

## A Facile Method of Superhydrophobic Coating on Rubberwood to Improve its Anti-Mildew Performance

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Rubberwood, which can be regarded as a natural polymer composite, presents many difficulties in the process of material modification. In this study, a convenient method was developed to impart superhydrophobicity to the rubberwood surface. SiO<sub>2</sub>-PS composite film was formed on rubberwood surface by simple short-term impregnation. The obtained superhydrophobic rubberwood exhibited a water contact angle (WCA) of ~155.6° at room temperature. The superhydrophobic coating surface provides the abilities of self-cleaning and anti-bacteria for rubberwood. Combined with a small amount of 3-iodo-2-propyl-butyl carbamate (IPBC), the control effect of the four fungi reached 75% (*Botryodiplodia theobromae*), 100% (*Trichoderma viride*), 95% (*Penicillium citrinum*), and 100% (*Aspergillus niger*), respectively. The developed method herein features environment-friendly raw materials, facile processing, and large-scale fabrication. It provides a new solution for long-term timber protection processing.

*Keywords:* Superhydrophobic surface; Self-cleaning; Anti-bacteria; Rubberwood

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### INTRODUCTION

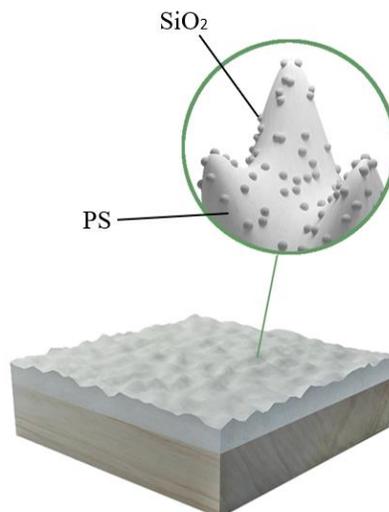
The rubber tree (*Hevea brasiliensis*) is widely planted in subtropical regions, such as Thailand, Malaysia, and Indonesia, as an economic tree species (Sik *et al.* 2010). Rubber plantations account for about 18% of global forest plantations (ITTO 2009). However, the use of rubberwood is limited because the wood is readily deteriorated by mold fungi, decay fungi, and insects (Ratnasingam *et al.* 2012). In order to expand the application range of rubber wood, it is important to protect the wood from degradation, mildew, and insects. For instance, studies have been conducted on utilizing essential oils to provide protection of rubberwood against decay (Matan 2012; Jantamas *et al.* 2016). To protect wood from mold fungi, many methods have been applied to deal with mold attack. At present, the anti-mold treatment of wood is mostly carried out together with the wood anti-degradation treatment, because moldy wood is often also susceptible to fungal attack. In the past, one such method for treating wood used chromated copper arsenate (CAA), which belongs to a group of inorganic waterborne preservatives. According to the relative proportions of metals, three CCA formulations are designated as three types A, B, and C. Though CCA was widely considered as the most effective wood protectant (Hingston *et al.* 2001), it has been banned all over the world due to its harm to humans and the environment (Balasoïu *et al.* 2001; Zagury *et al.* 2003).

As a substitute for CAA, the preservatives of copper as the main agent, including ammoniacal copper quaternary (ACQ), copper azole (CA), and copper dimethyldithiocarbamate (CDDC), show good protective effects on wood. Copper-based preservatives have been widely studied (Chung *et al.* 2005; Ye *et al.* 2015). However, copper ions will be gradually lost during the process, which will also cause serious pollution to the environment. Analogously, antiseptic boron compounds have not been shown to significantly affect the decay resistance of wood, and they present favorable environmental characteristics (Nami *et al.* 2007). However, borates are easily leached when treated woods are located in a relatively high humidity environment (Baysal *et al.* 2006). Several strategies have been proposed to resolve the above leaching problem of inorganic preservatives (Ratajczak and Mazela 2007; Obanda *et al.* 2008; Thévenon *et al.* 2010), such as micronized copper (McIntyre and Freeman 2009) and boron fixative agent (Mohareb *et al.* 2011). Although a large number of organic mold inhibitors, such as 4,5-dichloro-2-octyl-isothiazolo-lone (DCOIT) (Feng *et al.* 2017), tebuconazole 3-iodo-2-propyl-butyl carbamate (IPBC) (Muin and Tsunoda 2003), *etc.* (Sun *et al.* 2012), have been developed, they have problems of being degraded by microorganisms (Wallace *et al.* 2008). Meanwhile, they could also be decomposed under high humidity conditions (Schultz *et al.* 2007).

