




A lipid coating on cotton fibers with enhanced adsorption capability for fabric functionalization

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Abstract Cotton is often modified to minimize its disadvantages during textile processing. However, most finishing methods suffer from difficulties in finding finishing reagents and chemical aids. Here, we report a novel strategy based on a combination of the pad-dry-cure method and the construction of surface coating to expand the scope of the finishing reagent and simplify the finishing process. Polyacrylic acid (PAA) oligomer was first grafted onto cotton fiber surfaces via esterification between the carboxyl groups of PAA and the hydroxyl groups of the cellulose molecules on the cotton fiber surface. Hexadecanol was then reacted with the residual carboxyl groups of

the PAA to build a lipid layer on the cotton fiber surfaces. The enhanced adsorption capability for hydrophobic molecules was verified via three reagents: stearyl trimethyl ammonium bromide (STAB), 9,10-dihydro-9-oxa-10-phospho-10-oxide (DOPO), and 3,5-di-tert-butyl-4-hydroxybenzoic acid hexadecyl ester (Cyasorb 2908), which they offered bacterial-, flame-, and UV-resistance to the cotton fabrics, respectively. This strategy can lead to finishing technologies suitable for scale-up in the textile industry.

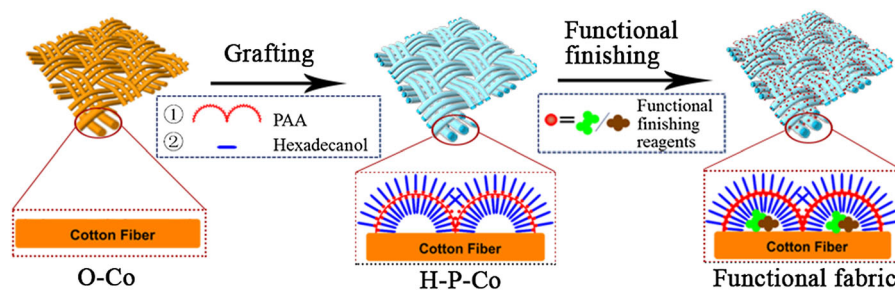
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Graphic abstract



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Introduction

Cotton is widely used in clothes and household textiles due to its air permeability, flexibility, and comfort (Liu et al. 2017; Peng et al. 2019; Qi et al. 2016a; Sandin and Peters 2018; Zhang et al. 2016). It also has several limitations such as flammability, microbial contamination, and poor UV blocking that have been identified as problems in its practical applications (Lazar et al. 2020; Xu et al. 2017; Zhou et al. 2018a). Many efforts have been devoted to fabricate antibacterial, flame-resistant, and UV-resistant cotton fabrics (Duan et al. 2020; Huang et al. 2020; Ortelli et al. 2018; Qin et al. 2019; Qiu et al. 2018; Yu et al. 2020; Zhang et al. 2019; Zhang et al. 2020; Zhou et al. 2019; Zhu et al. 2017). Textile engineering has led to additional functions to address concerns regarding environmental protection, health, and safety as well as help to develop wearable devices (Agrawal et al. 2020, 2019; Chen et al. 2020; Ghasemlou et al. 2019; Li et al. 2020a, 2017; Qi et al. 2016c; Sam et al. 2019; Yang et al. 2018; Zhou et al. 2018a). However, most finishing strategies suffer from limited stability, energy consumption, narrow scope of the finishing reagents and chemical aids, expensive costs, and challenges in scaling.

The pad-dry-cure method is a traditional finishing technology for cotton fabrics (Qiang et al. 2017; Xu et al. 2019a, b; Xu et al. 2018b; Zhou et al. 2018b). It is still recognized as a convenient, efficient, cheap, and

widely applicable way to impart cotton fabrics with different functions. Here, there are strict requirements on the chemical reactivity and structure of the finishing reagents. There are extensive efforts to develop finishing reagents that are suitable for the simple pad-dry-cure approach to prepare functional fabrics. For instance, Su et al. synthesized a borneol-based antibacterial agent to combat multi-drug resistant bacteria (Yang et al. 2020). This reagent can be grafted onto cotton for a sustained antibacterial activity that is higher than commercially available finishing reagents.

We recently described a clean technology using betaine as the finishing reagent for the preparation of antibacterial fabrics (Duan et al. 2020). The antibacterial efficiencies of the modified fabrics against both *Staphylococcus aureus* (*S. aureus*) and *Escherichia coli* (*E. coli*) are higher than 99.0%. Besides, an ammonium salt of diethylene glycol phosphonate was synthesized to realize flame retardance on the cotton fabric. The resulting fabric exhibited an optimum LOI value of 43.7%, which is significantly higher than that of untreated cotton fabric (Tian et al. 2019). More recently, a cyclophosphazane derivative and a triazine-based flame retardant were also reported to improve the flame retardancy of cotton fabrics (Liu et al. 2019a; Sun et al. 2020). In contrast, only a few organic UV-absorbers suitable for cotton textiles have been reported. Yan et al. found that cotton fabrics finished with 5-(carbonyloxy succinic)-benzene-1,2,4-tricarboxylic acid (BSTA) have a remarkable UV protective effect (Qi et al. 2016a, b, c).

In addition to conventional methods, surface coatings have been constructed on cotton fibers and are an effective method to achieve various functions on cotton (Chen et al. 2018; Gavrilenko and Wang 2019;

Guo et al. 2020; Holder et al. 2017; Li et al. 2020b; Mai 2018; Qiu et al. 2018; Xu et al. 2017). This strategy is especially effective for antibacterial, flame-retarding, and UV-protective functions because these effects are primarily moderated by the fiber surface. However, the vast majority of efforts over the last decade have focused on inorganic NPs such as Ag, TiO₂, CuO, and ZnO NPs (Cao et al. 2020; Chavali et al. 2020; Chen et al. 2018; Cheng et al. 2018; Choi et al. 2018; Eid et al. 2019; Irfan et al. 2019; Radetic and Markovic 2019). Somewhat surprisingly, polymer coatings on cotton for additional functionalities are far less explored (Li et al. 2020b; Pan et al. 2018; Song et al. 2019; Xu et al. 2020).

