

Article

Highly Hydrophobic and Self-Cleaning Heat-Treated *Larix* spp. Prepared by TiO₂ and ZnO Particles onto Wood Surface

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Abstract: The deposition of TiO₂/ZnO on heat-treated wood was prepared by a hydrothermal reaction and sol-gel method. Highly hydrophobic wood was successfully prepared with low surface free energy. The surface-modified wood samples were characterized by 3D-laser shape measurement microscopy, scanning electron microscopy, energy-dispersive spectroscopy, and Fourier transform infrared spectroscopy for the microstructure and chemical composition investigation. The deposited TiO₂ or ZnO markedly made the wood surface brighter, which was demonstrated by visual observation and spectrophotometer. The TiO₂/ZnO particles were successfully loaded onto the surface of the wood, proven by SEM-EDS and FTIR analyses. The contact angle of TiO₂ and ZnO-modified wood reached 123.9° and 134.1° respectively, which is obviously higher than that of the control at 88.9°. The hydrophobic properties of the TiO₂/ZnO modified wood samples were directly related to the shapes of clusters and spheres of particles, which increased the roughness of the wood surface. This study shows the hydrophobic properties of the TiO₂/ZnO-modified wood and provides the color and roughness changes for the painting process of heat-treated wood.

Keywords: heat-treated wood; TiO₂; ZnO; hydrophobic; roughness

1. Introduction

Wood heat treatment is commonly used in furniture and timber architecture to solve the problem of poor dimensional stability, grey color, decay, and so on [1–3]. The heat-treated wood is exposed to ultraviolet radiation, rain, microorganism, termites, and attack by drilling animals for outdoor occasions [4–6], while inorganic-wood composites give new functions to wood. Synergies between wood and inorganic components enhance the thermal stability, color fastness, mechanical performance, and dimensional stability of the wood [7–9]. Previous studies have shown that hybrid inorganic–organic film can effectively reduce the moisture absorption of the wood [10]. Recently, wood can be used as a template for nucleation and growth of inorganic particles such as TiO₂ or SiO₂ in a water-based environment [11–13].

The inorganic particles are usually generated from their monomeric precursors containing reactive organic groups to build up chemical bonds to the hydroxyl groups of wood [14]. A monomer precursor forms inorganic particles and reactive organic groups by hydrolysis reaction, which forms intact and solid coatings by continuous condensation reactions and are covered on wood [15,16]. Nano-TiO₂ is widely used in solar cells and photocatalysis; it is also used for improving fire resistance as well as the antifungal and antiweathering properties of wood. In these applications, the role of TiO₂ is directly determined by the morphological and the size of the particles [17].

Heat treatment significantly improves the dimensional stability, durability, and color of the wood. Therefore, heat-treated wood is widely used in indoor and outdoor applications, such as

landscape boards, fences, stairs, furniture, and other decorating cladding materials [18–20]. The outdoor environment (solar radiation, temperature, and humidity changes) significantly affects the color and chemical compositions of heat-treated wood [21,22]. The UV resistance of heat-treated wood was improved by the nano-TiO₂ or ZnO coating modification in a previous study [23,24]; changes to color and hydrophobic properties of heat-treated wood prepared by TiO₂ and ZnO particles research is rarely reported. The changes of color and hydrophobic properties play an important role in the painting process.

The purpose of this paper is to study the change of surface color, contact angle, and roughness of heat-treated wood before and after TiO₂ or ZnO deposition, all of which supply the fundamental parameters for the painting process of landscape boards and other materials.

2. Materials and Methods

2.1. Materials

Larix gmelinii (Rupr.) is a fast-growing species that is widely planted in Northern and Eastern China. A 15-year-old *Larix* spp. was purchased from Harbin, Heilongjiang Province, China. The samples were well prepared from the 2.5–3 m height of the same log. The dimension of the samples are 20 mm × 20 mm × 20 mm (longitudinal, radial, and tangential dimensions, respectively). The samples were firstly oven dried and then placed in a plastic bag for heat treatment.

2.2. Heat Treatment

The wood samples were heat-treated at 190 °C for 6 h with nitrogen as the shield medium, as in our previous study [25]. After heat treatment, the samples were cooled and placed in a plastic bag for surface modification, shown in Table 1. The temperature and duration time used in the heat treatment process are commonly used as modification to improve the dimensional stability of wood.

