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Comparative study of repellent finishes based on C6 and C8 chemistry on cotton fabric

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The present study aims at exploring the various aspects that contribute to an effective and reliable automobile seat cover by using heavy GSM textile fabric, rather than rexine/leather which gives water repellency, anticrease and softness. An attempt has been made to analyse the effects of repellents based on C6 and C8 chemistry on cotton fabric. Along with this, anticrease agent, silicone softener and catalyst are used to bring in multi-functionality to the material. The conventional perfluorocarbon used for fluid-repellent finish is based on C8 chemistry and is an established process but the chemical is highly persistent in environment while the C6 are far less bio-accumulative. To compare the effectiveness between the two, exhaustive trials are taken with varying concentration, curing time and temperature. A modified dimethyloldihydroxy ethylene urea based cross-linker is co applied along with softener in the same bath as that of the repellent finish by pad-dry-cure technique. Box-Behnken response surface design of experiment has been used to study the individual and interactive effects of process parameters in terms of water vapour permeability and crease recovery. The impact of finish on the whiteness is also measured. The strength loss measurement, scanning electron micrographs and energy-dispersive X-ray (EDX) have been carried out for selected samples. Fluorine retention on fabric is established by the EDX tests. Wash durability for C6 ones is found to be comparable to that of C8, thus showing the former a sustainable alternative.

Keywords: Bio-accumulative, Cotton fabric, Crease recovery, Fluorochemical, Fluid repellency, Multifunctional finishing

1 Introduction

Functional finishes on textiles are gaining importance in their applications. Multi functionality of textile products is the key to success in the present day market. At the same time, the material should be sustainable from process and environmental points of view. Health hazards of many chemicals have been rediscovered over a period of time and more so in recent years. In this context, it is observed that more than four million substances are registered with the CAS (Chemical Abstract Service), out of which impact of 80% of the chemicals on human health is not yet known. Basic compounds like benzene are not allowed to be used in excess of 5ppm in free state due to toxicity[1]. Hence, it becomes imperative to strike a balance between functionality offered by a product and its sustainability.

Fluorocarbons, also known as fluorochemicals, have been very effective in imparting fluid repellent finishes on textile fibres. Fluorocarbons essentially refer to per- and poly-fluoroalkyl substances (PFASs), which are fluorinated compounds containing at least one fully fluorinated methyl or methylene carbon atom. Their unique properties are due to the amphiphilic structure and strong carbon-fluorine bonds. The high level of hydrophobicity and oleophobicity offered by PFASs is because of the low surface energy provided by the orientation and packing of the terminal –CF_3 end groups within the side-chains[2-4]. However, long-chain perfluorocarbons (PFCs) are non-biodegradable and bio accumulative in the environment[5,6]. Conventionally, fluid repellent finishes based on C8 chemistry have been used. Although very effective, they are now found to be highly persistent and are virtually indestructible when released to environment[7]. They even move through soil to drinking water. Although fluorinated products have a profound greenhouse effect with a high Global Warming Potential (GWP), C6 based fluorochemical is a better alternative to C8. The C6 chemistry based product when used, produces a PHFA (perfluorohexanoic acid), which is supposed to be 40 times less bio-acccumulative than PFOA (perfluorooctanoic acid). Although C6 is close to C8 chemically, but doesn't contain PFOA and hence compounds based on the former, break down in the environment[1,8-10]. However, the C6 ones are likely to be less effective than that of C8 and hence both are still in industrial use. But the C8 chemistry used for repellent finishes is to be phased out sooner than later and hence prompts the present comparative study undertaken.

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Besides this, achieving multi-functionality in single finish treatment makes the process economically sustainable and some end users have a requirement for such properties. Upholstery, particularly for automobiles including two wheelers requires stain and water repellent in addition to good wrinkle recovery. Textile fabrics with such properties would make a good substitute to leather or rexine often used for the said purposes. For crease recovery, DMDHEU (Dimethyloldihydroxy ethylene urea) and modified DMDHEU are the preferred chemicals with high efficiency and relatively environmentally benign-properties[11,12]. In the present work, one such ultra low formaldehyde anticrease finish along with fluorochemical and softener are applied on a heavy gsm cotton fabric to get the desired effect.

2 Materials and Methods

2.1 Materials

Ready for dyeing 100% cotton fabric with 3/1 twill weave, 80 EPI, 48 PPI and 280 g/m^2 weight was used for the study. Oil and water repellents of C6 and C8 types, DMDHEU based anticreasing agent (Exsolink ECO Spl) and silicone softener all provided by Intexso Biochem, Mumbai and lab grade $MgCl_2.6H_2O$ were used for the finish application.

