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ORIGINAL RESEARCH

# A reactive fluorine-free, efficient superhydrophobic and flame-retardant finishing agent for cotton fabrics

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**Abstract** A novel finishing agent, N,N-dimethyl-octadecyl phosphate acrylamide (NDOPA), was synthesized with octadecanol, phosphorous acid, formaldehyde and acrylamide, and applied to cotton fabrics by ultraviolet curing to endow cotton fabrics with superhydrophobicity and flame retardancy. Results showed that NDOPA could be grafted onto cotton fabrics through C–O–C covalent bonds during the ultraviolet curing process. The contact angle of cotton fabric treated with the 10% NDOPA agent could reach 157.5°. The treated cotton fabrics exhibited clear flame retardancy. The treated cotton retained strong hydrophobicity and flame retardancy after 30 laundering cycles, indicating that the treated cotton fabrics had excellent durability. Thermogravimetric analysis revealed that the treated samples had flame-retardant properties. The results of Fourier-transform infrared spectroscopy, energy dispersive X-ray analysis and scanning electron micrography showed that NDOPA was successfully grafted onto the cotton fabrics; X-ray diffraction analysis indicated that the crystallized structure of treated cotton fibers nearly did not change.

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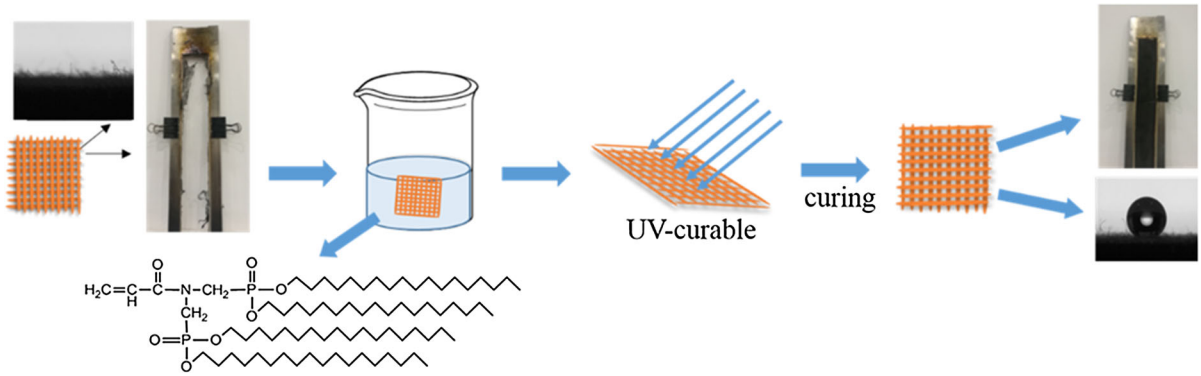
**Graphical abstract**




**Keywords** Superhydrophpbicity · Flame retardant · Cotton fabrics · Efficient · Durability

## Introduction

Cotton is widely used in the textile industry as a natural material because of its superior properties such as comfortableness, excellent mechanical properties, and breathability (Lin et al. 2018; Nguyen et al. 2013). However, cotton fabrics are flammable and hydrophilic, limiting their use in some areas (LTD 1934; Xue et al. 2012).

Hydrophobic textiles, especially superhydrophobic textiles, have been used in many industrial fields due to their unique properties, such as in stain-resistant fabrics for medical personnel, outdoor sportswear, and others (Sun et al. 2017). Surfaces with a water contact angle (WCA) higher than 150° and a sliding angle less than 10°, have been found to exhibit non-wetting and superhydrophobic surface characteristics (Lee et al. 2011). The structure (roughness) and chemical composition of a surface determines whether it possesses hydrophobic properties (Maciejewski et al. 2015). Therefore, a superhydrophobic cotton surface is commonly produced through two steps: (1) creating rough structures on cotton substrate with a nanoscale material; and (2) chemically modifying the substrate surface with low-surface-energy materials. Fluorinated compounds are widely used in the preparation of hydrophobic surfaces due to their low surface energy (Mckeen 2006; Wei et al. 2016). However, fluorine is potentially harmful to organisms and the environment (Keil et al. 2008). Therefore, preparing hydrophobic surface with fluorinated compounds have been prohibited by many countries (Tragoonwichian et al. 2011). A combination of micro- and nanoscale particles with low-surface-energy materials can be used to prepare superhydrophobic surfaces, but coating nanoscale particles and low-surface-energy material on cotton fabrics can make the durability of treated samples poor because the coated material does not combine with cellulose fibers with covalent bonds. Recent studies have identified self-healing properties for superhydrophobic surfaces as a mean of addressing durability (Li et al. 2010; Huang et al. 2018).

High flammability is a major drawback of cotton fabrics. Generally, flame-retardant cotton fabrics can be obtained by finishing flame retardants (Li et al. 2017). Most flame retardants are classified into halogen-based, inorganic metallic oxide, organophosphorus-based and nitrogen-based flame retardants (Chang et al. 2014). Halogen-based flame retardants have been banned due to toxicity. Durable flame-retardant cotton fabrics prepared by phosphorus-based flame retardants such as Proban and Pyrovatex CP release formaldehyde during usage. Scholars should seek novelty phosphorus-based reactive flame retardants without formaldehyde to produce durable flame-retardant cotton fabrics. Recently, bio-based flame retardants have been reported, such as DNA and phytic acid (Annalisa et al. 2016; Feng et al. 2017). Some new nanomaterials have also been used as flame retardants, such as graphene (Jing et al. 2018), nano-silicon dioxide (Qian et al. 2018), and carbon nanotubes (Pan et al. 2015). Emerging materials have also been added onto cotton fabrics through physical

coatings. Although these methods are easy to operate, flame-retardant cotton fabrics obtained using them are not especially durable.

Although many methods can impart cotton fabrics flame retardancy or hydrophobicity, it is rare to achieve both functions simultaneously. However, cotton fabrics with flame-retardant and superhydrophobic properties are in demand in many areas. Thirumalaisamy Suryaprabha et al. prepared superhydrophobic and flame-retardant cotton fabrics with copper, 2,5-dimercapto-1,3,4-thiadiazole, and silver. The coated samples had excellent superhydrophobicity and good rubbing fastness but poor washing durability (Suryaprabha and Sethuraman 2018). Zhang et al. (2017) prepared flame-retardant and hydrophobic cotton fabrics using self-assembly and in situ sol–gel technology, but washing durability failed to meet requirements for practical applications. Lin et al. (2019) fabricated a superhydrophobic and flame-retardant coatings on cotton fabric via sol–gel reaction. The cotton fabrics were imparted superhydrophobic properties and excellent flame retardancy. But the durability of treated cotton fabric was not discussed. Flame retardants and low-surface-energy coatings have not yet been combined with cellulose through covalent bonds.

In this work, a multifunctional fluorine-free hydrophobic flame-retardant finishing agent (NDOPA) was synthesized. The finishing agent with long carbon chain and organic phosphorus group, could endow the finishing agent with low surface energy and flame retardance. Then, the cotton fabrics were finished with this reactive flame retardant by the ultraviolet curing method. Ultraviolet curing could induce the bulk polymerization of NDOPA and the graft reaction between NDOPA and cotton fabrics. The finished cotton fabrics showed excellent hydrophobicity and certain flame retardancy. The finished samples had excellent durability.

