

Research Article

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Synthesis of a phosphoramidate flame retardant and its flame retardancy on cotton fabrics

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Abstract: A phosphoramidate flame retardant (dimethyl *N,N*-bis(2-hydroxyethyl)phosphoramidate, DMBHP) was synthesized and applied to cotton fabrics for enhancing the flame retardancy. The structure of DMBHP was characterized by FT-IR and NMR. The flame retardancy and combustion behavior of the treated cotton fabrics were evaluated using the vertical flammability test (VFT), limiting oxygen index (LOI), and the cone calorimetric test. Moreover, to further analyze the flame retardant action of DMBHP in cotton fabrics, thermal degradability of the treated fabrics, as well as the chemical structure, surface morphology, and element contents of the char residue of the DMBHP-treated fabrics were also evaluated. The results show that, after treating with DMBHP, the cotton fabrics acquired a LOI value from 18.1 to 31.1 with the concentration increasing to 30% and self-extinguished in VFT tests when treated with up to 15% DMBHP. Besides, the total heat release and the peak heat release rate of DMBHP (30%)-treated fabric decreased obviously compared with the pure cotton along with more residue retained. TG, SEM, and EDS results of treated cotton fabric and the corresponding residue after burning showed that DMBHP has the capability

of enhancing char formation. In addition, DMBHP will confer cotton fabrics a certain durability against washing with the help of 1,2,3,4-butanetetracarboxylic acid (BTCA) and citric acid (CA).

Keywords: phosphoramidate, cone calorimeter, thermal stability, SEM-EDS, cotton

1 Introduction

Cotton is a natural cellulose fiber that is used widely in apparel, upholstery, and industrial textiles as it possesses several desirable properties such as softness, comfort, warmth, recyclability and biodegradability. However, the high flammability of cotton fabric is a major concern that greatly restricts its practical application. To overcome this issue and reduce the potential fire hazard of cotton fiber, various compounds and formulations containing halogens, phosphorus, nitrogen, and silicon or their mixtures have been developed in terms of improving the flame resistance of cotton fabrics (1–5). Among all these flame retardants, the halogen-containing compounds, although providing excellent flame retardancy, had been forbidden due to releasing toxic gases during combustion and inducing potential bioaccumulation and persistence in organisms (6–8). In this context, a great deal of phosphorous-containing compounds has been developed, which are recognized as the most promising candidates for flame retardants due to their low toxicity and efficiency (8–10). This class of flame retardants turns out to be effective in promoting char formation catalyzed by polyphosphoric acid at high temperature and have great potential in flame retardant application for various polymers including cotton fabrics (11–13). Among phosphorous-containing compounds, various phosphorous–nitrogen-containing compounds were developed to provide flame retardancy for cotton fabrics as numerous studies show that the presence of nitrogen confers a synergistic enhancement in the phosphorous-containing flame retardants (12,14–16). In recent years, the phosphoramidates attracted a large quantity of attention in the field of flame retardant agents

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(3,13,14,17–19), which could be easily synthesized in one step from chlorophosphoramidates (or phosphites) and amine compounds (3,14,18–20). The application of phosphoramidates obviously improved the flame retardant capability of cotton fabrics, exhibiting a better FR property than phosphates. By choosing different starting materials, various phosphoramidates with different substituents on the oxygen and nitrogen atoms can be obtained. Structure effects of phosphoramidates on the flame retardancy of the treated cotton fabric have been carried out. Researchers found, in most cases, that the increased number of nitrogen atom bonded to the phosphoryl group enhanced the flame retardancy of the phosphoramidates. In addition, Nguyen et al. synthesized phosphoramidates with different *O*-alkyl groups and revealed that phosphoramidates with smaller *O*-alkyl groups exhibited superior flame retardant activity (20). Furthermore, Gaan et al. investigated the influences of different alkyl substituents at the nitrogen atom and found that reactive terminal hydroxyl groups could catalyze the decomposition of the flame retardants as well as promote the char formation of cellulose (14).

In this study, we synthesized a phosphoramidate with two methoxyl groups and hydroxyl groups in the substituents at the nitrogen atom (dimethyl *N,N*-bis(2-hydroxyethyl)phosphoramidate, DMBHP). The chemical structure of DMBHP was characterized using FT-IR and NMR. Flame retarded cotton fabrics with different concentrations of DMBHP were prepared, and the flame retardancy was evaluated using vertical flammability test (VFT), limiting oxygen index (LOI), and cone calorimetry. The thermal stability was determined by the thermogravimetric analysis (TGA). The char residues of the FR-treated cotton were also investigated using FTIR, SEM, and EDS. In addition, the two alkyl-hydroxyl groups, which is similar to those in cellulose molecules, make it possible to introduce polycarboxylic acid as crosslinking agents between DMBHP and cotton by esterification (21,22). Therefore, in washing durability test, 1,2,3,4-butanetetracarboxylic acid (BTCA) and citric acid (CA) were used to improve the washing fastness of the DMBHP-treated cotton fabric.

2 Experimental

2.1 Materials

Mercerized cotton fabric (140 g/m², 524 × 283, 14.74 tex × 14.76 tex) supplied by Weifang Qirong Textile Co. Ltd (Weifang, China) was used for the experiments. Dimethyl

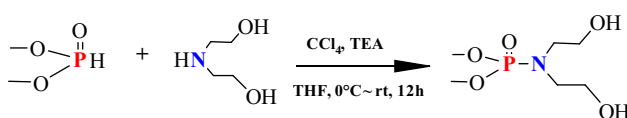
phosphite (DMP) and diethanol amine were purchased from Macklin Biochemical Co. Ltd, Shanghai, China. Tetrahydrofuran (THF), carbon tetrachloride, trimethylamine (TEA), citric acid (CA) and sodium hypophosphite (SHP) monohydrate were purchased from Sino-pharm Chemical Reagent Co. Ltd, Shanghai, China. 1,2,3,4-Butanetetracarboxylic acid (BTCA) was supplied by Shouguang Lisheng Chemical Co. Ltd, Shandong, China. All the chemicals were of reagent grade and were used in the experiments without further purification.

