



## Full Length Article

# One-step approach to prepare superhydrophobic wood with enhanced mechanical and chemical durability: Driving of alkali

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## ABSTRACT

Practical application of superhydrophobic wood remain a great challenge because of their poor durability and complicated production procedures. In this work, high-wear-resistance superhydrophobic wood was prepared by a facile, alkali-driven method using SiO<sub>2</sub> nanoparticles (NPs) and vinyltriethoxysilane (VTES). The obtained wood had a contact angle (CA) of 156.6° and a sliding angle (SA) of 1.8°. It also exhibited a high tolerance to harsh mechanical damages such as sandpaper abrasion. The mechanical durability of material was intelligently controlled by tuning the amount of alkali. The results showed that the wood prepared using 24 mL of sodium hydroxide solution (0.1 mol/L) retained its original superhydrophobicity over an abrasion length of 270 cm, whereas that treated with the solution containing 6 mL of sodium hydroxide solution could only resist an abrasion length of 60 cm. Further examinations showed that the alkali treatment provides more hydroxyl groups on the wood surface. These hydroxyl groups readily catch hydrophobic SiO<sub>2</sub> NPs, forming a rough hierarchical structure that supported by SiO<sub>2</sub> NPs and micropores. The presence of the alkali ensures abundant SiO<sub>2</sub> NPs anchoring on the wood substrate via chemical bonding, which contributes to the formation of a superhydrophobic coating with sufficient thickness and high bonding strength in support of mechanical stability.

## 1. Introduction

As a natural and green material, wood possesses many special advantages. It is a renewable resource with aesthetic appeal, mechanical strength, thermal isolation, and interesting sound-absorption properties, which results in a wide arrange of applications in areas such as building and decoration, furniture production, and railway construction. However, wood easily absorbs water as a result of abundant hydroxyl groups on its surface. This moisture change leads to wood deformation, decay, and strength drop, significantly reducing the wood's service life. The design of artificial superhydrophobic surfaces on wood substrates has been considered as a promising method to enhance water repellency [1]. Superhydrophobic surfaces with water contact angles (CAs) larger than 150° and sliding angles (SAs) below 10° [2,3], have been used in many areas, including anti-freezing [4,5], self-cleaning [6,7], and anti-corrosive applications [8,9] as well as drag reduction [10] and oil–water separation [11–13]. Such surfaces need to be

textured with a hierarchical structure and then modified with low-surface-free-energy substances [14–15]. To fabricate the hierarchical structure, methods such as chemical vapor deposition [16], layer-by-layer assembly [17], and sol–gel [18], hydro-thermal [19,20], and electrochemical processes [21] have been reported. Materials such as organosilanes [22], perfluorinated compounds [23], fluorosilane [24], and stearic acid [25] have been used to perform the chemical modification.

On the basis of this design strategy, there are many papers reporting the successful preparation of superhydrophobic surfaces on wood. Gan et al. [26] fabricated superhydrophobic wood by depositing CoFe<sub>2</sub>O<sub>4</sub> nanoparticles on the wood surface and then modifying the CoFe<sub>2</sub>O<sub>4</sub> layer with a layer of octadecyltrichlorosilane (OTS). The obtained wood demonstrated superhydrophobic properties with a CA of 150°. Sun et al. [27] prepared a rough structure by growing ZnO nanorod arrays on the wood surface and then reduced the surface energy using a thin layer of n-dodecyltrimethoxysilane (DTMS). The final wood surface had a CA of

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about 156°. Wang et al. [28] fabricated a micro–nano rough structure using an immersion process followed by chemical modification with stearic acid; the CA of the as-prepared wood reached 151°. All of these superhydrophobic wood surfaces have been obtained by complicated processes, which highly limit their industrial production.

Recently, it has been possible to prepare superhydrophobic wood surfaces by simplified methods. For example, Liu et al. [29] fabricated such surfaces through a convenient solution-immersion method using potassium methyl silicate. Heish et al. [30] prepared superhydrophobic wood by simply spraying a mixture containing silica nanospheres and perfluoroalkyl methacrylic copolymer. Tu et al. [31] successfully fabricated a superhydrophobic wood coating by simple dip-coating. Most reports have focused on the preparation of a superhydrophobic surface on the wood substrate; however, the obtained materials usually have poor mechanical stability. Therefore, it is urgent to prepare durable superhydrophobic wood surfaces through a simple and efficient method.

Here, superhydrophobic wood has been prepared by a simple, one-step method. The obtained material has a contact angle of 156.6° and a sliding angle of 1.8°. It also demonstrates excellent mechanical stability, which is driven by an alkali. Also, the working mechanism of the alkali has been systematically studied by scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS), Fourier-transform infrared (FTIR) spectroscopy, and water contact angle measurements.

## 2. Experimental

### 2.1. Materials

Chinese fir (*cunninghamia lanceolata*), obtained from Guangzhou, Guangdong province, was processed into experimental samples (longitudinal × radial × tangential: 3 × 10 × 10 mm<sup>3</sup>). All chemical reagents were used as received, without any further purification. The cedarwood samples were ultrasonically rinsed with deionized water for 1 h and then dried in an oven at 60 °C for 24 h. Sodium hydroxide (NaOH, 96%) was supplied by Huihong Reagent Co. Ltd. (Hunan, China), and a 0.1 mol/L NaOH solution was prepared using deionized water. Vinyltriethoxysilane (VTES) was supplied by Antian Composite Technical Industry Co. Ltd. (Nanjing, China), the silica nanoparticles (commercial SiO<sub>2</sub> NPs, 35 nm average particle size) were obtained from Shuitian Material Co. Ltd. (Shanghai, China), and ethanol absolute (EtOH, AR) was supplied by Damao Chemical Reagent Co. Ltd. (Tianjin, China).

