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Superhydrophobization of Cotton Fabric with Multiwalled Carbon Nanotubes for Durable Electromagnetic Interference Shielding

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Abstract: In this work, durable electromagnetic interference shielding cotton fabrics were obtained via superhydrophobic finishing with Nafion-MWCNTs coating. Nafion, a perfluorosulfonated polymer, exhibits low surface energy and good dispersing performance for MWCNTs. The uniform distribution of MWCNTs is beneficial for not only forming connective conductive network, which enhances electrical conductivity and shielding performance of the cotton fabric, but also constructing a nano-micrometer dual scale structure, which is necessary for superhydrophobic surface. After 6-cycles of Nafion-MWCNTs deposition, the resultant fabric possesses a favorable shielding effectiveness of 9.0 dB and a water contact angle of 154.6 ^{o}. Besides of the satisfied shielding ability and superhydrophobic surface, more importantly, the fabric exhibits good durability in EMI shielding after immersing in water for 96 h or washing with AATCC standard due to the superhydrophobicity and good chemical stability of the Nafion-MWCNTs coating.

Keywords: Cotton fabric, Carbon nanotubes, Electromagnetic interference shielding, Superhydrophobicity, Durability

Introduction

Electromagnetic interference (EMI) shielding fabrics have recently attracted much attention for their wide use in protecting people from harm by a variety of electromagnetic radiation sources such as electric appliances, microwave oven, TV set, communication devices, mobile phone, mobile phone and TV towers. There are two typical strategies to prepare EMI shielding fabrics. One is to blend or interweave metal fibers with conventional fibers [1-4], which is inconvenient because of their high stiffness, high specific density and vulnerability to corrosion. The other is to coat a conductive layer on fabrics to endow with shielding property. A thick coating will bond the fibers together, making the fabric rigid and uncomfortable, while a thin coating could prevent the fabric from damaging its comfortability and flexibility [5]. Kaynak *et al.* prepared EMI shielding polyester Lycra fabric coated with a thin layer of polypyrrole in the submicron range, which endowed the fabric with flexibility [5]. Apart from conductive polymers, carbon materials, such as carbon black, carbon nanofiber, carbon nanotubes, graphite and graphene, as promising EMI shielding materials, have attracted more and more attention because of their lightweight, versatile processability, excellent electrical conductivity [6-12]. As a one-dimensional nano material, compared with zero-dimensional nanoparticles, carbon nanotubes (CNTs) have high aspect ratios, allowing them to intimately contact with fibers to form more complete, consistent and reliable conductive network with the same filler loading, and thus are suitable for improving textiles electrical conductivity and EMI shielding property. Alimohammadi *et al.* prepared EMI shielding cotton fabrics via polycarboxylic acid/MWCNTs coating, exhibiting an EMI shielding effectiveness (SE) value of ~1.5 dB in the frequency range of 5-8 GHz [13]. Gultekin and Usta coated cotton fabrics with Polyamide 6/MWCNTs for EMI shielding application, demonstrating a maximum EMI SE value of 3.7 dB in the frequency range of 15-3000 MHz [14]. Bonaldi *et al.* chose CNTs as conductive reinforcement to fill up the vacancy of the conductive network formed by nanoparticles (Ag or polypyrrole), further increasing the shielding effectiveness of fabrics to 15-40 dB [15]. However, it is still a big challenge to make the shielding performance of fabrics sustainable when suffering from repeated daily washings. Up to now, there are few reports on the water washing stability of shielding performance of fabrics [16,17].

