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ORIGINAL PAPER

Fabrication of durable and cost effective superhydrophobic cotton textiles via simple one step process

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Abstract Superhydrophobic surfaces are highly hydrophobic, i.e., extremely difficult to wet. Such surfaces have water contact angle (WCA) exceeds 150° and water sliding angle (WSA) < 10°. This is known as "the superhydrophobic effect" or "the Lotus effect". Superhydrophobic cotton fabric was prepared via a novel one step solution immersion process using silica nanoparticles and hexadecyltrimethoxysilane. The method is simple, cost-effective and can be applied on the large industrial scale. Improvement of treatment durability was attained by the incorporation of silane coupling agents. A new substance, ethylenediaminetetraacetic acid (EDTA), was used for the first time in this study to improve the durability of the prepared superhydrophobic fabric and its performance was compared with that of silane coupling gents. The surface morphology and hydrophobic properties of the prepared superhydrophobic cotton fabrics were characterized by scanning electron microscopy, energy dispersive X-ray spectroscopy and Fourier transform infrared spectroscopy. The wettability of the developed superhydrophobic cotton fabrics was evaluated by WCA and WSA measurements. The modified cotton fabrics exhibited superhydrophobicity with WCA of 159.8° and WSA of 4.0°. Furthermore, the durability efficiency of samples was quantitatively evaluated using standard washing test. Results showed that both silane coupling agents and EDTA could greatly enhanced washing durability. EDTA provided higher stability than silane coupling agents with repetitive washing cycles which considers very promising alternative to improve the durability of the superhydrophobic cotton textiles.

Keywords Cotton · HDTMS · Silica nanoparticles · Superhydrophobicity · Durability · EDTA

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Introduction

Surfaces that exhibit water contact angles >150° and contact angle hysteresis <10° are inspired by natural surfaces such as the lotus leaf on which water drops remain almost spherical and easily roll off removing dirt particles in their path, these surfaces are usually called superhydrophobic surfaces (Li et al. 2007; Richard et al. 2013). The surface of lotus leaf was first examined by Barthlott in 1970 using scanning electron

microscopy and it was found that the surface has small micro-protrusions covered with nano-hairs which are covered with low surface free energy wax substances (Balani et al. 2009). Superhydrophobic surfaces have attracted significant attention within the scientific community over the last two decades because of their unique water-repellent, self-cleaning properties and their potential for practical applications (Xu et al. 2010; Zhang et al. 2008). The interest in self-cleaning property has been driven by the need to fabricate surfaces such as satellite dishes, solar energy panel, automobile windshields and high performance textiles for different domestic and industrial applications such as curtains, bed and table sheets and upholstery fabrics (Song and Rojas 2013; Zimmermann et al. 2008). Superhydrophobic surface on cotton fabrics had been fabricated by combining the surface roughness and lowering the surface free energy. Surface roughness on cotton fabric is created by the incorporation of various nanoparticles such as silica nanoparticles (SNP), zinc oxide and titanium dioxide. Surface energy of cotton substrate could be modified by compounds with low surface free energy such as fluorocarbons or silicones (Xue et al. 2010). For examples, superhydrophobic cotton fabrics had been prepared by the combination of the (SNP) and a cost-effective water-repellent agent (Bae et al. 2009; Lehocky et al. 2007). Superhydrophobic ZnO nanorod array film on cotton substrate was fabricated via a wet chemical route and subsequent modification with dodecyltrimethoxysilane DTMS (Bi and Cai 2008). In last decade, the preparation of superhydrophobic fabric does not become the major issue but the adhesion of hydrophobic coating to the fiber surface is crucial for the practicality on textiles. The loss of hydrophobic coating on the surface of cellulose fibers may be caused by mechanical action during washing. However, the swelling of cellulose fibers during washing process decreases the compatibility of the heterogeneous interface between inorganic silica coating and organic cellulose fiber. As a result, the silica coating cracks and the hydrophobic layer on silica coating is broken away from cellulose fabric (Huang et al. 2011).