## Experimental

### Materials

The 100% cotton fabrics (100% woven cotton fabrics, 115 g/m$^2$, 570 × 340) were supplied by Chaotianmen market in Chongqing, China and desized in a 10 g L$^{-1}$ NaOH solution at 98 °C for 1 h. Phosphoric acid (> 99%), acrylamide (> 99%), and formaldehyde (37–40%) were obtained from Chengdu Kelong Chemical Reagent Co., Ltd (Chengdu, China). Octadecanol was provided by Macklin Biochemical Co., Ltd (Shanghai, China). All chemicals were of reagent grade and used without further purification.

### Synthesis and characterization of finishing agent

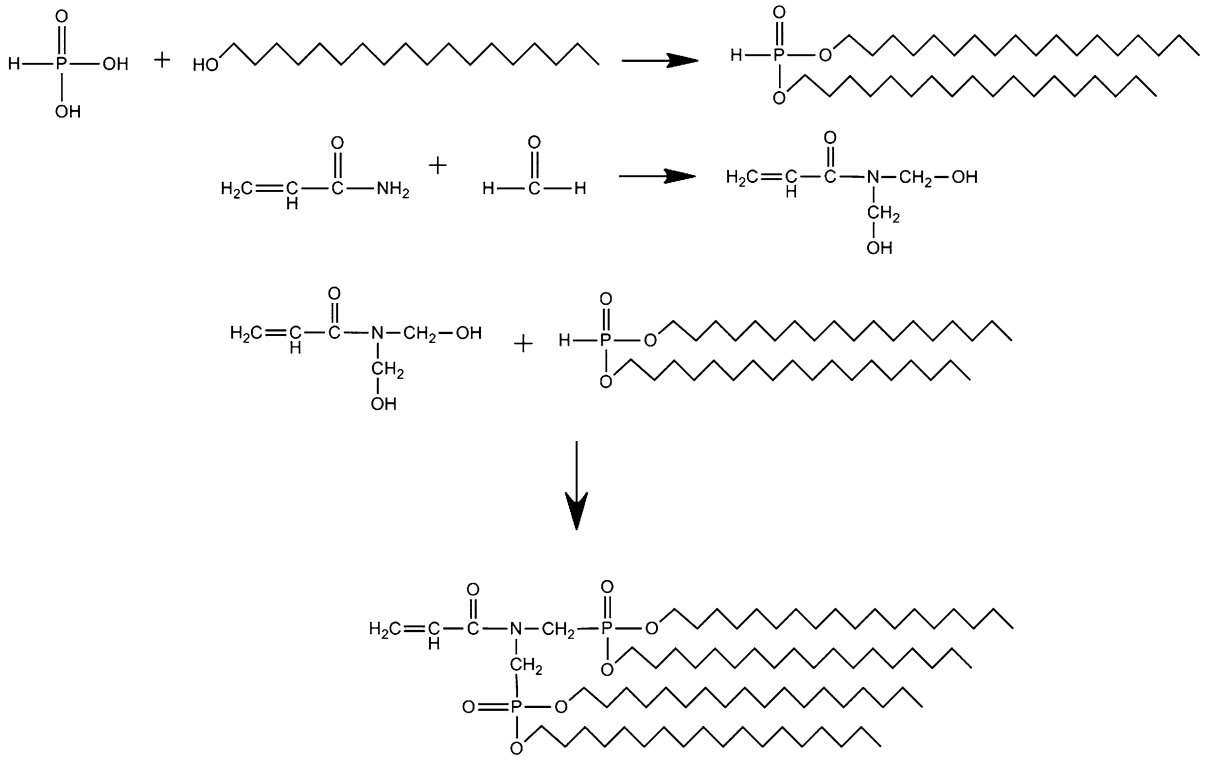
Octadecanol (18.43 g, 0.068 mol) and phosphoric acid (2.788 g, 0.034 mol) were added to a round-bottomed flask (100 mL), which was set in an oil bath pot with magnetic stirrers. The mixture was heated to 130 °C and reacted for 1.4 h. The white solid particles gradually become colorless and transparent liquid during the reaction process. Then the octadecyl phosphate ester has been produced. Dissolved acrylamide (1.208 g, 0.017 mol) in a round-bottomed flask (250 mL) containing 10 mL of distilled water. Then, formaldehyde solution (2.54 mL) was added to the solution and stirred for 2 min to synthesize N,N-dimethylol acrylamide. Next, N,N-dimethylol acrylamide solution was poured into the octadecyl phosphate ester solution and then stirred for 2 h at 70 °C. Then N,N-dimethyl-octadecyl phosphate acrylamide (NDOPA) has been obtained. Appropriate amount of absolute ethanol was added to the NDOPA solution to prepare various concentration hydrophobic flame-retardant solution at 60 °C. Scheme 1 shows the synthesis reactions.

### Flame-retardant finishing of cotton fabrics

Cotton fabrics were dipped into NDOPA with a mass concentration of 6%, 8%, and 10% at 70 °C for 20 min. The bath ratio was 1:20 (w/v). Then, the cotton fabrics were set under an ultraviolet ray lamp (2000 W). The distance between the ultraviolet lamp and cotton fabrics was approximately 36 cm, and the irradiation time was 3 min. Subsequently, the fabrics were cured at 130 °C for 6 min. Fabrics were washed under running water for 2 min and dried at 60 °C for 1 h (Scheme 2). NDOPA was polymerized and grafted onto the cotton fabrics through C–O–C bonds via ultraviolet ray irradiation.

### Characterization

Nuclear magnetic resonance ($^1$H NMR, $^{13}$C NMR, and $^{31}$P NMR) spectra of NDOPA were measured on a Bruker AV III 600 spectrometer (USA) with TMS as the



**Scheme 1** Synthesis of NDOPA

internal standard using $CDCl_3$. The WCAs of cotton samples were measured on an OCA15EC instrument (Dataphysics, Germany) at room temperature according to the standard. The water drop volume was 3 μL.

The surface morphology images of untreated samples, treated samples, washed samples and the char residues of treated samples were taken by a scanning electron microscope (SEM; Hitachi S-4800, Netherlands) at an accelerating voltage of 10 kV. The working distance was 6–8 mm. Energy dispersive X-ray analysis (EDX) was used to analyze elements on the surfaces of samples.

X-ray diffraction (XRD) spectra of the control and treated cotton were recorded on a Rigaku XD-3 wide-angle diffractometer (Beijing Purkinje General Instrument Co. Ltd., Beijing, China) at 36 kV and 20 mA. The diffractogram scattering angle ranged from 5° to 50° with a step size of 0.02° (λ = 0.154 nm).

The Fourier-transform infrared (FT-IR) spectra of NDOPA, the cotton dipped in a 10% mass concentration of NDOPA, untreated cotton and the cotton treated with NDOPA were acquired using a spectrum GX infrared spectrometer (PE. Co., USA) over the wavenumber range of 4000–500 $cm^{-1}$ using KBr pellets. The resolution was 2.0 $cm^{-1}$.

The flammability of samples was explored using a YG815B vertical fabric FR tester (Nantong Sansi Electromechanical Science & Technology Co. Ltd., China) in accordance with the ASTM D6413-99 standard.

