Preparation of Super-hydrophobic Cotton Fabrics with Conductive Property based on Graphene/Ag Composite Aerogels

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Abstract

In this work, graphene/Ag (GA/Ag) composite aerogels with the micro rough porous structure and conductive property and polydimethylsiloxane (PDMS) with the low surface energy were used to prepare super-hydrophobic cotton fabric (GA/Ag/PDMS@cotton) with conductive property was successfully prepared. The surface morphologies, chemical composition, wettability, conductive property and microwave absorption property of GA/Ag/PDMS@cotton fabric were characterized. It was shown that GA/Ag and PDMS were successfully coated onto the surface of cotton fabrics. The water contact angle of GA/Ag/PDMS@cotton was 157°. And it could make the luminous diode shining continuously. The treated cotton fabrics exhibited excellent super-hydrophobic and conductive properties. The minimum reflection loss of GA/Ag/PDMS@cotton fabric was −28dB, and it had a larger effective absorption bandwidth. In addition, the treated cotton fabric also showed excellent anti-fouling and self-cleaning properties. The GA/Ag/PDMS@cotton fabric cannot be wetted and polluted by water, fruit juice, milk and black tea. And it also had excellent laundering durability, acid and alkali corrosion and storage stability.

1 Introduction

At present, electromagnetic waves generated by electronic devices are a serious threat to people's health\(^1\)–\(^3\). The free electrons in the conductive material can form resonance with the frequency of the electromagnetic wave, and the absorption and reflection of the electromagnetic wave are both carried out\(^4\)–\(^6\). Endowing textiles with conductivity can solve the problem of electromagnetic wave pollution. However, the conductive textiles are susceptible to be polluted during application and its lifespan is reduced. Super-hydrophobic and conductive textiles with breathability and flexibility, not only have the characteristics of self-cleaning, anti-fouling and waterproofing\(^7\),\(^8\), but also absorb and reflect electromagnetic waves, which is expected to prolong the lifespan. It can be widely used in the field of electromagnetic shielding textiles, such as protective sleeves for precision electronic instruments, electromagnetic radiation protective clothing, etc\(^9\),\(^10\). And multi-functional textiles with high additional properties emerge gradually, which could better meet people's needs.

From an academic point of view, a super-hydrophobic surface refers to a surface with a static water contact angle greater than 150° and a rolling angle less than 10°\(^11\),\(^12\). Construction of super-hydrophobic and conductive textiles generally needs the microscopic rough structure, low surface energy and conductivity. Graphene aerogel is a three-dimensional material with a hierarchical porous rough structure\(^13\),\(^14\). And there is a large amount of air in its pores\(^15\),\(^16\), which is conducive to the construction of super-hydrophobic materials. However, graphene aerogels were currently widely used in dye wastewater adsorption\(^17\)–\(^19\) and other fields. Their application in super-hydrophobic and conductive textiles is rarely studied. The porous structure of graphene aerogel can make the electromagnetic wave reflect multiple times inside the material, which further improves the wave-absorbing performance. Ag is considered to be the metal material with the better conductivity\(^20\). Compounding graphene aerogel and Ag nanoparticles to prepare graphene/Ag composite aerogels could not only solve the problems of single
conduction mechanism and impedance matching\textsuperscript{[21–23]}, but also protect Ag from external oxidation. Moreover, the graphene/Ag composite aerogels were beneficial for the preparation of super-hydrophobic and conductive textiles due to the unique porous structure of graphene aerogels and the inherent properties of Ag nanoparticles. Zheng et al.\textsuperscript{[24]} fabricated super-hydrophobic and conductive cotton fabrics by assembling carboxylated and aminated multi-walled carbon nanotubes and modifying them with polydimethylsiloxane. The cotton fabric possessed a water contact angle of 162° and a resistivity value of about 8.57kΩ/cm. Liao et al.\textsuperscript{[25]} prepared super-hydrophobic and conductive polydimethylsiloxane@silver nanowires (PDMS@AgNWs) cotton fabrics by a simple dip-thermal curing method. Due to the combination of the rough structure of AgNWs and low surface energy PDMS, the prepared cotton fabric exhibits super-hydrophobicity with a water contact angle of 156° and excellent conductivity. In this work, the super-hydrophobic and conductive cotton fabric with synergistic self-cleaning and wave-absorbing properties were prepared by combining graphene/Ag composite aerogels with low surface energy material polydimethylsiloxane(PDMS).

