The preparation of flame-retardant poly (ethylene terephthalate) fabric by ionic agent

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Abstract. In this paper, PET fabric was modified by the flame retardant HOP to improve flame retardance. The results showed that the flame retardance of PET fabric improved a lot. With the increase of the HOP concentration, the limiting oxygen index could increase to 34%, the horizontal burning length could decrease to 0 mm, the horizontal burning rate could decrease to 0 mm/s, and the vertical ignite time could increase to 5 s. The infrared spectroscopy showed the modification of HOP made PET fabric gain phosphate groups. Moreover, the breaking strength, stiffness and crease recovery angle were kept well.

1. Introduction

Poly (ethylene terephthalate) (PET) is wildly used in many fields, such as clothing, home furnishings, engineering domain and so on due to its many outstanding properties like good chemical stability and excellent mechanical properties [1]. However, PET fabric is combustible fabric and it may cause fire which will end up with bodily injury and financial loss [2]. Therefore, the flame retardant modification has great significance and there are many studies intending to impart PET fabric with flame retardance [3-5].

It is commonly to use intumescent flame retardant and phosphorus-based flame retardant to improve the flame retardance of PET fabric. The flame retardant modification by using intumescent flame retardant belongs to gaseous phase flame retardancy that has high flame retardant efficiency [6], while the flame retardant modification by using phosphorus-based flame retardant belongs to solid phase flame retardancy that reduces combustible gas [7]. Considering of the safety of some intumescent flame retardants, phosphorus-based flame retardants are mainly applied in fabric flame retardant.

The aim of this study was to impart high-efficiency flame retardance to PET fabrics using the flame retardant HOP. The flame retardant HOP can introduce phosphorus into PET macromolecular by chemical reaction in order to improve the flame retardance of PET fabric. This method for modifying PET fabric is not only rapid but also efficient.

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2. Experimental

2.1. Material

Polyester fabric (27.0tex×22.2tex/500×360) was bought from Chongqing market. HOP [self-synthesized, $(HO)_2OP\text{-}R\text{-}PO(OH)_2$, molecular weight is 392, which react with PET fabrics to generate -PO(OH)-PET] was a flame retardant agent.

2.2. Flame retarded modification of PET fabrics

Flame retardant agent HOP could be dissolved in water. HOP solutions with different concentrations ranging from 10 g/L to 30 g/L were made with distilled water.

The PET fabric (50 cm × 70 cm) was immersed into HOP solution for 5 minutes at room temperature and the bath ratio was 1:20. After PET fabric was soaked, the PET fabric was dried at 60^{o}C for 30 min and baked in an oven at 125^{o}C for 25 min. The baked PET fabric was then washed and dried at 60^{o}C. This procedure was repeated thrice.

2.3. Test for flame retardant

2.3.1. Burning behavior. The limiting oxygen index (LOI) values of different treatments PET fabrics were measured according to an international standard ASTM D2863-2000 with M606B digital oxygen index apparatus from Qingdao Shanfang Instrument Co. Ltd., Shandong, China.

The horizontal burning behavior of PET fabrics was measured according to ASTM D 5132-2004, the standard test method for horizontal burning rate of polymeric materials used in occupant compartments of motor vehicles with the YG815D horizontal fabric FR tester from Nantong Sansi Electromechanical Science & Technology Co. Ltd., China.

The vertical ignite time of the PET fabrics treated by HOP were measured by the YG815B vertical fabric FR tester from Nantong Sansi Electromechanical Science & Technology Co. Ltd., China, according to ISO 6940:2004, Textile fabrics-Burning behavior-Determination of ease of ignition of vertically oriented specimens, MOD.

2.3.2. Characterization of the Modified PET Fabric Samples. Fourier transform infrared (FTIR) spectra of the fabric samples were acquired with a Spectrum GX spectrometer (PE Co., U.S.). The spectra were acquired over the range 2500-400 cm^{-1} using the reflection-absorption technique.

2.3.3. Mechanical properties. The tensile strength of fabric sample was measured according to ASTM 5035-2006 standard with an Electronic fabric tension tester, HD026N (Nantong Hongda Experiment Instruments Co. Ltd., China).

Based on ASTM D 1388-96 (2002), the standard test method for stiffness of fabrics, the stiffness of fabric was measured using a fabric stiffness tester (Changzhou, China).

