


# In situ reduction of TiO<sub>2</sub> nanoparticles on cotton fabrics through polydopamine templates for photocatalysis and UV protection

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**Abstract** In this work, titanium dioxide (TiO<sub>2</sub>) nanoparticles were immobilized on the surface of cotton fabrics by in situ reduction on polydopamine (PDA) templates. Evidenced by the results of scanning electron microscopy/energy-dispersive X-ray analysis (SEM–EDX), Fourier transform infrared and X-ray photoelectron spectroscopy (XPS), PDA films were successfully formed on the surface of cotton fabrics as the templates for the in situ reduction. The morphology, chemical composition and crystal structure of the coated cotton fabrics after in situ reduction of TiO<sub>2</sub> nanoparticles were characterized by SEM–EDX, XPS and X-ray diffraction, respectively. These results confirmed that anatase TiO<sub>2</sub> nanoparticles were well-dispersed on the surface of cotton fabrics. The

photocatalysis experiment results indicated that about 90% of methylene blue solution was degraded after UV illumination of the coated cotton fabrics. The thermogravimetric analysis results demonstrated that the TiO<sub>2</sub> nanoparticles had little effect on the thermal stability of cotton fabrics. The results of UV protection showed that the value of the ultraviolet protection factor (UPF) of the coated cotton fabrics was 127.2. The UPF value was maintained at 108.8 after five cycles of laundering, indicating excellent durability of the coating in terms of UV protection.

**Keywords** TiO<sub>2</sub> nanoparticles · Polydopamine · Cotton fabrics · Photocatalytic activity · UV protection

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## Introduction

Cotton has been widely used in clothing for its high wettability, permeability and comfort. But its disadvantages, such as poor ultraviolet resistance and breeding bacteria, confine its application potential (Gao et al. 2017). Functionalization of cotton fabrics is thus necessary for developing protective clothing. Researcher have focused on this area, and especially TiO<sub>2</sub> has been used to functionalize cotton fabrics with antimicrobial properties and UV protection together with antistatic, oil repellency and self-cleaning properties (Li et al. 2015; Karimi et al. 2014; Mishra and

Butola 2017). Although there are several methods to immobilize TiO<sub>2</sub>, the durability of the TiO<sub>2</sub> nanoparticles on cotton fabrics is still a big challenge.

Researchers have done extensive work to improve the durability of functional coatings on textiles by physical etching and chemical crosslinking. Sobczyk-Guzenda et al. (2013) implemented a radio frequency plasma enhanced chemical vapor deposition technique to deposit TiO<sub>2</sub> nanoparticles on cotton fabrics for self-cleaning and wettability. Li et al. (2017a) adopted the low temperature atomic layer deposition (ALD) technology to deposit TiO<sub>2</sub> nanoparticles on fabrics for infrared and thermal insulating properties. Bonet et al. (2015) employed acrylic resin and polycarboxylic acid as bonding agents to improve the durability of sprayed TiO<sub>2</sub> nanoparticles on cotton fabrics. Khajavi and Berendjchi (2014) modified the surface of cotton fabrics to introduce dicarboxylic acid with different chain lengths, and then TiO<sub>2</sub> nanoparticles were captured by the –COOH groups to generate excellent self-cleaning properties against washing. Yu et al. (2013) immobilized TiO<sub>2</sub> nanoparticles covalently on cotton fabrics with by co-grafting polymerization of 2-hydroxyethyl acrylate under  $\gamma$ -ray irradiation, and the affinity between the nanoparticles and fibers were greatly improved to withstand 30 cycles of washing. Even though the affinity between nanoparticles and fibers can be improved by either physical etching or chemical crosslinking, the methods are rather complicated as special equipment is required and the treatment conditions are hard to control.

Dopamine can be polymerized on the surface of inorganic and organic materials to form a functional polydopamine (PDA) layer (Lee et al. 2007; Jiang et al. 2011). The method is simple and environmental friendly with moderated reaction conditions and a controllable reaction process. Strong affinity between deposited nanoparticles and fabrics can be achieved by applying polydopamine as a template, and this is due to the ‘bridging’ effect of PDA. Wang et al. (2012) formed a layer of silver nanoparticles on polydopamine coated polyester fibers, the resulted fibers showed excellent electrical conductivity. Xu et al. (2016) deposited silver nanoparticles on polydopamine coated PET fabrics, and the modified fabrics exhibited excellent anti-wetting and self-cleaning properties after fluorination. Sa et al. (2014) deposited silver nanoparticles on polydopamine coated aramid

fibers for excellent electric conductivity with durability. Xu et al. (2013) achieved durable antimicrobial properties for cotton fabrics by the deposition of silver nanoparticles on polydopamine. Wang et al. (2015) deposited silver nanoparticles on polydopamine coated kapok fabrics, and highly-efficient antibacterial properties against staphylococcus aureus were developed. He et al. (2014) conducted dopamine polymerization to form an adhesive coating for improving the color fastness of fabrics. Liu et al. (2015) coated fabrics with polydopamine-encapsulated octadecylamine by in situ polymerization, so as to endow the fabrics with self-cleaning and self-healing abilities. According to the literature, there is no reports on the application of polydopamine as a template for deposition of TiO<sub>2</sub> nanoparticles on cotton fabrics.

In this study, dopamine was self-polymerized to form surface-adherent polydopamine thin films on cotton fabrics, and a durable and well distributed layer of TiO<sub>2</sub> nanoparticles was further deposited on the PDA-coated cotton fabrics by in situ reduction. The TiO<sub>2</sub>-coated cotton fabrics were characterized by SEM, XPS and XRD. Moreover, the photocatalytic activity, thermal stability together with UV protective properties of the TiO<sub>2</sub>-coated cotton fabrics were investigated.

