

# Preparation of hydrophobic and conductive cotton fabrics using multi-wall carbon nanotubes by the sol–gel method

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**Abstract** In this paper, a simple method is presented for making a conductive and hydrophobic cotton fabric using a multi-walled carbon nanotube (MWCNT). The method is known as ‘the sol–gel method’. The conditions for the sol–gel process such as the solution pH, silane agent concentration, polymerization time, amount of MWCNT, and its fixation procedure were optimized. The hydrophobic properties of the modified fabric were characterized in terms of water contact angle (WCA) and water droplets penetration time. The surface morphology of the treated samples was evaluated using a scanning electron microscope. The surface resistivity of the prepared fabric was measured according to the 76-1995 AATCC test method. The abrasion and washing fastness of the fabric were also studied. In optimum conditions, the prepared cotton fabric proved to have such features as  $WCA = 146^\circ$  and surface resistance =  $40 \text{ k}\Omega \text{ cm}^{-2}$ .

**Keywords** Sol–gel process · Surface resistance · MWCNT · Hydrophobic cotton

## 1 Introduction

The sol–gel technique is a relatively novel process to confer special properties on textiles and is associated with minimized environmental impacts [1]. The treatment of fabric surfaces by means of sol–gel deposition from a solution has been described as an encouraging approach. This is because it can lead to the formation of organic–inorganic hybrid coatings at near room temperature. Sol–gel processes are based on two steps involving hydrolysis and condensation reactions starting from (semi)metal alkoxides, such as tetraethoxysilane, tetramethoxysilane, and titanium tetraisopropoxide [2]. The sol–gel approach has been proposed for introducing new functional properties, such as antimicrobial function [3] UV radiation protection [4], dye fastness [5], anti-wrinkle finishing [6], superhydrophobicity [7], and biomolecule immobilization [8]. Besides, particular interest is taken in the self-organization of organosilane molecules, which creates ordered hybrid materials with hydrophobic properties. Transparent and durable superhydrophobic silica coating films on cotton at low temperatures have been reported by Pipatchanchai and Srikulkit [9]. During the past years, many studies have been carried out to fabricate multifunctional textiles with several features [10].

Berendjchi et al. [11] fabricated an antibacterial and superhydrophobic cotton fabric by introducing Cu nanoparticles into silica sols. They found that, due to their chemical activities, such nanoparticles can change the morphology and arrangement of silica nanostructures and, thus, improve the antibacterial activity of cotton fabrics. Paul et al. [12] developed UV-protecting cotton fabrics using ZnO and TiO<sub>2</sub> nanoparticles by the sol–gel technique. Tuttolomondo et al. [13] used the sol–gel method for immobilization of an enzyme on surface. This sol–gel

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immobilized *Pseudomonas* sp. was able to enzymatically reduce azo groups and could be used for decolorization of water contain azo dyes. It was observed that immobilized bacteria produced more than seven times higher amounts of extracellular enzymes involved in the biodegradation of azo dyes. Ardanuy et al. [14] presented an industrially viable procedure for the fabrication of durable insecticide textiles with the sol–gel technique. Permethrin was incorporated on cotton fabrics as a silicon oxide coating applied by conventional padding followed by curing. Messaoud et al. [15] improved the antibacterial properties of a cotton textile via the sol–gel technique using quaternary ammonium-based composite particles. Mahltig et al. [16] prepared mg/TiO<sub>2</sub> composites through the sol–gel procedure and coated them on textiles. They claimed that the prepared textile coatings could exhibit a high capability for dye decomposition under the chosen arrangement of investigations.

Recently, textiles combined with nanoscale materials have attracted considerable attention for their potential application in wearable displays, biomonitoring, and power storage [16–18]. Carbon nanotube (CNT) is one of the most versatile nanomaterials. The versatility is due to its superior properties and responsiveness to a wide variety of environmental stimuli including changes in the temperature, pH, and bio/chemical or mechanical stress [19, 20]. CNT-coated cotton fabrics exhibit super hydrophobic properties, enhanced conductivity, and improved physical properties [21, 22]. However, these CNT-coated textiles are expensive due to the high cost of CNTs. At the same time, CNT coatings make textiles dark and stiff after treatment.

