

Development of multifunctional cotton using fluorocarbon resin

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Abstract

An attempt has been made to develop multifunctional cotton fabric, possessing water repellent, stain repellent, shrink resistance and quick dry properties using fluorocarbon resin. The hydrophobicity of cotton fabric was determined by carrying out water repellency test, taking scanning electron microscopic photographs and measuring water contact angle. The durability of hydrophobicity of cotton was tested till 20 washes and found satisfactory. Oil repellency was determined employing hydrocarbons resistance test. The air permeability of cotton fabric was also determined keeping in view the impact on breathability of treated cotton and was found quite good. The untreated and treated cotton fabric was subjected to repeated domestic laundry condition, and shrinkage was measured, which indicated excellent shrink resistance behaviour because of its water repelling characteristic. This hydrophobicity of cotton also added to its quick dry behaviour even at low temperature and high relative humidity. The physical properties of treated dyed cotton fabric samples were compared with untreated, and no significant changes were observed in colour fastness to washing, rubbing, perspiration and light. The tensile and tear strength showed good retention even at higher concentration of fluorocarbon resin. This work is of great industrial importance for textile products used in home textiles. The textile industry can fetch more export earnings by doing multiple value addition using the same chemical. The work reported in the literature is about using fluorocarbon and developing water- and oil-repellent fabrics. In the present work, apart from water and oil repellency, shrink resistance and quick dry behaviour of cotton textile has also been established using same fluorocarbon because of hydrophobicity imparted to cotton.

Keywords

Hydrophobic, water repellency, oil repellency, shrink resistance, quick dry cotton

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Introduction

Various methods have been reported in the literature to get water-repellent cotton.^{1–4} Among these, the most common method is to use fluorocarbon, which impart both water and oil repellency (OR) to cotton because of its ease of application. Apart from this, fluorine has got a unique characteristic of lowering down the surface energy and making cotton fabric both water and oil repellent.^{5,6}

Any liquid drop on the fibre surface will have two types of interaction, namely the internal cohesive interaction within the liquid and the adhesive interaction between the liquid and the fibre surface. The liquid will spread only when the interaction with fibre surface is more than the cohesive interaction within the liquid. The internal cohesive interaction or surface tension of water is 73 mN/m.

Any finish that will reduce the surface energy of a cotton surface below the surface tension of water will make it water repellent. Similarly, the surface tension of oil is in the range of 10–20 mN/m. To make cotton oil repellent, its surface energy must be lower than that of oil. The major advantage of fluorocarbon is that both water and OR can be attained, because of the lower surface energy of cotton than

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the surface tension of oil. A fluorocarbon polymer sheath formed around the cotton fibres reduces the surface energy of cotton. This is accompanied by the increase in the contact angle of liquids on cotton surface.⁷ Fluorocarbon surfactants based on perfluoro alkyl hydroxyl alkyl siloxane compounds are capable of withstanding numerous washing and dry cleaning cycles.⁸

The water contact angle (WCA) at the liquid/solid interface on any solid surface determines its hydrophilic and hydrophobic characteristics. If WCA at the liquid/solid interface becomes lower than 90°, the surface becomes hydrophilic (water loving). On the other hand, if WCA becomes higher than 90°, the solid surface becomes hydrophobic (water hating). However, any solid surface will attain super hydrophobic characteristics, if WCA is greater than 150°.⁹

There are many examples of super-hydrophobic surfaces in nature. The wings of butterflies,^{10,11} the feet of water striders¹² and the leaves of plants^{10,13} are few examples. The development of techniques such as scanning electron microscopy (SEM) have made it possible to study the surface morphology at ultra-micro-scale and find out the possible explanation for super-hydrophobicity. The conclusion drawn is that both low surface energy and nano-roughness contribute directly to attain super-hydrophobic nature. Any solid surface will exhibit wettability and repellency behaviour depending upon the surface energy and roughness of material.^{11,14–18}