Based on the research background, we propose here a well-designed combination of the pad-dry-cure method and a surface coating can expand the scope of finishing reagents and simplifying the finishing process. To test this idea, a hydrophobic layer with robust adsorption capability was established on the cotton fiber surfaces over two steps: First, polyacrylic acid (PAA) oligomers were grafted onto cotton fibers via the esterification between a part of the carboxyl groups of PAA and the hydroxyl groups of the cellulose molecules on the cotton fiber surface. Second, hexadecanol was esterified with the residual carboxyl groups of the PAA. The lipid layer grafted on the cotton fiber would enhance the adsorption capability for hydrophobic finishing reagents, offering a versatile adsorbing fabric that can achieve various functions through a simple adsorbing treatment. The structure of the lipid layer was characterized utilizing Fourier transform infrared (FTIR) spectroscopy, X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), and Field emission scanning electron microscopy (FE-SEM). The enhanced adsorption capability was verified using three functional reagents, stearyl trimethyl ammonium bromide (STAB), 9,10-dihydro-9-oxa-10-phospho-10-oxide (DOPO) and 3,5-di-tert-butyl-4-hydroxybenzoic acid hexadecyl ester (Cyasorb 2908). Experimental data show that the grafting layer gives the cotton fabric good antibacterial, flame-resistant, and UV-resistant functions than the original cotton, if they have been finished with the same processes. Together with the simple adsorption process, this strategy can be scaled up to an industrial finishing production allowing for the development of finishing technologies in the textile industry.

Experimental

Materials

Sodium dodecyl sulfonate (98.0%), polyacrylic acid, (PAA, 50% aqueous solution, average molecular weight: 3,000), liquid paraffin (99.7%), N-hexane (97.0%), hexadecanol (98.0%), stearyl trimethyl ammonium bromide (STAB, 99.0%), 9, 10-dihydro-9-oxa-10-phospho-10-oxide (DOPO, 97.0%) were purchased from Shanghai Aladin Co., Ltd (China). 3,5-Di-tert-butyl-4-hydroxybenzoic acid hexadecyl ester (Cyasorb-2908, 97.0%) was obtained from J&K Chemicals (China). All of the reagents were applied without further purification. Cotton fabrics were provided by Testfabrics, Inc. (USA) and washed in 0.2 wt% sodium dodecyl sulfonate aqueous solution for 30 min and ethanol for 30 min, respectively.

Grafting lipid layer on cotton fabrics

10 wt% of PAA aqueous solution was selected according to the effect of the concentration on the tensile strength of the cotton fabrics and the water contact angle (WCA) of the modified cotton fabrics (Fig S1 and S2). Original cotton (O-Co) (10 pieces, 5 cm × 5 cm) was immersed into a PAA aqueous solution (250 mL, 10 wt%) for 10 min, squeezed to a wet weight of 200 ± 2 wt%, heated at 180 °C for 5 min, rinsed with deionized water (250 mL, 3 times), and dried at 100 °C for 1 h to obtain the sample P-Co. The P-Co (10 pieces, 5 cm × 5 cm) was immersed into a liquid paraffin solution of hexadecanol (250 mL, 10 wt%), heated at 175 °C for 30 min, ultrasonically rinsed with N-hexane (100 mL, 2 min, 3 times), and dried at 80 °C for 1 h to obtain sample H-P-Co.

Functional finishing processes

Antibacterial cotton fabric: H-P-Co (3 pieces, 5 cm × 5 cm) was immersed into a Na₂CO₃ aqueous solution (200 mL, 0.002 mol/L, pH = 10.2) for 10 min, rinsed with deionized water for 4 times, immersed into another STAB aqueous solution (500 mL, 0.1 mg/L) for 10 min, rinsed with deionized water (250 mL, 40 ± 5 °C, 4 times), dried at 100 °C for 1 h to obtain sample H-P-Co/STAB. As the reference sample, O-Co (3 pieces, 5 cm × 5 cm) underwent the same process to prepare sample O-Co/

STAB. In order to evaluate antibacterial renewability of H–P–Co fabric, H–P–Co/STAB (3 pieces, 5 cm × 5 cm) was washed using deionized water (250 mL, 40 ± 5 °C, 40 times) and then dried at 100 °C for 1 h to obtain sample H–P–Co/Wa. Then, the washed fabric samples (H–P–Co/STAB/Wa, 3 pieces, 5 cm × 5 cm) were immersed into a STAB aqueous solution (500 mL, 0.1 mg/L) again for 10 min, rinsed with deionized water (250 mL, 40 ± 5 °C, 4 times), and dried at 100 °C for 1 h to obtain sample H–P–Co/STAB/Re.

Flame retardant cotton fabric: H–P–Co (5 pieces, 5 cm × 5 cm) were immersed into a DOPO solution in dioxane (200 mL, 200 g/L) for 10 min, squeezed to wet weight of 230 ± 5 wt%, heated at 150 °C for 5 min, rinsed using deionized water (250 mL), dried at 100 °C for 1 h to obtain sample H–P–Co/DOPO. As the reference sample, O–Co (5 pieces, 5 cm × 5 cm) went through the same process to prepare sample O–Co/DOPO.

UV resistant cotton fabric: H–P–Co (3 pieces, 5 cm × 5 cm) was immersed into a Cysorb 2908 solution in acetone (100 mL, 3 wt%) for 10 min, squeezed to a wet weight of 200 ± 5 wt%, dried at 80 °C for 1 h, rinsed using deionized water (250 mL, 3 times), and dried at 100 °C for 1 h to prepare sample H–P–Co/C-2908. As the control sample, O–Co (3 pieces, 5 cm × 5 cm) underwent the same process to prepare sample O–Co/C-2908. Then, H–P–Co/C-2908 (3 pieces, 5 cm × 5 cm) was washed using sodium dodecyl sulfate solution (250 mL, 0.2 wt%) in a beaker for 10 min (300 rpm, 10 times), then washed with deionized water (250 mL, 3 times) and dried at 100 °C for 1 h to obtain sample H–P–Co/C/Wa. As the control sample, O–Co/C-2908 (3 pieces, 5 cm × 5 cm) was washed in the same way to obtain sample O–Co/C/Wa.

General characterization

Water contact angle (WCA) analyses were carried out on a contact angle measuring instrument (DSA 20, Kruss, Germany). The surface morphology of the cotton fabrics was examined using a field emission scanning electron microscope (FE-SEM) set (Ultra-55, Zeiss, Germany) to 3.0 kV after gold coating. Thermal gravimetric analyses were conducted via a thermo gravimetric analyzer (TGA/ DSC-1, Mettler-Toledo Corp., Switzerland) from 30 to 800 °C at a

heating rate of 10 °C /min with a nitrogen flow (40 mL/min). Attenuated total reflection fourier transformed infrared (ATR-FTIR) spectrum were recorded by PerkinElmer Frontier in the range of 400–4000 cm⁻¹ at a resolution of 1 cm⁻¹. XPS analyses were conducted by X-ray photoelectron spectrometer (Thermo ESCALAB 250XI, USA). XRD analyses were performed using an X-ray powder diffractometer (Ultima IV, Neo-Confucianism co. LTD, Japan) at a scanning speed of 5°/min from 10° to 80°.