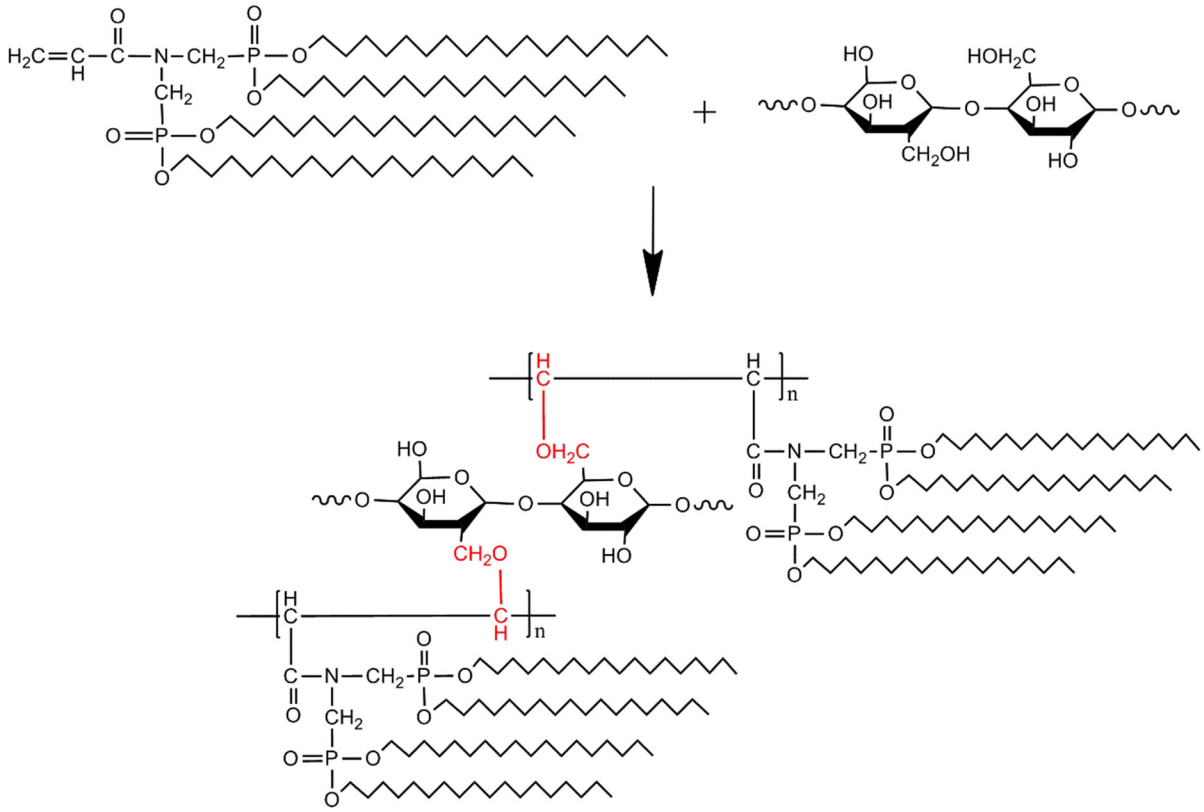
Thermal stability tests of cotton fabrics were carried out on a Pyris 1 thermogravimetic analyzer (Perkin-Elmer, USA) from 40 to 800 °C with a heating rate of 20 K $min^{-1}$ in nitrogen.

The washing durability of treated cotton fabrics was analyzed based on the AATCC 61-2006 standard (Roaches Co., England). In this method, samples were washed at 49 °C in a 0.15 wt % sodium dodecyl benzene sulfonate solution using a soaping fastness tester.

## Results and discussion

### Synthesis and characterization of NDOPA

The chemical structure of NDOPA was characterized by $^{1}H$ NMR, $^{13}C$ NMR, and $^{31}P$ NMR. Figure 1



**Scheme 2** Polymerization of NDOPA on cotton fabric

presents the $^{1}$H NMR spectrum of NDOPA. The peaks at 6.14 ppm and 5.74 ppm belong to hydrogen atoms (a, b, c) in $CH_2$=CH–. The peak at 3.64 ppm is attributed to hydrogen atoms (d) in N-$CH_2$. Peaks at 4.02, 1.68, and 1.57 ppm correspond to hydrogen atoms (e, f, g) in O–$CH_2$–$CH_2$–$CH_2$. Peaks at 1.26–1.29 ppm are assigned to hydrogen atoms (h–u) in the long carbon chain of NDOPA. The peak at 0.88 ppm corresponds to hydrogen atoms (v) in –$CH_3$.

The $^{13}$C NMR spectrum is shown in Fig. 2. Peaks at 104.05 and 116.85 ppm are attributed to carbon atoms (C1, C2) in $CH_2$=CH–. The peak at 128.69 ppm belongs to carbon atoms (C3) in –C(=O)–N. Peaks at 63, 59, and 31.92 ppm belong to the carbon atom (C4) in N–$CH_2$–P(=O), C5 in O–$CH_2$– and C6, which connects to C5. The peaks around 30.00–29.10 ppm correspond to carbon atoms (C7–C19) in the long carbon atoms; the peaks at 25.84, 22.67, and 14.06 ppm belong to C20–C22 in the long carbon atoms.

Figure 3 presents the $^{31}$P NMR spectrum of NDOPA. The peak at about 8.20 ppm is assigned to the phosphorus atoms of NDOPA.

## Hydrophobicity of samples

The surface wettability of cotton fabrics is displayed in Fig. 4. As shown in Fig. 4a, when water droplets dripped onto the surface of normal cotton, the fabric surface was wetted rapidly due to hydrophilic hydroxyl groups on cotton cellulose, and the contact angle was 0° after 10 s. Cotton fabrics after treatment showed excellent hydrophobic property (Fig. 4b–f). The WCAs of cotton fabrics treated with mass concentrations of the finishing agent solution at 6%, 8%, and 10% were 147.4°, 151.0°, and 157.5°, respectively. The WCAs of samples treated with 8% and 10% NDOPA were higher than the superhydrophobic standard angle (150°). As the concentration of the finishing agent increased, the contact angles of the treated cotton fabric gradually increased as well.

