Effects of Shellac Treatment on Wood Hygroscopicity, Dimensional Stability and Thermostability

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Abstract: Dimensional stability proves to be an important factor affecting the quality of wooden products. As a sort of crude and thermoplastic resin, shellac excreted by lac insects demonstrates water-repellent and environmental-friendly features. The research impregnated shellac with wood at room temperature and with a vacuum-pressure procedure. Efforts were made to examine how shellac treatment affected the dimensional stability, moisture absorption, chemical structure, thermostability, as well as morphological characteristics of wood. Results indicated that shellac treatment was a type of efficient solution useful in the enhancement of the dimensional stability of wood. Shellac solution had good permeability in the wood, and the weight percentage gain reached 13.01% after impregnation. The swelling coefficients of treated wood in the tangential and radial directions decreased by 20.13% and 24.12%, respectively, indicating that impregnation could improve wood dimensional stability. The moisture absorption of shellac-treated wood was reduced by 38.15% under 20 °C and 65% relative humidity. Moreover, shellac treatment significantly modified wood structure, although there were no drastic changes in the spectra. Specimens for shellac treatment ran across two decomposition peaks at 350 and 390 °C, and specimens in the control group saw one more common derivative thermogravimetric curve when the sharp peak approached 355 °C. After impregnation of shellac into wood, the shellac blocked pits and hardened on the intrinsic layer of the wood for fear of hygroscopicity. The practice was applicable to a variety of wood products, such as buildings, furniture, and landscape architecture.

Keywords: shellac treatment; swelling coefficients; thermostability; wood dimensional stability

1. Introduction

Wood has a porous structure and is made up of natural, organic macromolecular materials [1–3]. Consequently, when the temperature and humidity of surroundings change, wood can absorb or release moisture [4]. This general ability of wood to adjust to the environment has made it an important material in the field of building engineering, furniture manufacturing, and renovation [5,6] among other recent applications. However, if wood has defects, such as cracks, as moisture is absorbed and
released, it can be transformed and decay [7], which significantly impairs the life span and utilisability of wood products and narrows the scope of application.

Reinforcing the dimensional stability, hydrophobicity, and durability of wood by reducing its hygroscopicity has become a major focus of research in recent years [8–12]. Reductions in hygroscopicity may be realized through a variety of means, such as steam-heat treatment [13–15], acetylation and silylation treatment [16], methyltrimethoxysilane modification [17], styrene modification [18], boron and compatibilizer modification [19], siloxane modification [20], alkoxysilanes modification [21], silicone oil heat treatment [6,22], and tricine and bicine modification [23]. However, thermal processing is likely to darken the original color of wood, undermine its mechanical functions [1,24–26], and induce chemical modifications that are environmentally deleterious. Besides that, existing methods of improvement are quite complicated and energy-intensive.

Shellac belongs to a natural, thermoplastic resin secreted by lac insects that mostly grows on host trees across countries such as China, India, Malaysia, and Thailand [27]. Shellac is a natural mixture consisting of resinous material and odoriferous composite containing waxy components, and a combination of both polar and nonpolar dyes. The resin portion of shellac contains esters (approximately 30%, soft resin) and polyesters (approximately 70%, hard resin) originating from sesquiterpenic and hydroxy fatty acids [28–32]. Nevertheless, the composition of shellac varies relying on the species of insects and host tree where the raw material is obtained [32]. Shellac has been adopted for centuries in the art sector, primarily as a varnish to protect the surface of wooden artworks, such as wooden furniture restoration and musical instruments; for its ability to form films; ease of application; high adhesiveness to wooden surfaces; aesthetic appearance of its coatings; protective properties; and low toxicity [5,33]. More recently, shellac has been applied in a wide variety of domains (pharmaceuticals, food treatment, toys for children, electrical insulation) due to its peculiar characteristics, namely low toxicity, thermoplasticity, insulating properties [5,34–37]. In addition to these features, shellac is characterized by good film forming and protective properties, ease of application, and a high adhesion to the wood surface and provides aesthetically excellent coatings [37], although, to date, it has not been commonly applied to promote the moisture absorption of wooden products.