Superhydrophobic surfaces are capable of strong repellency to water, which could effectively prevent water penetrating into a substrate and consequently efficiently protect it from degradation of performance. They have been integrated with other functions, such as water-oil separation, fluorescence property, antibacterial property, electroconductivity, ultraviolet resistance, and flame-retardant property. Through the fusion of different biological solution, Jin *et al.* fabricated metallic foams with superhydrophobicity and oil/water separation capability [18]. Jiang *et al.* spin-coated trimethylsiloxane functionalized SiO_2 onto a precoated polyurethane layer to prepare superhydrophobic, transparent and fluorescent multifunctional materials [19]. Interestingly, superhydrophobic surfaces on fabrics were also constructed combining with other functions. Vilčnik *et al.* designed superhydrophobic cotton fabrics to enhance the washing fastness [20]. Shateri-Khalilabad and Yazdanshenas prepared superhydrophobic

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cotton textiles for improving the stability of antibacterium and electroconductivity [21,22]. Wang and coworkers obtained reliable ultraviolet-blocking cotton textiles via superhydrophobization [23]. More recently, Chen *et al.* attempted at resolving the problem of washing stability of flame-retardant treatments by synthesizing self-healing superhydrophobic coating on a cotton fabric [24]. Based on above-mentioned studies which implied that superhydrophobic surfaces as barriers could efficiently defend other properties against damage, we believe that it is reasonable to integrate the superhydrophobicity with shielding property into fabrics for improving its durability of shielding properties.

In this study, superhydrophobization of cotton fabric was employed to enhance the durability of its EMI shielding by means of Nafion-MWCNTs coating. The durability was assessed via comparing the shielding effectiveness of the fabric before and after washing. Vector network analyzer (VNA) was utilized to measure the EMI shielding property of the fabrics with Nafion-MWCNTs deposition. Scanning electron microscope (SEM) images were taken to reveal the deposition of Nafion-MWCNTs on cotton fabrics. The surface resistance and water contact angle (WCA) of the Nafion-MWCNTs coated cotton were also measured after each deposition to evaluate their electrical conductivity and superhydrophobic property. Furthermore, the chemical stability of the superhydrophobic fabric was also evaluated at a pH range of 2 to 12.

Experimental

Materials

MWCNTs were provided by Chengdu Organic Chemicals Company, (diameter: 8-15 nm, length: 50 μm and purity: >95 %). Desized, scoured and bleached plain woven cotton fabrics, with an areal density of 96.4 g/m^2 were purchased from Shandong Lutai Textile Co., Ltd. Commercial Nafion solution was bought from Du Pont Company (5 wt%, 0.93 g/cm^3, 70,000-120,000 Da). All other chemicals were analytic reagent and used directly without further purification.

Preparation of Nafion-MWCNTs Cotton Fabrics

Dispersion of Nafion-MWCNTs was prepared as follows: 1.28 wt% Nafion solution used to disperse MWCNTs was prepared by adding same volume of deionized water and anhydrous ethyl alcohol. The nanoink of MWCNTs dispersed in aforementioned Nafion solution at a concentration of 2.5 mg/m*l* was then treated by ultrasonic cleaner with a power of 50 W and a frequency of 53 kHz for 2 h. The stable nanoink was used for the coating process.

Cotton fabrics with a dimension of 15×15 cm^2 were first cleaned with acetone, then rinsed with deionized water several times and finally dried in an oven. Then the cleaned fabrics were dipped into aforementioned nanoink for 5 min and dried in an oven at 80 °C for 10 min. This dip-drying process was repeated to increase the MWCNTs mass on the cotton for up to 6 cycles. For simplicity, the samples were coded as D1 to D6 representing 1 to 6 deposition cycles.

Characterizations

A Hitachi S-4800 field-emission scanning electron microscope (FE-SEM) was used to observe the surface morphologies of cotton specimens before and after coating with MWCNTs at an accelerating voltage of 5 kV. Each specimen was coated with gold prior to the observation.

An OCA15EC type contact angle tester was utilized to test the WCA of the MWCNTs coated cotton fabrics. A microliter syringe was utilized to place the distilled water droplet on the fabrics. A 3 μ*l* distilled water droplets were dropped carefully onto the cotton fabrics. Each of the contact angle value represents the average of at least five measurements at different positions on the sample.

Standard two-probe method was employed to measure the surface resistance of the samples referring to AATCC 76-2005 by means of Fluke 15B multimeter, which is recommend for not highly conductive textiles materials [22]. The surface resistance was calculated according to the following equation:

$$R_s = RW/L \tag{1}$$

where R_s is the surface resistance (Ω/□), R is the resistance (Ω) of the fabric recorded by the multimeter, L and W are the length and width of the test area between the two probes (here, L=W=50 mm), respectively.