Wood has a strong hydrophilic nature, which not only leads to the size change of wood after exposure to water, but it also makes wood more susceptible to degradation by fungi and decay fungi (Kocaefer *et al.* 2015). In recent years, research on preparation of superhydrophobic structures has been widely carried out (Liu *et al.* 2015; Rao *et al.* 2016; Jia *et al.* 2018; Tu *et al.* 2018).

The superhydrophobic interface not only has the functions of water repellency, self-cleaning, anti-freezing, but also has certain antibacterial function. Superhydrophobic surfaces showing high resistance to bacterial contamination have been studied by many researchers (Heinonen *et al.* 2014; Feng *et al.* 2015). Superhydrophobic interfaces do not contain germicidal materials that destroy microorganisms. Instead, they prevent bacterial growth by reducing adhesion. Superhydrophobicity reduces adhesion between bacteria and a solid surface, making it easier to remove bacteria before they form a thick biofilm on the surface (Kartal *et al.* 2007; Zhang *et al.* 2013). In addition, superhydrophobic surfaces are found to have low protein adsorption and easy deproteinized properties (Koc *et al.* 2008). However, the existing methods of constructing hydrophobic structures are complex. In addition, there are no reports about the use of hydrophobic structures in conjunction with other antifungal agents.

Herein, we report a simple method to fabricate mechanically durable, self-cleaning, and multifunctional superhydrophobic surfaces on rubberwood by impregnating the specimens in the PS/THF solution and SiO<sub>2</sub>/acetone emulsion (Fig. 1). Superhydrophobic rubberwood is fabricated by nano-/micron- composite structure, which is combined with organic mildew inhibitor (IPBC) to improve its mildew resistance. Hydrophobic silica nanoparticles were partially embedded into PS micro-protrusion to construct robust superhydrophobic surfaces on the wood through a simple dissolution and resolidification method, which improved its water repellency and anti-biodeterioration performance. In addition, there is a process of simulating the loss process of mold inhibitor to demonstrate the auxiliary role of superhydrophobic interface in preventing wood. The film prevents moisture from contacting the wood while reducing the loss of fungicide in order to improve the wood's anti-degradation and anti-bacteria performances.



**Fig. 1.** Schematic diagram of super hydrophobic coating structure on rubber wood

## EXPERIMENTAL

### Materials

Rubber wood was purchased from Dongguan Jilong timber market. 3-iodo-2-propynyl butylcarbamate (IPBC, MW=281.09), polystyrene (PS, MW=104.14), and nano fumed silica ( $\text{SiO}_2$ , hydrophobic type-260, MW=60.08,  $\geq 99.8\%$ , particle size: 7-40 nm) were purchased from Aladdin Chemical Reagent Co. (Shanghai, China). Tetrahydrofuran (THF,  $\geq 99\%$ ) and acetone ( $\geq 99.5\%$ ) were purchased from Guangzhou chemical reagent factory (Guangzhou, China).

### *Fabrication of nanocomposite surfaces on wood substrates*

Rubber wood samples (20 $\times$ 20 $\times$ 20 mm) were dipped in the IPBC-EtOH solution, which was stirred for 2 h at a solid/liquid (g/mL) ratio of 1:1000, for 10 min at room temperature and pressure. Five grams PS and 50 mL THF were mixed in a 100 mL glass beaker for 20 min to form a homogeneous solution. Then the wood samples were dipped in the pre-PS solution for 5 s and kept at room temperature for 3 h to evaporate THF.  $\text{SiO}_2$  and acetone were stirred with a magnetic stirrer for 2 h. The emulsion was, respectively, divided into five different concentrations of 0.4, 0.8, 1.2, 1.6, and 2.0 wt%. The above wood samples were soaked in the emulsion again. Finally, the above samples were soaked in the emulsion for 5 s and then placed at room temperature for 3 h to obtain a nano-micro hierarchical structure.