Cotton absorbs moisture in the amorphous region and water molecules then act as lubricant and helps in the movement of internal polymer chains. The existing hydrogen bonds are disrupted, and new hydrogen bonds are formed in the swollen state of cellulose in new configuration and are locked after drying. Therefore, the wrinkled appearance of the cellulose fabric persists even after drying, in contrast to non-swelling synthetic fibres¹⁹ and also the fabric shrinks.²⁰

To produce non-swelling, durable press or shrink-resistant cellulose fabrics, the following two different chemical approaches have been used. The first approach is to block the pores of the fibres by incorporating polymeric finish. It inhibits penetration of water. The second approach is using multifunctional cross-linking agents. The multifunctional cross-linking agents reacts with the hydroxyl groups of the nearby cellulose molecules, thereby hindering the swelling of the cellulose fibre.^{21,22}

The work reported in the literature is about using fluorocarbon and developing water and oil-repellent fabrics. In the present work, apart from water and OR, shrink resistance and quick dry behaviour of cotton textile has also been established objectively using same fluorocarbon because of hydrophobicity imparted to cotton.

The cellulose has been made hydrophobic using fluorocarbon polymer. The hydrophobic cellulose repels water and inhibits its penetration even in the amorphous region. This will result in the development of shrink-resistant

cotton textiles. Since the water-repellent property has been imparted to cotton and penetration of water is inhibited, it enables cotton textiles to dry quickly even at low temperature and at high relative humidity (RH).

The micro-roughness on the surface of cotton has been studied taking pictures from SEM. The hydrophobicity, water repelling and oil repelling characteristics of cotton have been established by determining WCA and carrying out water repellency (WR) and OR tests, respectively. The anti-shrinkage behaviour and quick dry characteristics have also been established by carrying out repeated washings and measuring shrinkage and drying time in comparison to the untreated fabric.

Materials and methods

Materials

The plain weave fabric used for experimentation was of two types: 100% bleached cotton and 100% dyed cotton with reactive dyes, having weight 140 g/m², without any finishing agent.

The water-repellent chemical used was fluorocarbon resin emulsion with C8 chemistry, supplied under the trade name of Bioguard 581X by Biotex Ltd, Malaysia, along with Biocat M as universal catalyst.

Methods

Application of water-repellent chemical

The 100% bleached cotton fabric was padded with solutions of 10, 20, 30, 40, 50 and 60 g/l, Bioguard 581X along with catalyst Biocat M, with concentrations varying from 2, 4, 6, 8, 10 and 12 g/l, respectively, at pH 5 to 6 using acetic acid. The following padding conditions were used to get 80% pickup:

Mangle pressure: 4.5 bar

Speed: 2.50 m/min

After padding, the treated fabric samples were dried in Shirley Development Limited (SDL) mini dryer (UK) at 150°C for 2 min, followed by curing at 185°C for 1 min.

The 100% dyed cotton fabric sample was treated with 60 g/l, Bioguard 581X along with 12 g/l Biocat M catalyst at pH 5 to 6 maintained using 0.5 g/l acetic acid. The padding conditions such as mangle pressure, speed and %wet pickup was same as above.

Determination of wet pickup

The wet pickup of 100% cotton fabric was determined at different pressure readings (2, 3, 4 and 5 bar), at constant mangle speed of 2.50 m/min, using two-bowl laboratory vertical padding mangle, manufactured by Mathis, Switzerland.

The wet pickup was found by calculating the weight difference of wet and dry fabric and expressed in percentage on the basis of weight of dry fabric. The padding mangle pressure of 4.5 bar was used to get 80% pickup in the application of water-repellent chemical.

Determination of WR

The WR of treated bleached cotton fabric samples were determined employing SDL water spray tester (UK) using AATCC test method 22-2001.

Determination of OR

The OR of treated bleached cotton fabric samples were determined using hydrocarbon resistance test, AATCC test method 118-2002.

