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TEXTILE TECHNOLOGY

Preparation and Performance of Cotton Fabric with Antibacterial and Hydrophobic Properties Based on Click Reaction

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ABSTRACT

A simple modification method for creating cotton fabrics with hydrophobic and antibacterial surface properties is proposed. Silicon dioxide nanoparticles were obtained and added to a mixed solution of ethanol and silver nitrate to form a composite solution, and the SiO_2/Ag^+ composite solution was used to modify the cotton fabric. Then, the fabric was modified by self-assembly of (3-mercaptopropyl) triethoxysilane (MPTES), and the modified fabric was completed by grafting dodecafluoroheptyl methacrylate using click chemistry technology. A scanning electron microscope (SEM), Fourier transform infrared spectrometer (FTIR), thermogravimetric/differential thermal analyzer (TG), and contact angle measuring instruments were used to characterize the surface morphology and wettability of the cotton fabric. The results showed that the modified cotton fabric had a contact angle of 114.3 ° and still had good hydrophobicity after being subjected to multiple frictions. The modified cotton fabric also showed good antibacterial properties against *Escherichia coli* (*E. coli*) in a Petri dish.

Characteristics of original cotton fabrics, such as the ability to absorb moisture and good air permeability, often lead to the growth of bacteria (Dhandapani et al., 2014). The proliferation of microorganisms will destroy many excellent properties of cotton fabrics which can lead to yellowing and the reduction of mechanical strength (Shahidi et al., 2014). Traditional cotton fabrics can no longer meet the needs of modern production and clothing. The recent increase in infectious diseases caused by bacteria and viruses, such as Covid-19, has increased our demand for clean and hygienic fabrics. Consequently, there is an urgent need to produce antibacterial and hydrophobic cotton fabrics (Arain et al., 2013).

Up to now, in order to make cotton fabric hydrophobic, various methods have been proposed. For example, using hydrophobic compounds to modify the surface of the fabric (Yu et al., 2016), or a coating with low-surface-energy fluoride film to form a hydrophobic film on the surface of cotton fabric (Maity et al., 2010). There is also a method of preparing polyhexafluorobutyl methacrylate (PHFBMA) by free radical polymerization to modify the coating. The contact angle of the modified fabric is very high (Yang et al., 2018). The greater the water contact angle on the cotton fabric, the greater the hydrophobicity (Zhang et al., 2003). In fact, there are two main forms of hydrophobic finishing of cotton fabrics: the first is to start with raw materials and directly produce hydrophobic fibers. This fabric can be prepared by a blending method, or polymerization method, and then fibers are woven to prepare a hydrophobic fabric. This is also the main method for preparing hydrophobic synthetic fibers. The second method is to directly use the hydrophobic agent to finish the fabric to improve its performance. In order to make cotton fabrics antibacterial, nano-ZnO finishing agents are used in the preparation process (Li et al., 2007). There are also treatment methods for silver nanoparticles. The treated cotton fiber has a synergistic antibacterial effect on *Pseudomonas aeruginosa* (Kang et al., 2016).

However, most traditionally modified cotton fabrics usually only have a single hydrophobic or antibacterial property and have various defects such as poor friction resistance (Zhou et al., 2013). The fragility of superhydrophobic surfaces limits their applicability (Verho et al., 2011). Therefore, there is an urgent need to develop a material with excellent performance. Cui et al. (2009) conducted research on

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hydrophobic and antibacterial cotton fabrics, and the prepared superhydrophobic surface had excellent durability. Rana et al. (2016) prepared a multifunctional cotton fabric with Ag/AgBr-TiO_2 nanocomposite coating, which had good hydrophobicity, through a simple spraying process. The treated fabric also had good antibacterial properties.

In order to make cotton fabrics with hydrophobic and antibacterial properties, substances with different characteristics are often introduced into cotton fabrics, so that their surface properties are changed (Yeqiu et al., 2005). Recently, as a reaction to Covid-19, antibacterial and hydrophobic cotton fabrics have gained popularity in practical applications (Shateri-Khalilabad and Yazdanshenas, 2013). Cotton fabrics are filled with a dodecyl mercaptan-terminated nano-silver solution to achieve antibacterial characteristics. Silver nanoparticles have very strong antibacterial properties. The treated cotton fabric has excellent antibacterial properties against *E. coli*. (Tarimala et al., 2006). Antibacterial cotton fabrics can inhibit the growth and prevent the spread of microorganisms (Thomas et al., 2011). However, the cumbersome preparation process and high production cost limit their practical application. Therefore, an easy to prepare, low-cost method of antibacterial and hydrophobic cotton fabric has broad prospects.