2.2 Methods

The bleached fabric was first thoroughly washed with hot water in a lab winch and then dried. Then preliminary trials with the chemicals were taken to find out the effective range of chemical concentration and process conditions on the heavy gsm fabric. The procedure of pad –dry –cure was followed. All padding operations were carried out in the lab padding mangle at 80% expression followed by drying in the circulatory lab oven at 105°C and then curing for 3 min. Then three factors were chosen, namely liquid repellent concentration (A), cross-linker concentration (B) and curing temperature (C) separately for the C6 and C8 based fluid repellents. The low, medium and high levels of the factors were selected (Table 1). To study the individual and interactive effects of these variables, Box Behnken response surface design was used. The three variables taken were expected to potentially influence the multifunctional effect and hence selected for the study. In each finish formulation, 30 g/L Exosil Magna, a high performing silicone softener and $MgCl_2.6H_2O$ at one-fifth concentration of cross-linker were used. Crease recovery angle (CRA) and water vapour transmission rate (WVTR) were taken as the responses. Shirley crease recovery tester was used to find the CRA (warp+weft) while Systester WVTR –C3/C6 was used to check the water vapour transmission rate as per ASTM E98 standard. The sample is cut with the specimen cutter and then placed in a cup filled with 30 mL distilled water, sealed with a sealing ring and secured with the lid and screw. The tests are performed in cycle mode, with three 1800 second cycles for recording the data. The loss of mass of water from the cup with time is measured by the instrument continuously and rate of loss of water per unit surface area is used for calculation of water vapour transmission rate (WVTR) of the fabric samples[13].

Besides these, whiteness index was measured to check any dulling caused by the finish treatments. The measurements were done using a Premier colorscan 5100 model computer colour matching system with specular inclusion and the whiteness was measured in CIE system. Any change in tensile strength was monitored by doing the measurement on a universal tensile tester instrument working in CRE principle. ASTM D5034 was followed using ravelled strip method for the test. The same instrument was used for the measurement of tearing strength by the single rip tear strength approach following ASTM D2261 standard. In each case, 5 samples were tested for both warp and weft directions and average strength was calculated. The optimised samples were tested for EDX and SEM to detect elemental composition and chemical deposition on substrate respectively.

3 Results and Discussion

According to Box-Behnken design, 17 experimental runs are performed to prepare 17 varieties of both C8

Table 1— Process parameters and their levels

Factors	Levels					
	Low (-1)		Medium (0)		High (+1)	
	C6	C8	C6	C8	C6	C8
Repellent concentration (A), g/L	90	60	120	75	150	90
Anti-crease agent concentration (B), g/L	80	80	100	100	120	120
Curing temperature (C), °C	130	130	150	150	170	170

and C6 based samples. All these coated samples are measured for crease recovery angle (CRA) and water vapour transmission rate (WVTR). The results are given in Tables 2 and 3.

3.1 Analysis of Crease Recovery Angle of C8 Samples

The influence of process parameters, such as water repellent concentration (A), cross-linker concentration (B), and curing temperature (C) on crease recovery of C8 based coated textiles has been investigated by employing Box-Behnken Design and response surface methodology. All main effects and two factor interaction effects as obtained by ANOVA are shown in Table 4.

The Model F-value of 10.77 implies that the model is significant. There is only a 0.07% chance that a "Model F-Value" this large could occur due to noise. Values of "Prob> F" less than 0.05 indicate that the model terms are significant at 95% confidence interval. In this case, B and C are significant model terms. The model equation, which has coefficient of determination (R^2) 0.8660, that denotes goodness of fit and can well predict crease recovery angle (CRA) for any given unknown parameters, is given below:

$$\text{CRA} = 212.24 + 1.88A + 8B + 10.13C + 3.75\text{AB} + 0.50AC + 1.75BC \quad \ldots (1)$$

The trends obtained (Fig.1) show the influence of the process parameters on crease recovery. With increase in anti-crease agent concentration, there is rise in CRA. The change in repellent concentration doesn't appear to influence wrinkle recovery at lower concentration of anti-crease agent, while it has a positive effect at higher concentrations of the cross-linker, as evident from Fig 1(a). However, curing temperature and resin concentrations primarily affect CRA as both at higher

Table 2 — Experimental runs as per 3^3 Box-Behnken design and value of responses involving C8 based repellent