The quality, flexibility, water vapor permeability, water absorptivity, whiteness index, and tensile strength of the fabric samples were carried out according to our previous reports (Touhid et al. 2020; Xu et al. 2017, 2019b), as detailed in the “Supporting Information” part, which also introduces in detail the test methods of the cytotoxicity analysis, antibacterial property, flame retardant property and UV resistant property of the cotton fabrics.

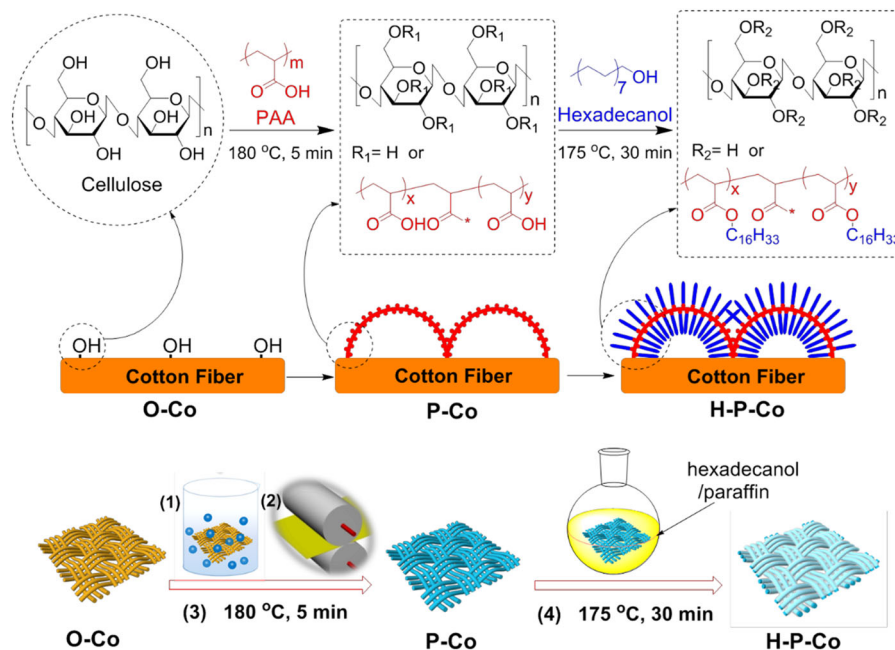
Results and discussion

Grafting the lipid layer on cotton fabric

The modification process undergone by cotton fabric is illustrated in Scheme 1. Having part of the carboxyl groups involved in the reaction with the hydroxyl groups of the cellulose molecules on the cotton fiber surface, the PAA chains were covalently linked on the fiber surface through ester groups. When the residual carboxyl groups of the PAA chains were further involved in the reaction with hexadecanol molecules, a layer of lipid coating was constructed on the fiber surface.

The WCA value was measured to evaluate the surface wettability of the original cotton fabric (O–Co) and the modified fabric that have undergone the entire modification process. As shown in Fig. S3a and S3b, the water droplets on O–Co and P–Co disappeared instantly, thus making it impossible to measure the certain WCA value. In contrast, the WCA value on the modified fabric, H–P–Co, was measured as 126.6° (Fig. S3c), indicating that the cotton fabric surface shifted to being hydrophobic.

ATR-FTIR is referred to as a representative technique applied to characterize the chemical bonds in a surface layer with its depth ranging from several



Scheme 1 The schematic diagram and reactions to construct the lipid layer on cotton fiber surface

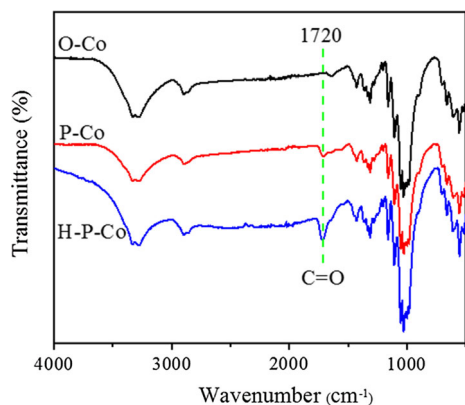


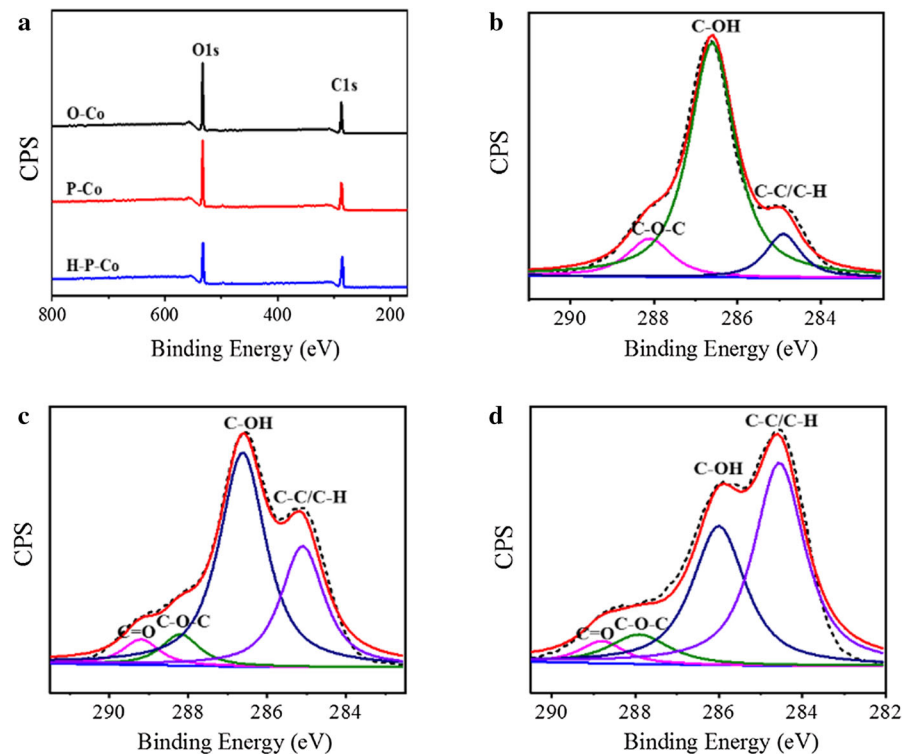
Fig. 1 ATR-FTIR spectrum of O-Co, P-Co and H-P-Co fabrics

hundred nanometers to several microns. In this study, ATR-FTIR technique was adopted to explore the chemical structure of the modified fiber surface. Figure 1 shows the ATR-FTIR spectrum of O-Co, P-Co and H-P-Co fabrics. The peak at 1720 cm^{-1} correspond to the C=O bond (Chen et al. 2018; Qi et al. 2016c; Xu et al. 2018a, 2016; Zhang and Fan 2010). The appearance of the C=O bond in the ATR-FTIR spectrum of P-Co indicated that the PAA chains were connected with the cellulose molecules by ester bands.

