AJHS
Asian Journal of Home Science
Volume 13 | Issue 1 | June, 2018 | 289-294
DOI: 10.15740/HAS/AJHS/13.1/289-294
e ISSN-0976-8351 ■ Visit us: *www.researchjournal.co.in*

Research Paper

Characterization of post FR treatedtextile materials: A comparative study

■ Archana Bahuguna **and** Shailaja D. Naik

Received: 22.12.2017; **Revised:** 15.04.2018; **Accepted:** 03.05.2018

■ **ABSTRACT :** Cotton and polyester fabrics were subjected to Atmospheric Pressure Plasma (APP) followed by Flame Retardant (FR) treatment using dielectric barrier discharge plasma with He-O_2 gas mixture by padding mangle; dried and cured on hot air stenter. The main aim of the study was to assess the effect of plasma-FR treatment on fibre topography, structural and functional properties.Surface topography of treated test samples assessed under SEM and revealed about surface erosion of both the fibres through pictorial presentation. Positive enhancement not only in structural properties of FR finished fabrics *viz.,* cloth count, thickness, GSM and dimensional stability but also the flame retardancy of cotton and polyester were noticed. Further, the qualitative improvements induced due to plasma-FR treatment were found to be sustainable on multiple washes. Thus, it is concluded that APP as a pre-treatment increases the fixation of FR agent into the etched fibre surface and induces sustainability of finish at greater levels.

■ **KEY WORDS:** Cotton, Flame retardant (FR), Helium-Oxygen plasma treatment, Polyester, Surface topography

■ **HOW TO CITE THIS PAPER :** Bahuguna, Archana and Naik, Shailaja D. (2018). Characterization of post FR treatedtextile materials: A comparative study. *Asian J. Home Sci.,* **13** (1) : 289-294, **DOI: 10.15740/HAS/AJHS/13.1/289-294.**

See end of the paper for authors' affiliations

Archana Bahuguna
Department of Textile and Apparel Designing, College of Rural Home Science, University of Agricultural Sciences, Dharwad (Karnataka) India
Email : archana5337bahuguna@gmail.com

Textile finishing is a basic requirement for every material either woven or knitted, refers to the processes that converts the material into a usable and more specifically to any process performed after bleaching to improve the look, performance, or hand (feel) of the finish textile or clothing.Nevertheless, this is the right time for textile industries to focus on environmentally safer methods of processing and finishing fabrics. In fact technologists are constantly working on innovative novel techniques to cater diverse applications by modifying fibre morphology. The surface modification is under exploration in order to afford desirable textile performances without deteriorating the fundamental properties. Every textile material has its own distinctive and appreciable textile properties which can be further enhanced by special finishing techniques. Among all natural fibres, cotton is the purest source of cellulose noted for its hydrophilicity, appearance, performance and natural comfort. Whereas, in contrast to cotton, polyester is the hydrophobic synthetic fibre derived from petroleum via condensation reaction of diols and dicarboxylic acids. Both of the fabrics and their blends are abundantly used by the consumers especially for children garments, kitchen wears, home textiles and industries owing to their respective characteristics *viz.*, comfort (cotton) and strength (polyester) but on the contrary both of fibres are highly combustible. Coating of cotton and polyester with non-flammable chemicals is an effective and appropriate approach to reduce inflammability especially for children's wear to avoid fire accidents and damages. Therefore, chemical treatment is necessary to prevent ignition of fire by small flames, which often cause degradation of fabrics at lower temperatures through the process of dehydration (Siriviriyanun *et al.*, 2008 and Wakelyn *et al.*, 2004).

Plasma is a dry processing technique and is an effective means to reduce the use of chemicals, water and energy thus considered eco-friendly. Modification of textile physics by plasma treatment represents great opportunity for improvement on conventional, energy demanding and less eco-friendly technologies. The general reactions to be achieved by plasma treatment are the oxidation of surface substrate, the generation of radicals, and the etching of the surface; when using special gases a plasma-induced deposition polymerization may occur. Moreover, both surface chemistry and surface topography may be influenced to result in improved adhesion properties as well as in the confinement of functional groups to the surface. The flexibility of plasma surface modification has opened up many possibilities for using it in textile processing as a stand-alone process or as a pre-treatment for improving the efficiency of the next process, also known as plasma-assisted processing (Bhat *et al.*, 2011). Thus, the present study is conducted with the objective to assess the effect of laundering on the structural and functional properties of post FR treated cotton and polyester fabrics.