Exp. runs	Factor levels			CRA	WVTR
	A, g/L	B, g/L	C, ºC	deg	g.m^{-2}day^{-1}
Control	-	-	-	155	4896.88
1	75	80	170	214	4013.63
2	90	100	170	226	3969.30
3	90	120	150	232	4254.83
4	60	80	150	205	4318.61
5	90	80	150	205	3566.05
6	75	100	150	206	4278.45
7	75	80	130	200	4410.99
8	75	100	150	211	4278.45
9	75	100	150	207	4298.25
10	75	100	150	206	4271.42
11	60	120	150	217	4458.39
12	60	100	130	203	3981.96
13	75	100	150	210	4283.92
14	90	100	130	202	4166.16
15	60	100	170	225	4372.43
16	75	120	170	230	4235.52
17	75	120	130	209	3959.21

Table 3 — Experimental runs as per 3^3 Box-Behnken design and value of responses involving C6 based repellent

Exp. runs	Factor levels			CRA	WVTR
	A, g/L	B, g/L	C, ºC	deg	g.m^{-2}day^{-1}
Control	-	-	-	155	4896.88
1	120	80	170	212	4128.76
2	150	100	170	221	3907.31
3	150	120	150	235	4164.37
4	90	80	150	213	4402.87
5	150	80	150	215	3621.65
6	120	100	150	220	4312.36
7	120	80	130	203	4457.18
8	120	100	150	218	4234.29
9	120	100	150	209	4217.22
10	120	100	150	212	4310.34
11	90	120	150	225	4497.63
12	90	100	130	205	4126.91
13	120	100	150	208	4298.57
14	150	100	130	211	4126.32
15	90	100	170	225	4478.27
16	120	120	170	232	4189.66
17	120	120	130	215	3968.49

Table 4 — Analysis of variance (ANOVA) of crease recovery angle of C8 treated samples

Source	Sum of squares	DF	Mean square	F value	Prob> F	
Model	1429.75	6	238.29	10.77	0.0007	Significant
A	28.13	1	28.13	1.27	0.2859	
B	512	1	512	23.14	0.0007	Significant
C	820.13	1	820.13	37.06	0.0001	Significant
AB	56.25	1	56.25	2.54	0.142	
AC	1	1	1	0.045	0.8359	
BC	12.25	1	12.25	0.55	0.474	
Residual	221.31	10	22.13			
Lack of fit	199.31	6	33.22	6.04	0.0517	Not significant
Pure error	22	4	5.5			
Total	1651.06	16				

values will promote more cross linking with cellulose hydroxyl group. The interactive effect among parameters is also insignificant, as indicated by their p values exceeding 0.05. With satisfactory increase in CRA, repellent finish based on C8 chemistry, however, doesn't appear to hamper the cross-linking of anti-crease agent with cellulose hydroxyl group.

3.2 Analysis of Water Vapour Transmission Rate of C8 Samples

The effect of process parameters on water vapour transmission rate of C8 based coated textiles is discussed here. Table 5 displays all main effects and two factor interaction effects as determined by ANOVA. The Model F-value of 1.99 implies that there is a 18.84 % chance that a "Model F-Value" this large could occur due to noise. Values of "Prob> F" less than 0.05 indicate that the model terms are significant at 95% confidence interval. In this case, A is significant model term. The model equation, which has coefficient of determination (R^2) 0.7189, which denotes goodness of fit and can well predict water vapour transmission rate (WVTR) for any given unknown parameters, is given below:

$$\text{WVTR} = 4282.10 - 146.88A + 74.83B + 9.07C - 82.50A^2 - 50.13B^2 - 77.13C^2 + 137.25AB - 146.83AC + 168.42BC \quad \dots (2)$$

The water vapour transmission rate decreases with increase in repellent concentration as shown in Fig. 2 (a). This is very much on expected lines while increase in curing temperature appears to be mildly reducing the transmission rate. The change in cross-linker concentration in the recipe has negligible influence on repellency properties as evident from the surface plots. There is no interactive effect among the process parameters also, as indicated by their p-values. The C8 based repellent appears to be singular contributor for fabric protection from fluids.