Table 1. The process of the surface-modified wood.

Process	Heat Treatment	Surface Modification
Control	Heat-treated at 190 °C for 6 h	-
TiO ₂ modified	Heat-treated at 190 °C for 6 h	Surface modification of the TiO ₂ nanoparticles
ZnO modified	Heat-treated at 190 °C for 6 h	Surface modification of the ZnO nanoparticles

2.3. Surface Modification of the TiO₂ Particles

A hydrothermal method was used for the TiO₂ surface modification of the wood, where 3 g surfactants (P123, analytical pure, Sigma-Aldrich Corporation, Saint Louis, MO, USA) was added in a 100 mL HCl solution (2 mol/mL), stirring for 2 h. Then, Ti(OBu)₄ was quickly added, and stirring was performed for 1 h. The heat-treated wood samples were immersed in this solution into a 150 mL stainless steel hydrothermal reactor lined with Teflon. The hydrothermal reactor was sealed in the oven and heated at 150 °C for 8 h. Then, it was naturally cooled after removal, and wood samples were oven dried at 80 °C for 24 h.

2.4. Surface Modification of the ZnO Particles

Solution A: 0.01 mol ZnAc and 0.03 mol NaOH were dissolved in 1 L methanol at room temperature and stirred at 60 °C for 2 h.

Solution B: 0.025 mol ZnNO₃ and 0.025 mol hexamethylenetetramine were dissolved in 1 L water stirred for 1 h at room temperature.

Heat-treated wood samples were immersed into the solution A for 4 h. The samples were removed and oven dried at 103 °C for 3 h. Then samples were immersed into the solution B at 90 °C for 4 h and rinsed with distilled water for 4 min. Finally, the samples were oven dried at 60 °C for 3 h.

2.5. Wood Photography

Wood samples were oven dried, and photos were taken under the same natural light environment by iPhone X.

2.6. Roughness

The 3D-laser shape measurement microscopy system (VK-X150, Keyence Company, Shanghai, China) was used to evaluate and analyze the roughness parameter (Ra) of the wood. A three-dimensional measurement analysis can be carried out directly to complete the measurement and evaluation of line roughness and surface roughness. The surface roughness was evaluated by an arithmetical mean deviation of the profile (Ra) measured by laser mode. Then, 200, 400, and 1000 magnification were selected to measure and analyze the surface roughness of the wood. The 16 bit ultra-fine CMOS image system (65536 grayscale) is composed of an induction photomultiplier and a confocal-ND filter optical system. Testing the roughness of the wood samples was performed 3 times.

2.7. Colormeter

A handheld spectrophotometer (Konica Minolta Sensing, Inc., Osaka, Japan D5003908) was equipped with an integral bulb spectrophotometer based on the CIE $L^*a^*b^*$ color spectrophotometer (ICCC). We tested 10 different positions on the tangential sections of the wood samples, and their average values were obtained by using a round hole probe with a colorimeter diameter of 8 mm. Calibration of the device was performed before each chromaticity test. Colors are represented by brightness and chromaticity, where chromaticity is the color constant that reflects the hue and saturation of the color.

$$\Delta L^*_t = L^*_t - L^*_0 \quad (1)$$

$$\Delta a^*_t = a^*_t - a^*_0 \quad (2)$$

$$\Delta b^*_t = b^*_t - b^*_0 \quad (3)$$

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

where L^*_0 , a^*_0 , and b^*_0 represent the color test value of the specimen, and L^*_t , a^*_t , and b^*_t represent the color test value of the specimen after surface modification. The 4 site locations of the measurements were 0.5 cm away from the edge of the tangential sections.

2.8. Scanning Electron Microscopy

Before analyzing the samples using SEM, the wood surface was coated with a layer of gold, the size of which was 8 mm × 8 mm × 3 mm for 110 s with an applied current of 23 mA. The sample was then mounted onto an aluminum pile with conductive paste. Surface morphology of the wood cell walls was determined by SEM (FEI, Quanta 200, Hillsboro, OR, USA).

2.9. Wood Contact Angles

The wood surface was characterized by the static contact angle under stable conditions (room temperature 20 ± 5 °C, relative humidity $65 \pm 10\%$). The wood samples were monitored by a video contact angle optical instrument (OCA20). The liquid was distilled water, and the volume of the droplet was 5 µL.

