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# Soil release performance of cotton finished with oleophobol CPR and CMC-Na salt

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## Abstract

Soiling of textiles is an irresistible process that deteriorates the appearance of fabric caused by gradual accumulation of soil on it. Cotton fabrics were finished with two different types of soil releasing agents, viz. Oleophobol CPR (fluorocarbon based) and Carboxymethyl cellulose sodium salt (CMC-Na) using Box-Behnken and Full factorial designs, respectively and the effect of factors and their levels on soil release property of fabric was studied. Finished fabrics were tested for two different soils i.e., mustard oil and hot coffee. From soiled and laundered fabrics soil release was evaluated for soil release rating as well as instrumental prediction with K/S value to measure discolouration of fabric due to soil. The CMC-Na salt finished cotton fabric picked up significantly more soil compared to that with Oleophobol CPR which resisted deposition of soil on fabric. Cotton fabrics finished with both of these showed excellent soil release with mustard oil and had better finish durability with Oleophobol CPR. Physical properties were comparable except stiffness which was high in CMC-Na finished cotton.

**Keywords:** Oleophobol CPR; Fluorocarbon; CMC-Na salt; Soil release rating; K/S value; Box Behnken design; Full factorial design

## Introduction

Soiling of textile is a natural and undesirable phenomenon. Soil can be solid or liquid or in mixtures (Cooke 1987; Kissa 2001). Soil release finish is important to get complete and satisfactory cleaning of soils with household detergents. This finish enhances hydrophilic surface characteristics of fabric allowing the soil to penetrate up to limited extent into the fabric and developing its activity during laundering, where its special functional groups remove the soil from the fabric and transfer it to the detergents (Bille et al. 1969). It also enhances fabric properties like protection from soil re-deposition during laundering and absorbency or transport of liquid water (Schindler and Hauser 2004).

Based on the chemical structures different types of soil release finishes based on polymers containing carboxyl groups or oxyethylene or hydroxyl groups are starch, carboxy-methylcellulose (CMC), polyvinyl alcohol (PVA), methyl cellulose, ethyl cellulose, hydroxypropyl starch, hydroxyethyl cellulose, hydroxypropylmethyl cellulose and hydrolysed cellulose acetates etc were formulated. Fluorine based compounds containing hydrophilic moieties such as polyoxyethylene, hybrid fluorochemical etc. act as soil release agents. The performance characteristics of fabrics treated with these

soil release finishes can be evaluated through oily soil-release testing (Cooke 1987; Schindler and Hauser 2004; Heywood 2003, Kissa 1984; Buck and Schubert 2009).

Fluorocarbon based compounds forming a thin film coating around fibers are used for soil release or stain repellent finishes. They alter the fabric surface properties by lowering the surface tension of the fabric which repels spill on contact by providing hydrophobic barrier to water and soils (Easter and Ankenman 2004, 2005; Hashim 1986). They are organic compounds consisting of perfluorinated carbon chains with more fluorine than hydrogens attached to carbon, having thermal and chemical stability. They have alternating hydrophilic and oleophobic units along a single hydrocarbon polymer backbone that gets strongly adsorbed and even forms covalent bond to the treated surface and orients perpendicular to the surface providing a low surface energy 'barrier' to both water and oil-based staining agents (Grajeck and Petersen 1962; Buck and Schubert 2009; Read and Culling 1967; Mather and Wardman 2011). Hybrid fluorochemicals are used with dual action of both soil-repellency and soil-release properties in air and in water (Kissa 2001). Critical surface tension for fluorocarbon surfaces is  $6 \text{ mN m}^{-1}$  ( $-\text{CF}_3$ ) to  $28 \text{ mN m}^{-1}$ , for bleached cotton it is  $44 \text{ mN m}^{-1}$  and surface tension value of water is  $72.8 \text{ mN m}^{-1}$  (Latta and Sello 1981; O'Lenick 1999). Typically, textile fluorochemicals are cationic in nature but can also be in their non-ionic or anionic forms. They are aqueous dispersions of a fluorinated polymer with some processing aids such as organic solvents or surfactants selected according to desired performance properties required like repellency or release etc., along with new advancement which does not affect other properties of the fabric (Arunyadej et al. 1998; Easter and Ankenman, 2004; Hashim 1986).

Oleophobol CPR, a soil release chemical is made up of dispersion of a perfluorinated polymer compound having non-ionic/cationic nature. It is based on C6 chemistry which is superior to previously used C8-based fluorocarbons that releases perfluorooctane sulfonate (PFOS) and perfluorooctanoic acid (PFOA) which are highly hazardous and toxic. PFOS is classified as a persistent, bioaccumulative, and toxic (PBT) substance by 'The Organisation for Economic Co-operation and Development' (OECD). Whereas C6 chemistry based fluorocarbons exclude the possibility of fluorocarbon products to break down into PFOS and PFOA and the resultant compound perfluorohexanoic acid (PFHA) has 40 times less bioaccumulative effects than that with PFOA (Holme 2012).

