



Hierarchical mesopore wood filter membranes decorated with silver nanoparticles for straight-forward water purification

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Abstract Woods with a three-dimensional mesoporous structure comprise numerous oxygen-containing functional groups, which can be used to reduce metal nanoparticles in situ. Here, we report a new wood filter membrane which was combined with silver nanoparticles (Ag NPs) for water filtration. The wood blocks were immersed in silver nitrate solution to compose the Ag NP/wood membrane and were directly used for filtration sterilisation. In addition, the bacteriostatic effect of the Nano-silver wood filter membrane was measured by filtering the river water. In 5 L of the river water, after being filtered through three wood membranes at the same time, the bacteria and fungi were killed. Meanwhile, the bacteriostatic test corroborated that the diameters of the bacteriostatic circles of *Staphylococcus aureus*, *Escherichia coli*, *Pseudomonas aeruginosa*, *Bacillus subtilis* and *Candida albicans* were all larger than 1 cm. Therefore, Ag NP wood membranes possess good filtration and antibacterial properties.

Keywords Silver nanoparticles · Wood membrane · In situ reduction · Water treatment · Bacteriostatic activity

Introduction

Water is the source of life. Nonetheless, contaminated drinking water can lead to outbreaks of massive infectious and parasitic diseases, which remain one of the leading causes of death globally. How to obtain cheap point of use (POU) to obtain daily drinking water and reduce the probability of waterborne pathogenic microorganism-induced diseases must be investigated, especially in developing countries (Dankovich et al. 2016). Chlorination and ozonation are two of the conventional disinfection methods to control microbial water pollution. In addition, conventional disinfectants are usually strong oxidants that can react with many natural substances to produce toxic, harmful or even carcinogenic substances (Lemus Pérez and Rodríguez 2017). Therefore, acquiring intelligent POU methods to generate clean drinking water is an urgent issue that must be solved urgently worldwide. The use of green materials to prepare nano-materials for POU sterilisation systems by simple methods has attracted considerable interests (Baruwati et al. 2009; Ayad et al. 2018). Nano-materials with bactericidal functions, such as silver nanoparticles (Ag NPs) (Jędrzejczyk et al. 2018; Lin et al. 2013), carbon nanotubes (Bradyestévez et al. 2010) and copper nanoparticles (Dankovich and Smith 2014), have been used for POU water treatment. Ag NPs not only have strong inhibition and killing effects on dozens of pathogenic microorganisms, such as *Escherichia coli* and *Staphylococcus aureus*, but also

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can effectively promote tissue repair and regeneration at the same time. Moreover, nano-silver materials are generally used in water treatment (Dankovich et al. 2016), medical applications (Howes et al. 2014), freshness preservation, catalysis (Dong et al. 2015) and photonics (Choi et al. 2013) due to their excellent bactericidal properties. Ag NPs are currently the most widely used commercial nano-particle products given their wide spectrum, nondrug resistance and insensitivity to pH and other properties (Xiu et al. 2012). However, with the high surface energy of nanoparticles, agglomeration occurs easily under the influence of the Van der Waals force when the nanoparticles are close (Hakim et al. 2005). Consequently, Ag NPs have been bound to various antibacterial materials in different ways to enhance their biocidal efficiency. For instance, the antimicrobial activities of Ag-TiO₂ nanoparticles and complex of nano-silver and cationic polymers have been reinforced after embedding with Ag NPs (Li et al. 2011; Song et al. 2012). Therefore, rapid, green, efficient and recyclable processes into nano-silver material synthesis for removing bacteria and other microorganisms have attracted great interest (Alahmadi et al. 2018; Dankovich 2014).

Woods, as the most common structural materials, have the characteristics of being natural, pollution-free, simple and easy to obtain. Woods consist mainly of cellulose fibres, hemicellulose and lignin to form a special microporous 3D cavity network structure (Ermeydan et al. 2014). Normally, there are two major categories of wood, broad-leaved trees (hardwood) and coniferous wood (softwood). Here we used was the hardwood-longan wood. And the hardwood cell wall and cellulose aggregate into microfibrils made up the basic structure of the wood. The average diameter of the microfibrils was 16 nm, the minor diameter was between 5 and 10 nm, and its shape had elliptical spaces. These elliptical spaces consist the structure of the lignin molecules deposited afterwards. The point is that lignin has carbonyl, hydroxyl and aldehyde, and reductive functional groups can be used as for metal nanoparticle in situ synthesis (Mansouri and Salvadó 2007). Any wood has a natural 3D pore structure and water delivery capacity. Thus, it can be made into a high-efficiency device to support the synthesis of nanoparticles, disperse metal nanoparticles and purify water (Sens et al. 2015). And in this way, the hierarchical nanoscale structures in woods are preserved, and efficient water treatment equipment

can be obtained directly without the need for complex manufacturing processes. In addition, it has to be mentioned that a macro-tube carbon structure decorated with Nano-silver (Ag/C) can be synthesized by high temperature carbonation when the filtration and sterilization efficiency of filter membrane is very low (Ji et al. 2015). In this way, not only zero pollution, but also efficient utilization of Nano-silver wood again.

