance, such as interference with adhesive curing and the formation of a weak boundary layer. Furthermore, the thermal modification process transforms the surface chemistry of the

wood, making it more hydrophobic. This change, coupled with the reduction in surface tannins, creates a more favorable bonding environment for adhesives. The linseed oil treatment further enhances this effect by acting as a sealant and reducing the interaction of residual tannins with the adhesive, leading to an overall increase in bond strength. Therefore, the improved adhesion strength observed in this work was judged to be a direct consequence of the thermal-induced reduction of tannins on the wood surface. Images of samples that were heat treated and then coated with LO are given in Fig. 6.

CONCLUSIONS

This study investigated the effects of heat treatments applied to tannin-rich sessile oak and Anatolian chestnut woods at different temperatures (160, 180, and 200 °C) on surface properties (color, gloss, and roughness) and examined the subsequent influence of linseed oil coating on adhesion performance. The experimental findings clearly demonstrated that increasing heat treatment temperature led to darker color, higher roughness, and lower gloss and adhesion strength. Statistical analyses (ANOVA and Duncan tests) confirmed that these changes were significant across all treatment levels. The graphical presentations of color changes, gloss, roughness, and adhesion strength consistently revealed a temperature-dependent trend, with the most pronounced changes occurring at 200 °C. From an industrial perspective, the combination of a moderate heat treatment temperature (160 °C) followed by linseed oil primer coating can be expected to provide the most balanced performance in terms of surface stability and adhesion strength. This recommendation is particularly relevant for furniture and coating applications where both aesthetic and mechanical performance are required.

The innovative contribution of this work lies in the systematic evaluation of thermal modification and natural oil coating specifically for tannin-rich hardwoods such as oak and chestnut. While previous studies often focused either on heat treatment alone or on coatings, this study provides an integrated analysis linking chemical degradation, physical surface changes, and adhesion behavior. Future studies incorporating microscopic imaging, wettability tests, and spectroscopic analyses are encouraged to further validate these interpretations and to deepen the mechanistic understanding of adhesion in thermally modified tannin-rich woods.

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