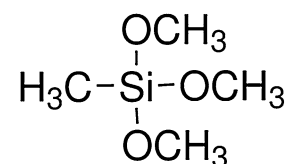
The objective of this research is to develop a simple method for the fabrication of hydrophobic conductive surfaces on textiles using a sol–gel technique. In this paper, we present a simple procedure for coating cotton goods using multi-walled carbon nanotubes (MWCNT) and methyltrimethoxy silane by the sol–gel method in order to achieve several features such as high conductivity and hydrophobic properties simultaneously.

## 2 Materials and methods

### 2.1 Materials

Methyltrimethoxysilane (MTMS) (Scheme 1) was supplied from Merck Company (Germany). The other chemical reagents such as HCL, methanol (with a purity grade of 98 %), sodium carbonate, nonionic detergent sodium hydroxide, sodium silicate, and hydrogen peroxide were purchased from Merck (Germany). MWCNT were purchased from nanolab Co. (America). The purity of the

**Scheme 1** The chemical structure of methyltrimethoxysilane



MWCNT materials was >95 % as received, and its outside and inside diameters were 10–20 nm and 5–10 nm respectively. The length of these MWCNTs was 10–30 μm. The cotton fabric was kindly supplied by Taban Weaving Co.

### 2.2 Preparation of cotton fabrics

Greige cotton (i.e. untreated or raw cotton) contains various non-cellulosic impurities present in the cuticle and primary cell wall of the fiber. Non-cellulosic materials include pectin (0.4–1.2 %), waxes (0.4–1.2 %), proteins (1.0–1.9 %), ashes (0.7–1.6 %), and other miscellaneous compounds. It is now generally accepted that pectin and wax contents and the distribution of the latter are responsible for the non-wetting behavior of greige cotton by water. Scouring can be done to remove pectin and waxes from the surface of a cotton fiber, which endows it with improved bleachability and dyeability. The cotton samples (4 g) were scoured in a bath including 2 g l<sup>-1</sup> NaOH, 5 g l<sup>-1</sup> Na<sub>2</sub>CO<sub>3</sub>, and a 3 g l<sup>-1</sup> nonionic detergent with a liquid-to-fabric ratio of 50:1 at 98–100 °C for 60 min, followed by rinsing with DI water and dried at room temperature. Then, the scoured samples were bleached in a solution of 2 g l<sup>-1</sup> NaOH, 10 ml H<sub>2</sub>O<sub>2</sub> and 1 ml sodium silicate with a liquid-to-fabric ratio of 30:1 at 98–100 °C for 60 min, followed by neutralization in a 0.5 % solution of acetic acid for 5 min. Finally, the samples were rinsed completely with DI water and dried in a preheated oven at 120 °C for an hour.

### 2.3 Methods

For preparation of the sol, 18 ml of methanol was added to 2 ml of DI water. Then, to this mixture was added 4.0 % v/v MTMS. Before adding silane monomer, the solution pH was adjusted by adding several drops of diluted hydrochloric acid at 4.0. The solution was placed on a magnetic mixer to be stirred at a moderate speed. The cut samples (3 × 3 cm) were added to the primary solution. To optimize the sol–gel parameters, an investigation was made of the sol pH from 2 to 9, the time range from 1 to 120 min, and the different concentrations of MTMS (0.25, 0.5, 1.00, 2.00, 2.5, 3.00 and 4.00 % v/v). In the next step, in order to create appropriate properties, MWCNT were used during the process. Also, different concentration of

MWCNT (0.01, 0.015, 0.02, 0.025, 0.03 and 0.04 g ml<sup>-1</sup>) were investigated, so were different fixation methods of carbon nanotube to be applied on the fabric surface.

#### 2.4 Characterization of surface morphologies

Microscopic investigations were carried out on the specimens using a JXA-840 scanning electron microscope (SEM) operated at a pressure of  $-1.0 \times 10^{-5}$  Torr at room temperature. The operation voltage was adjusted at 10 kV. The specimens were mounted on conductive carbon adhesive tabs and examined after gold sputter (SC 7620 EMITECH) coating.

#### 2.5 Hydrophobicity of samples

The hydrophobicity of the samples was determined in two ways; that is, by considering water droplet penetration time (WDPT) and water contact angle (WCA). The WDPT method is a commonly used way of determining the degree of surface water repellency. The method involves depositing a water droplet on the surface and then recording the time it take to penetrate [23].

