(REFEREED RESEARCH)

INVESTIGATION OF BURNING BEHAVIOR OF POLYESTER FABRIC WITH USING NATURAL STRUCTURED FLAME RETARDANT AGENT

DOĞAL YAPIDA GÜÇ TUTUŞUR MADDE KULLANIMI İLE POLYESTER KUMAŞIN YANMA DAVRANIŞININ İNCELENMESİ

Zeynep ÖMEROĞULLARI*, Dilek KUT

Uludağ University, Department of Textile Engineering, Bursa, Turkey

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ABSTRACT

This paper presents the use of natural structured flame retardant (FR) agent in padding treatment to confer a fire-resistant character to polyester fabrics. The flame retardant agent was obtained from limestone and consisted of any industrial additives and chemical materials. The surface of treated polyester fabric was characterized by FT-IR (ATR) and (SEM). The fire retardant character of the treated fabrics was investigated by LOI measurements. The flame retardancy effect and melt dripping behaviors of FR agent were also investigated. Washing resistances of padded fabrics were examined. According to the results, it was observed that there was a 39.5 % increase in the LOI value and treated polyester fabric was burned in a long time and without dripping.

Key Words: Flame retardant, LOI, Limestone, Polyester, Padding.

ÖZET

Bu çalışma, polyester kumaşa güç tutuşurluk özellik kazandırmak için emdirme yöntemi ile doğal yapıda güç tutuşur madde kullanımını sunmaktadır. Güç tutuşur madde kireç taşından elde edilmiş olup, herhangi bir endüstriyel katkı ve kimyasal madde içermemektedir. İşlem görmüş polyester kumaşın yüzey özelliği FT-IR (ATR) ve SEM tarafından karakterize edilmiş olup, güç tutuşurluk özelliği ise LOI ölçümleri ile değerlendirilmiştir. Aynı zamanda, güç tutuşur maddenin erime ve damlama davranışı da araştırılmıştır. Emdirme işlemi yapılmış kumaşların yıkamaya karşı dirençleri incelenmiştir. Sonuçlara göre, LOI değerlerinde % 39.5'luk bir artış görülmüş ve işlem görmüş polyester kumaşın daha uzun sürede damlama yapmadan yandığı gözlenmiştir.

Anahtar Kelimeler: Güç tutuşur, LOI, Kireç taşı, Polyester, Emdirme.

* Corresponding Author: Zeynep Ömeroğulları, zeynepomeroglu@uludag.edu.tr, Tel:+90 224 2942087 Fax:+90 224 2941903

1. INTRODUCTION

Flame-retardant processes provide textiles with an important performance characteristic. While firefighters and emergency personnel require protection from flames as they go about their duties; floor coverings, upholstery, drapery, commercial carpet, transportation, military and professional racers' garments, bedding and children's sleepware requirements also need flame-retardant textiles to protect themselves from the possible hazards caused by fire (1,2).

The requirements for a commercially successful flame retardant textile product have been given as meeting flammability requirements: having little or no adverse effect on the textile's physical properties; retaining the textile's aesthetics and physiological properties; being produced by a simple process with conventional equipment and inexpensive chemicals; being environmentally friendly and non-toxic (1,3).

Although polyester has a good thermal stability, chemical resistance, and excellent mechanical properties, the flammability and the poor anti-dripping property of PET restrict the range of their applications where specific fire resistance performance is required (6,7). In general, halogen and halogen–antimony systems tend to be flame inhibitors, the phosphorus and boron systems tend to enhance charring and formation of surface barrier layers (2,6). The halogen-containing flame retardants play an important role due to their high efficiency but their toxicity and corrosion cause environmental problems. Some of them have been restricted to use in Europe. Therefore, there is an increasing interest in the development of new environmentally friendly flame retardants to reduce the usage of the common Sb–Br formulations (3-6,8). Phosphorus containing flame retardants for PET are generally concerned because of not causing problems of smoke, toxicity and corrosion. However, melting dripping is still a big problem while burning. In fact, it will bring about a second fire (6). Because of halogen-containing flame retardants are being banned for ecological reasons and new kinds of flame-retardant chemistry which is based on organic phosphonate derivatives are much more expensive; their usage should be limited to the absolute minimum (9).

