

Research Article

Durable Flame-Retardant Cotton Fabric Modified by a Novel Reactive P-N Intumescent Flame Retardant

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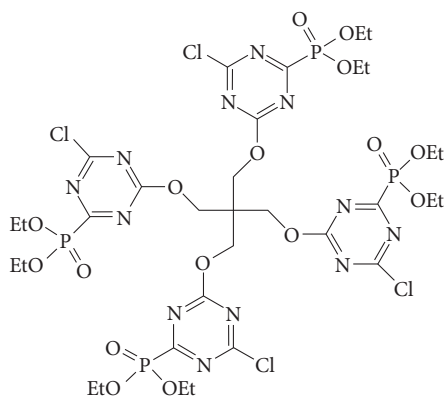
A novel reactive P-N intumescent flame retardant tetroxo (3-triethylphosphine-5-chlorine-1-triazine) neopentane (TTCTN) was prepared, and its structure was determined by Fourier transform infrared (FTIR) and nuclear magnetic resonance (NMR). The flame retardance and physiological comfort properties of TTCTN-treated cotton fabrics (TTCTN-CF) was researched. The CF treated with 20 wt% TTCTN (TTCTN(20)-CF) showed the limiting oxygen index (LOI) was up to 28.6%, and the flame retardancy reached the class B1 of national standard of flame retardant fabrics, and its peak heat release rate and total heat release were reduced by 55.7% and 37.4% comparing with untreated CF, respectively. The FTIR proved the TTCTN was grafted on the CF matrix. Thermogravimetric (TG) showed TTCTN could reduce the decomposition speed of the cotton fabric and formed the stable char residue at high temperature to protect the remaining CF from complete dehydration. After 50 washing cycles, the LOI of TTCTN(20)-CF was 26.2%, still reaching class B1 of national standard of flame retardant fabrics. Scanning electron microscope showed the TTCTN was still bounded to the matrix of CF after 50 washing cycles which further proved TTCTN(20)-CF had excellent durable flame retardancy.

1. Introduction

Cotton fabrics (CF) are widely used as an important biopolymer due to their excellent properties, for example, biodegradability, mechanical properties, hydrophilicity, and availability in large quantities [1, 2]. However, the ease of their thermal flammability restricted their specific applications in many fields [3]. Therefore, it is indispensable to enhance the flame resistance of the CF.

The halogen-containing flame retardants are used for CF in the early days, for example, tris(2-chloroethyl) phosphate, they exhibit excellent flame retardancy. But they may produce corrosive smoke during combustion and cause serious environmental pollution [4, 5]. The classic cotton flame-retardant agents can improve flame retardancy of CF, such as tetrakis(hydroxymethyl) phosphonium chloride (THPC) and *N*-methylol dimethylphosphono propionamide (MDPA), but the serious free formaldehyde problem of these flame retardants is harmful to human health [6–8]. So, it is urgent for developing environmentally friendly flame retardant for CF.

In recent years, P-N intumescent flame retardants, which are composed of acid source, air source, and carbon source, are paid great attention due to environment friendly and excellent flame retardance for polymer. This is because phosphorus atom is usually used as acid source can form the polyphosphoric acid in combustion, which can promote the carbon-rich compounds, used as carbon source, dehydration and carbonization, enhancing the formation of the protective char layer during combustion and further prevents the pyrolytic process of polymer [9–11]. Moreover, a constant content of the phosphorus atom is combined with the additional nitrogen atom, resulting in the generation of the phosphorus–nitrogen synergism. Nitrogen in an intumescent system is usually used as gas source, which may strongly promote the cross-linking with carbon and produce the incombustible gases during decomposition. Furthermore, the gases produced above can dilute the concentration of the flammable gases and form the foamed-char layers, which serve as the superiorly protective barriers against flaming and heating [12, 13]. At present, the P–N intumescent flame retardants have been used in CF and



SCHEME 1: The chemical structure of TTCTN.

present excellent flame retardancy and demonstrate that they are high-efficiency and eco-friendly for CF [14–17].

Triazine derivatives possess abundant nitrogen atoms and are often used as environmentally flame retardants [18, 19], and the triazine-based flame retardants can endow CF excellent durable flame retardance [20], because the C–Cl of triazine-based flame retardants can react with –OH of CF to form C–O–C covalent bond, the flame retardants are bonded in the matrix of CF. Hence, it is worth researching to develop the new P–N intumescent flame retardants containing triazine group for high-efficiency durable flame retardance of CF based on the advantage of P–N intumescent flame retardants. However, the research on flame retardance of CF treated with P–N intumescent flame retardants containing triazine group is rarely reported.

In this work, a novel P–N intumescent flame retardant containing triazine group (TTCTN) was synthesized with pentaerythritol as carbon source, cyanuric chloride as gas source, and triethyl phosphite as acid source (The structure is presented in Scheme 1). Simultaneously, a 1 mol TTCTN contains 4 mol C–Cl, which can react with the –OH of cotton fibers to form covalent bonds, and the TTCTN is bonded the matrix of cotton fibers, imparting outstanding durable fire resistance to CF.

2. Experimental

2.1. Materials. The pentaerythritol, cyanuric chloride, triethyl phosphite, and diisopropylethylamine were obtained from the Tianda Chemical Reagent Co., Ltd. (Taiyuan, China). Sodium hypophosphite, boric acid, anhydrous ethanol, ethyl acetate, and petroleum ether was purchased from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). CF were supplied by Chun Guang Textile Co., Ltd. (Taiyuan, China).