The contact angle (CA) of water droplets were measured using a goniometer apparatus coupled to a CCD camera (Kruss G10, KRUSS GmbH, Hamburg, Germany). The measurements were carried out in an ambient atmosphere at room temperature. The volume of the applied droplets of distilled water was 5  $\mu$ l. In view of the fact that, in some samples, water droplets could not be detached from the needle, each droplet was dropped to the sample surface from a distance of 0.2 cm by vibrating the needle. CA ( $h$ ) was determined according to the following equation:

$$\tan \theta/2 = 2h/d \quad (1)$$

where  $h$  is the droplet height, and  $d$  is the base diameter. The droplet height and the base diameter were measured using the optical images of the water droplet. Four measurements were made on each fabric surface in different positions to determine the average CA values.

#### 2.6 Surface resistance measurements on conducting textiles

Conductive polymer coatings on textiles cannot be characterized by the volume resistivity due to the thickness of the conductive coating being much smaller than the thickness of the textile substrate. In the case of coated fabrics, rather than resistivity in  $\Omega$ /m, surface resistivity (RS) in  $\Omega$ /sq (ohms per square) is used to express the electrical property. The surface resistivity of the conducting fabrics was measured by AATCC test method 76-1995 using a 34401A multimeter (Agilent Technologies) after conditioning in a standard atmosphere (i.e. 20 °C, 65 %

RH). This method involved two rectangular copper electrodes (20 mm  $\times$  30 mm) being pressed onto the fabric surface with a 10 N pressure. The measurement device was placed on a flat fabric, and a relaxation time of 120 s was allowed prior to each resistance measurement. This was repeated at least ten times for each sample.

#### 2.7 Washing and abrasion fastness

Evaluation of the washing fastness and the abrasion fastness of the prepared samples was performed by ISO-105-X12 1993 (E) test method and ISO-105-C01 1992 method respectively.

