

Thermal, Waterproof, Breathable, and Antibacterial Cloth with a Nanoporous Structure

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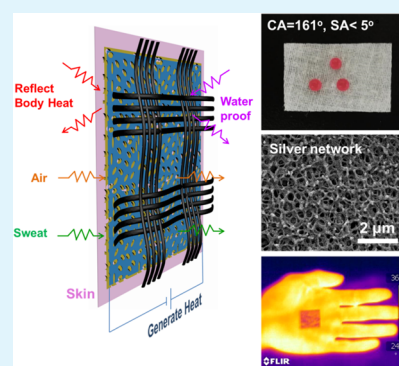
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Supporting Information

ABSTRACT: Wearable thermal management materials have attracted increasing attention because of the potential in energy conservation and the possibility to meet the need of smart clothes. An ideal cloth for cold areas has to be lightweight, warm, waterproof but breathable, and antibacterial. Herein, we present a multifunctional cloth starting from a cotton fabric, for which one side is modified to be superhydrophobic by introducing a silica nanoparticle/polydimethylsiloxane (PDMS) layer, while the other side is coated with a nanoporous cellulose acetate layer followed by depositing a thin silver film. The porosity allows the fabric to be breathable, and the silver film plays three important roles as a perfect infrared reflector, a flexible heater, and an antibacterial layer. Such a multifunctional fabric might be potentially useful in outdoor coats and other facilities.

KEYWORDS: porous structure, superhydrophobic, silver network, thermal control, breathability



INTRODUCTION

Clothing is a basic necessity of life.¹ Providing warmth for the human body is one of the main requirements, especially in temperate and frigid zones of the globe.² Cotton,³ wool,⁴ feather,⁵ and chemical fibers^{6,7} have been widely used to make warm clothes. Traditional clothes provide warmth by trapping the air around the human body to decrease heat transfer via convection and conduction. However, the high emissivity (0.75–0.9) of common clothes provides little radiative insulation effect.⁸ A commonly used way for further heat insulation is to increase the thickness of the clothes or wear more layers of clothes which on the other hand make the user uncomfortable. Besides, waterproofness is also an important aspect for such clothes. Cotton and other traditional clothes exhibit poor waterproofness, which would decrease the heat insulation performance of clothes when exposed to rain, fog, or snow.^{9–11} It is well-known that metals are a class of good infrared (IR) reflectors. For example, aluminized Mylar blankets perform well in blocking the radiative loss, such that they can be used as sun shields for cars. However, for clothing purpose, other performances such as light weight, high waterproofness, and high breathability should also be considered, which are not achievable by simply using metal foils or metal membranes.

Presently, a concept of “personal thermal management” is proposed as an optimal energy-saving method directly focusing on maintaining the warmth in the human body.^{12–14} A superior

thermal management material should be wearable like common clothes.^{15,16} An effective approach for fabricating thermal management materials is adding conductive nanomaterials on or inside textile fibers. Typically, conducting nanomaterials with a low emissivity that form a network could effectively reflect IR back to the body to reduce the heat loss. Such nanomaterial networks only have little or a small adverse effect on the breathability of the fabrics because of the percolation network. In general, the nanoscale conductors used for thermal management include carbon nanotubes (CNTs),¹⁷ graphite nanoplatelets,¹⁸ graphene,^{19,20} and metallic nanoparticles,²¹ among which silver nanowires are a superb choice because of the high IR reflection efficiency and the high electrical conductivity of silver that allows it to be potentially used as a heater. However, silver nanowires are expensive, typically at a price of ~\$400 per gram, about 3 orders of magnitude higher than bulk silver while the IR reflection efficiency of a silver nanowire network is still far lower than that of metal foils or films. In addition, silver nanowires may also present the disadvantages of poor adhesion to the textile.²²

In this study, we used a normal cotton textile as the substrate to fabricate a multifunctional cloth by constructing a hydrophobic coating on one side and a nanoporous structure