In this work, a simple modification method to create cotton fabrics with hydrophobic and antibacterial surface properties is proposed. The SiO_2 nanoparticles were obtained and added to the mixed solution of ethanol and silver nitrate to form a composite solution, and the SiO_2/Ag^+ composite solution was used to modify the cotton fabric. The fabric was modified by self-assembly of (3-mercaptopropyl) triethoxysilane (MPTES), and the modified fabric was obtained by grafting dodecafluoroheptyl methacrylate using click chemistry technology. A scanning electron microscope (SEM), Fourier transform infrared spectrometer (FTIR), thermogravimetric/differential thermal analyzer (TG), and contact angle measuring instruments were used to characterize the surface morphology and wettability of the cotton fabric. Fig. 1 illustrates the modification scheme of cotton fabrics and the intuitive effects of antibacterial and hydrophobic properties of antibacterial and hydrophobic cotton fabrics. Modified cotton fabrics with antibacterial and hydrophobic properties could be widely used in practice.

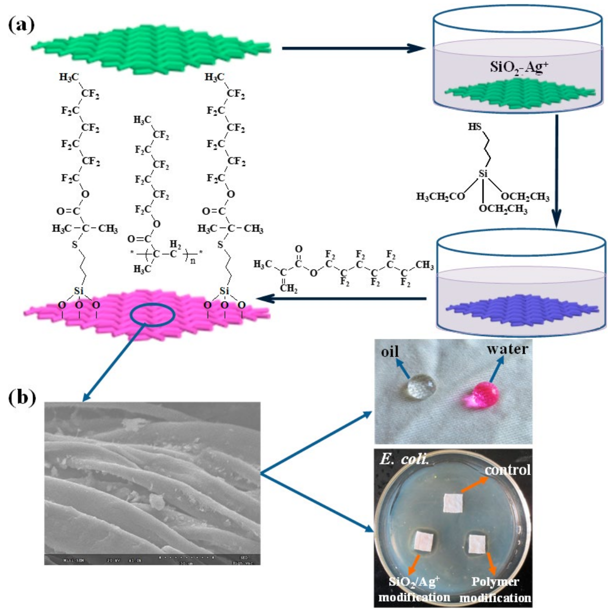

Figure 1. (a) Schematic diagram of modification of cotton fabric, (b) antibacterial and hydrophobic properties of the prepared cotton fabric.

MATERIALS AND METHODS

Materials. Dodecafluoroheptyl methacrylate ($C_{11}F_{12}H_8O_2$), MPTES ($C_9H_{22}O_3SSi$), absolute ethanol (C_2H_6O), azobisisobutyronitrile ($C_8H_{12}N_4$), ammonium hydroxide ($NH_3{\cdot}H_2O$), ethyl orthosilicate ($Si(OC_2H_5)_4$), toluene (C_7H_8), azobisisobutyronitrile (AIBN), and silver nitrate ($AgNO_3$) were provided by Aladdin Industrial Co., Ltd. (Shanghai, China). Arowana (Shanghai, China) soy oil was purchased from supermarkets.

SiO_2/Ag^+ Modified Cotton Fabric. The original fabric was first ultrasonically cleaned with ethanol and deionized water to remove impurities. After washing, the fabric was dried in an oven at 60 °C. Then, the cotton fabric was cut to the specified size according to the requirements of the ratio of 1:20. A solution of 49.5 mL of absolute ethanol, 2.0 mL of ammonium hydroxide (25% ammonia solution), 6.3 mL of water, and 2.23 mL of ethyl orthosilicate was combined into a flat-bottomed flask. The solution was stirred at room temperature and dried at 60 °C for 12 h to obtain silica nanoparticles. Then 0.2 g of silicon dioxide, 50 mL of absolute ethanol, and 0.12 g of silver nitrate were placed in a flat-bottomed flask and stirred at room temperature to prepare a SiO_2/Ag^+ composite solution. The cotton fabric was immersed in the above solution with a bath ratio of 1:20, so that the surface was loaded with

SiO_2/Ag^+. The weight of SiO_2-Ag^+ modified fabric increased by 1.19% compared with the original fabric by calculation.