2.10. FTIR

Fourier infrared spectrometers (Waltham, MA 02451, USA) were equipped with attenuated total reflection (ATR) accessories. The wood samples were oven dried at 103 °C for 24 h and tested directly. The Fourier infrared spectrometer has a scanning wavelength of 4000–400 cm^{-1} , a scanning resolution

of 4 cm^{-1} , and a scanning number of 32 times. The chemical compositions of wood samples were measured by FTIR.

3. Results

3.1. Visual Observation

The visual and color changes of three sections of wood before and after being surface modified (TiO_2 or ZnO) are shown in Figure 1. After surface modification, the surface of three sections of all wood specimens faded and became brighter obviously. The surface-deposited TiO_2 or ZnO particles markedly changed the wood surfaces. The color changes in the ZnO -modified one was greater than the TiO_2 modified one. However, the visual changes of surface-modified wood are hard to analyze only by visual observation, and thus further measurements were performed.

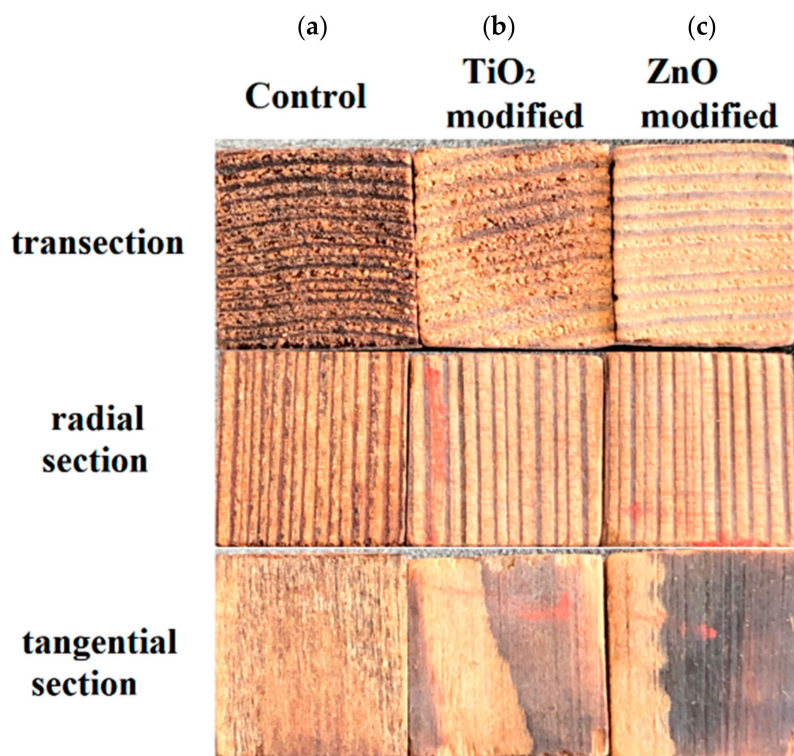


Figure 1. The photos of transection, radial and tangential sections of wood samples: (a) Control; (b) TiO_2 -modified wood; (c) ZnO -modified wood.

3.2. Color Changes

Figure 2 shows the lightness (L^*), redness (a^*), and yellowness (b^*) of the wood samples before and after surface modification. As shown in Figure 2, after surface modification of TiO_2 , the wood lightness increased significantly from 35.62 to 39.47; the red–green color index a^* decreased from 13.08 to 12.69, and the yellow–blue color index b^* increased from 13.61 to 17.63. The results showed that the surface modification of TiO_2 had slightly brightened and yellowed the surface of the wood samples, which were consistent with visual observation. After surface modification of ZnO , the lightness of the wood increased from 35.62 to 44.25; the red–green color index a^* decreased from 13.08 to 11.68, and the yellow–blue b^* color index increased from 13.61 to 15.28. This indicates that the wood samples became brighter and slightly greener after the surface modification of ZnO . TiO_2 or ZnO modifications had a certain impact on the color of the wood surface, which is slightly whitened. In general, the overall color changes were not significant, and the surface-modified wood basically retains its original grain and color.