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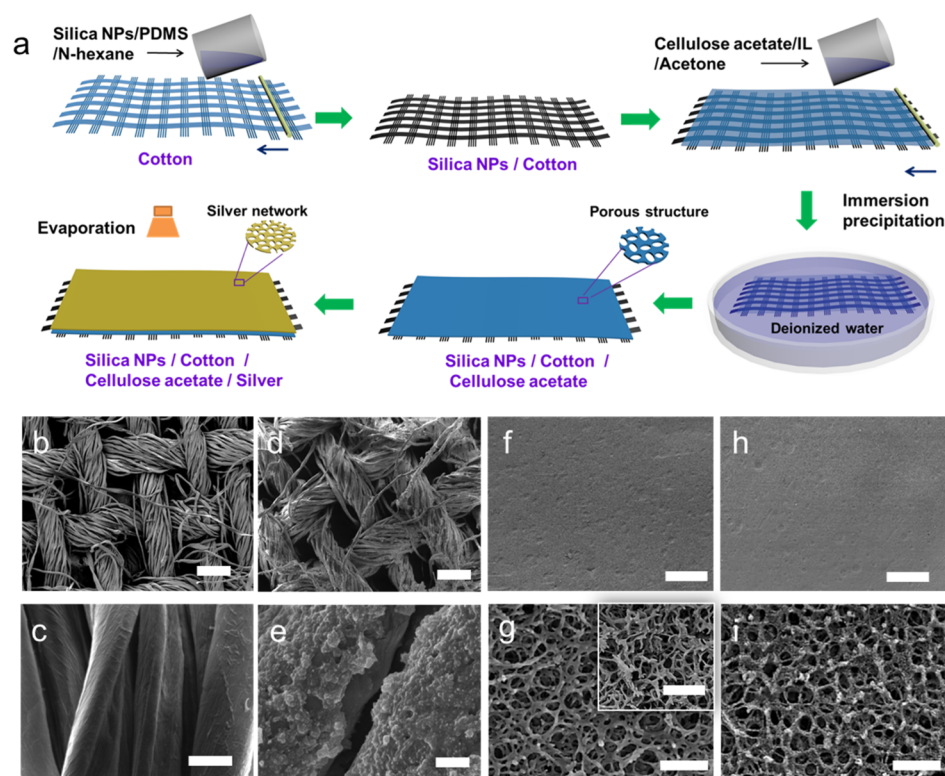


Figure 1. Preparation of the multifunctional cloth. (a) Schematic of the preparation process. (b) Low-magnification SEM image of a piece of normal cotton cloth. (c) SEM image of cotton yarn. (d) SEM image of the silica NP/PDMS-treated fabric. (e) Detailed morphology of the silica NP/PDMS layer. (f,g) SEM images of the surface morphology of cellulose acetate at different magnifications. (h,i) SEM images of the metallic silver network on the cellulose acetate porous pattern with different magnifications. Scale bars: 200 μm in panels b,d; 10 μm in panels c,e; 50 μm in panel f; 2 μm in panel g; 1 μm in panels h,i; and the inset in panel g.

followed by depositing a metallic film on the other side. The outer layer exhibits a water contact angle of 161° and a sliding angle of less than 5° , which effectively prevent the entry of water from the outside. The porous layer is a cellulose acetate thin film with dense nanopores that are beneficial to the high breathability and the improvement of the surface flatness of the whole textile, and the metallic silver network deposited on the porous layer can effectively reflect IR from the human body and also be used as a heater to provide additional Joule heat beyond the passive insulation by loading a small voltage. In this way, the multifunctional cloth can not only maintain the original nature of the cotton textile with high breathability but also possess significant properties including waterproofness, anti-fouling property, IR reflection, Joule heat generation, and antibacterial activities. Such a fabric might find wide applications in clothing, outdoor sports, and so forth.

■ PREPARATION OF THE MULTIFUNCTIONAL CLOTH

The preparation of the multifunctional cloth is shown as a schematic in Figure 1a. A common cotton textile is used as the substrate for the following procedure. First, we modify the surface of the normal cotton textile by introducing a silica nanoparticle (NP)/polydimethylsiloxane (PDMS) coating to make the surface superhydrophobic. After that, a cellulose acetate/acetone/IL solution is coated on the other side of the cotton textile and immersed into a water bath, leading to the formation of a porous cellulose acetate film. A silver film is then deposited on the porous structures by using thermal

evaporation, forming a silver network following the shape of the porous cellulose acetate film (Figure S1).

