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Flame Retardancy and Dyeing Fastness of Flame Retardant Polyester Fibers

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1. Introduction

This is a continuation of previously published paper^{1, 2, 3)} in which the dyeing properties and chemical resistances of the flame retardant polyester fiber according to the type of flame retardants were reported.

Polyester, mainly poly(ethylene terephthalate) fiber is widely used for textile apparel, industrial fiber, tire cord, etc., and the demands and supplies are growing annually by 9%. As interest in the danger of fire, the demand for flame retardant polyester fiber has been strong and there have been many researches and developments to improve the flame retardant polyester.

In the our previous publication^{1, 2, 3}, we report the relationships between phosphorous flame retardants and the products such as polymer, fiber according to the type of flame retardants. Flame retardant polyester fiber is used for other purposes not as a normal PET fiber. Normal PET fiber is primarily used in the textile apparel whereas the flame retardant polyester fiber is mainly used for industrial applications such as the upholstery or car interior. Therefore, flame retardant polyester fiber goods are exposed to the open air more than a normal PET fiber. If the flame retardant polyester fiber vulnerable to UV, the application of the fiber is limited. So, the flame retardant polyester fiber must have good weatherability such as good light fastness and anti-UV properties.

In general, Polyester fiber use deluster to reduce the transmission of light. Deluster, commonly titanium dioxide, shows good protection of UV in the fields, preventing the passage of light. However, the titanium dioxide has low band gap, between the ground state and the excited state, it is deteriorated in high dosage and for long periods.⁴⁾

Dyeing of polyester products primarily conducted at high temperature and pressure using disperse dyestuff. Disperse dyestuff is divided into nitrodiphenyl, amine, azo and anthraquinone dyestuff greatly depending on its chemical structure.⁵⁾

In this chapter, flame retardancy and dyeing fastness of flame retardant polyester fiber were compared with those of normal PET fiber in accordance with contents of the flame retardant and deluster. Increasing deluster(titanium dioxide) content in the flame retardant polyester fiber, flame retardancy is lowered, but dyeing fastness shows almost same level. Besides light fastness, dyeing fastness shows similar level with increased phosphorous content compared with normal polyester fiber.

2. Experiments & results

2.1. Fiber preparation

Flame retardant polyester polymers using phosphorous flame retardant were prepared in a same method as the previous report ²⁾. 3-(Hydroxyphenyl phosphinyl) propanoic acid was used as a phosphorous flame retardant, and was supplied from I company in Korea and used without further purification. The phosphorous content was adjusted 0.65wt% as a phosphorous atom on the basis of flame retardant polyester, and the contents of titanium dioxide were adjusted 0, 0.3, 2.5wt% respectively. Generally, the polyester fibers were called the as bright, semi dull and full dull according to the contents of deluster(titanium dioxide). The brief chemical formula of the flame retardant polyester is shown in Figure 1.²⁾

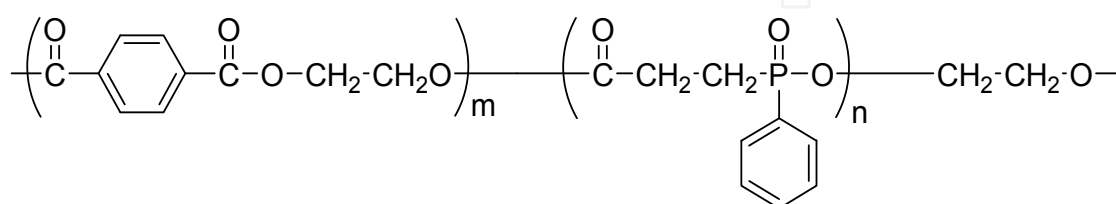


Fig. 1. Chemical formula of the flame retardant polyester

The flame retardant polyester polymers were dried in vacuo, the humidity was less than 25ppm based on polymer weight. And as previously reported, the 75d/36f filament yarns were prepared by spin-draw method.

Dyeing were conducted using hose-knitted sample and under the same condition as previously report. Commercially available anthraquinone disperse dyestuff was used for the dyeing test.