■ RESEARCH METHODS

The test samples selected for the study were plain woven white cotton (85.92 g/m^2) and polyester fabric (50.80g/m^2). The plasma experiments were carried out in the dielectric atmospheric pressure plasma equipment PLATEX-600 (GRINP S.R.L., Italy) at BTRA, Mumbai. Both the sides of the test samples were treated with helium and oxygen gas with a flow rate of 5 l/min and 0.5 ml/min, respectively, at a power 2.5 kW (cotton) and 1.5 kW (polyester) for 30 sec by keeping the electrodes at a distance of 1 mm. Right after plasma treatment the fabric was allowed to pass through finishing range consisted of padding mangle and hot air stenter for drying and curing. Flame retardant finish was carried out by Pad-Dry-Cure method using 250 gpl PYROVATEX® CP NEW (FR) and 20 gpl KNITTEX® FEL (melamine resin). Further, samples was dried and cured for 2 min at 90º C and 160º C, respectively.

Surface topography:

The test samples were analysed for surface topography by S-3700N Scanning Electron Microscope (SEM) with an accelerating voltage of 20kV and magnification power of 2,500X and 10,000X for assessment upto 20 and 5 µm level, respectively.

Laundering:

Treated substrates were hand washed using 2 gpl of surfactant, rinsed well and finally shade dried to find out the durability of finish on multiple washings. The test samples were subjected for a total of 10 washes and quality characteristics were assessed after every 5th wash.

Characterization :

Structural properties:

Cloth count, cloth thickness and GSM were examined as directed in BS 2862:1957, BS 2544:1954 and IS 1964:2001 test methods, respectively. Whereas, cloth shrinkage percentage was measured by using following formula:

$$S = \frac{L_o - L_a}{L_o} \times 100$$

where, Lo – Initial length and La – Final length

Functional property (Flame retardancy):

Ease of ignition and relative ability to sustain the combustion, measures the flammability characteristics of a material was studied by 45° flame test, in terms of seconds as directed in ASTM D1230-94.

Statistical application:

Single factor ANOVA was used to find out the effect of post-FR finish on structural and functional properties of the test samples.

Hypothesis:

The hypotheses set for the study were:

– Atmospheric pressure plasma treatment alters the inherent characteristics of cotton and polyester fabrics

– Laundering does not alter the structural and functional properties of the post FR treated fabrics.

■ RESEARCH FINDINGS AND DISCUSSION

The results obtained from the present investigation as well as relevant discussion have been summarized under following heads :

Surface topography :

Plates 1 and 2 reveal about the surface characteristics of untreated and plasma treated flame retardant cotton fabric; whereas Plates 3 and 4 display the alteration in surface topography of untreated and plasma treated flame retardant polyester fabricat 20 and 5 µm levels, respectively.On one hand, cotton fibre showed a twisted ribbon-like structure caused by spiraling of cellulose fibrils. Conversely on the other hand, polyester fibre observed with smooth tube like structure. But after post FR treatment the morphology of both the fibres changed due to etching thus creating surface roughness. Due to formation of continuous micro-cracks and pores parallel to fibre axis the fibre surface was severely eroded. Moreover, high deposition of finishing agents, resulting from application of FR finish, slightly acid in nature could be observed on the thickened and wrinkled fibre surface (Lam *et al.*, 2011).