Figure 1b,c shows the scanning electron microscopy (SEM) images of the morphology of the cotton textile material. The material is basically a loosely woven textile structure which consists of cotton yarn of 200 μm diameter with an interyarn space of 200 μm . The surface of each fiber is generally smooth. After introducing the silica NP/PDMS layer, small silica NPs are distributed on the surface of the cotton fibers, as shown in Figure 1d,e. Here, PDMS plays as an adhesive wrapped around the silica NPs on the cotton fibers, and the hydrophobic silica NPs generate a dual-size surface roughness to improve the hydrophobicity of the cloth. The detailed preparation procedure of the superhydrophobic layer is shown in Figure S2. On the other side of the textile, the porous cellulose acetate film looks smooth when inspected using SEM at a low magnification (Figure 1f). However, the high magnification SEM image indicates that the film consists of dense pores with an average diameter of ~ 500 nm and a wall thickness of ~ 70 nm (Figure 1g), as a result of phase separation. It is worth noting that the pores are penetrated throughout the cellulose acetate membranes, and the pore interconnectivity is exhibited in the cross-sectional image shown in the inset of Figure 1g. Immersion precipitation has been employed for the fabrication of the polymeric porous film.^{23–26} The precipitant is usually miscible with the solvent but immiscible with the polymer. During the phase separation, the solid polymer-rich phase forms the walls and the liquid polymer-lean phase forms the pores.²⁷ Herein, the cellulose acetate/acetone/IL solution is coated on the cotton textile and immersed into a water bath to

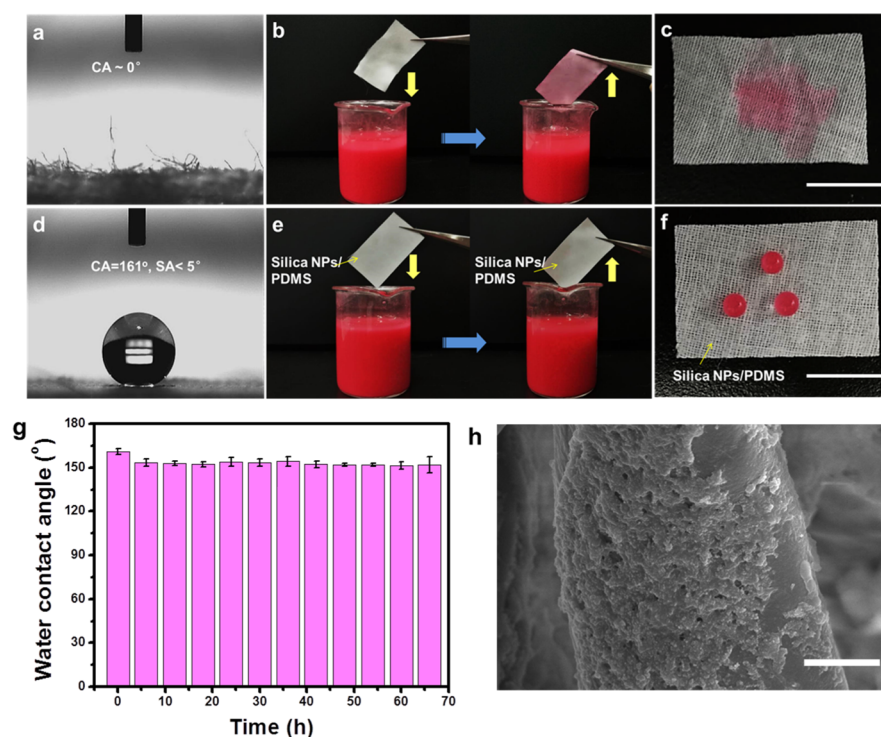


Figure 2. Wettability of the cotton textile before and after coating silica NPs. (a) Water drop ($2 \mu\text{L}$) on the untreated cotton textile, showing a contact angle close to 0° . (b) Untreated normal cotton cloth is polluted when immersed in dirty water. (c) Untreated cotton textile is completely infiltrated with dirty water drops. (d) Water drop ($2 \mu\text{L}$) on the cotton textile with silica NPs, showing a contact angle of $\sim 161^\circ$ and a sliding angle less than 5° . (e) After hydrophobic treatment, the cotton fabric becomes antifouling to dirty water. (f) Dirty water drops on the treated fabric, showing a remarkable antifouling property. (g) Change of water contact angle with ultrasonic cleaning duration. (h) SEM image of the coating surface after 66 h of ultrasonication. Scale bars are 1 cm in panels c,f and $5 \mu\text{m}$ in panel h.