In this paper, the super-hydrophobic and conductive cotton fabrics were prepared with hierarchical-porous graphene/Ag composite aerogels and low surface energy PDMS by spraying them onto cotton fabrics. The surface morphology, chemical composition, super-hydrophobic property, conductive property, microwave absorption property and durability of super-hydrophobic and conductive cotton fabrics were investigated. The obtained cotton fabric finally exhibited excellent super-hydrophobic property with water contact angle of 157° and good microwave absorption property with reflection loss of -28dB. Additionally, the super-hydrophobic and conductive cotton fabrics possessed self-cleaning property and anti-fouling property. It could be wetting and polluted by water, fruit juice, milk and black tea. It also exhibited good resistance to washing, acid and alkali corrosion and storage stability.

**2 Materials & Methods**

**2.1 Materials**

Graphene oxide(GO) was purchased from Nantong Qiangsheng Safety Protection Technology Co., Ltd, Jiangsu, China. Ammonia solution(NH\textsubscript{3}·H\textsubscript{2}O, 25%-28%), ethylenediamine(EDA, ≥ 99.0%), ethanol, silver nitrate(AgNO\textsubscript{3}, ≥ 99.8%), tetrahydrofuran(THF) and hydrazine hydrate aqueous solution (N\textsubscript{2}H\textsubscript{4}·H\textsubscript{2}O, 50%) were bought from Sinopharm Chemical Reagent Co., Ltd. (SCRC, China). Polydimethylsiloxane (PDMS, Sylgard 184) and its curing agent were bought from Dow Corning, America.

**2.2 Preparation of graphene/Ag composite aerogels**

By hydrothermal reaction at 180°C for 12h with addition agent(ethylenediamine and ammonia), the graphene aerogel(GA) was prepared successfully. The graphene/Ag composite aerogel was prepared by immersing the obtained graphene aerogel into AgNO\textsubscript{3} solution (12wt%) for 50min, and then dipping into hydrazine hydrate solution for 30min. After drying at 60°C for 3h in an oven, the obtained composite aerogels were denoted as GA/Ag.
2.3 Preparation of super-hydrophobic and conductive cotton fabrics

First, the prepared GA/Ag was ground into powder, and a certain amount of powder was dispersed in THF to prepare a GA/Ag dispersion with a concentration of 3wt%. On the other hand, a certain amount of PDMS and its curing agent were added into THF to form a homogeneous PDMS solution with a concentration of 3wt%. Finally, the GA/Ag dispersion and PDMS solution were sequentially sprayed on the surface of a clean cotton fabric with a size of 3cm×3cm. The super-hydrophobic and conductive cotton fabric (GA/Ag/PDMS@cotton) was prepared successfully. Additionally, PDMS@cotton was also prepared to serve as control for GA/Ag/PDMS@cotton.

2.4 Characterization

In this work, the scanning electron microscope (SEM, Hitachi S-4800) was used to evaluate the surface microstructures of the GA and GA/Ag. The surface chemical structure and elements of samples were analyzed by fourier infrared spectrometer (FT-IR, Avatar 380) and X-ray photoelectron spectroscopy (XPS, Escalab250Xi). The nitrogen adsorption-desorption isotherms and pore size distribution of GA and GA/Ag were analyzed by automatic specific surface area and porosity analyzer (BET, Autosorb-iQ). The super-hydrophobicity of pristine cotton, PDMS@cotton and GA/Ag/PDMS@cotton were explored by a video optical contact angle measuring instrument (KRUSS DSA30). The microwave absorption properties of GA/Ag/PDMS@cotton was measured by vector network analyzer (AV 3672B).