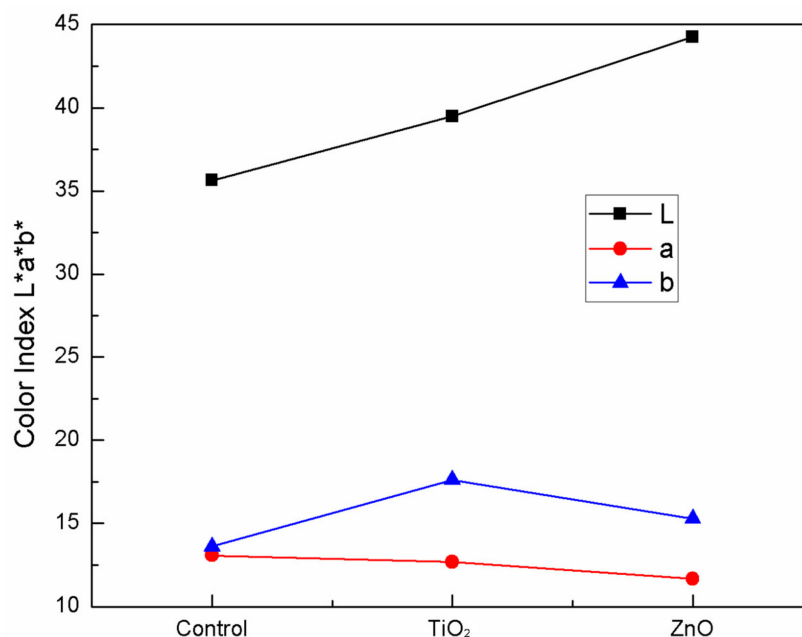


Figure 2. Changes of wood color index before and after surface modification.

3.3. Roughness

This study of wood roughness mainly focused on the changes of wood surface characteristics before and after surface modification, and comparisons were made with untreated wood. Effective surface modification improved fungal degradation, corrosion resistance, and color stability of wood in UV-VIS environment in previous studies [26–28]. Figure 3 shows the changes in the average surface roughness (Ra) of the wood before and after surface modifications. The wood roughness (Ra) increased slightly from $10.5 \pm 0.72 \mu\text{m}$ to $19.9 \pm 1.26 \mu\text{m}$ and $18.7 \pm 0.58 \mu\text{m}$ after TiO₂ and ZnO surface modification compared to untreated. This may be caused by the deposition of TiO₂ or ZnO particles on the wood surface. Overall, surface modifications had little effect on the roughness (Ra) of the wood, and the slight increase of roughness improves the bonding and coating performance of the wood [29].

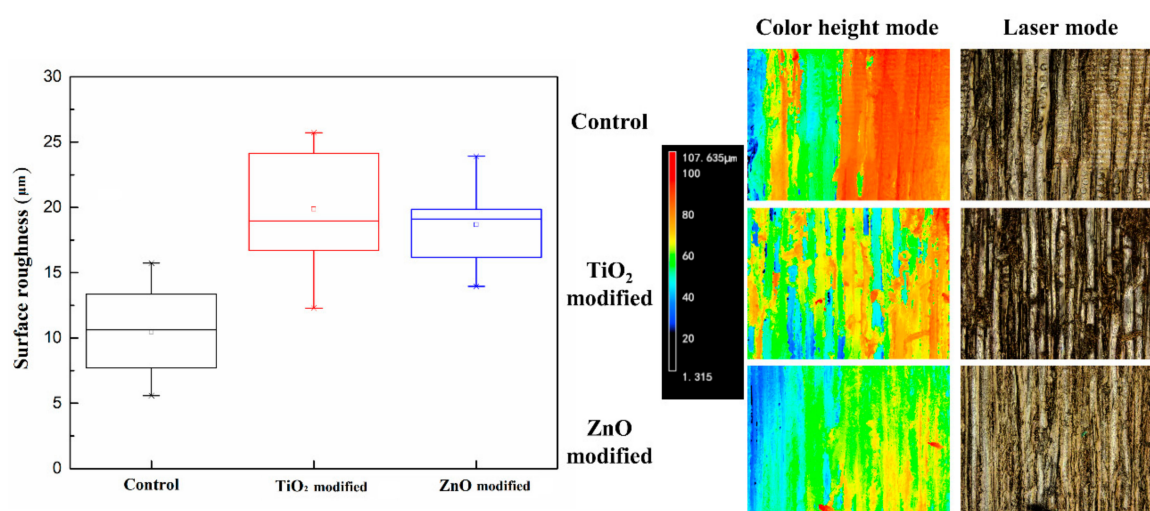


Figure 3. Roughness (Ra) of the wood samples before and after surface modification.