Flame retardancies and Dyeing fastnesses were evaluated by FITI Testing & Research Institute in Korea on the standard of LOI(limit oxygen index, KS M ISO 4859-2:2001) and KS K 0903, respectively.

2.2 Flame retardancy with deluster contents

LOIs according to the titanium content in the flame retardant polyester fiber were shown in Figure 2.

LOI is linearly decreased along with increase of titanium dioxide contents.

Decrease of flame retardancy due to titanium dioxide is not clearly defined, however, it is assumed that by photocatalytic property of anatase type of titanium dioxide. Titanium dioxide used in this experiment was anatase type produced from Sachtleben Company in Germany under the brand name of Hombitan LWSU. In polyester fiber industry, anatase type titanium dioxide is usually adopted. Another type of titanium dioxide, rutile type, is not adopted in polyester fiber industry. Between both types of titanium dioxides(anatase vs rutile) , anatase type titanium dioxide has a bandgap of 3.2eV ^{6,7)} as shown in Figure 3.

In general, most of the phosphorous compounds, including phosphorous flame retardants in this chapter, are known to lower the photocatalytic activity of titanium dioxide. It is assumed that the flammability is decreased by increase amount of titanium dioxide which has photocatalytic activity⁸⁾. However, if the good has the LOI value over 30, it is known that it is good flame retardant performance.