Self-Assembly Modification of SiO_2/Ag^+ Modified Cotton Fabric. Approximately 1.0 g MPTES and 40 mL toluene were shaken with a KQ-500 ultrasonic cleaner at 40K Hz until mixed into a homogeneous mixture. The SiO_2/Ag^+ modified cotton fabric was then immersed in the mixture. The treated fabrics were sealed with plastic wrap and stored at room temperature for more than 24 h to obtain the MPTES modified cotton fabrics. The weight of MPTES modified cotton fabric increased by 2.27% compared to the SiO_2/Ag^+ modified fabric by calculation.

Preparation of Antibacterial and Hydrophobic Cotton Fabric. The MPTES-modified cotton fabric, 20 g of dodecafluoroheptyl methacrylate, 0.2 g of AIBN, and 80 g of absolute ethanol were combined in a round bottom flask and stirred in a water bath at 75 °C for about one hr. Finally, the modified fabric was obtained after washing with absolute ethanol and drying under 60 °C. The weight of the modified fabric increased by 10.85% compared to the MPTES-modified cotton fabric by calculation.

Characterization. The changes of the molecular structure of the samples before and after modification were measured by FTIR (Shimadzu Corporation, Japan), and the types of the internal functional groups were analyzed. The wave number range is 500-4,000 cm^{-1}. The changes of the surface morphology of the samples before and after modification were analyzed by SEM (SNE-3000, SEC, Korea). The voltage of the SEM was 20 KV and the magnification was 1 k. About 5 mg of sample was added to the crucible of the comprehensive TG (TG/DTA 6300, Japan) and the heating rate was from 30 °C to 600 °C at 10 °C/min increases with a selected gas condition of air. The thermal stability of the cotton fabric before and after modification was compared and analyzed.

The water contact angle (WCA) was used to characterize the hydrophobicity of cotton fabrics by an OCA micro 50 machine (Data-Physics, Germany). The sample was stretched across the sample stage so that the sample surface was flat. Two μL of distilled water was added with a syringe to the fabric. Observations of the state of water droplets on the surface of the sample were recorded. The hydrophobicity of cotton fabrics before and after modification was compared and analyzed.

An electronic rubbing color fastness meter (M238BB) was used to determine the rubbing resistance of modified cotton fabrics. The samples were placed flat on sandpaper and fixed with a slip iron plate to ensure that the samples were flat and wrinkle-free. The rubbing cloth was fixed on the rubbing head and secured with iron clips. Rubbing tests were conducted on the modified cotton fabric with a rubbing head. Then the contact angle test was performed on the modified cotton fabrics with different rubbing cycles to compare the changes of hydrophobic properties before and after rubbing and to obtain the rubbing resistance of the modified cotton fabrics.

Antibacterial properties of the modified fabric were tested by pouring 15 mL of sterilized nutrient agar medium into a standard Petri dish. A sterile pipette was used to place 0.2 mL of bacterial solution on the plate and was spread with a sterile glass rod. The Petri dish was placed flat on a test bench for 20 – 30 min to allow the bacterial solution to penetrate the surface layer of the medium. Modified fabric was cut into 1 cm^2 pieces and placed on the surface of the agar with sterile forceps. Immediately after the fabric was placed on the agar medium, the Petri dish was placed in an incubator at 37±2 °C for 18 – 24 h. The area without bacterial growth at the boundary between the agar medium surface and the specimen in contact is called the inhibition zone. this area was observed and recorded.

RESULTS AND DISCUSSION

Fig. 2 shows the FTIR spectra of original cotton fabric, SiO_2/Ag^+-loaded cotton fabric, and the antibacterial and hydrophobic cotton fabric. The infrared bands at about 2900 cm^{-1} were attributed to the stretching vibration of -CH_2, and this area increased significantly due to fabric modification. The peak at 1247 cm^{-1} corresponded to the C-F stretching vibration peak, indicating that fluorine had been grafted on the surface of the cotton fabric. In addition, compared with the original cotton fabric, the hydrophobically modified cotton fabric has a C=O stretching vibration peak at about 1750 cm^{-1}. These results confirmed that the hydrophobic polymer had been successfully grafted onto the cotton fabric.