3.4. Microstructure

The morphology and chemical elemental compositions of the *Larix* wood before and after surface modification are presented in Figure 4. As shown in the SEM images of tangential sections of the wood,

abundant particles are present on the wood cell wall. The deposition made the wood cell wall uneven, which was also proven by a roughness test.

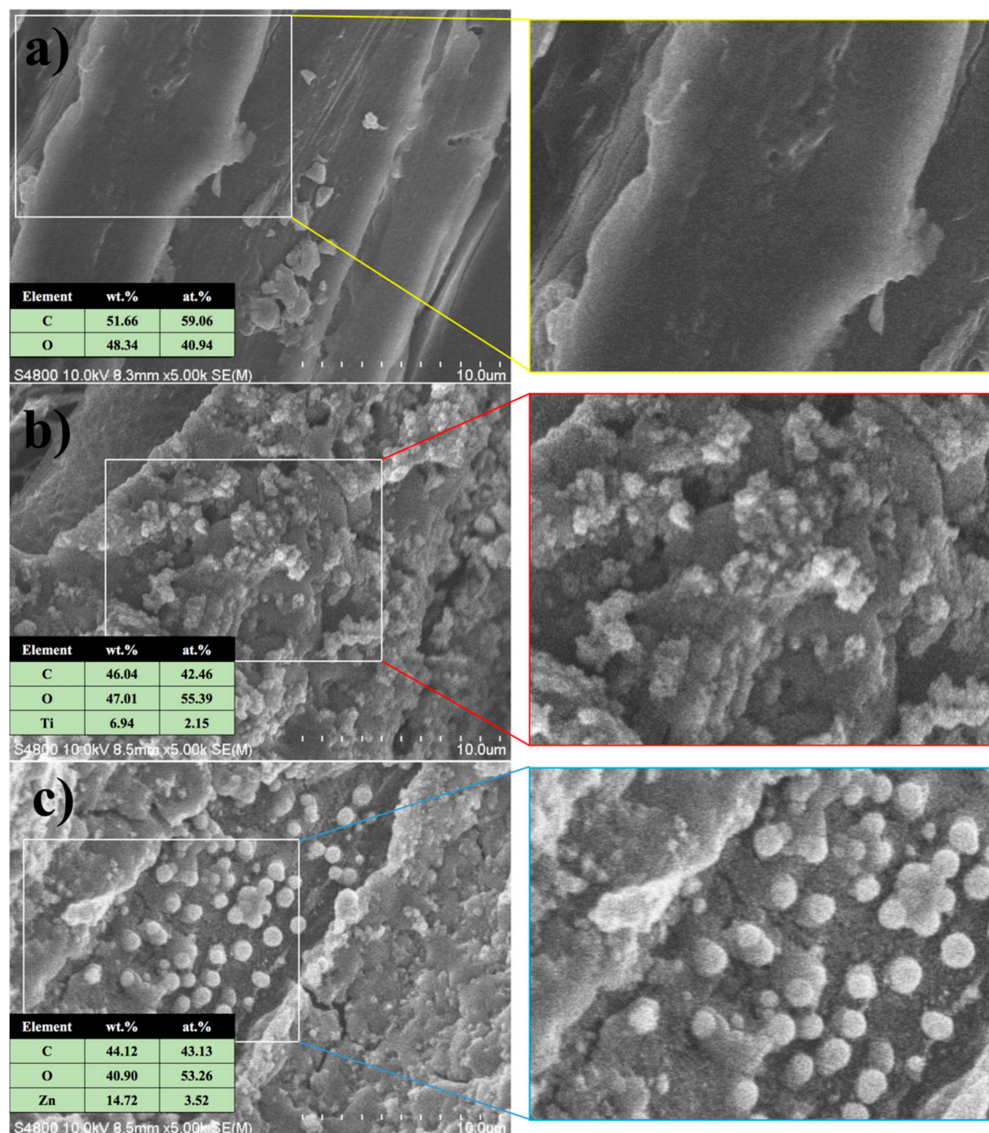


Figure 4. Scanning electron microscopy images of *Larix* spp. wood samples before and after surface modification: (a) Control; (b) TiO₂ modified wood; (c) ZnO modified wood.