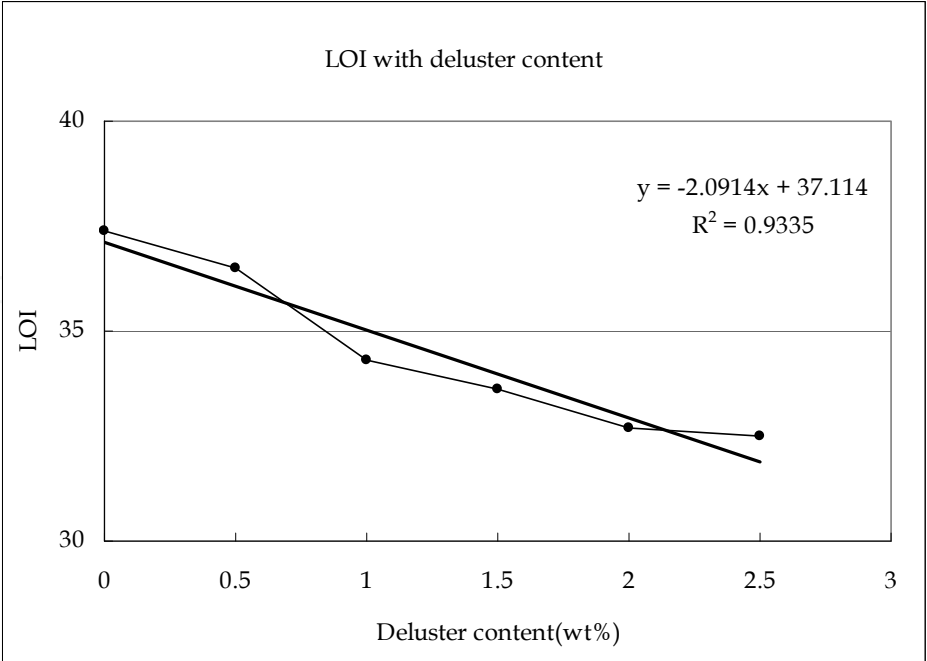


Fig. 2. Flame retardancies with deluster contents

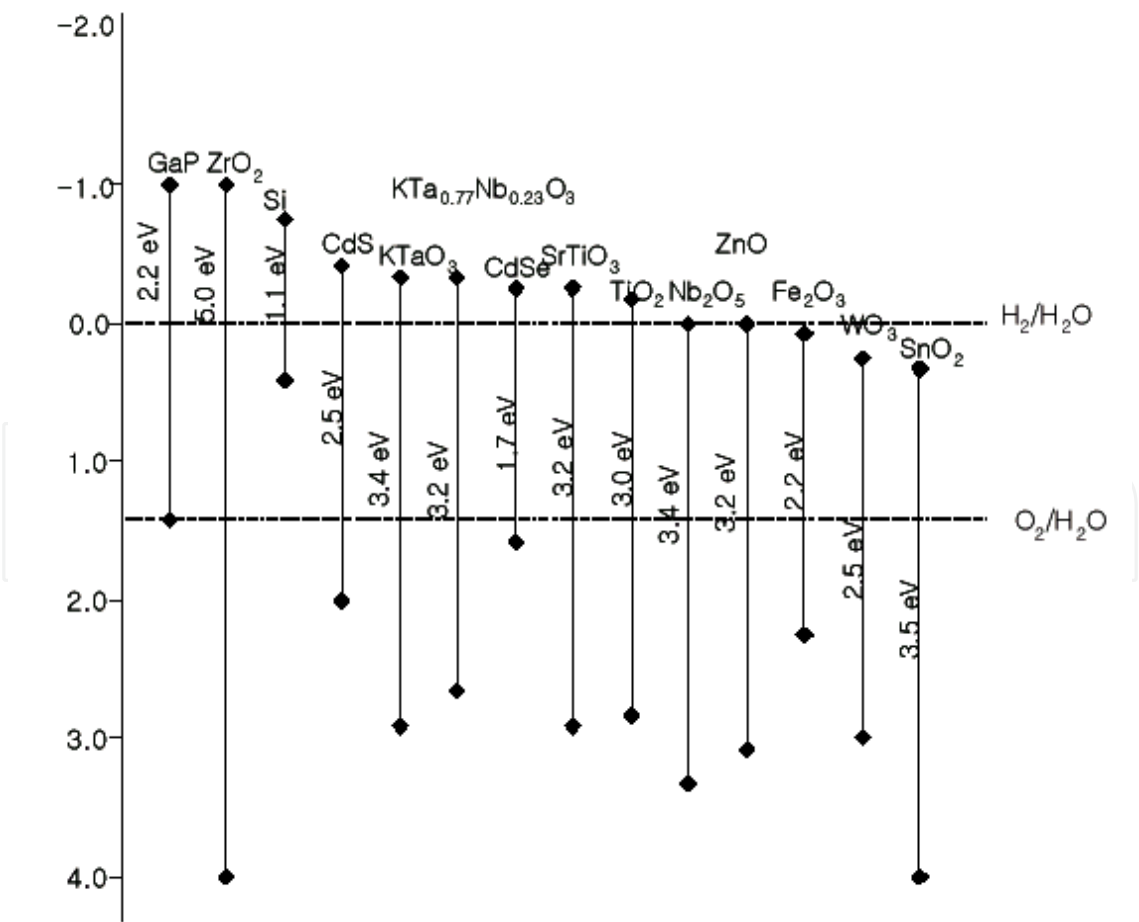


Fig. 3. Bandgaps of various semiconducting metals

2.3 Dyeing fastnesses with deluster contents

Table 1 shows dyeing fastness of the flame retardant polyester fibers along with the contents of titanium dioxide used as Deluster.

Deluster conent(wt%)	Washing	Rubbing	Sublimation	Light
0%	4~5	4~5	4~5	3
0.3%	4~5	4~5	4~5	3
2.5%	4~5	4~5	4~5	3
Normal PET(0.3%)	3~4	4~5	4~5	5

Table 1. Dyeing fastnesses(grade) with deluster contents.

Among dyeing fastnesses, rubbing and sublimation fastness showed similar trend with Normal PET, but washing fastness was better than Normal PET, light fastness was lower than Normal PET.

It is assumed that washing fastness is related to chain mobility of polyester polymer caused by the difference of glass transition temperature. Flame retardant polyester fiber has lower glass transition temperature than normal PET. Therefore, it has better chain mobility than normal PET, and the uptake of the dyestuff at the same temperature is higher than that of normal PET. 2)

Decrease of light fastness is assumed that the influence of photocatalytic activity of titanium dioxide above mentioned. Under the same condition, light fastness using azo disperse dyestuff has 3~4 grade of level which is somewhat better than that using anthraquinone disperse dyestuff, but the dyeing fastness of the flame retardant polyester fiber shows still inferior property compared to normal PET.

