Preparation and Test of UV Resistant and Flame Retardant Cotton Fabric With Phytic Acid, Tannic Acid and Diethylenetriamine as Raw Materials

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Abstract

Although cotton fabric is widely used in various fields because of its unique advantages, it has the disadvantages of flammability and poor ultraviolet protection. By combining diethylenetriamine(DETA) and phytic acid (PA) and tannic acid(TA) on cotton fabric, a chemical reaction intumescent flame retardant cotton fabric with anti-ultraviolet and anti-flame retardant was developed. The flame retardant and ultraviolet resistance of cotton fabric were characterized by limiting oxygen index (LOI) test, vertical combustion test, cone calorimetry test and ultraviolet resistance test. Scanning electron microscopy (SEM), X-ray photoelectron spectroscopy (XPS), Fourier transform infrared spectroscopy (FT-IR) and other tests were used to analyze the chemical composition, surface morphology and residual carbon after combustion of the cotton fabric, and it was confirmed that the modified cotton fabric has good ultraviolet resistance and flame retardant performance. In this study, an eco-friendly cotton fabric treatment method was proposed, which made cotton fabric have anti-ultraviolet and flame retardant properties, and a new application of tannic acid and phytic acid in ultraviolet protection and flame retardant of fabric was put forward.

1. Introduction

Characterized by great comfort, air permeability, hygroscopicity and tenderness, the cotton fabrics, which have an affinity for human skin, are widely used in clothes, decoration, technical fabrics and packaging(zhang et al. 2021; Sittinun et al.2021;Li et al.2020). Due to its rapid combustion, the cotton fabrics are very dangerous while burning. Fire disasters caused by them have posed serious threats to the life and property security of humanity(Li et al.2020;Nabipour et al.2020;Guo et al.2020;Li et al.2019; Taherkhani et al.2018;Cheng et al.2020). What's more, compared to synthetic textiles, the cotton textiles have a poorer protection capacity against ultraviolet rays, and the UV protection factors of them are often lower than 15(Kan et al.2014). Appropriate exposure to sunshine is favorable for health while inappropriate exposure to sun may do some harm to human body. According to different wave length, the UV radiation is divided into three types: UVA,UVB and UVC. Usually, the ultraviolet rays and the main part of the high energy ultraviolet from the sun will be absorbed by ozone layer. UVA and UVB, which have high energy and could penetrate deeply into dermis, are the two main ultraviolet rays that could reach the ground. Long-time exposure to ultraviolet rays will accelerate the skin aging of humanity, causing premature wrinkles(Kappes et al.2006;Choi et al.2010;Honig et al.2018). And it will increase melanin and lead to various kinds of skin diseases and skin cancers. Presently, however, there are only few researches of modification that enable cotton textiles to have great fame retardation and UV resistance, most could only have one capacity. It is rarely reported that cotton fabric has flame retardancy and UV resistance at the same time. Therefore, it has become an important issue that how to empower flame retardation and UV resistance to cotton textiles.

But in the past few decades,The most common and widely used productive method is to use fire retardant that is based on halogen and includes methanal to make cotton textiles that have the capacity of flame retardation(Zheng et al.2019;Castellano et al.2019). Halogen-based flame retardants, which

produce a large amount of harmful gases during combustion, are gradually replaced by phosphorus-containing flame retardants(Shen et al.2013;Zhong et al.2007;Li et al.2019). In recent years, intumescent flame retardant system has attracted more and more attention because of its synergistic flame retardant. Due to the synergistic effect of its three components, namely acid source, gas source and carbon source, it shows remarkable flame retardancy(Ge et al.2012;Fang et al.2015). In this paper, chemical reaction intumescent flame retardant cotton fabric was prepared with matrix cotton fabric as carbon source, diethylenetriamine as gas source and phytic acid as acid source. In order to improve the flame retardant effect and applicability of the intumescent flame retardant system, tannic acid was added as another carbon source. After tannic acid was added, the system also enjoyed the anti-ultraviolet performance.