Additionally, the strong absorption peak of the ester bond in the ATR-FTIR spectrum of H-P-Co suggested that there were hexadecanol molecules linked to H-P-Co by ester bonds.

Those structural changes occurring to the fiber surface were further investigated using XPS. Figure 2 shows the wide-range XPS spectrum of the O-Co, P-Co and H-P-Co fabric samples, the elemental contents (C and O) of which in the fiber surface layers are summarized in Table S1. In comparison with the original cotton, the O elemental content in the surface of P-Co declined slightly from 41.1% to 39.1%, before a further reduction to 31.6% following the modification made to H-P-Co fabric. This result is considered reasonable for the two reagents, PAA and hexadecanol, because their O elemental content was in an order of cellulose > PAA > hexadecanol. Moreover, the high resolution C 1s XPS spectrum of O-Co (Fig. 2b), P-Co (Fig. 2c) and H-P-Co (Fig. 2d) were compared. The C 1s XPS spectrum of the original cotton fabric were divided into three peaks with a binding energy of 288.1 eV (C–O–C, 12.22%), 286.6 eV (C–OH, 76.8%) and 284.9 eV (C–C/C–H, 11.0%), respectively (Duan et al. 2020; Freire et al. 2006; Xu et al. 2017). In contrast, the C 1s spectrum of P-Co exhibited an additional peak at the binding

Fig. 2 XPS spectrum of O-Co, P-Co and H-P-Co fabrics (a); high resolution C1s XPS spectrum of O-Co (b), P-Co (c) and H-P-Co (d) fabrics



energy of 289.2 eV (C=O, 5.4%), with a reduction to the intensity of the characteristic absorption of bond C-OH (57.5%), these results indicated that the PAA chains were covalently linked with the cellulose molecules on the cotton fiber surface through ester groups. In contrast to that of P-Co, the intensity of the characteristic absorption of C-C/C-H bond in the C 1s spectrum of H-P-Co was observed to rise sharply from 30.2% to 50.4%, suggesting the successful grafting reaction of hexadecanol. In addition, the high resolution O 1s XPS spectra of O-Co, P-Co and H-P-Co were shown in Fig. S4. There was only one peak at 533.3 eV in the O 1s XPS spectrum of O-Co, corresponding to C-O bond. In contrast, there was a new peak in both the O 1s XPS spectrum of P-Co (532.4 eV) and H-P-Co (531.9 eV), representing the C=O bond (Chandrasekaran et al. 2020; Suryaprabha and Sethuraman 2016). Moreover, the relative contents of the C-O bond and the C=O bond in the O 1s XPS spectra were consistent with those in the C 1s XPS spectra, further suggesting the successful grafting of PAA and hexadecanol.

Figure 3 shows the XRD spectrum of the fabric samples. There were four peaks observed at 2θ 14.5°, 16.4°, 22.5° and 34.2°, corresponding to the crystal

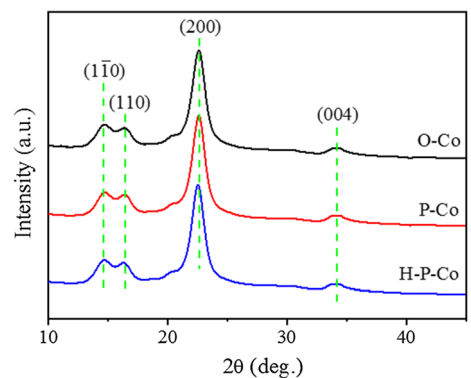


Fig. 3 XRD patterns of O-Co, P-Co and H-P-Co fabrics

face 1₁₀, 110, 200 and 004 of the typical cellulose crystal structure (Chen et al. 2019; Gaspar 2014; Liu et al. 2019b), respectively. In addition, the XRD spectrum of O-Co, P-Co and H-P-Co shows almost no change, implying that the modification process did not damage the crystalline structure of cotton fibers.

The surface morphology of O-Co (Fig. 4a, d), P-Co (Fig. 4b, e) and H-P-Co (Fig. 4c, f) were detected by SEM. The low-magnification SEM images (Fig. 4 top) showed no significant difference. Besides, as shown in the SEM image of H-P-Co (Fig. 4c), the

