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Fabrication of plasma treated functional cotton fabric

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Abstract
Cotton is the only chief natural fibre that has innumerable advantages of which to be mentioned are high wet tensile strength, abrasion resistance, moisture absorption, breathability, conductor of heat, dye pick up & quick drying and no static problems. But, it burns easily with large yellow flame and propagates when ignited is one of the limitations of this fibre. Hence, it becomes imperative to manage the propagation of fire by selecting suitable eco-friendly finish. Plasma is the innovative technology introduced for textile industries that offers several advantages over conventional wet processing. Plasma is a dry finish which eliminates the recurring expenses incurred on effluent treatments. The fibre morphology of cotton is altered from flat ribbon structure to micro-cracked porous surface at nano-meter level by plasma finish that develops a new dimension for cotton fabric to perform as flame retardant. Though the tensile strength, elongation and abrasion resistance of plasma treated cotton fabric reduced to some extent after multiple washes, but the level of flame retardancy remained elevated when compared to its control value.

Keywords: Cotton fabric, Flame retardant (FR), Plasma treatment and Surface morphology

1. Introduction

Textile materials have tremendous application as clothing; furnishings as well as household goods therefore, play an irreplaceable role in day to day life of every individual. Traditionally, it is known that the major cause for fire spreading is the textile material because of their inflammability as well as ubiquitous presence in our daily lives, in the form of clothing, bed linen and furnishing materials (Siriviriyanun *et al*., 2008) [8]. Among all textile fibres, cotton is used by almost every consumer especially for children garments, bed linen, table linen, kitchen wears and industries because of its inherent characteristics *viz*., comfort, high tensile strength, good abrasion resistance, high moisture absorption, conductor of heat, dye pick up & quick drying and no static problems; but has higher combustibility. Coating of cotton with non-flammable chemicals is an easy and effective approach to reduce inflammability mainly in children's wear. Therefore, chemical treatment is necessary to prevent ignition of fire by small flames or sparks by which often cause degradation of fabrics at lower temperatures through the process of dehydration (Siriviriyanun *et al*., 2008; Wakelyn *et al*., 2004) [8, 9].

Recently, due to increased environmental awareness and stringent effluent norms, textile industries are gradually moving towards implementation of environment friendly processes. Plasma being a dry process is a means to reduce the use of chemicals, water and energy. Modification of textile physics by plasma treatment offers great opportunity on improvement of conventional, energy demanding and less eco-friendly technologies. In plasma surface modification, the changes are principally attributed to the physical or chemical changes in the material because of the high-energy bombardment of plasma or plasma enhanced reactions. The active species produced in plasma do carry a high energy that causes sputtering, cleaning or etching effect, which modify the characteristics of fibre surface.

Thus, the present study is conducted with the objective to develop flame retardant cotton fabric using He - O_2 plasma treatment followed by FR finishing.

2. Material and methods

The details on the materials and methods are presented here under:

Test sample: Bleached plain woven white cotton fabric (85.92 GSM)

Plasma treatment: 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 sample 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 for 30 sec by keeping the electrodes at a distance of 1mm.

Flame retardant finishing: 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, sample was dried and cured for 2 min at 90 ºC and 160 ºC, respectively.

Assessment of Surface Morphology: The test samples were analyzed for surface morphology using S-3700N Scanning Electron Microscope (SEM) at 5 µm level with magnification power of 10,000X.

Laundering: Treated substrate 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.

Assessment of quality characteristics – performance, durable and functional

Performance properties: Cloth bending length and crease recovery angle were assessed as directed under BS 3356:1961 and IS 4681:1968, respectively.

Durable properties: Cloth tensile strength and elongation percentage were tested on Instron tensile strength tester in accordance with ASTM test method: 12616-1989. Whereas, flat abrasion resistance was assessed by Martindale's abrasion tester as directed under IS test method 12673:1989.

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

Statistical Analysis: Single factor ANOVA was used to find out the effect of post-FR finish on performance, durable and functional properties of the test samples.

Hypothesis: Post-Flame Retardant finish alters the performance, durable and functional properties of the test sample.