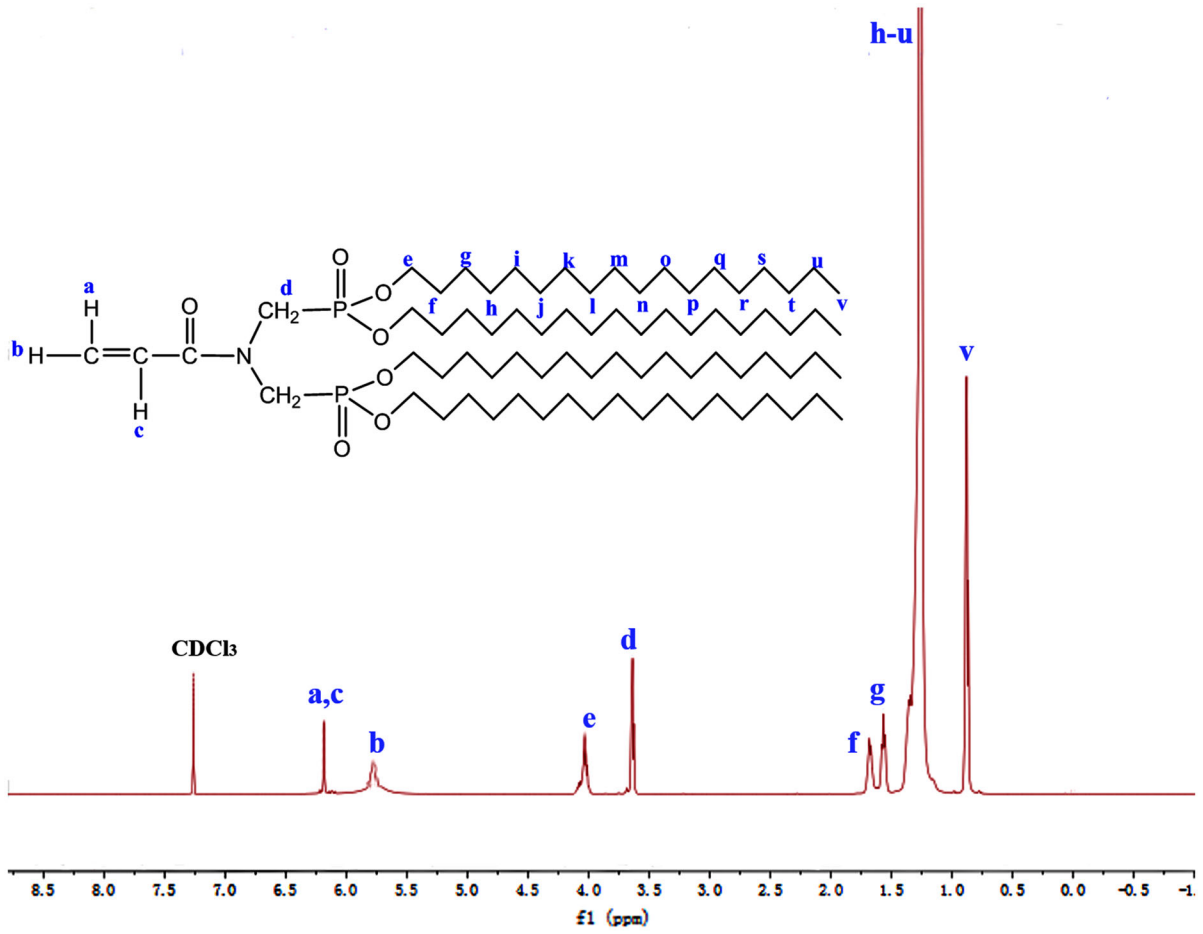



**Fig. 1** $^{1}$H NMR spectrum of NDOPA

These results showed that the hydrophobic flame-retardant finishing agent can impart cotton fabrics with excellent hydrophobic properties.

## Vertical flammability tests

Vertical flammability tests were carried out on control samples and samples treated with 6%, 8%, and 10% of the finishing agent to evaluate their flame-retardant properties. Figure 5 and Table 1 present the test results. All samples burnt completely. However, the treated cotton fabrics retained a relatively complete carbon frame after combustion, whereas the untreated cotton fabric retained only a small proportion of carbon frame. The after-flame time and after-glow time of untreated fabrics were 7 s and 9 s. The after-flame time of fabrics treated with 6%, 8%, and 10% of the finishing agent were 4 s, 2 s, and 2 s, respectively. All treated cotton fabrics had no smoldering after extinction. The control cotton fabric burned violently, whereas the flame on the treated cotton fabric was slight. Treated cotton fabrics released fewer flammable volatiles in the combustion process and retained a complete char form and fabrics structure due to the NDOPA containing the flame-retardant element (P element) was grafted onto the cotton fabrics through C–O–C bond. Therefore, cotton fabrics treated with the finishing agent had certain flame-retardant properties.

The LOI results of the treated samples and the treated samples after 30 LCs are shown in Fig. 6. The LOI values of 6%, 8% and 10%-NDOPA- treated samples were 19.8%, 20.5% and 21%, respectively. After 30 LCs, the LOI values of 6%, 8% and 10% samples maintained 19.0%, 19.6% and 20.1%, which were all higher than that of the original cotton (17.8%).

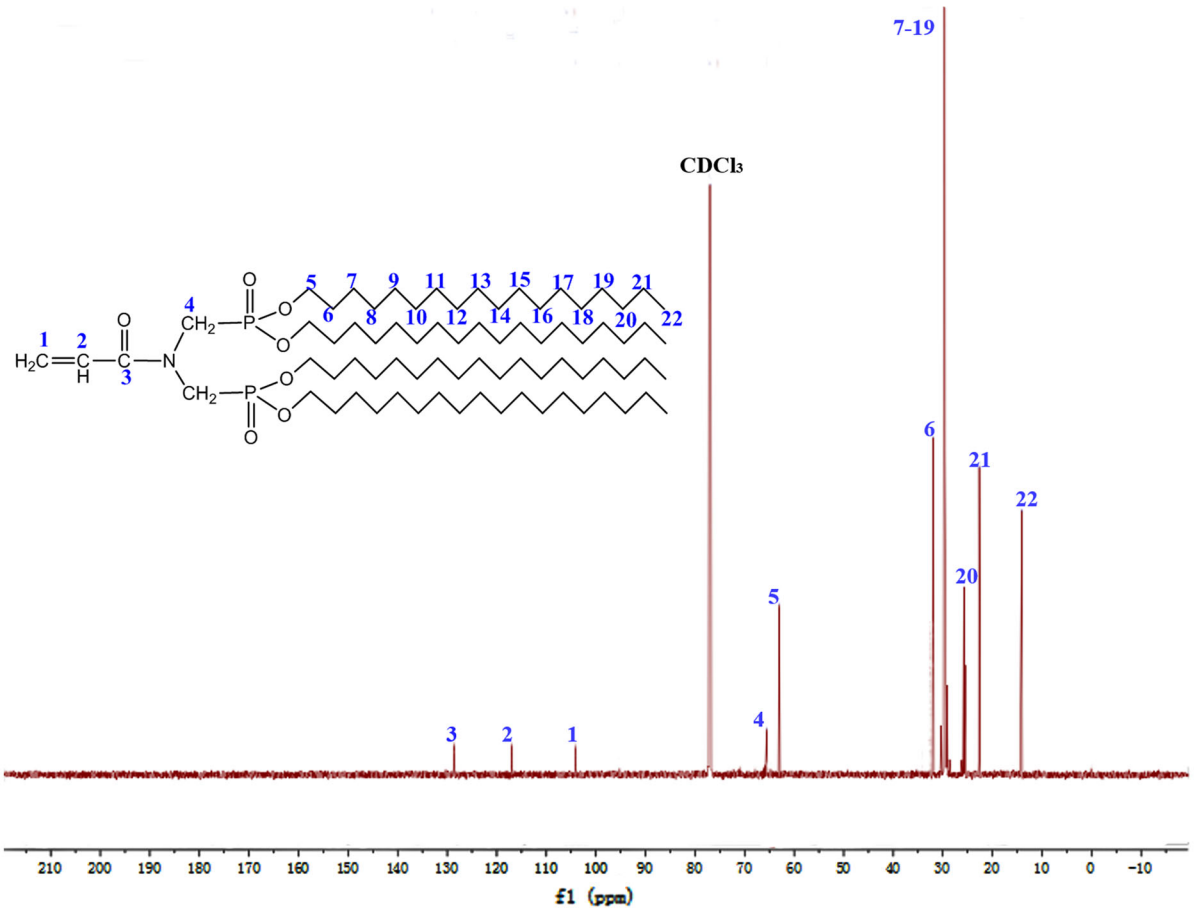



**Fig. 2** $^{13}$C NMR spectrum of NDOPA

This indicated that the treated samples had certain flame retardancy and semi-durability.