The conventional strategy employed to improve the hydrophobic coating durability involves crosslinking the coating layer and establishing covalent bonding between the coating and cotton substrate. However, only limited researches have been achieved in this area. For example, cotton fabrics grafted by 1H,1H,2H,2H-nonafluorohexyl-1-acrylate under simultaneous radiation-induced graft polymerization had been synthesized, which showed stable superhydrophobicity (Cai et al. 2012). Superhydrophobic cotton textiles had been prepared by a complex coating of amino- and epoxy-functionalized silica nanoparticles on epoxy-functionalized cotton textiles to generate a dual-size surface roughness, followed by hydrophobization with stearic acid, 1H,1H,2H,2H-perfluorodecyltrichlorosilane (Zhang et al. 2008; Xue et al. 2009). Functionalized silica SiO_2 particles not only generated a firm dual-size rough surface but also facilitated its further hydrophobization. Others investigated the performance of polycarboxylic acids (PA) in improving superhydrophobic durability (Huang et al. 2011). In their works, different PA were used and the results showed that PA with the highest number of carboxylic acid groups showed the best durability result. PA has the ability to esterify with cellulose –OH and co-condense with silica OH groups leading to the formation of interfacial ester bonds (Mao and Yang 2001). Therefore, PA used as heterogeneous crosslinker to improve the adhesion of the inorganic–organic interface (Hernández-Padrón et al. 2004; Huang et al. 2011). But, fabric yellowing at high temperature, their high cost and the need of catalyst to initiate their crosslinking with cotton fabric are considered the main drawbacks of PA which limits their applications.

In the present study, superhydrophobic cotton fabric was prepared via a facile one step process by the incorporation of SNP and HDTMS via solution immersion process. The treatment durability was enhanced by the incorporation of EDTA as a tetracarboxylic acid which is applied in this study for the first time. EDTA has numerous advantages; it has low cost, locally available and non-toxic material which showed a promising alternative for achieving durable hydrophobic fabrics. The obtained cotton fabrics were characterized by SEM, EDX and FTIR analysis. The hydrophobicity was evaluated by WCA and WSA measurements.

Experimental

Materials

Desized, scoured, bleached, and mercerized woven 100 % cotton fabric, weight per unit area 218.5 g/m^2

was supplied by Misr Amryah Company for Spinning and Weaving, Alexandria, Egypt. Hexadecyltrimethoxysilane (HDTMS) was purchased from Zhejiang Feidian Chemical Co. Ltd, China. 3-aminopropyltriethoxysiloxane (APTS, 98 %) and 3-glycidoxypropyltrimethoxysilane (GPTMS, 97 %) were purchased from Alfa, Germany. Silicon dioxide nanopowders (SNP 99.5 %, 15 nm) were purchased from MKNANO, Canada. Ethylenediamintetraacetic acid (EDTA) was purchased from El Gomhoriya Company, Egypt. Absolute ethanol and absolute methanol were purchased from Sigma-Aldrich, Germany. All the reagents in the experiment were used as received without further purification.

Methods

Preparation of functionalized silica nanoparticles

Functionalized silica nanoparticles (FSNP) are (SNP) treated with silane coupling gents. FSNP were prepared by using two different silane coupling agents as discussed elsewhere (Xue et al. 2009). Briefly, SNP were subjected to sonication bath in absolute ethanol for 30 min at room temperature. The whole suspension was divided into two equal volume solutions. One solution was used for amino functionalization (ASNP), and the other for epoxy functionalization (ESNP). Both ASNP and ESNP were separated by centrifugation at 6000 rpm for 30 min followed by washing with methanol several times, dried at 50 °C overnight and stored in a cleaned and well-sealed glass bottles.

Developing of superhydrophobic cotton fabric

Durability was improved by applying either silane coupling agents (FSNP) or EDTA. The latter was applied either in one or two steps procedures. Superhydrophobic cotton fabrics were prepared through one of the following different treatments.

Treatment with silane coupling agents Fabrics were immersed in 3.0 wt% ASNP for 60 min and then in 3.0 wt% ESNP for 60 min. After each step the fabrics were cured at 120 °C for 60 min. Fabrics treated with FSNP were immersed in 3.0 wt% silanol solution of HDTMS for 60 min followed by curing at 120 °C for 60 min.

Treatment with EDTA in two steps process Fabrics were immersed in different concentrations of EDTA solution (2.0 and 5.0 wt%) for 15 min followed by curing at 120 °C for 60 min, this is called pretreatment step. Hydrophobic solution composed of SNP (3.0 wt%) and HDTMS (3.0 wt%) were prepared. Cotton fabrics pretreated with EDTA were immersed in the prepared hydrophobic solution for 60 min followed by curing at 120 °C for 60 min.