3. Results and Discussion

Surface morphology

The surface morphology of control and Post-FR treated cotton samples are presented in Plate 1 (a) and (b), respectively observed under ESM at 5 µm level. The cotton fibre showed a twisted ribbon-like structure caused by spiralling of cellulose fibrils in Plate 1 (a). Conversely, in Plate 1 (b) the fibre showed surface roughness caused due to etching on post-FR treatment indicating change in the morphology of the fibres. It is apparent that formation of continuous micro-cracks and pores parallel to fibre axis and the fibre surface was severely eroded. Moreover, high deposition of FR finishing agents that is slightly acid in nature could be observed on the thickened and wrinkled fibre surface (Lam *et al.*, 2011) [5].

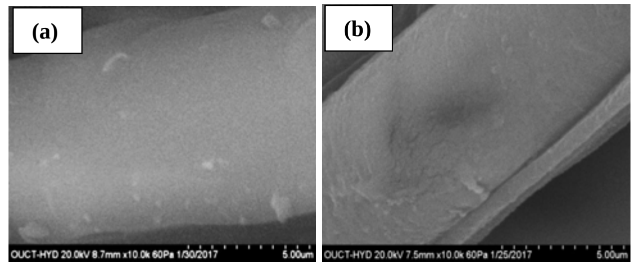

Plate 1: Surface morphology of (a) untreated and (b) post-FR treated cotton fabric

Performance properties

The warpway (02.33 cmle slightly reduced after post-RF treatment compared to its corresponding control values (02.37 cm and 01.95 cm, respectively) and is mainly due to sputtering action of plasma and deposition of flame retarding material into the micro-grooves that induced some percentage of stiffness to the fabrics. This result is in line with the study conducted by Chinnammal and Arunkumar (2014) [1] who stated that all the plain woven plasma treated samples have greater stiffness than their originals except the post dyed samples. Further, these results are also supported by the study conducted by Lam *et al*. (2014) [6] who reported that increased inter yarn friction due to the plasma etching enhanced the fabric softness when subjected to flame retardant treatment. Consequently, on subsequent washes the test samples became further soft and pliable in both directions may be due to removal of some percentage of FR agents that probably reduced the cloth stiffness of the treated fabrics significantly. And the values at 5th and 10th washes are significant at 1 per cent level of significance.

Cloth crease recovery is complimentary to cloth bending length and describes the resiliency of the test sample. The results indicated in Table 1, clearly showed that the weftway (97**) recovery is greater than its corresponding warpway (82**) on post-FR treatment when compared to its control values (76° and 64°, respectively). But there is decrease in the crease recovery angle of FR treated cotton fabric on multiple washes. The change in these values found to be highly significant. It means the level of stiffness reduced on washing, making the test sample more soft and pliable. This may be due to presence of FR agent within the abraded fibre area that in turn would probably restrict the molecular movement of fibre (Palaskar *et al*., 2014) [7].

Further, these results are also supported by the study conducted by Kan (2014) [2], which indicated that etching effect on fabric surface caused by plasma treatment provides a new pathway for the FR finishing agent to enter in to the fibre resulting in increasing the wrinkle recovery angle. Hence, the null hypothesis set for the study that 'post-FR finish alters the performance properties of the test sample' is accepted. The tensile strength, elongation percentage and abrasion resistance are the durable properties assessed after Post-FR treatment.

Table 1: Performance properties of post-FR treated cotton fabric

Cotton samples	Performance properties				
	Cloth bending length (cm)		Cloth crease recovery angle (degree)		
	Warpway	Weftway	Warpway	Weftway	Cloth recovery
Untreated	02.37	01.95	64	76	70
Treated(Plasma + FR)	02.33	01.93	82**	97**	89**
5th wash	01.95**	01.18**	79**	93**	86**
10th wash	01.73**	01.18**	68**	85**	79**

**- Significant at 1 % level of significance

Durable properties

The tensile strength, elongation percentage and abrasion resistance are the durable properties assessed after Post-FR treatment, and the results are presented in Table 1. It is evident from this Table that there was reduction in both warpway and weftway tensile strength (21.90** and 08.89**, respectively) and elongation percentage (07.21** and 04.98**, respectively) of cotton fabric and found to be highly significant after plasma treatment.

These results are supported by the study conducted by Lam *et al.* (2011) [5] where it is mentioned that though roughening effect of plasma creates more contact points in fibres microscopically resulting in increased inter-yarn and inter-fibre friction; but on Post- FR finishing, the cross linking agent (CL) used to bind FR-agent to fibre surface, reduces the strength of textile fabrics. Moreover, drop in breaking load is also attributed to acidity of FR finishing agent; therefore neutralizing the flame-retardant-treated textile specimen with alkali is indispensable.