3.3 Analysis of Crease Recovery Angle of C6 Samples

In this analysis, all main effects and two factors interaction effects, as obtained by ANOVA, are studied (Table 6). The model is significant as indicated by F-value of 5.36. There is only a 1.02% chance that a "Model F-Value" of this large could occur due to noise. Values of "Prob> F" less than 0.05 indicate model terms are significant at 95% confidence interval. In this

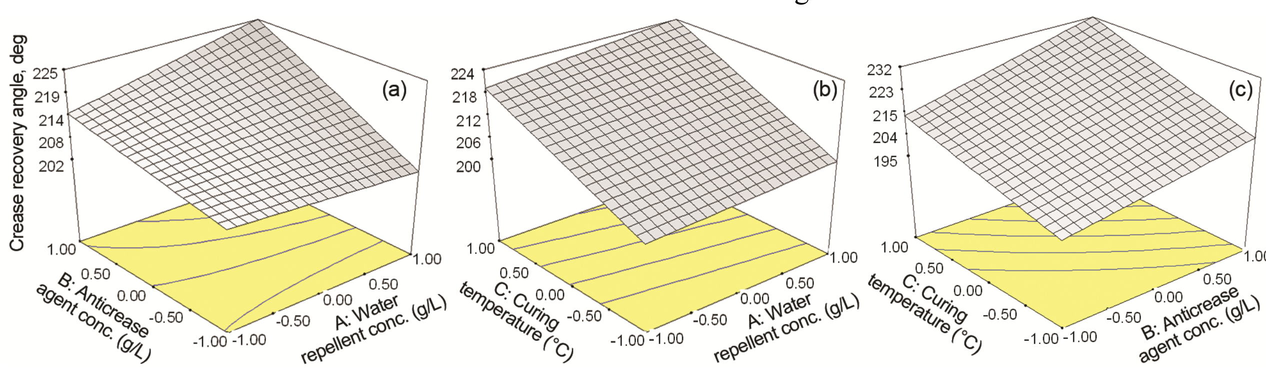

Fig. 1 — Effect of process parameters on crease recovery of C8 treated sample

Table 5 — Analysis of variance (ANOVA) of water vapour transmission rate of C8 treated samples

Source	Sum of squares	DF	Mean square	F value	Prob> F	
Model	5.645×10^5	9	62725.23	1.99	0.1884	
A	1.726×10^5	1	1.726×10^5	5.47	0.0519	Significant
B	44800.72	1	44800.72	1.42	0.2721	
C	658.12	1	658.12	0.021	0.8892	
A^2	28658.94	1	28658.94	0.91	0.3722	
B^2	10579.65	1	10579.65	0.34	0.5806	
C^2	25051.17	1	25051.17	0.79	0.4024	
AB	75350.25	1	75350.25	2.39	0.1661	
AC	86239.13	1	86239.13	2.73	0.1422	
BC	1.135×10^5	1	1.135×10^5	3.60	0.0997	
Residual	2.208×10^5	7	31535.98			
Lack of fit	2.203×10^5	3	73449.02	725.71	< 0.0001	
Pure error	404.84	4	101.21			
Cor Total	7.853×10^5	16				

Table 6 — Analysis of variance (ANOVA) of crease recovery angle of C6 treated samples

Source	Sum of squares	DF	Mean square	F value	Prob> F	
Model	985.50	6	164.25	5.36	0.0102	Significant
A	24.50	1	24.50	0.80	0.3924	
B	512.00	1	512.00	16.70	0.0022	Significant
C	392.00	1	392.00	12.78	0.0050	Significant
AB	16.00	1	16.00	0.52	0.4866	
AC	25.00	1	25.00	0.82	0.3878	
BC	16.00	1	16.00	0.52	0.4866	
Residual	306.62	10	30.66			
Lack of fit	191.42	6	31.90	1.11	0.4825	Not significant
Pure error	115.20	4	28.80			
Cor total	1292.12	16				