## Experimental

### Materials

Cotton fabrics (plain, 135 g/m<sup>2</sup>, density: 570/10 cm for warp and 280/10 cm for weft) were purchased from Wuhan Yudahua Textile Co., Ltd, by the fabrics were cleaned by acetone and deionized water before use. 3-hydroxytyramine hydrochloride (dopamine hydrochloride) was purchased from Aldrich Chemical Co. (Milwaukee, USA). Tris(hydroxymethyl) amino-methane (Tris), ammonium fluotitanate ((NH<sub>4</sub>)<sub>2</sub>TiF<sub>6</sub>) and boric acid (H<sub>3</sub>BO<sub>3</sub>) were obtained from Aladdin Chemical Co. (Shanghai, China). All the chemicals were in analytic grade and were used without further purification.

### Polymerization of dopamine on cotton fabrics

A dopamine solution with the concentration of 10 mM was dissolved in the Tris buffer solution. Then, the pH

value of the solution was adjusted to 8.5 by adding HCl. Cotton fabrics were dipped into the freshly prepared dopamine solution at room temperature. After stirring for 24 h, the fabrics were washed thoroughly with deionized water followed by hanging to dry.

### In situ reduction of TiO<sub>2</sub> nanoparticles

In situ synthesis and loading of TiO<sub>2</sub> nanoparticles on the PDA-coated cotton fabric are illustrated in Scheme 1. A PDA-coated cotton fabric was immersed into an aqueous solution containing 0.1 M (NH<sub>4</sub>)<sub>2</sub>TiF<sub>6</sub> and 0.3 M H<sub>3</sub>BO<sub>3</sub>, and then the solution with the fabric were placed on an overhead shaker (at 40 °C) with a speed of 100 rpm for 8 h. After in situ reduction, the cotton fabrics were rinsed with deionized water and dried in a vacuum oven.

### Characterizations

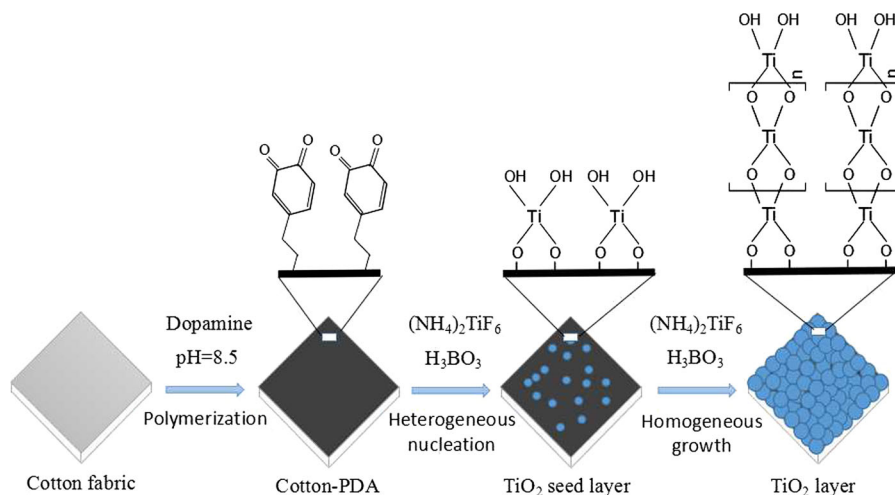
The surface morphology and elemental composition of cotton fabrics were observed on a scanning electron microscopy (SEM, JSM-5600LV, JEOL, Japan) with an energy dispersive X-ray spectrum (EDX, Oxford Instruments, Oxford, UK). Fourier transform infrared (FT-IR) spectra were obtained on a Nicolet 5700 instrument (Nicolet Co., US) accompanied with a wavenumber range of 4000–400 cm<sup>-1</sup> and a resolution of 2 cm<sup>-1</sup>. The amount of TiO<sub>2</sub> coated on cotton fabrics was measured by an inductively coupled plasma-mass spectrometry (ICP-MS, Agilent 7900,

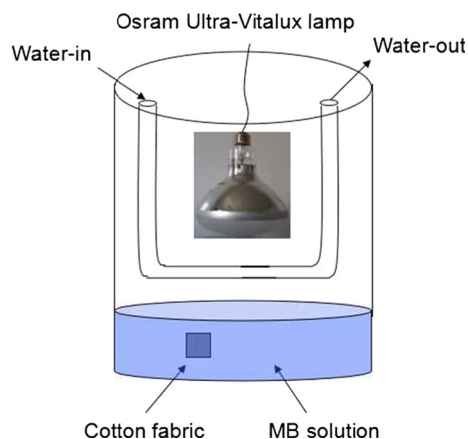
Agilent Technologies, Santa Clara, USA). XPS measurement was performed on a PHI 5000C ESCA system with a Mg K $\alpha$  source at 14.0 kV and 25 mA (Perkin-Elmer, America). A transmission electron microscopy (2100F, JEOL Inc, Japan) was used to observe the TiO<sub>2</sub> nanoparticles extracted from the coated cotton fabrics by ultrasound. The X-ray diffraction (XRD) spectra were collected on an X-ray diffractometer (D/max 2500, Rigaku, Japan) using Cu K $\alpha$  radiation with the diffraction angle range  $2\theta = 10^\circ\text{--}80^\circ$ , at 40 kV and 200 mA.

### Photocatalytic activity

The photocatalytic activity of the TiO<sub>2</sub>-coated cotton fabrics was measured by degradation of MB in an aqueous solution under UV illumination by a 300 W Osram Ultra-Vitalux lamp (mixture of lights, wavelength range 365–450 nm). Photocatalysis experiments were carried out on a self-regulating UV irradiation instrument (Fig. 1). For irradiation experiments, a 100 mL of MB solution (10 mg/L) with samples immersed in were taken into the photoreactor. The degradation was monitored by sampling the solution (2 mL) at a regular interval during the irradiation. The concentration of dye in each sampling solution was measured by recording its absorbance on an UV–visible spectrophotometer (U-4100, Hitachi, Japan) at the wavelength of 664 nm.