A Rohde & Schwarz ZVL6 type vector network analyzer (VNA) was used to measure EMI shielding effectiveness of MWCNTs deposited cotton fabrics in the frequency range of 3.9-6.0 GHz (C-band). In order to fit a waveguide holder, the sample was cut into a rectangle with a dimension of 48×22 mm^2.

To avoid the influence of temperature and humidity on the testing results, all measurements were completed in a standard testing condition of 65±2 % relative humidity and 21±1 °C.

Results and Discussion

Deposition of Nafion-MWCNTs on Cotton Fabrics

The cotton fabric coated by Nafion and MWCNTs is schematically illustrated in Figure 1(a). Nafion was used as a linker between hydrophilic cotton and hydrophobic MWCNTs because of its polar side chains and hydrophobic backbone, in addition to providing a homogenous distribution of MWCNTs. The process of deposition with Nafion-MWCNTs can be repeated on cotton fabrics because of the van der Waals interactions between the coating components of Nafion and MWCNTs. The repeatabilility of Nafion-CNTs on cotton yarns was realized by Shim *et al.* [25]. Furthermore, Pasta *et al.* [26] repeatedly deposited CNTs ink to increase the CNTs loading on cotton fabric via the van der Waals interactions

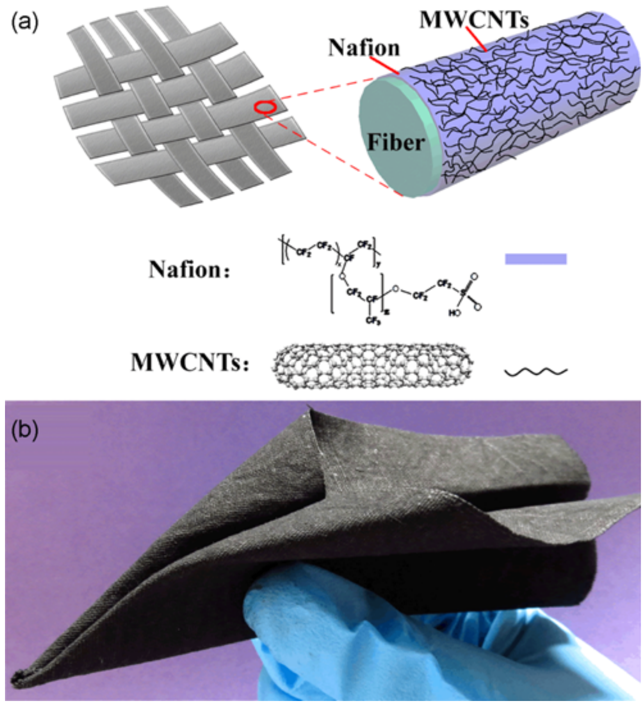

Figure 1. Structural schematic (a) and (b) photograph of cotton fabric coated with Nafion-MWCNTs.

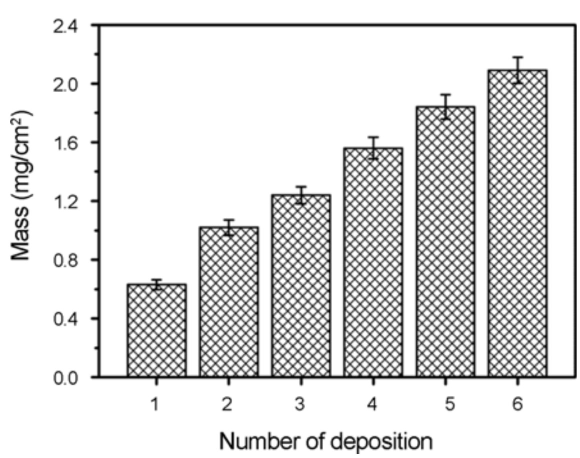

Figure 2. Growth of Nafion-MWCNTs coating with different number of deposition.

between cotton fiber and CNTs. The fabric coated with six times of Nafion-MWCNTs deposition is still very flexible (Figure 1(b)). With the deposition of Nafion-MWCNTs, the mass of coating increased from 0.63 mg/cm^2 to 2.09 mg/cm^2 as shown in Figure 2, which is beneficial to improve the electrical conductivity of the fabric similar to the result obtained by Shim *et al.* [25].