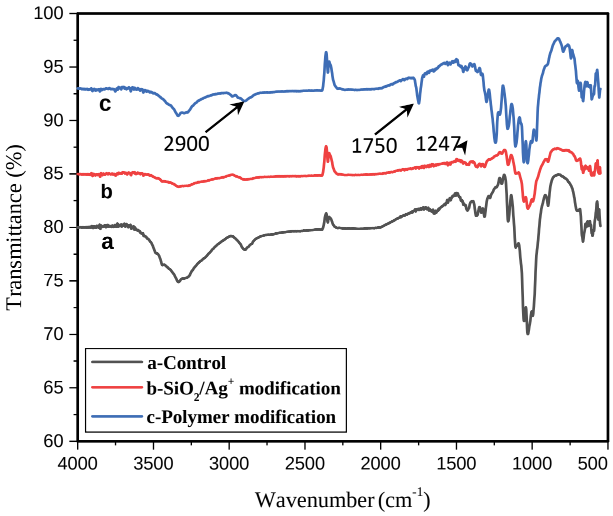

Figure 2. Fourier transformed infrared spectrometer (FTIR) spectra of the cotton fabrics: (a) original cotton fabric, (b) SiO_2/Ag^+-loaded cotton fabric, (c) polymer modified cotton fabric.

The morphological structures of the original cotton fabric, SiO_2/Ag^+-loaded cotton fabric, and antibacterial hydrophobic cotton fabric were studied by SEM. It can be clearly seen in Fig. 3a and 3b that the untreated cotton fabric and the SiO_2/Ag^+-loaded cotton fabric had a smooth appearance with obvious natural twists. As shown in Fig. 3c, the surface of the antibacterial and hydrophobic cotton fabric changed significantly. The surface of the modified cotton fabric has an obvious rough structure. This difference also proved that modification has occurred on the surface of the cotton fabric.

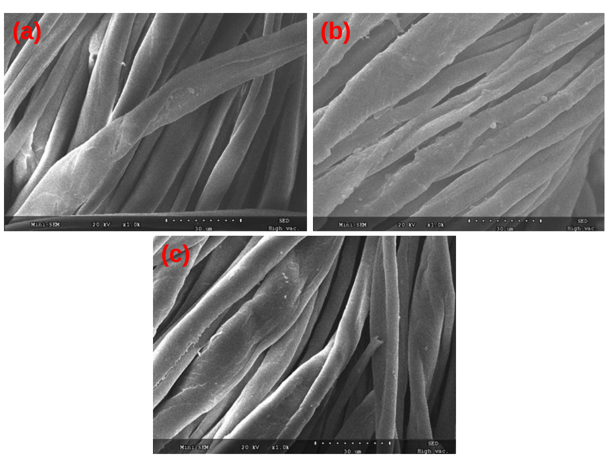

Figure 3. Scanning electron microscope (SEM) images of the cotton fabrics: (a) original cotton fabric, (b) SiO_2/Ag^+-loaded cotton fabric, (c) polymer modified cotton fabric.

The comprehensive TG was used to conduct research on the original cotton fabric, the cotton fabric loaded with SiO_2/Ag^+, and the antibacterial and hydrophobic cotton fabric (Fig. 4). The maximum temperature of decomposition for the three cotton fabrics, in the first stage occurred between 30 °C and 100 °C, which was the result of the loss of moisture. In the second stage, compared with the original cotton fabric, the initial decomposition temperature of the antibacterial and hydrophobic cotton fabric was reduced to 266 °C, which was caused by the decomposition of the fluoropolymer attached to the surface of the fabric. The initial decomposition temperature of the main peak decreased. The results showed that the thermal stability of the modified cotton fabric was lower than that of the original fabric and the fluoropolymer successfully adhered to the surface of the fabric.

