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ORIGINAL RESEARCH

Dynamic hydrophobic behavior of water droplets impact on the cotton fabrics coated with POSS block copolymer

Que Kong · Zuoliang Zhang · Zhiguang Li · Xuehong Ren

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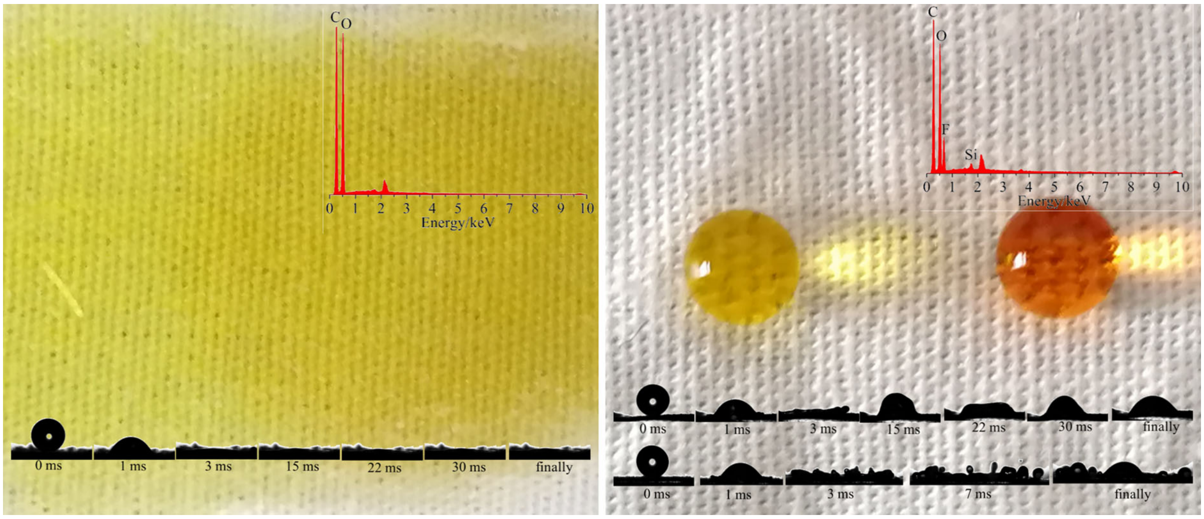
Abstract In this paper, fluorinated polyhedral oligomeric silsesquioxanes block copolymer with trifluoroethyl methacrylate as arms was synthesized through atom transfer radical polymerization, and blended with poly(vinylidene fluoride) for the preparation of an organic–inorganic hybrid water-repellent material. The organic–inorganic hybrid material was coated on cotton fabric for the hydrophobicity. The surface morphologies, surface chemical composition and contact angles of the coated cotton surface were measured. Furthermore, the dynamic hydrophobic behavior of water droplet impact on the cotton fabric was investigated. The bounce tendency after the water droplets hitting the coated fabric was obvious, and the morphology variation, spreading diameter, bouncing height and energy conversion of the water droplets were examined. In addition, a model of sticking and splashing of water droplet on the cotton surface was proposed. The coated hydrophobic fabrics displayed good water-repellent and durability, and maintained hydrophobicity even after treatment with acid and alkaline compounds, scraping with a knife, rubbing, and tape adhesion.

Q. Kong · Z. Zhang · Z. Li (✉) · X. Ren (✉)
Key Laboratory of Eco-textiles of Ministry of Education, College of Textiles and Clothing, Jiangnan University, Wuxi 214122, China
e-mail: lizg@jiangnan.edu.cn

X. Ren
e-mail: xhren@jiangnan.edu.cn

Graphic abstract



Keywords Polyhedral oligomeric silsesquioxanes · Cotton fabric · Dynamic hydrophobic behavior · Durability

Introduction

Cotton is one of the most widely used fabrics due to its soft, comfortable and breathable properties, which makes it occupy a very considerable market. However, cotton fabrics are easily wetted by water or stained. Once stained, large amounts of water with detergent are consumed for the cleaning, which will cause some pollution problems to the environment. (Cao et al. 2016; Li et al. 2015; Xue et al. 2016) Therefore, it is necessary to develop cotton fabrics with stain-resistant and self-cleaning functions by hydrophobic treatment.

In general, hydrophobic surfaces can be achieved by the construction of surface roughness or surface modification using low surface energy materials. (Kang et al. 2018; Liu et al. 2010; Ragesh et al. 2014) Organic–inorganic hybrid materials have low surface energy which containing fluorine and silicon. Inorganic components can provide good thermal stability, strength and oxidation resistance for hybrid polymers, and organic components can modify materials at the molecular level, which can influence the property of the material surface. The hydrophilic fabric can be treated by the organic–inorganic hybrid materials to obtain hydrophobic property. By means of this type of hydrophobic fabric treatment, the resistance of the fabric to water could be improved while establishing a self-cleaning effect (Hou et al. 2015; Kong et al. 2014; Xue et al. 2015; Xue et al. 2013a).