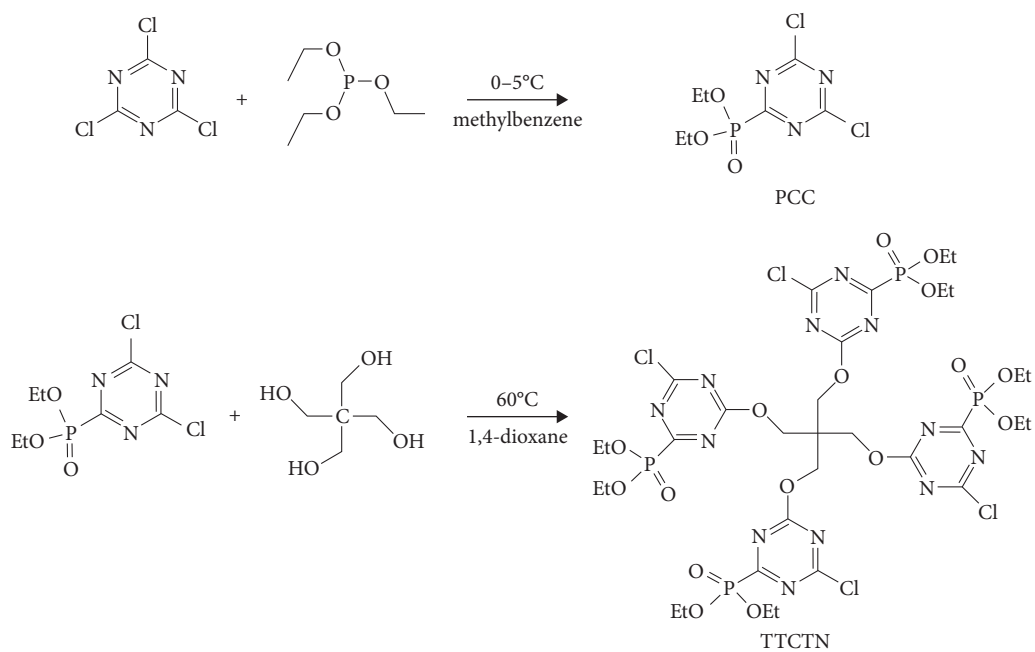
2.2. Preparation of the TTCTN. The intermediate (PCC) was prepared according the literature [21]. Pentaerythritol (1.36 g, 0.01 mol) was added into a 250 mL four-necked flask, then the 60 mL 1,4-dioxane was added. The PCC (12.87 g, 0.045 mol) was dissolved in 100 mL 1,4-dioxane and dropwise added into the four-necked flask at 60°C with vigorous stir. The pH was keeping at 7–8 with *N,N*-diisopropylethylamine during the reaction. After 9 hr, the reaction was stopped and the reaction

solution was filtered and concentrated. The filtrate was chromatographed on the silica gel column eluting with ethyl acetate/petroleum ether (v/v = 1 : 20). The yellow oily liquid was obtained (TTCTN, 9.67 g, yield 85.3%). The synthetic route is presented in Scheme 2.

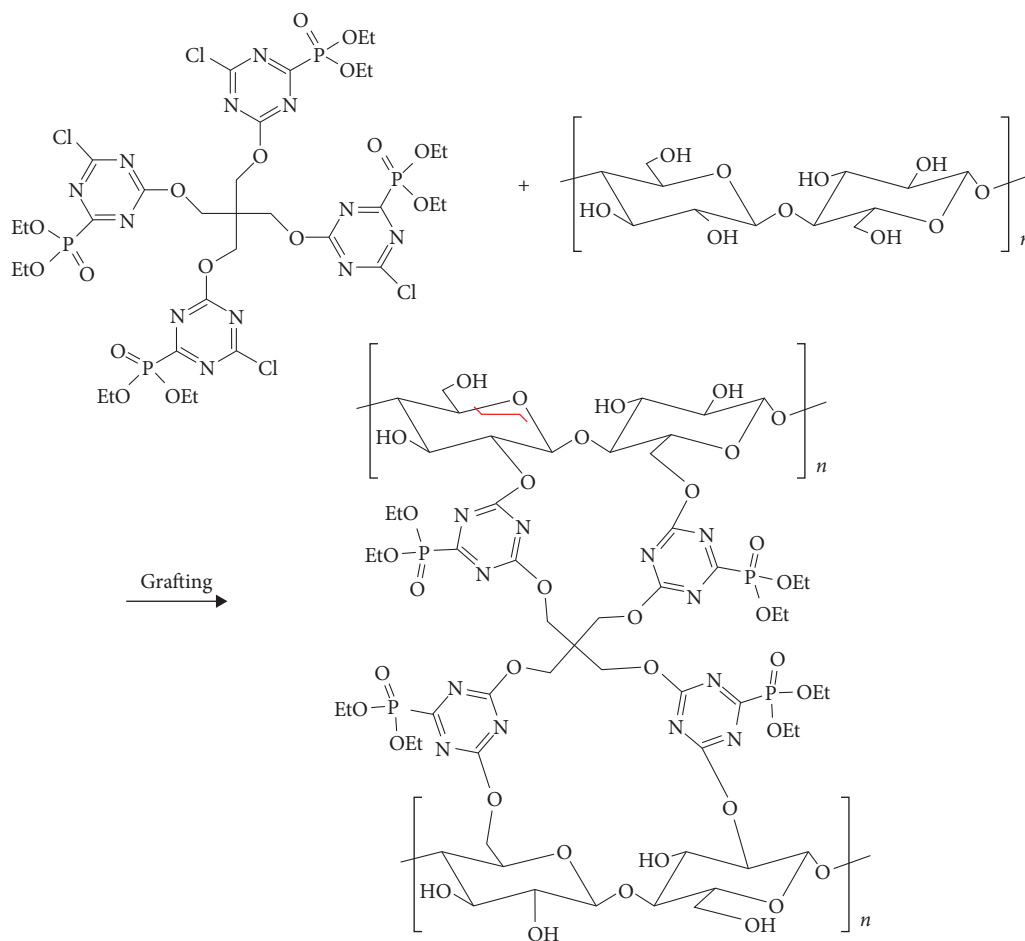
2.3. Fire-Resistant Finishing of Cotton Fabrics with TTCTN. First, the 12 g CF were washed with 25% NaOH solution for three times and dried naturally. Then, TTCTN, sodium hypophosphite and boric acid was added into H₂O/EtOH (v/v, 1 : 4) solution to prepare finishing solution containing different content TTCTN, 5% sodium hypophosphite (SHP) and 5% boric acid (BA). The CF were impregnated in finishing solution stirred for 2 hr at 60°C, then the CF were treated with two impregnations and two presses through a laboratory-scale padder to keep an approximately 80% of wet pickup. The CF treated with TTCTN were dried at 80°C for 5 min and cured at 160°C for 3 min. The grafting reaction between the cellulose and TTCTN is presented in Scheme 3.

2.4. Characterization. The structure and thermal property were characterized by Fourier transform infrared (FTIR) spectroscopy, nuclear magnetic resonance (NMR) spectrometer, and thermogravimetric (TG) analyzer. The TTCTN was characterized by FTIR (Vector-22 FTIR, Bruker, Germany) with the resolution of 4 cm⁻¹ in the range of 400–4,000 cm⁻¹ and NMR (AVANCE III 600, Bruker, Germany) using deuterated dimethyl sulfoxide (DMSO-d₆) as the solvent. Thermal degradation of TTCTN was researched by thermal analyzer (TG-209F3, Nexo, Germany) with heating rate of 10°C min⁻¹, temperatures ranging from 25 to 600°C under N₂ atmosphere.