Determination of the durability of WR effect

The durability of WR effect of bleached cotton fabric samples treated with various concentrations of fluorocarbon were determined by subjecting to 10 and 20 washing cycles employing DIN EN ISO 6330 standard for domestic laundry using launder-meter supplied by Mesdan Lab, Italy.

After completing 10 and 20 washing cycles, the water-repellent efficacy test was done employing SDL water spray tester using AATCC test method 22-2001.

SEM photographs

The SEM photographs were taken using SEM supplied by Carl Zeiss, model-EVO 18 and at a voltage of 20 kV. The photographs were taken at 1000 \times and 10,000 \times magnification of untreated and treated cotton samples.

Determination of WCA

The WCA of water-repellent cotton fabric samples were determined by spreading cotton samples on a flat table. One fine drop of water was placed on fabric samples using medical syringe of 0.5 ml capacity. The pictures were taken using Kodak 15 \times zoom camera (USA). Horizontal and tangential lines were drawn on pictures and WCAs were measured using protractor.

Determination of the shrinkage of untreated and treated cotton samples

The shrinkage of untreated and treated bleached cotton samples were determined employing DIN EN ISO 6330 standard for domestic laundry using the launder-meter.

Determination of quick drying behaviour of hydrophobic cotton

The drying behaviour of untreated and treated cotton samples were studied by washing the samples according to DIN EN ISO 6330 method, followed by uniform hydro-extraction. The excess water was extracted by padding the samples at 3.5 bar at a speed of 2.5 m/min, followed by drying at 65% RH and at 30 $^{\circ}$ C using Mesdan Spa, Type M250-RH conditioning chamber (Italy).

Determination of air permeability

The air permeability of various untreated and treated samples were determined using FX 3300 type calibrated air permeability tester supplied by TexTest AG, Switzerland, employing ASTM D737-96 method.

Determination of colour fastness to washing

The colour fastness to washing of both untreated and treated dyed cotton fabric samples were tested using ISO-2 test in launder-meter supplied by Mesdan Lab.

Determination of colour fastness to crocking

The colour fastness to crocking of both untreated and treated cotton fabric dyed samples were tested employing AATCC test method-8-2004 using crock meter supplied by Mesdan Lab.

Determination of colour fastness to perspiration

The colour fastness to perspiration of both untreated and treated cotton fabric dyed samples were tested employing ISO 105-EO4 1994 (acid and alkaline perspiration) using perspirometer supplied by Mesdan Lab, Electrical Heat Therostatic Culture Box, Model DH-4000B.

Determination of colour fastness to light

The natural sunlight source was used for determining the colour fastness towards light of both untreated and treated cotton fabric dyed samples.

The light fastness rating system was based on the rate of fading of eight blue-dyed wool standard samples which were rated from 1 (*poor*) to 8 (*excellent*). All the eight blue wool standard samples were exposed to natural sunlight source along with untreated and treated cotton fabric. The light fastness rating was given comparing the fading of blue wool standard number and tested samples placed parallel to blue wool standard.

Determination of tear strength

The tear strength of both untreated and treated cotton dyed fabric samples were determined employing ASTM D 1424

Table 1. WR of cotton fabric before and after domestic laundry.

Concentration of fluorocarbon (g/l)	Water spray rating		
	Before wash	After 10 washes	After 20 washes
10	100	90	80
20	100	90	80
30	100	100	90
40	100	100	90
50	100	100	90
60	100	100	100

WR: water repellency.

Table 2. OR of cotton fabric before and after domestic laundry.

Concentration of fluorocarbon (g/l)	OR grade		
	Before wash	After 10 washes	After 20 washes
10	5	4	3
20	5	5	4
30	5	5	5
40	5	5	5
50	5	5	5
60	5	5	5

OR: oil repellency.

method, using SDL Atlas M008E Digital Elmendorf Tester.

Determination of tensile strength

The tensile strength of both untreated and treated cotton dyed fabric samples were determined employing ISO-13934/1-EN 13534/1, using fabric traction strip method with Tenso Lab Strength Tester, Mesdan Lab.