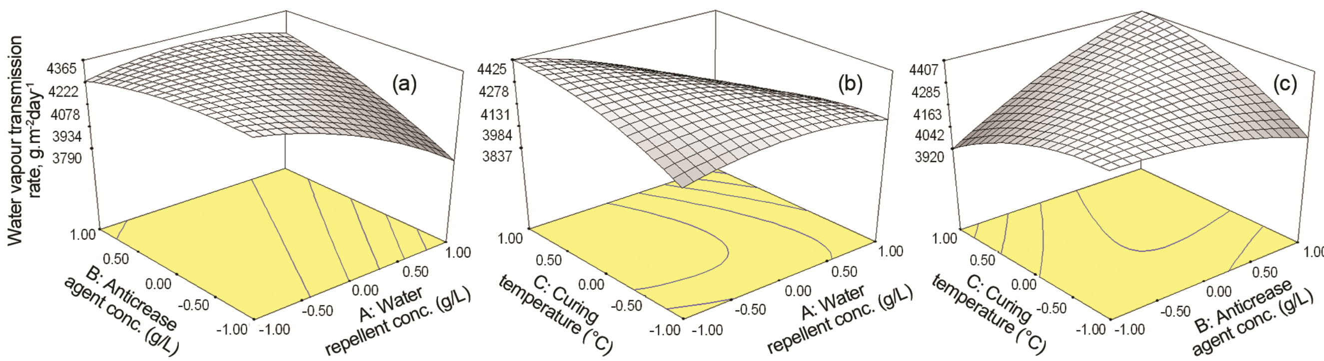

Fig. 2 — Effect of process parameters on WVTR of C8 treated sample

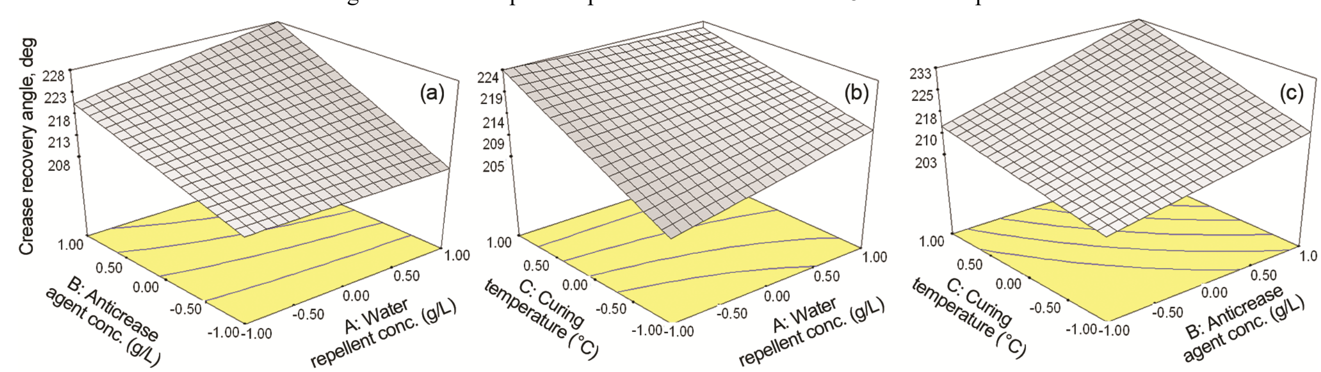

Fig. 3 — Effect of process parameters on crease recovery of C6 treated samples

case, B and C are significant model terms. The model equation is shown below, which has coefficient of determination (R^2) 0.7627, showing goodness of fit and the model can thus predict crease recovery angle (CRA) for any given unknown parameters:

$$\mathrm{CRA} = 216.41 + 1.75A + 8B + 7C + 2AB - 2.50AC + 2BC \quad \ldots (3)$$

In the recipe containing C6 based repellent, the CRA increases with increase in concentration of anti-crease agent as well as curing temperature. As it appears from the surface plots (Fig. 3), there is no perceptible change in the creasing behavior with repellent concentration. Although the influence appears to slightly varying with the level of resin content, but no particular synergy appears to exist between the two chemicals, as far as influencing the CRA is concerned. However, no interference is observed between their actions. The p-value for factor A (Table 6) further endorses it.

3.4 Analysis of Water Vapour Transmission Rate of C6 Samples

Box-Behnken design and response surface methodology have been used to study the impact of process parameters on the water vapour transmission rate of C6 based coated textiles. Table 7 shows every main effect and two factor interaction effect that the ANOVA revealed.

Table 7 — Analysis of variance (ANOVA) of water vapour transmission rate of C6 treated samples

Source	Sum of squares	DF	Mean square	F value	Prob> F	
Model	5.679×10^5	6	94653.66	4.08	0.0249	Significant
A	3.553×10^5	1	3.553×10^5	15.30	0.0029	
B	5496.24	1	5496.24	0.24	0.6371	
C	78.75	1	78.75	3.391×10^3	0.9547	
AB	50167.04	1	50167.04	2.16	0.1724	
AC	81330.48	1	81330.48	3.50	0.0908	
BC	75512.29	1	75512.29	3.25	0.1015	
Residual	2.323×10^5	10	23225.24			
Lack of fit	2.241×10^5	6	37342.89	18.23	0.0071	
Pure error	8195.08	4	2048.77			
Cor total	8.002×10^5	16				