There are few studies in literature reporting about non-dripping flame retardant additive aimed at PET. It is worthwhile to design an environmentally friendly and effective FRs for PET which combines non-dripping and fire-proofing property. Therefore, in padding treatment, we used a natural flame retardant agent obtained from limestone which improved the melt dripping behaviors and flame retardancy properties of polyester. The flame retardancy effect, flammability and melt dripping behaviors were investigated in this study.

2. MATERIAL AND METHOD

2.1. Material

In this study, 100 % polyester woven curtain fabrics were used as experimental samples to investigate the flame retardancy properties. The fibres of the fabrics were dyed with Dianix S disperse dyestuff at 130^{0}C. The properties of polyester fabric used in this study were given in Table 1.

Table 1. Properties of polyester fabric used in this study.

The properties of fabric		Weave polyester fabric used in the study
Fabric weight		230 g/m^2
Warp yarn	Density	80 warp/cm
	Fineness	75 denier-36 filament
	Twisting	572 Z Tex
	Cross section	Flat
	Color	Semi-dull
Weft yarn	Density	72 weft/cm
	Fineness	150 denier-288 filament
	Twisting	183 Tex
	Cross section	Flat/consists of microfibers
	Color	Semi-dull

The flame retardant agent (Firetex) used in this study was natural structured, consisted of any industrial additives and chemical materials, officially registered by Turk Patent Institute (patent number: 2003 36166). It is colorless, odorless and a clear liquid which has pH value of 8.2. Firetex was obtained from the conversation of limestone rocks into water. Limestone is a sedimentary rock composed largely of the minerals calcite and aragonite, which are different crystal forms of calcium carbonate ($CaCO_3$). The solubility of limestone in water and weak acid solutions leads to karst landscapes. Limestone often contains variable amounts of silica and varying amounts of clay, silt and sand carried in by rivers. Limestone is partially soluble, especially in acid (11).

According to brand registration certificate, it is indicated that Firetex improves the flame retardant properties of wooden products and also textiles especially in the usage area of curtain fabric, woolen carpet and blanket. In addition, due to the toxicity tests, it has not harmful effects onto environment, human beings and animals. As a result of ICP-AES analysis, Ba, Ni and Mg materials were determined in its structure.

Another flame retardant agent (Ruco-flam PSY) which is commonly used in textile industry was supplied by Rudolf Duraner Company (Turkey) and used as a finishing chemical to compare the flame retardant properties with Firetex. The chemical structure of flame retardant agent is alkyl-phosphonate and neutral. Specific weight of FR agent is 1.27 g/cm^3 at 20^{0}C. The pH value of it is 2-5. It has high resistance to acids and high water. It has no pigments; it provides flame retardancy and gives softness to finished fabric handle. The chemical structure of flame retardant agent is alkyl-phosphonate and halojen and antimony trioxide free.

2.2. Finishing Process

During the study, it was realized that the flame retardancy effect of Firetex on polyester fabrics was decreased when Firetex was diluted with water before padding treatment. For this reason, using 300 ml Firetex without water was found appropriate and treated with polyester fabrics in padding bath. Then fabrics were dried at 150^{0}C for 2 min.

Some of the polyester fabrics were also padded with 100 g/L Ruco-flam PSY (pick-up ratio was 70 %), where the pH of the padding bath was between 5-6 pH, the treated fabric was dried at 130^{0}C and cured at 185 ^{0}C for 1-2 min. 100 g/L Ruco-Flam PSY is the maximum concentration of flame retardant agent recommended by the company.

2.3. Washing process

In order to investigate the effect of multiple washings, the treated polyester fabrics were washed five times after the finishing process in Arçelik 4120 S home type washing machine at short washing program at 40 ^{0}C for 60 min with domestic detergent.

2.4. LOI test

To determine the flame retardancy characteristics of polyester fabric after padding with Firetex, LOI test was carried out due to the national American standard of ASTM D 2863-77. A numerical index, the 'LOI', is defined as the minimum concentration of oxygen in oxygen – nitrogen mixture, required to just support downward burning of a vertically mounted test specimen. Hence, higher LOI values represent better flame retardancy (10).