**Scheme 1** Schematic diagram and chemical structure of dopamine and TiO<sub>2</sub>-coated cotton fabric





**Fig. 1** Diagram of the self-regulating UV irradiation instrument

### Thermogravimetry analysis

Thermogravimetry analysis (TGA) was conducted on a Netzsch TG209 F1 thermal analyzer, with a heating rate of 10 °C/min from 30 to 700 °C under a nitrogen atmosphere at a flow rate of 60 mL/min. The instrument calibration included the balance correction and temperature correction, and the temperature was reproducible to  $\pm 0.1$  °C and the mass to  $\pm 0.1\%$ .

### UV protective property

The UV protective property of the TiO<sub>2</sub>-coated cotton fabrics was measured using a UV-1000F ultraviolet transmission analyzer, according to the standard AS/NZS 4399:1996. The coated samples were washed in an automatic washing machine to analyze the durability of coating, in accordance with the AATCC Test Method 135-2000.

## Results and discussion

### Characterization of PDA-coated cotton fabrics

Dopamine was self-polymerized in an alkaline aqueous solution to coat the surface of cotton fabrics. The polymerization process was initiated by adjusting the pH value of the aqueous solution. The surface morphology of the PDA-coated cotton fabrics was obtained by SEM, as shown in Fig. 2. The photographic images of the cotton fabrics before and after

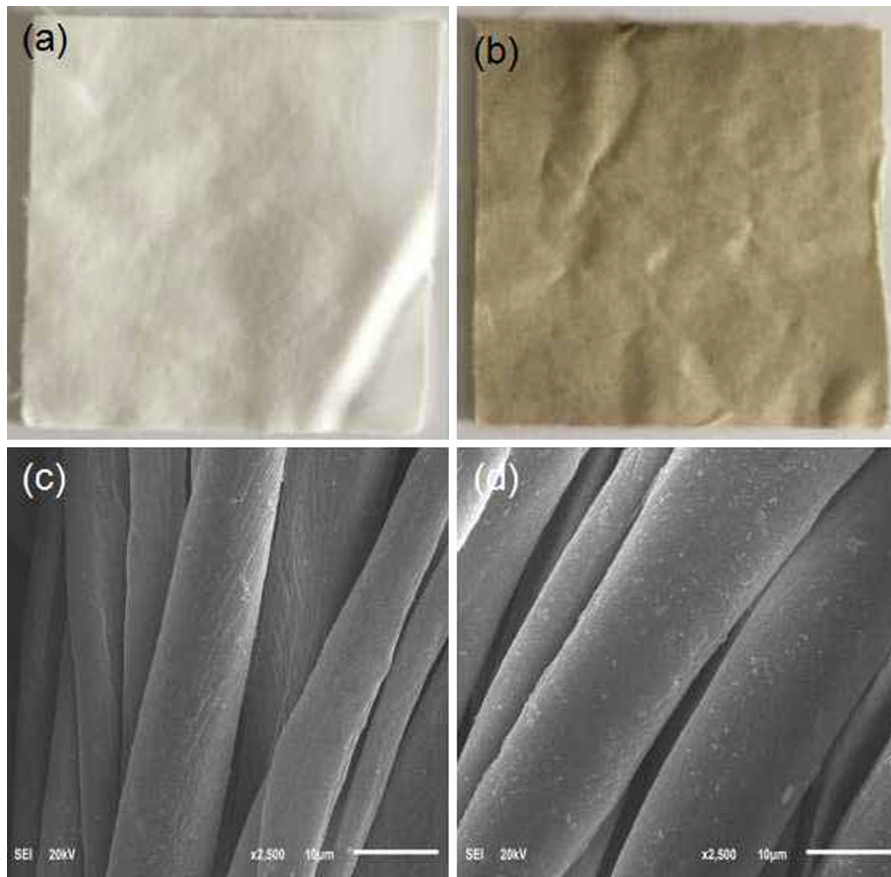
treatment are depicted in Figs. 2a, b, respectively. The color of cotton fabrics turned from white to yellow/brown after the self-polymerization procedure, and this was due to the formation of melanin from the oxidization of the catechol groups of dopamine (Bernsmann et al. 2011). The SEM images of Fig. 2c, d exhibit the surface morphology of the pristine and PDA-coated cotton fabrics, respectively. The morphology of pristine cotton fabrics depicts a smooth surface with characteristic grooves. However, the grooves of cotton fibers were covered with the polydopamine film after the polymerization, resulting in a coated layer on the surface of fibers.

The PDA-coated cotton fabrics were characterized by EDX to determine the chemical elements, as shown in Fig. 3. Compared to the spectrum of the pristine cotton fabric, the spectrum of the PDA-coated cotton fabric exhibited the signal of nitrogen with a weight percentage of 6.0%. It was evident that the nitrogen was originated from dopamine, suggesting the successful polymerization of dopamine on the cotton fabrics.