### *Characterization*

The water contact angle (WCA) on the surface was measured using a contact angle system OCA (Dataphysics, OCA20, Germany) at ambient temperature. The WCA was recorded 10 s after a droplet of deionized water (5  $\mu\text{L}$ ) was placed on the surface. The microscopic morphology of the superhydrophobic surfaces was observed using field-emission scanning electron microscopy (FE-SEM, Hitachi SU-70, Japan) in high vacuum mode at a working voltage of 2.0 kV. Prior to the observation, thin aurum films were

sputtered on all samples by using a low-conductivity sputtering coater (Hitachi E-1010, Japan) to improve conductivity.

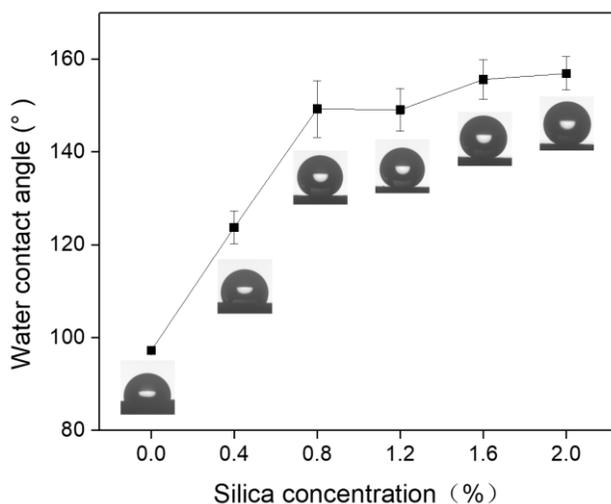
### Anti-mold test

In order to investigate the anti-mold performance of superhydrophobic rubberwood and the protective effect of superhydrophobic layer on IPBC, four groups of specimens, including control group (RW), superhydrophobic rubberwood (SRW), IPBC-treated rubberwood (IRW), and IPBC-treated superhydrophobic rubberwood (SIRW), were tested. Six wood samples of each type were used. Malt extract agar was used as the culture media for growing mold captured from air. Deionized water-mold solution was sprayed on the surface of specimens. In addition, IRW and SIRW primarily underwent a 72-h high humidity environment process (UVTest, ATLAS, Chicago, IL, USA), which was designed to spray pure water on the specimen surface for 5 min and then for the surface to be dried at 40 °C for 120 min. The proportion of the growth area of the mold on the surface of specimens was calculated as the criterion after 4 weeks.

## RESULTS AND DISCUSSION

### Hydrophobicity

As shown in Fig. 2, the average water contact angle (WCA) of the samples treated with PS was 126.1°, while that of the samples treated with 0.8% SiO<sub>2</sub>-acetone emulsion was 149.3°. When the concentration of silica was 1.6%, the WCA reached 155.6°. Therefore, the solid-liquid ratio of silicon dioxide particles to acetone is the key to controlling the microstructure and roughness of the hydrophobic surface, which is the basis for obtaining adjustable water repellency of treated wood.



**Fig. 2.** Variations of WCAs with silica nanoparticle contents in the acetone-SiO<sub>2</sub> emulsion

The prepared wood surface not only exhibited superhydrophobicity, but also had self-cleaning properties. As shown in Fig. 3, carbon-powder contaminates on the surface could be easily removed by dripping water onto the wood surface. When water droplets fell onto a slightly sloping surface, they rapidly rolled off the superhydrophobic wood surface because of the small sliding angle of the surface and removed surface contaminants.