2.2 DMBHP synthesis

As illustrated in Scheme 1, the flame retardant DMBHP was synthesized in a single step from dimethyl phosphite and diethanol amine. A solution of dimethyl phosphite (0.1 mol) and carbon tetrachloride (0.1 mol) was prepared in tetrahydrofuran (THF), and then cooled to 0–5°C while stirring, following which, a solution of diethanol amine (0.1 mol) and triethylamine (0.1 mol) in tetrahydrofuran was added to it drop by drop for 1 h using an addition funnel. Subsequently, the reaction mixture was allowed to warm up to the room temperature while continuing the stirring for 11 h. When the reaction was over, a white precipitate (TEA·HCl) was filtered out, and then, the solvent was evaporated, producing a light-yellowish oil (the flame retardant, DMBHP) with a yield of 98%. The structure of the synthesized flame retardant was confirmed using NMR and FT-IR.

2.3 Fabric treatment

The synthesized DMBHP compound was dissolved in distilled water to produce aqueous solutions with different DMBHP concentrations (wt%, 10%, 15%, 20%, 25%, and 30%). Cotton samples were soaked in the prepared solutions, followed by padding using a laboratory padder. The wet pickup after two dips and two nips was 100%. Subsequently, the fabric samples were mounted on a pin frame, dried at 80°C for 5 min, and finally cured at 160°C



Scheme 1: Synthesis of DMBHP.

for 3 min. Drying and curing were performed inside a setting machine (Shanghai Longcolor Textile Equipment Co. Ltd). The cotton samples treated with different DMBHP concentrations (10%, 15%, 20%, 25%, and 30%) were labeled as C10, C15, C20, C25, and C30, respectively. The weight gains (WG) of the treated samples were calculated using Eq. 1:

$$WG = \frac{W_i - W_0}{W_0} \times 100, \quad (1)$$

where W_0 and W_i denote the weights of the cotton fabrics before and after the completion of the treatment.

3 Characterization

3.1 ^1H NMR and ^{31}P NMR

^1H NMR and ^{31}P NMR of the synthesized DMBHP were evaluated in a Bruker AVANCE III HD 400 MHz spectrometer (Bruker, Germany) using CDCl_3 as a solvent. TMS and 85% aqueous phosphoric acid were employed, respectively, as a reference.

3.2 Fourier transform infrared (FT-IR) spectroscopy

The chemical groups in the structure of the synthesized DMBHP, control cotton, DMBHP-treated cotton, and the residue of treated cotton were analyzed through FT-IR spectroscopy using a Nicolet iS 50 FT-IR spectrometer (Thermo Fisher Scientific, USA) in the range of $500\text{--}4,000\text{ cm}^{-1}$ with a resolution of 4 cm^{-1} .

3.3 Vertical flammability tests and limiting oxygen index

Both the control and finished cotton fabrics were subjected to vertical flammability tests (VFT) and limiting oxygen index (LOI) test. The vertical flammability tests (VFT) were conducted in accordance with GB/T 5455–2014 using a vertical combustion apparatus (LFY-601 A, Shandong Textile Science Research Institute, China). The size of the samples was $300\text{ mm} \times 80\text{ mm}$, and the ignition time was 12 s. The afterflame time, afterglow time and char length were measured and recorded.

The LOI values of the cotton fabrics were evaluated in accordance with GB/T 5454-1997 using an oxygen index tester (LFY-606 B, Shandong Textile Science Research Institute, China), and for each sample, the fabrics were cut into $150\text{ mm} \times 58\text{ mm}$ pieces for testing.

3.4 Cone calorimetric test

The combustion behavior of the cotton samples was analyzed in accordance with the ISO 5660 using a cone calorimeter device (FTT007, Fire Testing Technology, UK). Samples of size $100\text{ mm} \times 100\text{ mm}$ were prepared, and three pieces of the fabrics with one layer above the other were wrapped with aluminum foil along with specific grids and burned at a heat flux of 35 kW/m^2 .

3.5 Thermogravimetric analysis (TGA)

Thermal degradation properties of the cotton fabrics were analyzed using a TG analyzer (STA6000, PerkinElmer, USA). The samples weighing approximately 5 mg were investigated under nitrogen atmosphere at a temperature range of $35\text{--}800^\circ\text{C}$ and a heating rate of 10°C/min .

3.6 SEM-EDS

The surface morphology of control cotton fabrics and the finished fabrics before and after burning was observed using a scanning electron microscope (VEGA 3, TESCAN, Czechoslovakia) at an accelerating voltage of 30 kV. The element contents and the distribution of phosphorus (P), nitrogen (N), carbon (C), and oxygen (O) in the DMBHP-treated finished cotton fabrics before and after burning were measured using an energy dispersive spectrometer (EDS).

3.7 Washing durability

In the washing fastness test, the flame retardant-treated cotton fabrics were soaked in a shaking aqueous solution containing 0.15 g/L AATCC 193 standard detergent for 10 min. The liquor ratio of fabrics to the washing solution was 1:50. The solution temperature was 50°C . Subsequently, the samples were removed, rinsed with distilled water, and then dried at 80°C for 1 h. Then, the LOI value was tested, respectively.