2.5. SEM micrographs

The surface morphology of untreated and treated polyester fabrics were scanned by SEM using ZEISS/ EVO 40 electron microscope at 10 kV. SEM micrographs of different areas of fabrics were taken under a high vacuum, with BAL-TEC SCD 005 coating device, gold-palladium (Au-Pl) coating at 40-50 nm thickness.

2.6. FT-IR (ATR) spectroscopy

FT-IR ATR spectrums of treated and untreated polyester fabrics were investigated by a Thermo Nicolet 6700 device in a wave number range between 525- 4000 cm^{-1}.

2.7. Color Spectrum

Color spectrums of treated and untreated polyester fabrics were investigated by a Konica Minolta Spectrophotometer CM-3600 d.

3. RESULTS AND DISCUSSION

3.1. LOI test results

LOI % of the untreated and treated polyester fabrics were shown in Table 2. According to LOI test results, it was determined that LOI value was increased from 19.7 to 27.5 and an increase of 39.5 % was observed in the LOI value of polyester fabric which padded with Firetex. In case of polyester fabric treated with Ruco-Flam PSY, LOI % was 32.5 after padding and there was an increase of 64.9 %. After washing process, it was indicated that LOI % values of all padded polyester fabrics decreased. But it should be emphasized that the LOI % values of fabrics padded with Firetex and Ruco-Flam PSY were nearly the same after washing process.

Table 2. LOI results of untreated and treated polyester fabrics.

Variation of application / Chemicals and amounts	LOI % of Unwashed polyester fabrics	LOI % of Five times washed polyester fabrics
No FR agent	19.7	19.7
100 g/L Ruco-Flam PSY	32.5	22.1
300 ml Firetex	27.5	22.8

On the other hand, burning behavior of fabrics treated with Firetex was investigated and compared with untreated polyester fabric. As a result of this investigation, it was determined that the time required for burning whole fabric (6x14 cm) which treated with Firetex was 210 sec, while the burning of untreated polyester fabric took approximately 11 sec. So, it was clearly seen that the burning time of fabric treated with Firetex was nearly 20 times bigger than the time required for burning of untreated fabric. To make a right comparison, this burning behavior of fabrics treated with Firetex was also compared with fabrics treated with alkyl-phosphonate-structured FR agent (Ruco- Flam PSY) which is commonly used in flame retardant finishing process. According to results, the time required for burning whole fabric treated with 100 g/L alkyl-phosphonate-structured FR agent was 40 sec. 100 g/L was the maximum concentration recommended by the producing company. It was obviously seen that the burning time of fabric treated with Firetex was 5 times bigger than fabric treated with alkyl-phosphonate-structured FR agent.

Although LOI % of fabric treated with Firetex was lower than LOI % of fabric treated with alkyl-phosphonate-structured FR agent (32.5 %), polyester fabrics burned so slowly and symmetrically. Moreover, no dripping and melting of polyester fabrics treated with Firetex could be seen in the study, despite the fact that they are the typical characteristics of polyester.

As there was no melting or dripping, only carbonization was observed during burning of fabrics treated with Firetex. But, at the end of pad-dry method, it was realized that the fabric treated with Firetex adsorbed moisture and gave a sense of wetness after waiting for half an hour in laboratory conditions. The photograph of polyester fabric treated with Firetex after burning was shown in Figure 2.

3.2. SEM micrographs results

SEM micrographs of untreated, Firetex treated polyester fabrics and 100 g/L Ruco- Flam PSY treated polyester fabrics were shown in Figure 2 (a), (b) and (c), respectively. The residues shown in Figure 2 (b) are denser than Figure 2 (a) and (c). This result can be attributed to materials like Ba, Ni and Mg which present in chemical structure of Firetex.