Treatment with EDTA in one step process One step process eliminates the complexity of the two steps involved in the conventional methods and reducing the energy consumed in the multiple steps of the curing process. Two different hydrophobic solutions composed of SNP (3.0 wt%), HDTMS (3.0 wt% solution) and EDTA (2.0 and 5.0 wt%) were prepared. Fabrics were immersed in the hydrophobic solutions for 60 min followed by curing at 120 °C for 60 min.

Treatment with EDTA in one step process without SNP New hydrophobic solution composed of HDTMS (3.0 wt% solution) and EDTA (2.0 wt%) was prepared. Fabric was immersed in the hydrophobic solution for 60 min followed by curing at 120 °C for 60 min.

Characterization

WCA and WSA were measured using distilled water droplets at room temperature. All the angles were determined by averaging values measured at five different points on each sample surface. The surface morphology of the cotton fabrics before and after treatment was examined by scanning electron microscope (JEOL-JSM-5300). Samples were coated with a thin layer of gold using a sputtering machine prior to examination with SEM. An elemental analysis of cotton textiles before and after treatment was implemented using an environmental scanning equipped with an energy dispersive spectroscope (FEI-Inspects Company). Infrared spectra were recorded using FTIR-spectrophotometer (Thermo-Nicolet 380). Samples were cut into very small pieces and then grounded and compressed with KBr salt into a thin pellet to form KBr disc. Different spectra were collected over the range of 500–4,000 cm^{-1}. Washing durability test was carried out according to ISO 105-C06: 2010 standard method using a standard washing laundering machine

(Gyro-wash: Model 81520 N). According to the standard test method, one wash provides an accelerated washing treatment corresponding to five home washings. The washing cycle duration was 45 min at a temperature of 40 °C. The durability of the superhydrophobic treatment was assessed by measuring the contact angle of the washed samples after each washing cycle.

Results and discussion

Durable and cost effective superhydrophobic cotton fabric was developed by simple one step treatment using solution immersion process in hydrophobic solution of EDTA and HDTMS.

Cotton hydrophobicity and treatment durability

The wettability of the cotton fabrics was examined by WCA and WSA measurements. The pristine cotton fabric can be completely wetted by water due to the abundant hydroxyl groups in its structure. On contrary to the pristine cotton fabric, the cotton fabrics after different pre-treatments and subsequent HDTMS modification showed superhydrophobicity as shown in Table 1.

As indicated in Fig. 1, cotton fabric pretreated with high concentration of EDTA (5.0 wt%) showed high WCA of 158.8° and low WSA of 1° but did not exhibit any hydrophobic effect after the first washing cycle, while cotton fabric pretreated with lower concentration (2.0 wt%) showed WCA of 157.3°, low WSA of 4° and provided better washing durability with WCA of 131.9° after six cycles. The same results were

Table 1 Water contact/sliding angles of different treatments

Treatment	WCA (°)	WSA (°)	Washing cycles (WCA)[a]
Silane coupling agent (FSNP)	159.5	3	12 (141.5°)
EDTA (5.0 wt%, two steps)	158.8	1	1 (0)
EDTA (2.0 wt%, two steps)	157.3	4	7 (114)
EDTA (5.0 wt%, one step)	158.8	3	2 (0)
EDTA (2.0 wt%, one step)	160.2	2	7 (121)
EDTA (2.0 wt%, one step, without SNP)	159.8	4	12 (145.8°)

[a] WCA at failure

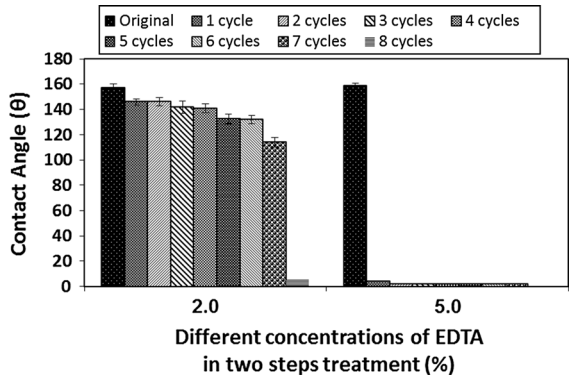

Fig. 1 Washing durability of cotton fabrics pretreated with EDTA in two steps process