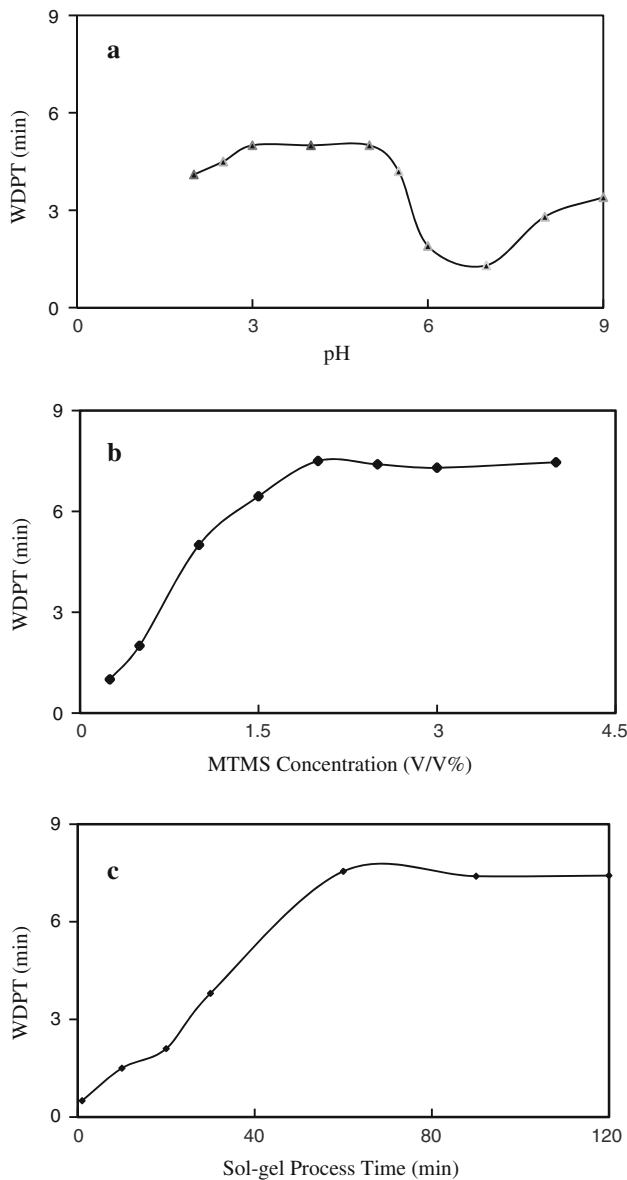
### 3 Results and discussion

#### 3.1 Making hydrophobic cotton fabrics by a sol–gel method

##### 3.1.1 The effect of sol pH on water drop penetration time

As the first step of the experiment, the initial solution pH was investigated in order to optimize the sol–gel conditions. To accomplish this step, some solutions were prepared with the concentration of 1.00 % v/v of MTMS in methanol at different pHs in a range from 2 to 9. In order to examine the formation of a polymer layer on the sample surface, the WDPT was used to refer to the complete penetration of water droplets into the samples. For this, 5  $\mu$ l drops of distilled water were placed on the surface of the treated sample at different pHs, and the complete penetration time of water into it was measured. The results are given in Fig. 1a.

The results presented in Fig. 1a show that the WDPT first increased by the increase of the pH and then reached its maximum in a mildly acidic environment (pH = 3–4.5). In this environment, the hydrolysis was preceded by the protonation of the OR group and converted to ROH (a weak alkaline), which is an excellent leaving group. So, the polymerization rate was enhanced. Then, the fixation rate of the silane polymer was decreased on the sample surface by the increase of pH toward a neutral environment. To perform the polymerization process, OR<sup>-</sup> (a strong alkaline) was inappropriate as a leaving group in a neutral environment. At the same time, the nucleophilic group was stronger in an alkaline environment, and the conditions were improved for the polymerization process. Thus, WDPT was slightly increased. But, as observed, the rate of acid hydrolysis of the silane monomer was significantly greater than the base hydrolysis, which is consistent with the findings of the shi et al. [24] and Arkles et al. [25].



**Fig. 1** The effect of different parameters the sol–gel process on penetration time, **a** solution pH versus penetration time (MTMS concentration = 1.00 % v/v, polymerization time = 90 min), **b** MTMS concentration versus penetration time (pH = 4.0, polymerization time = 90 min) and **c** sol–gel process time versus penetration time (pH = 4.0, silane concentration = 2.00 % v/v)

### 3.1.2 The effect of MTMS concentration on the water droplet penetration time

In the second step of optimizing the sol–gel conditions, the concentration of the silane monomer was evaluated as one of the most important factors in the sol–gel process. To this purpose, some solutions were prepared with different concentrations of MTMS in methanol (as a solvent) in a range from 0.25 to 4.00 % v/v. The samples were treated with each solution for 90 min, then removed from the solution and rinsed in a cold ethanol solution, and finally dried in a laboratory oven at

100 °C for 60 min. At this stage, for the evaluation of the hydrophobic properties of the samples, the WDPT (droplets of 5  $\mu$ L) was measured. Figure 1b shows the diagram of the silane polymer concentration versus the WDPT.

Based on the results shown in Fig. 1b, firstly, the amount of the polymer film formed on the surface of the cotton fabrics was negligible in spite of the low concentrations of the monomer. So the water droplets penetrated very fast into the fabrics. Secondly, the polymerization process increased by an increase in the concentration of the silane monomer and, thus, the sol–gel process was done at a better speed this time. Once the monomer concentration reached 2.00 % v/v, the WDPT was the longest and, therefore, the water permeability into the treated cotton fabrics was at the lowest. The WDPT remained approximately constant with an increase of the monomer silane concentration. Thus, based on the obtained results, a silane concentration of 2.00 % v/v was used in later stages.

### 3.1.3 The effect of the sol–gel time on the water droplet penetration time

In the third stage of optimizing the conditions of the sol–gel process, polymerization process time was studied. The polymerization process of the monomer was performed in a mildly acidic environment within different time durations from 1 to 120 min in the presence of cotton fabrics. As in the previous sections, the fabric removed from the sol–gel environment was rinsed with a cold ethanol solution and then dried in an oven at 100 °C for an hour. The WDPT into the fabric containing the silane polymer was measured. The results are given in Fig. 1c.

As shown in Fig. 1c, WDPT the treated fabrics increased by an increase in the time of the sol–gel process. This trend continued for 60 min. It seems that the formation of the polymer film on the fabrics stopped after the polymerization of the silane monomers was completed and they were, thus, actively posited on the fabrics.