Fluorine-containing polyhedral oligomeric silsesquioxanes (POSS) block copolymer is an organic–inorganic hybrid material which can be prepared through atom transfer radical polymerization (ATRP). (Zhang et al. 2017; Zhang and Müller 2013) POSS has an inorganic core with a Si–O–Si structure which has excellent thermal properties and good performance in terms of hydrophobic properties. (Gao et al. 2010; Wang et al. 2011) The side chain may be an organic material which has good compatibility with the POSS core. The organic group could have a great selectivity such as amino, hydroxyl, and vinyl, which can be used for the preparation of hybrid polymers with different functions. Therefore, POSS provides a chemical method to connect organic and inorganic components, which in turn can change the structure and property of the material. On the other hand, fluoropolymers are valued as a class of low surface energy materials which attract increasing attention. (Hong et al. 2016; Li et al. 2014; Xue et al. 2013b) The fluorine-containing polymer has excellent thermal stability, chemical stability, and hydrophobicity. The fluorine-containing groups tend to concentrate and align on the surface of the film during the film formation process and effectively reduce the surface free energy of the film. The low surface energy gives excellent surface hydrophobic and antifouling properties, which makes fluoropolymers promising high performance coating materials. Poly(vinylidene fluoride) (PVDF) is an excellent fluoropolymer which has high mechanical strength, low surface energy, excellent chemical stability and abrasion. (Kuila et al. 2017) Therefore, it has been regarded as one of ideal materials for preparing functional films.

Water impact behavior is used as a sufficient and necessary identification for superhydrophobic soft porous materials. Hence, the water droplet impact behavior test is a comprehensive method to characterize surface wetting. (Crick and Parkin 2011) The impact of droplets is followed by spreading, receding, bouncing or splashing which depend on the impact viscosity, surface tension, density and radius of the droplet, and the surface properties. (Zang et al. 2013) To further expand the application of the coated textiles, the morphology variation, spreading diameter, energy conversion, and adhesion of the impacted droplet need to be investigated. Consequently, the water impact behavior is an effective indicator for the surface properties.

In this study, we report the novel organic–inorganic hybrid water-repellent materials of POSS-poly(trifluoroethyl methacrylate)$_8$ (POSS-(PTFEMA)$_8$)/PVDF by one-step coating method to construct hydrophobic cotton fabrics. The organic–inorganic hybrid materials were applied onto cotton fabrics via a conventional "dipping-drying-curing" method with no need of pretreatment. This method can enhance the surface roughness and durability of hydrophobicity simultaneously without reducing fabric wearability. The dynamic hydrophobic behavior was analyzed by the water droplet impact test. The morphology variation, spreading diameter, bouncing height and energy conversion of the water droplets impacted on the coated fabric surface were examined. The experimental and theoretical combination methods were used to clarify the hydrophobic mechanism of the coated fabrics. The relationship between dynamic behavior of water droplets and the surface structure of fabric were investigated. Finally, the chemical and mechanical stability of the coated cotton fabrics were evaluated.

Experimental section

Materials

Cotton fabrics were purchased from Guandong Textile Dyeing Garment Co., Ltd. (Zhejiang, China). The POSS block copolymer POSS-(PTFEMA)$_8$ was synthesized through ATRP as previously reported (Li et al. 2017b). The molecular weight of POSS-(PTFEMA)$_8$ was 107,150 and the polymer dispersity index was 1.57. ANOSET® Yellow 4GN was provided from Shanghai Anoky Group Co., Ltd. (Shanghai, China) and MEGAFIX® red B-2BF was supplied by Shanghai Matex Chemicals Co., Ltd. (Shanghai, China). PVDF was purchased from Dongguan Xuanyang Plastic Material Company (Guangdong, China).

Preparation of hydrophobic cotton

Cotton fabric samples (3 cm × 3 cm) were washed with deionized water. A certain amount of POSS-(PTFEMA)$_8$ (0.2 g and 0.4 g) and PVDF (0.3 g) were dissolved in 20 mL N,N-dimethylformamide. The cotton fabrics were dipped into the coating solution and dried in an oven at 90 °C for 40 min, and then cured at 150 °C for 90 s. The coating process is shown in Fig. 1.

Characterization

Surface morphologies of the cotton fabrics were characterized by a scanning electron microscope (SEM) (HITACHI, SU-1510, Tokyo, Japan), and energy dispersive X-ray spectroscopy (EDS) was used to analyze the surface element compositions. Water contact angle (CA) was tested 5 times (PT-602A, Dong Guan Precise Test Equipment CO., LTD). The water droplet (12 μL) was dropped from a certain height (130 mm) to the surface of the fabric. The evolution of water droplet from the beginning to the static state was recorded by high speed CCD (American Trouble Shooter HR, 10,000 FPS). The chemical durability of the coated cotton fabric was evaluated by immersing into solutions of pH = 1 or pH = 13 which was adjusted by hydrochloric acid or sodium hydroxide. The physical durability of the coated cotton fabric was evaluated by scraping with knife repeatedly, rubbing by hands many times and sticking with tape.