In this study, we invented an efficient wood filter device for water filtration sterilisation by in situ forming Ag NPs penetrated the inner pipeline of longan wood. The various types of tissues fulfil the functions of mechanical support, conduction of liquids and storage, and the dense distribution of channel networks in the wood makes the Ag NPs ubiquitous, which provides possibilities for the microbes in the water to contact with the anchored Ag NPs during water treatment. Moreover, the wood also has a water transfer characteristic, which ensures the wood membrane's longevity and stabilisation for water treatment. The Ag NPs have excellent bactericidal properties, which can protect the wood membrane from attacking by bacteria, fungi and insects. Furthermore, using wood as the carrier of Ag NPs not only can avoid agglomeration but also can filtrate and sterilise liquid. The use of hard wood as a nano-material carrier and a filter element is green, highly efficient, low cost and renewable. Therefore, the preparation and application of Nano-silver wood membranes has a broad application prospect.

Experiment

Materials, chemicals and bacterium and fungus species types

The basswood used in this study was cut from a local (Zhangzhou, Fujian, China) longan tree, and river water (Chiu-Lung River) was used as the filtered water during the water bacteriostatic treatment. In addition, the size of wood samples was 50 mm diameter × 5 mm thickness. Silver nitrate was the analytical reagent, and 15.2 mol/L hydrogen nitrate, 12 mol/L hydrochloric acid and 30% perhydrol were the guaranteed reagents. Moreover, silver nitrate, hydrogen nitrate, hydrochloric acid and perhydrol were purchased from Sinopharm Chemical Reagent Co., Ltd., China, while the Luria–Bertani (LB) culture

media and potato dextrose agar (PDA) culture media were purchased from Beijing Land Bridge Technology Co., Ltd., China. Furthermore, deionised water and ultrapure water were used as solvents. *S. aureus*, *Pseudomonas aeruginosa*, *E. coli*, *Bacillus subtilis* and *Candida albicans* were purchased from the Guangdong Culture Collection Center.

Preparation of Ag NP/wood membranes

The wood pieces were immersed in a AgNO_3 (5 mg mL^{-1}) solution and subjected to ultrasonic reaction for 30 min and heating in a water bath at 85°C for 14 h to prepare the Ag NPs/wood. The solution changed from being colourless to dark yellow turbid liquid, and the block turned into black.

Characterisation

The longan wood morphology was observed by high-magnification scanning electron microscopy (SEM) (JSM-6010LA, JEOL, Japan). The Ag NP/wood filter membrane morphology and particle size were characterised by transmission electron microscopy (TEM) (JEM 2100F, JEOL, Japan) at 200 kV, and the wood membrane samples for TEM test were prepared by mechanically grinding into 500 mesh powder. In addition, the powder was put in anhydrous ethanol and dispersed for 1 h to acquire dispersed Ag NPs, and the functional groups on the surface of materials were monitored by an IS 10 attenuated total reflection–Fourier transform infrared (FTIR) spectrometer (TA Co., Ltd., USA). Each spectrum ranging from 4000 to 400 cm^{-1} was scanned 32 times at a scanning resolution of 4 cm^{-1} , and the crystal structure of the materials was characterised by an Ultima IV X-ray diffractometer (XRD, Rigaku Corporation, Japan) from 5° to 80° at $4^\circ/\text{min}$. Furthermore, the amount of Ag in the wood membrane samples was measured by inductively coupled plasma mass spectrometry (ICP-MS, Agilent-7500cx, Agilent, America), and the samples for the ICP-MS test were treated by mechanically grinding the membrane into powder, dissolving 1.0000 g of the powder in 10 mL of hydrogen nitrate for 2 h and adding 2 mL of hydrogen peroxide for microwave digestion. Ultimately, the resultant solution was filtered by a $0.22 \mu\text{m}$ filter membrane and diluted to a ppb level of 100 for ICP-MS analysis.

