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Atmospheric pressure plasma treatment of cotton using hexamethyldisiloxane

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Abstract
Cotton is a pure cellulosic fabric inherently hydrophilic, thus provides maximum comfort to the wearer. But, high moisture content of this natural fibre possesses low resistance to staining and micro-organism attack. Hence, there is an urge for suitable eco-friendly finish that could modify cotton into hydrophobic fabric. Plasma is the innovative technology introduced for textile industries that offers several advantages over conventional wet processing. Plasma is a dry finishing technique and is an effective means to reduce the use of chemicals, water and energy which eliminates the recurring expenses incurred on textile effluent treatments. The plain woven bleached cotton fabric was treated with helium plasma along with a monomer, hexamethyldisiloxane (HMDSO) which modified the surface morphology of the substrate thus, converting the hydrophilicity of cotton into hydrophobicity without hampering its useful inherent properties. Results of the study revealed that, stiffness and abrasion resistance of treated fabric decreases with increase in resiliency. On subsequent washes there was further increase in resiliency, decrease in abrasion resistance whereas stiffness remains same. Laundering also affect the hydrophobicity to some extent, but the plasma treated cotton fabric did not revert back to hydrophilicity.

Keywords: plasma, atmospheric, treatment, cotton, hexamethyldisiloxane

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. In textile manufacturing process, finishing is the final step or the last chance to provide the properties that customers will value. It is also called the beautification process of fabric. Finishing completes the fabric’s performance and gives it special functional properties including the final ‘touch’. However, this is the right time for textile industries to focus on environmentally safer methods of processing and finishing fabrics.

Among all natural fibres, cotton is the purest source of cellulose noted for its versatility, appearance, performance and natural comfort. Cellulose has highest molecular weight and structural order, hence is highly crystalline, oriented and fibrillar compared to other plant fibres thus, viewed as a premier fibre and biomass (Hsieh and Gordon, 2007) [3]. The hydroxyl groups render surface reactivity during wet treatments thus exhibit excellent wettability and wickability. However, high moisture content of this natural fibre possesses low resistance to staining and microorganism attacks. Thus, several finishing are aimed to control these undesirable properties.

It is evident that the conventional wet processing utilizes enormous amounts of water and the effluents of which cause water pollution that ultimately affects the ecology. This is the time when the industries have to look for ‘environmentally safe’ processes for textile processing. Plasma is a dry processing technique which controls the use of chemicals and energy. Modification of textile chemistry by plasma treatment represents great opportunity for improvement on conventional, energy demanding and less eco-friendly technologies. In plasma surface modification, the changes are principally attributed to the physical or chemical structure of the material due to high-energy bombardment of plasma or plasma enhanced reactions. The active species produced in plasma carry high energy that causes a sputtering, cleaning or etching effect, which modify the characteristics of fibre surface. Plasma processing can modify the surface of substrates without compromising the properties of bulk material. 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) [1]. Thus, the present study is conducted with the objective to assess:

1. Surface morphology characterization of plasma treated cotton fabric
2. Effect of laundering on the performance, durable and functional properties of plasma treated cotton fabric

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 gas and a monomer hexamethyldisiloxane (HMDSO) with a flow rate of 5 l/min and 1 ml/min respectively, at a power of 3.5 kW for 60 sec by keeping the electrodes at a distance of 1mm.

Surface morphology characterization: The test samples were analysed for surface morphology using S-3700N Scanning Electron Microscope (SEM) at 20 and 5 µm level with magnification power of 2,500X and 10,000X respectively.

Laundering: Laundering is another aspect of the study where the plasma treated cotton samples were hand washed using 2 gpl surfactant, rinsed well in cold water and finally shade dried. The washed sample was assessed to find out the impact of multiple washes on different quality parameters *viz.*, performance, durable and functional properties of treated substrates. The fabrics were subjected for a total of 10 washes and the quality parameters were assessed after every 5th wash.

Assessment of quality parameters – 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: The water repellency was assessed according to 'spray rating' directed in AATCC test method: 22-2005. However, the ability of a fabric to absorb water, by 'wicking' or 'capillary action', observed by timing, the rate at which water rises into the narrow strip of fabric suspended vertically with its lower end dipped into the water.