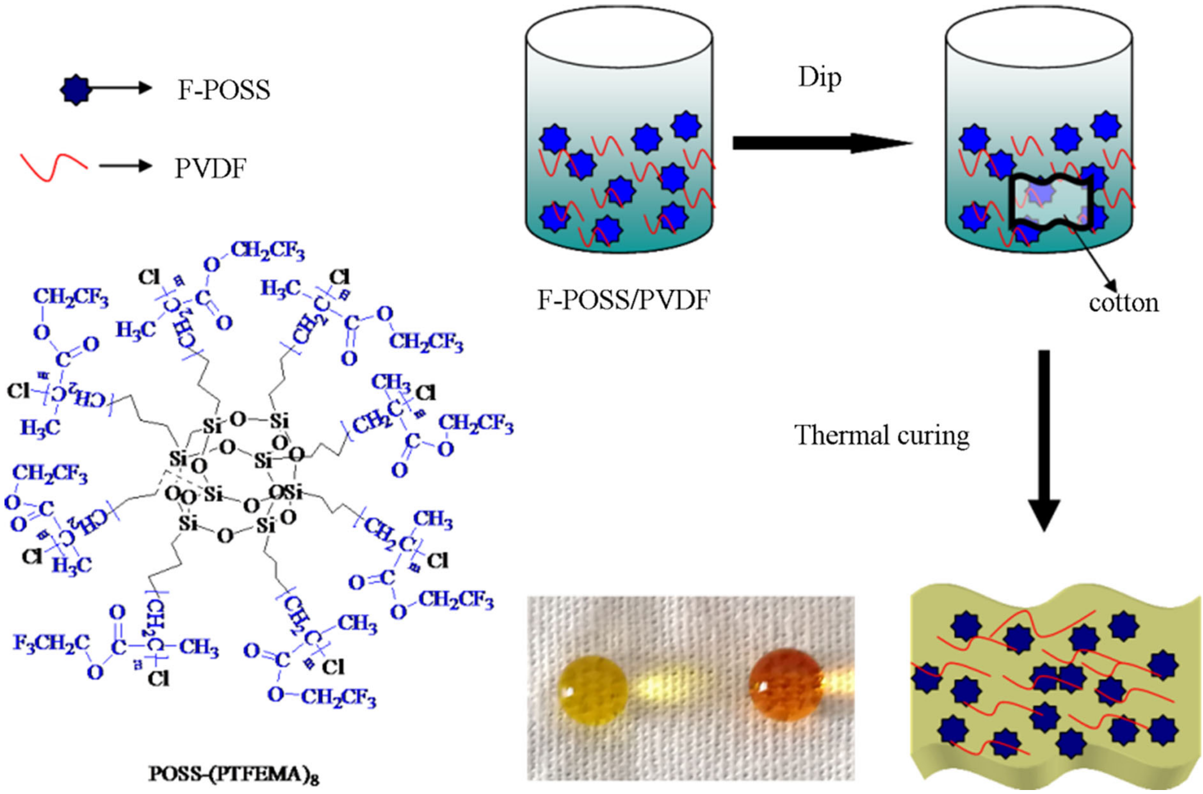


Fig. 1 Schematic illustration of the preparation of the coated cotton fabrics

Results and discussion

Characterization of the coated cotton fabrics

The SEM images of the surface morphologies of the untreated cotton fabric and coated cotton fabric are shown in Fig. 2. There is a certain difference between the untreated cotton fabric and coated cotton fabric. The surface of the coated cotton fabric appeared to be rougher due to the POSS block copolymer and PVDF coated on cotton fabric, which can enhance the hydrophobic property. On the other hand, the chemical composition also affects the surface hydrophobic property, and the EDS analyses are illustrated in Fig. 3.

As exhibited in Fig. 3a, the untreated fabric has only two peaks of C and O. While new peaks for F and Si are observed for the coated cotton fabric as illustrated in Fig. 3b. Si is from POSS block copolymer and the weight percent is 0.70%. F is from POSS block copolymer and PVDF and the weight percent is 11.19%. These results suggest that the organic–inorganic hybrid materials appear on the cotton surface. Images of the point distribution of chemical elements on the untreated and coated cotton fabric are displayed in Figs. 4 and 5.

As depicted in Figs. 4 and 5, the distribution of C and O elements of the untreated cotton fabric is uniform and the area ratio is 51:49. On the other hand, the C, O, F, and Si elements distribution ratios on the coated cotton are 45:38:12:6. Clearly, the POSS block copolymer and PVDF are covering the surface, and the element distribution of F and Si are uniform.

Hydrophobic properties of the coated cotton fabrics

Figure 6a shows the contact angles of the coated cotton fabrics. With the enhancement of POSS block copolymer, the contact angles increased from 120.3° ± 1.2° to 134.4° ± 1.4°. The POSS block copolymer has the Si and F elements which have low