## TGA

The thermal processes of samples were examined by thermogravimetric analysis under a nitrogen atmosphere (Fig. 7a). The control and cotton fabrics treated with 10% NDOPA proceeded through three stages of weight loss. When the temperature was below 100 °C, weight loss was attributed to the dehydration process. For the control cotton fibers, the initial decomposition temperature which the weight of the sample lost 5% ($T_5$) was 281 °C. Thermal decomposition of cellulose mainly occurred at temperatures ranging from 342.9 to 410.7 °C, with a residue of 15.5%. In the third stage of thermal decomposition (410.7–700 °C), the weight-loss rate of cotton fabrics slowed, and at 700 °C, only 5.5% residue remained. However, the initial decomposition temperature ($T_5$) of treated cotton fabrics was 239.4 °C. The severe temperature range of treated cotton fabrics for thermal weight loss was 288.1–349.3 °C, much lower than that of the control cotton. The residue of treated cotton fabrics at 700 °C was 20.9%. There was a great reduction in the decomposition of treated cotton; phosphorus-containing components in finishing agents turned into phosphoric acid, which has good dehydration properties during the combustion process and can accelerate cellulose dehydration and carbonization (Alongi et al. 2012a).

The TG curves of the control sample and the treated sample under air are shown in Fig. 7b. The weight of the samples belowed 100 °C decreased slightly because of the evaporation of water. For the control sample, the weight loss including water evaporation

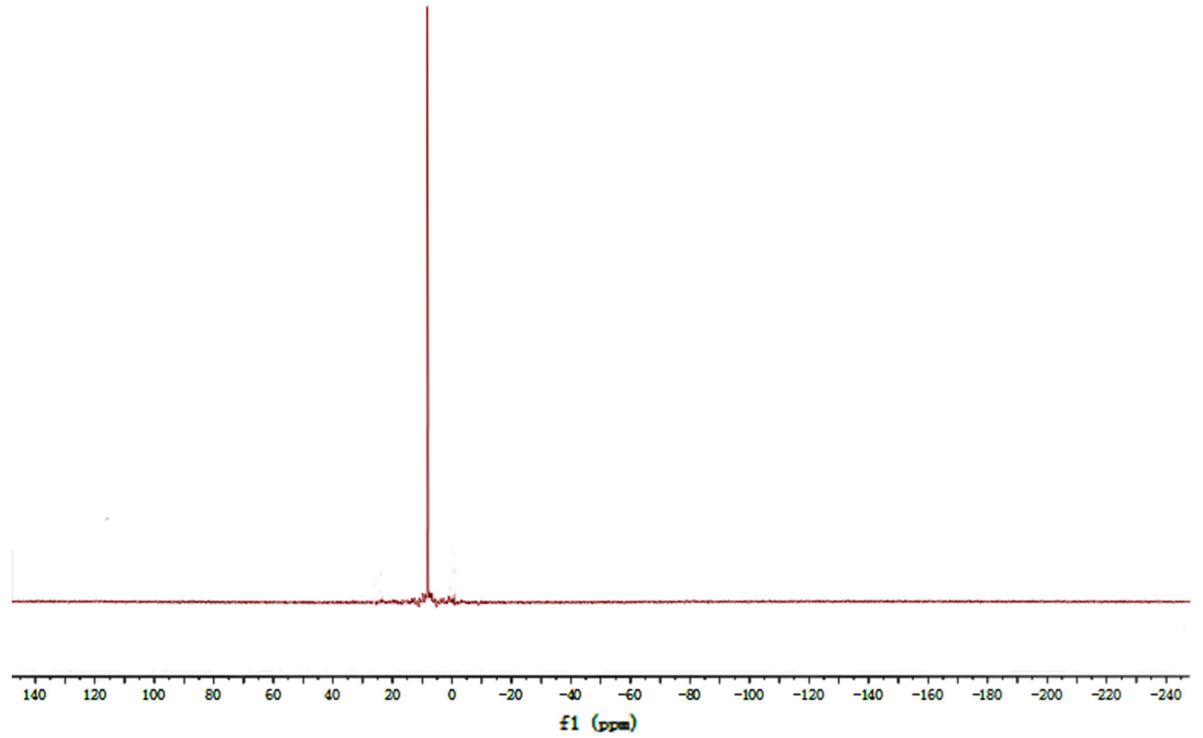



**Fig. 3** $^{31}$P NMR spectrum of NDOPA

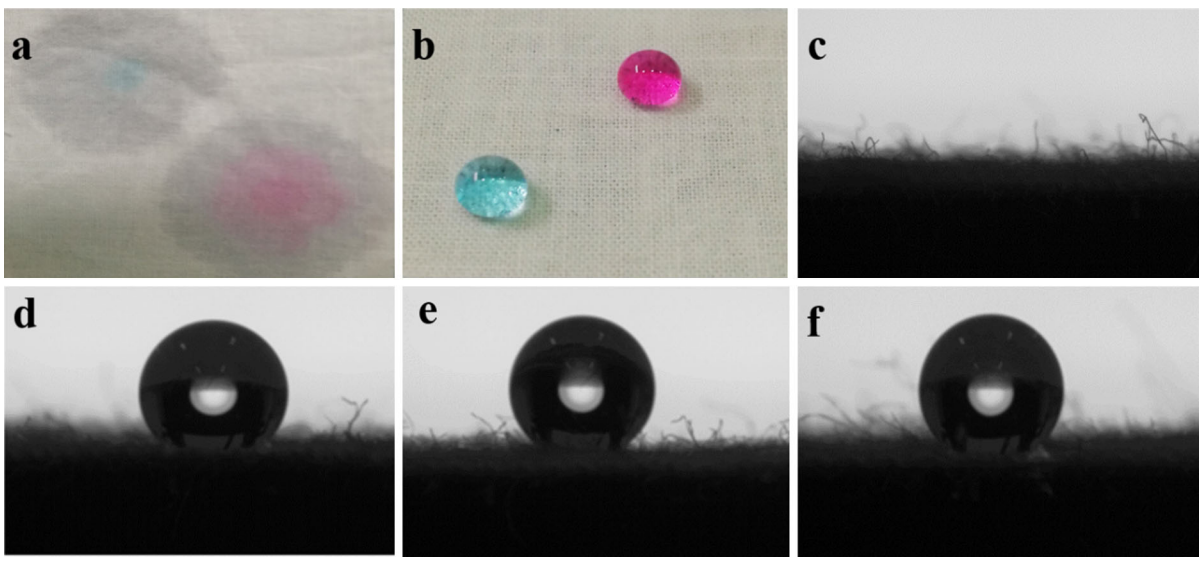



**Fig. 4** Photographs of water droplets on **a** pristine cotton fabric, **b** cotton fabric treated with 10% NDOPA, and water contact angles of the **c** control sample and samples treated with **d**–**f** 6%, 8%, and 10% NDOPA

can be divided into four stages. Its initial decomposition temperature ($T_5$) was 231.5 °C. The main thermal decomposition of the control sample occurred in the range of 328.0–397.3 °C. The weight of control sample lost 75.1% at 397.3 °C. In this stage, the volatiles and aliphatic char (char I) formed due to the