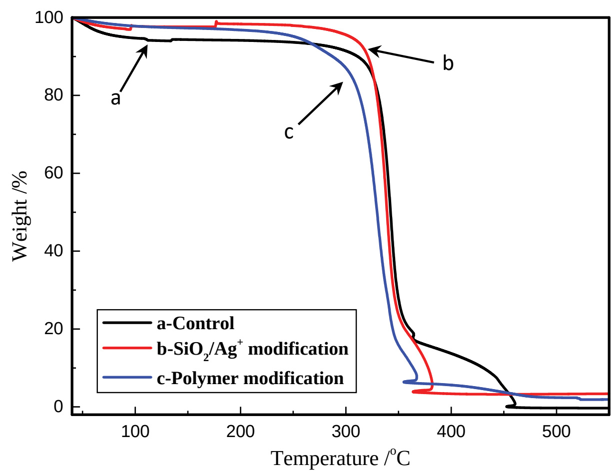

Figure 4. Thermogravimetric/differential thermal analyzer (TG) curves of the cotton fabrics: (a) original cotton fabric, (b) SiO_2/Ag^+-loaded cotton fabric, (c) polymer modified cotton fabric.

The contact angle measurements were performed to evaluate the surface hydrophobicity of the original cotton fabric, the SiO_2/Ag^+-loaded cotton fabric, and the antibacterial and hydrophobic cotton fabric. The results are shown in Fig. 5. The water droplets were absorbed as soon as they contacted the original and SiO_2/Ag^+-loaded cotton fabrics indicating that both fabrics had good hydrophilicity. The contact angle when the water came into contact with the original fabric or the SiO_2/Ag^+-loaded cotton fabric was close to 0 ° which meant that it would quickly become absorbed by the fabrics (Fig. 5). These results proved that the original cotton fabric and the antibacterial modified cotton fabric were hydrophilic.

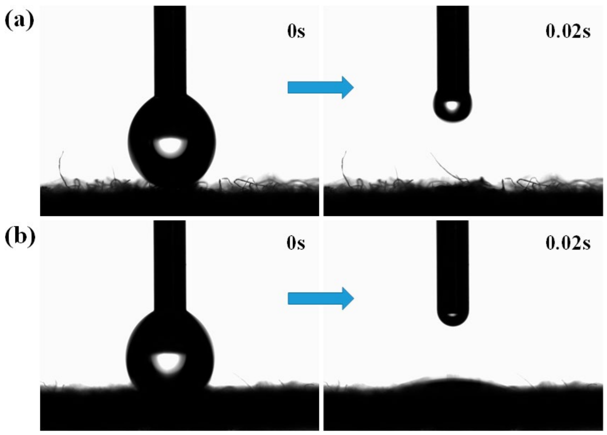

Figure 5. Contact angle tests of the cotton fabrics: (a) original cotton fabric, (b) SiO_2/Ag^+-loaded cotton fabric.

When the water droplets contacted the surface of the antibacterial and hydrophobic cotton fabric, the droplets retained a spherical shape as shown in Fig. 6. As the number of droplets increased, the shape of the water droplets became more and more circular. This phenomenon indicates that the water contact angle of the cotton fabric increases with the increase of the number of hydrophobic treatment impregnations. The contact angle was 114.3 ° after two treatments and increased slowly after continuing the reaction.

A piece of modified cotton fabric with an initial contact angle of 128.8 ° was selected for washing resistance testing. The contact angle was tested and recorded after a varying number of washes. After 20 washes, the hydrophobicity of the modified cotton fabric was still good, and the contact angle tested was still 124.6 °. This also indicates that the modified cotton fabric has certain washing resistance.

Fig. 7 shows the surface properties of the original cotton fabric, the SiO_2/Ag^+-loaded cotton fabric, and the antibacterial and hydrophobic cotton fabric. The surface properties of the cotton fabric were demonstrated by the presence of water droplets and soybean oil on the surface of the cotton fabric. The results show that when soybean oil and red water droplets were dropped on the original cotton fabric and the SiO_2/Ag^+-loaded cotton fabric, they did not maintain the spherical shape. They were directly absorbed once in contact, indicating that the fabrics were hydrophilic and lipophilic. When soybean oil and red water droplets were dropped on the antibacterial and hydrophobic cotton fabric, they remained spherical on the surface and were not absorbed. Thus, indicating that the antibacterial and hydrophobic cotton fabric had both hydrophobic and oleophobic properties which gives it broad prospects as self-cleaning materials.