Water sterilisation experiment

The bacteriostatic property of the nano-silver wood membrane was tested by filtrating the river water through it, and the continuous filtering of 5 L of river water with 1, 2, 3 and 4 pieces of wood was performed. Each filtrate (1 mL) was cultured on the LB and PDA culture media to test if bacteria or fungi survive, and the bacteria were cultured in the LB medium at 37°C for 24 h, whilst fungi were cultured in the PDA medium at 28°C for 48 h. In addition, the bacteriostatic inhibition zone experiment was used to prove the inhibition effect of Ag NPs/wood on five types of bacteria (*S. aureus*, *P. aeruginosa*, *E. coli*, *B. subtilis* and *C. albicans*), and the Ag NPs/wood was ground into 100 mesh powder, and 0.3000 g of the powder was added into 6 mm holes in the culture media of five different strains. Silver nitrate served as the positive control and aseptic water as the negative control. Moreover, the size of the inhibition zone was determined by the cross method after culturing in an incubator, and the inhibition zone $> 1 \text{ mm}$ means good bacteriostatic effects. Conversely, the inhibition zone $< 1 \text{ mm}$ means that the membrane has no bacteriostatic effect.

Stability of the Ag element in wood membranes

The binding stability of the Ag element in the Ag NP/wood was evaluated by testing the Ag elemental mass percentage before and after the water treatment by using ICP-MS measurements.

Results and discussion

Synthesis mechanism of Ag NP/wood membranes

Ag NPs in situ formed within the wood after the block immersed in the AgNO_3 solution, in which lignin acted as the reducing agent. Many scholars have corroborated that lignin is a natural phenolic resin and the second largest component of woods after cellulose. Lignin can be redox and depolymerised into aromatic compounds, which can significantly reduce the bond energy of the main chemical bonds and promote the conversion into highly functional lignin monomers, such as vanillin, eugenaldehyde and hypersandrin (Coccia et al. 2012; Mansouri and Salvadó 2007;

Simonović et al. 2011). In addition, the particular 3D structure of longan wood is used as a vessel to support the Ag NP growth everywhere that can enhance the antibacterial property of the wood membrane, and the entire wood block turned into black because of the plasmonic effect (Zhou et al. 2017). These special properties of lignin have also been proven to be useful for the in situ reduction of Pd(II) to Pd NPs in the wood for water treatment (Chen et al. 2014, 2017). The results of this study also affirmed that Ag^+ was reduced to Ag NPs in situ, in which silver atoms could bind to the hydroxyl groups of cellulose and hemicellulose in wood pipes (Shen et al. 2014; Goswami et al. 2018). The special structure of the hardwood itself and this possible bonding will make the Ag NPs stable inside the wood. Therefore, the Ag NPs would not be washed out easily. And the mechanism diagram of preparation and application of Ag NP/wood Membranes was given (Fig. 1).

Figure 2 shows the SEM images of natural longan wood and the Ag NP/wood. Wood was cut in the direction of growth, which can clearly show the

intricate channels inside. In addition, the axial parenchyma of the longan wood was annular tubular, annular bundle or whorl, and wood end nodes were thickened (Fig. 2c). The Ag NP/wood filter membrane possessed a uniform black colour (Fig. 2b), which proved the Ag NP ubiquity in the wood channels, and the samples were tested by TEM to determine the morphology and particle size of the synthesised Ag NPs in the wood. Figure 3 depicts that the Ag NPs had good dispersion and an average particle size of 9.45 nm. Research has confirmed that the nanoparticle aggregation issue appears as the Ag nanoparticle size decreases and that the specific surface area increases and the surface energy increases (Huang et al. 2016). Thus, numerous studies have bound Ag NPs in different types of carriers, such as porous carbon material, alumina ferric, oxide and polymer, to prevent nanoparticle agglomeration (Castle et al. 2011). In this study, the in situ reduction of Ag NPs in the small and complex cavity structure of wood could perfectly avoid the agglomeration of Ag NPs.