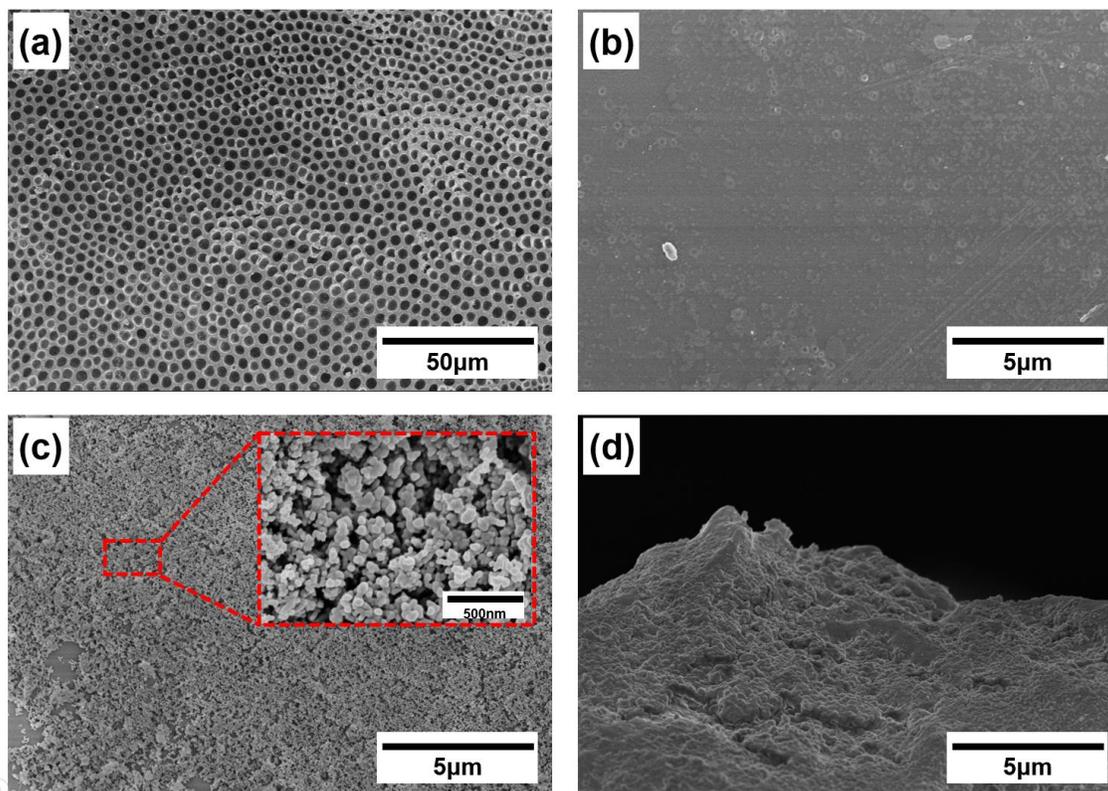


**Fig. 3.** Snapshots of the self-cleaning process on the superhydrophobic wood surfaces

### Microstructure and Surface Morphology

Figure 4a presents the morphology of the wood substrate after the evaporation process using only PS-THF solution, and the image was shot by FE-SEM. The figure shows that a honeycomb membrane was formed on the surface of specimen, which constitutes a coarse structure of micron size, after the evaporation process. The micro-scale roughness of the wood substrate was increased by PS-THF. The parenchyma cells were concealed by the PS layer ( $\sim 130^\circ$ ), which provides a degree of hydrophobicity. The wood substrate, however, was not thoroughly covered due to void structure. One serious drawback of this structure is that there are many pores in the membrane, which are the most likely locations for microorganisms to hide. Hence, the less soluble solvent acetone was used to redissolve the PS layer fixed on rubberwood surface to form a continuous dense film. Even at higher magnification, it is difficult to detect structural defects in the newly formed coating surface (Fig. 4b).