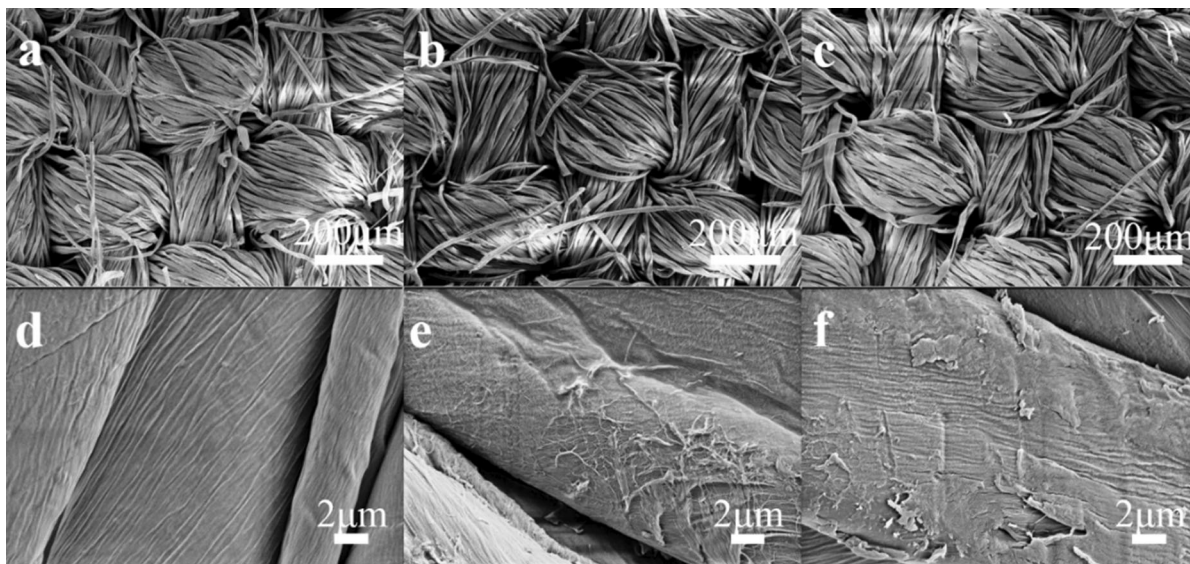


Fig. 4 FE-SEM images of the O-Co (a, d), P-Co (b, e) and H-P-Co (c, f) fabrics

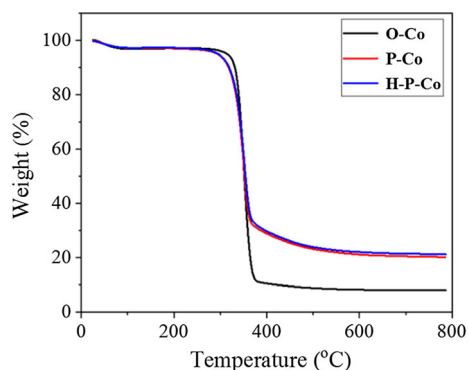


Fig. 5 TGA curves of the O-Co, P-Co and H-P-Co fabrics

gaps between the yarns and the interspaces between the fibers were not blocked by the grafted polymers. These void spaces played a crucial role in the breathability of cotton fabric. As shown in the high-magnification SEM images (Fig. 4 bottom), the original cotton fibers had a relatively smooth surface with numerous natural wrinkles. After the modification made to O-Co with PAA, the fiber surface became rough, despite the fiber surface being covered with two dimensional reticulation, suggesting that the PAA polymeric chains were successfully grafted onto the cotton fiber surfaces. Furthermore, after the modification with hexadecanol to prepare H-P-Co, the fiber surface was covered with a rough and film like coating.

Figure 5a shows the TGA curves of O-Co, P-Co and H-P-Co. The original cotton fabric showed a relatively low carbon yield ratio at 8.1%. After the grafting of PAA molecules, however, the carbon yield ratio of P-Co increased significantly to 20.2%. When hexadecanol modification was complete, the carbon yield ratio further increased to 21.2%. The three fabric samples were weighted (Table S2), 232 ± 1.5 mg, 249 ± 6.3 mg and 252 ± 3.1 mg for O-Co, P-Co and H-P-Co fabric (1 piece, $5 \text{ cm} \times 5 \text{ cm}$), respectively. Therefore, the improvement of carbon yield can hardly be attributed to the grafted coatings. It is speculated that the PAA chains grafted on the fiber surface underwent thermal decomposition into carboxylic acid (the thermal decomposition mechanism of the PAA chains was shown in Fig. S5), which may be associated with the dehydration of the cellulose molecules of the cotton fibers and ultimately results in the increase of carbon yield ratio.

The vitro cytotoxicity assessments were carried out by means of CCK-8 assay and apoptosis assay to evaluate the safety of O-Co and H-P-Co (Zhang et al. 2020; Zheng et al. 2021). We configured leachate solutions with different concentrations to examine the effects of dosage, the original leachate solutions of O-Co and H-P-Co were labeled O-Co-1 and H-P-1, and the leachate solutions of O-Co and H-P-Co diluted twice with normal saline were labeled O-Co-2 and H-P-2, respectively. The effect of the leachate solutions

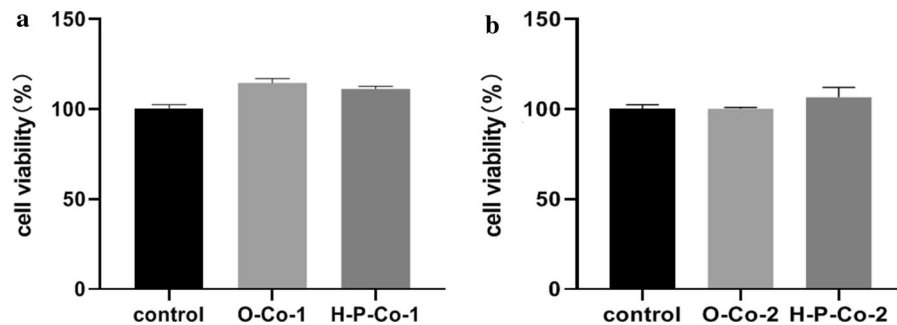


Fig. 6 Cytotoxicity evaluation by CCK-8 assay (**a**, **b**) of the leachate solutions from O-Co and H-P-Co fabrics. The leachate solutions without dilution (**a**). The leachate solutions were diluted twice (**b**)

on the cell proliferation of NIH/3T3 cells was detected by CCK-8 assay. As shown in Fig. 6a, b, the cell viability of O-Co and H-P-Co reached a similar level to the control group in both cases of the leachate solution either not diluted or diluted twice. According to the results obtained, the leachate solutions were neither toxic nor dosage dependent. Then, the leachate solutions were further subjected to apoptosis detection. An induction of apoptosis was detected by Annexin V-FITC/PI Apoptosis Detection Kit. The test results of the undiluted leachate solutions (Fig. S6a) and the leachate solutions diluted twice (Fig. S6b) suggested a similar percentage of apoptosis cells (Annexin V + /PI - and Annexin V + /PI +), which is less than 9%. This was insignificantly different from the control group. These results demonstrated that the leachate solutions of O-Co and H-P-Co were non-toxic, suggesting that the H-P-Co fabric was safe to human health, as like the original cotton fabric (O-Co).