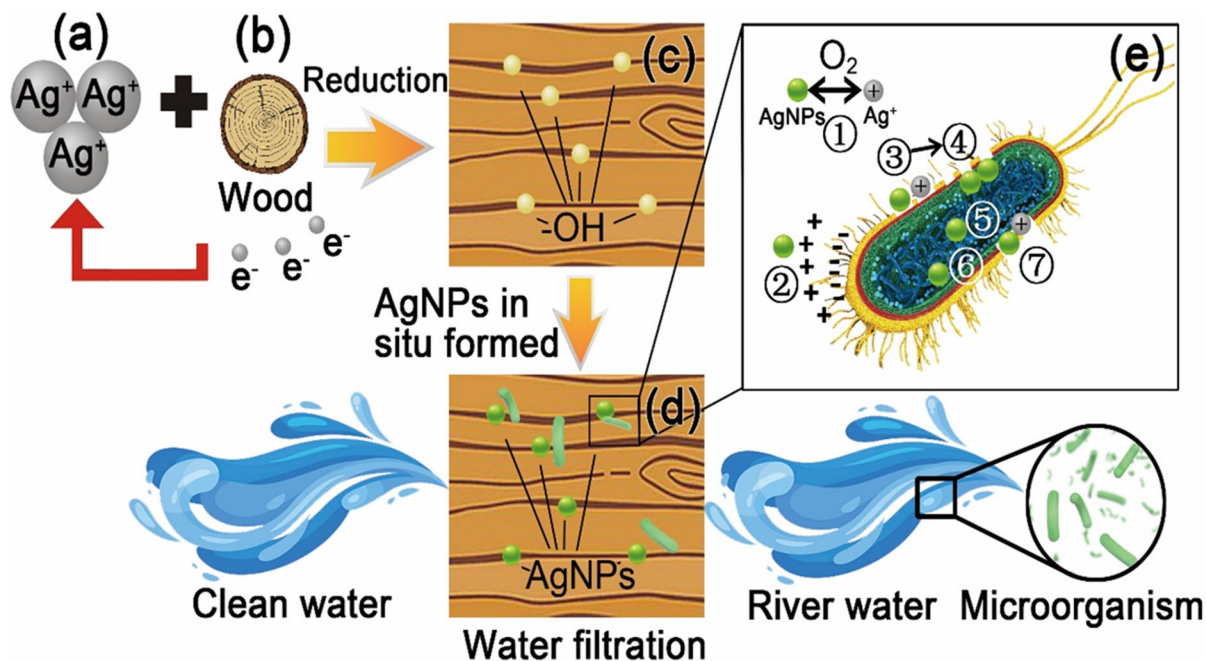


Fig. 1 Mechanism diagram of preparation and application of Ag NP/wood membranes. **a–c** The process of in situ reduction of Ag NPs in wood internal pipeline, **d** filtration and sterilization process of Ag NPs wood membrane. **e** Several ways of sterilization of Nano-silver. ① Under the action of O_2 , Ag^+ was released from Nano-silver wood membrane for

bacteriostatic; ② Negative electricity on the surface of bacteria, so killed bacteria by electrostatic action; ③–④ Ag NPs anchored the cell wall and penetrated to inactivate the cell; ⑤ Effect on DNA; ⑥ Acting on enzymes and their respiratory chains; ⑦ Destroyed the permeability of cell membrane

Fig. 2 Longan wood decorated with Ag NPs. **a** Observation of the radial tissue of the wood by SEM. A block of natural wood (diameter 5 cm, thickness 0.5 cm) was cut perpendicular to the growth direction, in which the internal organisational structure remains the same. **b, d** the wood changed to black after immersing in the AgNO_3 solution. **c** The partial enlarged detail shows the inner structure of the wood, which has axial parenchyma plant tissues and numerous long channels