### 2.2. Preparation of the wood samples

The wood samples were prepared by a previously reported method [32]. First, the reactant solutions were prepared (the chemical compositions and different ratios are shown in Table 1). The specific sample preparation process is described as follows: The chemical components were first mixed in a beaker and then ultrasonically dispersed at 30 °C

**Table 1**  
Synthetic condition for preparing the samples.

Sample	CH <sub>3</sub> CH <sub>2</sub> OH (mL)	VTES (mL)	SiO <sub>2</sub> (g)	NaOH(mL)
S1	50	3	0	3
S2	50	0	0.2	3
S3	50	3	0.2	3
Wood-0	50	3	0.2	0
Wood-6	50	3	0.2	6
Wood-12	50	3	0.2	12
Wood-18	50	3	0.2	18
Wood-24	50	3	0.2	24
Wood-27	50	3	0.2	27
Wood-30	50	3	0.2	30

for 1.5 h. Then, the reactant solution (1.5 mL) was added into a weighing bottle containing the wood sample. It should be noted that the wood sample was completely immersed in the reactant solution. The weighing bottle was subsequently transferred to the 250 mL glass bottle, which was sealed with a lid. Finally, this glass bottle was heated in an oven at 100 °C for 1.5 h to obtain the sample.

### 2.3. Characterization

The CAs and SAs were measured using a 4 μL distilled water droplet on an OCA20 contact angle analyzer at room temperature. The CA and SA were obtained as averages of measurements taken from at least five different positions on each sample's surface. The surface morphology of the as-prepared wood surface was examined using a scanning electron microscope (Quanta 450, FEI) operated at 15.00 kV. Since wood has poor electrical conductivity, the sample surface was sputtered with a thin gold layer using a sputtering coater (SC7620, Quorum, UK). This treatment improved the conductivity of the samples. Surface chemical composition was determined by EDS (Oxford Instruments, INCA X-ACT250) and FTIR (IRAffinity-1, Shimadzu Corporation, Japan).

## 3. Results and discussion

### 3.1. Fabrication of superhydrophobic wood

It is well known that surface roughness is the most important factor for the successful fabrication of superhydrophobic surfaces. SEM was applied to study the surface morphology of the as-prepared samples. The morphology of a sample treated with VTES is shown in Fig. 1a. It can be seen that a smooth film covers the wood surface. The as-prepared wood had a CA of 115°, higher than that of untreated wood (80°, shown in the inset of Fig. 2b). The improvement in water repellency of the VTES-treated wood can be attributed to the modification of low-surface-energy materials, appearing as a smooth film (Fig. 1e). Owing to the lack of surface roughness, the VTES-treated wood demonstrated hydrophobic rather than superhydrophobic properties. The morphology of a sample that was treated only with SiO<sub>2</sub> is shown in Fig. 1b. As it can be seen, the SiO<sub>2</sub>-treated wood surface is covered by a thick and highly dense clay-like coating. In the higher magnification image (Fig. 1c), it was found that the SiO<sub>2</sub> piles were uneven, which improved the roughness of wood surface. However, the SiO<sub>2</sub>-treated wood surface shows superhydrophilicity, which is demonstrated by a CA of 0° (Fig. 1f), which is consistent with the result of previous literature [33]. It has been reported that superhydrophilicity results from the presence of plenty hydroxyl groups on nanosilica surfaces [34]. Fig. 1d shows the SEM image of a sample treated with VTES and silica. In contrast to the surface of the SiO<sub>2</sub>-treated wood, the VTES–SiO<sub>2</sub>-treated wood surface was covered with separate SiO<sub>2</sub> NPs. Moreover, micro/nanopores were created between the SiO<sub>2</sub> NPs. Thus, a rough structure was confirmed on the VTES–SiO<sub>2</sub>-treated wood surface. The obtained wood surface demonstrated superhydrophobicity with a CA of 150° and an SA of 4.8° (Fig. 1g). The surface morphology confirms the change in roughness of wood with different treatments. The wettability (CA, SA) indicates the change in surface chemistry of different wood samples. EDS spectra further confirms the different surface chemistry according to the changing atomic percentage of C element (Fig. 1h–k). Therefore, it can be safely concluded that the SiO<sub>2</sub> NPs and VTES afford a rough texture and a low-surface-energy layer, respectively.

The use of an alkaline solution also had a significant effect on the wettability of the as-prepared wood surfaces. A series of wood samples was prepared using different amounts of sodium hydroxide. As shown in Fig. 2, the CA of sample wood-0 decreased from 80° to 0°, whereas that of sample wood-6 increased to 150.8°. This shows that a superhydrophobic surface was obtained when sodium hydroxide was added to the reactant solution. Therefore, sodium hydroxide has a decisive effect on the preparation of superhydrophobic surfaces. With increasing

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