The flame retardancy of TTCTN-CF were evaluated by limiting oxygen index (LOI), vertical burning test, and cone calorimeter test. LOI value were obtained using the HC-2 oxygen index analyzer (Cangzhou Lisheng Test Instrument Co., LTD) according to GB/T 5454-1997. Vertical burning test was performed by the CZF-5 vertical burning tester (Cangzhou Lisheng Instrument Co., LTD) according to GB/T 5455-1997. CF were classified into different flame retardant rating according to test results: B1 rating (char length ≤150 mm, after-flame time ≤5 s, and after-glow time ≤5 s), B2 rating (char length ≤200 mm, after-flame time ≤10 s, and after-glow time ≤10 s). The cone calorimetry tests (Fire Testing Technology) was performed according to test standard ISO 5660, the samples (100 × 100 mm) were tested under a heat flux of 35 kW m⁻². Thermal decomposition property of TTCTN-CF was researched by thermal analyzer (TG-209F3, Nexo, Germany) with heating rate of 10°C min⁻¹, temperatures ranging from 25 to 600°C under both nitrogen and air atmospheres. The washing durability of TTCTN-CF was evaluated by a soaping fastness tester (Cangzhou Lisheng Instrument Co., LTD) in accordance with AATCC-61-2013 (1 A), and 3.7 g L⁻¹ washing detergent was used. The samples were immersed into the solution containing detergent for 45 min at 40°C, then, the samples were rinsed and dried at 60°C. One washing cycle of accelerated washing treatment corresponds to five times domestic washing. The tensile strength of TTCTN-CF was tested by the HD026 N electronic



SCHEME 2: The synthetic routes of TTCTN.



SCHEME 3: The grafting reaction between cellulose and TTCTN.

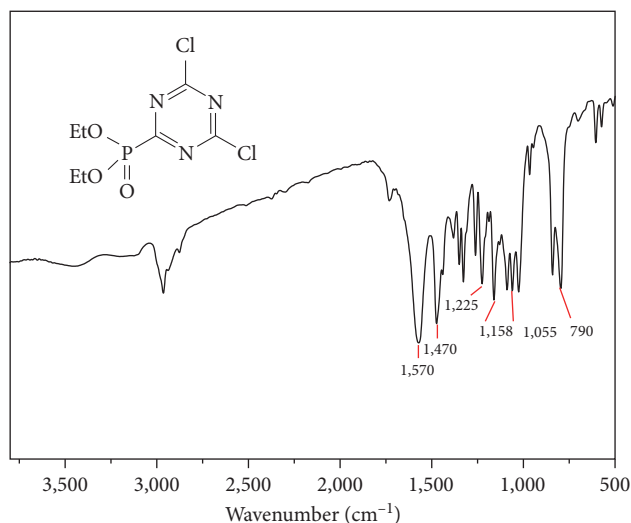


FIGURE 1: The FTIR spectrum of PCC.

fabric tester (Nantong Hongda Experiment Instruments Co., LTD) in accordance with ASTM 5035/2006 standard. The air permeability of TTCTN-CF was tested by the air permeability tester (FX 3300, TEXTTEST AG, Switzerland) according to standard ASTM D2986. The air flow passing through the fabric at 200 Pa pressure was measured. The stiffness of TTCTN-CF was measured by LLY-01B stiffness tester (Shaoxing Electromechanical Technology Co., LTD) according to standard GB/T 18318-2001 to measure the bending force of samples and then stiffness was calculated. Prior to testing of physiological comfort properties, the samples were conditioned in $65 \pm 2\%$ relative humidity and $20 \pm 2^\circ\text{C}$ atmospheres for 24 hr. The surface morphology of TTCTN-CF and char residue was tested by the scanning electron microscopy (SU1510, Hitachi, Japan). For avoiding charge accumulations, all samples were sputter-coated with gold.

3. Results and Discussion

3.1. Characterization of PCC. The chemical structure of PCC was characterized by FTIR and elemental analysis. As Figure 1 shows, the characteristic peaks of PCC were assigned as follows: 839 cm^{-1} (C–Cl), $1,055$ and $1,470\text{ cm}^{-1}$ (C–N and C=N of triazine ring, respectively), $1,158$, $1,225$, and $1,570\text{ cm}^{-1}$ (P–O, P=O and C–O–P of (O)P(OEt)₂, respectively). The assignment of these characteristic peaks showed the PCC should be synthesized.

The structure of PCC was further characterized by elemental analysis, the contents of C, H, and N in PCC are shown in Table 1. The actual contents of C, H, and N were 29.42%, 3.54%, and 14.77%, respectively, which were very close to the theoretical value. The analysis results of FTIR and elemental analysis showed the PCC was prepared successfully.

3.2. Characterization of TTCTN. The chemical structure of TTCTN was characterized by FTIR and NMR. As Figure 2 shows, the characteristic peaks of TTCTN were assigned: 837 cm^{-1} (C–Cl), $1,056\text{ cm}^{-1}$ (C–O–C), $1,161\text{ cm}^{-1}$ (P–O), $1,228\text{ cm}^{-1}$ (P=O), $1,469\text{ cm}^{-1}$ (C=N), and $1,560\text{ cm}^{-1}$

TABLE 1: The elemental analysis results of PCC.

	C	H	N
Actual content (%)	29.42	3.54	14.77
Theoretical content (%)	29.39	3.52	14.73

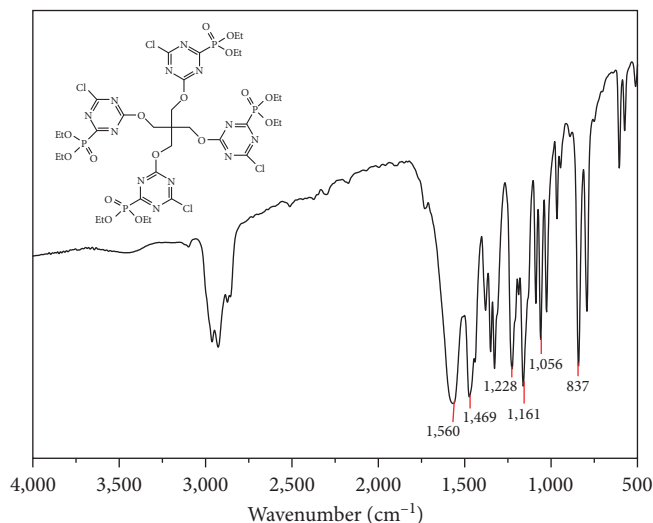


FIGURE 2: The FTIR spectrum of TTCTN.

(C–O–P). The appearance of these characteristic peaks indicated that TTCTN was synthesized.