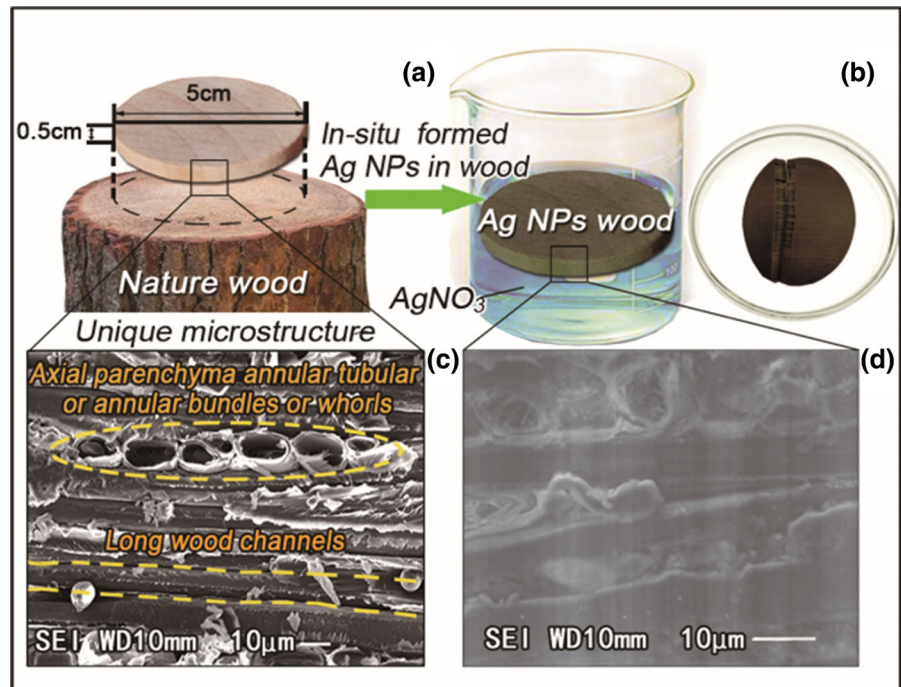
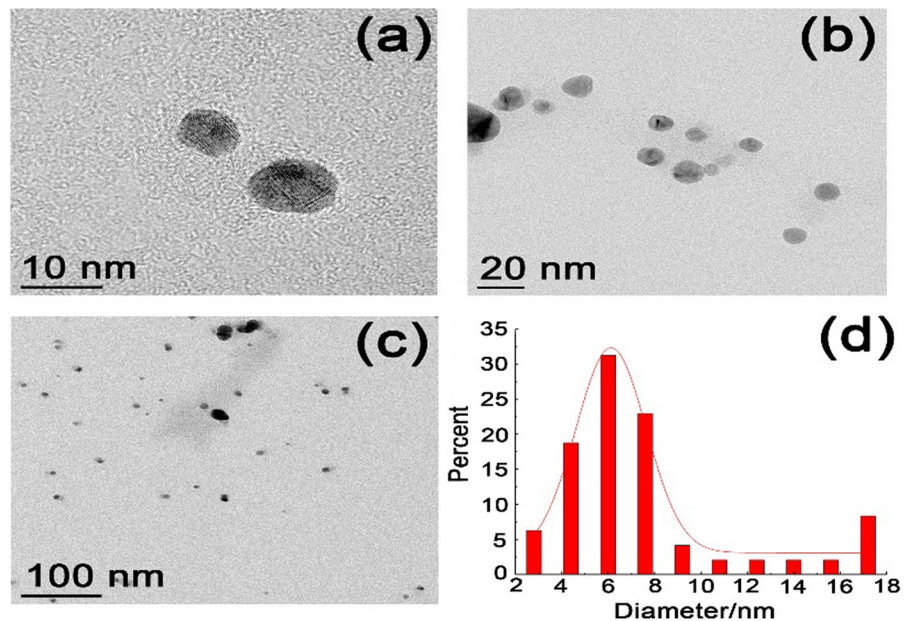


Fig. 3 **a–c** TEM images of Ag NPs decorated in the wood. **d** Size distribution of Ag NPs in the wood



The crystalline structure of woods is similar to that of cellulose given that the main components of woods are lignin, cellulose and hemicellulose, which are inherently amorphous structures (Lionetto et al. 2012). In addition, the peaks observed at $2\theta = 15^\circ$ and $2\theta = 22.4^\circ$ are designated (101) and (002) planes of

cellulose, which both exist in natural wood and the Ag NP/wood (Fig. 4). The XRD spectrums of the Ag NP/wood clearly show the Ag crystalline peaks at 38.1° (111), 44.3° (200), 64.4° (220) and 75.9° (311) of Ag nanoparticles (Fig. 4a). All the crystalline peaks can be exactly determined from the face-centered-

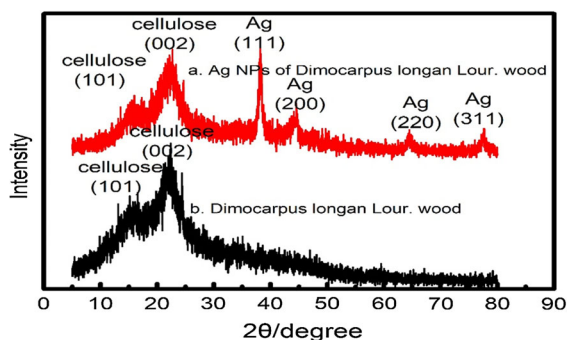


Fig. 4 XRD spectrum of longan wood and Ag NP/wood membranes

cubic structure of Ag (JCPDS Card No. 4-783), and the intensity of diffraction peaks can reflect the loaded amount of silver.