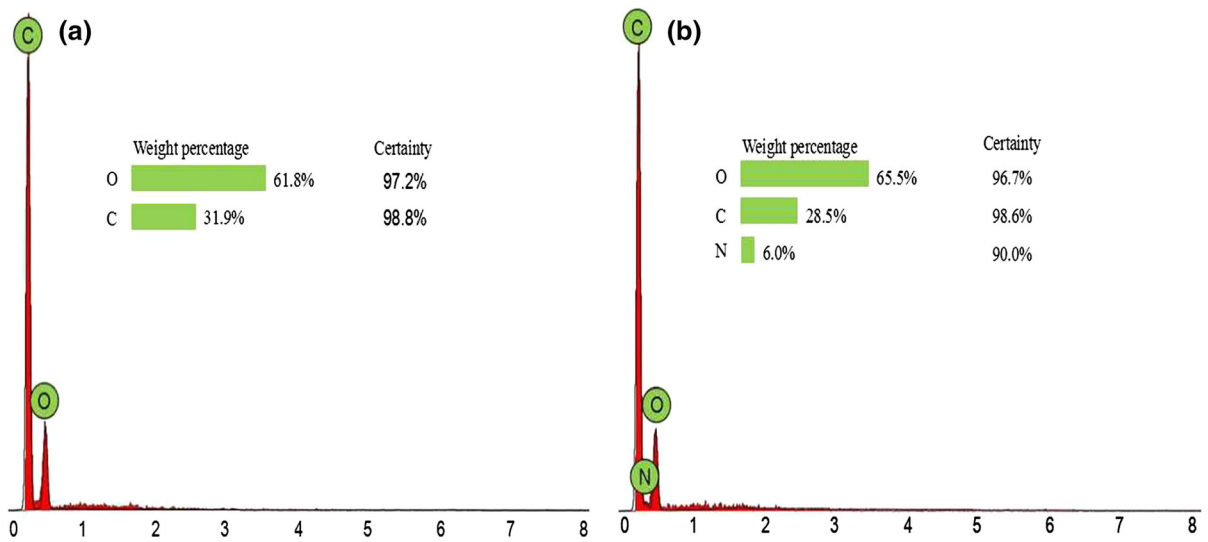
The formation of dopamine on cotton fabrics has been further confirmed by FTIR spectroscopy and XPS spectrum, as shown in Fig. 4. Compared to the spectrum of the pristine cotton fabrics, the FTIR spectrum of the PDA-coated cotton fabrics contains a high intensity peak at 1614 cm<sup>-1</sup>, which is attributed to the stretch vibration of aromatic rings of dopamine (Ma et al. 2011a; Xu et al. 2016). The XPS survey spectra reveal the presence of C1s (at 286.2 eV) and O1s (at 532.7 eV) signals for the both the pristine and PDA-coated cotton fabrics. A new signal at 400 eV corresponding to N1s (7.53%) was noticed after the self-polymerization of dopamine on the cotton fabrics, and the newly detected signal was attributed to the amino groups of dopamine (Chen and Feng 2014). These results further demonstrate that the polydopamine film has been coated on the surface of cotton fabrics.

### Characterization of TiO<sub>2</sub>-coated cotton fabrics

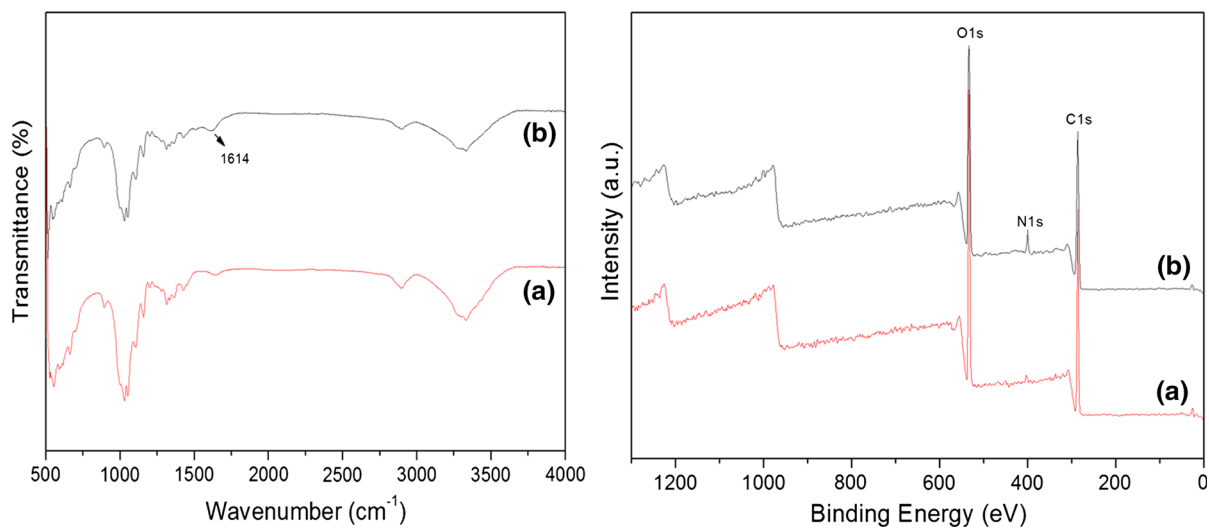
After the in situ reduction of TiO<sub>2</sub> nanoparticles on the template of polydopamine, an evenly distributed film of nanoparticles was formed on the surface of cotton fabrics. To show the deposition of TiO<sub>2</sub> nanoparticles on the PDA-coated cotton fabrics, the TiO<sub>2</sub>-coated cotton fabrics were characterized by SEM, as shown in



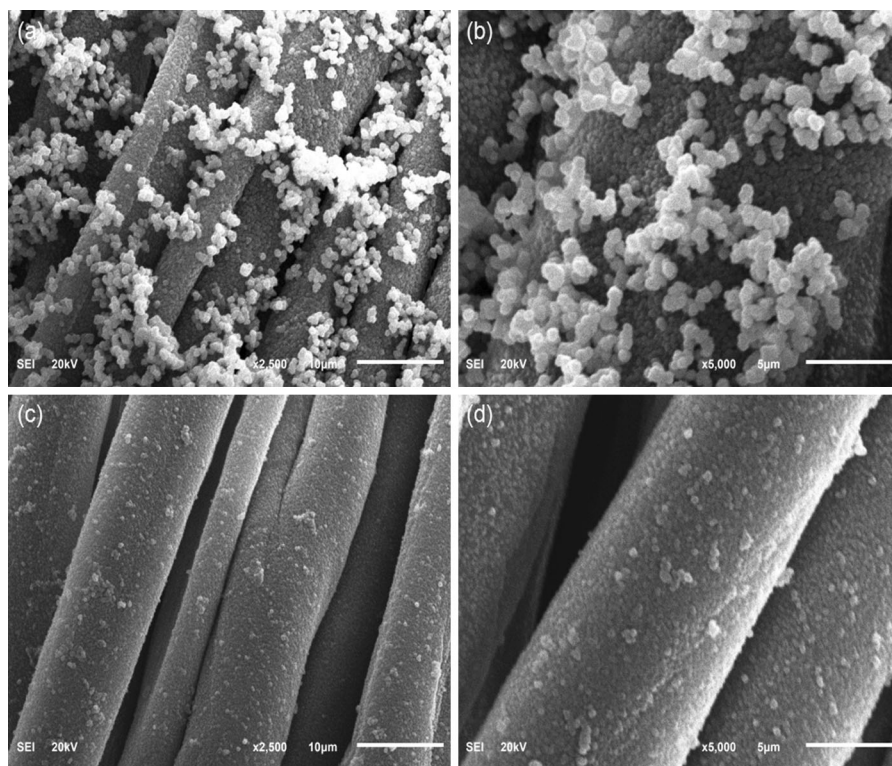
**Fig. 2** Photographs and SEM images of cotton fabrics (a, c) and PDA-coated cotton fabrics (b, d)