4 Results and discussion

4.1 Synthesis and structural characterization of DMBHP

The FT-IR spectra of the synthesized DMBHP are displayed in Figure 1. The absorption peak at $3,366\text{ cm}^{-1}$ was attributed to the stretching vibration of the O–H group, while the peaks at $2,986$ and $2,852\text{ cm}^{-1}$ were assigned to the vibrations of the $-\text{CH}_3$ and $-\text{CH}_2$ groups. The peaks at $1,225$ and $1,184\text{ cm}^{-1}$ were attributed to $\text{P}=\text{O}$ absorption (3,17). The characteristic peak at $1,003\text{ cm}^{-1}$ was for $\text{P}-\text{O}-\text{C}$. When the spectrum of DMBHP was compared to the that of DMP, besides the peaks at $3,366\text{ cm}^{-1}$, two other absorption peaks appeared at $1,159$ and $1,358\text{ cm}^{-1}$, respectively, which were attributed to $\text{P}-\text{N}$ and tertiary $\text{C}-\text{N}$ bending vibration, while the $\text{P}-\text{H}$ absorption peak at $2,430\text{ cm}^{-1}$ (as shown in the spectrum of DMP) disappeared in the spectrum of DMBHP. These results indicated that the target phosphoramidate flame retardant depicted in Scheme 1 had been synthesized successfully.

To further confirm the structure of the synthesized flame retardant, the ^1H -NMR and ^{31}P -NMR spectra were obtained. As depicted in Figure 2, ^1H NMR spectrum gave the proton signals with the following peaks: 4.47 ppm (s, 1H), $3.70\text{--}3.57\text{ ppm}$ (m, 5H) and $3.22\text{--}3.03\text{ ppm}$ (m, 2H). The singlet at 4.47 ppm (labeled as d) was attributed to $-\text{OH}$, and the peaks at $3.70\text{--}3.57$ (labeled a + c) were attributed to the protons in $-\text{CH}_3$ and $-\text{CH}_2$ near the $-\text{OH}$ group. The peaks around $3.22\text{--}3.03\text{ ppm}$ corresponded to $\text{N}-\text{CH}_2-$. In addition, an integration ratio of the peaks d, (a + c), and b was determined to be $1:5.07:2.02$, which is quite close to the numbers of corresponding hydrogen protons in the DMBHP molecule ($2:10:4$). The single peak at 13.56 ppm in the ^{31}P NMR spectra was attributed to the

only phosphorus in the synthesized flame retardant, indicating that DMBHP had been synthesized successfully.

4.2 Flammability of DMBHP-treated finished fabrics

LOI evaluation and vertical flammability tests were performed to assess the flame resistance of the fabrics treated with different concentrations of the flame retardant synthesized in the present study. Table 1 enlists the LOI values and the results of the vertical flammability tests for control cotton and treated cotton fabrics (C10, C15, C20, C25, and C30). It was observed that the LOI value of the treated fabrics increased with the increasing DMBHP concentration. Pure cotton is combustible, with a LOI value of just 18.1, while the sample treated with 15% DMBHP presented an LOI value of 26.5, which implied that it would be hard to burn in air (23). Furthermore, the finished fabric presented an LOI value of 31.1 when the concentration reached 30%. In the vertical flammability test, the control cotton sample was observed to burn out with an after-flame time of 15.3 s, an afterglow time of 25 s, and nearly no residue at the completion of the test. In regard to the finished fabrics, the fabrics treated with 10% and 15% DMBHP exhibited excellent flame-retardant properties; although the samples exhibited an after-flame burning time of 1.5 and 1.1 s, respectively, they also displayed improved char-forming properties. As the concentration of DMBHP was increased to 20%, the WG of C20 reached 15.1%, with no after-flame and afterglow burning observed, and the finished fabrics exhibited a char length of 12 cm, which could satisfy the maximum level requirements (10–15 cm) and pass the vertical flammability test (20). Clearly, a further increase in the DMBHP concentration led to better flame retardant properties and less char length for the finished cotton fabric. Overall, both LOI measurements and VFT ascertained the effectiveness of DMBHP treatment for cotton fabrics.

4.3 Cone calorimetric test

To further assess the flame retardancy of the treated cotton fabric in real fire scenarios, the cone calorimetry was employed (2,15,24). The curves of total heat release (THR), the residue, and the pictures of the residue after the tests are presented in Figure 3.

From Figure 3, we can see that the THR value of finished cotton significantly decreased, compared to the control

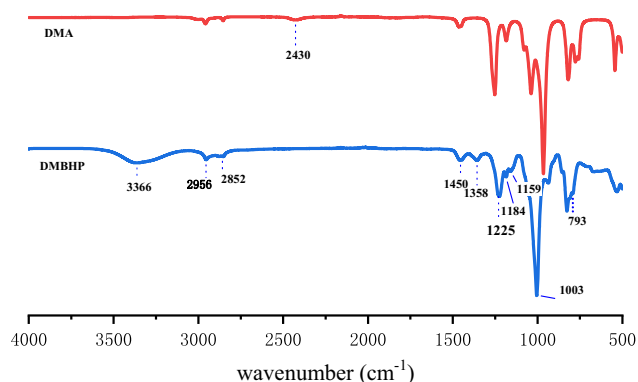


Figure 1: FT-IR spectra of DMBHP and DMP.