The surface morphologies of the cotton fabrics coated by Nafion-MWCNTs are shown in Figure 3. The surface of the pristine cotton fibers is smooth with convolution, possessing grooves and veins (Figure 3(a), (b)). Figure 3(c), (e) and (g) present the SEM pictures of the cotton fiber after one, three and six times of deposition at a low magnification, respectively. With the deposition of Nafion-MWCNTs, a thin layer of coating was clearly observed. Especially, Figure 3(d), (f), (h) clearly shows that the individual cotton fibers are fully covered by MWCNTs with little aggregation, which constructs the nanoscale structure on the microscale fiber. Moreover, the amount of MWCNTs on cotton fibers increased with the number of deposition, which is consistent with the growth of Nafion-MWCNTs coating (Figure 2).

Superhydrophobicity of Cotton Fabric

With respect to superhydrophobicity, large-surface roughness and low-surface energy are the two essential key factors. Generally, fabrics are fabricated with yarns and each yarn is comprised of many fibers. The hierarchical structure of a fabric and nanostructure of MWCNTs endow the MWCNTs coated fabric with a micro-nano dual-scale structure, which is beneficial for increasing roughness. In addition, Nafion, a perfluorosulfonated polymer, could provide low surface energy [27]. Therefore, it is rational that fabricating a superhydrophobic surface is possible with Nafion-MWCNTs on hydrophilic cotton fabrics.

As expected, the water dyed with rhodamine B was absorbed rapidly to the original cotton fabric (Figure 4(a)) because of containing abundant hydroxyl groups which provide perfect hydrophilic character. However, after treatment with Nafion only or Nafion-MWCNTs nanoink, the cotton fabric surfaces were transformed from hydrophilic to hydrophobic (Figure 4(b), (c)). Furthermore, the surface wettability of the cotton fabric was estimated via WCA measurement. As shown in Figure 5, WCAs of the fabrics increased with the increase of deposition cycles. Fabrics with three deposition cycles achieved superhydrophobic level, i.e., WCA larger than 150^{o}. The WCA of fabric with six deposition cycles increased further to 154.6^{o}.

There are two classical models, namely Wenzel's model and Cassie's model, to explain superhydrophobicity. Generally, the Wenzel's model is suitable for homogeneous interface without any air pockets. However, cotton fabric is a porous substrate, even after it was coated with Nafion-MWCNTs, which could be seen from SEM images (Figure 3). In this situation, air could be trapped below the drop, forming air pockets, which was expected to be modeled by Cassie's equation as follows.

$$\cos\theta_r = f_1\cos\theta - f_2 \qquad (2)$$

where θ_r is the apparent WCA on a rough and porous surface, i.e., the observed WCA, θ is the intrinsic WCA on the corresponding flat surface, f_1 and f_2 are the liquid/solid interface area and liquid/air interface area beneath the droplet divided by the projected area (f_1+f_2=1), respectively. This equation was further modified to explain the surface