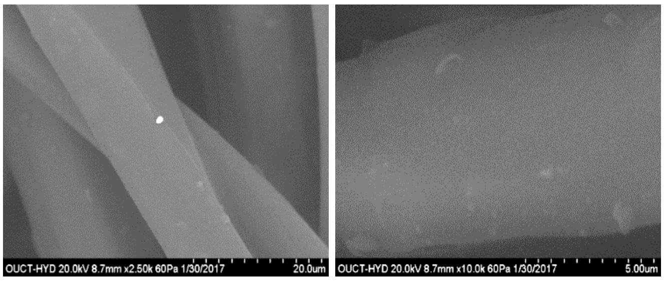
Statistical Analysis: One way ANOVA was used to find out the effect of plasma treatment on performance, durable and functional properties of the treated test samples.

Hypothesis: Laundering does not alter the performance, durable and functional properties of plasma treated cotton fabric.

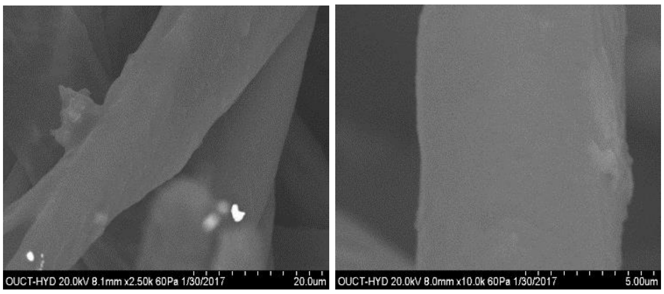
Results and Discussion

Characterization of surface morphology

Alteration in surface topography of cotton after plasma treatment to induce hydrophobicity is clearly evident from Fig 1(b) when compared with untreated sample presented in Fig 1(a) at both 20 and 5 µm levels, respectively. The smooth surface of cotton has changed to rough texture on plasma treatment and is due to physical abrasion; which is a predominant reaction of plasma that induced a distinct surface morphology to the substrate. The action of helium gas in surface etching proposed to be the prime factor contributing to the surface hydrophobicity (Tsoi *et al.*, 2011) [6]. Moreover, a thin layer of monomer hexamethyldisiloxane (HMDSO) covers the entire fibre and characterized as a globular structure somewhat similar to bumping structure of lotus leaf (Twardowski *et al*., 2012) [7].



(a)



(b)

Fig 1: Surface morphology of (a) untreated and (b) plasma treated cotton fabric

Performance properties of plasma treated cotton fabric

Effect of laundering on performance properties *i.e.* Cloth bending length and crease recovery angle of plasma treated cotton sample is presented in Table 1.

Bending length of the fabric is one of the important performance properties describes the pliability and hand feel characteristics. It is learnt from Table 1 that the warpway bending length was longer (02.36 cm) than its corresponding weftway length (01.95 cm), at control. However, a decrease in warpway (01.78 cm) and weftway bending lengths (01.18 cm) was observed after plasma treatment. Change in the morphology of the fabric may cause this reduction in both the directions of treated test sample due to etching, removal of the size and thickness from the fabric. These results are supported by the study conducted by Chinnammal and Arunkumar (2014) [2] who indicated that post dyed samples were soft and pliable compared to pre dyed samples. In other words the atmospheric pressure plasma treatment has reduced the stiffness of the cotton fabric. However, this reduction in bending length in both the directions is found to be significant at 1 per cent level of significance. Meanwhile, there was no much effect of washing on the decrease percentage in the bending length of treated fabric, which is evident from this table. Hence, null hypothesis set for the study that laundering does not alter the performance properties of plasma treated cotton fabric is accepted.

Crease recovery angle is one of the performance properties directly supports the bending length of that fabric and indicates the level of softness, pliability and resiliency of a fabric. The value of crease recovery angle of the test sample presented in Table 1 indicates that the warpway recovery angle (64°) is lower than its corresponding weftway recovery

(76°), at control. After plasma treatment, an increase in recovery angle of both warp (72°) and weft (80°) is noticed. Cloth crease recovery is the combined effect of recovery angles of both warp and weft yarns. It is evident that after plasma treatment, the recovery angle of cotton fabric is relaxed mainly due to etching of the surface and partial removal of size present on the cloth surface if any by 'cleaning of the surface'. Further, a slight increase in the crease recovery angle is observed both warpway (09.72 and 11.11 %) and weftway (16.25 and 17.50 %) after 5th and 10th washes, respectively. Therefore, the null hypothesis set for the study that laundering does not alter the performance properties of plasma treated cotton fabric is rejected.