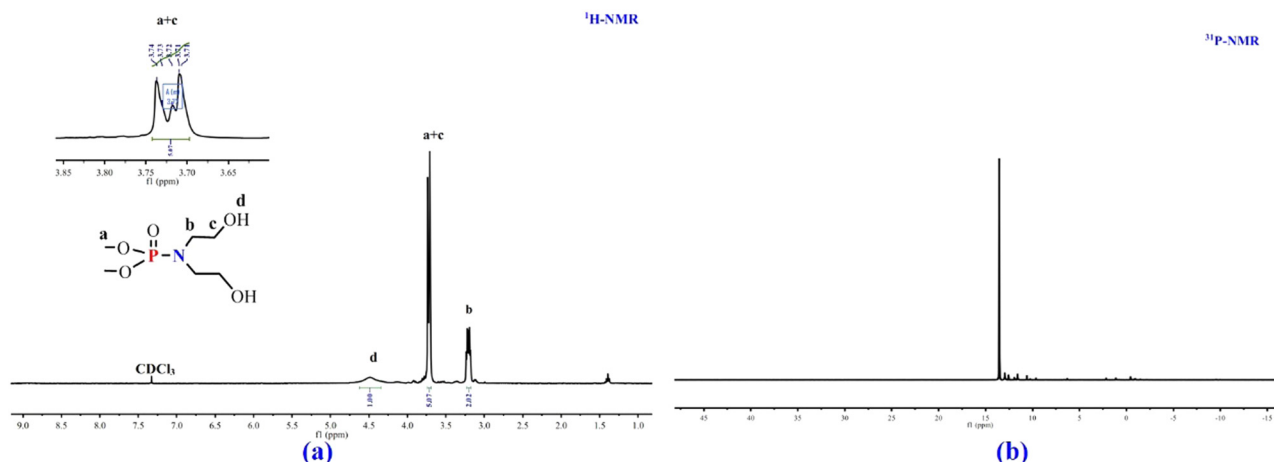


Figure 2: (a) ^1H -NMR and (b) ^{31}P -NMR spectra of DMBHP.

cotton, from 7.2 to 5.6 MJ/m², which was supposed to be attributed to the blocking of the heat transfer by the charred residue layer. In addition, the control cotton burned vigorously after the ignition, as shown in Figure 3e, and the HRR reached its peak with a PHRR value of 196.9 kW/m². After the incorporation of DMBHP, the PHRR of the treated cotton fabric decreased significantly to a value of 115.2 kW/m². These results might be due to the incorporation of flame retardant, which changes the decomposing path of cotton fabric (25), and the formed char layer could block the heat transfer and the oxygen supply for the underlying substrate. These results were also verified by the photographs of the fabrics after the cone calorimetry test, as shown in Figure 3a and b, almost nothing was left for the control cotton after the cone calorimetry test. While for the DMBHP-treated fabric, significant char was formed, with a residue of about 15.6% retained after the test, which is consistent with the results of TG and vertical flammability test.

4.4 Thermogravimetric analysis (TGA)

The thermal stabilities of control fabric and finished fabric were determined by performing TG and DTG in the nitrogen

atmosphere, and the results are presented in Figure 4. The corresponding data such as $T_{5\%}$ (the temperature of 5% weight loss, defined as the initial decomposition of the tested samples (13,18,26)), T_{max} (the temperature of the maximum rate of degradation), and the char residue at 800°C are presented in Table 2. It is clear from Figure 4 and Table 2 that control cotton had a $T_{5\%}$ value of 292°C, a prominent degradation stage at the temperature range of 280–390°C, and a T_{max} of 367°C. The pyrolysis of pure cotton in nitrogen is reported to be caused the dehydration and depolymerization of cellulose into volatile products involving levoglucosan (27,28). With an increase in temperature, degradation continued for the control cotton, and only 8.9% char residue was left at 800°C. Comparatively, all the finished fabrics exhibited lower initial decomposition temperatures as well as maximum rate decomposition temperatures, which was attributed to the formation of phosphoric acid derivatives because of degradation of the flame retardants and the subsequent dehydration of cellulose (13). Moreover, a large amount of residue was observed for the finished fabrics, and the residue amount increased as the concentration of FR increased. For instance, the treated fabric C10 presented a residue amount of 31.2% at 800°C, which was higher than that of the pure cotton (8.9%), even though the weight gain

Table 1: Results of LOI measurement and VFT for cotton treated with different concentrations of DMBHP

Samples	Concentration (wt%)	WG (wt%)	LOI (%)	After-flame time (s)	After-glow time (s)	Char length (cm)
Control cotton	0	0	18.1 ± 0.5	20.3 ± 1.2	15 ± 1.5	—
C10	10	7.9 ± 0.8	24.5 ± 0.5	1.48 ± 0.3	0	30
C15	15	11.8 ± 1.0	26.5 ± 0.1	1.11 ± 0.4	0	26 ± 0.5
C20	20	15.1 ± 1.2	28.1 ± 0.3	0	0	12 ± 0.4
C25	25	20.5 ± 0.9	30.2 ± 0.4	0	0	10 ± 0.2
C30	30	24.7 ± 1.1	31.1 ± 0.3	0	0	8 ± 0.5