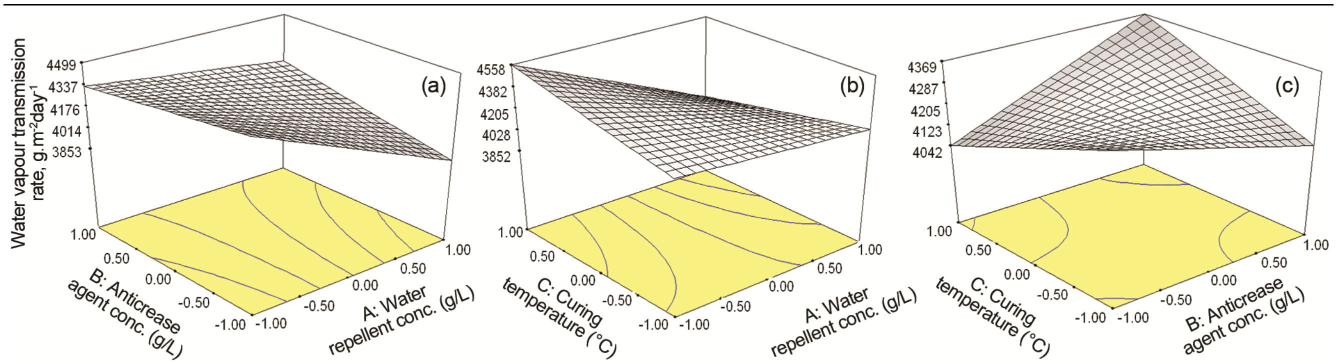

Fig. 4 — Effect of process parameters on WVTR of C6 treated samples

The Model F-value of 4.08 implies that the model is significant. The failure due to noise is as low as 2.49%. Values of "Prob> F" less than 0.05 indicate that the model terms are significant at 95% confidence interval. In this case, A is significant model term. Therefore, the model can well predict water vapour transmission rate (WVTR) for any given unknown parameters. The model equation is shown below, which has coefficient of determination (R^2) 0.7097, denoting goodness of fit:

$$WVTR = 4202.48 - 210.75A + 26.21B + 3.14C + 111.99AB - 142.59AC + 137.40BC \quad ...(4)$$

Higher repellent concentration in the recipe with C6 based fluid repellent appears to decrease the water vapour transmission rate [Fig. 4(a)]. But at lower curing temperature this effect is found to be less pronounced [Fig. 4(b)]. This could be attributed to lower polymerization of C6 chains due to inadequate conditions. However, the influence is prominent as the temperature is increased. The effect of curing temperature is further seen in Fig. 4(c), indicating its importance in getting the desired repellent action.

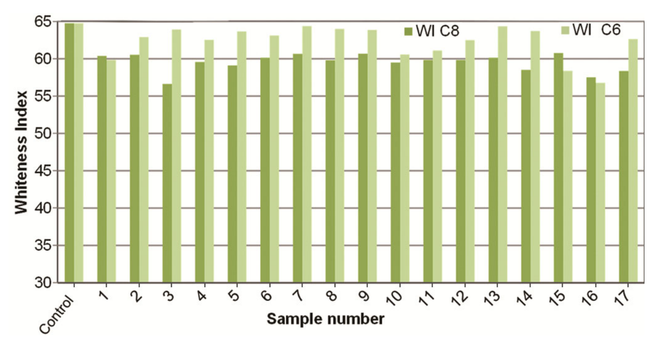

Fig. 5 — Whiteness trends in finished fabrics

3.5 Effect on Whiteness

The whiteness index is measured for the finished samples to study if any significant dulling or yellowing takes place on chemical application. However, there is not much change in these values (Fig. 5). Although there is slight reduction in whiteness in some of the treated samples compared to untreated samples, but it is not quite visually perceptible. There is also no specific trend between C8 and C6 formulations used.

3.6 Effect on Tensile and Tear Strength

Tensile properties of the samples when tested at an extension of 50 mm/min show a decreasing trend for

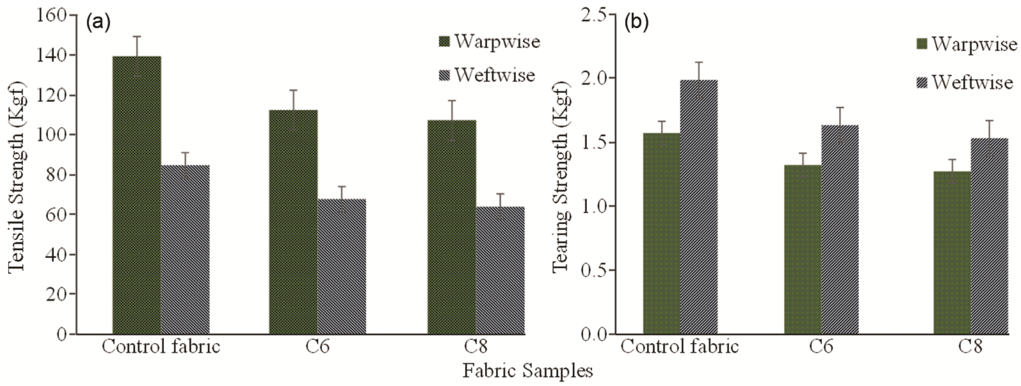

Fig. 6 — Strength loss in C6 and C8 based samples (a) tensile strength and (b) tear strength