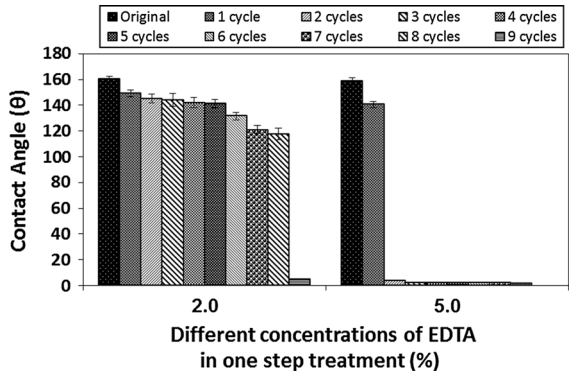

Fig. 2 Washing durability of cotton fabrics treated with EDTA in one step process

obtained for the one step treatment with EDTA in presence of SNP; higher EDTA concentration did not provide washing durability and failed after the second washing cycle while lower EDTA concentration showed a better durability up to seven cycles with WCA of 121°, as shown in Fig. 2. These results indicated that higher concentrations of EDTA provided superhydrophobicity, but it is not effective in improving the treatment durability. The failure of higher EDTA concentrations (5.0 wt%) in two steps treatment is attributed to the complete crosslinking of the carboxylic acid groups in EDTA structure with the free hydroxyl groups of cotton fabric which blocking the active sites on the cotton surface. Therefore, further treatment with the hydrophobic solution (SNP, HDTMS) is only resulted in a deposition of hydrophobic coating on the cotton surface without any chemical bonding between blocked cotton fabric and

hydrophobic solution. High concentration of EDTA with four carboxylic acid groups provides high reactivity to react with cotton fabric and as proved in the work done by Huang et al., it is clear that the more carboxylic acid groups in PA molecule the more interaction sites to bond with cotton fabric. The same result was obtained for higher EDTA concentrations (5.0 wt%) in one step treatment where EDTA was added to the hydrophobic solution of SNP and HDTMS. EDTA readily cross-linked with hydrophobic solution and only small amount of free carboxylic acid groups in EDTA or free hydroxyl groups in the hydrophobic solution that could bond with cotton fabric which resulted in a weak washing durability up to only two cycles. Lower concentration of EDTA (2.0 wt%) either in one or two steps treatment is more effective in improving the washing durability as shown in Figs. 2 and 3. Lower EDTA concentration cross-linked with hydrophobic solution with some carboxylic acid groups remained free to cross link with cotton fabric. The lower concentration did not cause the complete crosslinking as in the higher concentration which resulted in a better washing durability. Ester bridges introduced by the four carboxylic groups strengthened the adhesion between hydrophobic coating and cotton fabric and so the ester-bridges breakdown became difficult with repetitive washing.

Cotton fabric treated with hydrophobic solution in absence of SNP (2.0 % EDTA, HDTMS) showed high WCA of 159.8° and greatly improved washing durability with WCA > 145° after 12 washing cycles, as shown in Fig. 3. EDTA with four carboxylic acid groups readily grafted and cross-linked with large amount of hydrolyzed HDTMS with three hydroxyl groups. Therefore, when applied to cotton fabrics a huge number of ester bridges were bonded with cotton which resulted in a high durable superhydrophobic fabric. Schematic illustration for the preparation of superhydrophobic cotton fabric in one step treatment using EDTA and HDTMS is presented in Fig. 4. Comparing EDTA results with the work done by W. Huang et al., the best washing durability was attained by 1,2,3,4-butanetetracarboxylic acid (BTCA) which provided durability up to only six cycles (30 times) whereas EDTA could provide washing durability up to twelve cycles (60 times) with WCA of 145°. Therefore, EDTA as a crosslinking substance is considered as a promising alternative for improvement of treatment durability.