Following a recent study examining the dimensional stability of Tung oil-treated wood [38], this paper examines the efficacy of using shellac as an environmentally friendly modifier to improve the dimensional stability and hygroscopicity of wood. Specifically, a shellac alcohol solution was used as a modifier to impregnate wood at room temperature, and then, the experiment tested how the shellac affected the dimensional stability and physicochemical structures, and thermostability of wood.

2. Materials and Methods

2.1. Sample Preparation

Ailanthus (Ailanthus Desf.), a white-colored wood with a similar grain to a white oak commonly endemic to Sichuan Province of China was used. Specimens collected for the test were prepared from sapwood with dimensions of 20 × 20 × 20 mm³ (length × width × thickness) and a primary moisture content of 70% ± 5% in accordance with the GB/T 1931–2009 criterion [39]. The drying treatment was conducted at 103 °C.

2.2. Impregnating the Wood with Shellac

Shellac (98% purity, wax free) and ethyl alcohol (90%) were provided by Chuxiong DES Shellac Co., Ltd. headquartered in Chuxiong, Yunnan of China. Shellac was treated by impregnation inside one vacuum chamber in Shanghai Laboratory Instrumental Works Co., Ltd., Shanghai of China. Twenty dry specimens were weighed, immersed in shellac ethyl alcohol solution (20%), and processed inside one vacuum chamber under 0.01 MPa pressure at room temperature (20 °C) for 1.5 h. Then, pressure was allowed to equilibrate to atmospheric levels for 1.5 h, followed by vacuuming and performance of the reverse process three times to obtain samples impregnated with shellac solution. By contrast,
there was no impregnation operation in the control group. Specimens went through natural drying for 7 days until the alcohol evaporated completely and air-dried at 103 °C to take measurements of the oven-dried weight.

2.3. Weight Percentage Gain (WPG)

The determination of WPG depends on the weight before and after impregnation according to Equation (1) as below:

\[
WPG = \frac{w_w - w_0}{w_0} \times 100\% \tag{1}
\]

where \(w_0\) suggests the pretreatment oven-dried weight of specimens (g), while \(w_w\) states the posttreatment oven-dried weight of specimens (g). Then, a comparative study was performed on the treatment method and the t-test at \(p = 0.05\) in SAS (v. 9.4, SAS Institute, Cary, NC, USA).

2.4. Estimation of Wood Dimensional Stability

Swelling tests adhered to the GB/T 1931–2009 criterion \[40\]. Specimens from the treated group and the control group were all conserved in the temperature control chamber at 20 °C with 65% humidity after drying, so as to obtain the equilibrium moisture content (EMC). The scales and weights of the specimens were estimated before and after conditioning. Equation (2) was the algorithm for the swelling coefficient \[39\]:

\[
a = \frac{l_w - l_0}{l_0} \times 100\% \tag{2}
\]

where \(a\) means the swelling coefficient at radial, tangential, and longitudinal directions; \(l_0\) illustrates primary dimension of specimens; and \(l_w\) suggests the postconditioning dimension. Treatment was performed to compare with the t-test in SAS at \(p = 0.05\).

2.5. Moisture Absorption (MA)

The specimens were placed inside the chamber with the temperature presetting as the constant of 20 °C and humidity as 65% to meet the EMC as stipulated by the GB/T 1931–2009 criterion \[40\]. Following conditioning operations, the MA would be calculated via Equation (3):

\[
MA = \frac{w_a - w_b}{w_b} \times 100\% \tag{3}
\]

where \(w_b\) \((w_a)\) means denote the specimen weight before and after conditioning in the climate chamber (g). Treatment was performed to compare with the t-test in SAS at \(p = 0.05\).