3 Results And Discussion

3.1 Formation mechanism of GA/Ag/PDMS@cotton

The graphene/Ag composite aerogel exhibited excellent hierarchical porous micro-rough structure. Thus, the graphene/Ag composite aerogel was beneficial for the construction of surface roughness of super-hydrophobic materials, which can effectively increase the contact area between the air and the surface of the material. Polydimethylsiloxane was a hydrophobic organosilicon material with terminal methyl group, which had the advantages of high strength and good wear resistance. PDMS had a certain adhesion to textiles and could improve the firmness of textiles. First, the graphene/Ag composite aerogel was ground into powder and dispersed into THF solution to prepare the composite aerogel dispersion. And the cotton fabric with micro-rough structure was obtained. Then, the PDMS solution was prepared by dissolving PDMS into a THF solution. The treated cotton fabric with micro-rough structure and low surface energy was obtained. As shown in Fig. 1, the composite aerogel dispersion and the PDMS solution were sprayed on the surface of the cotton fabric successively. After drying, the super-hydrophobic and conductive cotton fabric (GA/Ag/PDMS@cotton fabric) was obtained.

3.2 Surface morphology analysis.
The surface morphology of the PDMS@cotton and GA/Ag/PDMS@cotton was investigated by SEM. As shown in Fig. 2(a), the surface of the cotton fabric treated only with PDMS was relatively smooth, and the fiber surface was covered with a dense and uniform PDMS film. However, when the cotton fabric was loaded with GA/Ag and PDMS, it could be found that the micro-roughness of the cotton fabric surface was significantly improved. A large number of composite aerogel particles covered the surface of the cotton fabric, and the particles were distributed evenly (Fig. 2b and Fig. 2c). The pores of the graphene/Ag composite aerogel were partly filled with PDMS, and GA/Ag and PDMS were closely connected to each other, forming a dense film on the surface of the fabric. It gave the surface of the cotton fabric a certain micro roughness and low surface energy. Good super-hydrophobic and conductive surface was obtained.

In addition, the surface elements of GA/Ag/PDMS@cotton were tested by EDS. As shown in Fig. 2(d-h), the five element mapping diagrams of C, N, O, Si, Ag were shown, respectively, which could be observed that these five elements were uniformly distributed on the surface of the GA/Ag/PDMS@cotton. The C element was mainly derived from GA and the Ag element came from Ag nanoparticles. The Si element indicated the existence of PDMS. These all showed GA/Ag and PDMS were coated onto the cotton fabric surface successfully.

3.3 Chemical composition analysis.

The chemical structure of cotton fabrics were studied by FT-IR. As shown in Fig. 3(a), the FT-IR curves of pristine cotton fabric, PDMS@cotton and GA/Ag/PDMS@cotton were similar. GA/Ag/PDMS@cotton fabric had a strong and wide absorption band near 3335cm$^{-1}$, which was the stretching vibration absorption peak of hydroxyl(-OH) in cellulose macromolecule of cotton fabric. The stretching vibration peak of -CH$_2$- group was located at 2850cm$^{-1}$. The strongest band was located at 1065cm$^{-1}$, and these bands were mainly generated by the stretching vibration absorption of C-O-C group in cellulose macromolecules. The peak located at 2967cm$^{-1}$ in the FT-IR curves of PDMS@cotton was -CH$_3$ group. And there was also a strong absorption peak at 1259cm$^{-1}$, which was the bending vibration peak of Si-C. The peak located at 790cm$^{-1}$ indicated the symmetrical stretching vibration peak of Si-O-Si group.

In this paper, the chemical composition of cotton fabric surface before and after coating were further analyzed by XPS. As shown in Fig. 3(b), it indicated the XPS spectrum of pristine cotton and GA/Ag/PDMS@cotton. The pristine cotton fabric had strong absorption peaks at 284.0eV and 532.0eV corresponding to C1s and O1s, respectively. On this basis, the two peaks located at 102.0eV and 152.0eV were the absorption peaks of Si 2p and Si 2s, which were attributed to the introduction of low surface energy substance PDMS. It could also be found that the exist of N1s peak at 399eV and the Ag 3d peak at 371eV. The N element came from the addition of EDA molecules during the preparation of GA. The appearance of Ag element was because of the Ag nanoparticles on the surface of GA/Ag. To sum up, the successful loading of GA/Ag and PDMS on the surface of cotton fabric was confirmed again.