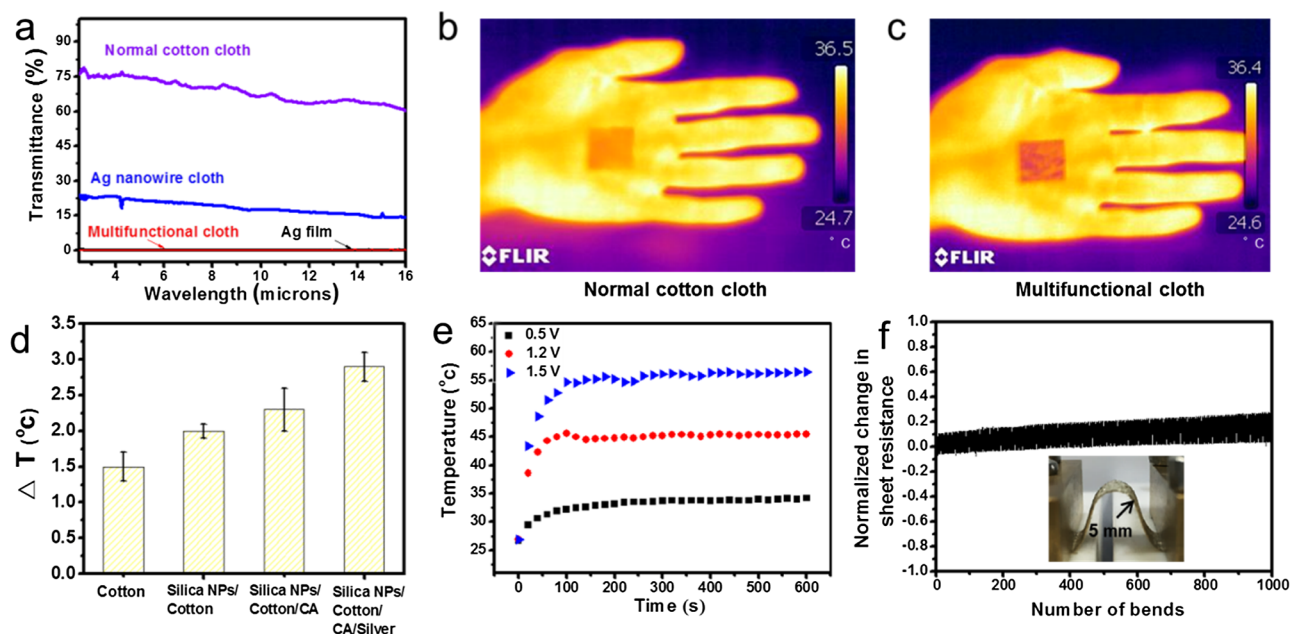


Figure 3. (a) Transmittance of the normal cloth, Ag nanowire cloth, Ag film, and the multifunctional cloth observed using a Fourier-transform infrared microscope. (b) Thermal image of a normal cloth on a hand. (c) Thermal image of a multifunctional cloth on a hand. (d) Heat transfer measurement by the temperature increase when placing the cloth at different fabrication stages on an open-air hot plate of 33°C . (e) Temperature change vs time after applying different voltages to a $2 \text{ cm} \times 2 \text{ cm}$ sample of the multifunctional cloth. (f) Change of the sheet resistance as a function of number of bends at a bending radius of 5 mm.

form the porous film (Figure S3). Note that this porous layer also fills up the gaps that are between the cotton yarn, and this allows the subsequent porous silver film to cover the entire

surface of the textile. The SEM images (Figure 1h,i) show that the silver film follows well the morphology of the porous cellulose acetate but with a pore size slightly smaller than the