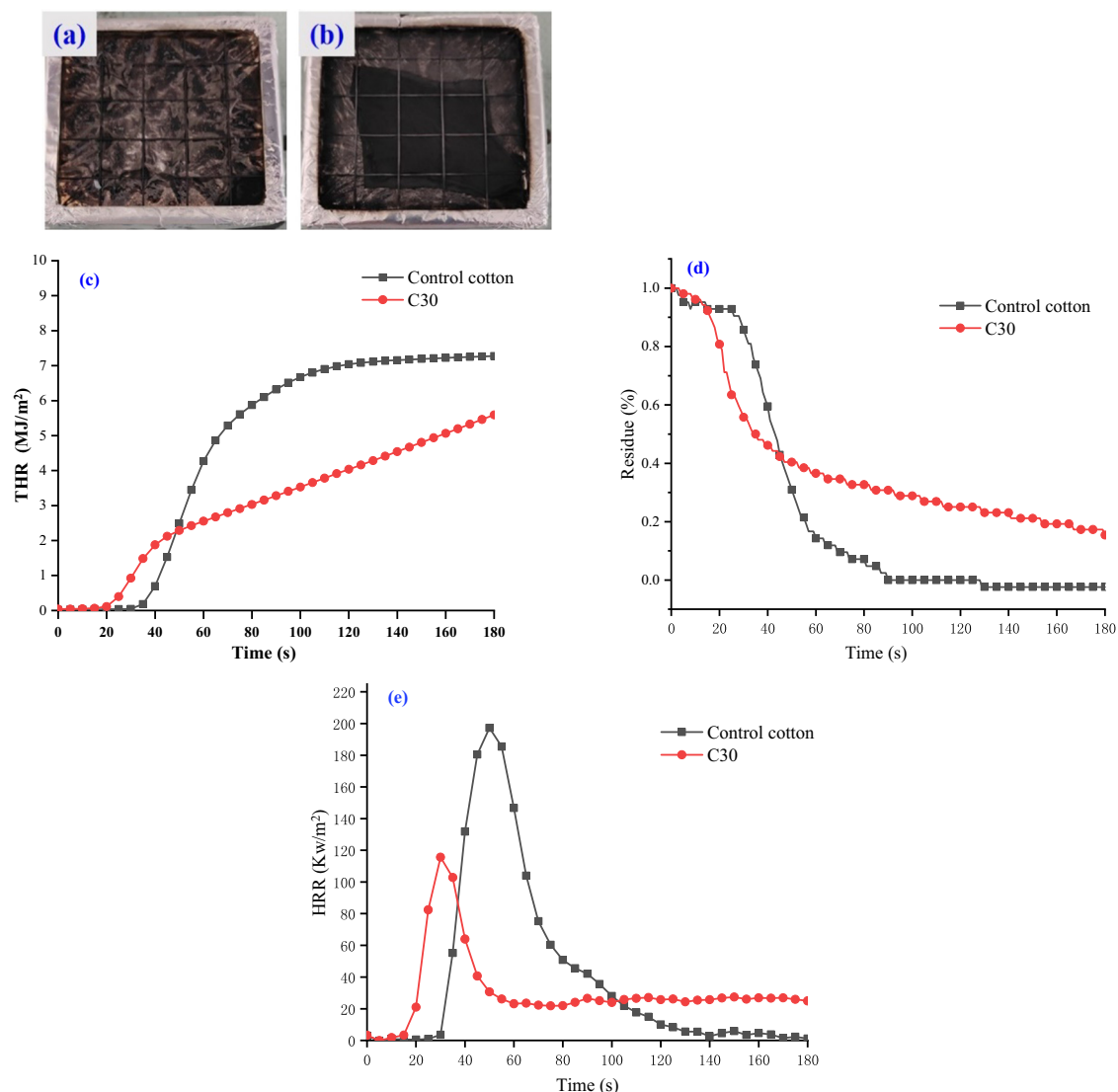


Figure 3: Typical curves from calorimetric tests: (a) control cotton, (b) DMBHP-treated cotton fabrics after cone calorimetry; (c) THR, (d) residue, and (e) HRR curves of the control cotton and DMBHP-treated fabric after the cone calorimetry test.

of DMBHP was just 7.9% (as presented in Table 2). This implied that the introduction of DMBHP greatly enhanced char formation via condensed phase mechanism (13,29). The formed charred residue could act as a protective barrier isolating the heat and mass transport from the fabric. However, it is noteworthy that when the concentration was increased beyond 10%, further improvement in flame retardant loading could not produce such a high increase in the amount of char residue as from 7.9% to 31.2%. Overall, the TG results demonstrated that treatment with DMBHP would alter the degradation mode of the cotton fabric by favoring char formation and lowering the initial decomposition temperature. The similar performance of the flame retardants containing phosphorous and nitrogen have also been reported

in various other materials and for different flame retardant agents (13,17,20,29), possibly due to the phosphorylation and catalytic dehydration of cellulose by the phosphorus-containing flame retardants.

4.5 Surface morphology and elemental constitution

SEM was employed to further analyze the surface morphology of the finished fabrics and the corresponding residue of the fabrics postburning after the vertical flammability test, and the images are presented in Figure 5. As expected,

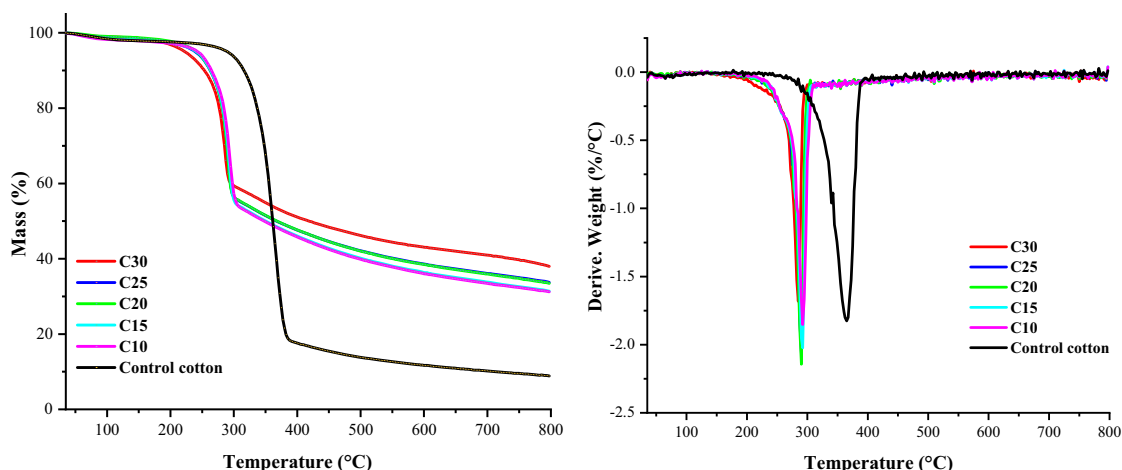


Figure 4: TG and DTG curves for control cotton and DMBHP finished cotton.