Carboxymethyl cellulose is one of the first antiredeposition agents used in detergents which acts as dispersion for soil (Cooke 1987; Noel et al. 1975). As a soil release agent it forms a protective coating over the fabric which gets easily desorbed and removed with every subsequent laundering along with soil from fibre (Heywood 2003). It is readily absorbed by cellulose fibres forming hydrogen bonds that smoothens the fibre surface, therefore, reducing soil adherence (Slade 1997). Its hydrophilic nature enhances cleaning efficiency. The carboxy groups present on it increase the electronegative charge on the fibre and dirt particles possess negative charge in aqueous bath thereby repelling each other (Pepperman et al. 1971; Beninate et al. 1966).

The present study was aimed at formulating soil release finish conditions to predict optimum soil release properties in terms of its soil release rating and K/S value on cotton with two different soil release chemicals viz., Oleophobol CPR and Carboxymethylcellulose sodium salt (CMC-Na salt) using mustard oil and hot coffee as soils. For Oleophobol CPR (a C-6 type fluorocarbon based product) Box-Behnken

design was used in conjunction with response surface analysis and regression methods; for CMC-Na salt full factorial design was used. Finished cotton was evaluated for its visual assessment (soil release rating) as well as instrumental assessment (K/S value) followed by durability of finish. Statistical analysis of both soil release finishes were carried out in order to determine and visualize the relative importance of the factors and also to obtain optimum conditions for finish. Effect on physical properties was also studied and compared.

## Methods

### Materials

In this study bleached and mercerized plain woven 100% cotton fabric was used having 126 g/m<sup>2</sup>, 140 ends per inch, 72 picks per inch, 40s warp and 40s weft count. Oleophobol CPR (fluorocarbon based) and Carboxymethylcellulose sodium salt (CMC-Na salt, high density 1100-1900cp) were procured from Huntsman and SD-Fine respectively.

### Application of soil release agents on cotton fabric

Cotton fabric was immersed in respective finish liquor for 30 seconds at room temperature with conditions mentioned in Table 1 for varying concentrations and pH, succeeded by padding on laboratory padder under controlled pressure and speed, to achieve 70-80% expression (owf). Fabrics were dried at 80°C for 5 minutes. Oleophobol CPR treated fabrics were cured under stretched conditions to form crosslinks whereas in case of CMC-Na salt no curing was required because of inability of CMC-Na to form crosslinks.

### Evaluation of properties

Soil release property for both untreated and treated cotton was evaluated with mustard oil and 2% hot coffee as two different soils through AATCC test method 130:2006 for soiling and rating. In this study the finished fabrics were soiled with soils those are encountered in use and laundered with detergent at room temperature by consumer during use. Therefore, the reference oil used in the study was mustard oil because of its wide availability, consistent colour and domestic usage. Along with this, hot coffee as water based particulate soil was also selected to see how soil release finishes behaves to it.

Soiled, aged and laundered fabrics were visually assessed against photographic standards and rated accordingly (1: severe staining to 5: no apparent stain). To rectify results with error arising out of visual assessment, instrumental evaluation was also done by measuring K/S value with spectrophotometer (Datacolor Check, US) at 400 nm ( $\lambda_{Max}$ ) for both soils. This method eliminates visual variables and even detects small amounts of residual soil.

**Table 1 Factors and levels for finishing with Oleophobol CPR and CMC-Na salt**

	Independent variables	Levels			Independent variables	Levels			
		# 1 soil release chemical	-1	0		1	# 2 soil release chemical	1	2
X1	Oleophobol CPR (gpl)	40	50	60	CMC-Na salt (gpl)	0.5	1	1.5	2
X2	pH	4	5	6	pH	4	5	6	x
X3	Curing temperature (°C)	140	150	160					
X4	Curing time (min)	3	4	5					

Other properties tested were whiteness index (WI), total crease recovery (TCRA, AATCC Test 66-2003), tensile strength (ISO 13934-1:1999), tearing strength (ASTM D1424-09), bending length (ASTM D1388) and air permeability (AP, BS 5636).