**Fig. 3** EDX spectra of cotton fabrics (a) and PDA-coated cotton fabrics (b)



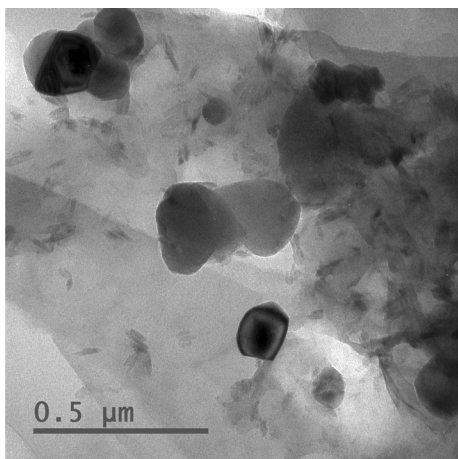
**Fig. 4** FT-IR and XPS survey spectra of cotton fabrics (a) and PDA-coated cotton fabrics (b)



**Fig. 5** SEM images of  $\text{TiO}_2$  nanoparticles deposited on cotton fabrics before (a, b) and after washing (c, d)

Fig. 5a, b. The  $\text{TiO}_2$ -coated cotton fabric was completely covered by nanoparticles, leading to the formation of a dense film of  $\text{TiO}_2$  nanoparticles on the surface of the fabric. The nanoparticles have a round shape with almost the same size (about 500 nm)

as seen from the SEM images. However, the size of the extracted  $\text{TiO}_2$  nanoparticles from the coated fabric lies from 100 nm to 250 nm in the TEM image (Fig. 6). One possible reason is that the nanoparticles



**Fig. 6** The TEM image of the extracted  $\text{TiO}_2$  from the  $\text{TiO}_2$ -coated cotton fabrics

have aggregated to some extent during the coating process (Li et al. 2017b).

The SEM images in Fig. 5c, d show the surface morphology of the  $\text{TiO}_2$ -coated cotton fabrics after five washing cycles. It can be seen that the nanoparticles are well dispersed on the fiber surface, but some free nanoparticles have been washed out from the surface of the film. This has been further confirmed by the results of ICP-MS, in which the amount of  $\text{TiO}_2$  changed from 0.284 to 0.259 mg/g after five washing cycles. As a whole, the film of nanoparticles has been well preserved after laundering, indicating the excellent durability of the coating. Since the  $\text{TiO}_2$  nanoparticles were synthesized by the hydrolysis of  $(\text{NH}_4)_2\text{TiF}_6$  and  $\text{H}_3\text{BO}_3$  (Ou et al. 2010; Liu et al. 2017), the hydroxyl groups of polydopamine can react with the metal ion to form metal oxide nanoparticles (Li et al. 2016; Ma et al. 2011b). Owing to the chelation between  $\text{TiO}_2$  nanoparticles and polydopamine (Zheng et al. 2012), the  $\text{TiO}_2$  nanoparticles have been well dispersed on the surface of cotton fabrics with excellent affinity.

The EDX spectra of the  $\text{TiO}_2$ -coated cotton fabrics before and after washing are shown in Fig. 7, and before washing there are three detected peaks in the spectrum for carbon (C), oxygen (O) and titanium (Ti) elements with the weight percentages of 8.5, 72.6 and 18.9%, respectively. After five washing cycles, the weight percentage of detected titanium has decreased slightly to 17.2%. The laundering has resulted in a loss of about 9.0% for Ti element, which is consistent with

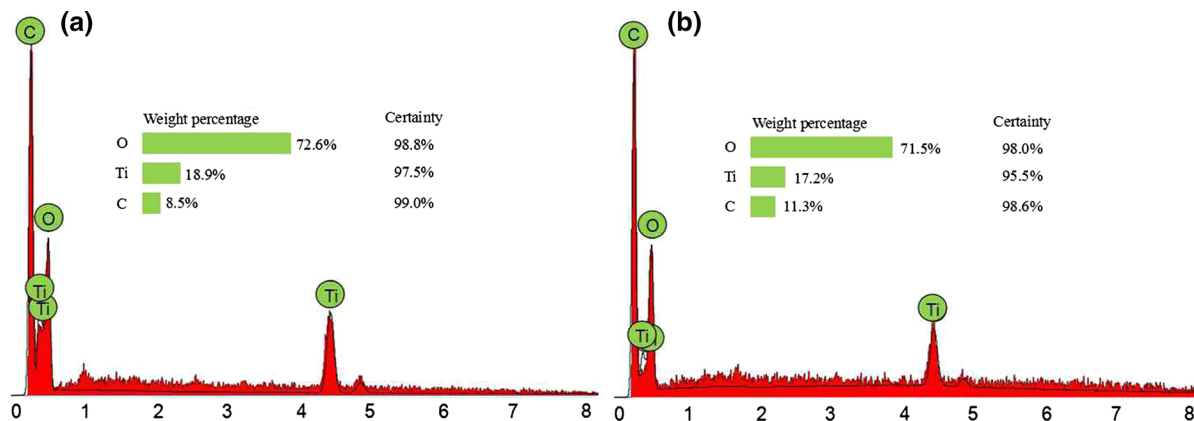
the ICP-MS results. The excellent durability is because that most of the nanoparticles have been adsorbed onto the surface of fabrics by chelation with strong binding force, and only few of the weakly adsorbed nanoparticles have been washed away.