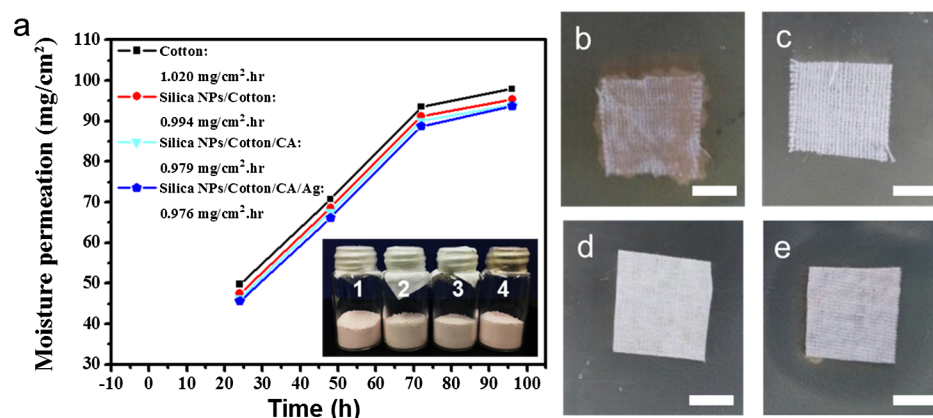


Figure 4. (a) Mass increase of desiccants sealed with different fabrics. Inset is the corresponding photograph of the bottles sealed by the following cloths: (1) normal cotton cloth, (2) silica NPs/cotton, (3) silica NPs/cotton/cellulose acetate, and (4) silica NPs/cotton/cellulose acetate/silver. (b–e) Antibiotic test of (b) normal cotton cloth, (c) silica NPs/cotton, (d) silica NPs/cotton/cellulose acetate, and (e) silica NPs/cotton/cellulose acetate/silver (multifunctional cloth, with the silver film facing down). Scale bars in panels b–e are all 0.5 cm.

original pore size (~ 500 nm) of the cellulose acetate pattern. The corresponding specimens at each stage are shown in Figure S4.

■ WATERPROOFNESS

The cotton fabric coated with silica NPs exhibits superhydrophobicity. As shown in Figure 2a, when a drop of water falls onto the untreated cotton fabric, the water wets the fabric immediately with a water contact angle approaching 0° because of complete infiltration. By contrast, after coating with a silica NP/PDMS layer, the water contact angle of the textile dramatically increases to 161° , while the sliding angle is less than 5° (Figure 2d), indicating an extraordinary waterproofness (shown in Figure S5). In addition, the treated superhydrophobic textile also exhibits antifouling function in dirty water, as indicated in the case with red chalk powders dissolved in water. As shown in Figure 2b,c, the original cotton cloth is dyed red by immersing into dirty water within a short time, whereas the superhydrophobic cotton fabric shows a remarkable antifouling ability in dirty water (Figure 2e,f). Furthermore, the washing durability was evaluated by ultrasonication in a mixed solution of water and ethanol. The change of the water contact angle is less than 9° after 66 h of ultrasonication, indicating that the cloth might be durable to washing (Figure 2g). The SEM observation reveals that the coating surface still retains its rough morphology after 66 h of ultrasonication (Figure 2h). This simple and low-cost coating shows a remarkable superhydrophobic property that is potentially useful for waterproof and self-cleaning protective textiles.

■ THERMAL CONTROL, ELECTRICAL PROPERTY, AND DURABILITY

The pore diameter of the conductive metallic silver network is far smaller than the wavelength of the blackbody radiation of the human body which is centered at $9 \mu\text{m}$, indicating that the cloth can effectively reflect human body radiation. The transmittance spectra of the normal cloth, Ag nanowire cloth (Figure S6), Ag film, and multifunctional cloth are compared in Figure 3a. The average transmittance in the range of $2.5\text{--}16 \mu\text{m}$ for the multifunctional cloth is close to $\sim 0\%$, which is significantly lower than that of the Ag nanowire cloth (20.6%) and normal cotton cloth (72.3%) and almost completely

overlaps with that of the continuous silver films. The low IR transmittance clearly indicates that the multifunctional cloth is a perfect IR reflector and, therefore, suitable to trap the thermal radiation around the human body. Figure 3b,c illustrates the thermal images of a human hand with a normal cloth and with a multifunctional cloth attached on, respectively. Both specimens are in thermal equilibrium on the palm before imaging at a palm temperature of 33.3°C and an atmosphere temperature of 25.0°C . For the normal cotton cloth, the textile emits IR in a temperature range of $31.7\text{--}32.7^\circ\text{C}$ as detected by using an IR camera. By contrast, the IR camera shows that the temperature of the multifunctional cloth on palm is lower, in the range of $30.4\text{--}31.1^\circ\text{C}$, indicating a decrease of $1.3\text{--}1.6^\circ\text{C}$ compared with that of the normal cotton cloth. Although the multifunctional cloth is tested under the same temperature as the normal cotton cloth, the low emissivity of the silver network induces lower thermal radiation making the multifunctional cloth cooler in the thermal images. The results prove that the multifunctional cloth is an effective IR reflector that can effectively decrease the heat loss from the human body. We have further compared the heat transfer property of the normal cloth and the multifunctional cloth by putting them on an open-air hot plate and testing the surface temperature. In principle, the larger the difference in temperature between the hot plate top and the upper surface of the sample, the better the thermal insulation is. The hot plate is set to 33°C to simulate the human skin temperature. From Figure 3d, the multifunctional cloth has a 93% and a 26% increase in temperature difference compared with the normal cotton cloth and the silica NP/cotton/cellulose acetate cloth, respectively, indicating that the thermal insulation is a collective effect of the silver network and the porous cloth.