Table 2: Data obtained from TG tests of control cotton and DMBHP-finished cotton in a nitrogen atmosphere

Samples	$T_{5\%}$ (°C)	T_{max} (°C)	Residue at 800°C (wt%)
Control cotton	292.3	366.7	8.9
C10	244.5	292.5	31.2
C15	241.5	292.4	31.4
C20	241.5	289.9	33.4
C25	238.1	289.9	33.7
C30	224.9	285	38.0

compared to the control cotton (Figure 5a), it was observed that the treated cotton fabric (Figure 5b–d) exhibited a rough and uneven surface, which was similar to a coating deposited on the fiber even though the synthesized flame retardant was soluble in water and the pad-dry-cure finishing process was employed to apply DMBHP onto the fabric. Similar fiber surface appearance of DMBHP-treated fabric has also been reported for other phosphoramidate derivatives (20). In addition, all the treated fabrics after burning retained the integral fabric construction of fiber arrangement (Figure 5f and g). There were numerous bubbles appearing on the residue surface for the fabric treated with 10% DMBHP (Figure 5f) possibly due to the release of various gases (15,30). A few of these bubbles became bigger because of continuous heating, causing the expansion of gas (31). Interestingly, it was observed that a denser molten-like layer was formed on the residue surface when a higher concentration (20%) of DMBHP was applied, as depicted in Figure 5g (indicated within the yellow circle). These results suggested that DMBHP display a flame retarding action in the condensed phase, through the formation of a protective carbon layer (30). The different surface properties of the residues

from the fabrics treated with different DMBHP concentrations could be explained as follows: large amount of gas was allowed to permeate into the air as the molten-like protective layer was thin when the *WG* of the flame retardant was low; however, when the weight gain of the flame retardant increased, the formed protective layer became thicker, and it became difficult to release the gas into the air, resulting in few bubbles.

The surface chemical compositions of fabrics treated with DMBHP (C30) and the residues of that after burning were determined using EDS. As depicted in Figure 6, besides C and O, the distribution of P and N was detected in DMBHP-treated fabrics (Figure 6a) as well as in the residues after burning (Figure 6b), which implied that the flame retardant was successfully introduced into the fabric. In addition, the weight percentages of C, O, N, and P elements were determined and compared. It was observed, after burning, that the content of O element decreased and that of C increased significantly due to the dehydration reaction and char formation during the combustion. Moreover, after burning, the elements P and N still retained, the weight percentage of P increased from 10.12 to 19.12, and the content ratio of N to P decreased dramatically after burning, which is possibly because of the P–N-containing flame retardants favoring char formation in cellulose and the release of NH_3 (32).

4.6 FT-IR of DMBHP-treated fabrics before and after burning

FT-IR spectra of the DMBHP-treated fabrics and control fabrics are presented in Figure 7. The treated fabrics

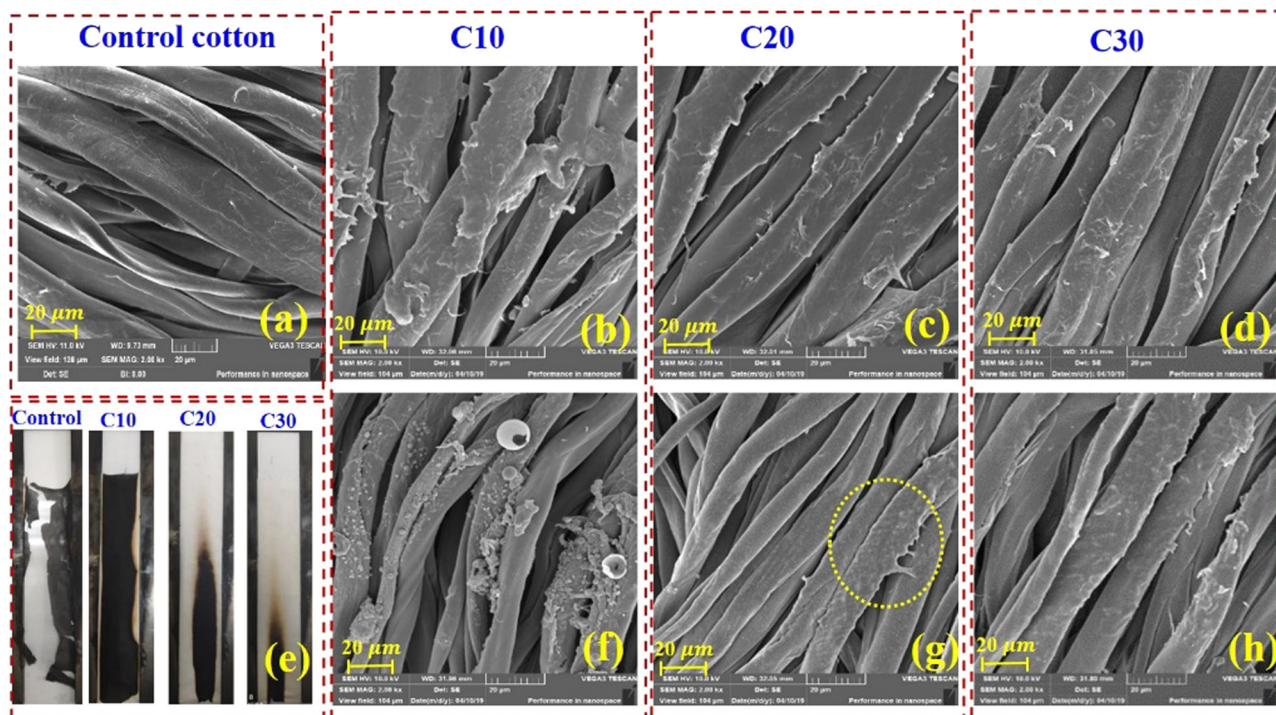


Figure 5: SEM images of (a) control cotton and (b–d) DMBHP-treated cotton and (e) photographs of DMBHP treated fabrics; (f–h) SEM images of the residue of treated cotton after the vertical flammability test.