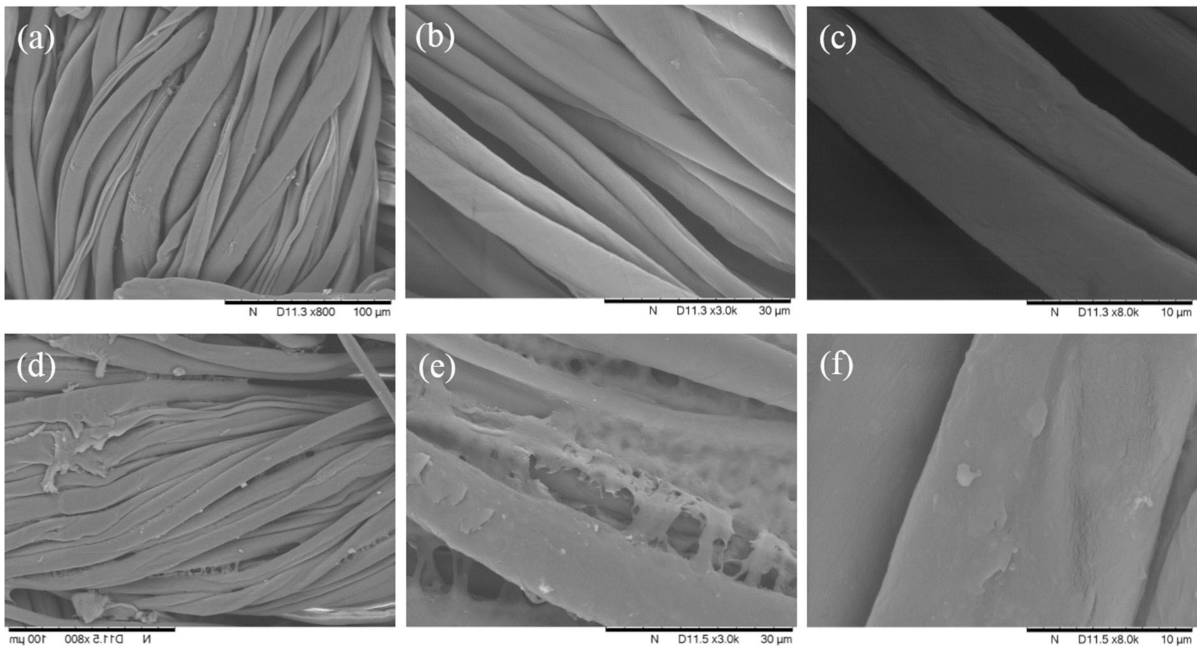


Fig. 2 SEM images of **a–c** untreated cotton fabric, **d–f** coated cotton fabric

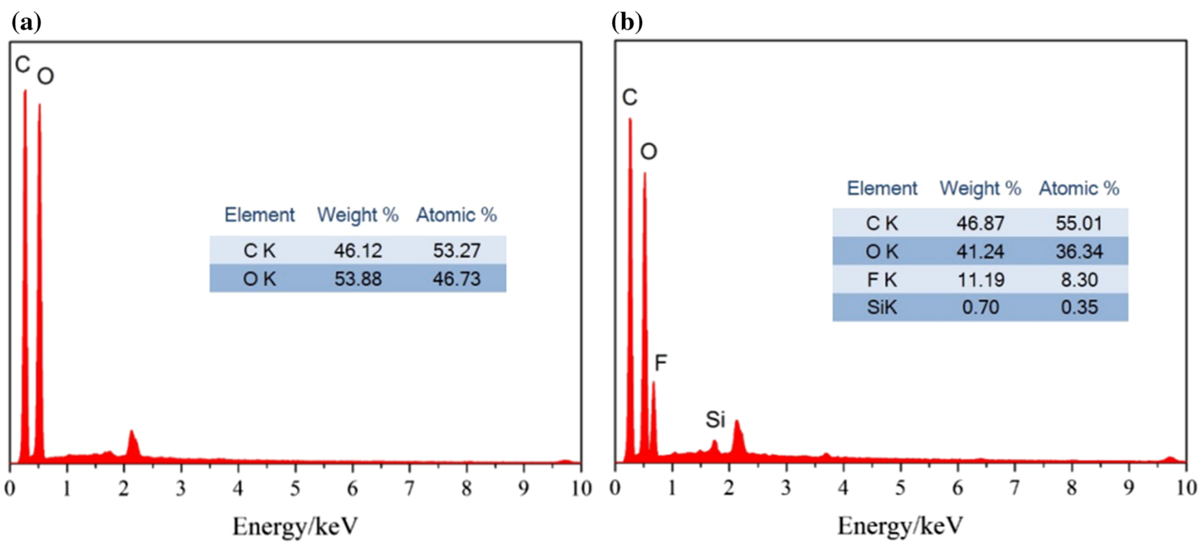


Fig. 3 EDS of **a** untreated cotton fabric and **b** coated cotton fabric

surface energy, resulting in the high water contact angle. With added PVDF, the contact angle enhanced to 139.9° ± 1.6° due to the cooperative effect of the POSS block copolymer and PVDF.

The untreated cotton fabric has very good hydrophilic properties. When the water droplets of ANOSET® Yellow 4GN and MEGAFIX® red B-2BF solutions are added to the surface of the fabric, the dyes are quickly absorbed into the cotton fabric and penetrated inside. As a result, the cotton fabric is dyed which is shown in Fig. 6b. However, the coated cotton fabric shows good hydrophobicity and resists

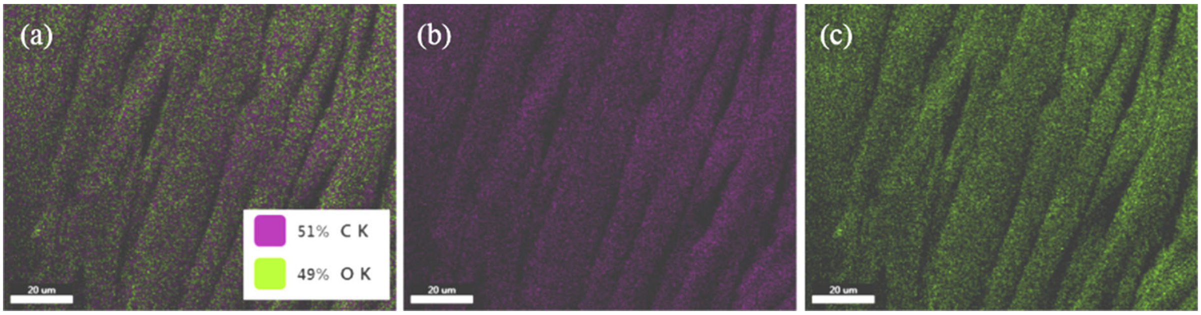


Fig. 4 Point distribution of C and O elements on the untreated cotton fabrics **a** the total point distribution of the C and O element, **b** the point distribution of C element, **c** the point distribution of O element