The silver network has a sheet resistance of $2.8\text{--}4 \Omega/\text{sq}$, and such a highly conducting layer can also be used as a heater. Figure 3e shows time-dependent temperatures of a $2 \text{ cm} \times 2 \text{ cm}$ sample at applied voltages of 0.5, 1.2, and 1.5 V. The low sheet resistance of the multifunctional cloth enables the generation of Joule heating at a low voltage. At a room temperature of 25°C , an applied voltage of only 0.5 V can induce a temperature up to 34.1°C , higher than the average skin temperature of 33°C . Note that the voltage we tested is safe for the human body. To identify the durability of the silver network under repeated deformations, we have measured the

Table 1. Comparison of the Properties of the Normal Cotton Cloth, Ag Nanowire Cloth, and Multifunctional Cloth^a

sample	CA (deg)	IR transmittance (%)	ΔT (°C)	moisture permeation (mg/(cm ² ·h))	temperature at 0.5 V (°C)	antibacterial
normal cotton cloth	~0	72.3	1.5 ± 0.2	1.02	25	no
Ag nanowire cloth	~0	20.6	1.9 ± 0.7	0.869	36.4	yes
multifunctional cloth	161 ± 2	~0	2.9 ± 0.2	0.976	34.1	yes

^aCA, water contact angle on the outside of the cloth; ΔT is the difference in temperature between the hot plate top (33 °C) and the upper surface of the cloth.

sheet resistance under real-time cyclic bending at a bending radius of 5 mm for 1000 cycles (Figure 3f), and the sheet resistance shows only a 18.4% increase. Such a small change in sheet resistance typically does not have a perceptible effect on heating. The wear rate of the cloth is also shown in Figure S7. Although the wear rate increases with the increasing number of abrasion cycles, it is still in an acceptable range, indicating that the cloth is durable under mechanical wear.

BREATHABILITY AND ANTIBACTERIAL PROPERTY

The multifunctional cloth has a silica NP/PDMS coating on one side and a porous cellulose acetate film covered by a silver film on the other side. Especially, the porous cellulose acetate film fills up the gaps between the cotton yarn, and it is therefore necessary to investigate the effect of the added layers on breathability. The breathability of both the multifunctional cloth and the normal cotton cloth is characterized by the mass increase of the desiccant in a bottle sealed by the respective cloth. Water vapor permeates through the cloth and gets absorbed by the desiccant, leading to a weight gain over time. The multifunctional cloth can still maintain its good breathability, with a weight gain rate reduction of only ~4% compared with the normal cotton cloth (Figure 4a). All samples were tested in the same time duration and under the same environment to avoid possible deviation in ambient temperature and humidity. The result indicates that the multifunctional cloth exhibits high breathability and ensures comfortable wearing feeling with the aid of porous nature. Besides, the multifunctional cloth also exhibits the antibacterial property probably because nanoscale silver is an effective antibacterial material. As shown in Figure 4b–e, an empty gap around the multifunctional cloth reveals its ability to suppress the growth of bacteria, whereas neither the normal cloth nor those without the deposition of silver show this antibiotic effect. The multifunctional cloth can restrain the reproduction of bacteria and lengthen the lifespan of the personal thermal management cloth.