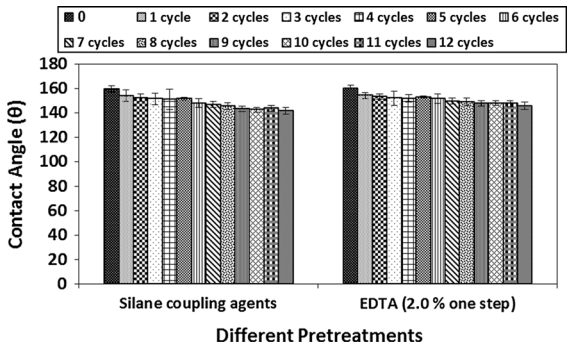

Fig. 3 Washing durability of silane coupling agents versus EDTA

On the other hand, silane coupling agents (FSNP) provided excellent surface roughness to cotton fabric and showed excellent superhydrophobicity with WCA of 159.5°. Treatment of cotton fabric with FSNP followed by hydrophobization with HDTMS exhibited washing durability up to twelve washing cycles with WCA > 140°, as shown in Fig. 3. The amino groups of the first layer of silica coating were utilized to react with the epoxy-functionalized silica particles. Therefore, there must be strong interactions not only among the silica particles, but also between the silica particles and the cotton textiles (Xue et al. 2009). SEM of cotton fabric treated with FSNP showed a dense cluster of silica layers deposited on cotton which resulted in a robust superhydrophobic cotton fabric and greatly improved washing durability result. From the above results, it is clear that treatments either with FSNP or EDTA provided excellent washing durability for cotton fabrics compared to the moderate durability exhibited by other PA with lower or same number of carboxylic groups. Photographs of the common household liquid droplets deposited on the surface of cotton fabrics with different treatments are shown in Fig. 5. All the droplets showed spherical shape with high WCA indicating the excellent superhydrophobicity of the treated fabrics.

Surface morphology

The microstructures of cotton fabrics before and after different treatments were studied by SEM, as shown in Fig. 6. The SEM image of untreated cotton fabric, Fig. 6a, b, shows a clear surface smoothness with grooves easily observed on the surface of the fibers due to the inherent characteristic of the cotton fibers

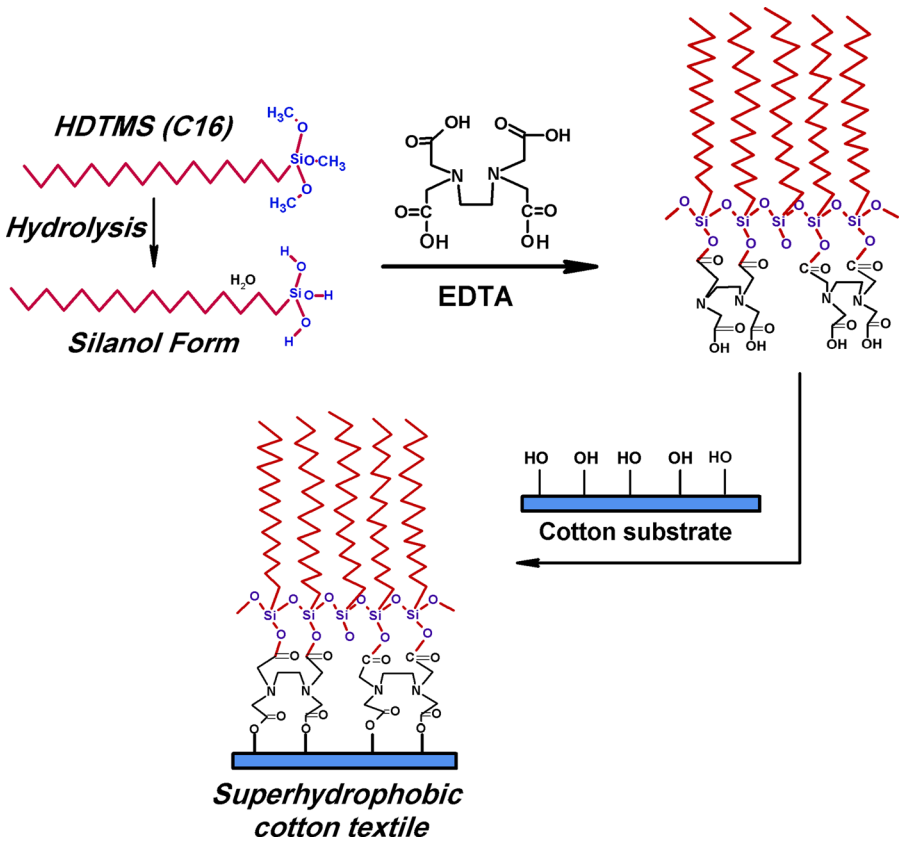

Fig. 4 Schematic illustration of one step process for preparation of superhydrophobic cotton textile