Crease recovery angles of treated fabrics were measured using a wrinkle recovery tester (Wenzhou, China) according to AATCC Test Method 66-2008 for wrinkle recovery of woven fabrics: recovery angle.

Table 1. The limiting oxygen index, horizontal burning behavior and the time to ignite the fabric of the polyester treated by HOP.

Concentration of HOP (g/L)	**0**	**10**	**20**	**30**
The limiting oxygen index (%)	**21**	**26**	**28**	**34**
Hrizontal burning length (mm)	**254**	**74.3**	**32.5**	**0**
Hrizontal burning rate (mm/s)	**5.5**	**4.5**	**3.7**	**0**
The time to ignite the fabric (s)	**1**	**1**	**2**	**5**

3. Results and discussion

3.1. Flammability of PET fabric modified by HOP

Table 1 showed the limiting oxygen index, horizontal burning behavior and vertical ignite time of the original PET fabric and the modified PET fabrics.

From the table, it can be observed that the limiting oxygen index increased when the concentration of HOP increased. The limiting oxygen index of original PET fabric was 21%, and it could increase to 34% when the PET fabric was modified by 30 g/L HOP solution for three times. The horizontal burning rate decreased quickly. The horizontal burning rate of original PET fabric was 5.5 mm/s and it decreased to 0 mm/s when the PET fabric modified by HOP with 30 g/L concentration for three times. The horizontal burning lengths of original and modified PET fabrics decreased quickly with the increase of the HOP concentration. The horizontal burning length of original PET fabric was 254 mm/s, the fire spread fast and the sample were burned out. The horizontal burning length could decrease to 0 mm when the PET fabric was modified by 30 g/L HOP solution for three times. And the time for the modified PET fabric to be ignited by vertical method became longer. It cost 1 s to ignite the original PET fabric, and the ignite time increased to 5 s after PET fabric was modified by 30 g/L HOP for three times. After modified by flame retardant HOP, the PET fabric was difficult to be ignited. The flame retardance of PET fabric improved greatly.

3.2. Characterization of the modified PET fabric samples

Figure 1 was the infrared spectroscopy of PET fabric before and after modified by HOP. The concentration of HOP was 30 g/L and the process was repeated thrice. As shown in the figure, the modified PET fiber had stronger absorption peaks at 570 cm^{-1}, 600 cm^{-1}, 650 cm^{-1}, 1270 cm^{-1} and 1610 cm^{-1}. The moderately strong absorption peaks 570 cm^{-1}, 600 cm^{-1}, 650 cm^{-1} were assigned to the P-C single bond, which indicated that there was a dehydration reaction between HOP and PET macromolecular, and phosphorus was grafted onto PET macromolecular. At the absorption peak of 1270 cm^{-1}, the absorption of modified PET fiber had strengthened. It was assigned to the P=O exists.

At 1610 cm^{-1}, a characteristic absorption peak of carboxylate appeared. It showed that there were transesterification and hydrolysis reaction between HOP and PET macromolecular. From the modification process, although PET fabric was soaked in HOP for 25 min, the water on the surface of fabric would rapidly volatilize in 125^{o}C. There were a few chances for PET fiber to be hydrolysis. Carboxyl was supposed to appear by transesterification.

HOP is a flame retardant agent containing phosphorus, the structure of HOP is $(HO)_2OP$-R-$PO(OH)_2$. HOP can react with PET fabrics to generate -PO(OH)-PET, as shown in scheme 1. Therefore, the modification of HOP made PET fabric gain phosphate groups, and the PET fabric could get flame-retardant phosphorus to improve the flame retardance.

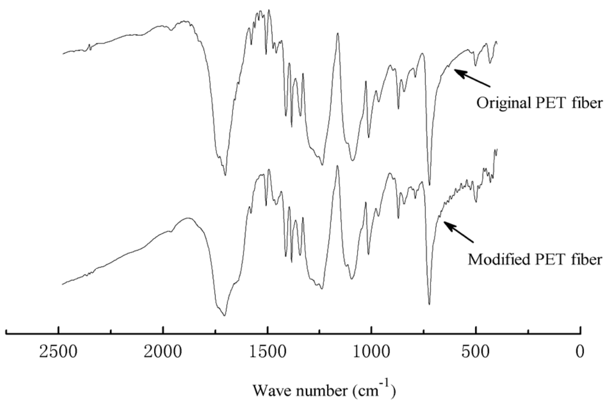
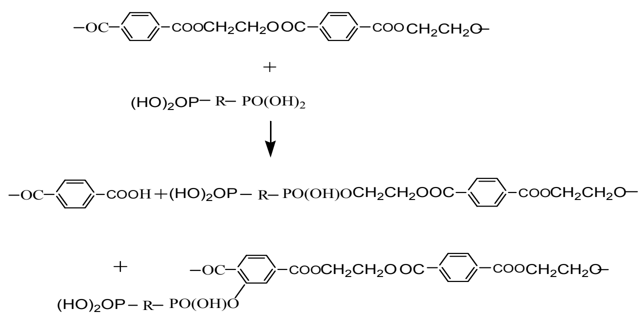