The contact angle of the coated rubberwood was  $100^\circ$ . Compared with PS-THF coating, this contact angle eliminates the pore structure, thereby reducing the probability of microbial adsorption. Although the pores of PS coating modified by acetone were reduced, their properties were also reduced accordingly. Therefore, nano-silica was embedded in PS coatings as an auxiliary, forming a new rough structure. In the process of acetone dissolving PS, the interface between the polymer and solvent behaved as a kind of viscous liquid. Thus, the silica nanoparticles added to acetone could be embedded in the interface overall or partially (Figs. 3c and 3d). A hierarchical structure was formed by reason of the partially-embedded nano-silica in relatively neater PS layer while the interface was resolidificated again. The addition of nanoparticles dramatically increased the roughness of specimens. Figures 4c and 4d show the morphology of the wood substrate subjected to the second dissolution-solidification using acetone. When the concentration of acetone-silicon suspension reached 1.6%, it can be seen that the rubberwood surface was covered almost completely with silica particles. This structure not only had good hydrophobicity, but also provided a more complete protection of the wood surface. A microscale obvious protuberance with multitude tiny peaks and valleys around it can be seen in Fig. 3d. The entire superhydrophobic layer was like a hill covered with trees, which provided an excellent multiple spatial structure, resulting in eminent hydrophobicity. Therefore, two times shaping treatment with PS-THF and silicon-acetone resulted in the formation of both micro- and nanohierarchical structures. Under the rough structure created in this study, water droplets in contact with the surface should be blocked by air retained in nano-grooves and particles, which prevents penetration of the water droplets into the wood surface. In addition, due to the absence of large-scale pores, microorganisms on the surface are easily carried away by water flow, making it difficult for them to reproduce and grow on the wood.



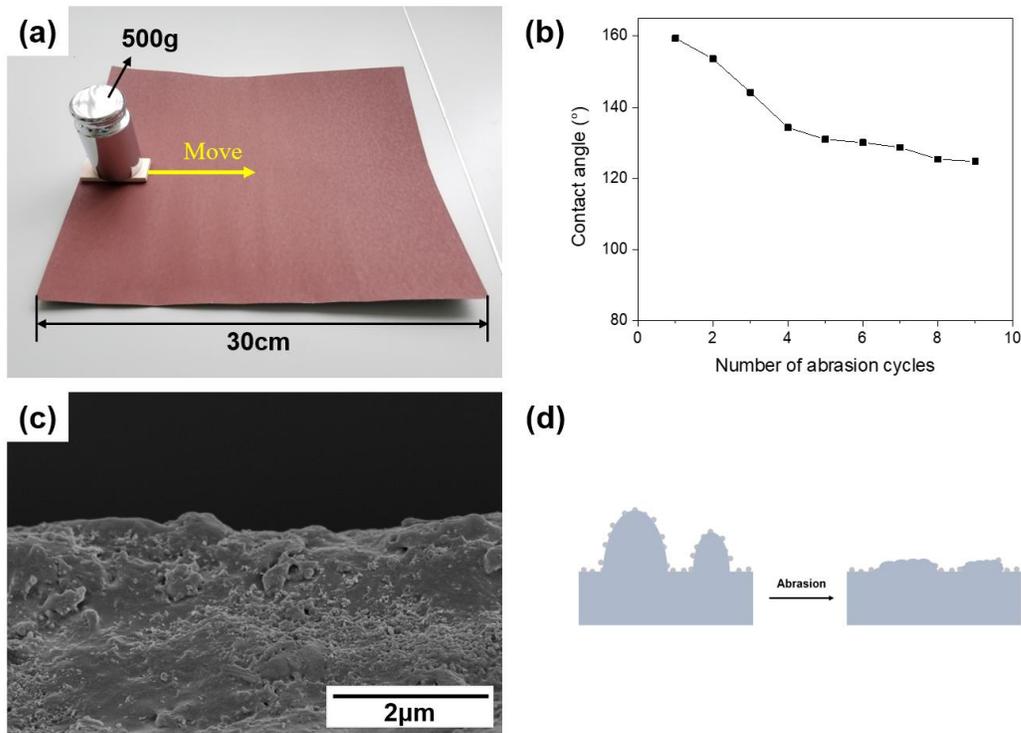
**Fig. 4.** FE-SEM images of (a) wood coated with PS, (b) PS-covered wood treated with acetone, and (c, d) modified wood with SiO<sub>2</sub>/acetone solid liquid ratio: 1.6 wt%