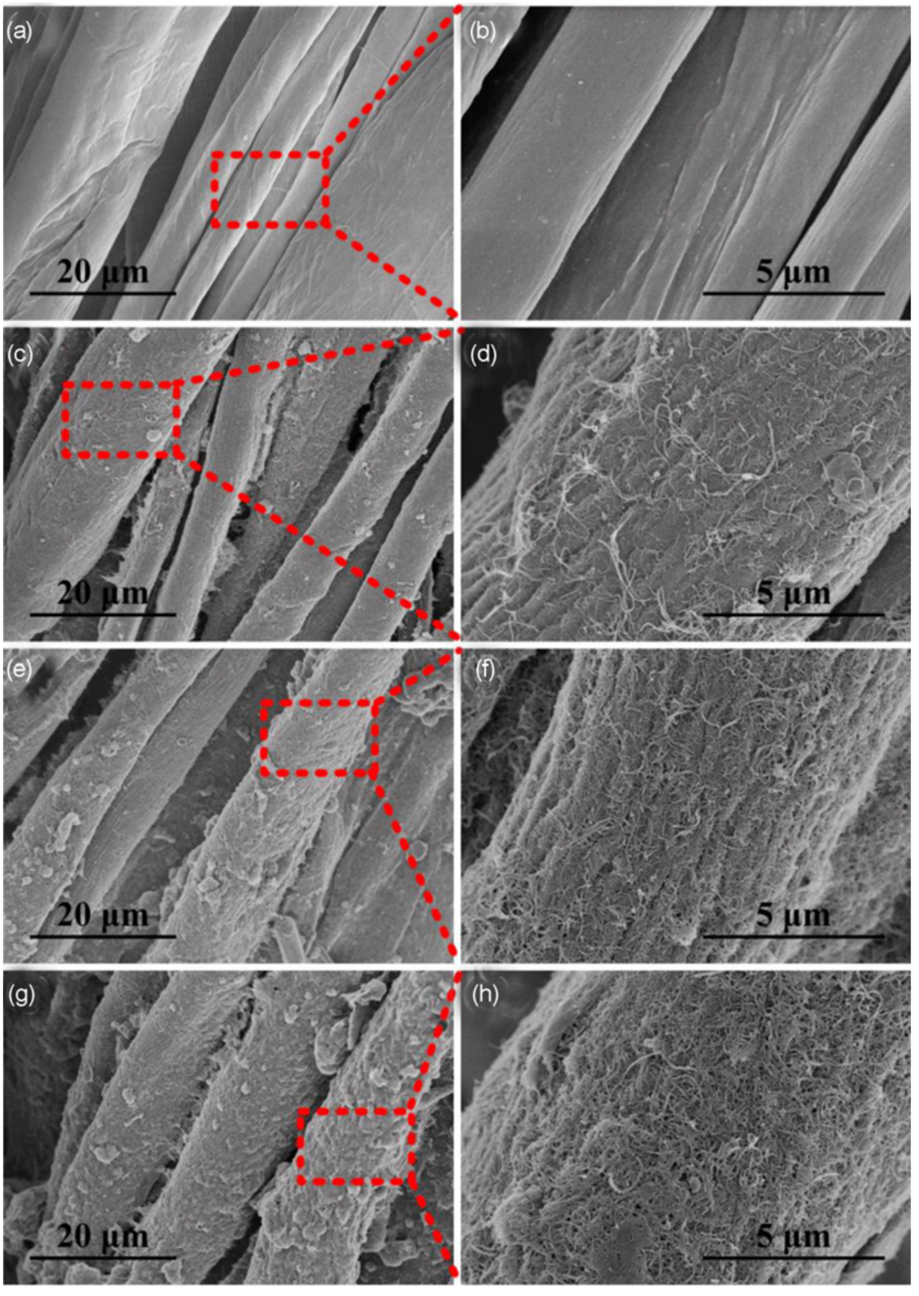

Figure 3. SEM images of pristine cotton fiber (a, b), cotton fiber with one (c, d), three (e, f) and six (g, h) deposition cycles.

Figure 4. Photographs of water droplet on pristine cotton (a), (b) cotton with Nafion-MWCNTs, cotton with Nafion only (c).

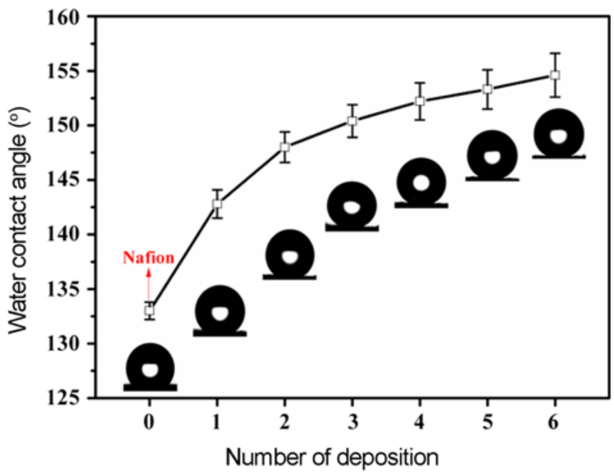

Figure 5. Water contact angle with various numbers of deposition.