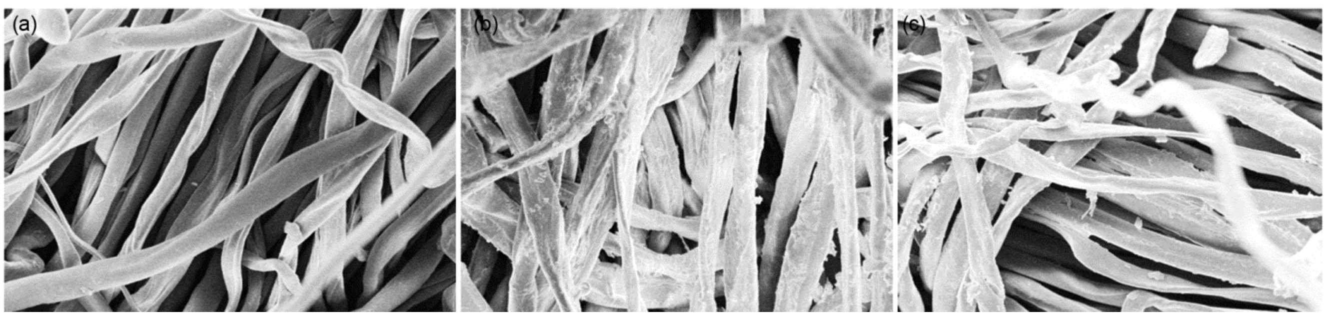

Fig. 7 — SEM images (500×) of (a) untreated, (b) C6 based and (c) C8 based samples

the finished fabrics. However, the decrease in strength is well within the acceptable limit of 25%. The loss in strength is attributed to acidic conditions due to mineral acid generation from catalyst during curing. Cotton, being sensitive to HCl liberated by magnesium chloride in the recipe, results in lowering of strength. Tear strength of the samples is tested at an extension rate of 2 in/min and the results show similar trends with relatively better results in C6 as compared to that in C8. The loss in tear strength is of course due to the same reason as in case of tensile strength. Results of a representative sample of both C6 and C8 recipes are shown in Fig 6.

3.7 Laundry Durability

The durability of the finish applied is tested after washing by ISO 105-CO2 wash fastness test method. This is done only for the optimised samples of C6 and C8 formulations. The repellency as well as wrinkle recovery properties are more or less retained in both the cases as compared to respective unwashed samples (Table 8). Also, the results between the two fluorochemical formulations are comparable, indicating the possibility for substitution of C8 with C6 type of fluid repellent in the multi-functional finish effect on the cellulosic substrate.

Table 8 — Water vapour permeability result before and after laundering

Washes	WVTR, $g.m^{-2}day^{-1}$		CRA, deg	
	C-6 based sample	C-8 based sample	C-6 based sample	C-8 based sample
Before laundering	3907.31	3969.30	221	226
After first cycle	4112.25	4208.56	214	223
After second cycle	4251.63	4362.12	212	218

3.8 SEM and EDX Results

The scanning electron micrographs indicate deposition of the chemicals on substrate as compared to untreated ones which have quite smooth appearance (Fig. 7). This indicates successful application of the finishes in C6 and C8 along with anticrease agent. Energy dispersive X-ray microanalysis for sample number 5 of both the formulations shows significant carbon, nitrogen and fluorine presence which is evident from Fig. 8 and Table 9. This is justified because of the chemicals used for the treatments. The crosslinker is carbon and nitrogen rich and forms an ether linkage with the substrate while the repellent finishes contain carbon and fluorine. Interestingly, the fluorine content in case of C6 finished fabric is more than in C8 finished one, possibly due to higher concentration of repellent used in the former.