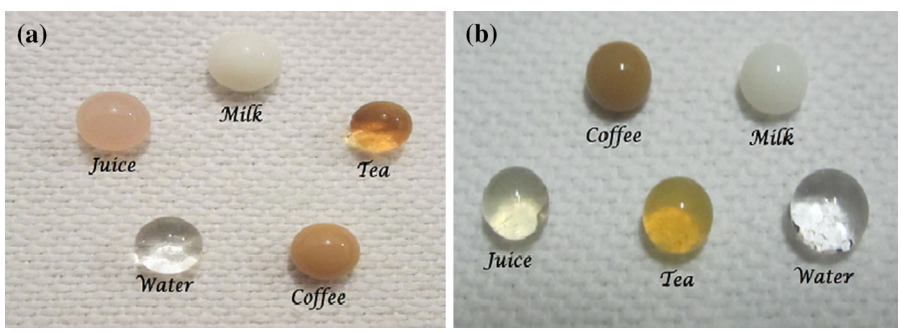

Fig. 5 Droplets of the common household liquids standing on the cotton fabric treated with **a** FSNP and **b** EDTA

(Zhang et al. 2013). EDX spectrum of pristine cotton gave only two strong signals for C and O. EDX of cotton fabric treated with HDTMS showed a new peak for Si which proved the successful incorporation of HDTMS on the cotton surface.

SEM image of cotton fabric treated with silane coupling agents (FSNP) shows silica clusters appeared on the surface of cotton fiber resulting in a rough surface with an increase in the WCA, as shown in Fig. 6c, d. This can be attributed to the complex coating of amino- and epoxy-functionalized silica nanoparticles (Xue et al. 2009), thus generating a dual-size surface structure on the cotton fabric which provided high WCA of 159.5° and low SA of 3.0°. The two layers of self-assembled FSNP improved the adhesion between HDTMS silanol groups and the hydroxyl groups on the cotton surface and exhibited heavier coating density of FSNP, as shown in the