3.4 Super-hydrophobic properties analysis.
In this work, GA/Ag composite material provided the micro-level roughness for the treated cotton fabrics. And the low surface energy of fabrics was given by PDMS. On this basis, a surface with excellent super-hydrophobic performance was constructed. As shown in Fig. 4(a), the water contact angle of pristine cotton fabric was 0° because of the inherently hydrophilic of cotton fabric. The water contact angle of PDMS@cotton was 123°, which didn’t have super-hydrophobicity. However, compared with PDMS@cotton, the water contact angle of GA/Ag/PDMS@cotton reached 157° due to the rough structure of GA/Ag and the low surface energy of PDMS. The GA/Ag material possessed excellent hierarchical porous structure, which was beneficial for the construction of surface roughness. As shown in Fig. 4(b), the different droplets (water, juice, cola and milk) were dropped on the surface of GA/Ag/PDMS@cotton, these droplets always kept stable spherical shape, and the fabric surface also couldn’t be wetting. However, these droplets on the pristine cotton fabric were quickly absorbed. The pristine cotton fabric soaked in water was wetting quickly so that it sunk to the bottom of the beaker due to its inherent hydrophilic properties. But, the GA/Ag/PDMS@cotton was always floating on water and couldn’t be wetting (Fig. 4c). In addition, the surface of GA/Ag/PDMS@cotton produced a dense air layer (“silver mirror” phenomenon), which effectively inhibited the penetration of water droplets (Fig. 4d).

Liquid repellency was also a distinguishing feature of super-hydrophobic textiles, which exhibited anti-adhesion properties to water droplets. As shown in Fig. 5, the continuously falling methylene blue dye aqueous solution can’t stay on the surface of GA/Ag/PDMS@ cotton, but roll off quickly. The surface of treated cotton fabric remained dry and clean and the surface was not wetting. The GA/Ag/PDMS@cotton fabric exhibited excellent liquid repellency. This phenomenon was attributed to the combined effect of GA/Ag and PDMS. GA/Ag provided the hierarchical porous rough structure and PDMS constructed the low surface energy of the treated cotton fabric.

The treated cotton fabric not only had super-hydrophobic properties, but also exhibited excellent anti-fouling properties and self-cleaning properties. As shown in Fig. 6, the GA/Ag/PDMS@cotton fabrics exhibited excellent anti-fouling properties when immersed in various liquids. When the treated cotton fabric was immersed in milk (Fig. 6a1-a3), fruit juice (Fig. 6b1-b3), dye (Fig. 6c1-c3) and black tea solution (Fig. 6d1-d3), the GA/Ag/PDMS@cotton fabric surface remained dry when it was pulled out. And small droplets were also not stained. As shown in Fig. 6e, a layer of soluble contaminants was spread on the inclined treated cotton fabric, and then the surface was flushed by water droplets. It could be seen that the water droplets quickly roll off the surface of the sample and could take away the dirt, keeping the surface clean.

### 3.5 Conductive properties analysis.

In order to explore the conductivity of GA/Ag/PDMS@cotton, PDMS@cotton and GA/Ag/PDMS@cotton were connected with a 5mm luminous diode under the voltage of 3V. As shown in Fig. 7(a), in the circuit formed by PDMS@cotton, the luminous diode was not lighting. This was attributed to the electrical insulation of PDMS. However, in the circuit formed by GA/Ag/PDMS@cotton, the diode was shining
constantly (Fig. 7b). This indicated that the GA/Ag/PDMS@cotton fabric had excellent electrical conductivity and stable electrical conductivity in the conductive path.

Graphene sheets were self-assembled layer by layer to form GA. There was a large π bond formed by sp² hybrid orbital, and there were free moving electrons in the π bond. Electrons could flow in a certain direction to form a current under voltage. Ag existed in GA/Ag with the form of a metal element. The metal atoms were bound to their valence electrons weakly. And some of the valence electrons were easily separated from the metal atoms to become free electrons. The bonds formed in this way were called metallic bonds. This theory was called free electron theory. The directional movement of free electrons in the Ag structure generated electric current, and metal atoms or ions hindered the movement of electrons to generate resistance. The composition of GA and Ag improved the conductivity of GA/Ag. Benefiting from the porous structure of aerogel and the synergistic effect of graphene and Ag, GA/Ag exhibited excellent conductivity. GA/Ag/PDMS@cotton fabrics were prepared by combining GA/Ag with PDMS. Although PDMS had electrical insulating properties, leading to the conductive effect of treated cotton fabrics decreases. In the conductive path, the diode was still brighten, indicating that GA/Ag/PDMS@cotton fabric still exhibited good conductivity.