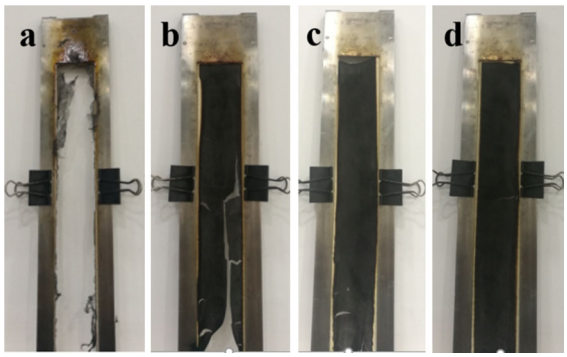



**Fig. 5** Vertical flammability test results of **a** control sample and samples treated with **b–d** 6%, 8%, and 10% NDOPA

dehydration and depolymerisation of cellulose (Horrocks 2013). The third stage was in the range of 397.3–542.8 °C. The weight of control sample decreased continuously and only 1.19% of the weight remained at 542.8 °C. The aliphatic char (char I) transformed into aromatic char (char II) in this stage

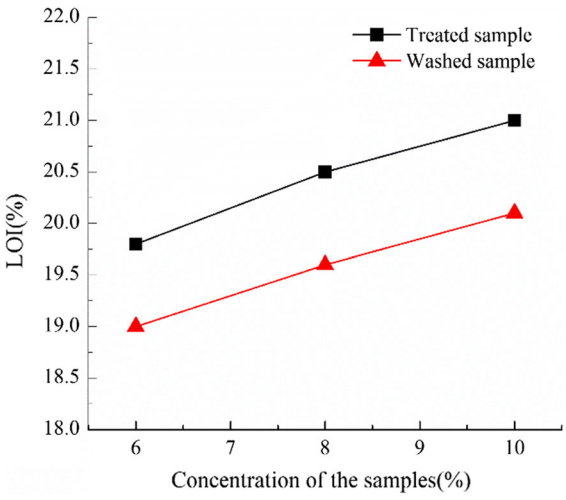



**Fig. 6** LOI values of the treated samples and washed samples

(Alongi et al. 2014). The last stage was in the range of 542.8–700 °C. The aromatic carbon decomposed into ethylene and the residue was 0.29% at 700 °C (Alongi and Malucelli 2015). As for the treated sample, the initial decomposition temperature was 173.0 °C and the main thermal decomposition occurred in the range of 211.7–285 °C. The decomposition temperature of the treated cotton is lowered compared with that of the control cotton because the flame retardant could decompose easily to generate phosphoric acid which would promote the decomposition of cellulose and the formation of carbon. The residue of treated sample at 550 °C was 5.4%, which was much higher than that of control sample. This implied that NDOPA changed the cellulose decomposition pathway, and the cellulose generated more char instead of volatiles gases.

## Surface morphology of cotton fabrics

The surface morphologies of the pristine cotton fibers, treated fibers, washed fibers and burned fibers treated with NDOPA are shown in Fig. 8. The treated and burned samples were treated with 10% mass concentration of NDOPA. Untreated fibers showed a flat ribbon structure with a smooth, clean surface (Fig. 8a–c). The surfaces of treated fibers became rough because the NDOPA molecule was a hydrophobic molecule, making it difficult for NDOPA molecules to enter the hydrophilic inner space. The NDOPA molecules could also polymerize to form macromolecules, which assembled to make the fiber surface rough (Fig. 8d–f). Therefore, hydrophobic flame-retardant finishing could fabricate a rough surface. The morphologies of treated samples after 30 LCs are shown in Fig. 8g–i. The NDOPA-treated samples could maintain rough surfaces which was advantageous to maintain the hydrophobicity of the samples after 30 LCs. SEM images of the burned fibers treated with NDOPA are displayed in Fig. 8j–l. The burned cotton fibers maintained the original structure and complete carbon.

**Table 1** Vertical flammability of control and finished samples

| Samples | After-flame time (s) | After-glow time (s) | Char length (mm) |
|---|---|---|---|
| 0 | 7 | 9 | 0 |
| 6% | 4 | 0 | 350 |
| 8% | 2 | 0 | 350 |
| 10% | 2 | 0 | 350 |

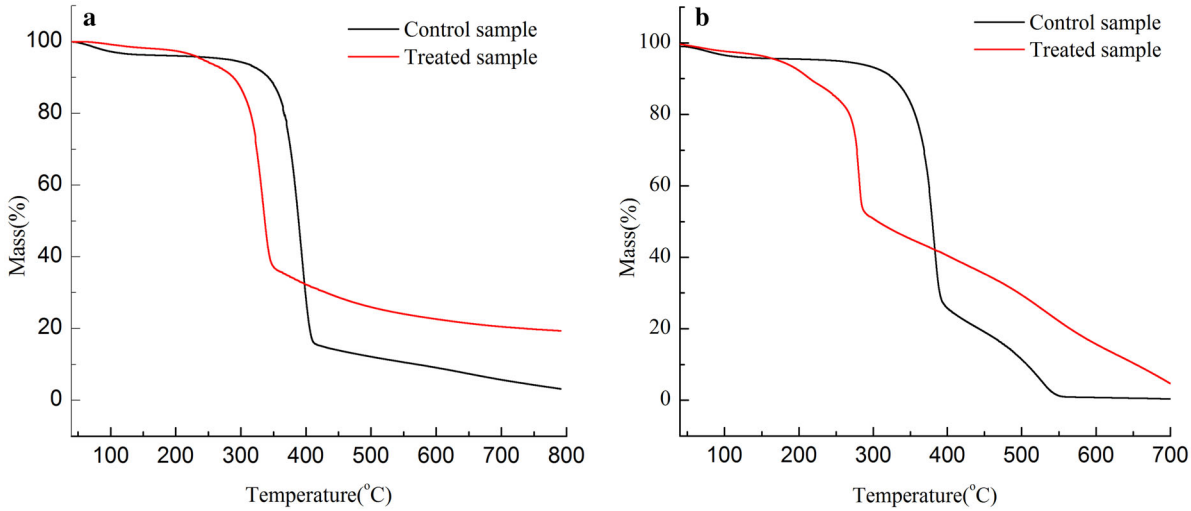



**Fig. 7** Thermogravimetric curves of the control and treated samples under **a** $N_2$ and **b** air

EDX

EDX results of the sample treated with 10% NDOPA and after 30 LCs are exhibited in Fig. 9a–c and Fig. 9d–f, respectively. The C, O, and P elements were distributed on the treated cotton fabrics. As shown in Table 2, the mass percentage concentrations of C on the treated sample was higher than on the control sample due to the long carbon chain in the NDOPA. These results revealed that NDOPA was on the cotton fibers. After 30 LCs, the treated cotton fibers still had a proportion of the P element, implying that the treated cotton had good durability.