As an environmental friendly, nontoxic biocompatible acid, phytic acid has been put into wide application(Jiang et al. 2012). Under thermal decomposition, it could induce cotton textiles to form coke by releasing carboxylic acid, phosphoric acid and sulfuric acid(Liu et al. 2019; Cheng et al.2020). The more we add phosphorus to cotton textiles, the better is their flame retardation ability, and the molecular weight of P of phytic acid is around 28% (C. F. Cullis et al.1992;Zhou et al.2015). Besides, according to the mechanism of flame retardation, the synergistic effect of N and P performs better in flame retardation(Gaan et al.2008).

Tannic acid, featured with special characters such as antibacterial property, inoxidizability, the ability to precipitate protein, reducing capacity and UV resistance, is a natural phenolic compound found in many plants, and most can be found in the bark and performs well in fire resistance(Baron et al.2019;Nam et al.2017;Tributsch et al.2008;Majumdar et al.2015;Ma et al.2017;Ekambaram et al.2016). While caught in fire, the acid could effectively reduce oxidizing agent and free radical and maximize the unburned solid and burned reserves, thus helping the trees survive(Tributsch et al.2008).

So, through a low-cost, simplistic, efficient and chemically stable method, we take ecologically friendly and sustainable materials like phytic acid, Tannic acid and diethylenetriamine as our raw materials to produce chemical reaction intumescent flame retardant cotton fabric that have the capacity of flame retardation and UV resistance. The phytic acid, when used alone, can not have an ideal effect in the improvement of the flame retardation of cotton textiles and is unable to guard against ultraviolet rays. However, after adding adding tannic acid and diethylenetriamine can form intumescent flame retardant system, which can improve the flame retardant performance of cotton fabric and make it have the ability of anti ultraviolet at the same time. Phytic acid, tannic acid and diethylenetriamine are grafted onto cotton fabric by chemical reaction, so the adhesion is more reliable. This article dives into the chemical structure, flame retardation capacity and UV resistant ability of modified cotton textiles as well as the relevant mechanisms.

2. Experimental

2.1 Materials and Methods

2.1.1 Reagents and materials

See Table 2.1 for details of reagents and materials.

2.1.2 Preparation of Anti-ultraviolet and Flame Retardant Cotton Fabric

The first is the preparation of aminated cotton fabric. $30 \times 8 \text{ cm}^2$ cotton fabric was soaked in a beaker containing a mixed solution of 30ml N,N-dimethylformamide (DMF) and 20ml epichlorohydrin, and the beaker was placed in an oil bath with a water temperature of 85°C to react for 1.5 h; Then, 1.5 ml of diethylenetriamine (DETA) was slowly dropped into a beaker, the solution was uniformly stirred, and the obtained cotton fabric was taken out after reacting for 1 h; The cotton fabric was washed several times with ethanol, and then the cotton fabric was repeatedly washed with distilled water to wash off the organic solvent on the surface of the cotton fabric; Finally, the sample was completely dried in an air drying oven at 70°C to obtain ammoniated cotton fabric (CF-DETA).

Ammoniated cotton fabric was added into 100ml 0.01 mol/L tannic acid solution for 1h, and put into an air drying oven at 70°C to completely dry, thus obtaining cotton fabric named CF-DETA-TA; 4ml of 50% phytic acid was added to 100ml of distilled water and stir evenly; Then, CF-DETA-TA was added into phytic acid solution, and the reaction was shaken in a shaking table for 2 hours; Finally, the cotton fabric was dried in an air drying oven at 70°C to obtain flame retardant cotton fabric named CF-DETA-TA-PA.

In addition, the influence of different concentrations of DETA-TA-PA (DTP) on the flame retardant properties of cotton fabrics was studied. The content of DTP was reduced to 25%, 50%, and 75% of the original, and the flame retardant cotton fabrics were prepared by the same preparation method.

2.2 Characterization and Performance Test of Modified Cotton Fabric

(1) Limiting oxygen index (LOI) test: LFY-606B oxygen index meter was used to check the LOI of cotton fabric and modified cotton fabric. The sample was cut to $150 \times 58 \text{ mm}^2$ according to the standard GB/T5454-1997.