The structure of TTCTN was further confirmed by NMR, the characteristic proton signals are displayed in Figure 3. δ 1.15 (t, 24H, 8CH₃), δ 3.46 (q, 16H, 8OCH₂), and δ 4.76 (m, 8H, 4CH₂), these signals were consistent with the structure of TTCTN. In addition, the ³¹P NMR showed a single peak at -20.27 ppm (Figure 4), which was characteristic signals of P elements of TTCTN. The results of NMR well proved the TTCTN was synthesized successfully.

3.3. TG Analysis of TTCTN. Thermal decomposition property of TTCTN was characterized under nitrogen atmosphere (Figure 5). It could be seen that there was one degradation stages of TTCTN, the initial degradation temperature was 190°C , the TTCTN was decomposed rapidly between 190 and 270°C , and the maximum decomposing rate temperature was 260°C , thermal decomposition completed about at 400°C for TTCTN. Generally speaking, the starting decomposition temperature of CF was 250 – 300°C [21], it could be seen that the starting decomposition temperature of TTCTN lower than the that of CF, TTCTN could be applied in CF as a flame retardant. During this stage, the TTCTN molecular skeleton was decomposed and released the water, ammonia, and phosphonic radicals. The phosphonic radicals could accelerated the TTCTN decomposing to form char [22]. Figure 5 shows the char residue of TTCTN was 43.7% at 500°C , such high char weight indicated that TTCTN was an efficient char forming agent.

3.4. The Analysis of Structure of TTCTN-CF. Figure 6 presents the FTIR spectra of TTCTN-CF and untreated CF. The characteristic peaks $3,350$, $2,750$ and $1,020\text{ cm}^{-1}$ were the

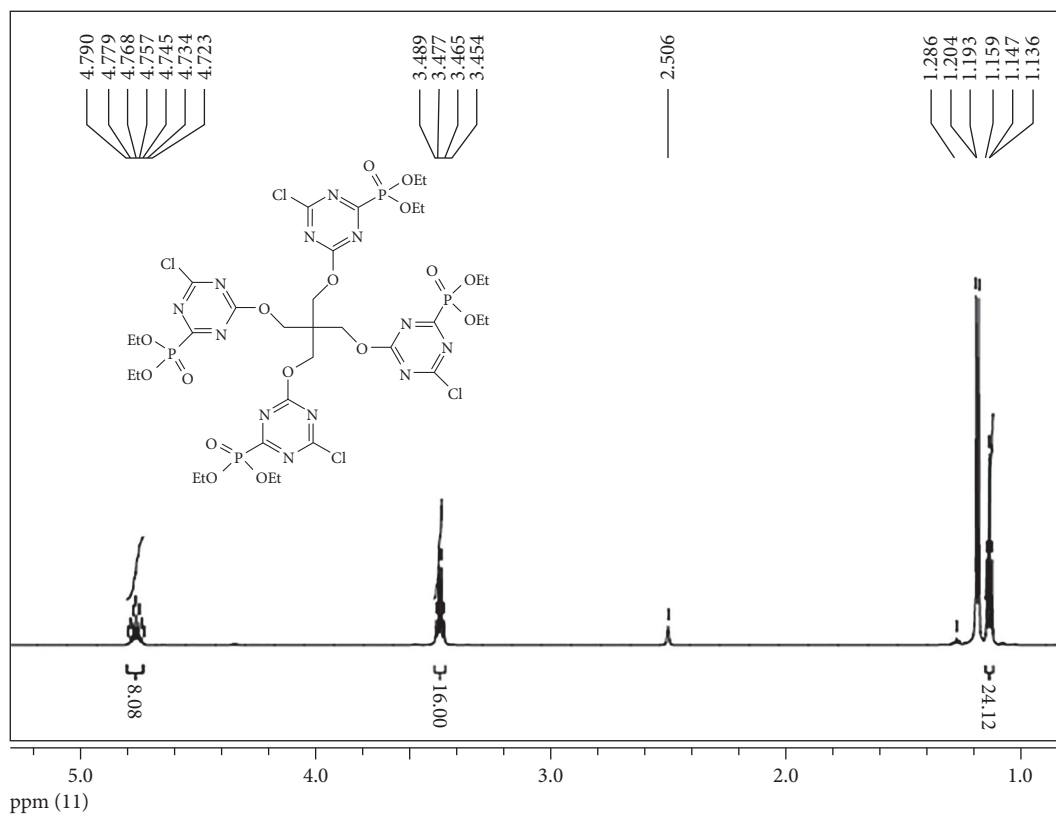


FIGURE 3: ¹H NMR spectrum of the flame retardant TTCTN.

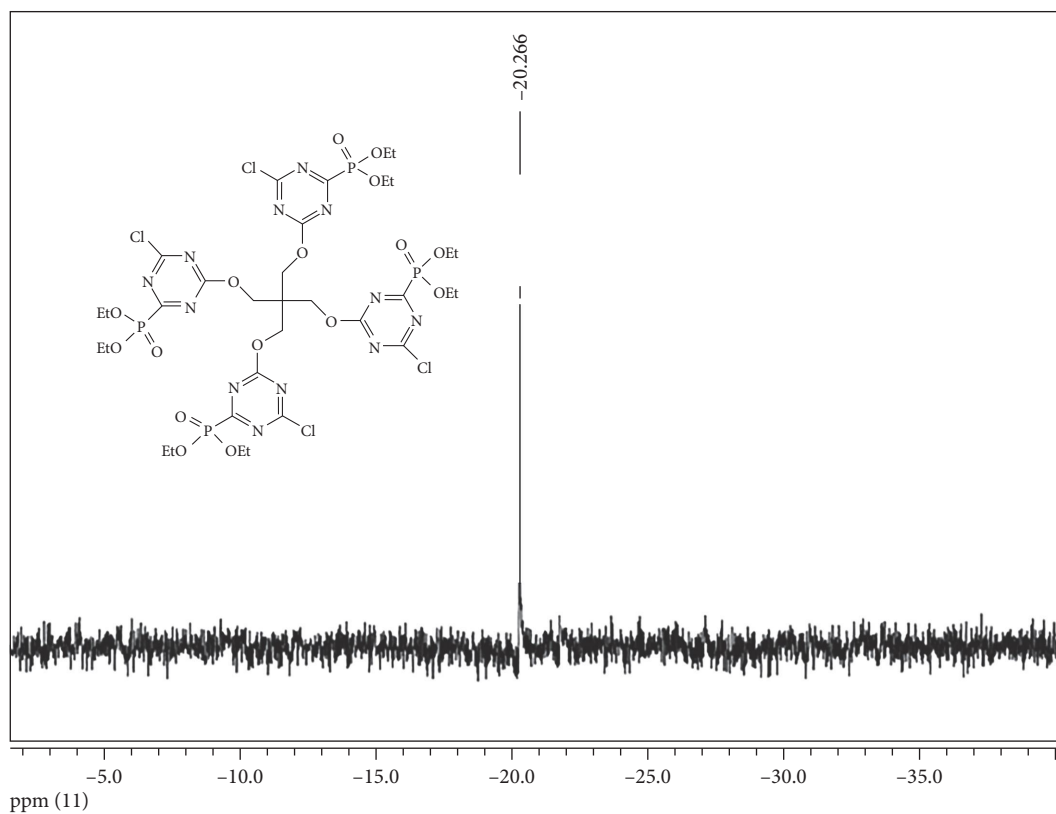


FIGURE 4: ³¹P NMR spectrum of the flame retardant TTCTN.

