THERMAL PROPERTIES OF FLAME RETARDANT COTTON FABRIC GRAFTED BY DIMETHYL METHACRYLOYLOXYETHYL PHOSPHATE

by

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Thermal properties of flame retardant cotton fabric grafted by dimethyl methacryloy-loxyethyl phosphate were investigated by the atom transfer radical polymerization method. Thermal gravimetric analysis was used to explore the thermal decomposition mode of flamed retardant cotton fabric. The weight loss rate of the flamed retardant cotton was bigger than that of the control cotton fabric, and a more final residual char of flamed retardant cotton was also observed. Flammability tests were used to study the flame retardance property of the flame retardant cotton fabric. The results showed that flamed retardant cotton fabric with 16.8% of weight gain could keep good flame retardance. Scanning electron microscope pictures were applied to investigate the morphology of residual char of the flame retardant samples.

Key words: cotton, flame retardance, thermal properties, thermal gravimetric analysis

Introduction

Cotton is the most abundant polymeric raw material in the world. This inexpensive, biodegradable, and renewable resource has been widely studied during the past decades. Cotton fabric has many useful properties, but cotton fiber is kind of quite combustible fiber – limited oxigen index (LOI) \approx 18.4%. Halogen-free phosphorus based compounds are the flame retardants most frequently used on cotton textiles. The most common method to impart cotton fabrics with flame retardance involves the polymerization or the grafting reactions [1]. In the present work, thermal properties of flame retardant cotton fabric (FR cotton) grafted by dimethyl methacryloyloxyethyl phosphate (DMMEP) via atom transfer radical polymerization (ATRP) method were investigated. DMMEP was self-made according to the reported reference [2].

Experimental analysis

Thermal gravimetric analysis

The thermal stability of FR cotton was measured by TGA (fig. 1). It could be seen that control cotton fabric and FR cotton with different weight gains had similar weight loss behavior. At low temperature (from 50 °C to 220 °C), the weight loss in this stage was slight, which was as-

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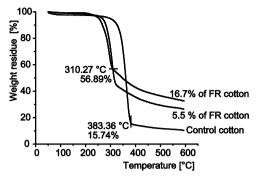


Figure 1. TGA curves of cotton samples

signed to the loss of the absorbed moisture of cotton fabric. From 220 °C to 390 °C, the weight loss was the most, which was attributed to the decomposition of cellulose. At 600 °C, the decomposition nearly finished, the remain was char residue. For control cotton fabric, the weight loss was 4.08% at 278 °C; while the weight loss intensified with the temperature increasing and reached 84.26% at 383 °C. Then the weight loss became slowly, the final residual char was 10.71% at 600 °C. Compared with the control cotton fabric, the weight loss velocity of the FR cotton was more rapid. This was due to the phos-

phorous based flame retardant could convert into phosphoric acid at high temperature, and it further turned into metaphosphoric acid, poly-metaphosphoric acid, which could catalyze the dehydration carbonization of cotton fibers [3]. The weight loss of 16.7% of weight gain FR cotton was 43.11% at 310 °C. The final residual char increased at 600 °C and reached to 26.12% and 33.08% for FR cotton with 5.5% and 16.7% of weight gain samples, respectively. This could be explained by the introduction of the phosphorous monomer (DMMEP) which helped to form more non-flammable char residue [4]. It could be concluded that the effect of DMMEP flame retardant on cotton fabric accorded with condensation phase retarding mechanism.

Flame retardance

The flame retardance of cotton before and after grafting is listed in tab 1. For FR cotton, the LOI values increased with the weight gain increasing. This was due to the phosphorus content on the cotton surface increased with the increase of weight gain.

Table 1. The flame retardar	ce of grafted cotton fabrics
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Weight gain [%]	LOI [%]		Char length [mm]	
	Before washing	After 5 times of washing	Before washing	After 5 times of washing
0	18.8	_	Burn out	_
5.5	25.1	24.4	132	149
6.9	25.4	24.8	128	142
8.0	26.1	25.2	120	135
16.8	28.1	26.6	115	130

Phosphorus flame retardant could generate phosphorous acid after heating, which formed a nonvolatile protective film and isolated the exterior oxygen. Thus the diffusion of flammable gas forming from the combustion was inhibited and flame retardant purpose was achieved. Accordingly, the increase of phosphorus content was helpful to enhance the flame retardance. The char length of cotton fabric decreased with the increase of weight gain. The FR cotton fabric with 16.8% of weight gain could keep good flame retardance with 26.6% of LOI and 130 mm of char length after 5 times of washing.

Morphology of the char residue

The micrographs of the char residue of cotton samples are shown in fig. 2. Compared with the control cotton fabric, there were many big bubbles on the outer and inner surfaces of charred layer of FR cotton samples.

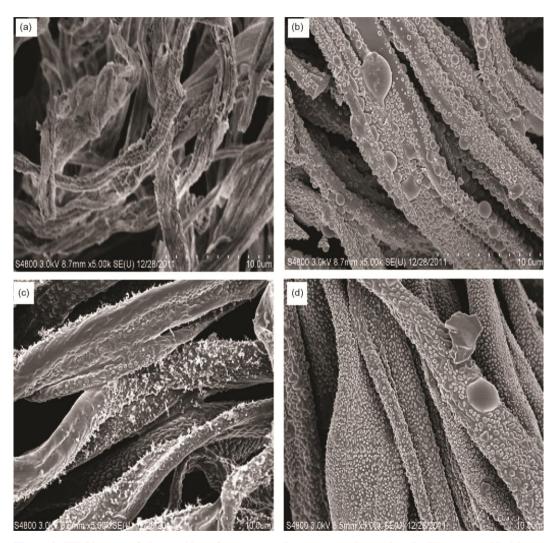


Figure 2. SEM images of char residue of cotton samples; (a) control samples, (b) FR cotton with 6.9% of weight gain, (c) FR cotton with 8.6% of weight gain, (d) FR cotton with 16.7% of weight gain

These bubbles char structure with a continuous outer surface and porous swollen structure could not only inhibit the release from cellulose degradation but prevent the heat source to convey heat for cellulose and insulate the oxygen source. Thus the spread of flame could be effectively prevented. With the weight gain increasing, the bubbles structure on the char residue surface became denser and had better flame retardance.

Conclusions

TGA and flame retardance measurements indicated that the flame retardant cotton fabric grafted by DMMEP via ATRP method had certain flame retardance and washing durability. DMMEP flame retardant on cotton fabric accorded with condensation phase retarding mechanism. SEM images of char residue of FR cotton fabric showed the bubbles char structure with a continuous outer surface and porous swollen structure, which could prevent the spread of flame.

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