The chemical composition of the  $\text{TiO}_2$ -coated cotton fabrics was determined by XPS, as shown in Fig. 8 the XPS wide scan and  $\text{Ti}2p$  core-level spectra. The XPS wide scan spectrum contains peaks of  $\text{C}1s$  at 285.1 eV,  $\text{O}1s$  at 532.1 eV and  $\text{Ti}2p$  at 458 eV, respectively. The presence of the strong signal of  $\text{Ti}2p$  peak confirms the presence of  $\text{TiO}_2$  nanoparticles. The XPS  $\text{Ti}2p$  spectrum shows the binding energy of  $\text{Ti}2p_{3/2}$  at 459.1 eV and  $\text{Ti}2p_{1/2}$  at 464.7 eV, and the doublet separation energy between these two peaks is 5.6 eV. These results represent the typical characteristics of  $\text{Ti}^{4+}$  from the anatase  $\text{TiO}_2$  (Huang et al. 2017).

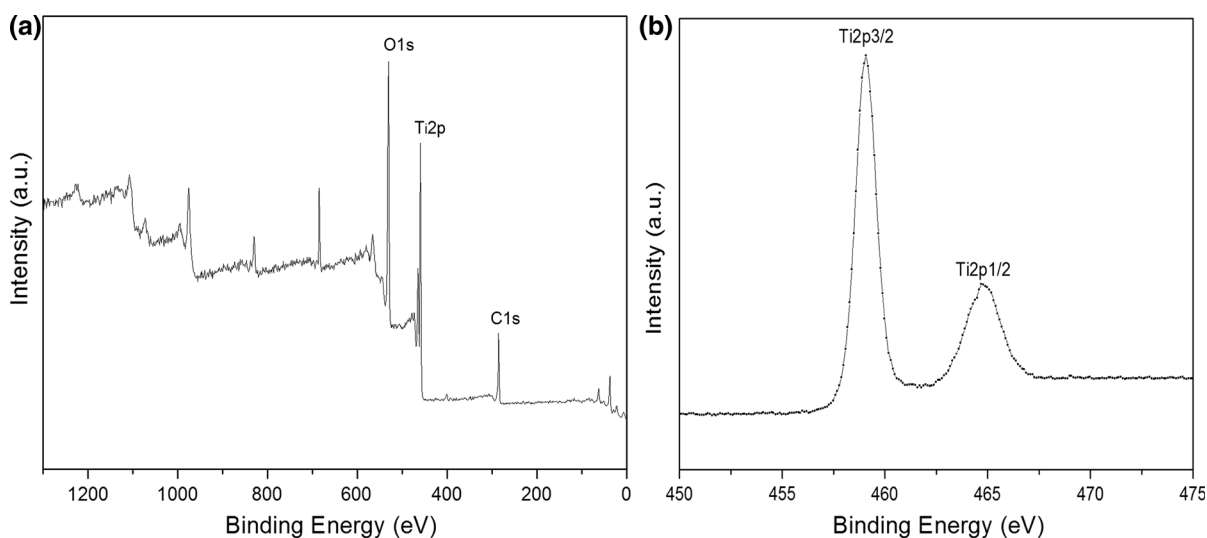
The crystal structure of all the coated cotton fabrics was analyzed by XRD, as presented in Fig. 9. The characteristic peaks of the crystal structure of pristine cotton fabric (Fig. 9a) can be found at  $2\theta = 14.7^\circ$ ,  $16.3^\circ$ ,  $23.1^\circ$  and  $34.4^\circ$ , corresponding to the crystallographic plane (1–10), (110), (200) and (004), respectively (Lu and Hsieh 2010). There is no extra diffraction peak for the spectrum of PDA-coated cotton (Fig. 9b), and this is mainly due to the amorphous structure of polydopamine (Zhang et al. 2012). An extra peak appears at  $25.5^\circ$  as a shoulder peak for the spectrum of the  $\text{TiO}_2$ -coated cotton fabric (Fig. 9c), and this peak is ascribed to the  $\text{TiO}_2$  anatase structure (Sadr and Montazer 2014; Pakdel et al. 2014).

#### Photocatalytic activity

The presence of the anatase crystal structure implies the potential photocatalytic ability of the  $\text{TiO}_2$ -coated cotton fabrics (Luttrell et al. 2014). The photocatalytic activity of the  $\text{TiO}_2$ -coated cotton fabric was evaluated by the degradation of MB under UV irradiation. The wavelength 664 nm was chosen as it is the wavelength for the main UV–Vis absorption peak of MB, and this peak can be easily monitored during the degradation (Yang et al. 2010). The photodegradation of MB solution in presence of pristine cotton fabric after UV illumination for 14 h is shown in Fig. 10a. As expected, the absorbance of residual MB solution at 664 nm changes slightly after UV irradiation,