ROLE OF THE NANOPOROUS CELLULOSE ACETATE FILM

The porous cellulose acetate film plays several important roles. First, the hydrophilic and high-porosity materials maintain the good hygroscopicity and breathability of the fabric. The well-interconnected nanopores with hydrophilicity penetrate throughout the whole film, allowing water vapor to diffuse from one side to the other (Figure S8), which is fundamental for heat balance and comfort.²⁸ The resistance to diffusion in perforated films has been studied,²⁹ given as

$$R_w = \frac{t}{\beta} + 0.71d \left(\frac{1}{\beta} - \frac{1}{\sqrt{\beta}} \right) \quad (1)$$

where t is the thickness of the film, d is the average diameter of the pores, β is the percentage area of the pores, and R_w is the

resistance of the film in centimeters of still air. From eq 1, the water vapor transmission through the surface increases as the pore size decreases on a constant porosity and thickness. Therefore, the interconnected nanopores contribute positively to the breathability of the cloth. This might be the reason that although the large gaps (between the fiber yarn, ~200 μ m) are filled up with the nanoporous film, the breathability of the textile has a little change. A cloth that is both waterproof and breathable provides the wearer with a high level of comfort in many situations.

Second, the porous film fills up the sub-millimeter holes between the cotton yarn and thus increases the effective area of the IR reflector with a factor of ~33% (the area fraction of the gaps in the original cotton fiber). In addition, the metal film deposits not only on the walls between the nanopores but also fills inside the nanopores, thus offering an ideal IR reflection effect close to that of a smooth metal film and better than that of silver nanowires. This is the reason that our cloth exhibits full IR reflectance.

Third, the porous structure acts as a template for silver deposition, and the corresponding silver network offers a better durability than loosely assembled one-dimensional silver nanowires. Typically, silver nanowire suspension is quite expensive, whereas depositing a metal film is relatively cheaper. The metal network can be formed not only by using vacuum deposition but also by chemical plating and other methods. The deposited metal type can be diverse: other cheaper metals such as copper, nickel, and aluminum could also be deposited (Figure S9).

MULTIFUNCTION OF THE CLOTH

Some literature has reported smart clothes that meet different requirements, such as waterproof clothes,³⁰ energy harvesting clothes,^{31–33} antibacterial clothes,^{34–37} and energy management clothes.³⁸ However, these clothes typically have only a single function. In comparison, the multifunctional cloth we developed has a series of properties including waterproofness, breathability, antibacterial activity, and personal thermal control (as given in Table 1). Such a cloth with multifunctions will allow for diverse applications under harsh conditions. In addition, although the superhydrophobic feature makes it difficult to wash as the cloth floats on water, the antifouling and antibacterial properties allows our cloth to be wash-free. We have also tried dry cleaning, and the electrical resistance of the cloth exhibits only a slight increase after several cycles of dry cleaning tests (Figure S10).

CONCLUSIONS

In conclusion, we have fabricated a multifunctional cloth with a superhydrophobic silica NP/PDMS layer on one side and a metallic silver network templated by a porous film on the other surface. The multifunctional cloth not only maintains the original nature of cotton textile with a high breathability and flexibility but also possesses other desired properties including

waterproofness, antibacterial activity, IR reflection, and the ability to generate Joule heat. The porous cellulose acetate film plays important roles: it increases the IR reflection efficiency and maintains the high breathability. This multifunctional cloth might have practical applications in wearable electronics and smart garments and offers a new design consideration for thermal control.

■ ASSOCIATED CONTENT

● Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acsami.7b16422.

Materials required; methods of preparation of the waterproof layer, cellulose acetate porous film, and Ag network layer; characterization analysis of the multifunctional cloth; cross-sectional SEM image of the multifunctional cloth; schematic procedure of the preparation of the superhydrophobic cotton textile; SEM images of the membrane surfaces with varying cellulose acetate concentrations; images of the textiles at different stages; waterproofness testing of the textile; wear rate plot for the multifunctional cloth; optical photographs showing time-dependent water contact angles; SEM images of the Ag nanowire cloth and nickel network cloth; and the change of resistance of the multifunctional cloth after several dry cleaning cycles (PDF)

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Author Contributions

Q.L. and C.F.G. conceived the idea and designed the experiments. Q.L. and J.H. fabricated the multifunctional cloth and measured the water contact angle, infrared spectrum, thermal imaging, breathability, bending experiment, and Joule heating. J.Z. and M.G. conducted the SEM characterization. Y.H. and Y.W. drew the schematic diagram. Q.W. conducted the antibiotic test. Q.L. and C.F.G. analyzed the data and co-wrote the paper. All the authors discussed the result of the whole paper.

Notes

The authors declare no competing financial interest.

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