2.4 Light fastnesses with phosphorous contents

We can see the dyeing fastnesses are almost same level of flame retardant polyester and normal polyester except for the light fastness as shown in Table 1.

The effect of phosphorous content in the flame retardant polyester on Light fastness was conducted by varying the phosphorous contents in the polymer(deluster contents are same as 0.3% by weight), and the result is shown in Table 2.

Phosphorous content (wt%)	0(Normal PET)	0.5%	0.65%
Light fastness	5	3~4	3

Table 2. Light fastness with phosphorous contents.

Light fastness of Flame retardant polyester fiber is inferior to that of normal PET. The difference is brought about by the content of phosphorous flame retardant, not by the content of titanium dioxide. For that reason, there is a necessity for flame retardant polyester

fiber to improve light fastness for using industrial applications such as upholstery and car interior, etc.

2.5 Light fastnesses improvement

To improve the light fastness of the flame retardant polyester fiber, following method was chosen and conducted ;

1. Increase of inorganic UV stabilizer(manganese acetate) in polymer
Manganese acetate is known to be incorporated into the polymer as UV stabilizers under polymerization of flame retardant polyester⁹⁾. Flame retardant polyester fibers incorporated manganese acetate as the basis of 60, 120, 200, 300 ppm of manganese metal in polymer were polymerized. And the polymers were spun in a same method as above mentioned. The results of the light fastness were shown in Table 3.

Manganese content(ppm)	60	120	200	300
Light fastness	3~4	3	3	3

Table 3. Light fastness with manganese contents.

Table 3 shows that the light fastness decreases with the content of manganese metal. Above all, the coagulation of the manganese acetate brought about the increase of filtration pressure in the polymerization and spinning process to conducting long period test. If the light fastness is improved, this method would be difficult to apply to commercial production of flame retardant polyester fiber.

2. Incorporation of organic UV stabilizer into polymer
To improve the light fastness of the flame retardant polyester fiber, some UV stabilizers were recommended by UV stabilizer maker. The basic properties of the UV stabilizers used in this test were shown in Table 4. The UV stabilizers were incorporated in polymerization reactor as in the state of ethylene glycol dope and the contents of the UV stabilizers were adjusted to 0.3 wt% based on the polyester polymer. The fibers are prepared as same method above mentioned.

Light fastnesses of all flame retardant polyester fibers incorporated various UV stabilizers how negligibly little difference as compared with that not containing the UV stabilizers.(same grade of light fastness of 3)

3. Using of UV absorber under dyeing
Benzotriazole type UV absorber as dyeing auxiliary was recommended form dyestuff maker and dyeing test was conducted. It is added to dyeing solution whose concentration is owf 1%, and the other conditions are same as the above dyeing method. The light fastnesses are increased to the grade over 4(at least 1 grade better that of not treated good).
To investigate the effect of benzotriazol UV absorber on the light fastness of polyester fibers, the UV absorption spectrum is shown in Figure 4.

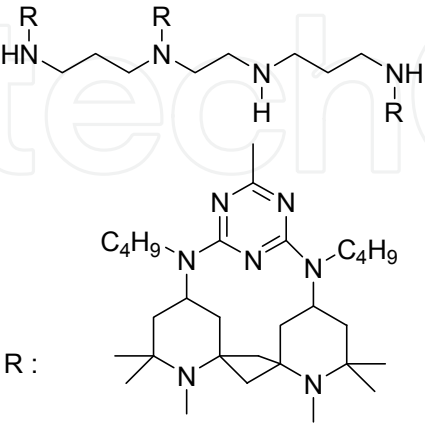
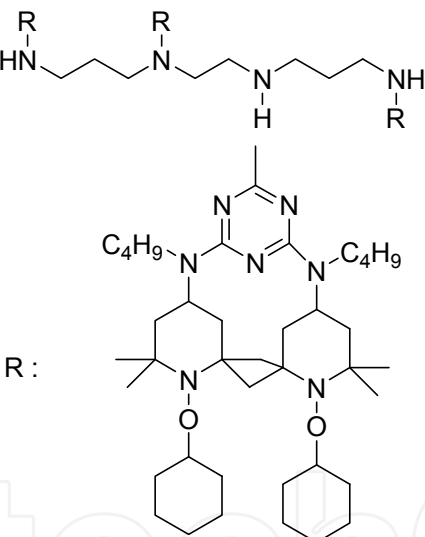
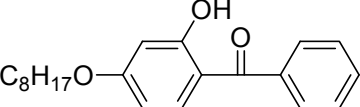
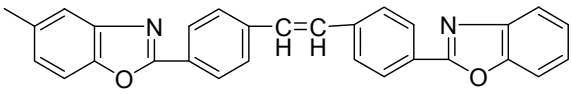
UV Absorber	Chemical structure	Molecular weight	Melting Point (°C)	Remark
Chimassorb 119FL	 R :	2286	115~150	Triazine derivative
Flamestab NOR 116	 R :	2261	108~123	Triazine derivative
Hostavin ARO 8P	 $C_8H_{17}O$	326	>48	Benzophenone derivative
Hostalux KS			250~300	Benzoxazole derivative