## 3.2 Enhancement of the hydrophobicity and conductivity of the cotton fabrics using MWCNT

The superhydrophobicity feature of the lotus leaf surface has been of interest since past decades. To achieve such a surface, surface chemistry must be modified so as to make the surface structure rougher. Using carbon nanotubes is an effective method to roughen the surface [24]. In this research, MWCNT was fixed to different methods on sample surfaces, and then the rates of hydrophobicity and conductivity were measured. In the next section, the amount of required MWCNT is investigated to achieve the above two goals.

### 3.2.1 The effect of fixation of MWCNT on surface resistance

The following ways were evaluated to determine the best way of fixating MWCNT and its effect on the rate of the conductivity and hydrophobicity of cotton fabrics:

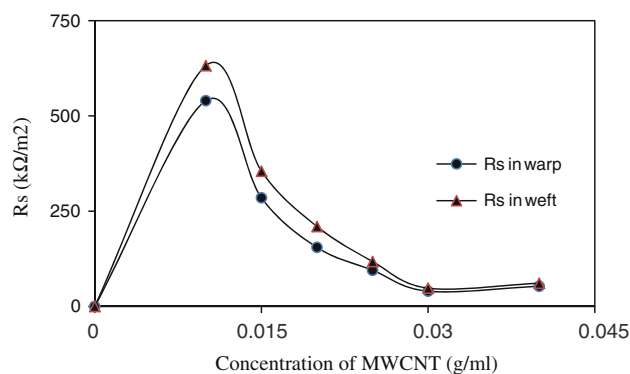
1. First, the cotton stuffs were treated by the sol–gel method in optimum conditions. Next, a mixture of MWCNT prepared with a concentration of  $0.02 \text{ g ml}^{-1}$  in DI water was placed in an ultrasound bath. The ultrasound bath was used to prevent the nanoparticles from aggregation and penetration into the fibers of the fabric. In this way, MWCNT was fixed by padding on the finished samples using the sol–gel method.
2. In this procedure, first a mixture of MWCNT prepared with a concentration of  $0.02 \text{ g ml}^{-1}$  in DI water was placed in an ultrasound bath. Then, it was fixed by padding on the samples. Next, the samples padded in the MWCNT emulsion were placed in a sol–gel bath, and the formation of silane polymers was performed in optimum conditions for 60 min.
3. In this procedure, the sol–gel process and the fixation of MWCNT on the cotton samples were carried out under ultrasound simultaneously.
4. In this process, all the steps were performed as in the third stage, and just magnetic stirring was used instead of an ultrasound bath. In fact, this procedure was a study of the effect of ultrasound waves on the fixation process of MWCNT and the formation of polymer films on the cotton stuffs.

Finally, the WDPT of the prepared samples and their surface resistivity were measured by the above procedures.

The results indicate that the rate of hydrophobicity of the prepared samples using procedure (2) is higher than that in the other procedures. It means that padding the blank samples with an MWCNT mixture in an ultrasound bath and then doing the sol–gel process provides better results. Also, in this way, the rate of the surface resistivity of the samples is significantly reduced. Therefore, approach (2) is mentioned as the best way to make hydrophobic fabrics and to increase textile conductivity. Besides, by comparing the third and the fourth procedures, one can realize the highly suitable influence of ultrasound waves during the finishing operation. In fact, the presence of ultrasound waves prevents the accumulation of MWCNT and increases its penetration into the samples.

### 3.2.2 The effect of the amount of MWCNT on surface resistance

In order to study the effect of MWCNT amount on increasing the conductivity and hydrophobicity of the



**Fig. 2** The diagram of effect the different amount of the MWCNT on surface resistivity (pH = 4, polymerization time = 60 min, MTMS concentration = 2.00 % v/v, procedure No. 2)

**Table 1** The results of measurement the surface resistance and water contact angle of prepared samples in present work and other works (pH = 4.0, silane concentration = 2 % v/v, polymerization time = 90 min and  $0.03 \text{ g ml}^{-1}$  MWCNT)