3.6 Microwave Absorption Properties analysis.

In this paper, we also studied the microwave absorption properties of different materials through vector network analyzer. As shown in Fig. 8, GA had the reflection loss value of -25.1dB at the frequency of 11.2GHz, and the effective bandwidth is 3.4GHz (9.7 ~ 13.1GHz). The graphene/Ag composite aerogel had the smallest reflection loss (-40dB) at 10.9GHz. The effective frequency band was 8.8-12.8GHz and the effective bandwidth was 4GHz. Due to the mutual synergy and impedance matching between GA and Ag nanoparticles, the composite aerogel exhibited superior microwave absorption property. The GA/Ag/PDMS@cotton was prepared by combining GA/Ag and PDMS on the surface of cotton fabric. The result showed that the GA/Ag/PDMS@cotton had a reflection loss of -28dB and a large effective absorption bandwidth. The reflection loss of PDMS@cotton was close to 0. It didn't have microwave absorption property. In addition, the microwave absorption property of GA/Ag/PDMS@cotton was worse than that of GA/Ag, which was mainly attributed to the electrical insulation and physiological inertness of PDMS.

The GA/Ag material possessed a hierarchical porous structure, when the electromagnetic wave from the external environment entered into the surface of GA/Ag, multiple reflections occurred inside the material, which was beneficial to improve the microwave absorption performance. And oxygen-containing groups in GA/Ag could produce Fermi energy levels and polarization relaxation, which could effectively prevent the generation of transmission waves. The electrical energy was dissipated in the form of heat, which further improved the wave-absorbing properties of the material. In addition, the porous structure not only effectively reduced the density of the material, but also caused multiple reflections of electromagnetic waves, accelerating the loss of electromagnetic energy.
3.7 Durability analysis.

Generally, super-hydrophobic textiles were exposed to the external environment for a long time, including regular washing, erosion of rainwater, etc., which would lead to the weakening of super-hydrophobicity and wave absorbing properties. In order to ensure the lifespan of the product and reduce environmental pollution, it was very important to endow the treated cotton fabrics with good durability. In this paper, the stability of the super-hydrophobic properties and microwave absorption properties of GA/Ag/PDMS@cotton in various environments was evaluated, including washing resistance, corrosion resistance, and placement stability.

The laundering durability of treated cotton fabrics was tested by ultrasonic washing. With the increase of ultrasonic time, the water contact angle of GA/Ag/PDMS@cotton fabric decreased slightly, but it was still above 150°, and the change of resistivity was also very slight (Fig. 9a). Since the nanosheets of graphene aerogel could tightly wrap the Ag nanoparticles, the synergistic effect of PDMS made it firmly adhere to the textile surface and behaved very robust. As shown in Fig. 9(b), after ultrasonic washing for 90 minutes, the reflection loss of the treated cotton fabric increased to -24dB, and the effective bandwidth was shortened, showing excellent washing resistance.

The treated cotton fabrics were immersed in acid-base salt solutions of different pH for 6 h, and the changes of water contact angle, resistivity and reflection loss were observed. The corrosion resistance of GA/Ag/PDMS@cotton was analyzed. It could be seen from Fig. 10(a) that compared with the ultrasonic washing treatment, the water contact and resistivity of cotton fabrics treated in acidic and alkaline solutions changed larger. When pH = 1 and pH = 13, the water contact angle of treated cotton fabrics was lower than 150°. The treated cotton fabric possessed good hydrophobic properties. After soaking in strong acid solution of pH = 3 for 6h, the reflection loss of GA/Ag/PDMS@cotton fabric increased about 4dB. After soaking in strong alkali solution of pH = 11 for 6h, the reflection loss of GA/Ag/PDMS@cotton increased about 4.5dB (Fig. 10b), showing better acid and alkali resistance.

Because the treated cotton fabrics were exposed to the external environment for a long time, the placement stability of the treated cotton fabric was observed by placing the GA/Ag/PDMS@cotton at room temperature for 30 days. It could be observed from Fig. 11(a) that with the increase of days, the water contact angle and resistivity of the treated cotton fabric hardly changed. Just the resistivity increased slightly after 20 days. And the fabric was placed for 30 days with negligible change in reflection loss (Fig. 11b). In conclusion, after the durability test, the GA/Ag/PDMS@cotton fabric showed little change in super-hydrophobic properties and microwave absorption properties. It showed excellent laundering durability, corrosion resistance and storage stability, which could be widely used in daily life and production.