## Experimental plan

### *Design of experiment*

A 4<sup>3</sup> Box Behnken design consisting of 27 runs with three replicates at central point was used to evaluate functional characteristics of Oleophobol CPR treated cotton. The characteristics of soil release were evaluated using two different soils (oil and water based) for its release rating and K/S value. The various parameters, viz, fluorocarbon concentration, pH, curing temperature and time were included in the research design and their levels are mentioned in Table 1. Results were analyzed with response surface plots and equations were formed for responses at 95% confidence level. The individual as well as interaction effect of the process factors on soil release rating and K/S value were examined. Analysis of responses related to independent variables was done using quadratic polynomial equation and model accuracy was verified by the coefficient of determination (R<sup>2</sup>).

$$Y = \beta_0 + \sum_{i=1}^4 \beta_i X_i + \sum_{i=1}^4 \beta_{ii} X_i^2 + \sum_{i < j=2}^4 \beta_{ij} X_i X_j$$

where Y is the predicted response for soil release rating or for K/S value,  $\beta_0$  is an intercept,  $\beta_i$ ,  $\beta_{ii}$  and  $\beta_{ij}$  are the coefficient of the linear, quadratic, and interactive terms of regression equation; and  $X_i$ ,  $X_{ii}$ , and  $X_{ij}$  represent the coded independent variables, respectively.

After studying the response surface plots, the conditions for soil release rating and K/S value were analyzed. Further from these 27 runs, only those combinations were selected for further study which had resulted in excellent soil release rating and lower K/S value to further evaluate durability and physical properties of fabric.

A full factorial research design was used to evaluate functional characteristics of CMC-Na finished cotton with two factors at varied levels giving 12 combinations (Table 1) that were studied for soil release rating and K/S value response. For each test performed, an Analysis of Variance (ANOVA) model was fit to the data and tests were conducted (at 95% confidence level) on the statistical significance of the main effects of both factors. All design formation and statistical analyses were done with Statistica 6 and Systat 12 softwares.

### *Soiling procedure*

Fabric soiling was done according to AATCC test method 130. On a bloating paper fabric specimen sized 4" × 4" was kept and approximately 0.2 ml of soil (mustard oil or hot coffee) was placed on it. On top of the soil pool, glassine paper was placed and a 2.3 kg weight with 2.5" diameter cylinder covering the soil and rested for one minute to force the soil into the fabric. Weight and glassine paper were removed and soiled fabrics were aged in open air for two hours before laundering.

### *Laundering*

Finished as well as soiled samples were laundered with 5 gpl laundry soap solution at 1:50 liquor ratio at room temperature (27 to 31°C) for a wash cycle of 12 minutes.

Fabrics were rinsed with water at room temperature for two minutes and were dried in air by drip drying.

### **Durability**

After laundering followed by soil release evaluation, the best finished combinations were further evaluated for finish durability by repeated soiling at same point, ageing and laundering. Durability of finish was also evaluated by subjecting finished fabric to five consecutive soiling-ageing-laundering cycles comparing soil removal characteristics of fabrics with both visual assessment (rating) and instrumental assessment (K/S value) at the end of each cycle.

## **Results and discussion**

### **Soil release performance of finished fabric**

Soil release rating and K/S value for 27 runs according to Box Behnken design with Oleophobol CPR finished cotton and for 12 different runs using full factorial design (4 levels of concentration and 3 levels of pH) with CMC-Na salt finished cotton are mentioned along with their design in Tables 2 and 3 respectively. In both the finishes, excellent soil release ratings were obtained for almost all the set of conditions when mustard oil was used. Interestingly, although the soil release rating was high with both the finishes with CMC-Na showing comparatively better results. Spectrophotometric assessment showed presence of small amounts of soil in the fabric which was not detected by visual assessment. CMC-Na finished cotton showed no prominent stain on finished fabrics; it formed a film on the fabric that was not permanent and showed poor wash fastness. K/S value of untreated cotton after first soiling (mustard oil)-ageing- laundering cycle was 0.1254, which was comparatively very high compared to that of finished fabrics i.e. 0.025 for Oleophobol CPR and around 0.06 for CMC-Na salt. Both CMC-Na and Oleophobol CPR showed poor soil release property with prominent stain on cotton with hot coffee showing release rating with Oleophobol CPR and CMC-Na as 2.5-3.5 and 2.5-3 respectively with corresponding K/S value of 0.2-0.3 (quite a high value). Coffee acts as a natural dye and has affinity to adhere with cotton. Due to poor results, further study on coffee as soil was dropped.