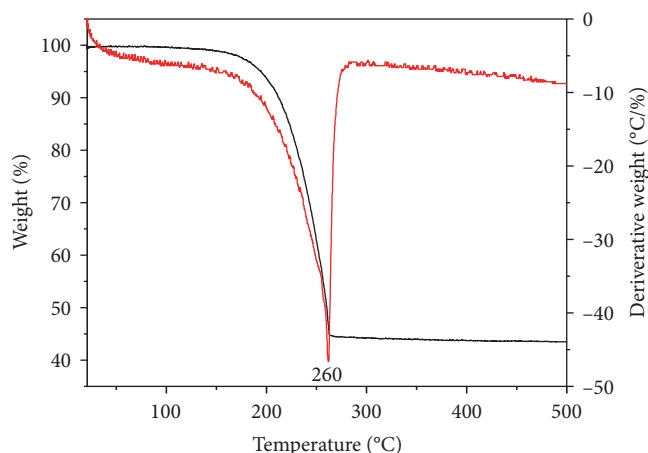


FIGURE 5: TG curves of TTCTN.

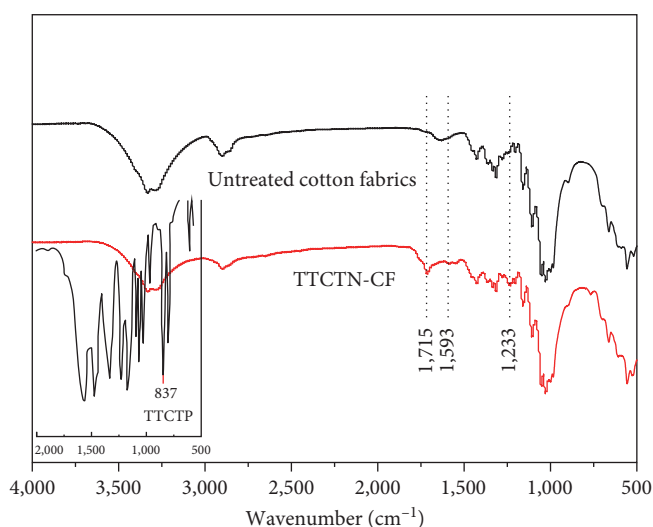


FIGURE 6: The FTIR spectrum of TTCTN-CF.

stretching vibration of the $-\text{OH}$, $-\text{CH}_2$, and $\text{C}-\text{O}-\text{C}$ groups in TTCTN-CF and untreated CF, respectively. It could be seen that there appeared the new peaks at 1,715, 1,593 and $1,233\text{ cm}^{-1}$ in spectrum of TTCTN-CF comparing with the spectrum of untreated sample, which are characteristic peaks of $\text{C}=\text{N}/\text{C}=\text{N}$ in triazin and $\text{P}=\text{O}$ in $(\text{O})\text{P}(\text{OEt})_2$ groups. Simultaneously, the $\text{C}-\text{Cl}$ characteristic peak at 837 cm^{-1} disappeared comparing with the spectra of TTCTN, which indicated that $\text{C}-\text{Cl}$ of TTCTN and $-\text{OH}$ of CF occurred chemical cross-linking reaction, the TTCTN was grafted in the matrix of CF.

3.5. Flame Retardance of the TTCTN-CF. To investigate the flammability properties of the TTCTN-CF with different TTCTN contents, the LOI and vertical burning test were performed. The results are given in Table 2. The LOI value of the TTCTN-CF were raised from 18.9% to 31.8% with the increase of the add-on of TTCTN. The result indicated that the TTCTN was an effective flame retardant. The vertical burning test was also carried out to assess the flammability of the TTCTN-CF, it can be seen that the CF had long

after-flame times and the entire fabric was burned out after combustion. At the same time, TTCTN processed cotton fabric had almost no after-glow burning, and the after-flame time decreases with the increase of TTCTN dosage. When TTCTN content was increased to 20 wt%, the after-glow time and after-flame time were 0 S, the char length was 13.6 cm, and the B1 rating could be reached. These phenomena demonstrated that TTCTN had good flame-retardant effect for CF.

3.6. Tensile Strength and Physiological Comfort Test of TTCTN-CF. Table 3 shows the changes of tensile strength in the warp and weft directions for the CF with different content TTCTN. The CF had tensile strength of 878.7 N in the warp direction and 480.4 N in the weft direction. Along with the increase in TTCTN concentration, the TTCTN treated CF had a slightly decreasing, TTCTN(25)-CF had tensile strength of 792.3 N in the warp direction and 437.2 N in the weft direction. Decreasing not more than 10% in tensile strength when the content of TTCTN was not higher than 25 wt%. The results suggested that TTCTN slightly affected the tensile strength of CF.

The air permeability and stiffness testing were carried out to know the physiological comfort properties of TTCTN-CF. Air permeability is the measure of air flow passed through a given area of a fabric and decides the physiological comfort to the great extent. Table 3 shows the effect of TTCTN concentration on air permeability of CF, it can be seen that there was no significant drop in air permeability with increase in TTCTN concentration up to 25 wt%, the small drop in air permeability of 18.2% was found for TTCTN(20)-CF. However, the air permeability was significantly dropped by 32.7% with further increase in TTCTN concentration beyond 30 wt%, this behavior could be explained that the TTCTN on the surface of cotton fabric which partially covered the interspaces between fibers and yarns and reducing the air flow through the fabrics.

Stiffness is a special property of the fabric for desirable draping and also influences the physical comfort of clothing. Table 3 shows the effects of TTCTN concentration on the bending properties of CF. It can be observed that the stiffness increased with increase in concentration of TTCTN. This could be associated with increased interfiber/yarn friction and abrasion at fiber/yarn crossover points due to TTCTN grafted in the matrix of CF [23]. However, the stiffness was not significantly increased with increase in concentration of TTCTN, which showed satisfactory physical comfort behavior.