Table 4. UV stabilizers used in the test

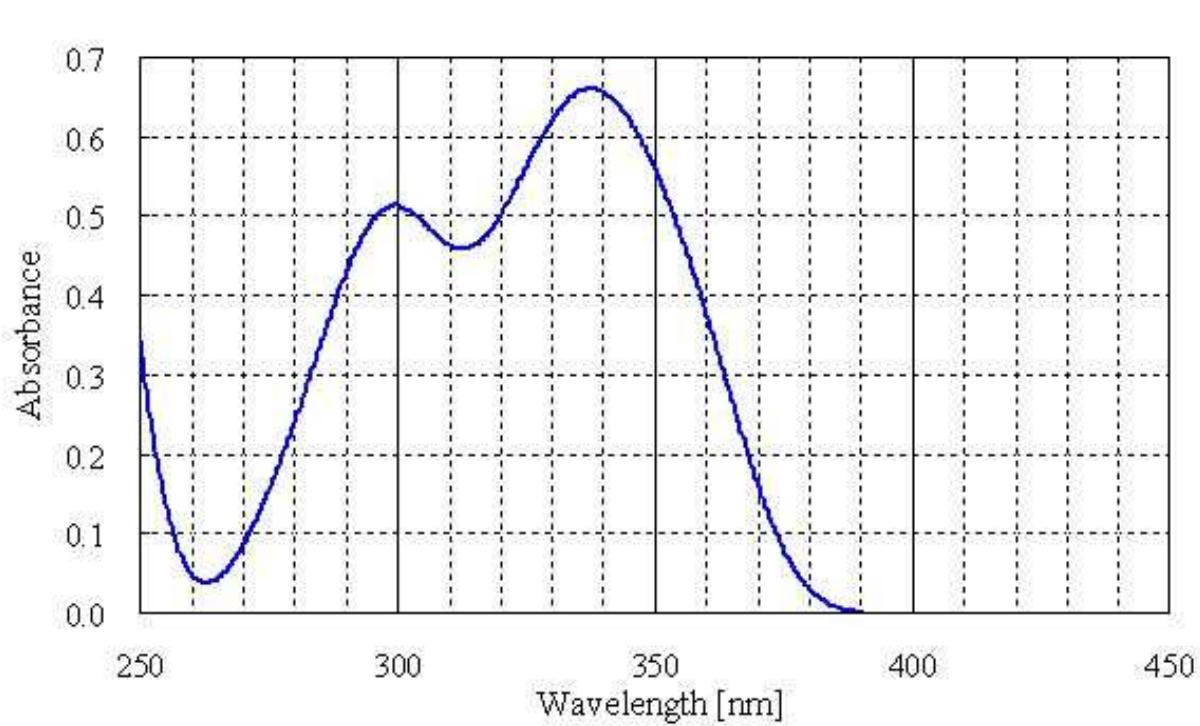


Fig. 4. UV absorption spectrum of benzotriazole UV absorber

Benzotriazole UV absorber is thought to improve the light fastness of the polyester fiber by absorbing the light around 350nm wavelength which is mainly absorbed in polyester. Improving the light fastness using benzotriazole UV absorber can be adopted not only to flame retardant polyester fibers but also to the normal PET fibers.