The elements Ti and Zn were detected by SEM-EDS obviously, which proves that TiO₂ and ZnO were successfully loaded onto the surface of the wood cell wall. Based on the EDS measurement, the weight percentage of Ti on the wood surface was 6.94% and the weight percentage of Zn was 14.72%. The deposition of TiO₂ particles on the wood cell wall tended to clump together in clusters, while the deposition of ZnO particles formed spheres on the surface of the wood cell wall.

3.5. Contact Angle

The water contact angles (CA) of the wood before and after surface modifications are shown in Figure 5. The surfaces of the original wood showed hydrophilicity with a contact angle of 88.9°, which is most likely caused by heat treatment. However, after surface modification, the contact angles of TiO₂ and ZnO modification reached 123.9° and 134.1°, respectively. This indicates that the TiO₂ or ZnO

particle deposition enhanced the hydrophobicity of the wood surfaces, which is most likely caused by the formation of rougher structures on the wood surface [30].

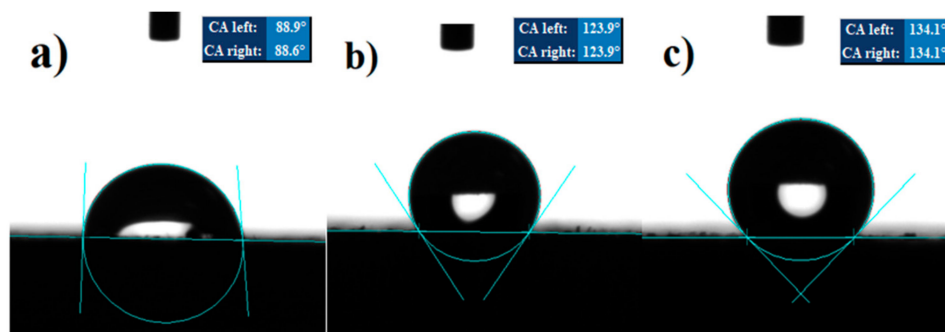


Figure 5. Water contact angles of (a) control; (b) TiO_2 modified wood; (c) ZnO modified wood.

Figure 6 shows the curves of the water contact angle of the wood and the surface-modified wood with time. Without surface modification, the water contact angle of the wood decreased rapidly. After 10 s, the CA was 29.3° and the CA was less than 20° after 40 s, which means the water was absorbed by the surface of the wood, while the CA of the TiO_2 and ZnO surface-modified wood decreased slowly and the CAs were still 113.1° and 127.6° after 60 s, respectively. This demonstrated that the surface-modified wood exhibited better water resistance varying over time. Compared with the untreated one, the heat-treated wood after surface modification is more conducive to the rapid drainage of rainwater.

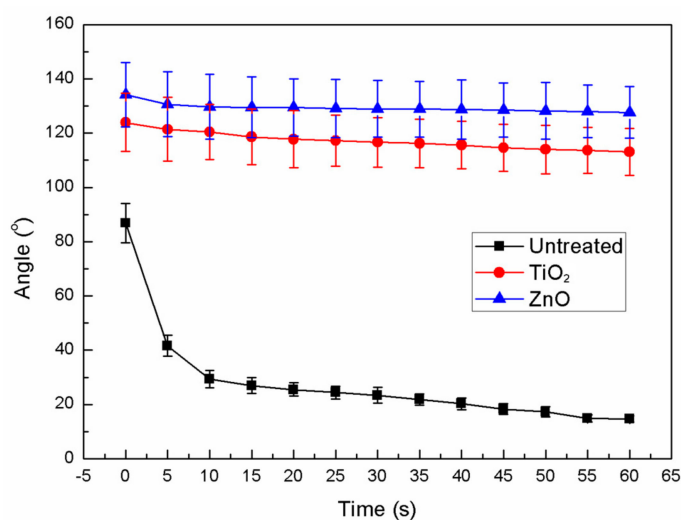


Figure 6. Curve of water contact angle of wood samples.