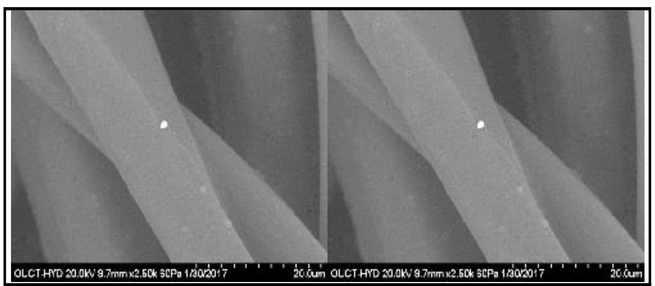

Plate 1 : Surface topography of untreated cotton fabric

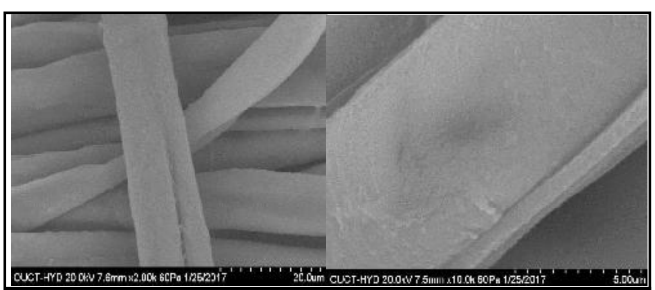

Plate 2 : Surface topography of post FR treated cotton fabric

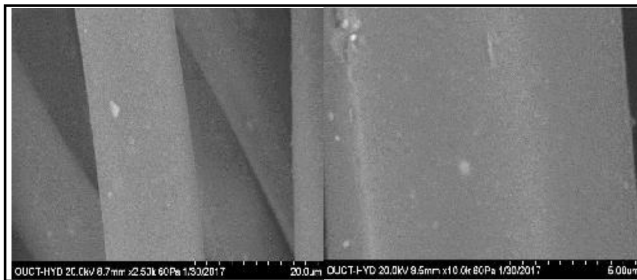

Plate 3 : Surface topography of untreated polyester fabric

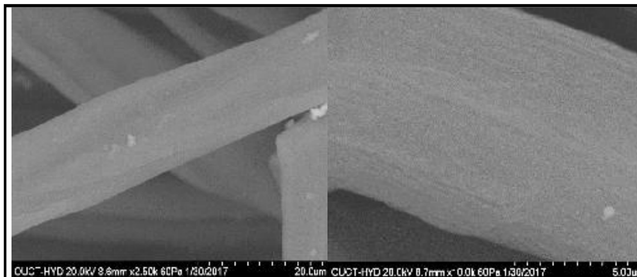

Plate 4 : Surface topography of post FR treated polyester fabric

Effect of washing on the structural properties of post FR treated cotton and polyester fabrics :

The effect of post FR treatment on various structural properties *viz.*, cloth count, cloth thickness, GSM and shrinkage percentage of cotton and polyester fabrics is displayed in Table 1; further the effect of multiple washings on these properties is assessed and compared with the values of treated sample.

Cloth count of the woven textile is the number of ends and picks per unit length and was calculated while the fabric is under zero tension and free from folds and wrinkles. The ends and picks per inch of cotton and polyester is 92:46 and 80:45, respectively at control but the number increased after plasma treatment followed by flame retardancy finish (93:47 for cotton and 81:46 for polyester), and is significant at 1 per cent level of significance.It is noted that the ends and picks of both the fabrics increased in number after plasma treatment followed by flame retardancy finish which is may be attributed to relaxation shrinkage and adjustmentin fibre topography. The sputtering action of plasma led to formation of micro-grooves on the surface of fibre irrespective of their specific inherent characteristics. Moreover, fixation of flame retarding agent onto these grooves altered the overall morphology and structure of fibres, thus enhanced the cloth count of both cotton and polyester fabrics significantly. Further, there was increase in the ends and picks per inch of cotton after 5th wash from 01.08 per cent to 02.15 per cent and 02.17 per cent to 02.12 per cent, respectively but remained unchanged thereafter; whereas there is no change in the cloth count of polyester fabric on subsequent washes. Helium along with oxygen plasma added more number of polar groups because of which cloth count of cotton again increased after 5th wash and thereafter remained constant. Whereas ends and picks per inch of polyester fabric remained unaltered even after subsequent washes. This opponent behavioural change of cotton and polyester on subsequent washes is basically due to the opposite inherent properties of respective fibres. Therefore, the modification is restricted only to the upper most layer of the substrate and did not affect the respective bulk properties (Sparavigna, 2008). Hence, the Null hypothesis set for the study that atmospheric pressure plasma treatment alters the inherent characteristics of cotton and polyester fabrics is rejected.