(2) Vertical combustion test: LFY-601A vertical combustion tester was used to carry out vertical combustion test on the sample. According to the standard GB/T5455-2014, the size of the tested cotton fabric is $300 \times 80 \text{ mm}^2$, and the flame length is 40mm.

(3) Cone calorimetry test: The combustion performance of raw cotton fabric and modified cotton fabric was measured by cone calorimeter, and the measured heat flux was 35 kW/m^2 . According to the standard ISO 5660-1, the size of the tested cotton fabric is $100 \times 100 \text{ mm}^2$, and each sample should be checked twice.

- (4) Ultraviolet protection performance test: The Ultraviolet Protection Factor (UPF) of the sample was measured on the HD902C ultraviolet protection and sun protection test system, with a wavelength of 280–400 nm and an interval of 5 nm.
- (5) Scanning electron microscope (SEM) test: The morphology of cotton fabric, modified cotton fabric and burned cotton fabric was characterized by JEOL JSM-IT300A SEM under 15 kV voltage.
- (6) X-ray photoelectron spectroscopy (XPS): The elements on the surface of cotton fabrics and modified cotton fabrics were characterized by Thermo Scientific ESCALAB 250 Xi spectrometer with Al K α X-ray source (1484 eV, 300 W).
- (7) Fourier Transform Infrared Spectroscopy (FT-IR): The FT-IR spectra of carbon residue in cotton fabric, modified cotton fabric and burned cotton fabric were obtained on a FTIR spectrometer (VERTEX70) equipped with ATR accessories. The FT-IR spectrum was scanned 64 times at intervals of 4.0 cm⁻¹ in the range of 500–4,000 cm⁻¹.
- (8) Thermogravimetric (TG) test: The thermal stability of the original and treated cotton fabrics was analyzed by Pyris 1 TG analyzer. Under nitrogen conditions, the heating rate was 20k/min and the temperature was 20–700°C.
- (9) Mechanical properties test: The tensile strength of samples was studied by HD026PC multifunctional electronic fabric strength tester. According to standard GB/T 3923.1–2013, the size of each sample was prepared as 30 cm × 6 cm. One end of the sample was fixed on the clamp, and the other end naturally fell on the next sample clamp for testing. Then the tensile strength and elongation at break of paper and paperboard at room temperature were tested at a speed of 25mm/min.

3. Results And Discussions

3.1 Analysis of Flame Retardant Performance

In order to visually show the flammability of modified cotton fabrics, LOI tests were carried out, and the results were summarized in Table 3.1. Compared with pure cotton fabric, the LOI value of modified cotton fabric is improved, which is as follows: the LOI value of unmodified cotton fabric was 16.5% (Zhang et al.2018); the flame retardant property of cotton fabric was slightly improved with the addition of DETA, and its LOI value was 20%; the LOI value of CF-DETA-TA was 25%, and that of CF-DETA-TA-PA was 34%. The cotton fabric treated with DETA-TA-PA (DTP) can not burn in normal atmosphere, which reaches the LOI value of flame retardant standard cotton fabric (26.0%). Through the vertical combustion test, it can be seen from Table 3.1 that the cotton fabric burns rapidly within 12 s after being ignited as it is, and the afterflame time and afterglow time are 12s and 14s respectively. The addition of DETA can improve the flame retardancy of cotton fabric, and the residual flame time and afterglow time are 10s and 10s, respectively, with burning time slightly shortened. Compared with the control sample, the flame retardancy of CF-DETA-TA is improved. Although CF-DETA-TA burns completely, the residual flame time is

lower than the control sample, and the afterglow time is 0 s. As can be seen from Table 3.1, the damage length of CF-DETA-TA-PA is 75 mm respectively, and the afterfire and afterglow time are both 0 s, indicating that the flame can be extinguished immediately after leaving the fire source. Therefore, the samples treated with DTP have excellent flame retardancy.