Thus, based on the influence of TTCTN on the flame retardance, tensile strength and physiological comfort properties of CF, the optimum concentration of TTCTN was confirmed to be 20 wt%, the flame-retardant performance of TTCTN(20)-CF could reached B1 grade, at the same time, the use of TTCTN did not significantly reduce the tensile strength of CF and could keep satisfactory physical comfort properties.

3.7. Cone Calorimetry Tests of TTCTN(20)-CF. The HRR and THR of CF and TTCTN(20)-CF are shown in Figure 7. The CF exhibit significantly higher HRR and THR values

TABLE 2: Flammability properties of the TTCTN-CF.

Sample	Content of TTCTN/wt%	After-flame time/s	After-glow time/s	Char length/cm	Flame retardant ratings	LOI/%
CF	0	13.0	32.5	—	—	18.9 ± 0.26
TTCTN(10)-CF	10	7.2	0.0	27.0	—	23.5 ± 0.36
TTCTN(15)-CF	15	5.6	0.0	19.3	B2	25.5 ± 0.24
TTCTN(20)-CF	20	0.0	0.0	13.6	B1	28.6 ± 0.28
TTCTN(25)-CF	25	0.0	0.0	10.1	B1	30.1 ± 0.26
TTCTN(30)-CF	30	0.0	0.0	6.0	B1	31.8 ± 0.34

TABLE 3: Tensile strength and physiological comfort test of cotton fabrics with different content TTCTN.

	Tensile strength		Air permeability (L/m ² S)	Stiffness (mm)
	Warp (N)	Weft (N)		
CF	878.7	480.4	581.3	39.6
TTCTN(10)-CF	857.1	468.8	492.2	41.7
TTCTN(15)-CF	846.7	459.3	490.9	42.4
TTCTN(20)-CF	824.3	449.7	475.7	43.9
TTCTN(25)-CF	792.3	437.2	450.9	44.7
TTCTN(30)-CF	765.5	415.6	390.7	46.3.

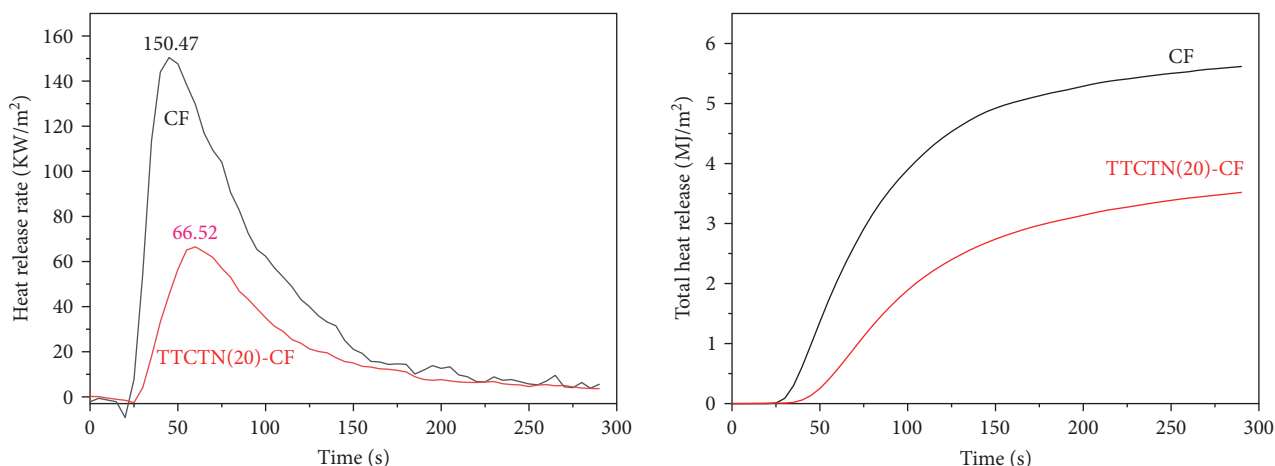


FIGURE 7: Heat release rate (HRR) and total heat release (THR) curves of TTCTN(20)-CF.

comparing with TTCTN(20)-CF. The PHRR (66.52 kW m⁻²) of TTCTN(20)-CF was much lower than the CF (150.47 kW m⁻²). Correspondingly, the THR decreased from 5.63 to 3.52 MJ m⁻². Comparing with CF, the PHRR and THR of TTCTN(20)-CF were reduced by 55.7% and 37.4%, respectively. The results indicated that TTCTN(20)-CF had excellent flame retardance.

3.8. Thermal Behaviors of TTCTN(20)-CF. TG and DTG of CF and TTCTN(20)-CF were conducted under nitrogen and air atmospheres, respectively, to investigate their thermal and thermo-oxidative stability. The results are shown in Figure 8 and the corresponding data are summarized in Tables 4 and 5. Figure 8(a) and 8(b) showed the thermal degradation curves under nitrogen atmosphere. The untreated CF began to decomposing at 297.3°C, the temperature at maximum weight loss rate was at 370.1°C, and the char yield was only

14.3% at 600°C. For the TTCTN(20)-CF, the initial decomposing temperature (T_{onset}) and maximum decomposing temperature (T_{max}) were lower than CF, TTCTN(20)-CF began to decomposing at 227.7°C and the temperature at maximum weight loss rate was at 301.9°C. The char yield of TTCTN(20)-CF was 40.2% at 600°C, which showed the char was stable at higher temperatures. Notably, compared with the untreated CF, a higher char yield was achieved and the temperature at maximum weight loss rate was decreased by about 74°C for TTCTN(20)-CF, which was attributed to the formation of polyphosphoric acid during the decomposing of TTCTN, and degrading the thermal decomposition reaction activation energy as a catalyst [24, 25]. TTCTN could decrease the temperature of maximum weight loss rate and formed the stable char residue at high temperature to protect the remaining CF from complete dehydration.