**Fig. 7** EDX spectra of  $\text{TiO}_2$ -coated cotton fabrics before (a) and after (b) washing



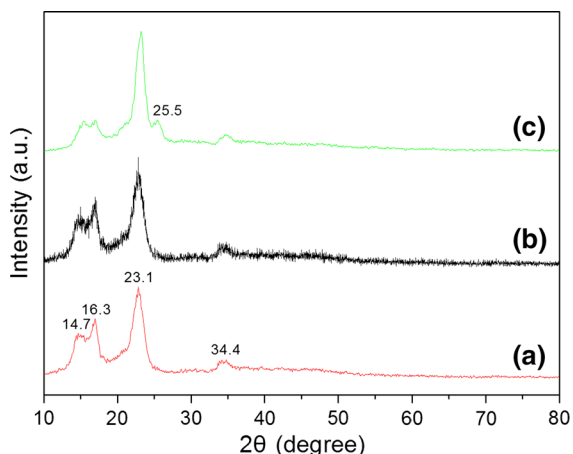
**Fig. 8** XPS wide scan (a) and  $\text{Ti}2p$  (b) spectra of  $\text{TiO}_2$ -coated cotton fabrics

indicating that the pristine cotton fabric exhibits no photocatalytic ability. Figure 10b shows the photodegradation of MB on the  $\text{TiO}_2$ -coated cotton fabrics, with the inset shows the absorbance of residual MB solution as a function of UV illumination time. The absorbance of residual MB solution at 664 nm decreases evidently with the increase of the UV illumination time, and about 90% of MB solutions have been degraded after of UV illumination for 14 h. The reason for this excellent photocatalytic activity is attributed to the presence of  $\text{TiO}_2$  nanoparticles on the coated cotton fabrics.

#### Thermal stability

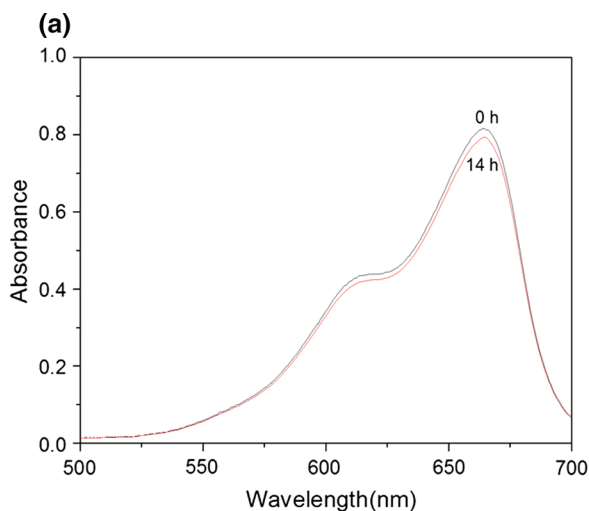
The thermal degradation properties of the coated cotton fabrics were measured by thermogravimetric analysis, as shown the TG and DTG curves in Fig. 11. The onset decomposition temperature for the pristine cotton fabric is 333.7 °C and the weight residue at 700 °C is 3.1%. The DTG curve also reveals that the temperature of the maximum degradation rate is 348.5 °C. After the coating of PDA and  $\text{TiO}_2$  nanoparticles, the onset decomposition temperature decreased to 332.8 and 307.6 °C, respectively. At the same time, the temperature of the maximum degradation rate decreased to 345.1 and 332.6 °C, respectively. The decrease of these temperature values is due





**Fig. 9** XRD patterns of pristine cotton fabrics (a), PDA-coated cotton fabrics (b) and TiO<sub>2</sub>-coated cotton fabrics (c)

to the deteriorated properties of the cotton fabric by the usage of H<sub>3</sub>BO<sub>3</sub> as the reducing agent. The weight residue at 700 °C is 4.5 and 18.5% for PDA- and TiO<sub>2</sub>-coated cotton fabrics, respectively. Thus, the weight percentage of TiO<sub>2</sub> deposited on cotton fabric (before washing) can be calculated as 14%. It can be seen that the polydopamine coating has little effect on the thermal stability of the fabric.

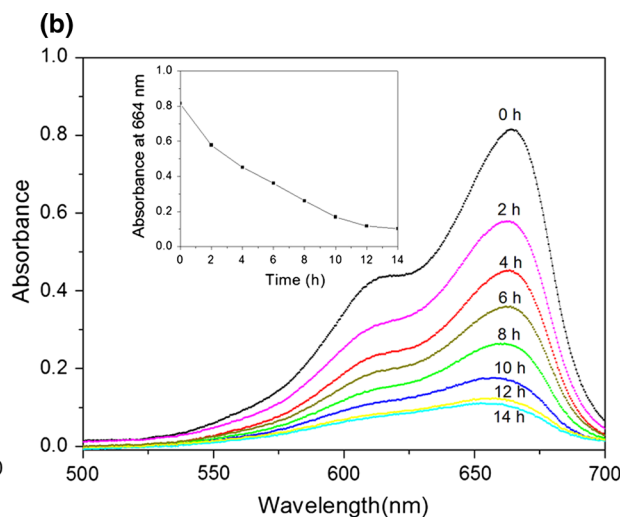


**Fig. 10** UV–Vis absorption spectra of photodegradation of MB solution in presence of pristine cotton fabrics after UV illumination for 14 h (a) and on TiO<sub>2</sub>-coated cotton fabrics at