3.6. FTIR

The chemical composition of the untreated wood surface and the TiO_2/ZnO surface-modified wood was measured by the Fourier transform infrared, which is shown in Figure 7. For the spectra of the heat-treated wood, the typical absorption peaks are present, with the broad band in $3500\text{--}3200\text{ cm}^{-1}$ assigned to the O–H and N–H groups [31]. The absorption peak at 2900 cm^{-1} was assigned to the symmetric and asymmetric stretching vibrations of the –CH groups [32]. The C=O stretching vibration of lignin and the C–O–C stretching absorptions were observed at 1730 cm^{-1} and 833 cm^{-1} , respectively [33]. Compared to surface-modified wood samples, the FTIR-ATR spectra of the heat-treated wood are slightly different from those of the surface-modified ones. Compared to the control sample, the higher strength band of Ti–O was seen at $770\text{--}650\text{ cm}^{-1}$, indicating the deposition of TiO_2 on the wood

surface [34], which is also proven by the SEM-EDS analysis. The band at 430 cm^{-1} was assigned to the bonding of Zn–O, which proves the formation of ZnO on the wood surface [35].

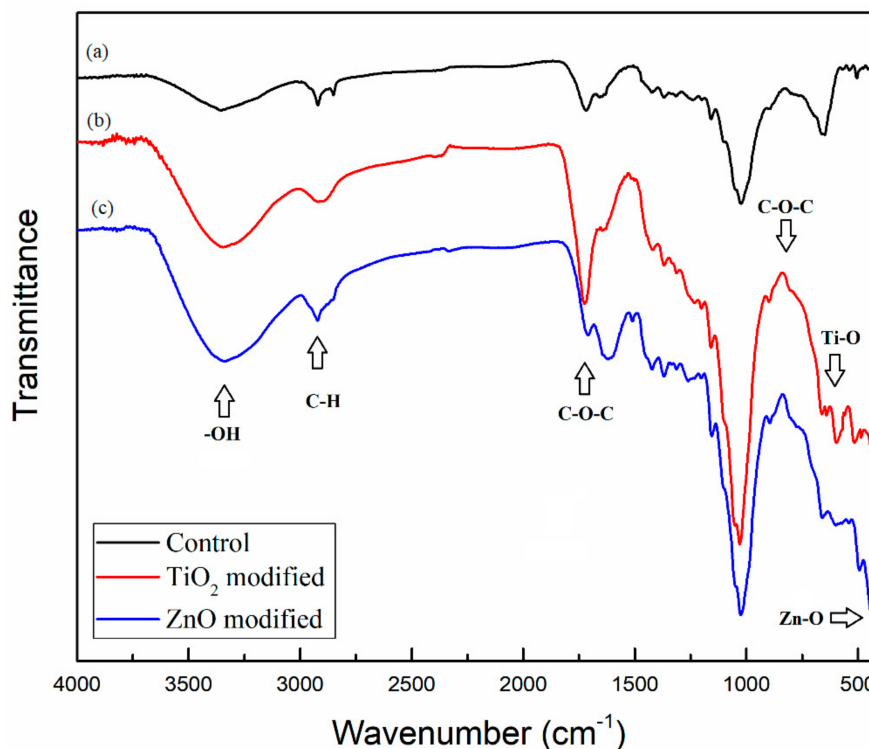


Figure 7. FTIR of *Larix* spp. wood samples before and after surface modification: (a) Control; (b) TiO_2 modified wood; (c) ZnO modified wood.

4. Conclusions

After the surface modification of TiO_2 and ZnO, the wood samples became brighter obviously, which was also proven by the CIElab color spectrophotometer analysis. The TiO_2 and ZnO particles were successfully deposited on the surface of heat-treated wood samples, which is proven by the SEM-EDS and FTIR analyses. This surface modification process achieved the uniformly dispersed nanoparticles on heat-treated wood surface. The deposition of TiO_2 particles clumped together in clusters, and the deposition of ZnO formed spherical particles. The TiO_2 and ZnO modifications increased the roughness of the wood, and it also made the surface of the heat-treated wood hydrophobic. This study revealed that the surface modifications of heat-treated wood have potentially achieved the hydrophobic and self-cleaning characteristics that are beneficial for exterior panels landscape, gardening materials, and timber architecture.

In this study, the hydrophobic characteristics of heat-treated wood were improved without significantly changing its original color. The surface modification is expected to be applied to the protection and restoration of ancient buildings by means of painting. This surface modification also improves the value and service life of heat-treated wood products. This study provides a theoretical basis for the realization of low-cost and high-efficiency industrial wood surface hydrophobic treatment.

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Conflicts of Interest: The authors declare no conflict of interest.

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