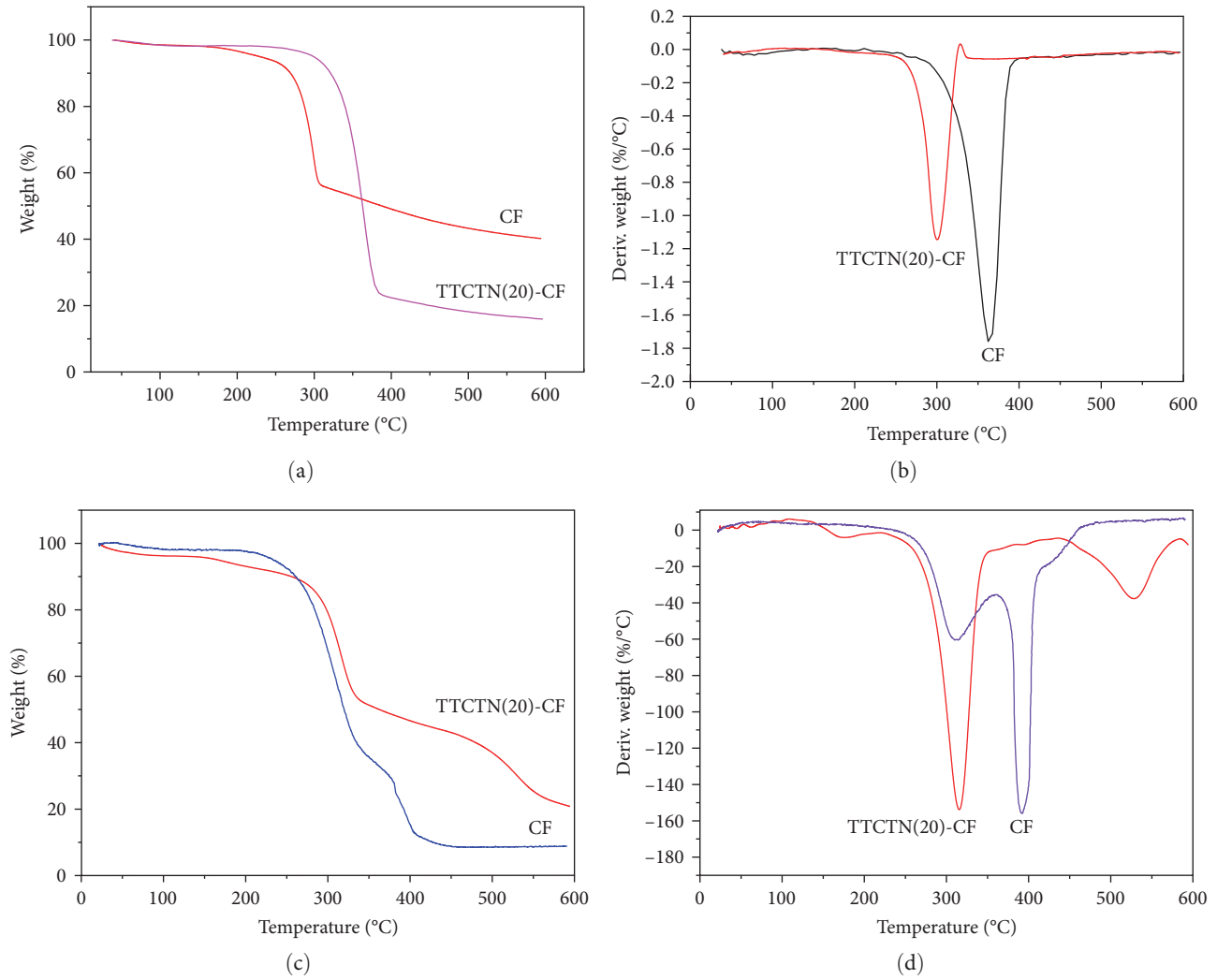


FIGURE 8: TG and DTG curves of TTCTN(20)-CF under nitrogen (a, b) and air atmospheres (c, d).

TABLE 4: Data of TG and DTG thermograms of TTCTN(20)-CF in nitrogen.

Samples	T_{onset} (°C)	T_{max} (°C)	Char residue at 600°C (%)
CF	297.3	370.1	14.3
TTCTN(20)-CF	227.7	301.9	40.2

TABLE 5: Data of TG and DTG thermograms of TTCTN(20)-CF in air.

Samples	T_{onset} (°C)	T_{max} (°C)		Char residue at 600°C (%)
		Stage 1	Stage 2	
CF	240.7	302.4	375.5	8.7
TTCTN(20)-CF	196.6	310.6	528.7	20.0

The thermal stability of untreated and treated cotton fabrics in air was also explored, as showed Figure 8(c) and 8(d). The thermal degradation of CF proceeded in two steps. The CF began dehydration at 240.7°C (T_{onset}), the first degradation occurred at

302.4°C, which attributed to the depolymerization of cellulose to produce volatile substances [7]. The second degradation temperature was the T_{max} of CF, which generated at 375.5°C, the second step was due to the oxidation of the char formed in the first step

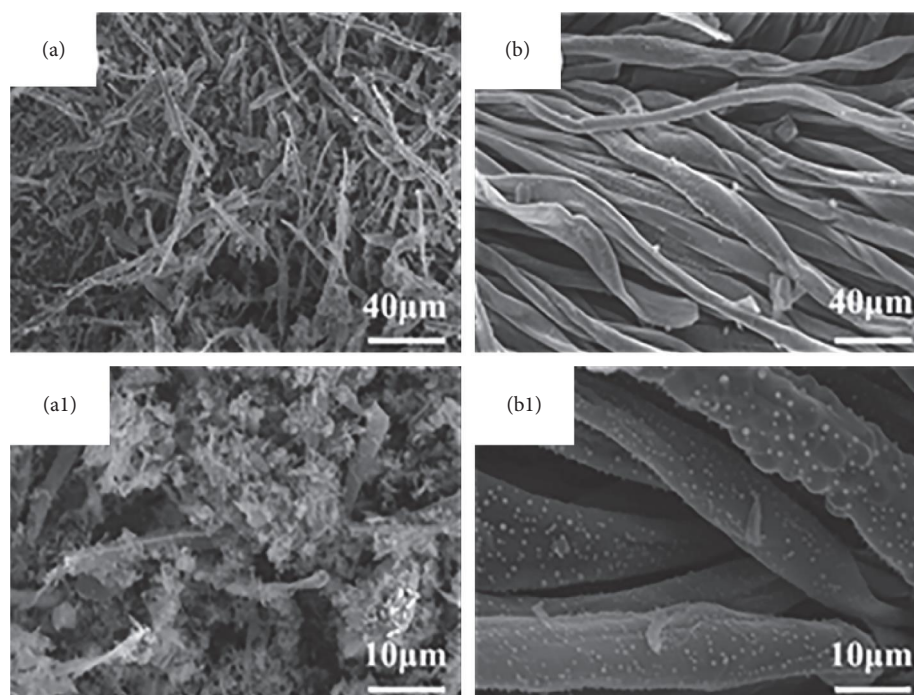


FIGURE 9: Morphology of char residue of untreated cotton fabric (a, a1) and TTCTN(20)-CF (b, b1).

TABLE 6: The flame retardance of TTCTN(20)-CF with different washing cycles.