exhibit peaks for P–O absorption at $1,225\text{ cm}^{-1}$ and P–N absorption at 796 cm^{-1} (17). These absorption peaks were also observed in the FT-IR spectrum of the synthesized flame retardant (depicted in Figure 1 DMBHP). In addition, certain new peaks appeared in the spectra of flame retardant-treated fabrics, among which the peak at $1,375\text{ cm}^{-1}$ was attributed to $-\text{CH}_3$ and the peak at $1,025\text{ cm}^{-1}$ was assigned to P–O–C absorption.

To further confirm the aforementioned stated assertions from the EDS analysis, the char residue of the finished fabrics C20 and C30 after vertical flammability tests was characterized using FT-IR, and the results were illustrated in Figure 8. In comparison to the spectrum of the finished fabric (C30) before burning, the absorption bands for hydroxyl groups around $3,350\text{ cm}^{-1}$ and those for the hydrocarbons appearing at $2,890$ and $1,385\text{ cm}^{-1}$ disappeared in the spectrum of the char residue. Moreover, the significant peaks for phosphorus compounds at $1,225$, $1,025$, and 794 cm^{-1} also disappeared. In addition, new peaks at $1,400$ – $1,700$ and 980 cm^{-1} appeared in the spectrum of the residue, which were attributed to the absorptions of C–C in aromatic groups (3,9) and to P–O–P stretching (13). It was concluded from these results that

the incorporation of DMBHP possibly promoted the dehydration of cellulose and the formation of char residue.

4.7 Washing ability

The washing ability of the treated fabrics is an important factor that influences their practical application. To evaluate the washing ability of the DMBHP-treated fabrics, the LOI values of the fabrics after the washing test were determined, and the results are presented in Figure 9. After the washing test, the LOI value decreased to 18.5, which is close to the value for pure cotton, implying that the flame retardant was not bound to the cotton fabric. To improve the washing ability of DMBHP-treated fabrics, two cellulose cross-linking agents, BTCA and CA, were involved in the finishing solution of 30% DMBHP, respectively, and sodium hypophosphite (SHP) was used as a catalyst. The treated cotton fabrics were tested in accordance with the procedure in 3.7. As shown in Figure 9, we can see that the LOI value of the flame retardant cotton fabric with the presence of BTCA and CA remained 26.6 and 25.2, respectively, after washing for 10 min, which