As shown in Table S2, a number of significant factors for the cotton fabric were characterized to assess the impact of the modification process on some natural properties of cotton. The bending height of a fabric is a commonly used index for fabric softness. As shown in Fig. S7, the bending height of H-P-Co exhibited only slight difference to the O-Co fabric, suggesting that the modification process made no significant difference to the softness of the cotton fabrics. Additionally, the whiteness indexes of H-P-Co showed no much difference with O-Co too (Table S2). Water absorptivity and water permeability are regarded as the crucial factors in the level of wearing comfort. As confirmed by the experiments, they were well maintained on the H-P-Co fabric after the modification processes and finishing treatments

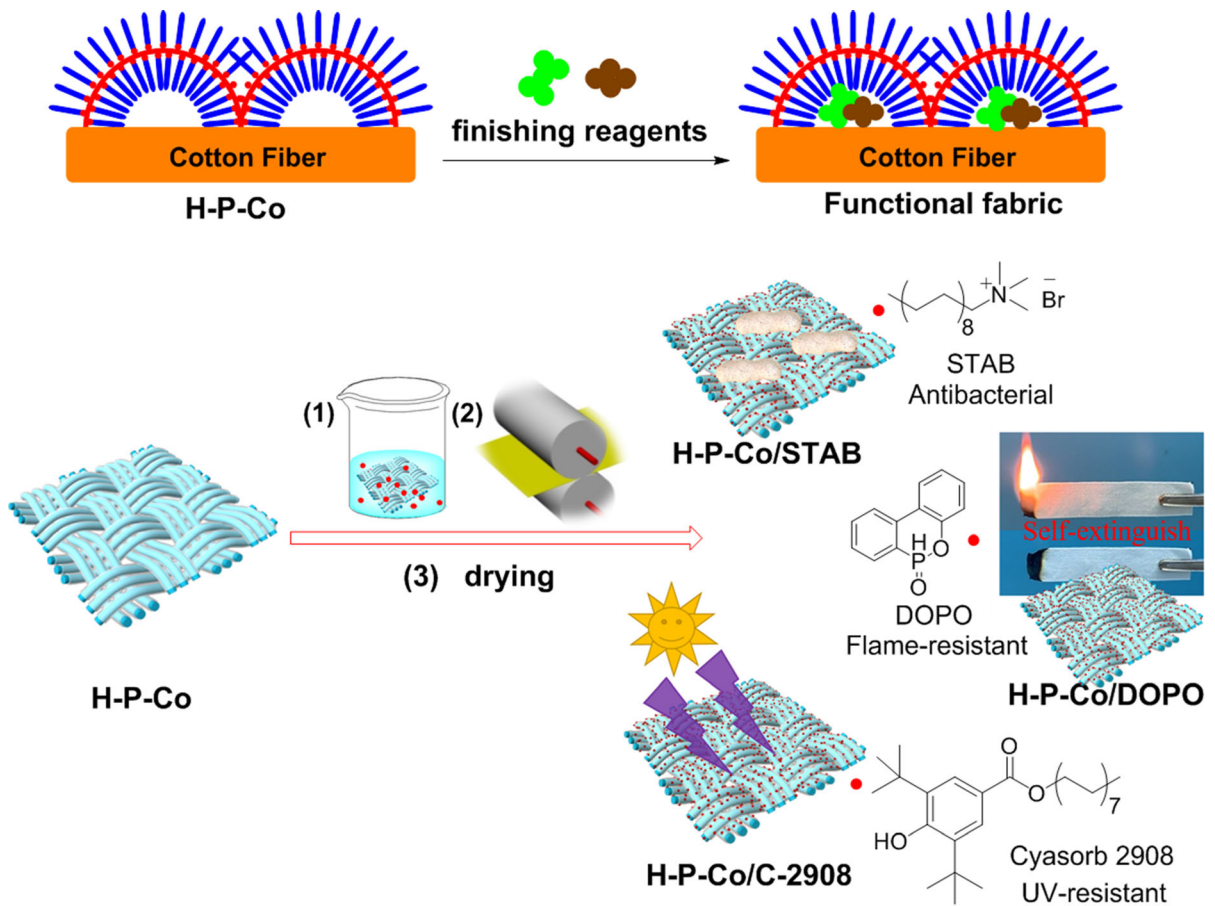
(Table S2). These results are highly consistent with the observation made in the low-magnification SEM images (Fig. 4c and Fig. S8 top) that the gaps between the cotton fibers were not blocked. Moreover, the breaking strength of H-P-Co (32.0 MPa) remained acceptable for daily use, despite being slightly lower than that of O-Co (43.6 MPa).

Functional finishing treatments

As mentioned in the introduction part, suitable reagents and extreme reaction conditions are usually required for a successful finishing process. However, they are often what hinders the development of functional fabrics. In this study, a lipid layer was prepared on the surface of cotton fabric using PAA and hexadecanol through an environmentally friendly process. All of the reagents used were low-toxic, inexpensive, and commonly used for industrial productions. Furthermore, the lipid layer on the surface of cotton fabric was designed to solve some obstacles in the general pad-cure finishing processes.

Hydrophobic finishing reagents can be adsorbed in the lipid layer, and chemical reactions are rarely needed in the finishing process. Based on the adsorption concept, this strategy can be adopted to widen the finishing reagent scope and simplify the treatment processes. To verify the effectiveness of adsorption by the grafting lipid layer, STAB, DOPO, and Cyasorb 2908 functional reagents were selected to achieve antibacterial, flame-resistance, and UV-resistant functions on the cotton fabric, respectively (Scheme 2).

Antibacterial performance is an important factor to consider for the fabric market because it matters for human health. STAB (Inacio 2016) has been applied as disinfectants and antiseptics for quite long, it is an



Scheme 2 The schematic diagrams of the functional finishing processes of H-P-Co fabric and their corresponding functionalities

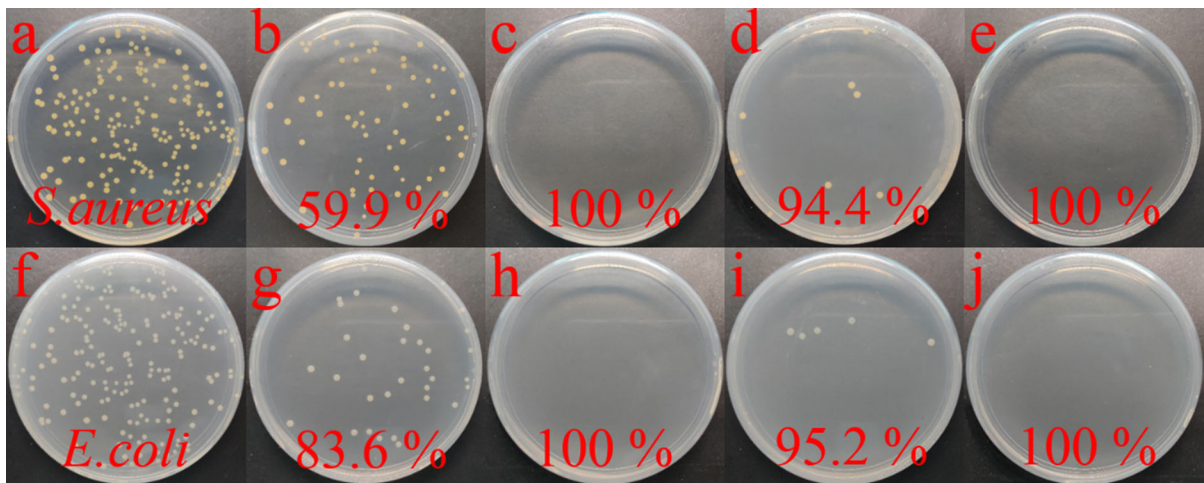


Fig. 7 Optical images of the antibacterial tests against *S.aureus* and *E.coli*, O-Co (a, f), O-Co/STAB (b, g), H-P-Co/STAB (c, h), H-P-Co/STAB/Wa (d, i), H-P-Co/STAB/Re (e, j)

amphiphilic molecule, with a positively charged quaternary ammonium polar head group that has one apolar chain attached to it. Such desirable advantages as broad-spectrum antimicrobial activity, chemical stability, cheap affordability, and less demanding requirements on storage make it fit for both general hygiene and clinical purposes.