The method of energy-dispersive X-ray spectroscopy (EDS) was considered to confirm if silver elements exist in the wood membrane. The EDS spectrum shows the peaks of C, O, Zr and Pt elements in the natural wood and the Ag NP/wood (Fig. 5). However, the peak of Ag elements existed in the Ag NP/wood only (Fig. 5a). Consequently, we can confirm that Ag has formed in the wood. The mass% of Ag in the Ag NP/wood was 1.64%, which was close to 1.89% (the result of ICP-MS).

The FTIR was recommended to test the changes in chemical bonds before and after the Ag NPs were formed for studying the synthesis process of the Ag NP/wood. The broad and strong absorption band at $\sim 3341\text{ cm}^{-1}$ was due to the stretching vibration of -OH and hydrogen bonds. In addition, the absorption peaks at ~ 2935 and $\sim 2892\text{ cm}^{-1}$ were assigned to the stretching vibration of $-\text{CH}_3$ and $-\text{CH}_2$, and the

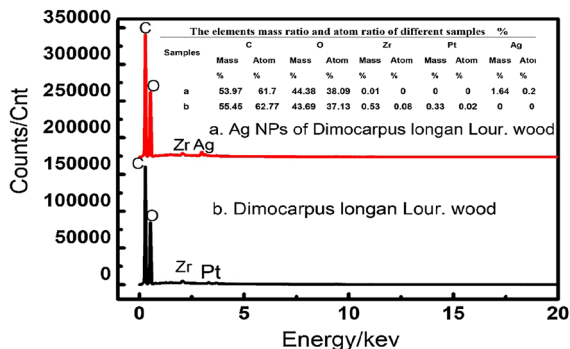


Fig. 5 Element distribution of longan wood and Ag NP/wood membranes

characteristic peak at $\sim 1737\text{ cm}^{-1}$ was caused by the stretching vibration of $\text{C}=\text{O}$. The absorption peaks at ~ 1652 and $\sim 1598\text{ cm}^{-1}$ were assigned to the $\text{C}=\text{O}$ of acid amides (Amide I band) and the bending vibration of $\text{N}-\text{H}$ (Amide II band). After immersing into AgNO_3 solution, an obvious decreasing band intensity was observed at $\sim 1626\text{ cm}^{-1}$, which indicated the silver generation by reducing the number of $\text{N}-\text{H}$ groups. Moreover, the absorption peak of $-\text{OH}$ shifted to a lower wavenumber ($\sim 3332\text{ cm}^{-1}$), and the Ag ions bonded to the functional groups, such as $-\text{OH}$ and $-\text{NH}_2$, and caused the changes in absorption peaks. The FTIR spectrogram not only confirmed the formation of Ag NPs within the wood but also showed the functional groups on the surfaces of the natural wood and Ag NP/wood (Fig. 6).

Bacteriostatic properties of Ag NP/wood membranes

As effective bactericides, Ag NPs have an excellent antibacterial activity against various microorganisms (Wei et al. 2013; Yang et al. 2014; Khatoon et al. 2015; Gopiraman et al. 2018). Qiu et al. (2014), Su et al. (2017) corroborated that Ag NPs exert a clear inhibitory effect on several commonly tested bacteria and that the inhibitory effect is influenced by the size, concentration and contact time of Ag NPs. Ag NPs also have good thermal stability and still exert a bacteriostatic effect after high-temperature treatment (Sun et al. 2006). Many studies have accordingly used nano-silver materials to prepare fresh-keeping and coating films of several food types (Emamifar et al. 2010; Yang et al. 2010; Hu et al. 2011; Herrera et al. 2018).