Samples	Specific resistance ( $\text{k}\Omega \text{ cm}^{-2}$ )	WCA ( $^{\circ}$ )	Ref.
Treated sample with conductive polymer of PEDOT	100–110	–	[10]
Prepared cotton sample with CNT and PVA	2.87	–	[27]
Treated sample with 0.015 alkyl trialkoxy silane (C16)	6,000	116	[26]
Treated sample with FOTS	–	146.8	[17]
Treated sample with %5 HDTMS and %3 fumed silica	–	121.6	[9]
SNP (4.0 wt%) and WR (10 wt%) agent treated PET fabric	–	158	[29]
Hydrophobic and conductive sample by procedure 2	40.1	146	This work

cotton materials, the mixtures containing different amounts of MWCNT were prepared in distilled water. Accordingly, mixtures with different concentrations of MWCNT, including 0.01, 0.015, 0.02, 0.025, 0.03 and  $0.04 \text{ g ml}^{-1}$ , were prepared. The blank samples were processed in these mixtures according to procedure 2. Then, WDPT and the surface resistivity were measured according to the method mentioned in the previous sections. The results are shown in Fig. 2.

Obviously, the surface resistance of the raw sample is too high. Based on the results of Fig. 2, the best condition can be achieved when  $0.03 \text{ g ml}^{-1}$  of MWCNT is used. In this condition, hydrophobicity and conductivity are at the highest level. Therefore, based on the results, concentration of  $0.03 \text{ g ml}^{-1}$  is considered as the best amount of

MWCNT. The results also show that the rate of MWCNT fixation at the sample surface rises first by an increase in the amount of MWCNT, so does the rate of hydrophobicity and conductivity of the samples. Following that, by an increase in the concentration of MWCNT from  $0.03 \text{ g ml}^{-1}$  onwards, hydrophobicity decreases while the surface resistivity increases. A possible interpretation of these results is that, with an increase in the concentration of MWCNT to more than  $0.03 \text{ g ml}^{-1}$ , there will be too much of it accumulated on the surface of the cotton fabrics, and this excessive accumulation of MWCNT reduces the efficiency of the prepared goods.

To have a better examination and comparison, in addition to the above test results, the results of some other

works [10, 26, 28] which have been done in this field are given in Table 1. By comparing the results, one can conclude that the proposed method has a relatively better performance than those referred to in other studies.

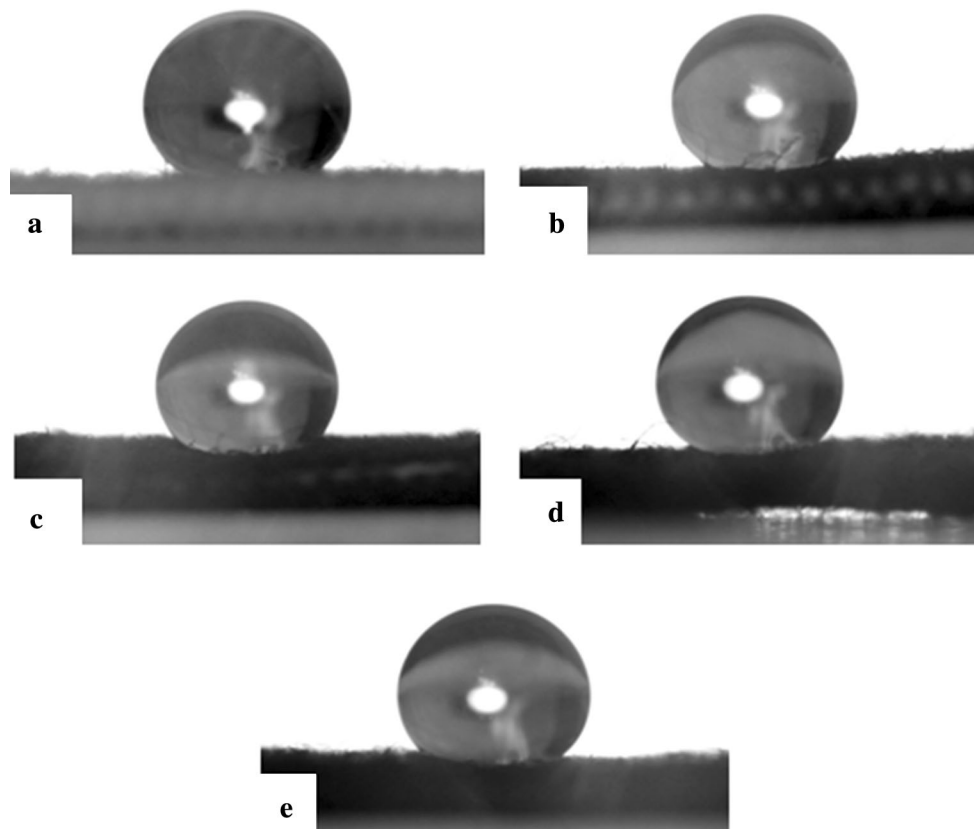
### 3.3 Evaluation of water contact angle

A method was used to measure the water droplet contact angle and to examine the hydrophobic property of the cotton materials. The result of this study is given in Table 2. Also, Table 1 was presents the measurement results of the WCA obtained in other works [9, 17, 26, 29]. Figure 3 shows the images of water droplets on the treated samples in different conditions.