The LOI values of samples at different DTP concentrations were further studied, as shown in Table 3.2. When the sample is treated with 50% DTP, the weight of cotton fabric increases by 7.30%, and the LOI value of cotton fabric is 27%, which is higher than the LOI value of flame retardant standard (26.0%). In addition, with the increase of DTP concentration, the LOI value of samples treated with DTP also increased. LOI test results show that the modified cotton fabric has excellent flame retardancy.

3.2 Cone Calorimetry Test

Cone calorimetry test is often used to evaluate the combustion behavior of different materials under specific conditions and provide complete flame retardant characteristics(Zhang et al. 2021;Xu et al.2020), and the test results are shown in Table 3.3. Fig. 3.1 is a graph of heat release rate (HRR), total heat release (THR), total smoke production (TSP), smoke production rate (SPR), COP and CO₂P of the sample. As can be seen from Fig. 3.1, the HRR and THR values of the modified cotton fabric are lower than those of the raw cotton fabric. As can be seen from Table 3.3, the Av-HRR value of the modified cotton fabric is reduced and the amount of carbon residue is increased, i.e. the carbon formed during combustion of the modified sample limits the transfer of combustible volatiles and heat, resulting in lower THR value. The PkHRR value of raw cotton fabric is 117.9 kW/m², while the PkHRR value of CF-DETA-TA-PA is much lower, which is 10.4 kW/m². Similarly, the TTI value of CF is 16s, while CF-DETA-TA-PA cannot be ignited. The results show that the flame retardancy of the modified cotton fabric is obviously improved.

Smoke intrusion is the main cause of death during fire (Giebultowicz et al.2017). As can be seen from Fig. 3.1 (c) and (d), the smoke production rate of CF-DETA-TA-PA in the first 50s is higher than that of the raw cotton fabric, but the SPR of the modified cotton fabric after 50s is very low, and the total smoke production is much lower than that of the control sample. In other words, CF-DETA-TA-PA not only has good flame retardancy, but also has low smoke exhaust amount, which may be due to the positive influence of phosphoric acid or polyphosphoric acid generated during combustion on the pyrolysis of cellulose, resulting in early pyrolysis of cellulose. As can be seen from Fig. 3.1 (e) and (f), the COP value of the modified cotton fabric is higher, while the CO₂P value is much lower than that of the control. The high COP value may be caused by incomplete combustion of coated cotton fabric, especially when phytic acid burns, phosphoric acid or polyphosphoric acid can be produced, which catalyzes cellulose to generate carbon residue, resulting in incomplete combustion of cotton fiber. The above results prove that the modified cotton fabric has not only flame retardant performance, but also smoke suppression effect.

3.3 Analysis of Anti-UV Performance

According to Table 3.4, the UPF value of raw cotton fabric is 11.1(Zhong et al.2007;Farouk et al.2021;Pandiyarasan et al.2017), and the UPF values of CF-DETA, CF-DETA-TA and CF-DETA-TA-PA are

20.7, 99.2 and 131.8 respectively, which are slightly improved. The ultraviolet transmittance of CF-DETA-TA-PA is less than 2.32%. According to AS/NZS 4455 miniclip UPF classification system, CF-DETA-TA-PA is considered to have good UV protection performance. The results show that the modified cotton fabric not only has flame retardancy, but also has a good potential application prospect in ultraviolet protection materials (Pandiyarasan et al.2017).

3.4 Analysis of Surface Morphology

Fig. 3.2 is a SEM image of raw cotton fabric and modified cotton fabric. The raw cotton fabric showed a smooth surface, but after grafting DETA, it was found that the concavity and convexity of the surface increased after the graft copolymerization reaction, indicating that the organic monomer was grafted into the cellulose framework, as shown in Fig. 3.2 (b₁, b₂). As shown in Fig. 2.2 (c₁, c₂), after covering the tannin layer, the surface of cotton fiber is rough, and the surface of CF-DETA-TA-PA is relatively smooth than that of CF-DETA-TA, but some cracks appear locally, which indicates that a layer of phytic acid is deposited on the surface of cotton fabric. The above results indicate that the coating was successfully deposited on cotton fabric (El-Shafei et al.2015;Li et al.2015).