FTIR

FTIR spectra of NDOPA, the control sample, sample dipped in a 10% mass concentration of NDOPA and sample treated with a 10% mass concentration of NDOPA are shown in Fig. 10. The absorption peaks of samples within 3500–3000 and 3000–2800 $cm^{-1}$ were assigned to hydrogen-bonded –OH stretching and C–H stretching (Rosace et al. 2017; Colleoni et al. 2013; Alongi et al. 2012b). Strong peaks at 2917 and 2850 $cm^{-1}$ were attributed to the stretching vibration of C–H of the long carbon chain grafted onto cotton. The peaks at 1638 $cm^{-1}$ which belongs to C=C groups in the sample dipped in a 10% mass concentration of NDOPA decreased obviously after the polymerization under ultraviolet ray irradiation (Shanti et al. 2016). The presence of the finishing agent on cotton fabrics was confirmed by peaks at 1467 and 1238 $cm^{-1}$, attributed to the stretching of C=O groups and P=O groups (Xing et al. 2011; Cheema et al. 2013). The peaks at 1111 $cm^{-1}$ of the control and treated sample spectra were C–O–C bonds of cellulose (Gao et al. 2015). For the finishing agent spectrum, peaks at 720 $cm^{-1}$ belonged to the P–C stretching vibration of the finishing agent (Zheng et al. 2016). These results suggested that NDOPA molecules were grafted onto cellulose.

XRD

The crystal profile of the control and 10%-NDOPA-treated cotton fibers was characterized by XRD analysis. Figure 11 illustrates that the curves of treated cotton fibers were highly similar to those of the control cotton fibers. The control and treated cotton fibers exhibited diffraction peaks at 14.43°, 16.04°, 22.60°, and 34.03°, assigned to the (1–10), (110), (200), and (004) planes (Jia et al. 2016; Zheng et al. 2016; French 2013). The intensity of the diffraction peaks of treated cotton fibers was nearly the same as that of the control sample. As the NDOPA molecule was hydrophobic, it was difficult for NDOPA to enter the inner polar space of cotton fibers; thus, the crystal structure of cotton fiber was nearly unaffected in the finishing process. NDOPA was therefore grafted primarily onto the surface layer of cotton fibers.

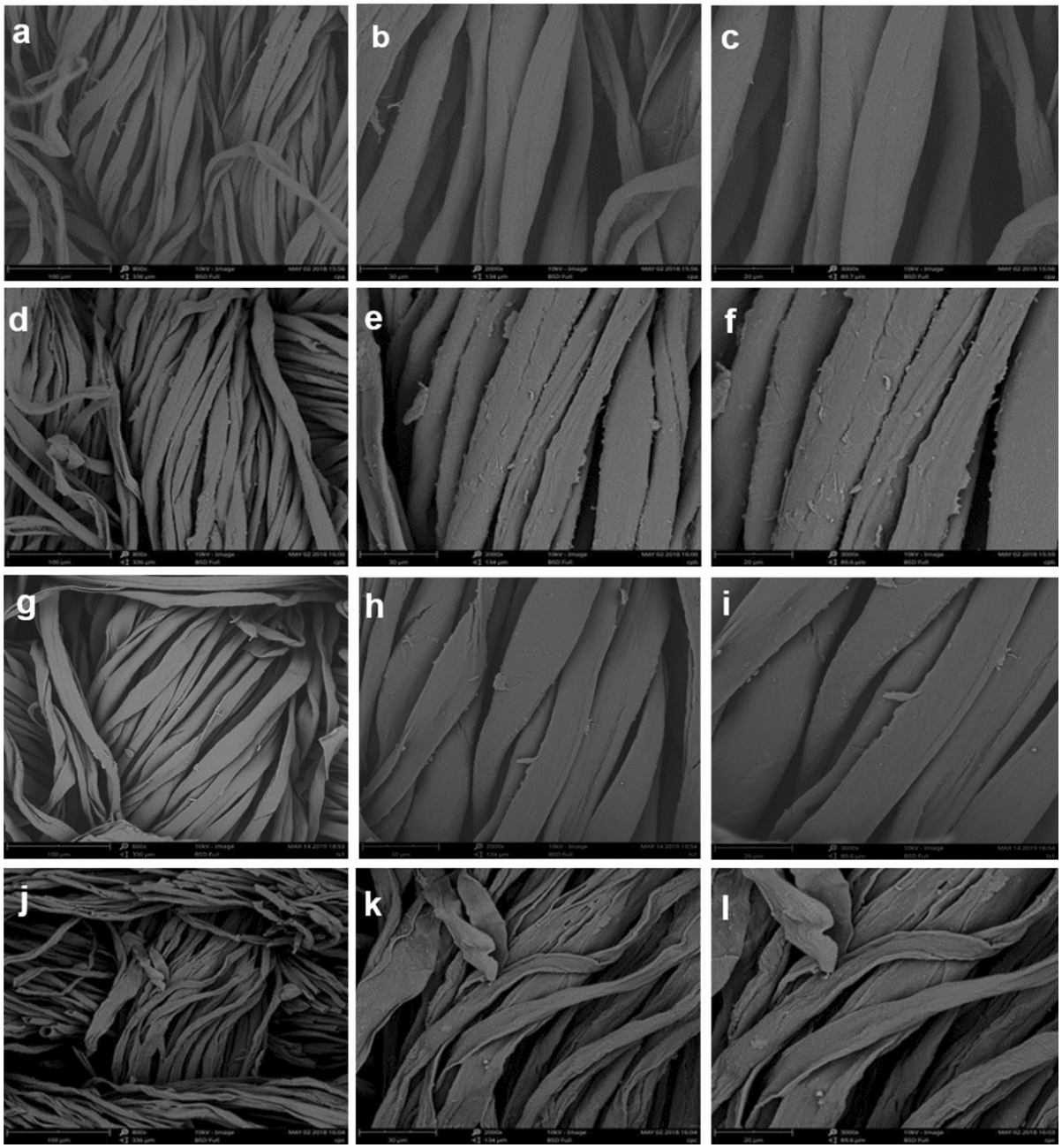



**Fig. 8** Scanning electron microscopy images of **a–c** control sample, **d–f** treated sample, **g–i** treated sample after 30 LCs and **j–l** burnt sample

## Durability

The laundering durability of samples was tested to evaluate whether superhydrophobic cotton textiles with flame-retardant properties could be used for an extended time without losing their function. Laundering durability was tested according to the AATCC 61-2006 standard. Figure 12 presents the WCAs of cotton fabrics treated with a mass concentration of 6%, 8%, and 10% finishing agent after 30 LCs. The 6%-, 8%-, and 10%-NDOPA-treated cotton fabrics had respective contact angles of 139.9°,

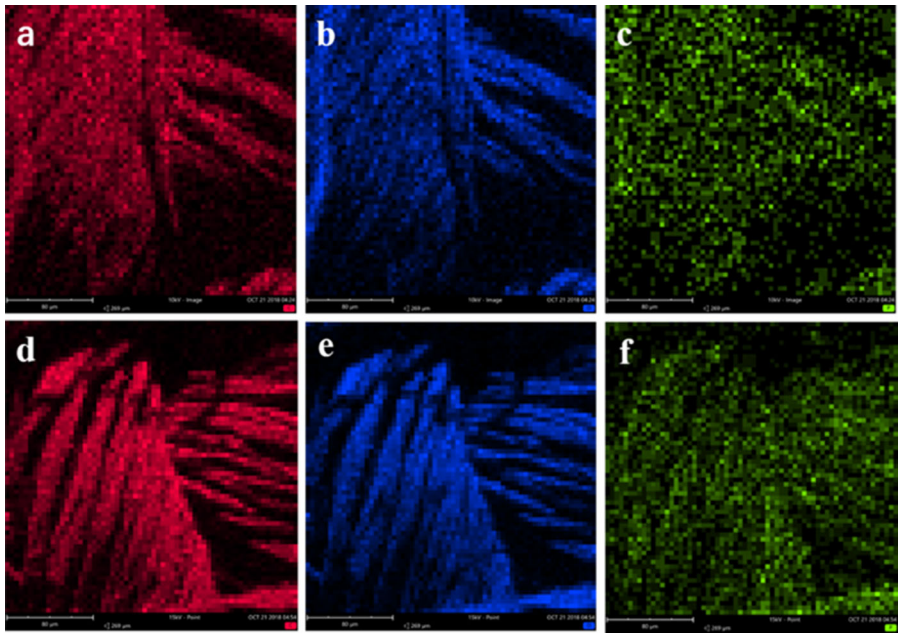



**Fig. 9** Treated cotton elements distribution of **a** C, **b** O, and **c** P; and treated cotton after 30 LCs elements distribution of **d** C, **e** O, and **f** P