Table 1: Effect of laundering on performance properties of plasma treated cotton fabric

Sl. No	Cotton samples	Performance properties			
		Cloth bending length (cm)		Cloth crease recovery angle (degree)	
		Warpway	Weftway	Warpway	Weftway
1	Untreated	02.36	01.95	64	76
2	Treated (hydrophobicity)	01.78**(24.57)	01.18**(39.48)	72**(12.50)	80**(05.26)
3	5th wash	01.78(00.00)	01.10**(06.78)	79**(09.72)	93**(16.25)
4	10th wash	01.78(00.00)	01.10**(06.78)	80**(11.11)	94**(17.50)

Figures in parenthesis indicate percentages **- Significant at 1 % level of significance

Durable property of plasma treated cotton fabric

The tensile strength and elongation percentage of plasma treated cotton samples is presented in Table 2 and found to be higher compared to control sample. This may be due to the frictional properties of fibres because of plasma sputtering action. These results are in line with the study conducted by Kan and Lam (2013) [4] who stated that the increase in cotton strength after plasma treatment is due to enhancement of inter yarn and inter fibre friction caused by etching effect of plasma. But on washing, both tensile strength and elongation declined due to removal of amorphous region during plasma treatment. Cotton is a fibre which gains strength when wet due to improvement in alignment of its long polymer in amorphous region, but impact of plasma on the amorphous region declined its property to gain strength on washing.

Cloth abrasion resistance is the wearing away of any part of material by rubbing against another surface. Table 2 displays the result of abrasion resistance of control and treated cotton fabric as well as after 5th and 10th wash. It is clear from this Table that, there was reduction in the abrasion resistance after plasma treatment (44.55 %). Further a trend of gradual decrease in the values is observed after 5th (10.71 %) and 10th (21.43 %) washes compared to treated samples, and the values are significant at 1 per cent level of significance. This reduction may be basically due to complete removal of impurities, protruding ends from fabric surface not only during treatment as well as while washings. Karahan *et al.* (2009) [5] revealed that hairiness of both wool and cotton fabric was significantly reduced by plasma treatments. This result is also supported by the study conducted by Bhat *et al.* (2011) [1] where it was clearly indicated that the protruding fibres become fragile due to etching and thus easily breakable. Hence, the null hypothesis set for the study that laundering does not alter the durable property of plasma treated fabrics is rejected as the treated cotton show significant reduction in abrasion resistance on subsequent washings.

Table 2: Effect of laundering on durable properties of plasma treated cotton fabric

S. No	Cotton samples	Durable properties				
		Cloth tensile strength (kgf)		Elongation (%)		Cloth abrasion resistance (cycles)
		Warpway	Weftway	Warpway	Weftway	
1	Untreated	22.62	09.40	07.85	05.86	101
2	Treated (hydrophobicity)	23.70**(04.77)	10.45**(11.17)	08.88**(13.12)	06.88**(17.41)	56**(44.55)
3	5th wash	23.53**(00.72)	10.15**(02.87)	08.17**(07.99)	06.69**(02.76)	50**(10.71)
4	10th wash	23.41**(01.22)	09.98**(04.49)	08.10**(08.78)	06.51**(05.38)	44**(21.43)

Figures in parenthesis indicate percentages **- Significant at 1 % level of significance

Hydrophobicity of plasma treated cotton fabric by spray test and wicking length

The impact of laundering on water repellency of plasma treated cotton fabric and control sample is presented in Table 3, which gives a clear picture about treated and untreated test samples. Cotton basically is hydrophilic in nature and absorbs moisture readily when come in contact with. And this is proved by the spray test rating scale which reads as 'zero' *i.e.* 'complete wetting of upper and lower surface'. On the contrary the plasma treated cotton was rated as 100 which expresses as 'No sticking or wetting of upper surface'; which clearly indicates that the treated sample has acquired a new dimension of 'water repellency'. However, the level of water repellency gradually reduced after multiple washes and is indicated as 70 after 5th wash *i.e.* 'partial wetting of whole of upper surface' and 70 to 50 after 10th wash *i.e.* somewhat 'partial to complete wetting of upper surface'. This indicates that, the level of water repellency gradually reduced after multiple washes; in other words there is shift from 'water repellency' to 'partial hydrophilicity' on subsequent washes. Therefore, it may be stated that though plasma finish followed by monomer treatment did turn the hydrophilicity in to water repellency and this change over is not permanent. Furthermore, the plasma treatment does not change the inherent inbuilt bulk characteristics of cotton but the change is restricted to morphology only. These results are supported by the study conducted by Tsoi *et al.* (2011) [6] that physical or morphological alteration is a crucial function that contributes to surface hydrophobicity of the substrate.