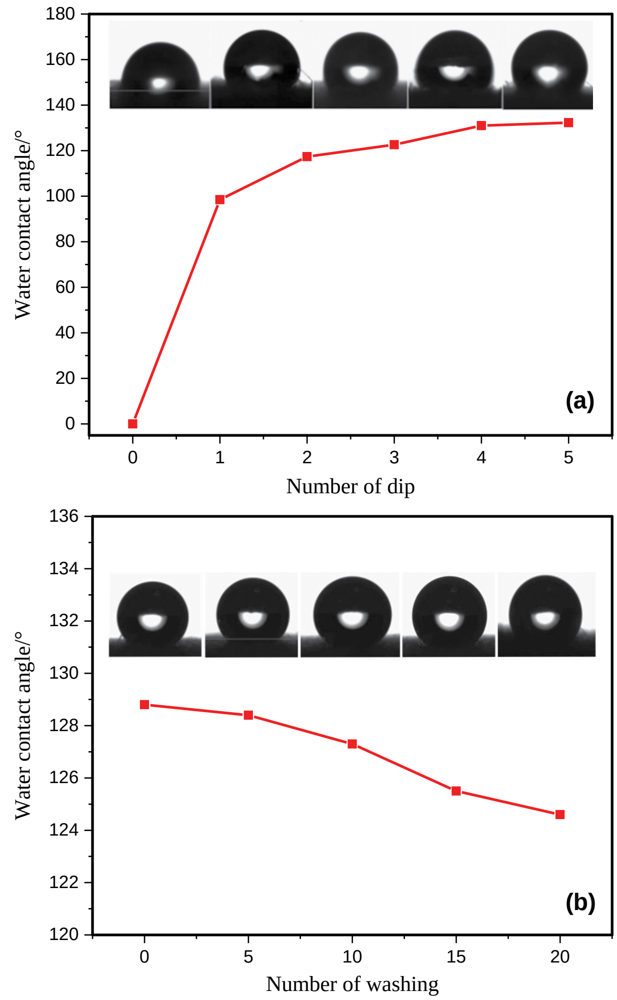

Figure 6. Effect of the number of dipping and washing on the water contact angle: (a) water contact angle for different number of dipping, (b) water contact angle for different number of washings.

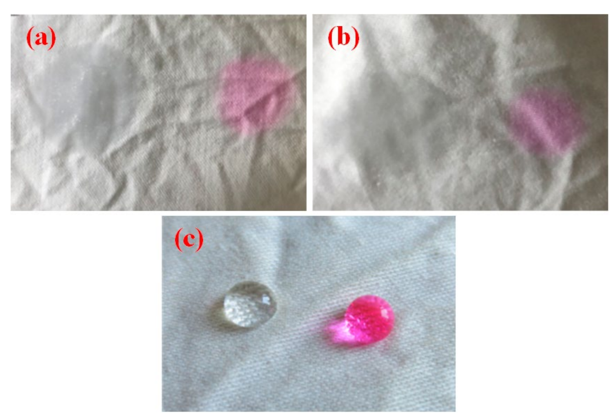

Figure 7. The surface properties of the cotton fabric: (a) original cotton fabric, (b) cotton fabric loaded with SiO_2/Ag^+, (c) polymer modified cotton fabric.

The antibacterial and hydrophobic cotton fabric were subjected to different rubbing cycles to test its rub resistance. As shown in Fig. 8, the SEM images of the modified cotton fabric were rubbed 100, 200, 400, and 600 times. The appearance of the rubbed cotton fabric changed significantly compared with the unrubbed cotton fabric (Fig. 8). The surface became rougher, and sol powder had appeared. As the number of rubbing cycles increased, more sol powder was produced. Some fibers were damaged by friction, and the sol powder had fallen off from a large area (Fig. 8c). It can be concluded that friction affects the performance of the treated cotton fabric, as increased rubbing led to more significant impacts on the performance of the cotton fabric.