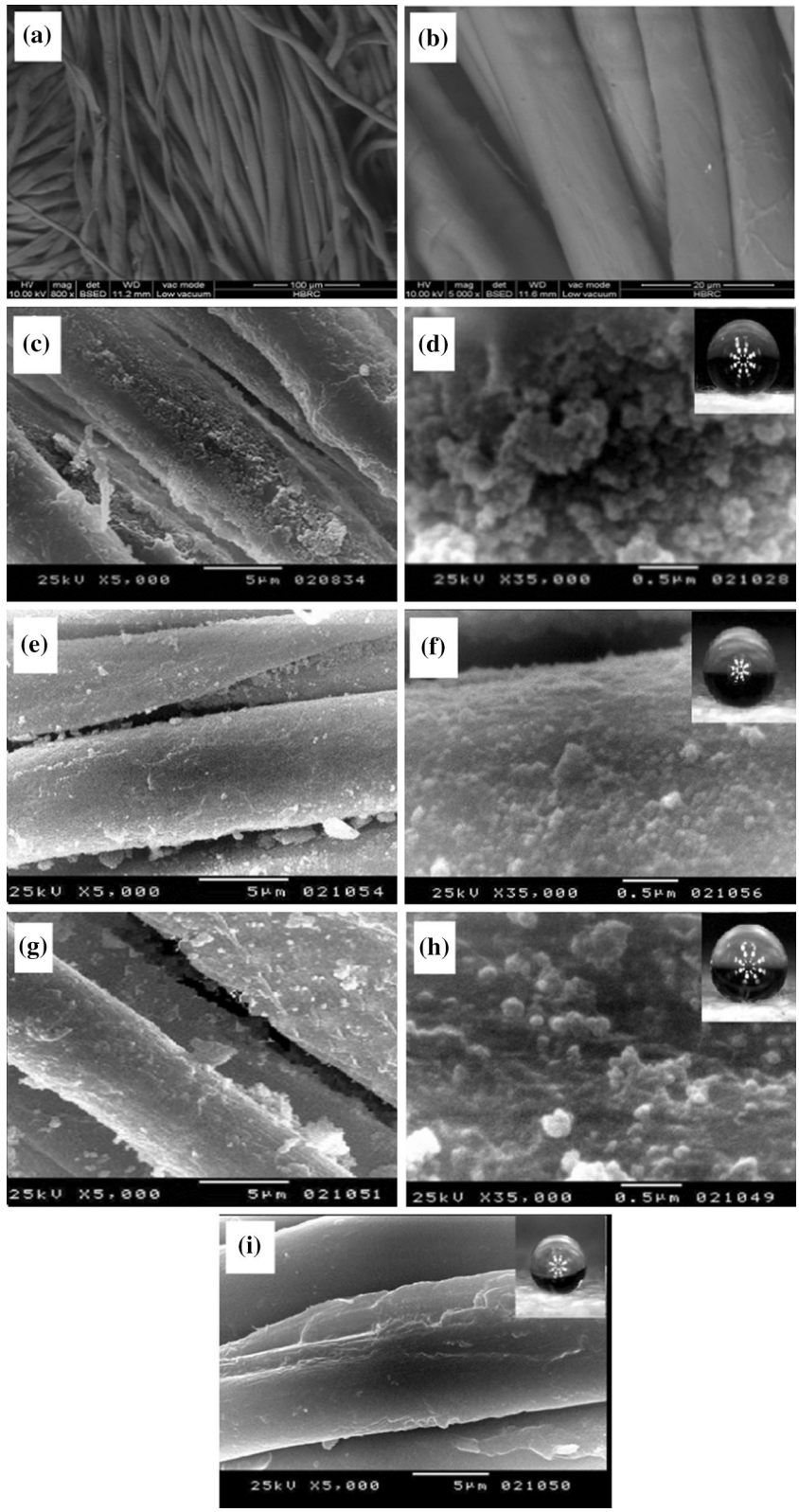

Fig. 6 SEM images of **a** pristine cotton, **c** one step treatment with SNP and HDTMS, **d** two steps treatment with SNP and HDTMS, **e** cotton fabric treated with FSNP and HDTMS, **g** two steps treatment with EDTA and HDTMS in presence of SiO_2, **i** one step treatment with EDTA and HDTMS in presence of SiO_2, **k** one step treatment with EDTA and HDTMS in absence of SiO_2, (**b**), (**f**), (**h**) and (**j**) are higher magnifications of (**a**), (**e**), (**g**) and (**i**), respectively

magnification image in Fig. 6d. The dense clusters of self-assembled FSNP coated with HDTMS greatly improved the treatment durability as proved from the washing test results. EDX of the cotton fabric treated with FSNP and HDTMS showed strong Si peak with an increase in the wt% of both O and Si which indicates the successful incorporation of FSNP and formation of polysiloxane layer that deposited on the surface of cotton fibers. SEM image of cotton fabric pretreated with EDTA followed by modification with hydrophobic solution of SNP and HDTMS showed a coating with a moderate surface roughness, as shown in Fig. 6e, f. EDTA Pretreatment blocked the active reaction sites on cotton surface. Therefore, hydrophobic silica solution did not react well with cotton and deposited on the surface without strong bonding which resulted in a weak washing durability as proved from washing results. One step treatment with lower concentration of EDTA (2 wt%) in presence of SNP showed a quite dense coating making the surface rougher than the surface in the two steps treatment, as shown in Fig. 6g, h. This indicates that the performance of EDTA in one step is better than the two steps treatment which resulted in a higher WCA of 160.2° and lower SA of 2.0°. This attributed to the better crosslinking of EDTA as a tetra-carboxylic acid with both SNP and HDTMS when added together in one solution and so showed a dense coating on the cotton surface. SEM of cotton fabric treated with EDTA and HDTMS in absence of SNP shows the surface coated with a dense oily layer of the hydrophobic solution of EDTA and HDTMS without any surface roughness, as shown in Fig. 6i. This treatment showed the best washing durability among all different treatments. EDTA with four carboxylic acid groups could cross-link with higher concentrations of HDTMS and when applied to cotton fabric dense layers of the self-assembled silanol solution was formed on the surface of cotton fabric. EDX result of this treatment showed an increase in the wt% of Si and O as a result of the deposition of HDTMS layers which cross-linked to cotton surface by EDTA by great number of ester bridges resulting in an excellent washing durability.