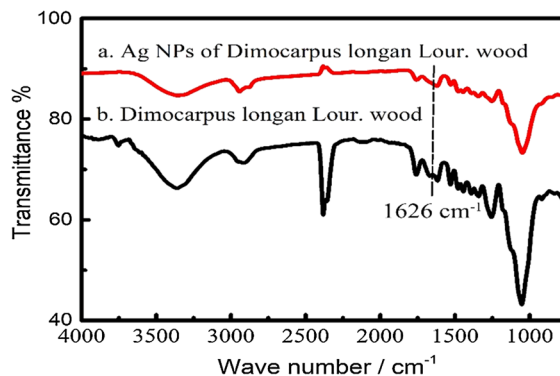


Fig. 6 FTIR spectrum of longan wood and Ag NP/wood membranes

Moreover, the assumption that the Ag NP/wood has strong capability to kill bacteria will be proven by filtrating river water through the membrane in this part (the filtration rate was $3.6 \text{ L m}^{-2} \text{ h}^{-1}$, with an eliminating bacterial and fungal rate up to 100% when three wood membranes were used). Compared with that in the filtrate by the natural wood membrane, no bacteria or fungi were found in the filtrate that passed through three pieces of Ag NP/wood membranes at the same time (Fig. 7). The Ag NP/wood membrane can also be used for liquid filtration sterilisation. The bacteriostasis inhibition zone diameter test was used to prove the bacteriostatic effect of the wood filter membrane in this study, and the test affirmed that nano-silver powder exerted an inhibitory effect on *E. coli*, *S. aureus*, *B. subtilis*, *P. aeruginosa* and *C. albicans*. The inhibition zone diameters of the five strains were as follows: *S. aureus* $1.89 \pm 0.01 \text{ cm}$, *E. coli* $1.57 \pm 0.01 \text{ cm}$, *P. aeruginosa* $1.35 \pm 0.01 \text{ cm}$, *B. subtilis* $1.25 \pm 0.01 \text{ cm}$ and *C. albicans* $1.20 \pm 0.01 \text{ cm}$ (Fig. 8). The Ag NP/wood membrane powder exhibited an inhibitory effect on bacteria and fungi.

Stability of the Ag element in wood membranes

The ICP-MS result contended that the prepared Ag NP/wood membrane contained 1.89 wt% of Ag element, with 0.06 wt% of Ag NPs loss after 5 L of river water was treated with the membrane. The average weight of one wood block we used was 7.0212 g. Therefore, after filtering 5 L river water with three pieces of wood, the concentration of silver in the filtrate was 0.048 mg/L, lower than 0.05 mg/L stipulated in China's hygienic standard (GB5749-2006) for drinking water. And the United States Environmental Protection Agency, recommends that the maximum dose of secondary controlled pollutant silver ions in drinking water is 0.10 mg/L. Furthermore, some studies have shown that the minimum inhibitory concentration of some kinds of bacteria and fungi were much higher than 0.048 mg/L (Kabir et al. 2011; Holla et al. 2012; Qiu et al. 2014). Therefore, the loss Ag NPs in water have no effect on our bacteriostatic experiment. From such a low loss rate of nano-silver, it can be seen that Ag elements are tightly bound to wood.

