Development of Flame Retardant sheets for Industrial Material (3) - Flame retardant finishing by one step dyebath process -

Youn Jung Park, Ye Jeong Kim, Hea In Kim, Soo Min Park

Department of organic material science and engineering, Pusan National University San 30 Jangjeon-dong Geumjeong-gu Busan 609-735, Korea E-mail: soominpark@pusan.ac.kr

1. INTRODUCTION

During latest two decades, the flammability of commercial polymers has received global attention. Poly(ethylene terephthalate)(PET) can be used to produce high-performance engineering thermoplastics or fibers because of its good thermal stability, chemical resistance, and excellent mechanical properties. However, like other organic polymeric materials, the flammability of PET is a shortcoming in some application. The improvement of the flame retardancy of PET fiber via the introduction of an additive flame retardant has been reported in the literature. Flame retardants are materials that inhibit or resist the spread of fire.

The means of combustion and the flame retardancy of polymeric materials are very complex processes. Despite tremendous progress in studying the flame retardancy of polymeric materials, ideal flame retardant finishing for PET materials are one of the underdeveloped technology in our industry sheet finishing fields, and to contribute to increase in domestic demand and export.

The halogen-containing flame retardants play an important role due to their high efficiency while added into polymeric materials, but their toxicity and corrosion cause environment problem. Some of them have been restricted to use in Europe. There for more and more phosphorus-containing compouds have been used as flame retardants, and especially organophosphorous compounds demonstrated good ability as flame retardants, not causing problems of smoke, toxicity and corrosion.

In this research we used flame retardant finishing by one step dyebath process. This method is constituted dying and flame retardant finishing process at one time. So, it has many advantages such as saving time, increasing production speed and productivity in comparison with existing method.

2. EXPERIMENTAL

2.1 Material

Commercial Poly(ethylene terephthalate)(PET) with an intrinsic viscosity of 0.64dL/g was provided by Dongjin Dyeing Co. Ltd. .

FR1, FR2, FR3 as flame retardant(FR) blended with bromo-FR and/ or Phosphor-FR were used according to its instructions.

2.2 Flame retardant finishing

The flame retardant finishing of PET fabrics was carried out by one step dyebath process with the 3 kinds of FRs at various concentrations. One step dyeing/finishing was processed in a bath containing 2, 4, 6, 8, 10 owf (%) FR, dyes and dyeing auxiliaries according as composition of Table 1.

Table 1. Composition of dying solution containing FR

Auxiliary agent (1 g /l)	FR* owf (%)
Cellow HL 0.189 Newdisper R-50A	0
	2
—	4
DLA	6
HL 0.1560 Dyffer PB	0
	8
	10
	Newdisper R-50A DLA

* FR1. FR2. FR3

The sample was prepared in the Infrared Dyeing Machine according to following procedure, Figure 1. It heated to 135° C with a heating rate of 1.5° C/min and after continued at $135 \,^{\circ}$ C for 40 min. And then the finished fabric was dried at room temperature and treated at 180° C for 2 min using a tenter.

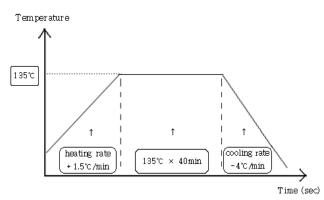


Figure 1. The preparation procedure of Flame retardant finishing by one step dyebath process

2.3 CHARACTERIZATION

2.3.1. FT-IR spectrophotometer

Chemical components of FRs were confirmed by the Fourier Transform infrared spectroscopy (FT-IR, JASCO, Japan). FT-IR spectra of FRs were recorded in the ranged of $4000 \sim 400 \text{ cm}^{-1}$ using a ATR (silicon crystal) at the resolution of 4 cm⁻¹ and 100 scans.

2.3.2. Color strength

Macbath Coloreye 700 spectrophotometer (illuminant D65, 10° observer) was used to evaluate samples based on the Kubelka-Munk analysis (K/S). The K/S values obtained after finishing by one step dyebath process indicate the change of the sample colors.

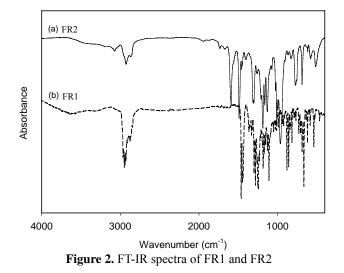
2.3.3. Phase stability

The evaluation of the dispersion stability for dyeing solutions containing FRs was carried out at room temperature using Turbiscan Lab (Formulaction, France)

2.3.4. Thermal property analysis

Thermogravimetric analysis was performed on 2-10 mg samples under air atmosphere at a heating rate of 10° C/min using a TGA 2050 thermogravimetric analyzer. DSC was carried out under nitrogen atmosphere at a heating rate of 10° C/min using a DSC (Shimadzu Co.) analyzer. And FMVSS 302(KSB ISO-3795) method was used for flame retardancy test.

3. RESULTS AND DISCUSSION



FT-IR spectra of FRs, FR1 and FR2, are presented in Figure 2. As seen in the Figure 2(a), the peaks at 1200 and 960 cm⁻¹ correspond to phosphorus-oxygen double bond stretching (P=O) and phosphorus-oxygen single bond stretching (P-O), respectively. The IR spectra indicate that FR2 is containing triphenyl phosphate. From the FT-IR spectrum of the Figure 2(b), it is notable that FR1 is containing hexabromocyclododecane, a bromonated flame retardant.

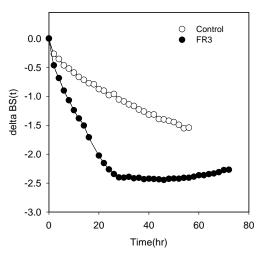


Figure 3. Variation of the mean value for each $\triangle BS(t)$ profile between 5mm and 40mm the samples for dispersions without or with FR3.

The variation in the backscattering (BS) of dispersions without or with FRs shows the sedimentation phenomena; the migrations due to the flocculation by coagulation among dyes, FRs, and dyeing auxiliaries. For the dispersions containing FR3 in Figure 3, $\triangle BS(t)$ values more rapidly decrease.

Table 2. ΔE for untreated PET of PET samples treated with different FRs at various concentrations.

Conc. FRs	2	4	6	8	10
FR1	0.182	0.166	0.148	0.428	0.275
FR2	1.608	1.641	2.894	2.896	4.637
FR3	0.450	1.212	1.970	2.022	1.985

 ΔE between control and treated sample with FRs shows low value in the case of FR1, a phosphate ester.

5. REFERENCE

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