Results and discussion

Effect of fluorocarbon concentration on WR of cotton

In order to make cotton hydrophobic, the bleached cotton samples were treated with various concentration of fluorocarbon emulsion along with the catalyst. The WR of treated fabric samples were tested before and after 10 and 20 washes, respectively, using SDL water spray tester. The results are given in Table 1.

The spray test rating of 100 before wash indicates that there was no sticking or wetting of upper surface by water even at lowest concentration of 10 g/l fluorocarbon. However, after 10 and 20 washes, a rating of 90 and 80, respectively, was obtained showing that there was slight random wetting of upper surface with water at spray point only. This clearly indicates that hydrophobicity of cotton fabric was maintained even after 20 washes at almost all concentrations of fluorocarbon.

Effect of fluorocarbon concentration on OR of cotton

The OR of treated fabric samples were tested before and after 10 and 20 washes employing hydrocarbon resistance test. The results are given in Table 2.

The OR grade of five before wash indicates a very good oil-repellent property even at a concentration of 10 to 20 g/l fluorocarbon. The same performance was maintained even after 10 washes at 20 g/l and above. After 20 washes, a grade of 4 at 20 g/l fluorocarbon indicates slight deterioration of performance. However, 30–60 g/l fluorocarbon-treated cotton showed a very good rating of five. This clearly indicates that the durability of OR of hydrophobic cotton fabric was maintained even after 20 washes at above 20 g/l fluorocarbon concentration.

SEM photographs of untreated and treated cotton fabric

SEM photographs of untreated and treated cotton fabrics were taken at 100 \times and 10,000 \times magnification as shown in Figure 1. The pictures clearly show that micro-roughness was developed on fluorocarbon-treated cotton in comparison to the untreated cotton fabric with smooth surface. This micro-roughness on treated cotton fabric is making cotton hydrophobic.

WCA of fluorocarbon-treated cotton before and after 20 washes

The WCA of treated cotton at various concentrations of fluorocarbon (20, 40 and 60 g/l) were measured before and after 20 washes.

It can be seen from Figure 2 that WCA of treated cotton fabrics were above 90 $^\circ$ both before and after 20 washes, indicating that cotton fabric has become hydrophobic. Another observation is that WCA are almost constant between 123 $^\circ$ and 127 $^\circ$ before washes. It shows that increase in concentration of fluorocarbon from 20 g/l to 60 g/l have minimal impact on hydrophobicity of cotton. After 20 washes, although there was reduction in WCA but in all cases, it was above 90 $^\circ$ indicating that hydrophobicity of cotton was maintained.

Shrinkage behaviour of hydrophobic cotton

It can be seen from Figure 3 that the shrinkage in warp direction increased with the increase in number of washes. The shrinkage became almost constant after seven washes. The untreated cotton showed shrinkage of 9.5% after eight washes. However, the fluorocarbon-treated cotton showed shrinkage varying from lowest 3.5% (at 60 g/l) to maximum 5% shrinkage (at 10 g/l) treatment. The fluorocarbon-treated cotton samples from 20 g/l to 50 g/l showed shrinkage varying from 4.0% to 4.5%. About 58% to 53% reduction in shrinkage has been observed in treated cotton samples at