2.6. Chemical Structure Analysis Using FTIR Spectroscopy

Both ATR-IR spectra of the control and shellac-treated wood milled specimens were collected using a standard FTIR spectrometer (Tensor 27, Bruker, Germany) via straightforward transmittance with the resolution of 4 cm\(^{-1}\) for 32 scans in a span of 700–4000 cm\(^{-1}\). Adjustment on light devices and background spectra was performed before measurements were taken. The spectra were averaged over six measurements for each treatment.

2.7. Thermogravimetric Analysis

For comparing the degradation properties of wood and shellac-treated wood, the experiment adopted one thermogravimetric analyzer developed by Netzsch STA449F3 in Germany. The wood and treated wood powder were heated at a speed of 10 °C/min in a nitrogen environment, with a final temperature of 800 °C.
2.8. Morphological Characteristics

The macroshape of specimens as well as variation in physical structure were assessed via morphological observations made with a scanning electron microscope (SEM) produced by Hitachi S-3400N II in Tokyo of Japan.

3. Results and Discussion

3.1. Weight Percentage Gain (WPG)

WPG indicates the net weight of the wood-extracted shellac (Table 1). WPG ultimately depends on the permeability of the shellac solution inside the *Ailanthus* wood. The oven-dried weight of unrefined specimens was 4.636 g, the average posttreatment oven-dried weight with shellac was approximately 5.239 g, and the WPG was 13.01% (Table 1). The specimen weights before and after shellac solution impregnation are shown in Table 1.

| Table 1. Weights of specimens before and after shellac solution impregnation. |
|-----------------------------|-----------------------------|-----------------------------|
| Wood Sample | Before Impregnation | After Impregnation |
| Weight (g) | 4.636 ± 0.221 | 5.239 ± 0.319 |

3.2. Estimation of Wood Dimensional Stability and Moisture Absorption (MA)

3.2.1. Wood Dimensional Stability

The dimensional stability of wood is an important factor affecting its utilization and quality, but this property depends on a variety of factors, such as tree species, tree age, and age of the wood. The tangential and radial swelling coefficients are more sensitive than those in the longitudinal direction and are thus important for estimating the dimensional stability of wood. Figure 1 shows that the shellac solution impregnating treatment influenced the dimensional stability of wood. The mean of the tangential swelling coefficient in the control group totaled 3.08%, while that in the treated group was just 2.46%. In comparison with the control group, the postshellac treatment tangential swelling coefficient in the treated group was lowered by 20.13%. The mean of the radial swelling coefficient in the control group and treated group was 2.28% and 1.73%, respectively. The radial swelling coefficient in the control group was 24.12% lower than that in the treated group. For this reason, shellac treatment clearly reduced the wood swelling coefficients in the tangential and radial direction but did not drastically influence the wood swelling coefficient in longitudinal direction (*p* = 0.1112). The pattern makes sense, given that wood tends to shrink and swell in the tangential and radial direction but remain stable in the longitudinal direction. The increase in wood dimensional stability stems from the fact that shellac is an excellent water repellent [40].

3.2.2. Moisture Absorption (MA)

Moisture content is an important factor affecting wood stability when it is below the threshold of fiber saturation. In order to further elucidate the effect of shellac solution impregnation on wood dimensional stability, the hydrophilicity of wood was evaluated via the measurement of MA. Figure 2 shows the specimen weight before and after conditioning inside one climate chamber, with the temperature set as 20 °C and humidity as 65%. The specimen weights before and after conditioning were 5.239 g and 5.178 g, respectively, and the MA was approximately 11.69%. In comparison with the control group, MA in treated group had reduced by 38.15%. In consequence, shellac treatment significantly lowered wood MA performance and thus promoted wood dimensional stability.
Wood dimensional stability is related to its hydrophilic chemical composition, which generally contains hydroxyl groups and other chemical components. By reducing these components, the dimensional stability of wood is improved [41,42]. FTIR-ATR spectrometry has been widely employed to reveal chemical changes associated with various treatments. Infrared spectra are sensitive indicators of chemical changes, as demonstrated by previous research on wood treatments [43–46]. Figure 3 presents the FTIR spectra for shellac, shellac treatment specimens, as well as the control group. In comparison with the control group, the specimen chemical structure after shellac treatment in the treated group was adjusted, with spectra remaining constant. The weakening of band intensity at \(1384 \text{ cm}^{-1}\) and \(2900 \text{ cm}^{-1}\) was matched with \(-\text{OH}\) stretching, while bands at \(1709 \text{ cm}^{-1}\) and \(1631 \text{ cm}^{-1}\) were allocated to \(\text{C}=\text{O}\) and aromatic carbon skeleton stretching vibration, separately. No changes occurred in the other chemical groups after shellac treatment. Though shellac treatment changed the chemical groups of wood to some extent, the overall structure of the wood remained unchanged.