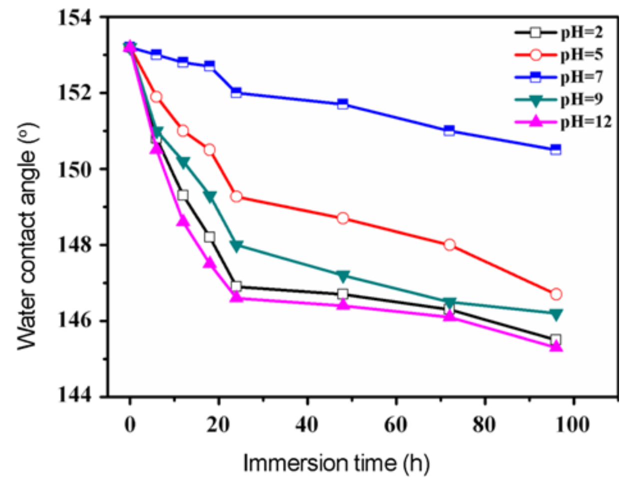

Figure 6. Water contact angle versus immersion time at various pH values on superhydrophobic cotton fabric.

roughness on the wetted area as follows (equation (3)) [28]:

$$\cos\theta_r = rf_1\cos\theta - f_2 \quad (3)$$

where r is the surface roughness factor of the wetted area ($r \geq 1$). WCA increases with augment of roughness. In our study, it was obvious that the roughness of the fabrics greatly increased with the introduction of MWCNTs, since raw cotton fiber was smooth (Figure 3(a), (b)) and became rougher and rougher after deposition of Nafion-MWCNTs (Figure 3(c)-(h)). In order to further prove this, a controlled experiment was conducted. A cotton fabric was modified by Nafion solution alone under the same conditions without MWCNTs. In this case, a 3 μl water droplet was softly dropped on the modified surface, and the apparent WCA was 133 ^{o} (Figure 5), smaller than that on the surface deposited with Nafion-MWCNTs. Therefore, MWCNTs play an important role in increasing roughness and forming nano-microscale dual structure, facilitating formation of superhydrophobic surface.

Stability of Superhydrophobic Cotton Fabric

It is necessary for practical applications that superhydrophobic surface possesses the environmental stability. In present, the chemical resistance of the Nafion-MWCNTs coated cotton fabric (D5) was evaluated by measuring the change of WCA values after being treated with aqueous solutions of varying pH (Figure 6) according to the method described previously by Li *et al.* [29]. Figure 6 reveals that superhydrophobic cotton fabric displays a high stability with WCA >145 ^{o} after treatment with acidic, neutral and basic solution for 96 h though WCA values decrease with prolonging immersion time. Furthermore, we found that the modified cotton fabric maintained the initial WCA value after 1 month storage in air, indicating that the superhydrophobic fabric had satisfactory long-term stability. This could be due to the fact that it is difficult for water to wet the superhydrophobic surface. Furthermore, the stable interaction between Nafion-MWCNTs and cotton is beneficial for improving the stability of the superhydrophobic surface. Such stable building may be due to the following reasons: (1) there are large van der Waals forces between MWCNTs and the cotton fibers [26]. Pasta *et al.* demonstrated that cotton fabric coated with SWCNTs exhibited excellent chemical resistance performance because of strong van der Waals forces existing between SWCNTs and textile fiber [26]. (2) The waterproof surface was formed, ascribed to Nafion bridging MWCNTs and cotton fibers and lowering cotton fabric's surface energy [27]. Shim *et al.* [25] also proved that it was impossible to remove the adsorbed Nafion-CNTs from the fibers by exposure to solvents, heat, or a combination of both [25]. (3) Flexible MWCNTs allow themselves to be conformally adhered to the surface of textile fibers, which maximized the surface contact area between MWCNTs and cotton fibers [25,26].