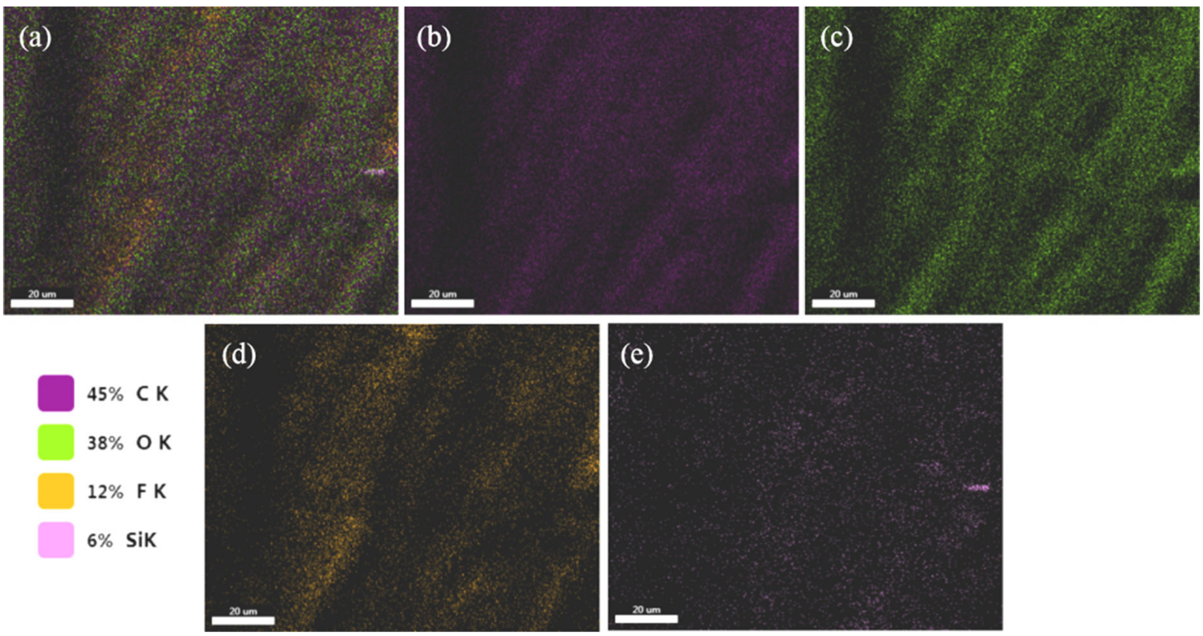


Fig. 5 Point distribution of C, O, F and Si elements on the coated cotton fabrics **a** the total point distribution of the C, O, Si and F elements, **b** the point distribution of C element, **c** the point distribution of O element, **d** the point distribution of F element, **e** the point distribution of Si element

the penetration of droplets, and the shape of the dye droplets is maintained on the coated fabric surface in Fig. 6c. (Xue et al. 2013a; Zhang et al. 2013)

The dynamic behavior of water droplet by hitting the cotton fabric by free fall from a certain height can further serve to measure the wettability. The morphology variation of the droplets impacting on the cotton fabric as time advances are displayed in Fig. 7.

As illustrated in Fig. 7, the droplets exhibit varied impact behaviors such as immersion, spreading, retraction, oscillation, and splashing. In Fig. 7a, the water droplet gradually immerses into the untreated cotton fabric within 3 ms. On the contrary, in Fig. 7b, the droplet spreads out and extends rapidly resulting from the inertia and capillary force, (Liu et al. 2014; Smith and Bertola 2010) and surface tension causes it to return to the center after the maximum spreading. The droplets move up to a certain height and then vibrate to the stationary state on the coated cotton fabric. There is no splash phenomenon in the whole process, and the water droplets remain intact. However, the water droplet breaks into some small droplets

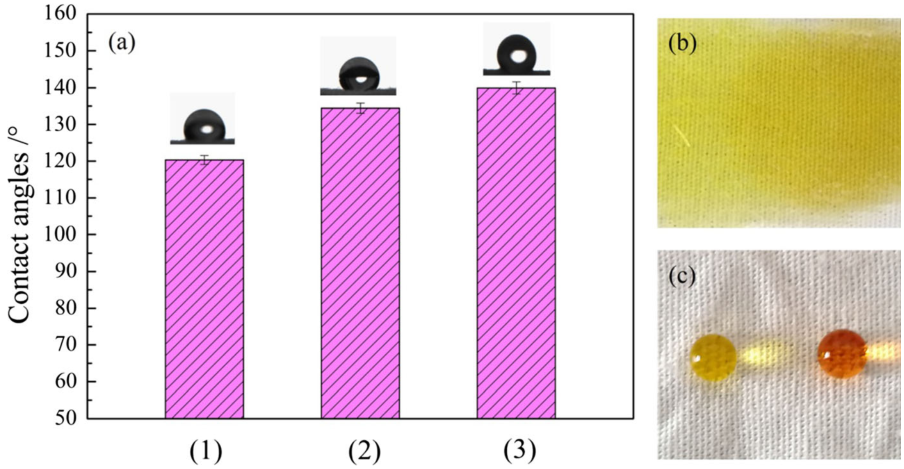


Fig. 6 **a** Water contact angles of cotton fabrics coated by (1) 10 g/L POSS-(PTFEMA)$_8$ (2) 20 g/L POSS-(PTFEMA)$_8$ (3) 10 g/L POSS-(PTFEMA)$_8$ and 15 g/L PVDF; Digital images of dye droplets after contact with the **b** untreated cotton fabric and **c** coated cotton fabric