Figure 1. The photograph of polyester fabric treated with Firetex after burning

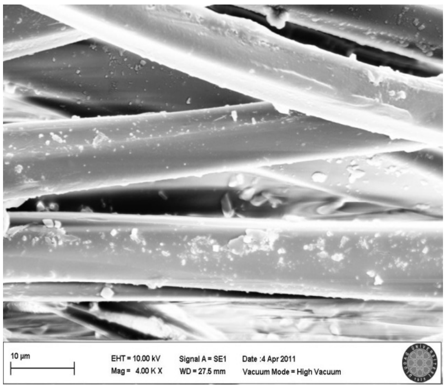

Figure 2. (a) SEM micrograph of untreated polyester fabric

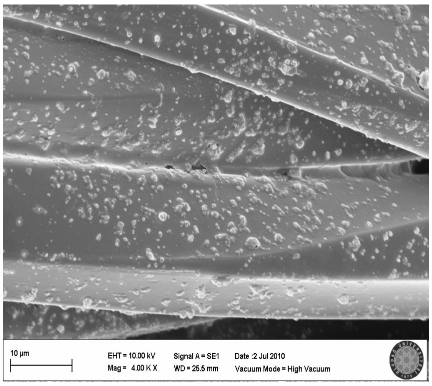

Figure 2. (b) SEM micrograph of Firetex treated polyester fabric

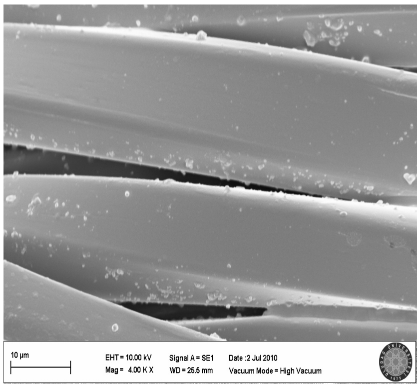

Figure 2. (c) SEM micrograph of 100 g/L Ruco-Flam PSY treated polyester fabric

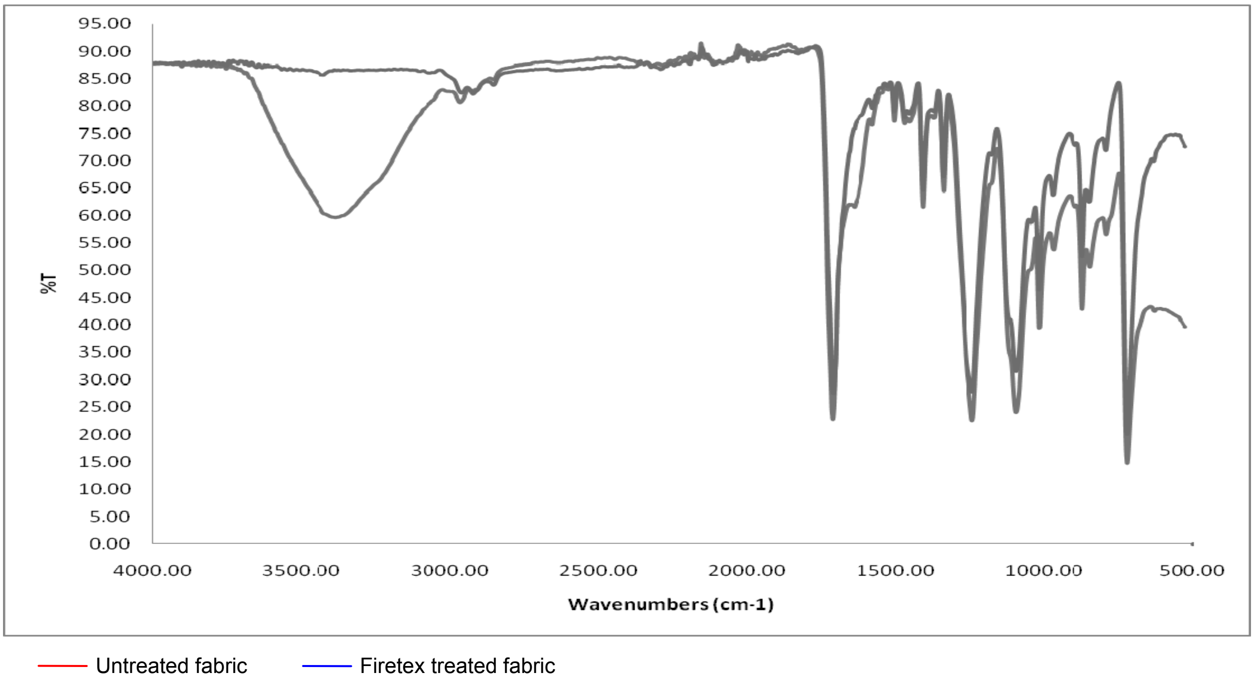

Figure 3. FT-IR (ATR) spectrum of untreated and Firetex treated polyester fabric

3.3. FT-IR (ATR) spectroscopy results

FT-IR (ATR) spectrum of untreated and Firetex treated polyester fabric was shown in Figure 3. The characteristic peaks of the polyester were seen at wave numbers of 1715 cm^{-1} (aromatic ester C=O tension vibration), 1242 cm^{-1} (aromatic ester C-C-O tension vibration) and 1096 cm^{-1} (aromatic ester O-C-C tension vibration).

Characteristic $CaCO_3$ peaks in the spectra do not show any appreciable effects due to the adsorption of water on the surface. However, as water adsorbs on the surface there is the appearance and growth of new bands associated with the bending (1646 cm^{-1}) and stretching (3000–3700 cm^{-1}) modes of adsorbed water (12). In the O–H stretching region, a broad absorption band grows in and this band has distinct structure. The distinct feature in this broad band appears at 3375.2 cm^{-1}. The bending mode absorption band also determines changes in the band structure as a function of humidity.

While the water pick of untreated fabric was at 3426.3 cm^{-1}, the water pick of Firetex treated fabric was at 3375.2 cm^{-1} because of its continents which provide humidity. Since the fabric treated with Firetex adsorbed moisture after pad-dry process, a sharp peak was obtained at the peak of 3375.2 cm^{-1} which symbolizes hydroxyl groups that are at the end of the linkages of polyester.

3.4. Color spectrums results

Color spectrums of treated and untreated polyester fabrics were reported below in Table 3.

As seen in Table 3, the color of fabric padded with Firetex was 23.2 % more dark when compared with untreated fabric. It is considered that the property of moisture adsorption of Firetex padded fabric which was observed during the study (after padding and drying) caused this high color of darkness. Whereas the colors of washed fabrics were lighter in comparison with untreated fabric, the color of fabric padded with Ruco-Flam PSY showed an increase of 7.0 % in color darkness.