EMI Shielding Effectiveness and Durability

Electrical conductivity is one of the important criteria for EMI shielding because EMI shielding materials need mobile charge carriers or holes. Surface resistances of samples with different numbers of deposition were shown in Figure 7. Surface resistance decreased from 6780 to 378 Ω/□ when the number of deposition increased from one to six, which means that electrical conductivity increased with MWCNTs deposition because more MWCNTs can form more and denser conductive network as shown in Figure 3. In comparison with other CNT-coated textiles, the surface resistance of the fabric with six depositions of Nafion-MWCNTs (378 Ω/□) was much lower than that of Nylon/CNT textiles (31 MΩ/□) [30] and that of MWCNTs/BTCA coated cotton fabric (2 kΩ/□) [13]. Therefore, it is expected that the microwave shielding efficiency of the fabric coated with MWCNTs could be more substantially improved.

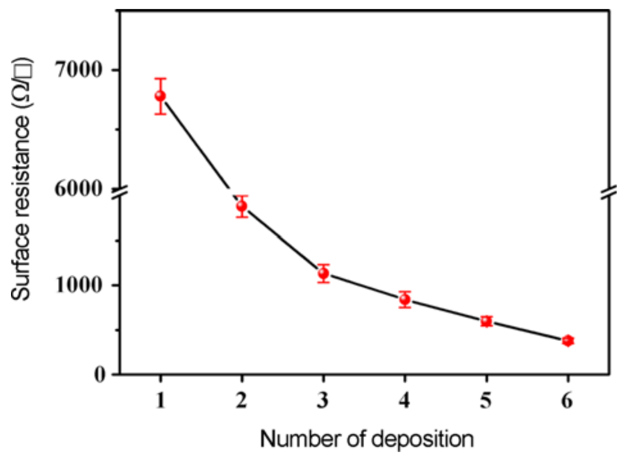

Figure 7. Surface resistance of cotton fabric with different number of deposition.

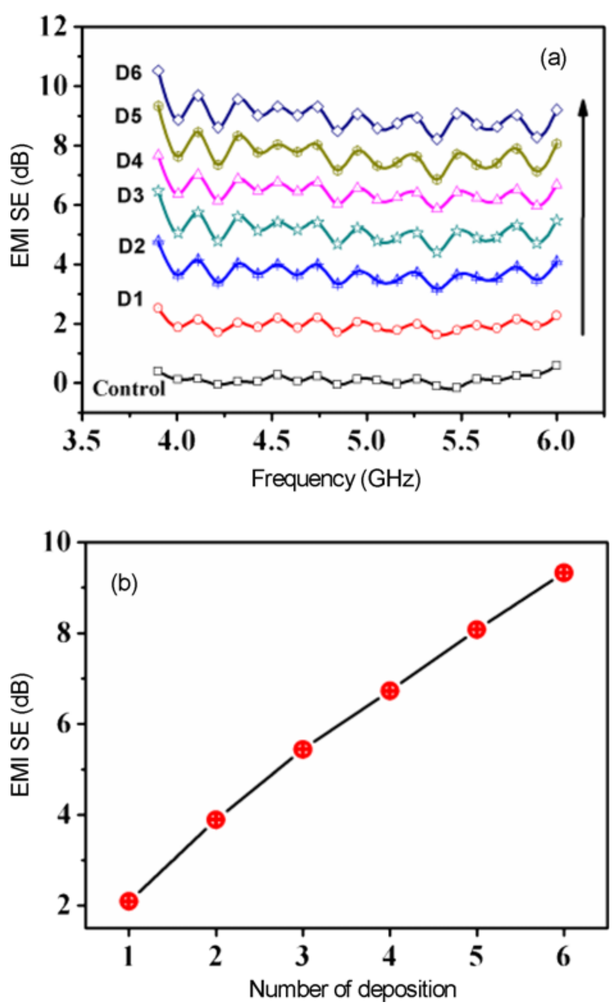

Figure 8. EMI SE of the fabrics with different number of deposition (a) in the frequency range of 3.9-6.0 GHz and (b) at 4.5 GHz.