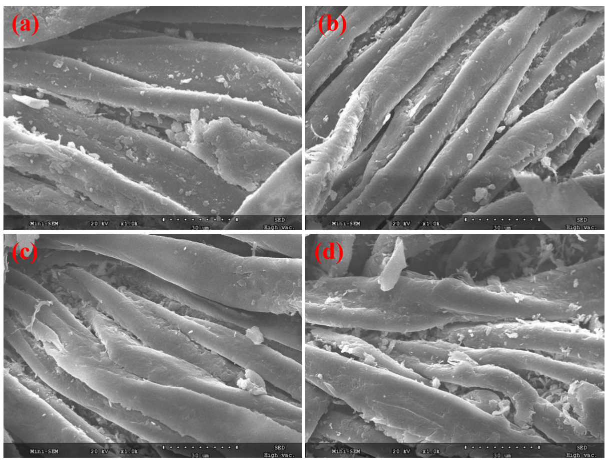

Figure 8. Scanning electron microscope (SEM) images of modified cotton fabrics with different friction times (a) 100 times of friction, (b) 200 times of friction, (c) 400 times of friction, (d) 600 times of friction.

In order to further verify the hydrophobic properties of the cotton fabric after friction, water droplets were deposited on each piece of cotton fabric to observe the surface wettability. Fig. 9, illustrates that although the modified cotton fabrics were rubbed 100 and 200 times, respectively, the water droplets were still spherical on the surface of the cotton fabrics, which proved that they still had good hydrophobicity. However, when the number of frictions reached 400, the water droplets could no longer maintain the spherical shape on the surface of the cotton fabric, and the hydrophobic performance of the fabric significantly deteriorated. The contact angle was tested on modified cotton fabrics after different rubbing cycles. It was found that the modified cotton fabric still had a water contact angle of 117.8 ° after 200 rubbing cycles. This showed that the modified cotton fabric could still maintain good hydrophobicity and a certain degree of friction resistance after being subjected to less than 200 rubbing cycles. At the same time, it also proved the conclusion drawn from the previous observation under the SEM.

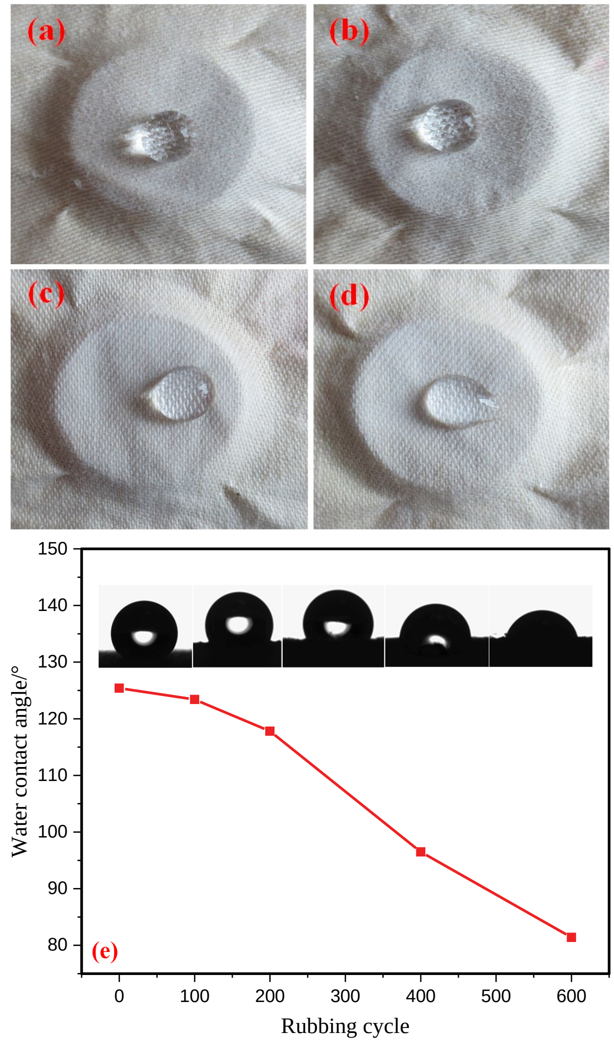

Figure 9. Photographs of water drops on the surface of modified cotton fabric after different rubbing cycles (a) 100 times, (b) 200 times, (c) 400 times, (d) 600 times, and (e) water contact angle after different rubbing cycles.