Statistical analysis was carried out for both the finishes with mustard oil. In case of Oleophobol CPR, response surface plots are shown in Figures 1(a-f) and 2(a-f) to see the effect of each independent factor, i.e., concentration, pH, curing temperature and time as mentioned in Table 1 and their interaction, as a function of two factors, whilst rest two factors were kept at a constant centre level on dependent variables i.e. soil release rating and K/S value. Significant influence of concentrations of Oleophobol CPR, pH and curing temperature was found on both responses studied. Regression equation for soil release rating and K/S data are mentioned in Table 4. The regression model showed significance with F value of 7.86 and 10.725 respectively for rating and K/S value implying that the models are significant as F value was less than that of the calculated one in both cases. The model showed statistically insignificant lack of fit for both responses, as is evident from the P value of 0.775 and 0.695 respectively, in case of soil release rating and K/S value. The lack of fit F-value of 0.575 and 0.747 in case of soil release rating and K/S value respectively showed the validity of the predictive

**Table 2 Box Behnken experimental design layout and observed responses for Oleophobol CPR\***

Run no.	Independent variable				Dependent variable**			
	X1: Oleophobol CPR (gpl)	X2: pH	X3: Curing Temperature (°C)	X4: Curing Time (minute)	Y1: Soil release rating	Y2: K/S	Y3: Soil release rating	Y4: K/S
					Mustard oil		Hot coffee	
1	40	4	150	4	4	0.0386	3	0.2521
2	60	4	150	4	5	0.025	3.5	0.2042
3	40	6	150	4	3.5	0.0412	2.5	0.3254
4	60	6	150	4	4	0.0349	3	0.2476
5	50	5	140	3	4	0.0359	3	0.2912
6	50	5	160	3	4.5	0.0281	3.5	0.2810
7	50	5	140	5	4.5	0.0328	3	0.2505
8	50	5	160	5	5	0.0257	3.5	0.2321
9	50	5	150	4	4.5	0.0308	3	0.2638
10	40	5	150	3	4	0.0356	2.5	0.2940
11	60	5	150	3	4.5	0.0318	3.5	0.2414
12	40	5	150	5	4	0.0362	3	0.2711
13	60	5	150	5	5	0.0261	3.5	0.2331
14	50	4	140	4	4	0.0313	3	0.2553
15	50	6	140	4	3.5	0.0391	2.5	0.2707
16	50	4	160	4	4.5	0.0298	3.5	0.2511
17	50	6	160	4	4	0.033	3	0.2748
18	50	5	150	4	5	0.0267	3.5	0.2601
19	40	5	140	4	3.5	0.0402	2.5	0.2961
20	60	5	140	4	5	0.0259	3	0.2662
21	40	5	160	4	4	0.0341	3	0.2901
22	60	5	160	4	5	0.025	3.5	0.2537
23	50	4	150	3	4.5	0.0299	3	0.2652
24	50	6	150	3	4	0.0371	2.5	0.2806
25	50	4	150	5	5	0.0256	3.5	0.2599
26	50	6	150	5	4	0.0324	3.5	0.2785
27	50	5	150	4	4.5	0.0303	3.5	0.2631

\*Soils used were mustard oil and hot coffee 2 (%).

\*\*All responses readings after laundry only.

Note: Untreated cotton soiled and laundered had rating 3-3.5 and K/S value 0.1254 for mustard soil; 2.5 rating and K/S value 0.3547 for coffee.

model. Significant model terms for soil release rating were X1, X2, X3, (X2)<sup>2</sup>, and (X3)<sup>2</sup> and for K/S were X1, X2, X3, X4, (X1)<sup>2</sup> and (X2)<sup>2</sup>.

In case of CMC-Na salt finish, the influence of independent factors, viz. concentration of CMC-Na salt and pH of the bath (Table 1) and their interaction on dependent factors i.e. soil release rating and K/S value were analyzed by regression data and ANOVA (Table 5). From statistical analysis it was found that concentration of CMC-Na salt played significant role in the soil release rating as well as K/S value showing significance of concentration in regression with  $\beta$  0.898 for rating and 0.88 for K/S value but pH didn't show any significant effect. F value calculated from the ANOVA table showed that at significance level of 5 (%) for both rating and K/S