The capability of the coated fabric to shield electromagnetic waves was measured in terms of its signal attenuation, defined as logarithmic function of the ratio of the transmitted power (P_t) to the incident power (P_i) of the electromagnetic wave and is represented by the equation (4) [31].

$$SE = -10\log(P_t / P_i) \text{ (decibel, dB)} \quad (4)$$

Figure 8(a) shows the EMI SE of cotton fabrics with various times of deposition in the frequency range of 3.9-6.0 GHz. It was revealed that the EMI SE value fluctuated with the frequency, which may be attributed to the irregular nature of conductive network formed in cotton fabric [32]. As expected, the EMI SE of fabrics rose with the number of deposition in the whole frequency range. We further analyzed the EMI SE of fabrics with different number of MWCNTs deposition at 4.5 GHz, as shown in Figure 8(b). The EMI SE increased from 2.1 dB to 9.3 dB as the number of deposition increased, due to more and more MWCNTs coated on the cotton fabric and more mobile charges available to interact with the electromagnetic fields in the radiation.

In order to understand the durability of EMI shielding for the cotton fabric coated with Nafion-MWCNTs, we immersed D5 and D6 samples into water for 96 h at room temperature and compared the difference of EMI SE before and after water immersion. As shown in Figure 9, the EMI SE changed little after such a long time immersion in water. In addition, we tested the EMI SE of D5 and D6 after washing with the AATCC standard (61-2013 test No. 2A), in which one wash is equal to five times of home machine laundering. The results indicated that the EMI SE of D5 and D6 declined insignificantly, and remained at 7.27 dB and 8.51 dB corresponding to the retention rates of 94.3 % and 95.0 %, respectively. This proved that the cotton fabrics coated with Nafion-MWCNTs possessed good shielding durability, owing to their superhydrophobicity and moderate chemical stability.

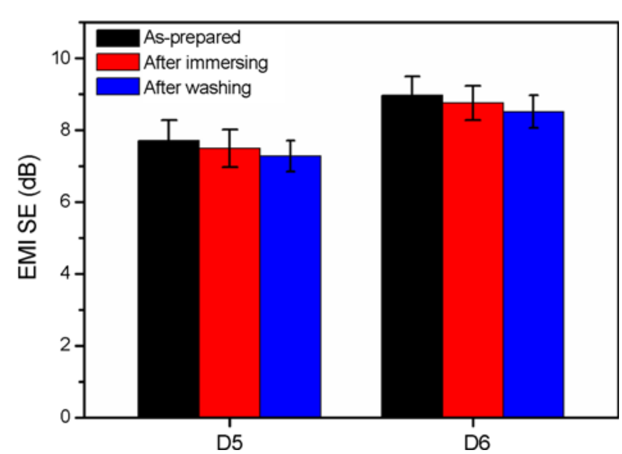

Figure 9. Average EMI SE in C-band of fabrics with five (D5) and six (D6) Nafion-MWCNTs deposition cycles before washing and after immersing in water for 96 h and after washing with AATCC standard.

Conclusion

In summary, durable EMI shielding cotton fabrics have been prepared by means of a superhydrophobic Nafion-MWCNTs coating. The fabric displays a superhydrophobic water contact angle of 154.6^{o} and an favorable shielding effectiveness of 9.0 dB in the frequency range of 3.9-6.0 GHz after six times of deposition with Nafion-MWCNTs. More importantly, the fabric remains almost the same shielding effectiveness after immersing in water for 96 h or washing with AATCC standard because of its superhydrophobicity and chemical stability, showing the excellent durability of Nafion-MWCNTs coating in EMI shielding. This work demonstrates the significance of superhydrophobicity for the practical applications in EMI shielding.

Acknowledgments

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