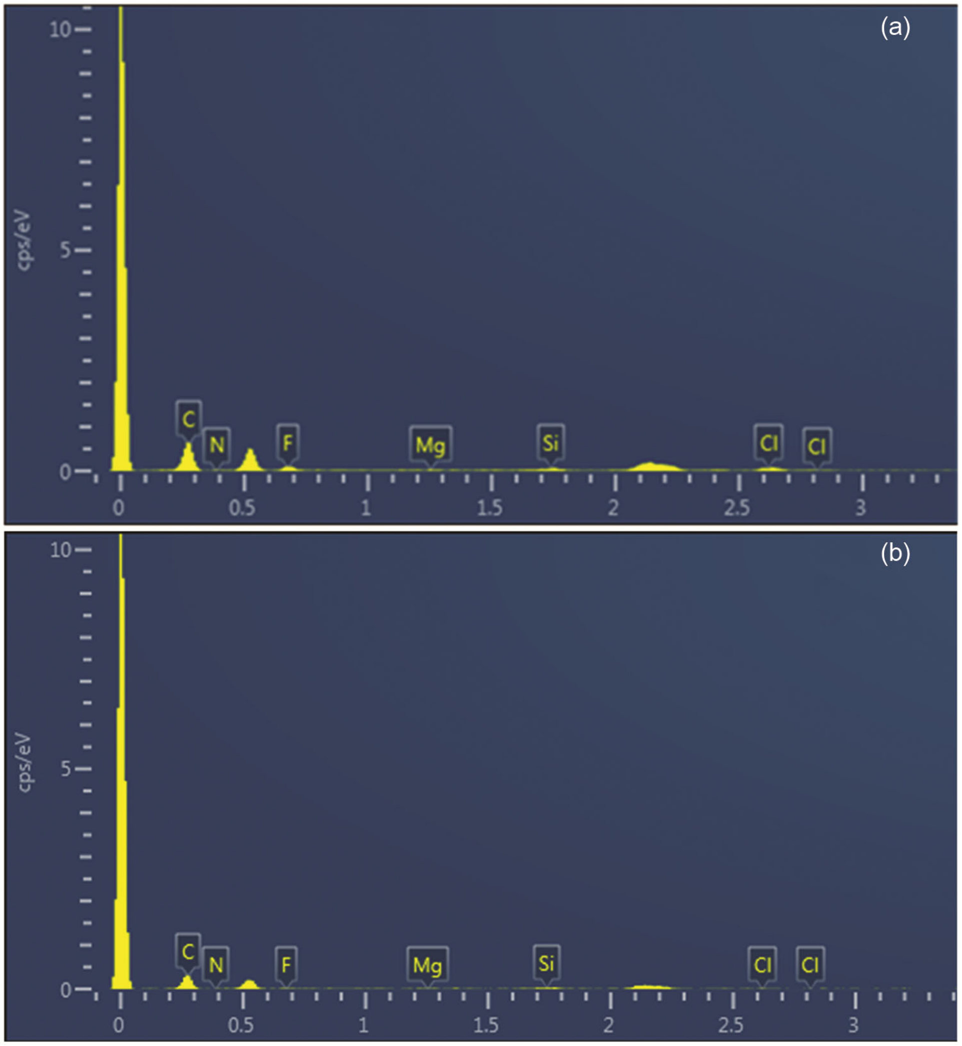

Fig. 8 — EDX analysis of treated samples (a) C8 based and (b) C6 based

Table 9 — Elemental analysis from EDX results

Element	Weight, %		Atomic, %	
	C-8 based sample	C-6 based sample	C-8 based sample	C-6 based sample
C	77.85	67.35	82.67	74.25
N	14.36	19.36	13.08	18.31
F	3.62	6.61	2.43	4.61
Mg	0.48	0.25	0.84	0.45
Si	2.63	1.19	2	0.94
Cl	1.06	0.38	3.83	1.43
Total	100	100	100	100

4 Conclusion

The aim to have multi-functionality by the finish formulation has been achieved to a great extent. The improvement in properties appears to serve reasonably well for upholstery usage. There is no significant difference between the C8 and C6 based liquid repellent treated cotton fabrics in terms of water transmission and wrinkle recovery. The modified DMDHEU based crosslinker has been suitably co-applied with the two variants of repellent finishes. The whiteness retention has also been quite satisfactory in both the cases post-finishing. Finishing, particularly curing, usually causes some dulling of whiteness, which is minimal in most cases in the present study. The tensile and tear strength loss caused by acid liberating agent in the form of catalyst for cross-linking is also with in manageable level. The durability to laundering exhibited by the samples is also quite satisfactory. The presence of chemicals in the substrate is visible in the SEM pictures. This is further endorsed by the elemental analysis with EDX. Although higher percentage of fluorine is seen in C6 treated samples, their bio-accumulation is far less compared to that of C8 ones. This makes C6 environmentally sustainable and hence a good substitute to C8 based liquid repellents. Also the evaluation of different properties makes C6 absolutely comparable to that of C8. The lone demerit is higher concentration of C6 based repellents is to be used for getting desired result.

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