Cloth thickness is the distance between upper and lower surface of the material, measured under the specified pressure. It is evident from same table that thickness of cotton and polyester fabrics at control was 00.33 mm and 00.25 mm, respectively; which was increased to 00.37 mm (cotton 12.12 %) and 00.28 mm (polyester 12.00 %), after flame retardant finish. This is mainly due to the deposition of FR agent on the abraded region of fibre surface. Meanwhile, the thickness reduced after 5th wash (cotton 02.70 % and polyester 07.14 %) and the values were highly significant. This may be apprehended that some per cent of FR agent was superficially held on the fabric surface during padding mangle and got released from the substrate during multiple washes. However, further laundering did not show any thickness loss which indicated the sustainability of FR finishing. These results are in line with the study conducted by Kan *et al.* (2011) where it was stated that FR coating was able to react directly with fabric through its N-methylol group to form a cross linked polymeric network with the bonding being highly resistant to hydrolysis during multiple home washings.

The findings of cloth weight revealed that there was remarkable increment in GSM of treated cotton and polyester fabrics, from 85.92 g/m^2 to 98.40 g/m^2 and 50.80 g/m^2 to 86.16 g/m^2, respectively. And this increase in GSM is highly significant. The GSM of control sample increased remarkably on post FR finishing and is attributed to increase in yarn density and deposition of FR agent on the etched fibre surface, formed due to sputtering action of highly ionized plasma. The results were supported by Bhat *et al.* (2011) where it was stated that the amount of material deposited, gradually increased the weight of fabric, during plasma treatment.Further, on 5th and 10th washes, the GSM of cotton fabric reduced by 00.89 and 01.38 per cent but however was much higher than control values. Similarly, the GSM of polyester fabric did reduce by 31.68 per cent and 38.07 per cent after 5th and 10th washes, respectively.The reduction in GSM values were corresponding to the values of cloth thickness.

Contraction in the direction of warp and weft refers to 'fabric shrinkage'. Table 1 does display about the cloth shrinkage of both cotton and polyester fabrics after flame retardant finish. The general observation from this Table

Table 1 : Effect of laundering on structural properties of post FR treated test samples

Samples	Structural properties					
	Cloth count (threads per inch)		Cloth thickness (mm)	Cloth weight (GSM)	Cloth shrinkage (%)	
	Warp	Weft			Warpway	Weftway
Cotton						
Untreated	92	46	00.33	85.92	02.54	00.80
Treated (Plasma + FR)	93**(01.08)	47** (02.17)	00.37** (12.12)	98.40** (14.53)	02.41*	00.67**
5th wash	95** (02.15)	48** (02.12)	00.36** (02.70)	97.52 (00.89)	01.34**	00.67
10th wash	95** (02.15)	48** (02.12)	00.36** (02.70)	97.04* (01.38)	01.34**	00.67
Polyester						
Untreated	80	45	00.25	50.80	00.80	00.80
Treated (Plasma + FR)	81** (01.25)	46** (02.22)	00.28** (12.00)	86.16** (69.60)	00.67**	00.67**
5th wash	81 (00.00)	46 (00.00)	00.26** (07.14)	58.86** (31.68)	00.67	00.67
10th wash	81(00.00)	46 (00.00)	00.26** (07.14)	53.36** (38.07)	00.67	00.67

Figures in parenthesis indicate percentages * and ** indicate significance of values at P=0.05 and 0.01, respectively is that the warpway shrinkage is greater than its corresponding weftway shrinkage in cotton fabric; whereas the percentage shrinkage in warp and weft directions in polyester fabric did remain same. Nevertheless, the shrinkage of control samples (cotton 02.54 % and 00.80 %; Polyester each 00.80 %) was highest compared to other stages of testing. This is because the first shrinkage that occurs is referred to as relaxation shrinkage where the warp yarns relax from the tension held during weaving process. The first shrinkage is always maximum; thereafter the shrinkage if any, is always in less percentage and is progressive, hence termed as progressive consolidation which finally leads to dimensional stability.A descending trend of shrinkage is observed in cotton fabric which attained stability after 5th wash. On the contrary, the polyester fabric did not show any shrinkage after plasma treatment (each 00.67 %) *i.e.* the fabric attained dimensional stability after plasma treatment. Due to different inherent properties of cotton and polyester fabrics the rate of shrinkage varied. Moreover, these results are supported with the corresponding values of thread per inch *i.e.* cloth count of respective fabrics.Hence, the null hypothesis set for the study that laundering does not alter the structural properties of post FR treated fabrics is rejected *i.e.* there is change in the threads per unit area, cloth thickness, cloth GSM and shrinkage percentage of plasma treated fabrics after plasma and FR finishing.