3.5 Analysis of X-ray Photoelectron Spectroscopy

Fig. 3.3 is a full-range XPS image of raw cotton fabric after modification, which shows that the surface chemical composition of cotton fabric has completely changed before and after modification. Two peaks of 285.5eV and 532.4 eV are shown in the spectrum, which are attributed to C 1s and O 1s, respectively. In contrast, the two peaks of CF-DETA at 402.5 eV and 399.4 eV correspond to N1s, which is due to the N element contained in DETA. Two new peaks of 191.5 eV and 133.9 eV appeared in CF-DETA-TA-PA, which correspond to P2s and P2p, confirming the successful grafting of DETA and phytic acid on the cotton fabric surface (Przybylak et al.2016).

Fig. 3.4 (a), (b), (c) and (d) are high resolution XPS images of C1s, O1s and N1s and P2p, respectively. In the C 1s high resolution XPS spectrum, it is divided into three carbon states at 284.5eV, 285.6eV and 288.1 eV, which are divided into C-C/C-H, C-O/C-N and C-O-C respectively. The O1s XPS spectrum and characteristic peaks are shown in Fig. 3.4 b. The O 1s peak is divided into three peaks of 533.1 eV, 532.6 eV and 532.0 eV, which are divided into C-O/P-O, C-O-C and P=O, respectively. The N 1s peak is divided into two peaks of 402.5 eV and 399.4 eV, corresponding to C-N-C and C-N, respectively, which are consistent with the previous report.

3.6 Analysis of FT-IR Spectra

Fig. 3.5 is the FTIR spectra of raw cotton fabrics and modified cotton fabrics. The peak of raw cotton fabric at 3330cm⁻¹ is attributed to the stretching vibration of hydroxyl groups in cotton fiber, the peak at 2895cm⁻¹ corresponds to the stretching vibration of C-C, the peak at 2876cm⁻¹ corresponds to the stretching vibration of C-H, and the peaks at 1431 cm⁻¹ and 1335 cm⁻¹ correspond to the bending vibration of CH₂ and CH on the main chain of cotton fabric, respectively, and the characteristic peak at

1031 cm^{-1} corresponds to the stretching vibration of C-O-C in cellulose. After DETA treatment, many peaks originally existing in cotton fabric were weakened: the peak formed at 1626 cm^{-1} corresponds to the stretching vibration of C-N, and the peak at 1261 cm^{-1} proves the existence of N-H on cotton fabric. The peak at 2963 cm^{-1} corresponds to the stretching vibration of the phenolic hydroxyl group after the addition of tannic acid, and the peak at 869 cm^{-1} corresponds to the bending vibration of the benzene ring plane. In CF-DETA-TA-PA, the absorption peak at 1760 cm^{-1} corresponds to the stretching vibration of P=O, and the peak at 1196 cm^{-1} corresponds to the stretching vibration of P-O. The change of absorption peak position and intensity indicates that some substances are loaded on the surface of cotton fabric, which is consistent with the previously reported results (El-Shafei et al.2015).

3.7 TG Analysis

The thermal stability and thermal oxidation stability of control samples and treated samples were evaluated by N₂ TG analysis. Fig. 3.6 is a TG and DTG diagram of the sample in N₂. Table 3.5 shows where the decomposition rate of the modified cotton fabric is lower than that of the control cotton fabric, and the carbon residues of the CF, CF-DETA, CF-DETA-TA, CF-DETA-TA-PA are 4.3%, 23.6%, 23.2%, and 43.0%, respectively. Phosphoric acid or polyphosphoric acid produced in the thermal degradation process of phytic acid is beneficial to the decomposition of cellulose into carbon slag, forming an insulating dense protective layer, which can prevent convection and conduction of heat and materials and reduce the decomposition temperature. The dense layer is also beneficial to increase the carbonization yield during combustion. The analysis results show that the decomposition rate of modified cotton fabric decreases obviously.