Based on the results in this section, as it was seen previously, procedure 2 is the best method for making hydrophobic–conductive cotton fabrics and obtaining a maximum contact angle. Also, a comparison of the tables in this study with those of other research works reveals that the proposed method is desirably capable of making hydrophobic cotton goods. It is also important to note that the method proposed in the present study is very simple and less time-taking.

**Table 2** The results of measurement the WCA on treated fabrics

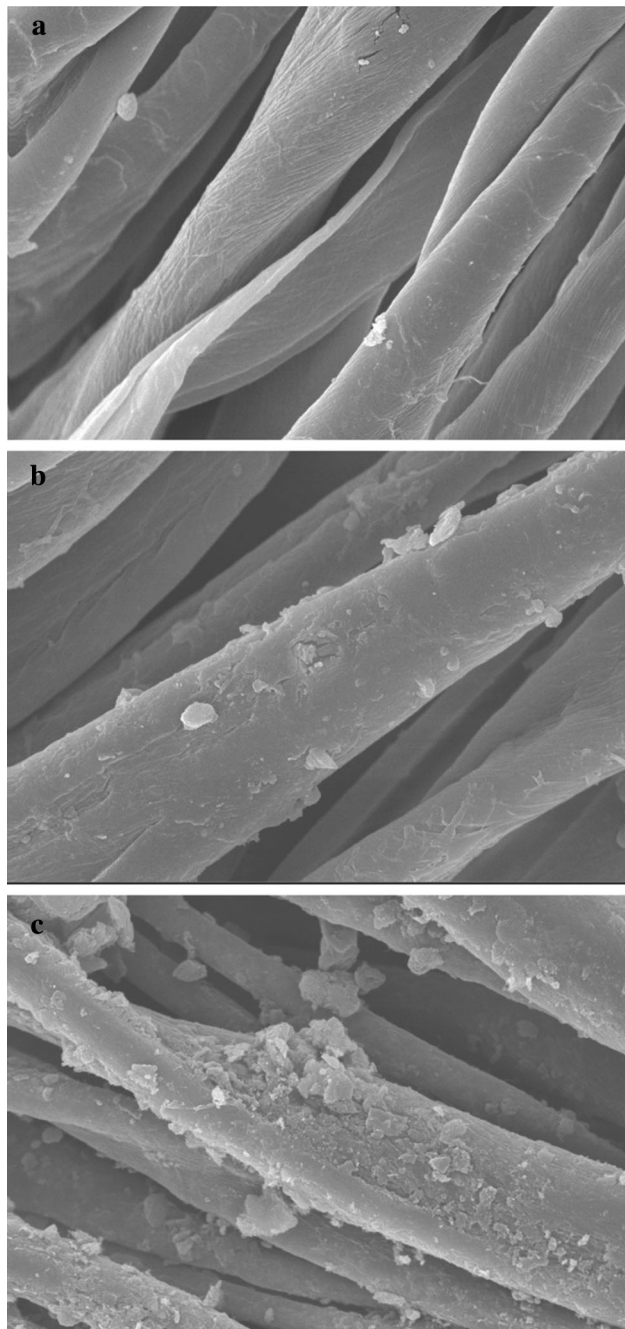
WCA (°)	Sample
138	Prepared sample by procedure 1
146	Prepared sample by procedure 2
135	Prepared sample by procedure 3
120	Padded sample in $0.03 \text{ g ml}^{-1}$ MWCNT



**Fig. 3** The image of a droplet on treated fabric with **a** 2.00 % v/v MTMS compound, **b** padded in  $0.03 \text{ g ml}^{-1}$  MWCNTs solution, **c** by SP 1, **d** by SP 2 and **e** by SP 3. (The picture was capture immediately after placing droplet on sample surface)