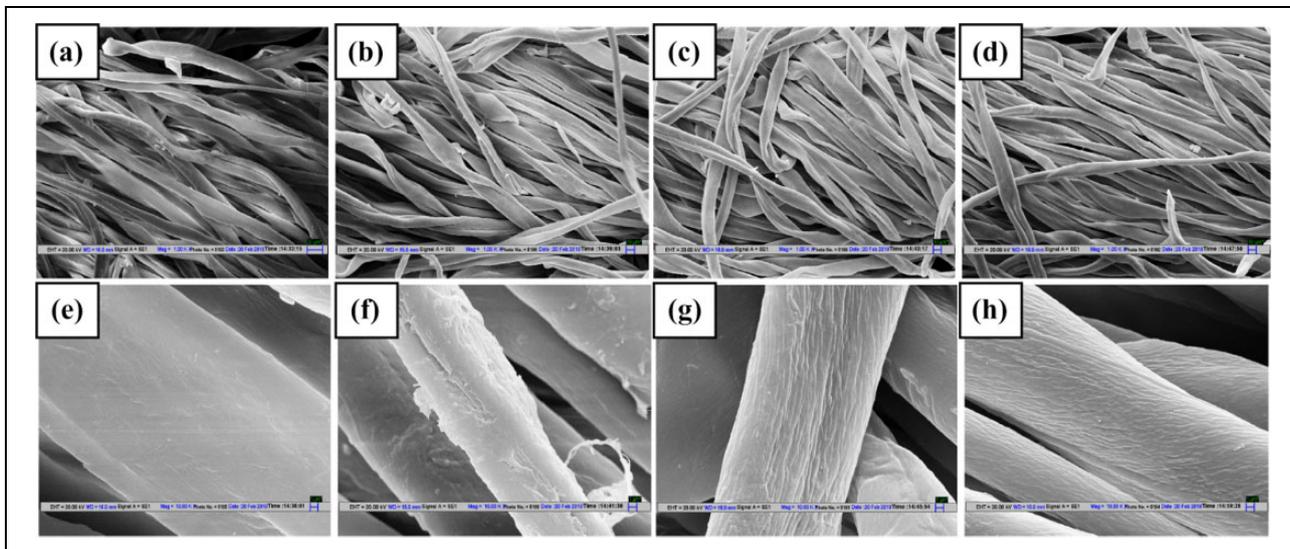


Figure 1. SEM photographs of untreated and treated cotton with fluorocarbon at $\times 100$ and $\times 10,000$ magnification: (a) untreated cotton at $\times 100$; (b) 20 g/l treated cotton at $\times 100$; (c) 40 g/l treated cotton at $\times 100$; (d) 60 g/l treated cotton at $\times 100$; (e) untreated cotton at $\times 10,000$; (f) 20 g/l treated cotton at $\times 10,000$; (g) 40 g/l treated cotton at $\times 10,000$; (h) 60 g/l treated cotton at $\times 10,000$ with fluorocarbon resin. SEM: scanning electron microscope.