On subsequent washing, there is a trend of reduction in warpway tensile strength (21.85** and 21.82**) and weftway tensile strength (08.80** and 08.79**) after 5th and 10th washes, respectively and the values are found to be highly significant. This meagre reduction may be due to degradation of FR agent to some extent.

Abrasion resistance is one of the fabric properties that determine the level of durability. The unit for expressing cloth abrasion is ‘cycles’ *i.e.* higher the number of cycles better the resistance is. It is evident from the same Table that, after plasma treatment abrasion resistance of cotton fabric reduced (55**) significantly due to reduction in hairiness caused due to plasma etching which makes the fibre more fragile (Karahan *et al*., 2009) [4]. Moreover, plasma finishing followed by pad-dry-cure and stentering, resulted into flat and smoother surface. Washing of FR treated fabrics further reduced its resistance power (42** and 28** after 5th and 10th washes, respectively) may be because of more distortion in fibre structure. Therefore, the null hypothesis set for the study that ‘post-FR finish alters the durable properties of the test sample’ is accepted.

Table 2: Durable properties of post-FR treated cotton fabric

Cotton samples	Durable properties				
	Cloth tensile strength (kgf)		Elongation (%)		Cloth abrasion resistance (cycles)
	Warpway	Weftway	Warpway	Weftway	
Untreated	22.62	09.40	07.85	05.86	101
Treated(Plasma + FR)	21.90**	08.89**	07.21**	04.98**	55**
5th wash	21.85**	08.80**	07.10	04.95	42**
10th wash	21.82**	08.79**	07.10	04.93	28**

**- Significant at 1 % level of significance

Flammability

A progressive burning of a fabric at a distance of 127 mm from a flame is deemed to be “burn time”, and at less than 7 seconds, is termed as failure of resistance to burning under specified conditions. The flame resistance of the fabric is assessed on the basis of burning time (sec) and char/melt length (cm) and expressed in terms of three classes of fabric where class I (best), and class III (not acceptable) established by Consumer Product Safety Commission (CPSC) (ASTM D1230-94).

Table 3 presents the burning time and char/melt length of post FR treated test sample. It is clear from this Table that untreated test sample could easily catch fire within 6 seconds with char/melt length of 15.00 cm, and finally degraded due to very high inherently combustible 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. In fact, after FR finish the test sample Did Not Ignite which clearly indicated that the post-FR finished fabric was highly resistant to fire. However, on subsequent washes, the time taken to burn the FR treated sample reduced from DNI to 21 (5th wash) and from 21 to 20 seconds (10th washes) compared to the corresponding untreated samples. This 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) [3].

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 fibre which may also positively influence the flame-retardancy of plasma treated sample. 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. Parallelly the test sample belonged to class I category even after 10 washes as the burning time was more than 7 seconds. Thus, the hypothesis set for the study that ‘post-FR finish alters the functional property of the test sample’ is fully accepted.

Table 3: Flame retardancy of post-FR treated cotton fabric

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

4. Conclusion

Plasma treatment followed by flame retardant finish altered the surface topography of cotton fabric. A certain level of reduction in the values of stiffness of cotton on post-FR finish is due to deposition of FR agent on the surface of cotton fibres. On subsequent washes, the cloth stiffness of the test samples reduced. Cloth crease recovery angle of the test sample after FR finishing enhanced compared to control samples. Though there was decrease in the recovery angle after multiple washes but the level of recovery was much better than its corresponding control recovery. Plasma treatment followed by flame retardancy (FR) finishing reduced the tensile strength and elongation of cotton samples but on washing, a meager percentage of reduction was noticed compared to treated sample. Sterilization of cotton during helium-oxygen plasma treatment followed by flame retardant (FR) finishing significantly reduced the abrasion resistance of the test sample. The untreated test samples could ignite and propagate flame at very low burning time whereas post-FR treated fabric 'Did Not Ignite' when approached with flame and the length of char/melt was ignorable; thus, categorized as class I (best). The treated test sample did burn after 5th and 10th wash but took long time to burn, much higher than the time specified for class I *i.e.,* 7 seconds; hence, still categorized under class I. The length of char or melt length of treated sample was relatively lower than the length of char/melt length of untreated sample. Finally it may be concluded that the post FR treatment induced flame resistance to cotton fabric a new venture in fabrication of plasma treated flame retardant cotton fabric, an innovatively engineered functional fabric.

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