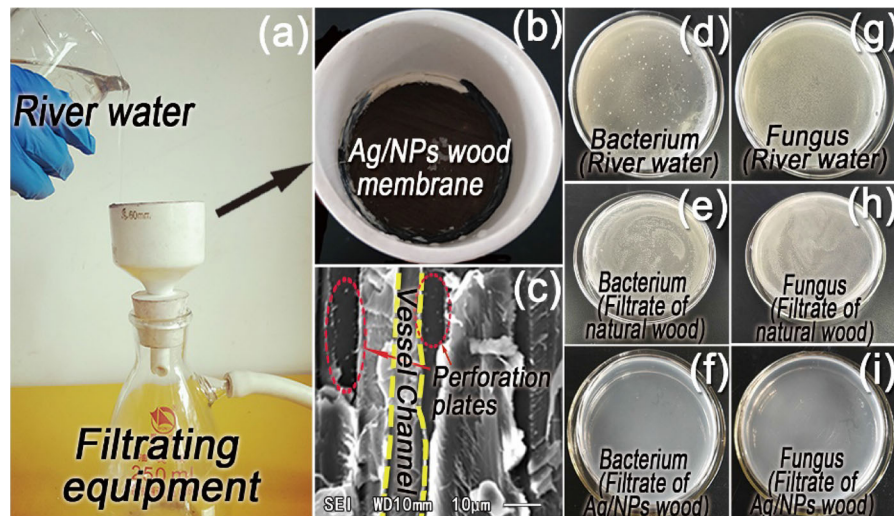
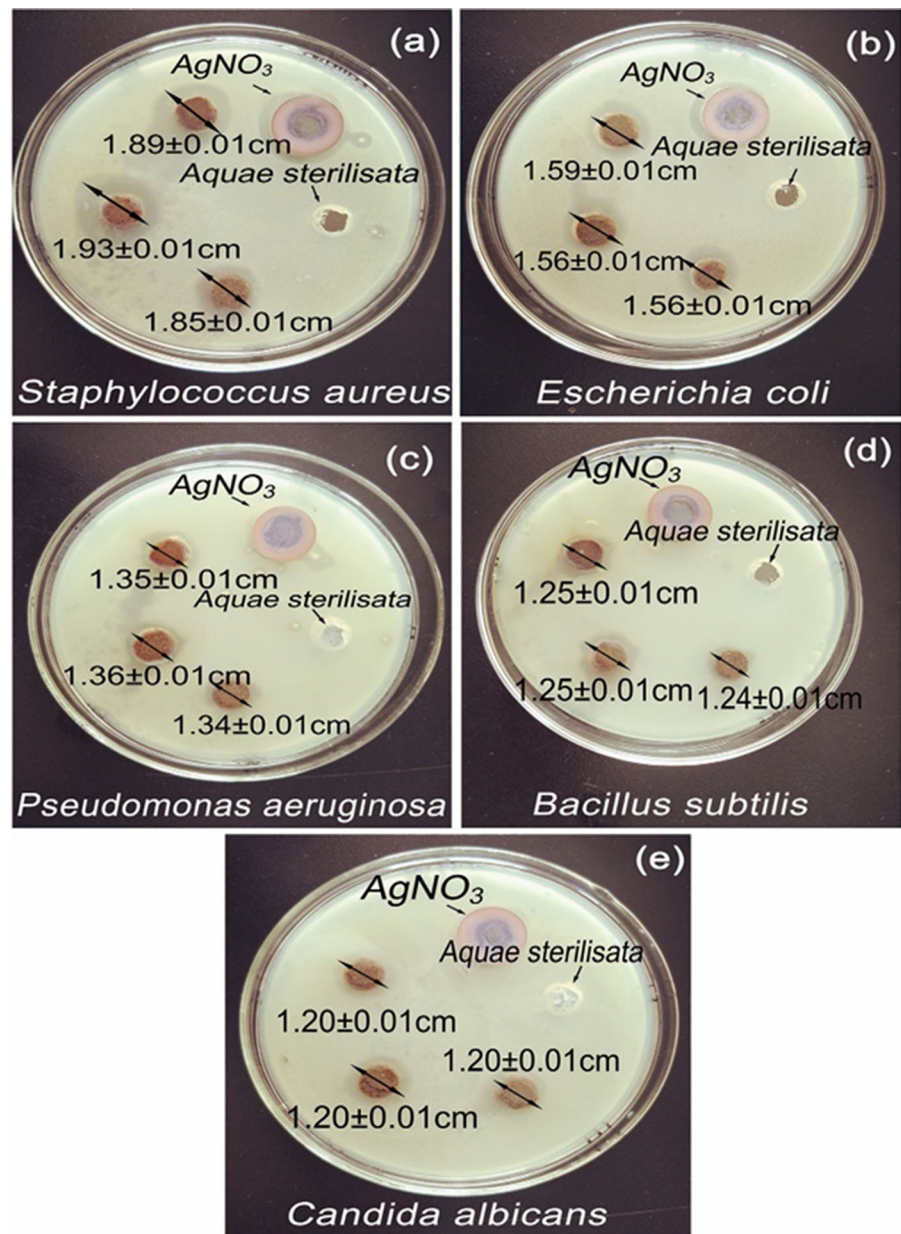


Fig. 7 Effect of Ag NP/wood membrane on removes the bacteria and fungi from river water. **a** River water filtrating equipment. **b** Ag NP/wood membrane sealed in a Buchner funnel with glass cement. **c** Perforation plates of Ag NP/wood when river water passed through. **d, e, g, h** Bacteria or fungi in

river water or filtered liquor passed through the natural wood after being cultured in culture medium. **f, i** The bacteria and fungi could not be found after river water passed through the Ag NP/wood membrane

Fig. 8 Inhibition zone diameters of four common bacteria and one common fungi



Conclusion

In this work, a hierarchical mesopore wood filter membranes decorated with silver nanoparticles can act as an excellent water purification device, which is due to the interaction between ubiquitous silver nanoparticles and special crisscross hardwood network channel structures. Natural wood not only promotes the in situ reduction of nano-silver on the internal pipeline, but also acts as a scaffold for continuous

water treatment to increase the exposure and interaction of microorganisms in water to Ag NPs. In this study, we tested the effectiveness of the Ag NP/wood membrane by filtering river water, and the result claimed that four common bacteria and one common fungus could be 100% killed after river water flowed through three Ag NP/wood membranes at a filtration rate of $3.6 \text{ L m}^{-2} \text{ h}^{-1}$. Furthermore, bacteriostatic circles also proved that the Nano-silver wood membrane exerted strong filtration and bacteriostatic

effects. Therefore, the wood membrane can be expanded to various fluid-filtering treatment applications. And also the wood can be decorated with other nanoparticles, such as palladium and copper nanoparticles, which could be used in other areas, such as photo catalytic degradation. In summary, the proposed easily prepared, low-cost and high-efficiency 3D hardwood membrane is extendible and available for broader industrial development and application.

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