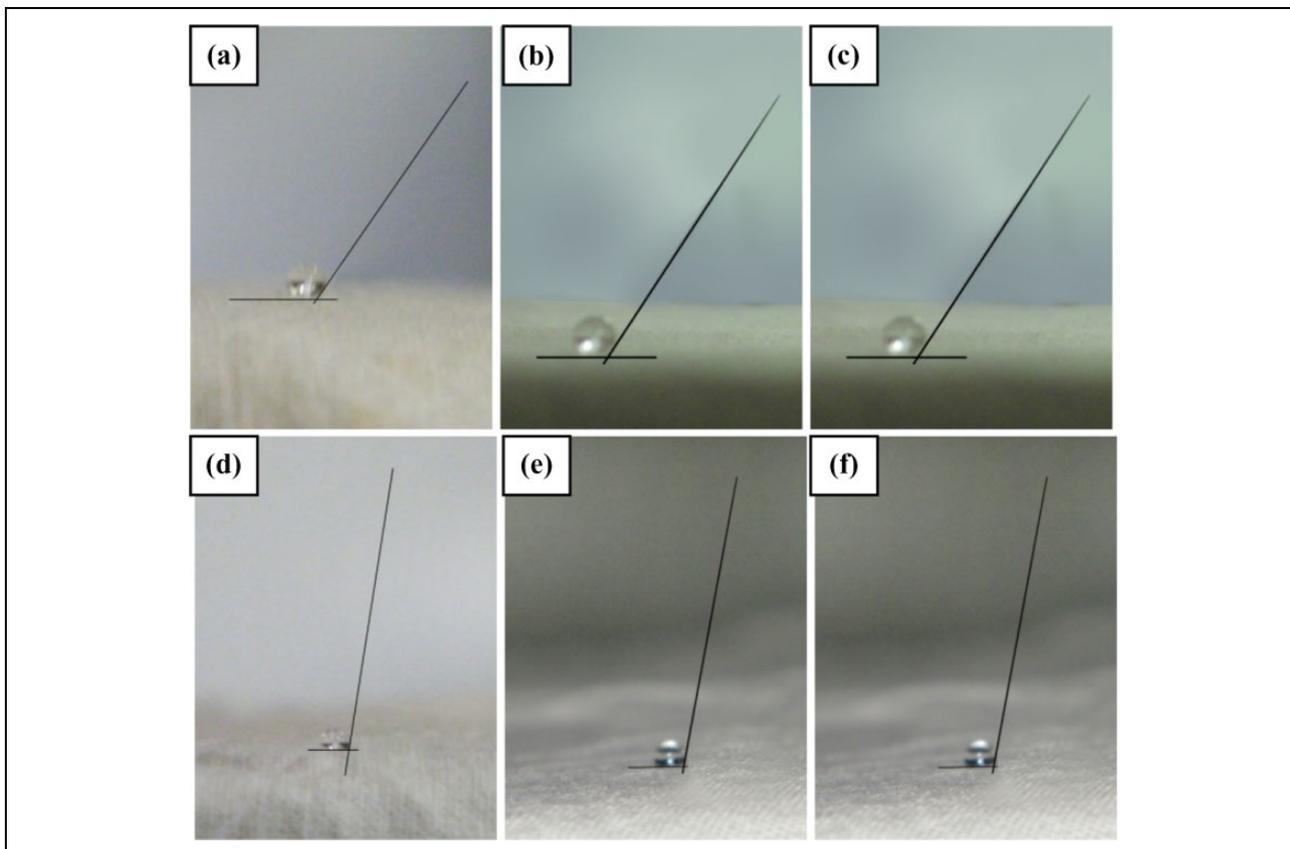


Figure 2. WCA of treated cotton with fluorocarbon before and after 20 washes. (a) 20 g/l unwashed WCA (123°); (b) 40 g/l unwashed WCA (123°); (c) 60 g/l unwashed WCA (127°); (d) 20 g/l 20 washes WCA (98°); (e) 40 g/l 20 washes WCA (101°); (f) 60 g/l 20 washes WCA (101°). WCA: water contact angle.

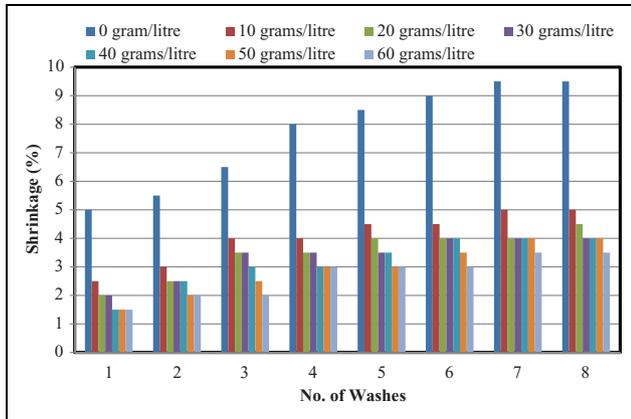


Figure 3. Warp-wise shrinkage of untreated and treated cotton with fluorocarbon.

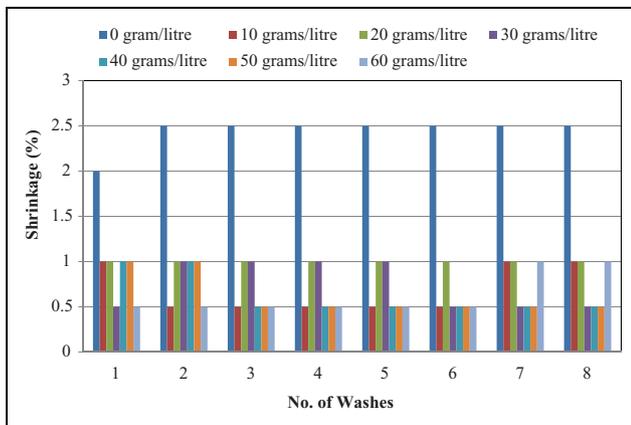


Figure 4. Weft-wise shrinkage of untreated and treated cotton with fluorocarbon.