**Table 3 Full factorial design layout with observed responses for CMC-Na \***

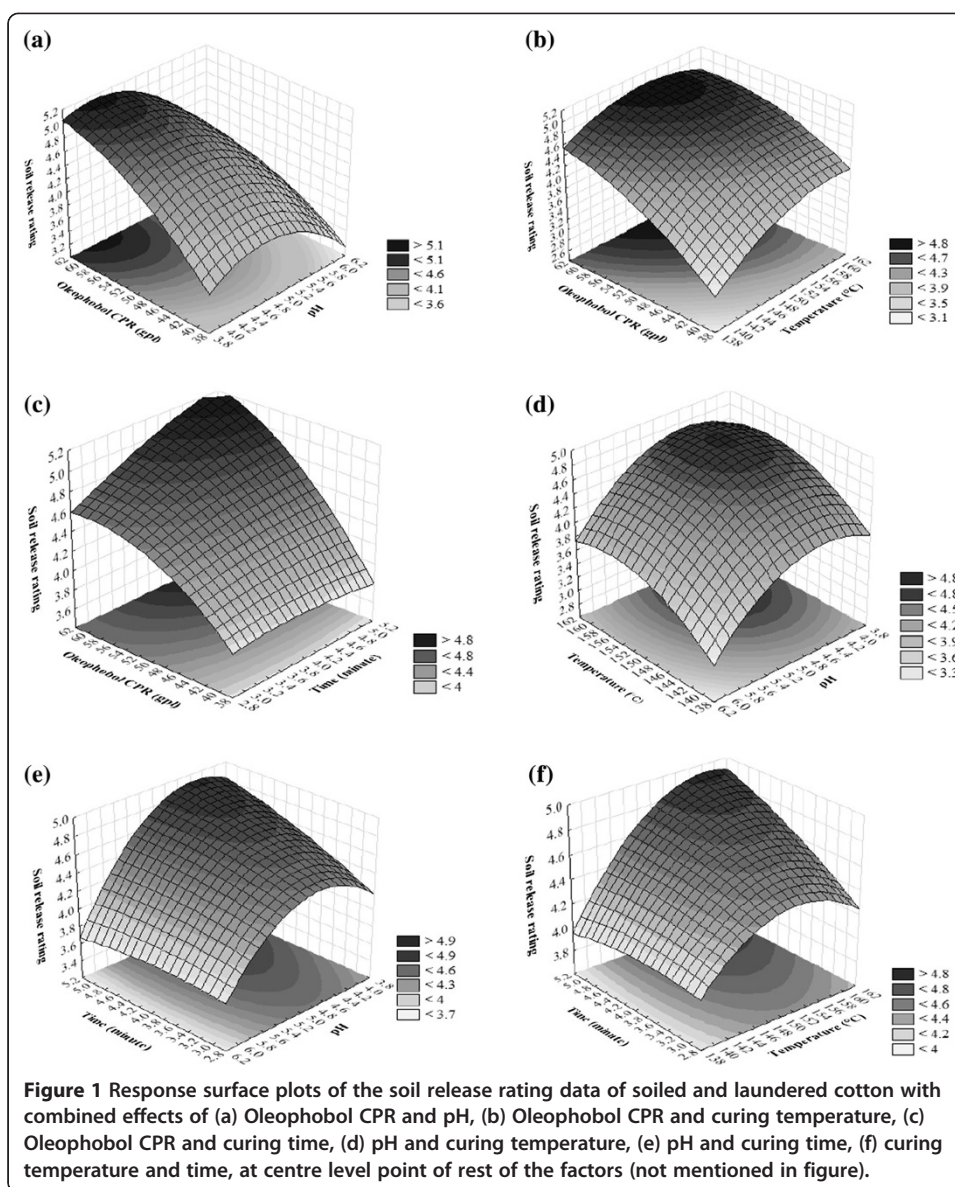
Run No.	Independent variable		Dependent variable				Add on* (%)
	X1: CMC –Na salt (gpl)	X2: pH	Y1: Soil release rating	Y2: K/S	Y1: Soil release rating	Y2: K/S	
			Mustard oil		Hot coffee		
1	0.5	4	4.8	0.0686	3	0.2523	0.55
2	0.5	5	4.8	0.0678	2.5	0.2731	0.48
3	0.5	6	4.7	0.0701	3	0.2131	0.68
4	1	4	4.9	0.0665	2.5	0.2781	0.88
5	1	5	4.9	0.0669	2.5	0.2716	0.95
6	1	6	4.8	0.069	2.5	0.2826	1.13
7	1.5	4	5	0.064	3	0.2223	0.98
8	1.5	5	5	0.0657	2.5	0.2787	1.08
9	1.5	6	5	0.0651	2.5	0.292	1.16
10	2	4	5	0.0644	2.5	0.2974	1.29
11	2	5	5	0.0652	2.5	0.2715	1.27
12	2	6	5	0.0635	3	0.2297	1.31

\*Add on % = [(dry weight of fabric after padding - initial dry weight of fabric) / (initial dry weight of fabric) × 100].

value, calculated F was greater than F tabulated. Hence it could be concluded that CMC-Na salt concentration had a significant effect on both soil release rating and K/S value.