Vertical wicking test method is used to evaluate the ability of vertically aligned fabric specimen to transport liquid along and/or through them, and is applicable for woven fabrics. From Table 4, it is evident that both warpway and weftway wicking length was maximum at control compared to plasma treated samples after 5th and 10th washes. In a fibre this may progress due to wicking which is driven by capillary action or capillarity. Capillary forces drive the liquid in capillary spaces and thus develops greater affinity between the liquid and the surface; and thus liquid attempts to spread across the surface. The plasma treated sample exhibited a total hydrophobic nature by expressing wicking length as 'zero'. And these results are supported by the values of 'Spray Test Rating Scale' presented in Table 3, where the spray test rating of plasma treated cotton fabric was 100, meant 'No sticking or wetting of upper surface'. Hydrophobicity can be expressed in terms of contact angle, which is greater than 90°. Zhang *et al.* (2003) stated that a post treatment at a high temperature was conducive to enhance the hydrophobicity and the recovery of water repellency after multiple washes, is mainly due to some physical and chemical changes in the fabric after plasma treatment. Since, the low temperature plasma treatment was the method adopted in the present study there is possibility of reversible wettability of plasma treated cotton fabric after multiple washes. And this fact is proved by the wicking length of plasma treated cotton sample after 5th and 10th washes. Though there is slight wicking due to capillary forces, the force responsible to drive the liquid in capillary spaces, but the length is much shorter than the wicking length of control sample, which indicates that the treated sample have retained the property of water repellency, if not hydrophobicity. Hence, the null hypothesis set for the study that laundering does not alter the functional property of plasma treated fabric is rejected.

Table 3: Effect of laundering on water repellency of plasma treated cotton fabric

Sl. No	Cotton samples	Spray test (ratings)
1	Untreated	Zero
2	Treated (hydrophobicity)	100
3	5th wash	70
4	10th wash	70 to 50

Ratings as per AATCC

100: No sticking or wetting of upper surface (ISO 5)
90: Slight random sticking or wetting of upper surface (ISO 4)
80: Wetting of upper surface at spray points (ISO 3)
70: Partial wetting of whole of upper surface (ISO 2)
50: Complete wetting of whole of upper surface (ISO 1)
0: Complete wetting of whole of upper and lower surfaces (0)

Table 4: Effect of laundering on wicking length of plasma treated cotton fabric

Sl. No	Cotton samples	Wicking length (cm)					
		1 minute		2 minute		5 minute	
		Warpway	Weftway	Warpway	Weftway	Warpway	Weftway
1	Untreated	03.00	02.30	04.10	02.70	05.60	04.00
2	Treated hydrophobicity)	00.00**	00.00**	00.00**	00.00**	00.00**	00.00**
3	5th wash	00.90**	01.30**	01.90**	02.00**	03.10**	03.20**
4	10th wash	01.00**	01.30**	02.00**	02.10**	03.20**	03.30**

**- Significant at 1 % level of significance

Conclusion

Helium plasma treatment along with monomer hexamethyldisiloxane (HMDSO) significantly altered the surface morphology of cotton fabric from smoother to globular structure. Cloth stiffness of the test samples reduced after treatment but on subsequent washes, the change was negligible. Whereas, cloth crease recovery angle of the test sample enhanced not only after treatment but also on multiple washes. Tensile strength and elongation percentage of cotton fabric enhanced significantly after plasma treatment, but did decline on multiple washes due to removal of amorphous region caused by sputtering action of plasma. Removal of upper thin layer of fabric surface due to sputtering action of plasma reduced the resistance for abrasion of treated substrate. A trend of reduction in abrasion was further observed after multiple washes also. Moreover, results of spray test and wicking test displayed the induced hydrophobicity is not permanent, at the same time did not revert back to hydrophilicity on multiple washes; but did retain the water repellency property. Thus plasma treatment is a sustainable finish to remarkable extent.

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