varying concentration of fluorocarbon (20–50 g/l) because of hydrophobicity imparted to cotton as shown in WR rating and WCA before and after washes. This hydrophobicity prevents the penetration of water in cotton, thereby imbibing the swelling of cotton and making it shrink resistant.

The weft direction shrinkage is shown in Figure 4. The weft direction shrinkage became almost constant after two washes. The untreated cotton showed maximum shrinkage of 2.5% after two washes. However, the fluorocarbon treated cotton showed shrinkage varying from 0.5% to 1.0% at various concentrations of fluorocarbons. About 60–80% reduction in shrinkage has been observed in treated cotton fabric samples at varying concentration of fluorocarbons in weft direction because of cotton becoming hydrophobic.

Quick dry behaviour of hydrophobic cotton

In Figure 5, drying behaviour of treated cotton samples have been studied against untreated at 65% RH and 30°C. The initial moisture of untreated cotton sample (0 g/l) after washing and hydro-extraction through padding mangle was 90%. In case of treated cotton samples after

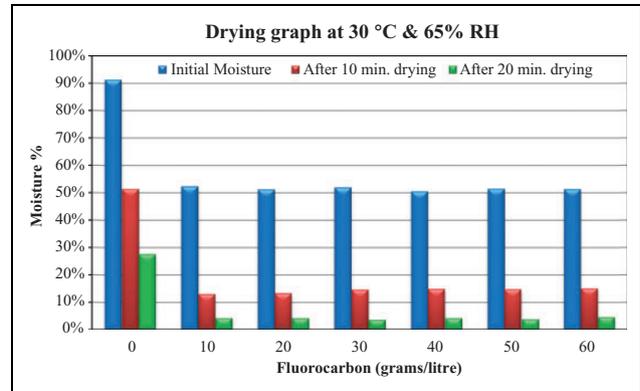


Figure 5. Drying graph of hydrophobic cotton.

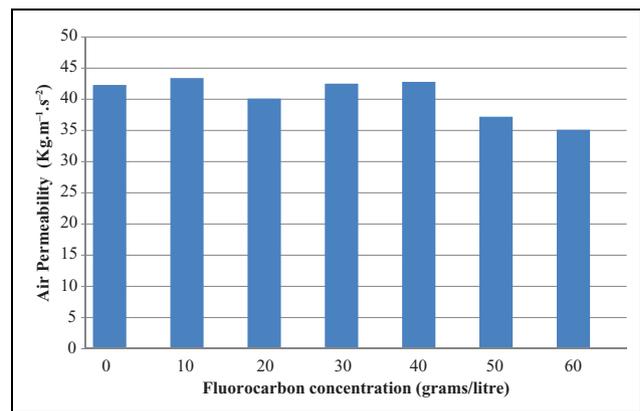


Figure 6. Air permeability of untreated and treated cotton with fluorocarbon at varying concentrations.

hydro-extraction, the initial moisture was around 50% irrespective of the concentration of fluorocarbon used because of hydrophobicity imparted to cotton with fluorocarbon.

After 10 min drying at 65% RH and 30°C, the untreated cotton showed moisture content around 50%, and all treated samples were almost dried to a moisture content between 13% and 15%. The reason for quick drying even after 10 min was because the initial water picked up by treated cotton was lower due to their hydrophobic nature. It shows reduction in drying time of treated cotton samples by more than 50%.