Table 3. Color spectrums of treated and untreated polyester fabrics

Polyester Fabric	CIE LAB			∆E (in comparison with untreated fabric)	Depth of Color (in comparison with untreated fabric)
	L*	a*	b*		
Untreated fabric	39.987	-12.931	-12.636		
Fabric padded with Firetex	29.942	-7.723	-9.576	10.877	23.2 % more dark
5 times washed fabric after padding with Firetex	40.016	-13.876	-13.246	1.524	2.6 % more light
Fabric padded with Ruco-Flam PSY	36.253	-11.123	-12.969	3.295	7.0 % more dark
5 times washed fabric after padding with Ruco-Flam PSY	39.474	-12.582	-14.534	1.99	1.2 % more light

4. CONCLUSION

As a result of this study, it was determined that Firetex improved the flame retardancy effect of polyester fabric after treatment. LOI values increased from 19.7 to 27.5. There was a 39.5 % increase in the LOI value.

The burning time of fabric treated with Firetex was 20 times bigger than required time for burning of untreated fabric. This burning behavior was also compared with fabrics treated with alkyl-phosphonate-structured FR agent which is commonly used in flame retardant finishing process. It was determined the burning time of fabric treated with Firetex was 5 times bigger than fabric treated with alkyl-phosphonate-structured FR agent. Washing resistances of treated fabrics were also examined. It was indicated that LOI % values of all padded polyester fabrics decreased after washing process.

The flammability behavior of polyester fabric was also investigated and realized that Firetex treated fabrics burned so slowly and symmetrically. Moreover, there was no dripping or melting, only carbonization was watched during burning. This natural structured flame retardant agent provided an increase in flame retardancy properties and improvement in burning time. However, it was realized that at the end of the pad-dry process, the fabric treated with Firetex adsorbed moisture and gave a sense of wetness after waiting for half an hour in laboratory conditions.

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