Effect of washing on the functional properties of post FR treated cotton and polyester fabrics :

Flame retardants are a key component in reducing the adverse effect of fires on people, property and the environment. Flame retardants are added or treated potentially flammable textile materials by means of chemical finishing that can prevent the ignition and propagation of fire by small flames, often causing the degradation of textiles at lower temperatures through the process of dehydration. Table 2 presents the burning time and char/melt length of plasma treated cotton and polyester finished with flame retardancy. It is clear from this Table that both cotton and polyester untreated test samples easily catch fire within 6 and 7 seconds, respectively with char/melt length of each 15.00 cm, and finally degraded due to very high combustible inherent property. In general, the flame spread on a microscopically raised fabric surface is more rapid than on a smooth fabric surface. In fact, the plasma treatment followed by flame retardant finish did remove the surface fibrils of both the test samples thus, making it smooth and in turn reduced the velocity of burning speed. Hence, elevated the level of test samples from class II to class I (Table 2). In fact, after FR finish the test samples did not ignite which clearly indicated that the post FR finished fabrics were highly resistant to fire. Further, on subsequent washes, the time taken to burn the FR treated samples reduced from DNI to 21 sec and from 21 to 20 sec (cotton); DNI to 19 sec and from 19 to 17 sec

Table 2 : Effect of laundering on flame retardancy of post-FR treated cotton fabric

Samples	Flame retardancy		
	Burning time (sec)	Char/melt length (cm)	Class
Cotton			
Untreated	6	15.00	II
Treated (Plasma + FR)	DNI	03.00	I
5th wash	21	10.00	I
10th wash	20	11.00	I
Polyester			
Untreated	7	15.00	II
Treated (Plasma + FR)	DNI	02.00	I
5th wash	19	10.00	I
10th wash	17	10.00	I

DNI - Did not ignite

According to Consumer Product Safety Commission (CPSC):

Class I : Fabric burning time more than 7 seconds

Class II : Fabric burning time between 4 - 7 seconds

Class III : Fabric burning time less than 4 seconds (polyester) compared to the corresponding untreated samples (cotton - 6 sec and polyester - 7 sec). The increase in phenomenon may be due to the etching effect on the fabric surface caused by plasma treatment. The helium-oxygen plasma removes all the contaminations from the fibre surface and thus may avoid interference of bonding between respective fibres and FR-CL linkages (Kaplan, 2004).

The etching effect reduces the weak boundary layers and increases the surface area thus, allowing greater number of FR molecules to get attached. The attachment of FR molecules ultimately improved the performance of flame retardancy. In addition, the oxygen plasma introduces more polar groups that in turn enhanced the wettability of cotton as well as polyester fibres which may also positively influenced the flame-retardancy of plasma treated samples. But on subsequent washes, degradation of FR-agent from the etched surface to some extent probably reduced the level of flame retardancy, thus making the fabric slightly flammable. But still these test samples belonged to class I category as the burning time was more than 7 seconds.Hence, the hypothesis set for the study that laundering does not alter the functional properties of the post FR treated fabrics is accepted.

Conclusion :

Plasma treatment followed by flame retardant finish significantly altered the surface topography of both cotton and polyester fibres. Due to surface erosion there was formation of micro cracks and pores all along the fibre axis. Post FR finish induced flame retardancy of greater level among both cotton and polyester fabrics. A slight increase in cloth count of both the test samples after plasma treatment was observed. But on multiple washings, the threads per unit area increased remarkably in cotton fabric whereas the values in polyester remained unchanged due to their different inherent characteristics. Deposition of flame retarding agent on microgrooves of cotton and polyester fibre surface and consolidation of yarns due to shrinkage, increased the cloth thickness and the correspondingly GSM. But on multiple washes there was drop in the values and is due to release of superficially held FR agent from the fabric surface to some extent. The relaxation shrinkage was maximum at control; but the percentage of progressive shrinkage gradually reduced on multiple washes. After plasma treatment shrinkage percentage of both fabrics reduced significantly in both the directions *i.e.* warp as well as in weft. The polyester fabric attained the dimension stability at much earlier stage of treatment compared to cotton fabric and is attributed to the respective inherent characteristics.

Authors' affiliations:
Shailaja D. Naik, Department of Textile and Apparel Designing, College of Rural Home Science, University of Agricultural Sciences, DHARWAD (KARNATAKA) INDIA

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