Figure 1. FTIR spectra of the original and modified PET fabrics.



Scheme 1. The possible reaction equation of the modification.

3.3. Breaking strength of modified PET fabric

The relationship between the HOP concentrations and the breaking strengths of original and modified PET fabric was shown in figure 2. From figure 2, it can be seen that the breaking strengths decreased gradually as the HOP concentration increased. The breaking strength of the original PET fabric was 834.00 N, and the breaking strength decreased to 709.20 N when the PET fabric modified by 30 g/L HOP for three times. Overall, the breaking strength of the modified fabric was still high enough to be used in textile.

3.4. Stiffness of modified PET fabric

Figure 3 showed the relationship between the HOP concentrations and the bending lengths. According to figure 3, the bending length increased a little as the HOP concentration increased. The bending length of the original PET fabric was 24.02 mm, and the bending length increased to 26.28 mm when the PET fabric modified by 30 g/L HOP for three times. This indicated that the modification process had only a small effect on the stiffness of the PET fabric, and the limpness was kept well.

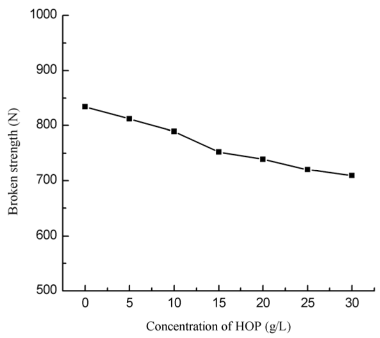


Figure 2. The relationship between the HOP concentrations and the breaking strengths.

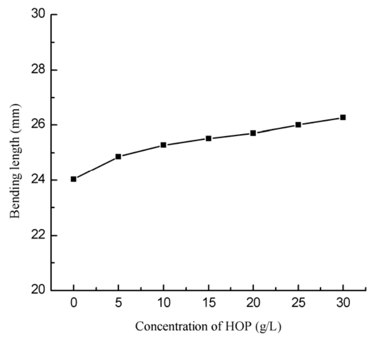


Figure 3. The relationship between the HOP concentrations and the bending lengths.

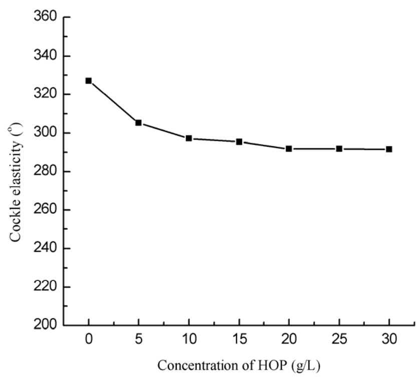


Figure 4. The relationship between the HOP concentrations and the crease recovery angles.

3.5. Crease recovery angle of modified PET fabric
Figure 4 showed the relationship between the HOP concentrations and the crease recovery angles. From figure 4, it can be seen that the crease recovery angle decreased a little as the HOP concentration increased. The crease recovery angle of the original PET fabric was 327.10^o, and the crease recovery angle decreased to 291.50^o when the PET fabric was modified by 30 g/L HOP for three times. The crease recovery angles decreased slightly, and they were still high enough actually.

4. Conclusions
The modification with flame retardant HOP could endow high flame retardance to PET fabric. The flame retardance of PET fabric improved a lot when the fabric was modified by 30 g/L HOP for three times. The modification of HOP made PET fabric gain phosphate groups to improve the flame retardance of PET fabric. When the concentration of HOP increased, the horizontal burning rate decreased rapidly, and the vertical ignite time increased quickly. When PET fabric was modified by 30 g/L HOP for three times, the horizontal burning rate decreased to 0 mm/s, and the vertical ignite time increased to 5 s. At the same time, the breaking strength, stiffness and crease recovery angle were kept well.

Acknowledgments
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