FTIR analysis

Figure 7 showed the different FTIR spectra of pristine and treated cotton fabrics. Figure 7a showed spectrum of pristine cotton with the characteristic bands of OH stretching at 3,500–3,000 cm^{-1} and the CH stretching at 2,800–2,900 cm^{-1} (Liu et al. 2014). FTIR spectrum of cotton treated with HDTMS or both SNP and HDTMS showed new band at 2,844 cm^{-1} and increased band intensity at 2,918 cm^{-1} of $–CH_2$ due to the C–H symmetric and antisymmetric stretching respectively indicated the introduction of long chain hydrocarbon of HDTMS, as shown in Fig. 7b, c. Spectrum b showed significant improvement of the OH stretching band around 3,418 cm^{-1}, which confirmed the presence of large amount of free silanol groups of HDTMS on cotton surface. The reduction in peak at 3,418 cm^{-1} in spectrum c proved the condensation reaction of HDTMS with SNP since the silanol stretching band decreased obviously. These changes

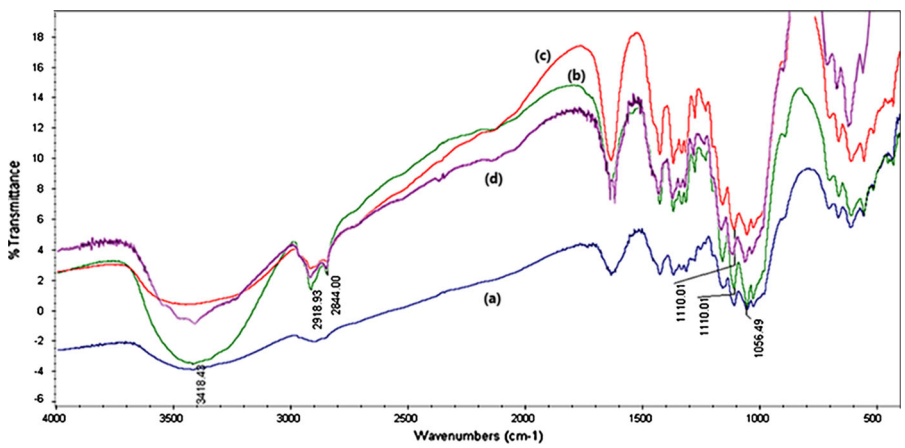

Fig. 7 FTIR spectra of **a** pristine cotton, **b** cotton treated with HDTMS, **c** cotton treated with SNP and HDTMS and **d** cotton treated with EDTA and HDTMS

proved that the total numbers of silanol –OH groups were reduced and hydrophobic SNP were formed on the surface of cotton fabrics. Besides, the improvement in peak intensity at 1,056 and 1,100 cm^{-1} is attributed to the introduction of Si–O–Si of silica nano-particles and HDTMS. Spectra of cotton fabrics treated with EDTA and HDTMS showed two peaks at 2,847.2 and 2,918.9 cm^{-1} corresponding to the asymmetric and symmetric stretching of CH_2 of long chain of HDTMS. Spectrum of cotton treated with EDTA is shown in Fig. 7d. In addition to the two characteristic peaks of the long chain hydrocarbon of HDTMS, two characteristic bands for the carboxylic carbonyl group of EDTA at 1,637 and 1,618 cm^{-1} appeared which indicated that EDTA was incorporated successfully in the treatment.

Conclusions

A simple and cost effective one step solution immersion process was applied to prepare superhydrophobic cotton fabric based on EDTA and HDTMS. This treatment showed WCA of 159.8° and SA of 4°. EDTA was used for the first time as a crosslinking polycarboxylic acid to improve the washing durability of the developed superhydrophobic fabrics. The durability test showed that both silane coupling agents and EDTA greatly improved the washing durability of the superhydrophobic fabrics. Unlike other polyacids, EDTA was used without catalyst, did not cause fabric yellowing and has low cost compared to the high price of both polyacids and silane coupling agents. Treatment with FSNP involves four separate steps with each step is followed by heat treatment which required too long treatment duration and this considered as the main drawback of its application. However, EDTA was applied in one step and showed the best hydrophobic washing durability. Therefore, EDTA showed very promising alternative for improving the superhydrophobicity of cotton textile and treatment durability.

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