**Table 2** Elemental composition of samples

| Element | Control sample | Treated sample | Treated sample after 30 LCs |
|---|---|---|---|
| C% | 45.59 | 50.16 | 47.92 |
| O% | 54.51 | 49.07 | 51.57 |
| P% | – | 0.77 | 0.51 |

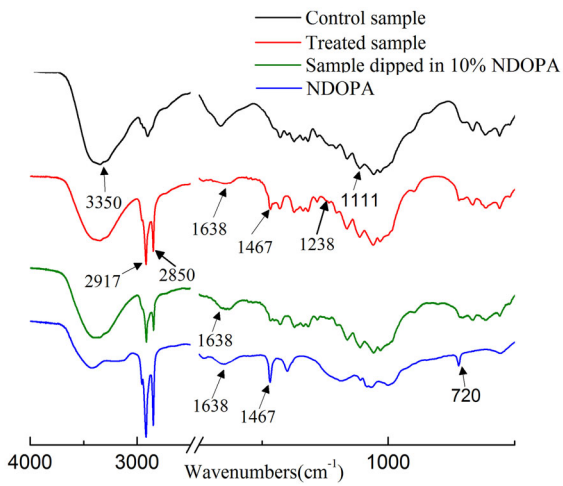



**Fig. 10** Fourier-transform infrared spectra of control sample, treated sample, sample dipped in 10% NDOPA and NDOPA

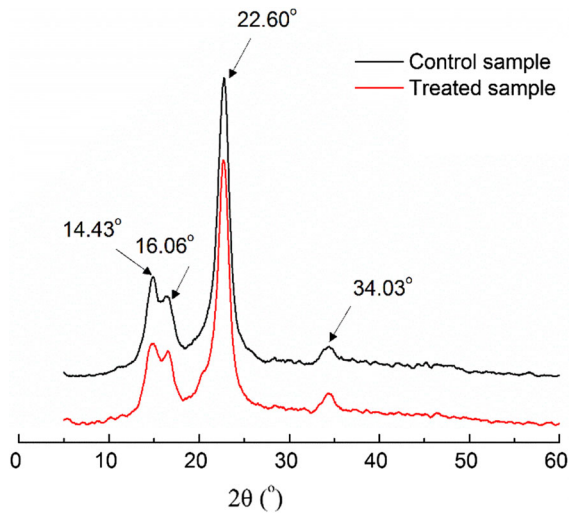



**Fig. 11** X-ray diffraction spectra of control sample and treated sample

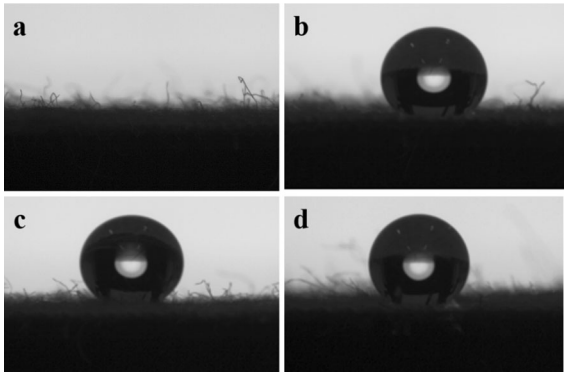



**Fig. 12** Water contact angles of **a** control sample and samples treated with **b–d** 6%, 8%, and 10% NDOPA after 30 LCs

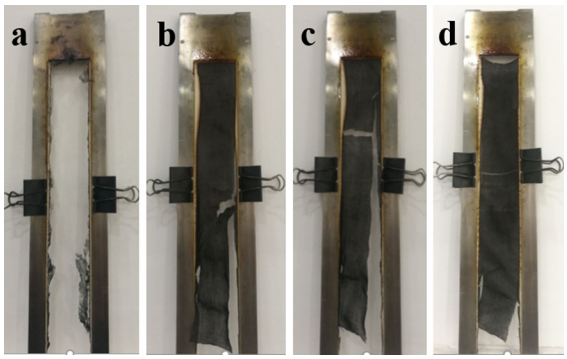



**Fig. 13** Vertical flammability test results of **a** control sample and samples treated with **b–d** 6%, 8%, and 10% NDOPA after 30 LCs

144.5°, and 149.5° after 30 LCs. Compared with the control cotton fabrics, the treated cotton fabrics maintained large contact angles and excellent hydrophobic properties after several washing cycles. Accordingly, the treated cotton fabrics effectively guaranteed the durability of hydrophobicity to laundering.

Figure 13 presents the vertical flammability test chart of the control cotton fabrics and cotton fabrics treated with 6%, 8%, and 10% finishing agent after 30 LCs. The damage length of all samples was 35 cm; the after-flame time and after-glow time of the control cotton fabrics were 8 s and 9 s, respectively; The after-flame time values of cotton fabrics treated with 6%, 8%, and 10% finishing agent were 6 s, 5 s, and 4 s, respectively; and the after-glow time of all treated fabrics was 0 s. The treated cotton fabrics maintained a relatively complete carbon frame. The flame-retardant properties of the washed treated cotton fabric decreased slightly compared with the unwashed treated cotton, but all samples retained an intact carbon structure. Therefore, cotton fabrics finished with the finishing agent exhibited durable flame-retardant properties (Table 3).

**Table 3** Vertical flammability of control and finished samples after 30 LCs

| Samples | After-flame time (s) | After-glow time (s) | Char length (mm) |
|---|---|---|---|
| 0 | 8 | 9 | 0 |
| 6% | 6 | 0 | 350 |
| 8% | 5 | 0 | 350 |
| 10% | 4 | 0 | 350 |

## Conclusion

A new fluorine-free hydrophobic and flame-retardant finishing agent was designed and synthesized. Cotton fabrics treated with the finishing agent demonstrated hydrophobicity and flame retardancy functions. The contact angle of the treated cotton fabrics could reach 157.5°, indicating that cotton fabrics treated with the finishing agent could be superhydrophobic. The contact angle was maintained at 149.5° after 30 LCs. The vertical flammability burning test showed that the treated cotton fabrics were able to maintain a complete carbon frame and a substantially complete carbon frame after 30 LCs. The results of the TG test suggested that the treated cotton fabrics had flame retardancy. FTIR and EDX indicated that the finishing agent was effectively covalently bonded to the cotton fiber. Thus, the cotton fabrics had durable hydrophobicity and flame retardancy. XRD and SEM showed that the structure and surface morphology of the cotton fabrics did not change much. In short, this new finishing agent can be applied to cotton fabrics as a durable and environmentally friendly hydrophobic flame-retardant.

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