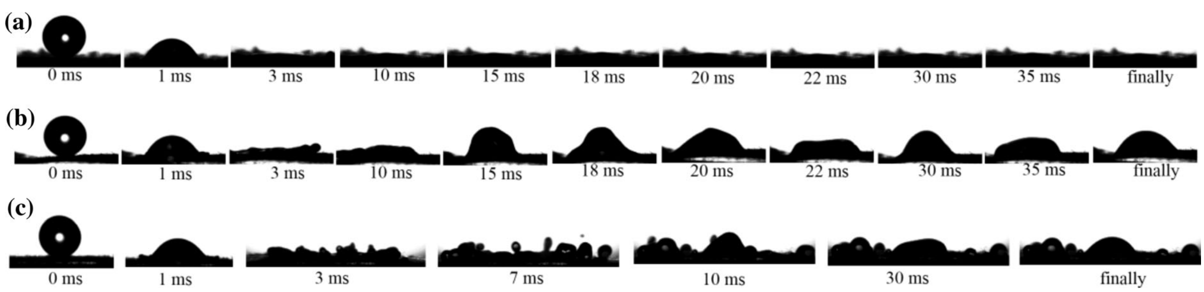


Fig. 7 Surface morphology variation of droplets impacting cotton fabrics, **a** untreated cotton fabric, **b** 10 g/L POSS-(PTFEMA)$_8$ and 15 g/L PVDF coated cotton fabric, **c** 10 g/L POSS-(PTFEMA)$_8$ coated cotton fabric. The initial time of water droplets begin to contact with the cotton fabric is determined as 0 ms

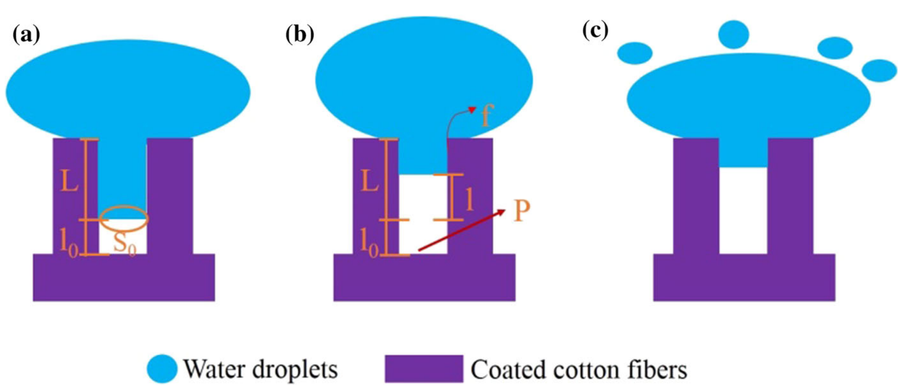


Fig. 8 Model to describe water impact behavior

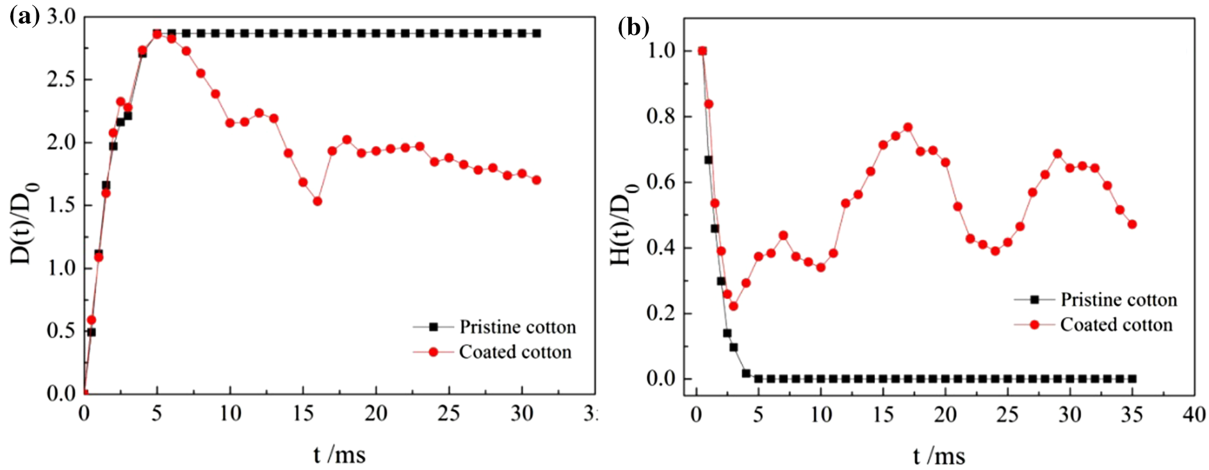


Fig. 9 The D(t)/D_0 values and H(t)/D_0 variations as a function of time of the untreated and coated cotton surface. D(t) and H(t) are the contact diameter and maximum height of the droplet after impact with time and D_0 is the initial diameter of the droplet

and the splash phenomenon occurs as shown in Fig. 7c. The movement of the largest droplet in the center is similar to the water droplet pictured in Fig. 7b.