Washing cycles	After-flame time/s	After-glow time/s	Char length/mm	LOI/%	Flame retardant ratings
0	0.0	0.0	135	28.6 ± 0.28	B1
10	0.0	0.0	135	28.1 ± 0.34	B1
20	0.0	0.0	138	27.7 ± 0.20	B1
30	0.0	0.0	140	27.3 ± 0.32	B1
40	0.0	0.0	143	26.8 ± 0.26	B1
50	0.0	0.0	147	26.2 ± 0.28	B1
60	0.0	0.0	150	25.3 ± 0.32	B2

[26]. The 8.7% of carbon layer could be retained at 600°C. It could be noted that TTCTN(20)-CF showed a lower onset degradation temperature (T_{onset} : 196.6°C) and had a major weight loss stage at 310.6°C, this stage included the formation of char to generate thermally stable cohesive phases which prevented the sample from further decomposition [27]. Comparing to CF, the oxidation of the char in second degradation was delayed to 528.7°C, and the degradation rate decreased obviously. The result proved that the thermo-oxidative stability of the TTCTN(20)-CF was significantly improved.

3.9. Surface Morphology of Char Residue for TTCTN(20)-CF. The surface morphology of char residue of TTCTN(20)-CF had been studied by SEM (Figure 9). The surface morphology of the char layer after burning was very loose and the char residues were completely destroyed for untreated cotton fabric. For the burned TTCTN(20)-CF, the surface of TTCTN(20)-CF was covered by a dense and swollen char layer, which can prevent the release of heat from the burning

of the cotton fabric and block contact of the flammable degradation products with external combustibles such as oxygen and protect effectively the internal structure [28]. Therefore, the integrity of the fiber structure could be maintained.

3.10. Washing Durability of TTCTN(20)-CF. The washing durability of TTCTN(20)-CF was studied. As shown in Table 6, the flame retardance of TTCTN(20)-CF was slightly decreased with the increasing of the washing cycles, the B1 flame retardant ratings and LOI = 26.2% could be still obtained after 50 washing cycles, which demonstrated that the chemically bonding connection was strong between TTCTN and cotton fabric matrix. This indicated that the TTCTN(20)-CF had excellent washing durability.

3.11. Surface Morphology for TTCTN(20)-CF with 50 Washing Cycles. The surface morphology of untreated CF, TTCTN(20)-CF, and TTCTN(20)-CF with 50 washing cycles was analyzed by SEM. As Figure 10 shows, the surface of the untreated CF

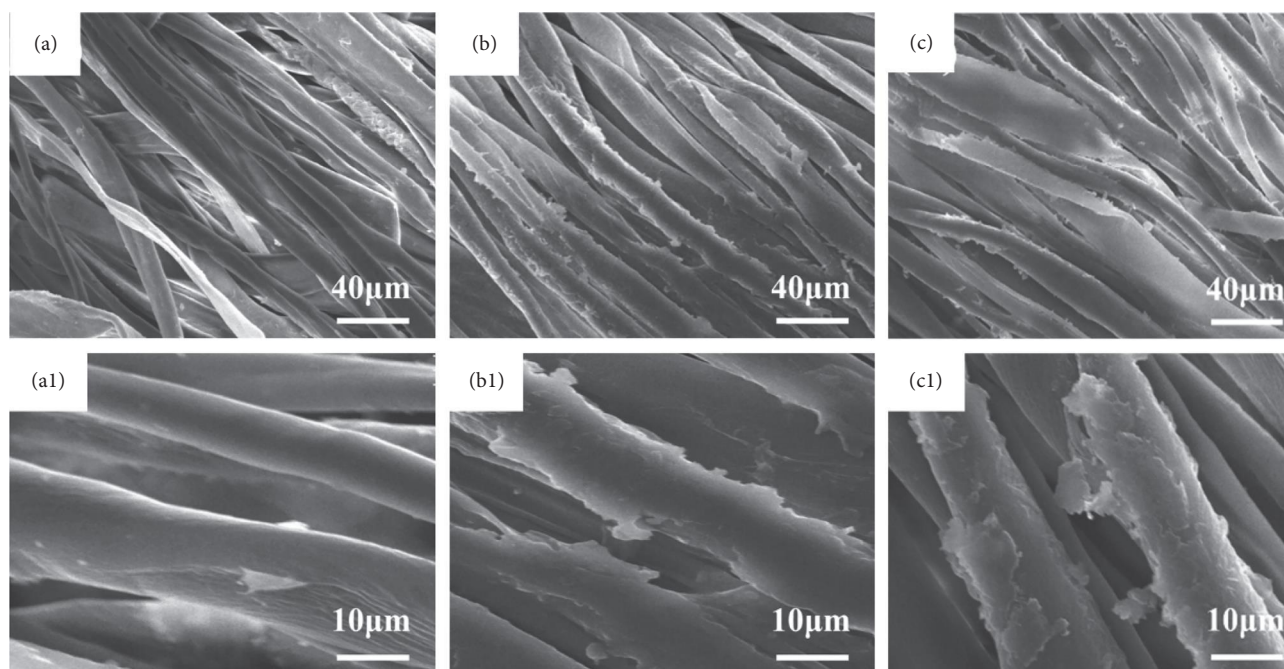


FIGURE 10: SEM of untreated cotton fabrics (a, a1), TTCTN(20)-CF (b, b1), and TTCTN(20)-CF with washing 50 washing cycles (c, c1).

was smooth, but the surface of TTCTN(20)-CF showed the rough surface comparing with the untreated CF, which was attributed to TTCTN grafted in fabric matrix by the cross-linking reaction during flame retardant finishing process of CF. After 50 washing cycles, SEM showed the TTCTN was still bounded to the surface of CF, this further proved that the presence of strong chemically bonding connection between TTCTN and cotton fabric matrices, which gave the TTCTN(20)-CF excellent washing durability.

4. Conclusions

A novel P-N intumescent flame retardants named TTCTN with reactive groups was successfully prepared. The CF treated with TTCTN had excellent flame retardance and physiological comfort properties. The LOI of TTCTN(20)-CF could reach 28.6% and the flame retardancy reached the class B1 of national standard of flame retardant fabrics. The HRR and THR of TTCTN(20)-CF were reduced by 55.7% and 37.4%, respectively. In addition, the TTCTN did not significantly affect the tensile strength of CF. Compared with untreated cotton fabric, TTCTN(20)-CF decomposed at a lower temperature and more protective carbon residues could be obtained. The TTCTN(20)-CF had excellent washing durability because TTCTN could be grafted on cotton fabric matrix by strong chemically bonding. The B1 flame retardant ratings and LOI = 26.2% could be still obtained for TTCTN(20)-CF with 50 washing cycles. CF treated with TTCTN had durable flame retardance and application potential.

Data Availability

The underlying data used to support the findings of this study are included within the article.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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