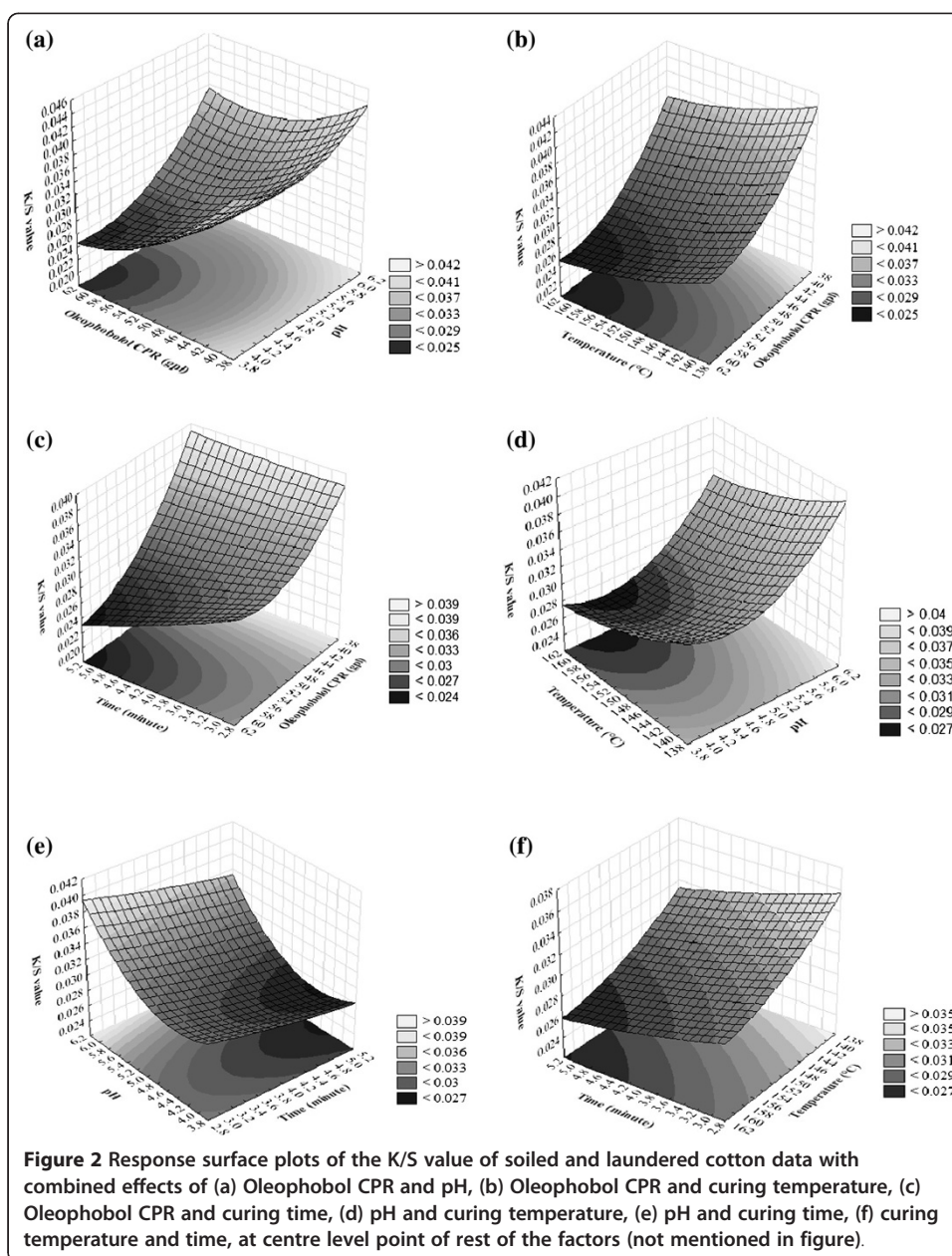
On studying the influence of parameters with response surface figures for Oleophobol CPR on soil release rating and K/S value as responses with mustard oil, it was found from Figure 1(a) that soil release ratings were excellent at all concentrations of (40-60 gpl) and at pH 4-5. At lower concentrations, e.g. 40-48 gpl and at pH 4-6, the soil removal performance was comparatively less. Rating increased especially with concentration greater than 48 gpl and at pH 4-5. Soil release rating was improved (Figure 1b) when temperature was simultaneously increased from 150 to 160°C at a concentration beyond 54 gpl. Lower concentration i.e. 40 to 50 gpl and curing time 3-5 minutes showed less effect on soil release rating (Figure 1c). In all cases maximum soil release rating was seen at pH 4-5. When temperature was increased beyond 150°C and pH was kept around 4 excellent soil release ratings were achieved with the maximum at 154-156°C and pH 4.4-4.7 (Figure 1d). With high curing time of 5 minutes at pH 6 soil release ratings obtained were not maximum but the latter was obtained at pH 4.5 and time > 4.4 minutes (Figure 1e). Soil release rating was not good even with maximum curing time at curing temperatures of 140-150°C (Figure 1f); substantial improvement in ratings was achieved at curing time of 4.4 minutes at temperature >150°C. From Figures 1(c, e, f) higher soil release ratings were obtained with curing time of 5 minutes, at concentration >50 gpl, temperature >150°C and pH 4-5. Concentration, pH and temperature played significant roles in soil release rating. Therefore, it could be concluded that Oleophobol CPR, only at high concentrations (>54 gpl) showed maximum soil release ratings at pH 4-5, high curing temperatures (150-160°C) and with enough time 4-5 minutes.