The maximum weight loss rate of the control sample at 353°C is 25.7%/°C, and about 4.3% thermally stable carbon residue is formed at 700°C. The modified cotton fabric is still a one-step thermal degradation process, and CF-DETA and CF-DETA-TA almost show the same thermal behavior. It can be seen from the table that the decomposition temperatures of cotton fabrics treated by DETA, DETA-TA and DETA-TA-PA are 210°C, 223°C and 208°C, respectively, which are significantly lower than those of control samples, indicating that flame retardants have significant influence on the decomposition behavior of products, which is beneficial to preferential decomposition and charring. Similarly, the T_{max} and $T_{10\%}$ values of the treated cotton fabric are also low. In addition, the R_{max} value of all coated cotton fabrics is low, which makes the modified cotton form more carbon residue than the control cotton, thus preventing the cotton fabric from further burning and releasing flammable volatile substances, and producing less volatile substances and combustible substances in the thermal degradation process. The data show that the modified cotton fabric has better thermal stability.(Alongi et al.2013;Lessan et al.2011)

3.8 Surface Morphology Analysis of Residual Carbon

Fig. 3.7 is an electron micrograph of CF and CF-DETA-TA-PA before and after combustion. Untreated samples are easy to break after burning, which can not keep the original morphology, indicating that pure cotton fabric is flammable. Fig. 3.7 (b₁, b₂) shows the morphology of modified cotton fabric after

combustion, that the shape of the sample remains intact and the residual carbon skeleton structure still exists clearly. There are many small bubbles on the surface of the carbon residue skeleton structure of CF-DETA-TA-PA, which is due to the decomposition of phytic acid to form phosphoric acid and pyrophosphate. (Tao et al. 2021)

3.9 Infrared Analysis of Combustion Samples

Fig. 3.8 shows the infrared spectra of CF and CF-DETA-TA-PA after combustion. For pure cotton fabric after burning, there is a peak at 1027 cm^{-1} , which corresponds to the stretching vibration of cotton fabric C-O-C. For the treated cotton fabric burning, the peak at 1013 cm^{-1} corresponds to the stretching vibration of the cotton fabric C-O-C, the peak at 2929 cm^{-1} corresponds to the stretching vibration of the cotton fabric C-C, the peak at 1219 cm^{-1} corresponds to the stretching vibration of P=O, and the peak at 1614 cm^{-1} corresponds to the stretching vibration of P-O. The analysis results show that the treated cotton fabric can keep a more complete structure after burning. (Jiang et al. 2019; Chen et al. 2019)

3.10 Analysis of Mechanical Properties

The mechanical properties of the control sample and the treated sample were tested. Fig. 3.9 is a stress-strain graph of cotton fabric before and after modification. The tensile strength of cotton fabric treated by DETA is higher than that of the original cotton fabric, and the tensile strength increases from 24.4 MPa to 27.1 MPa, but the elongation at break decreases. After adding tannic acid, the tensile strength of cotton fabric is 26.6 MPa, and that of CF-DETA-TA-PA is 24.0 MPa, which can be seen that the addition of phytic acid makes the tensile strength of cotton fabric decrease. Generally speaking, the tensile strength and elongation at break of cotton fabric decreased slightly after modification. (Tania et al. 2020; Muzaffar et al. 2021)

4. Summary

In this paper, a cotton fabric with anti-ultraviolet and flame retardant was prepared by grafting DETA, tannic acid and phytic acid on the surface of cotton fabric. The LOI value of unmodified cotton fabric is 16.5%, while that of treated cotton fabric can reach 34%, which meets the flame retardant standard of cotton fabric. TG test shows that at $700\text{ }^{\circ}\text{C}$, the residual mass of treated cotton fabric is 43.0%, while that of raw cotton fabric is 4.3%. THR, PkHRR and TSP of treated cotton fabric are obviously lower than those of untreated cotton fabric, which indicates that modified cotton fabric can effectively inhibit cotton burning. The UPF value of modified cotton fabric is 131.8, and the ultraviolet transmittance is less than 2.32%. The tensile strength of the treated cotton fabric has no obvious change, but the elongation at break decreases. The morphology and structure of burned cotton fabric were analyzed by SEM and FTIR, and it was found that the modified cotton fabric could keep a more complete structure after burning. Therefore, the improvement of flame retardancy and UV resistance of modified cotton fabric has broad application prospects.