In order to investigate the antibacterial properties of the modified cotton fabric, the inhibitory effect of the modified cotton fabric on *E. coli* in the Petri dish was observed. Fig. 10 showed the inhibitory effect of original cotton fabric (a), SiO_2/Ag^+-loaded cotton fabric (b), and antibacterial and hydrophobic cotton fabric (c) on *E. coli*. The raw cotton fabrics did not have antibacterial properties, and the antibacterial-treated cotton fabrics retained antibacterial properties after the hydrophobic treatment. These results indicated that the modified cotton fabric had good application prospects in hydrophobic and antibacterial aspects.

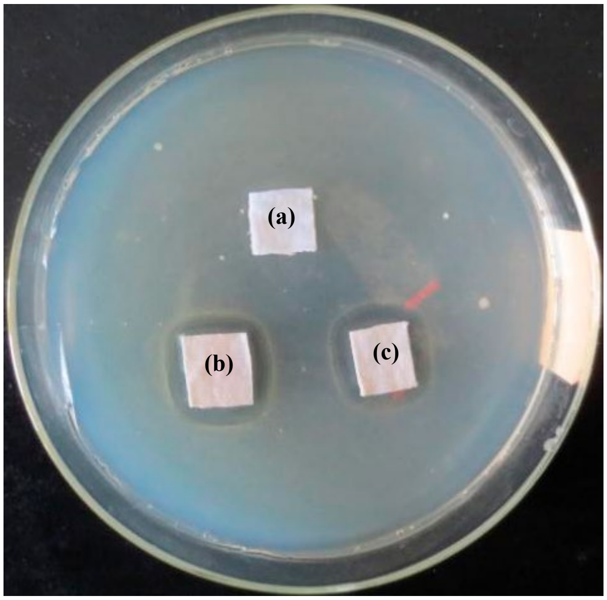

Figure 10. Comparison of antibacterial properties of cotton fabrics before and after modification (a) original cotton fabric, (b) SiO_2/Ag^+-loaded cotton fabric, (c) polymer modified cotton fabric.

In order to investigate the antibacterial properties of the modified cotton fabrics after washing and rubbing, the inhibition of *E. coli* in Petri dishes by the modified cotton fabrics was observed. The antibacterial and hydrophobic cotton fabric retained antibacterial properties after washing, but the antibacterial performance decreases significantly after 15 washes (Fig. 11). The antimicrobial performance of the modified cotton fabric with 200 repetitions of rubbing was also improved to some extent, probably because rubbing deteriorated the hydrophobic layer which exposed the SiO_2/Ag^+-loaded layer. As friction increased, antibacterial properties significantly decreased. These results indicate that the modified cotton fabrics have some resistance to washing and rubbing.

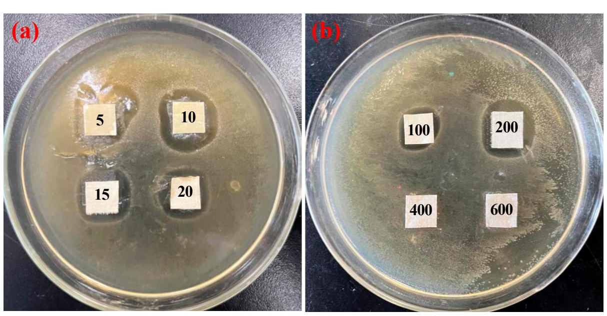

Figure 11. Investigation of antibacterial properties of modified cotton fabrics after washing and rubbing (a) after different number of washing cycles (5, 10 ,15, and 20 times), (b) after different cycles of rubbing (100, 200, 400, and 600 times).

CONCLUSIONS

In general, a modified cotton fabric with hydrophobic, oleophobic, and antibacterial properties was successfully prepared. It had a larger water contact angle, reaching 114.3 °. Moreover, it had a certain degree of friction resistance. Specifically, the modified cotton fabric maintained good hydrophobicity up to 200 rubbing cycles. In addition, the prepared cotton fabric also exhibited good antibacterial properties and had a significant inhibitory effect on *E. coli*. The modified cotton fabric with the above properties has broad application prospects in hydrophobic and antibacterial applications.

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