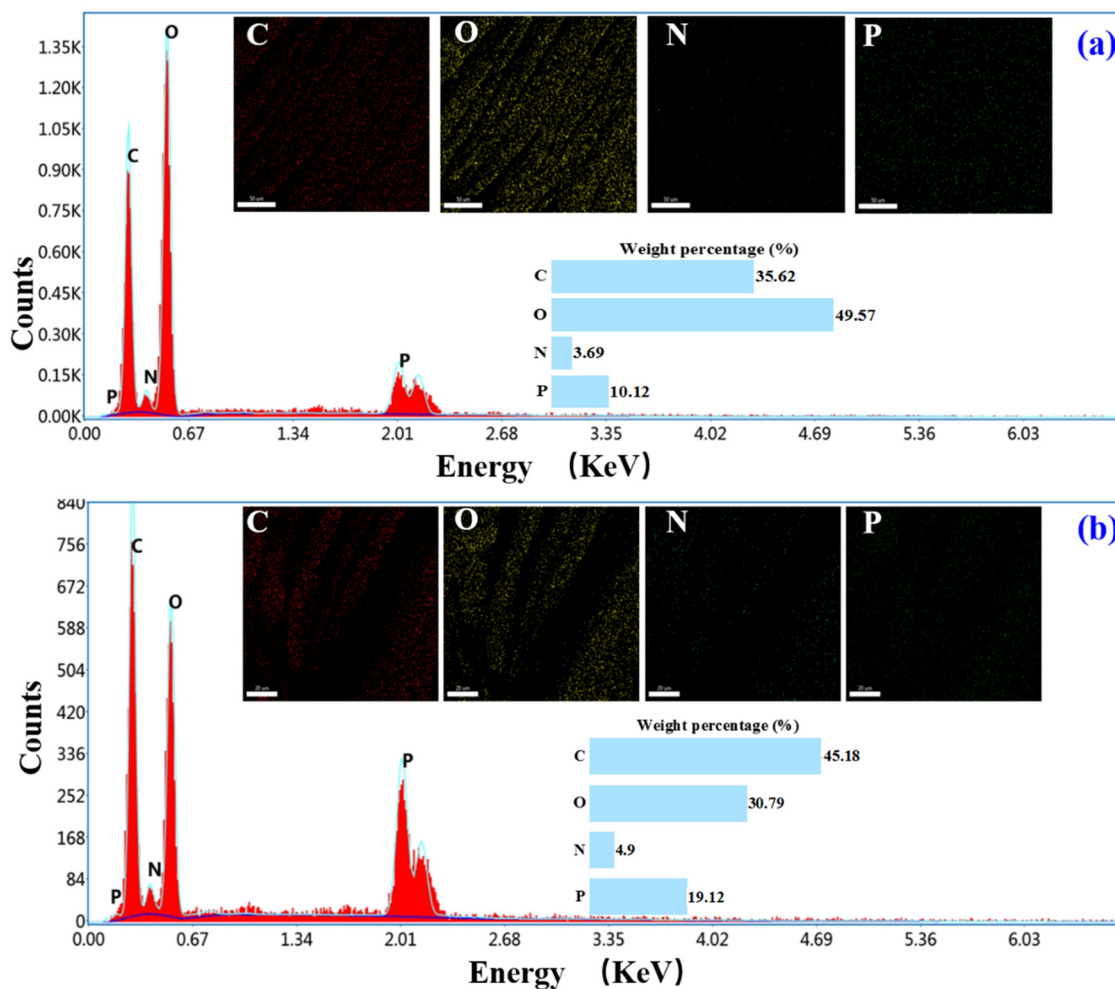


Figure 6: EDS results for DMBHP-treated cotton fabrics: (a) C30 and (b) burnt C30.

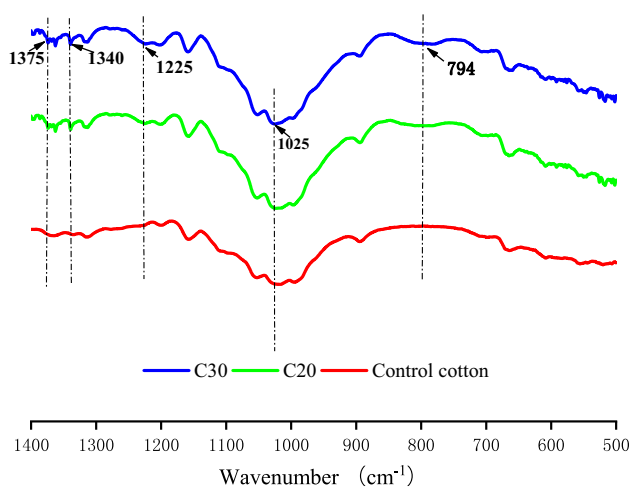


Figure 7: FT-IR spectra of control cotton and the cotton treated with DMBHP.

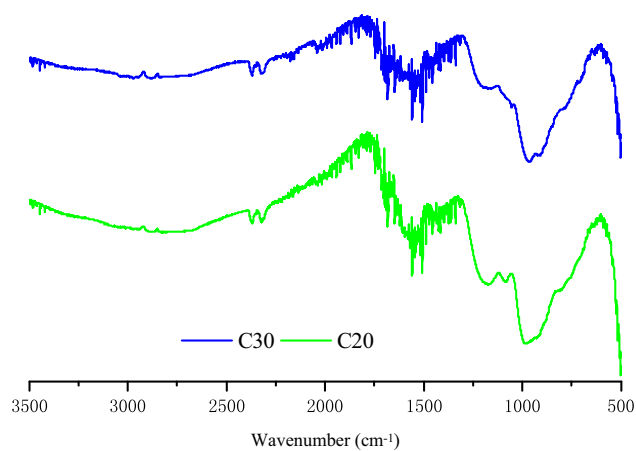


Figure 8: FT-IR spectra of the residues of DMBHP-treated fabrics.

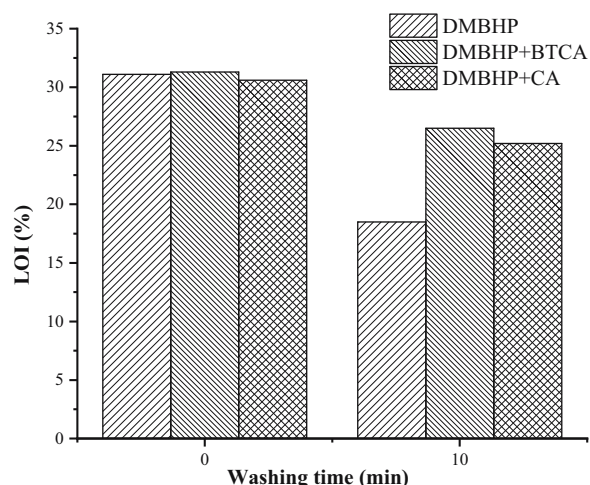


Figure 9: LOI values of the flame retarded cotton fabric (finishing solution: (a) 30% DMBHP; (b) 30% DMBHP + 8% BTCA + 4% SHP; (c) 30% DMBHP + 8% CA + 4% SHP). Other treatment process parameters of (b) and (c) are same with (a)).

implied that the utilization of BTCA and CA can endow the DMBHP-treated cotton a certain durability against washing.

5 Conclusion

A phosphoramidate flame retardant containing hydroxyl groups, named DMBHP, for cotton fabrics was synthesized successfully and characterized via FT-IR and NMR. The synthesized compound was applied onto cotton fabrics with different weight gains in the range of 7.9–24.7% using the pad-dry-cure method. The flammability test results revealed that the fabric with a weight gain of 7.87–24.86% could achieve a limiting oxygen index within the range of 24.5–31.1. The treated cotton fabric passed the vertical flammability test when the weight gain was 15.1%. The cone calorimetric test revealed that flame retardant finishing could significantly lower the heat release rate, and total heat release with increased residue retained. The TG and DTG results revealed that the treated cotton fabric exhibited a lower initial decomposition temperature and greater amounts of residue compared to the control pure cotton. SEM-EDS of the treated cotton fabric before and after burning demonstrated the flame retardant action of DMBHP in the condensed phase promoting char formation. Furthermore, the synthesized flame retardant would be bounded to the cellulose through the cellulose crosslinking agents as the alkyl hydroxyl groups in DMBHP are similar to that of cellulose.

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