Declarations

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Conflict of interest The authors declare that they have no conflicts of interest to declare.

Ethical Standards This article does not contain any studies with human participants or animals performed by any of the authors.

Informed consent None.

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Tables

Table 2.1 Reagents and materials

Experimental materials	chemical formula	Specifications	manufactor
tannic acid	$C_{76}H_{52}O_{46}$	Analytically pure	Aladdin Industrial Corporation
phytic acid	$C_6H_{18}O_{24}P_6$	Analytically pure	Aladdin Industrial Corporation
epoxy chloropropane	C_3H_5ClO	Analytically pure	Sinopharm Chemical ReagentCo., Ltd
N. N-dimethylformamide	C_3H_7NO	Analytically pure	Sinopharm Chemical ReagentCo., Ltd
Diethylenetriamine	$C_4H_{13}N_3$	Chemical purity	Sinopharm Chemical ReagentCo., Ltd
ethanol	C_2H_5OH	Analytically pure	Sinopharm Chemical ReagentCo., Ltd
acetone	CH_3COCH_3	Analytically pure	Sinopharm Chemical ReagentCo., Ltd

Table.3.1 Data from LOI and vertical burning tests of untreated samples and treated cotton fabric.

sample	LOI% ^a	Afterglow time ^b (s)	Afterglow time ^c (s)	Loss length ^d (mm)
CF	16.5	12	14	300
CF-DETA	20	10	10	300
CF-DETA-TA	25	8	0	300
CF-DETA-TA-PA	34	0	0	75

Table.3.2 LOI values of samples treated with different concentrations of DTP.

sample	Weight gain ^a (wt%)	LOI% ^b
25%DTP	3.11	25.5
50%DTP	7.30	27
75%DTP	12.72	31
100%DTP	16.72	34

Table.3.3 Cone calorimetry results of samples.

sample	TTI(s)	PkHRR(kW/m ²)	TTPHRR(s)	AvHRR(kW/m ²)	THR(MJ/m ²)	TSP(m ²)
CF	16	117.9	35	15.4	4.3	1.8
CF-DETA-TA-PA	-	10.4	265	6.0	1.8	0.6

Table.3.4 UPF values of the samples

样	UPF	T(UVA)%	T(UVB)%	T(UVR)%
CF	11.1	11.92	6.77	10.41
CF-DETA	20.7	5.94	10.53	7.32
CF-DETA-TA	99.2	2.57	3.07	2.74
CF-DETA-TA-PA	131.8	2.63	1.51	2.32

Table.3.5 Data from TG and DTG curves of the control and modified cotton fabrics in N₂.

Samples	T _{5%} (°C)	T _{10%} (°C)	T _{max} (°C)	R _{max} (%/°C)	Residue at 700°C(%)
CF	244	323	353	25.7	4.3
CF-DETA	210	243	290	5.2	23.6
CF-DETA-TA	223	247	289	4.9	23.2
CF-DETA-TA-PA	208	245	275	10.6	43.0

Figures

Figure 1

3.1 HRR (a), THR (b), SPR (c), TSP (d), COP (e) and CO₂P (f) curves of samples.

Figure 2

3.2 SEM images of (a1, a2) CF, (b1, b2) CF-DETA , (c1,c2) CF-DETA-TA and(d1,d2) CF-DETA- TA-PA.

Figure 3

3.3 XPS survey spectra of pristine cotton fabric and modified cotton fabrics.

Figure 4

3.4(a) C 1s, (b) O 1s, (c) N 1s and P 2p spectra of CF-DETA- TA-PA.

Figure 5

3.5 FTIR spectra of TA and TA-Cu

Figure 6

3.6 TG and DTG curves of control cotton and modified cotton fabric in nitrogen

Figure 7

3.7 SEM image of (a₁,a₂)CF and (b₁,b₂)CF-DETA-TA-PA after burning

Figure 8

3.8 FTIR spectra of TA and TA-Cu after burning

Figure 9

3.9 Strain–stress curves of control and treated samples