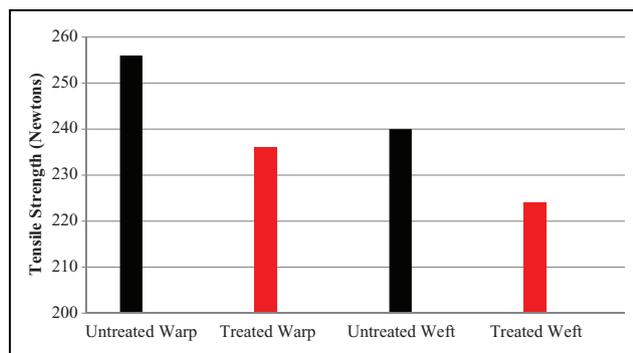
Air permeability of untreated and treated cotton with fluorocarbons

The air permeability of untreated and treated cotton with varying concentration of fluorocarbon was determined. The results are shown in Figure 6.

It can be seen in Figure 6 that untreated cotton (shown as 0 g/l fluorocarbon concentration) showed air permeability of 42 kg/m². The air permeability values of treated cotton with 10–40 g/l fluorocarbon varied between 40 kg/m² and 44 kg/m². It indicates that air permeability/breathability of treated cotton fabric was closer to untreated cotton up to

Table 3. Comparison of fabric properties between untreated and treated dyed cotton.

S. No.	Parameters	Untreated dyed fabric	Treated dyed fabric
1	Colour fastness to washing		
	Change in colour	4-5	4-5
2	Staining on adjacent Fabric	4-5	4-5
	Colour fastness to rubbing		
3	Staining on adjacent fabric		
	Wet rubbing	4-5	4-5
4	Dry rubbing	4-5	4-5
	Colour fastness to perspiration		
5	Acidic		
	Change in colour	4-5	4
6	Staining on adjacent fabric	4-5	4-5
	Alkaline		
7	Change in colour	4-5	4-5
	Staining on adjacent fabric	4-5	4-5
8	Colour fastness to light	>3	>3
	Change in colour		
9	Tensile strength (N)		
	Warp	256	236
10	Weft	240	224
	Tear strength (N)		
11	Warp	37.85	35.10
	Weft	37.52	34.34

**Figure 7.** Tensile strength of untreated and treated cotton with 60 g/l fluorocarbon.

40 g/l. However, at 50–60 g/l of fluorocarbon, little deterioration was observed. This further shows that no continuous film of fluorocarbon polymer was formed and porosity of fabric was sustained below 50 g/l fluorocarbon.

Comparison of colour fastness and physical properties of untreated and treated dyed cotton fabric

In order to compare various colour fastness and physical properties of untreated and treated cotton fabric, the dyed cotton fabric was treated with the highest concentration of fluorocarbon (60 g/l). The reason for the selection was that if the tested physical properties are comparable at this concentration of fluorocarbon, the properties will remain comparable at lower concentration of fluorocarbon also.

The results presented in Table 3 are self-explanatory and shows no significant change in properties, except little deterioration in tensile strength (below 8%) as shown in Figure 7.

Although the decrease in tensile strength is minimal, the probable reason for decrease may be due to the reaction of fluorocarbon resin with cellulose, which decreases little elasticity and flexibility of cellulose fibres. The durability of finish towards domestic laundry also confirms the reaction of fluorocarbon resin with cellulose.

Conclusions

It has become possible to produce multifunctional cotton textiles by making it hydrophobic using fluorocarbon polymer. The desired WR, OR, shrinkage resistance and quick dry properties can be achieved even at a lower concentration of 20 g/l of fluorocarbon. SEM photographs and WCA clearly established the generation of micro-surface roughness and hydrophobicity of cotton. The finishes imparted are durable and can withstand 20 domestic laundry cycles.

The air permeability test indicated that breathability of untreated and treated cotton fabric sustained, indicating no continuous film formation.

The fabric properties of untreated and treated dyed cotton fabrics at 60 g/l fluorocarbon were found similar in terms of various colour fastness and tear and tensile strength, indicating no deterioration of fabric performance even at higher concentration of fluorocarbons.

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