## UV protection property

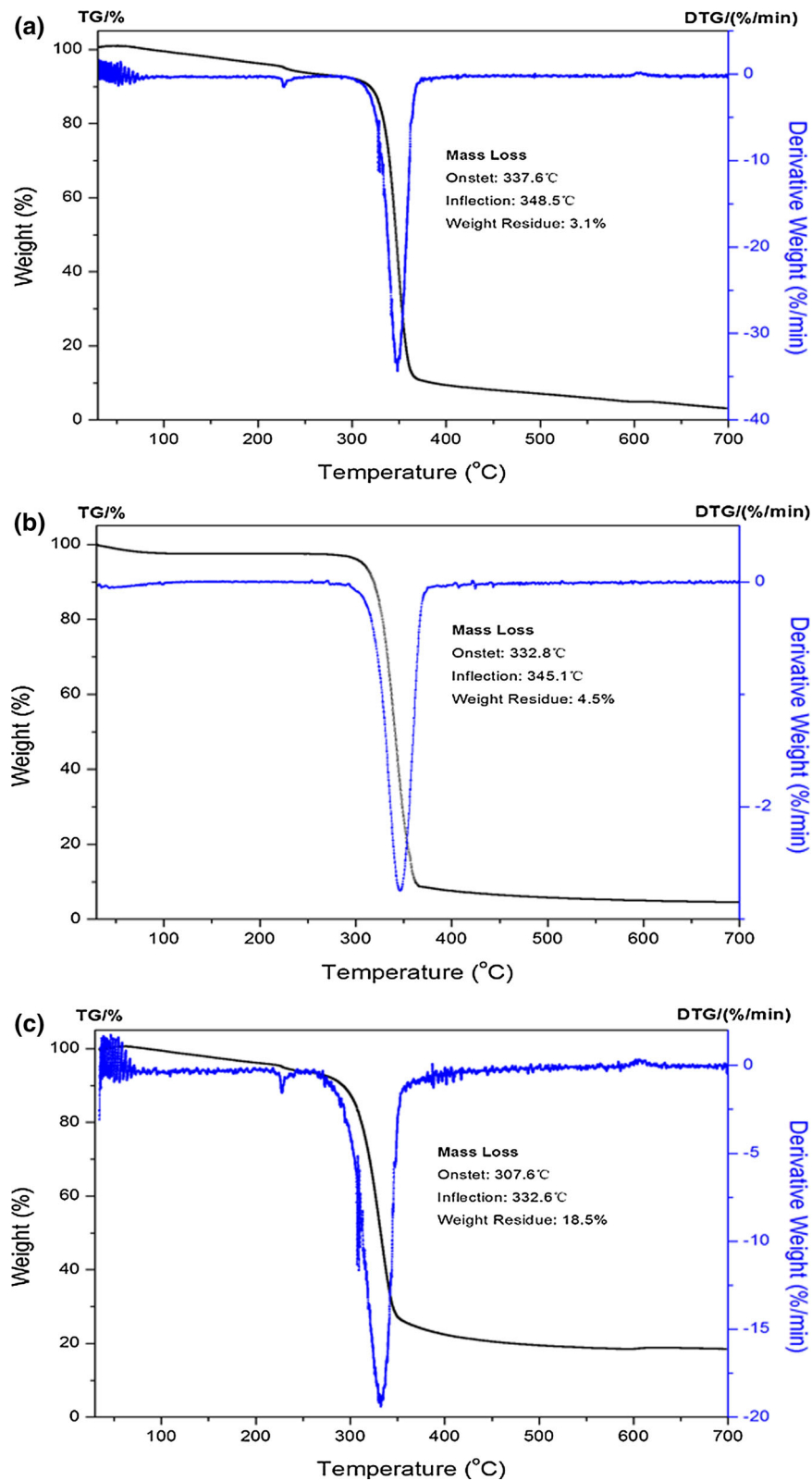
The UV protective property of the TiO<sub>2</sub>-coated cotton fabrics was evaluated by the ultraviolet protection factor (UPF), as shown in Fig. 12. The pristine cotton fabrics show a low UPF of 6.4 due to their poor inherent UV protection property. After the coating of TiO<sub>2</sub> nanoparticles, the UPF value increases rapidly to 127.2, indicating that coating of TiO<sub>2</sub> nanoparticles is very effective in UV protection for cotton fabrics.

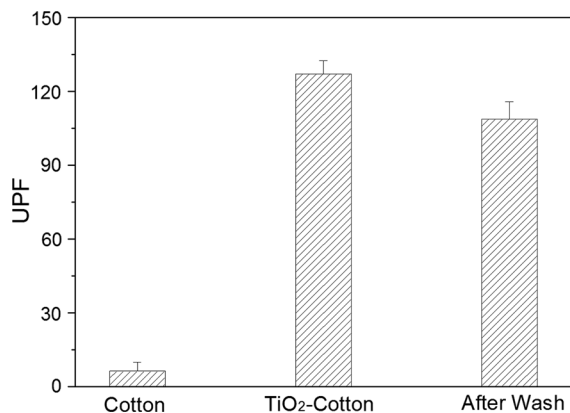
The coating showed excellent durability against washing in terms of UV protection. After five washing cycles, the UPF value decreases to 108.8 with a drop of less than 20. The calculated amount of TiO<sub>2</sub> nanoparticles on fabrics dropped from 0.284 to 0.259 mg/g after five washing cycles, resulting in the drop of UPF value. A UPF of 40 – 50 + yielded by clothing exhibits excellent protection against UV radiation (Gorjanc et al. 2014), so that excellent UV protective property has been maintained for the TiO<sub>2</sub>-coated cotton fabrics. The durability is attributed to the PDA template on the surface of cotton fabrics that serves as an adhesion layer. These results imply that the TiO<sub>2</sub>-coated cotton fabrics exhibit excellent and durable UV protection property.



different UV irradiation time (b) (inset: the absorbance of residual MB solution at 664 nm as a function of UV irradiation time)

**Fig. 11** TG and DTG curves of pristine cotton fabrics (a), PDA-coated cotton fabrics and TiO<sub>2</sub>-coated cotton fabrics (b)





**Fig. 12** UPF values of pristine cotton fabrics and TiO<sub>2</sub>-coated cotton fabrics (before and after washing)

## Conclusions

An innovative method was developed to immobilize TiO<sub>2</sub> nanoparticles on cotton fabrics by in situ reduction of TiO<sub>2</sub> nanoparticles on a polydopamine template. The polydopamine film has been coated on the surface of cotton fabrics, as confirmed by SEM–EDX, FT-IR and XPS. TiO<sub>2</sub> nanoparticles were successfully deposited on the PDA-coated cotton fabrics as evidenced by SEM–EDX, XPS and XRD. The TG and DTG results suggested that the thermal stability of the cotton fabrics has not been affected evidently after the deposition of TiO<sub>2</sub> nanoparticles. The TiO<sub>2</sub>-coated cotton fabrics exhibited excellent photocatalytic activity. The UPF value of the TiO<sub>2</sub>-coated cotton fabric was 127.2, and it was maintained at 108.8 even after five washing cycles. These results implied that the TiO<sub>2</sub>-coated cotton fabrics exhibited excellent photocatalytic activity and durable UV protection capacity.

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