The cotton surfaces can be identified as concave structures as shown in Fig. 8a. The pressure p in the air pockets determines the depth L, and l_0 is the length between the bottom of the surface and water. After the impact, the water droplet moved up to the maximum height as shown in Fig. 8b, where l is the distance between before and after impacting, and f is the force caused by water surface tension. (Lu et al. 2014) In this process, water experienced adhesion force F_s, which is caused by the concave structure. F_s can be exhibited as shown in Eq. (1)

$$F_S = p_0 S_0 - P S_0 \quad (1)$$

where p_0 and P are the pressures of one standard atmospheric and the concave structure, respectively. S_0 is the bottom area of the water droplet. Here we used the ideal gas law (Eq. (2)):

$$PV = nRT \quad (2)$$

where V and n are the volume and amount of substance of air in the concave structure, respectively. R is the ideal gas constant and T is the temperature, thus Eq. (1) can transformed to Eq. (3)

$$F_S = p_0 S_0 - nRT/(l_0 + l) \quad (3)$$

In Eq. (3), p_0, S_0, n, R, T and l_0 are all constants, therefore, the adhesion force increased as l got larger. When $l < L$ and $F_s < f$, the water just remained intact to the concave structure as shown in Fig. 8b. Furthermore, the water contact angle on the POSS-(PTFEMA)$_8$ coated cotton fabric surface is lower than that on the POSS-(PTFEMA)$_8$ and PVDF coated cotton fabric surface. As a result, the adhesion force of the POSS-(PTFEMA)$_8$ coated cotton fabric surface is higher than that of the POSS-(PTFEMA)$_8$ and PVDF coated cotton fabric surface. (Li et al. 2017c) In this case, if $l < L$ and $F_s > f$, the adhesion force will be greater than the force from water surface tension on the POSS-(PTFEMA)$_8$ coated cotton fabric surface. Hence the water cannot escape from the concave structure, and the water will be splash lead to some water staying on the surface (Fig. 8c).

During the water impact process, the kinetic energy is transformed to interfacial energy, causing the droplet to spread and deform. The droplet retracts after reaches maximum spreading due to the elastic energy. The speed of the retraction is confirmed by the interfacial energy. (Smith and Bertola 2010) However, the droplets could not rebound, and achieve equilibrium after several vibrations on the POSS and PVDF modified surfaces. (Caviezel et al. 2008) On the other hand, the droplets could splash due to the high velocity and strong adhesion (Joung and Buie 2015; Koishi et al. 2017; Xu 2007).

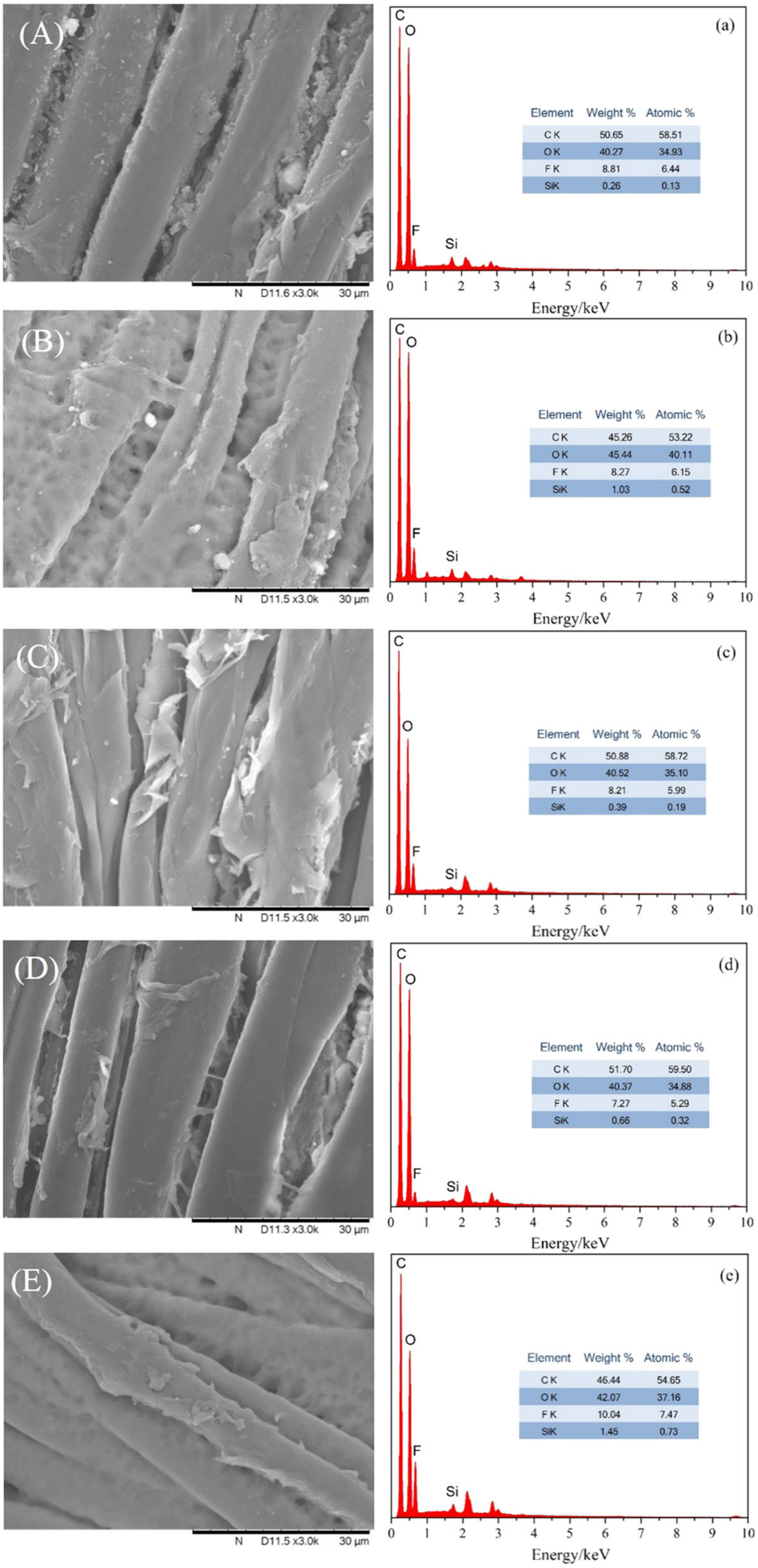


Fig. 10 The SEM images **A**–**E** and of the EDS analysis **a**–**e** surface morphologies of the coated cotton fabrics after stability tests. **A** and **a** acid treatment (pH = 1), **B** and **b** alkaline treatment (pH = 13), **C** and **c** knife scraping, **D** and **d** rubbing, **E** and **e** tape sticking