### 3.4 The surface characteristics of the treated fabrics

Scanning electron microscope technique was used to investigate the changes in the surface morphologies of the coated fabrics. The images related to the samples are shown in Fig. 4. The figure shows the surface of a raw fabric whose fiber surface is free of polymer particles. In part (b) of this figure, SEM images are shown of the coated



**Fig. 4** SEM images of **a** washed and bleached sample, **b** coated sample with 2.00 % v/v MTMS, **c** prepared sample by procedure 2 (magnification 3,500 $\times$ )

sample with 2.00 % v/v silane monomers. In Fig. 4c, the surface of the coated sample is shown with silane polymers and MWCNT under ultrasound simultaneously (i.e. an optimum condition). In this picture, the MWCNT particles and the polymer layer can be clearly seen.

### 3.5 The washing and abrasion fastness of the hydrophobic–conductive fabrics

As it was indicated in the previous sections, polymers can form a layer on fabrics. Since this layer may be destroyed due to abrasion or washing, its stability and durability is a matter of importance. To determine the durability of the layer, washing and abrasion fastness was measured using the standard test of color fastness devised for wear-and-wash textiles. Obviously, this test is not originally designed to measure the type of fastness as examined in the present study; therefore, it is necessary that new methods be developed for this purpose. This test can, nevertheless, be used for assessing the relative stability.

In accordance with standard test methods, the conductivity of the samples was measured after wear-and-wash operations, and it was compared with an unwashed sample. The results are shown in Tables 3 and 4.

**Table 3** The results of washing fastness of the hydrophobic–conductive fabrics in surface resistivity

Washing cycles	Rs after washing cycles ( $\text{k}\Omega \text{cm}^{-2}$ )				
	–	5	10	15	20
Prepared sample by procedure 1	78.6	600	900	1,095	1,600
Prepared sample by procedure 2	40	65.7	82	102	168
Prepared sample by procedure 3	366	860	1,028	1,467	1,900
Padded sample in 0.03 $\text{g ml}^{-1}$ MWCNT mix	43	254	590	708	1,023

**Table 4** The results of abrasion fastness of the hydrophobic–conductive fabrics in surface resistivity

Abrasion cycles	Rs after abrasion cycles ( $\text{k}\Omega \text{cm}^{-2}$ )					
	–	10	50	100	200	500
Prepared sample by procedure 1	78.6	88.9	90.6	120.2	160.6	306.3
Prepared sample by procedure 2	40	54	74	116	152	245
Prepared sample by procedure 3	366	410	523	674	750	874
Padded sample in 0.03 $\text{g ml}^{-1}$ MWCNT mix	43	180	310	754	968	1450

The obtained results suggest that the washing cycle conductivity of the samples is reduced. However, the samples somehow regain their conductive properties after 20 times of washing. The abrasion fastness of the coated samples was good after wear, but their conductivity reduced slightly after wear. The good abrasion fastness could be due to the depth of coating and the penetration of nanoparticles into the structure of the fabric and fibers. In fact, it can be stated that a polymer silane film serves as a protective layer on surfaces coated with MWCNT.

#### 4 Conclusion

In this paper, a hydrophobic–conductive cotton fabric was made using MTMS and MWCNT by a sol–gel method. To do this, at first, sol–gel process conditions, namely the solution pH, MTMS concentration, polymerization time, and, then, the amount of MWCNT and its fixation procedure were optimized. The properties of the prepared sample were evaluated from a hydrophobic point of view by measuring the WCA, the water droplets penetration time duration, and its conductivity. Next to that, the surface morphologies and the washing and abrasion fastness of the prepared samples were studied. The optimized conditions for preparing the conductive–hydrophobic cotton fabric were as follows: pH = 4, concentration of MTMS 2.00 % v/v, sol gel time of 60.0 min, and concentration of 0.03 g ml<sup>-1</sup> of MWCNT under ultrasound waves. The WCA and the resistivity of the padded sample in a 0.03 g ml<sup>-1</sup> solution of MWCNT treated with a 2.00 % v/v MTMS were found equal to 146° and 40 kΩ cm<sup>-2</sup> respectively. In SEM images, as it could be clearly seen, the surface of the cotton fibers was covered with a silane layer and MWCNT particles.

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