4. Comparison of the disperse dyestuff

It would be the best way to find a suitable dyestuff to improve light fastness of the flame retardant polyester fiber if it is commercially available. The effect of the disperse dyestuff on light fastness of the flame retardant polyester fiber was compared using the dyestuff kind according to their chemical structure.

From the standpoint of chemical structure, the disperse dyestuffs were categorized into nitrodiphenyl amine dyestuff, azo dyestuff and anthraquinone dyestuff. Among these dyestuffs, the azo and anthraquinone dyestuffs are mainly used in commercial scale.

Dyestuffs	Anthraquinone dyestuff	Azo dyestuff (Red)	Azo dyestuff (Blue)
Light fastness	3	Above 4	4

Table 5. Light fastness according to the dyestuff

In Table 5, there is a noticeable difference in light fastness dyed with between azo dyestuff and anthraquinone dyestuff. The light fastness dyed with anthraquinone dyestuff is much inferior to that with azo dyestuff.

It is assumed that insoluble dyestuff is reduced to leuco compound by the high reductivity of the phenyl phosphinic acid in the phosphorous flame retardant ^{2, 6)} as shown in Figure 5.

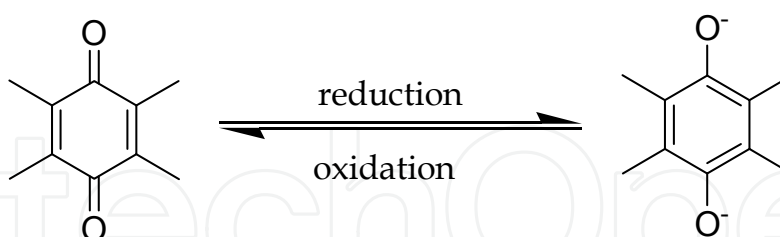


Fig. 5. The brief scheme of reduction state of anthraquinone dyestuff

3. Conclusion

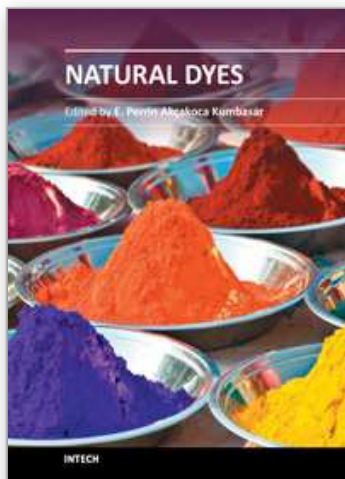
In this chapter, the flame retardancies with deluster contents and dyeing fastnesses with phosphorous contents were investigated.

Whereas titanium dioxide as the deluster affects the flame retardancy of the polyester, it does not affect the dyeing fastness. Titanium dioxide is semi conductor material whose band gap is 3.2eV. Titanium compound in polyester fiber is activated by light, so polyester fibers containing titanium dioxide has color shading problem(yellowing) for outdoor usage. To minimize the yellowing of the polyester goods of polyester fiber with titanium dioxide, phosphorous stabilizer was used. So it could be thought that a part of phosphorous compound act as the stabilizer to reduce the activity of titanium dioxide and the remnant reveals the flame retardancy.

Low level of light fastness of phosphorous flame retardant polyester fiber is due to reductivity of the phosphorous compound. The phosphorous compound shows acidity by the nature of phenyl phosphinic acid to fade out the disperse dye. Light fastness of the phosphorous flame retardant polyester can be minimized by benzotriazole stabilizer⁵. In commercial scale, it is a proper way to improve of light fastness of Flame retardant polyester fiber.

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Textile materials without colorants cannot be imagined and according to archaeological evidence dyeing has been widely used for over 5000 years. With the development of chemical industry all finishing processes of textile materials are developing continuously and, ecological and sustainable production methods are very important nowadays. In this book you can be find the results about the latest researches on natural dyeing.

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