### Mechanical Durability of the Superhydrophobic Surface

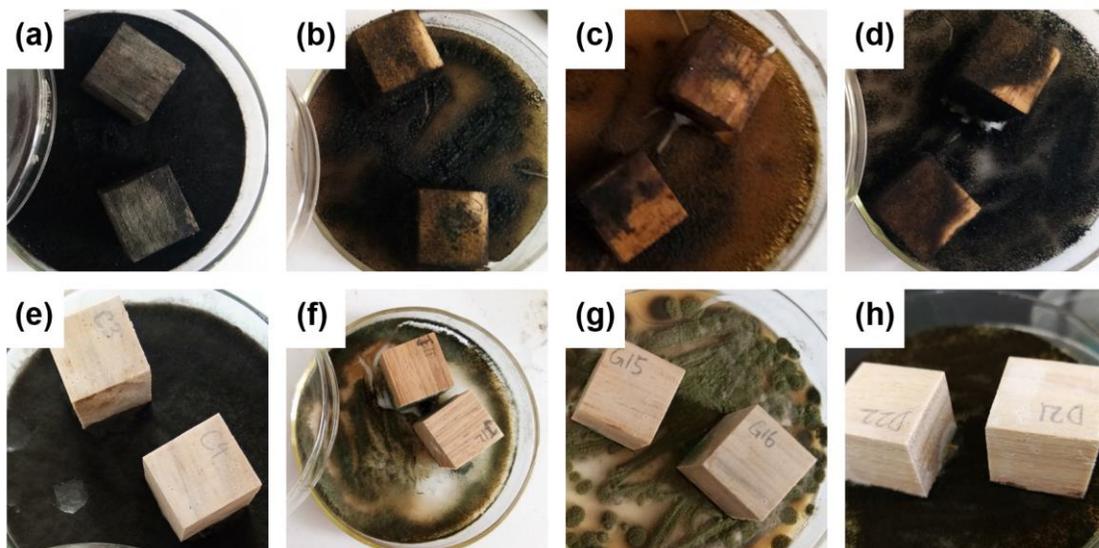
The main problem in the practical application of superhydrophobic wood is that the rough microstructure of the membrane is easily damaged by mechanical abrasion. Therefore, wear tests of super hydrophobic coatings on wood surface were carried out. As shown in Fig. 5a, a 500 g weight (5 kPa) was loaded on the resultant surface, facing 1500-mesh sandpaper as an abrading surface, and moved a distance of 15 cm along the ruler, which was applied to test the anti-abrasion ability. Figure 5b shows the change in CAs as a function of abrasion cycles for the prepared wood surface. It could be seen that the CAs of the treated wood decreased and remained at about 130° after being abraded over and over again (~1 to 9 times). After many times of wear, the microstructure of the hydrophobic layer was destroyed. As shown in Figure 5c and 5d, the "peak" of the film was severely depleted by mechanical friction, and the silica particles embedded therein was also lost. As a result, the surface was reduced to a similar topography of the micron structure built with a single PS. Nevertheless, it can still provide a hydrophobic function to prevent water from touching the following wood.

### Inhibition effect of superhydrophobic rubberwood on mold

Four kinds of molds were used in the mold proof experiment, namely *Botryodiplodia theobromae* (Fig. 6a and 6e), *Trichoderma viride* (Fig. 6b and 6f), *Penicillium citrinum* (Fig. 6c and 6g), and *Aspergillus niger* (Fig. 6d and 6h). The samples treated with IPBC-ethanol solution with concentration of 0.3% and 1.6% SiO<sub>2</sub>-acetone emulsion with concentration of 2.0% achieved the best mildew resistance, and the control efficacy of the four fungi reached 75%, 100%, 95%, and 100%, respectively.