To sum up, the GA/Ag/PDMS@cotton fabric had excellent laundering durability, corrosion resistance and storage durability. In the internal structure of graphene aerogel, the interconnection between its carbon atoms has a certain toughness. When the graphene aerogel was under the action of external force, its
carbon atomic surface was bent and deformed, and the carbon atoms could resist the external force without rearrangement. Thus it maintained the stable structure of the graphene aerogel. When Ag nanoparticles were deposited on the framework of graphene aerogel, the large lamellar structure of the aerogel could wrap Ag nanoparticles in the 3D network, which is protected from the external environment. When the GA/Ag material and PDMS were combined and treated on the surface of cotton fabric, the GA/Ag material was fastened onto the surface of cotton fabrics due to the strong adhesiveness of PDMS. In addition, PDMS had the characteristics of physiological inertness, good chemical stability, weather resistance and waterproof performance. PDMS could wrap the GA/Ag on the surface of cotton fabric. So GA/Ag/PDMS@cotton fabric exhibited excellent chemical stability.

4 Conclusions

In summary, GA/Ag/PDMS@cotton with excellent super-hydrophobic and conductive property was prepared by a simple spraying method with graphene/Ag composite aerogel as the micro-rough and conductive material and PDMS as the low surface energy material. The water contact angle of the treated cotton fabric is 157°. It exhibited excellent self-cleaning and anti-fouling properties, and couldn’t be wetting and polluted by water, juice, milk and black tea. The treated cotton fabric was connected with the light-emitting diode to form a channel, and the diode emits kept shining, which had excellent conductivity. In addition, the treated cotton fabric also had excellent microwave absorption performance. Its reflection loss was −28dB, and it had a large effective absorption bandwidth. It also had excellent laundering durability, acid and alkali corrosion and placement stability.

Declarations

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Declaration of competing interest

The authors have declared that no conflict of interest exists.

Author contributions

Yangchun Liu and Lihui Xu wrote the main manuscript text and Yangchun Liu prepared figures 1-11. All authors reviewed the manuscript.

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**Figures**
Figure 1

Fabrication process of GA/Ag/PDMS@cotton

Figure 2

SEM images of (a) PDMS@cotton, (b) GA/Ag/PDMS@cotton with high magnification, (c) GA/Ag/PDMS@cotton with low magnification. (d-g) Element mapping images for C, O, Si, Ag, respectively. (h) EDS energy spectrum

Figure 3

(a) FT-IR spectra of pristine cotton, PDMS@cotton and GA/Ag/PDMS@cotton. (b) XPS spectrum of pristine cotton and GA/Ag/PDMS@cotton
Figure 4

(a) The contact angle of pristine cotton, PDMS@cotton and GA/Ag/PDMS@cotton. (b) Different liquid droplets on the surface of the pristine cotton fabric and GA/Ag/PDMS@cotton. (c) Pristine cotton and GA/Ag/PDMS@ cotton soaked in water. (d) Silver mirror phenomenon.

Figure 5

Photographs of methylene blue dyestuff continuously rolling off the surface of GA/Ag/PDMS@cotton at different time stages.

Figure 6
Self-cleaning process of GA/Ag/PDMS@cotton

Figure 7

(a) Circuit diagram formed from PDMS@cotton. (a) Circuit diagram formed from GA/Ag/PDMS@cotton. (b) is a schematic diagram of the back connection of the circuit board.

Figure 8

Two-dimensional reflection loss graphs of different materials.

Figure 9

(a) Change of contact angle and resistivity of treated cotton fabric after ultrasonic washing. (b) Comparison of reflection loss before and after ultrasonic washing of treated cotton fabric for 90 minutes.

Figure 10

(a) Change of contact angle and resistivity of treated cotton fabric immersed in different pH solutions for 6h. (b) Comparison of reflection loss before and after finishing cotton fabric immersed in acid and alkaline solution for 6h, respectively.

Figure 11

(a) Change of contact angle and resistivity of treated cotton fabric after a period of time. (b) Comparison of reflection loss before and after finishing cotton fabric for 30 days.