Table 1 CAs of the coated cotton fabrics after treatments

Treatments	CA/°
Acid treatment (pH = 1)	138.1 ± 1.5
Alkaline treatment (pH = 13)	136.7 ± 1.7
Knife scraping	132.4 ± 1.1
Rubbing	133.9 ± 1.3
Tape sticking	139.5 ± 0.8

The residual energy (E) is calculated as shown in Eq. (4): (Li et al. 2016, 2017a)

$$E = E_1 - E_2 \quad (4)$$

Here, E_1 is the kinetic energy, and E_2 is the dissipation energy during the impact behavior. When a water droplet hits the coated cotton surface, the kinetic energy is converted to surface energy and dissipation energy. (Lee et al. 2012) Furthermore, the surface energy is related to surface roughness. During this process, the dissipation energy is resulting from vibration of the droplets against viscosity. Consequently, the residual energy after impact is lower than the gravitational-potential energy and the surface adhesion, and the droplet does not bounce off. (Li et al. 2017a) The $D(t)/D_0$ and $H(t)/D_0$ values as a function of time of the two surfaces are shown in Fig. 9a, b respectively.

As shown in Fig. 9a, the $D(t)/D_0$ values of untreated cotton fabric are gradually increased and finally stabilized as the droplet gradually penetrates into the interior of the cotton fabric. In opposition, $D(t)/D_0$ rises to the maximum value (D_{max}/D_0) first and then the vertical vibration occurs on coated cotton fabric. The D_{max}/D_0 value of the coated cotton surface is obviously controlled by inertial effect caused by kinetic energy. (Lee and Lee 2011) Furthermore, inertia induces the droplet to expand while the surface is apt to pull back the spreading droplet and dissipate energy limiting the value of D_{max}/D_0. (Visser et al. 2015) The constant $D(t)/D_0$ value indicates the droplet deposition, whereas the decreased $D(t)/D_0$ value indicates the receding motion of the droplet (Fu et al. 2016).

The residual energy influences the bouncing height [$H(t)/D_0$]. (Li et al. 2017c) The $H(t)/D_0$ values on the untreated and coated cotton surfaces as a function of time are exhibited in Fig. 9b. The $H(t)/D_0$ values of the untreated cotton fabric gradually decrease to 0 and tend to be stable within 5 ms. The coated cotton fabric exhibits high $H(t)/D_0$ value, indicating that the surface is hydrophobic. The values go up and down following the vibration of the droplets.

Durability of the coated cotton fabrics

A few durability tests including pH and mechanical stability of the coated cotton fabric are carried out. The SEM images of the surface morphologies and the EDS analysis of the coated cotton fabrics after stability tests are exhibited in Fig. 10. The CAs of the coated cotton fabrics are shown in Table 1.

As shown in Fig. 10, it causes a subtle change on the coated cotton fabrics, and the weight percent of F for all the five cotton fabrics are lower than that of the untreated cotton fabrics. As can be seen from Table 1, after the treatments of strong acid and alkali conditions for 24 h there is no significant change in the contact angle. The coated cotton fabric is stable under acid and alkaline conditions due to the good film-forming properties and excellent stability of POSS block copolymer and PVDF. In addition, the coated cotton fabric is hydrophobic, and acid and alkali are prevented from wetting the cotton fabric. Therefore, the cotton fabric could be protected from the attack of acid or alkali.

Additionally, the coated cotton fabrics are treated by knife scraping, rubbing and tape sticking. Although the weight percent of F is decreased, the surface roughness is changed from Fig. 10C–E. The hydrophobic properties are slightly reduced but no major changes occur. This slight decrease might be due to the partial loss of the surface roughness and the removal of the POSS block copolymer/PVDF coating. However, the wettability remains highly hydrophobic. POSS block copolymer and PVDF deposit on the fabric surface could also penetrate into the cotton fabrics. (Wu et al. 2016) The large amount of POSS block copolymer and PVDF embed in the cotton fabrics and the uniform distribution of F and Si elements on the surface seems to help prevent the loss of hydrophobic property during physical treatments.

Conclusions

Cotton fabrics were modified by organic–inorganic hybrid materials of POSS block copolymer and PVDF. The surface roughness of the coated cotton fabrics increased and the surface energy decreased due to the addition of Si and F elements. The coated cotton fabric was hydrophobic with the water contact angle of 139.9° ± 1.6°. Testing the dynamic hydrophobic behavior, the water droplets quickly penetrated into the untreated cotton fabric due to the excellent hydrophilic property, while the water droplets were vibrating or splashing on the coated cotton fabric due to the adhesion and the surface energy. The coated cotton fabric exhibited a more obvious bounce tendency, lower spreading diameter and higher bouncing height than the untreated cotton fabric. A model of the water droplet impact behavior with sticking and splashing and the adhesion was proposed. In addition, the coated cotton fabrics could withstand strong acid or alkali, knife scraping, rubbing, and tape sticking treatments.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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