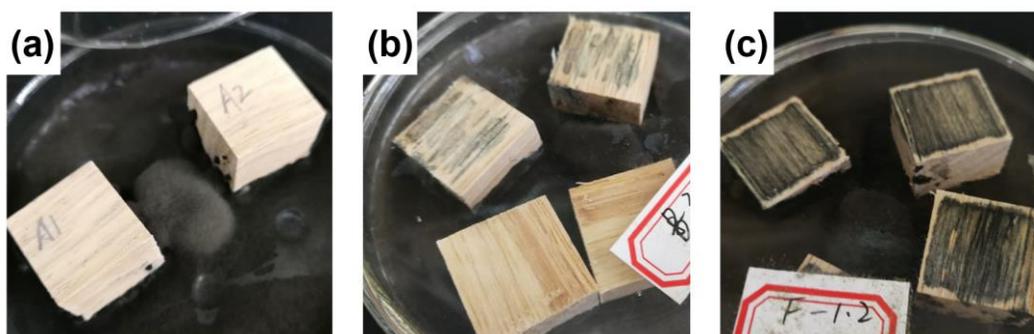
**Fig. 5.** (a) the setup used to test the mechanical stability of the prepared superhydrophobic wood against abrasion; (b) CAs as a function of number of abrasion cycles for the superhydrophobic wood surfaces; (c) SEM images of the rubber wood surface after 9 abrasion cycles; (d) schematic diagram of the abrasion-induced damage to the membrane on the wood surface



**Fig. 6.** Photograph of moldproof specimen, (a-d) RW, (e-h) SIRW

Figure 7a shows the condition of the test pieces in a mold environment. It can be seen that the surface of the superhydrophobic specimens remain dry due to the existence of a hydrophobic layer, which helps prevent the continuous growth of mold. Figure 7b and 7c show the internal conditions of the four specimens, respectively. There is no evidence of mold growth in the interior of SIRW (lower part of Fig. 7b). The diffusion of mold was inhibited on the surface of the specimen treated with IPBC only (upper part of Fig. 7b);

however, the mold gradually invaded the interior of the wood. This is mainly due to the fact that IPBC cannot enter the depth of the specimen only by soaking at normal temperature and pressure, and could only stay at a depth of about 6 mm. Without the protection of a superhydrophobic layer, organic fungicides would gradually leak out in the humid environment, leading to the possibility of mold invading the wood interior. Similarly, the specimen treated only by superhydrophobic treatment (upper part of Fig. 7c) also does not show a large number of mold on the surface, while a large number of mold appears inside. Although the superhydrophobic layer effectively inhibits the spread of mold on the surface, once mold spores reach the wood through the membrane, they begin to multiply inside. The control group (lower part of Fig. 7c) without any treatment was covered with mold inside and outside.



**Fig. 7.** Photograph of moldproof specimen, (a) the surface image of SIRW, (b) the internal image of IRW and SIRW (c) the internal image of SRW and RW

## CONCLUSIONS

1. Superhydrophobic films on rubberwood were prepared from polystyrene-tetrahydrofuran (PS-THF) solution and SiO<sub>2</sub>-acetone emulsion. Rubberwood with high hydrophilicity was transformed into superhydrophobic material has been developed by forming SiO<sub>2</sub>-PS composite film on rubberwood surface by simple short-term impregnation. The water contact angle (WCA) of the modified rubberwood reached 155.6°.
2. The samples treated with 3-iodo-2-propyl-butyl carbamate (IPBC)-ethanol solution with concentration of 0.3% and SiO<sub>2</sub>-acetone emulsion with concentration of 2.0% achieved the best test results., and the control efficacy of four fungi was 75% (*Botryodiplodia theobromae*), 100% (*Trichoderma viride*), 95% (*Penicillium citrinum*), and 100% (*Aspergillus niger*), respectively.
3. The treated rubberwood had excellent hydrophobicity, self-cleaning ability, and antibacterial properties, and presents broad application prospects in the field of outdoor wood materials.

## ACKNOWLEDGEMENTS

This work was financially supported by the Guangdong government science and technology project entitled, “Integration and demonstration of the key technology of

comprehensive processing and utilization of Thailand's rubberwood" (2017A050501030), as well as the Guangdong forestry science and technology innovation project entitled, "Study on oriented cultivation and efficient utilization of acacia of black wood" (2018KJCX004-03).

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Article submitted: May 13, 2019; Peer review completed: July 6, 2019; Revised version received and accepted: July 15, 2019; Published: July 18, 2019.  
DOI: 10.15376/biores.14.3.7111-7121