Herein, STAB was taken as an antibacterial finishing reagent for understanding the advantages of the lipid layer grafted onto the cotton fiber surfaces. After a simple process for the adsorption of STAB, the WCA of the H–P–Co dropped from 126.6° to 106.5° (Fig S3c), indicating that the STAB molecules were oriented and their hydrophilic groups were arranged towards the outside of the fiber surface. Besides, the bending height, ventilation property and water absorptivity of H–P–Co/STAB fabric (Fig S7c and Table S2) were similar to those of H–P–Co fabric, indicating that the wear comfort of the H–P–Co fabric was not damaged by the antibacterial finishing. In addition, SEM analysis were conducted to detect the surface morphology of the H–P–Co fabric before and after finished with STAB. As show in Fig. S8a, there was a layer of film found on the surface of H–P–Co/STAB, suggesting that this finishing process made no difference to the morphology of H–P–Co.

Figure 7 and Fig S9 show the bacterial reduction rate (BR) on the cotton fabrics. The H–P–Co/STAB fabric produced remarkable antibacterial effect against both *S.aureus* and *E.coli* as the BR values were both close to 100% (Fig. 7c, h). In comparison, the BR values of the O–Co/STAB samples reached merely 59.9% and 83.6% for *S.aureus* and *E.coli*, respectively (Fig. 7b, g). Those results led to the inference that H–P–Co can absorb more STAB than O–Co. In addition, the data shown in Fig. 7d, i was obtained after the repeated washing process. After 40 washing cycles, the antibacterial activity of the H–P–Co/STAB fabric was still as high as 94.4% and 95.2% for *S.aureus* and *E.coli*, respectively. However, through the re-adsorption of STBA, the antibacterial rate increased back to 100%.

As an oil-soluble, heterocyclic, halogen-free and organophosphorus compound, DOPO has received notable attention owing to its excellent flame retardant capability and environment compatibility (Vasiljević et al. 2015). It could play important role in flame inhibition in the gas phase and char enhancement in the condensed phase (Suryaprabha and Sethuraman

2020, 2018; Wang et al. 2016). In this study, it is a model flame retardant to evaluate the effect of the lipid layer-assisted finishing process. As show in Fig. S7d and Table S2, after being finished with DOPO, the bending height, ventilation property and water absorption of the H–P–Co/DOPO fabrics exhibited only slight difference to the H–P–Co fabric, suggesting that the flame-retardant finishing process made no significant damage on the comfort of the H–P–Co fabric. Besides, the layer of film on the surface of H–P–Co/DOPO was found smoother than the film on H–P–Co (Fig. S8e), and the WCA of H–P–Co/DOPO was 130.3°, slightly higher than that of H–P–Co, these phenomena may be caused by the presence of DOPO molecules on the surface of H–P–Co/DOPO.

The flame retardant capability of the modified cotton fabrics was estimated using two methods: a direct burning test and a LOI measurement. Figure 8 shows the time-dependent images recording the burning process of the cotton fabrics. The complete fire process is shown in Video S1. After ignition, the fabrics continued to burn violently until the material was consumed. The O–Co fabric burned actively, leaving a minimal amount of char (Fig. 8a). The P–Co and H–P–Co ignited immediately and burned rapidly similar to O–Co (Fig. 8b), however, they left more residue with a distinguishable weave structure (Fig. 8b, c). This agrees to the TGA results of the high carbon yield ratio, further demonstrating that the PAA chains grafted on the cotton fibers have a catalyst effect during the burning process. The O–Co/DOPO was slowly burned within 9 s, but it did not self-extinguish until the fabric was burned off (Fig. 8d). In contrast, H–P–Co/DOPO (Fig. 8e) shows the lowest flammability and the capability to self-extinguish (the flame automatically extinguished within 4 s). To quantify the combustion properties of the cotton fabrics, LOI tests were performed with representative data in Fig. 9. The LOI value for O–Co is 17.5%, meaning that the original cotton fabric is very flammable. In contrast, the LOI value of H–P–Co/DOPO increased to 23.6%, demonstrating the enhanced flame-retardant capability, due to the adsorption capability of the lipid layer on the original cotton fabric.

The UV region of the electromagnetic spectrum can be divided into three bands, namely, UVC (200 nm–290 nm), UVB (290 nm–320 nm) and UVA (320 nm–400 nm). Among them, UVA and UVB