Study of K/S values showed that concentration, pH and curing temperature are significant factors in soil release. Lower K/S value indicates less soil retention in fabric and more soil release from finished cotton. It was evident from Figures 2(a-c) that at any pH between 4-6, K/S value decreased with increase in concentration of Oleophobol CPR (>54 gpl); lowest K/S achieved with Oleophobol CPR with 58-60 gpl



at pH 4-4.5 (Figure 2a). With temperature >150°C or with curing time >4 minutes when concentration was >52 gpl (Figure 2b, c), K/S value obtained was close to the K/S value of original untreated fabric (0.025). K/S value decreased when pH was shifted towards acidic side i.e. 4 and increased when pH was 6 at lower temperatures (around 140°C) and with high curing time (5 minutes). A temperature beyond 153°C, pH 4.1-5 (Figures 2 d-e) and curing time >4.5 minutes (Figure 2f) showed decrease in K/S value. Lower K/S value ensured no soil present in the cotton and it was obtained with Oleophobic CPR >52 gpl at pH 4-5, curing temperature >150°C and time up to 5 minutes, as can be found in Figures 2(a-f). It could be concluded that Oleophobic CPR at higher concentrations showed excellent results at pH 4-6 and high curing temperature. Optimized conditions were obtained when Oleophobic CPR concentration was kept at 60 gpl, pH ~ 5, curing temperature ~ 160°C and time of 5 minutes.





In case of CMC-Na salt finished fabric two factors were studied for their influence on soil release rating and K/S value (Table 3). It was found that maximum soil release property was obtained at pH 4 along with all concentrations of CMC-Na salt selected. Comparing the results, fabrics with CMC-Na salt concentrations at 0.5 and 1 gpl showed better results in acidic pH 4-5 rather than at pH 6 which had slight oil marks with higher K/S value when compared with finished with it at same concentrations of CMC-Na salt at pH 4-5. It was observed that increase in concentration of CMC-Na salt caused difficulty in solubilizing in water; moreover, pad liquor became highly viscous. As the concentration increased from 0.5 gpl to 2 gpl there was increase in the viscosity of the CMC-Na salt solution. Concentration beyond 1 gpl had a visible wet layer on cotton and took nearly 10 minutes to dry at 80°C. The fabric got stiffer with increase in

**Table 4 Regression equation formed in terms of coded values for Oleophobol CPR**

Response (Y)	Regression equation with all factors	R <sup>2</sup>	Adj. R <sup>2</sup>	F- value
Soil release rating (Y1)	$Y1 = 4.667 + 0.458(X1) - 0.333(X2) + 0.250(X3) + 0.125(X4) - 0.167(X1)^2 - 0.354(X2)^2 - 0.229(X3)^2 - 0.042(X4)^2 - 0.125(X1 \times X2) - 0.125(X2 \times X4) - 0.125(X1 \times X3) + 0.125(X3 \times X4) + 0.125(X1 \times X4)$	0.902	0.787	7.868
K/S value (Y2)	$Y2 = 0.029 - 0.005(X1) + 0.003(X2) - 0.002(X3) - 0.002(X4) + 0.002(X1)^2 + 0.003(X2)^2 + 0.001(X3)^2 + 0.002(X1 \times X2) - 0.001(X2 \times X3) + 0.001(X1 \times X3) - 0.002(X1 \times X4)$	0.926	0.84	10.725

concentration (1.5 and 2 gpl), due to more add on % on cotton (Table 3). Based on this, durability of finish and physical properties were studied at lower concentrations of CMC-Na salt (Table 6), although at concentrations (1.5 to 2 gpl) the soil release rating was excellent with lower K/S value when compared to K/S values finished at lower concentrations.

In case of Oleophobol CPR all those sets of parameters resulting in excellent soil release rating as well as lower K/S value were studied further for durability of finish and physical properties (Table 6). In case of CMC-Na salt, although there was removal of soil, oil marks (translucent) could be seen, with high K/S value (after laundering) between 0.06-0.07. But this wasn't the case with Oleophobol CPR which had its K/S value between 0.025-0.04, and had no oil marks like CMC-Na salt after laundry.