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Figure 3. FTIR spectra of the samples treated by shellac and those in the control group.

3.4. Thermostability

In order to evaluate the thermal characteristics of wood before and after shellac solution impregnation treatment, the thermogravimetric (TG) and derivative thermogravimetric (DTG) curves of the specimens in treated group and control group were constructed, respectively, as shown in Figure 4. The first region expressed as “a” in the TG curve is referred to as the depolymerization and dehydration stage, which implies the presence of slight weight loss (approximately 6–8 wt%) in 30–120 °C for entire samples. During the stage, wood specimens may take in heat, leading to evaporation and loss of low-weight molecules in specimens. Additionally, no evidence saw an apparent degradation of wood components [44–46]. In regard to region “b”, with temperature varying in the range of 120 to 230 °C, wood components such as hemicellulose were possibly degraded [2,24,49,50], but those with sophisticated structures were very likely to have bond fragmentation. Most quality loss can be seen in region “c”, with temperature varying in the range of 230–490 °C. For region “c”, overall structural components, accounting for approximately 80% of the gross mass in specimens experienced thermal degradation. Additionally, in region “c”, specimens after shellac treatment had a different reaction, and two peaks occurred at 350 and 390 °C, respectively. On the contrary, control specimens in the more common DTG curve saw a sharp peak at 355 °C due to cellulose decomposition [51]. In region “d”, the gross quality loss of specimens was slow. In this stage, residuals in the treated group dropped by 12.01% in comparison with that in the control group (16.30 wt%). Reductions in residuals may be related to the degradation of most shellac, but several wood constituents were nondegradable.

Figure 4. Thermogravimetric (TG) and derivative thermogravimetric (DTG) curves of untreated wood samples and samples treated with shellac.
3.5. Morphology

SEM micrographs of the specimens in the treated group and control group are presented in Figure 5. Shellac treatment was found to be greatly influential in specimen structure, especially wood impregnated with shellac solution. Pits were the main moisture passages inside wood; these pits had to be blocked in the treated group, preventing MA in the condition that wooded product were placed in the environment, which improved wood dimensional stability. Shellac could, therefore, prevent MA when it covered intrinsic layers of wood, thereby enhancing wood dimensional stability.

![Figure 5. SEM micrographs of the control group (left) and the shellac treatment group (right).](image)

4. Conclusions

Shellac solution impregnation treatment seems to be a useful means in the enhancement of wood stability. The shellac solution had good permeability in the wood, and the WPG reached 13.01% after impregnation. After treatment of shellac, the swelling coefficients of wood in the tangential and radial directions decreased by 20.13% and 24.12%, respectively, improving wood dimensional stability. The MA of shellac-treated wood was reduced by 38.15% with humidity set as 65% and temperature as 20 °C. Moreover, the shellac treatment slightly altered wood structure, although there were no changes in the spectra. Specimens under shellac treatment experienced two decomposition peaks at 350 and 390 °C, respectively, and those in the control group witnessed one more common DTG curve in which the sharp peak occurred at 355°C in the TG analysis. Shellac impregnated with wood blocked the pits used as prime water passages and then hardened on the internal layer of the wood after ethanol vaporization, which prevented MA in the application of wooden products. Furthermore, shellac modification was performed at room temperature. Thus, shellac modification has become a solution featured by energy conservation and environmental friendly properties that warrants further study.

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