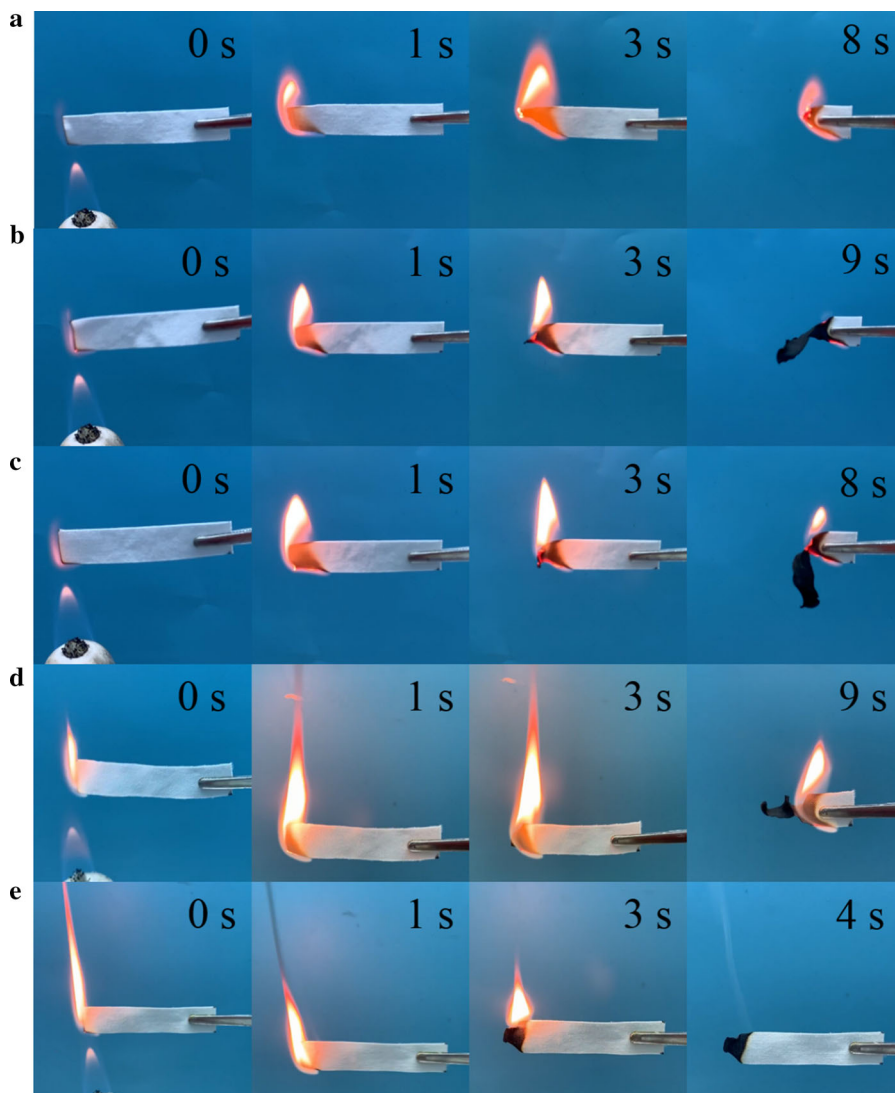


Fig. 8 Optical images of the cotton samples ignited with alcohol lamp, O-Co (a), P-Co (b), H-P-Co (c), O-Co/DOPO (d), H-P-Co/DOPO (e)

can penetrate human skin, thus causing diseases (Subbiah et al. 2019). Developed by American Cyanamid Company in the early 1980s, Cyasorb 2908 is an excellent light stabilizer that shows such advantages as low volatility, low coloring, chemical stability, acid and alkali resistance, and low-toxicity (Kuki et al. 2017). Herein, it was selected to improve the ultraviolet resistance of the cotton fabric. As shown in Fig. S7e and Table S2, after the UV-resistant finishing, the bending height, ventilation property and water absorption of the H-P-Co/C-2908 fabric almost has no difference to the H-P-Co fabric, indicating that the UV-resistant finishing process did not hurt the

comfort of the H-P-Co fabric. And the WCA of H-P-Co/C-2908 (129.2° , Fig. S3f) was slightly higher than that of H-P-Co/C-2908, which may contribute to the presence of Cyasorb 2908 molecules on the surface of H-P-Co/C-2908 fabric. In addition, there was still a layer of film on the surface of H-P-Co/C-2908 fabric (Fig. S8f), indicating that the UV-resistant finishing process did not damage the surface morphology of H-P-Co fabric.

Figure 10 and Table S3 show that the UPF value of O-Co was only 4.91 and slightly increased to 5.04 after the finishing process. Whereas, the H-P-Co fabric showed a UPF value of 11.91, and increased to

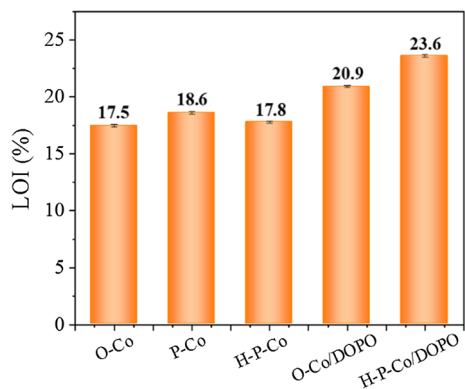


Fig. 9 The limiting oxygen index values of the cotton fabrics

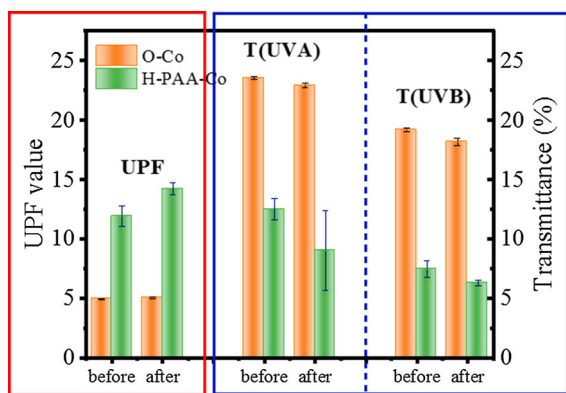


Fig. 10 UPF value, T (UVA) and T (UVB) graphs of the cotton fabrics before and after the adsorption treatment with Cyasorb 2908

14.20 via the simple adsorption of Cyasorb 2908. The increase in the UPF value of the H-P-Co is relatively higher than that of the O-Co, demonstrating that the H-P-Co can absorb more Cyasorb 2908 molecules than the O-Co. Furthermore, after washing for 10 times, the UPF value of H-P-Co/C/Wa (12.94) remained significantly higher than that of the H-P-Co, while the UPF value of O-Co/C/Wa (4.93) was almost identical to that of the O-Co, suggesting that the UV resistance achieved by going through the same UV-resistant finishing processes would last longer on the H-P-Co fabric than on the O-Co fabric.

Conclusion

A lipid layer was constructed on cotton fiber surfaces through a simple two-step esterification process that

involved first covalently linking PAA chains to the cellulose molecules on the fiber surface. Next, the residual carboxyl groups of the PAA chains reacted with the hexadecanol molecules. The hydrophobic coating grafted on cotton fiber surfaces offers a versatile approach to fabricate functional fabrics via the simple pad-dry-cure process. Upon adsorption of STAB, a remarkable antibacterial effect (i.e., near 100% BR values against both *S. aureus* and *E. coli*) was achieved on the cotton fabric. The pad-dry-cure process using DOPO impacted the cotton fabric with outstanding flame retardant capability. The LOI value of the modified fabric was enhanced to 23.6 versus 17.5 for the untreated sample. Similarly, the UV-resistant function was established by adsorbing the common light stabilizer Cyasorb 2908. All of these results demonstrate that the lipid layer grafted on the fiber surface has great potential for a wide range of functional fabrics.

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Declarations

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

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