It may be concluded here that cotton fabric finished with CMC-Na salt at 0.5 gpl was enough but in case of Oleophobol CPR, a minimum concentration of 50 gpl was essential at pH 4-5 along with curing temperature (150°C-160°C) for comparable soil release rating. CMC-Na salt formed a layer on fabric and required no curing as it doesn't produce any crosslinks with cellulose and pH played no significant role in its finish properties.

It was also observed that Oleophobol CPR finished fabrics resisted the oil or any soil (e.g. chocolate syrup, soya sauce, mobil oil, tea and ink which were also studied but not reported) to penetrate without any external pressure in the fabric i.e. it had made the surface of the fabric resistant to the oil as well as other soils (Table 7). Application of external pressure on soil already on the fabric caused it to penetrate; whereas, in the case of CMC-Na salt finish, the soil (oil or coffee) easily and immediately spread out without any external pressure and diffused inside the fabric.

**Durability of finished fabrics**

The durability of both the finishes towards soil release (mustard oil) were studied by subjecting finished fabrics to five consecutive soiling-ageing-laundering cycles, succeeded by assessment of soil removal both visually as well as instrumental prediction after each cycle. The results are shown in Figures 3 and 4. For both finishes there was decrease in soil release rating and increase in K/S value with every soiling-ageing-laundering cycle.

**Table 5 Statistical analysis of data for CMC-Na salt**


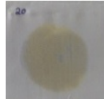
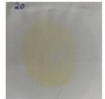
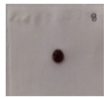

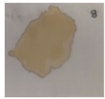
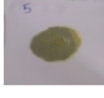

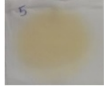



Dependent variable	Soil release rating	K/S value
Multiple R <sup>2</sup>	0.845	0.817
Adjusted Multiple R <sup>2</sup>	0.811	0.777
F- ratio	24.56	20.11

**Table 6 Physical properties of Oleophobic CPR and CMC-Na salt finished cotton with excellent soil release rating**

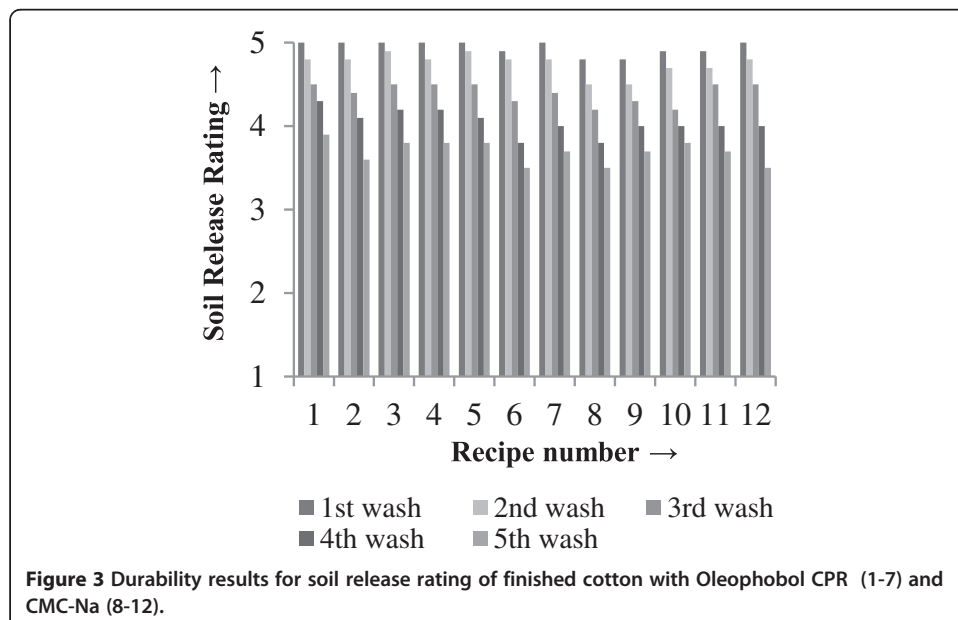
Recipe No.	Oleophobic CPR (gpl)	CMC-Na salt (gpl)	pH	Curing temperature (°C)	Curing time (minute)	Tensile strength (% retention)		Tear strength (% retention)		Bending length (cm)		TCRA*	WI*	AP* (cm <sup>3</sup> /cm <sup>2</sup> /s)
						Warp	Weft	Warp	Weft	Warp	Weft			
UN*	-	-	-	-	-	100	100	100	100	2.3	1.52	155	78.8	17.96
1	60	-	4	150	4	82.01	95.8	109.38	105.33	2.16	1.51	171	74.5	17.16
2	60	-	5	140	4	84.13	96.2	111.86	111.09	2.26	1.53	162	73.6	16.43
3	60	-	5	160	4	85.93	95.18	107.46	110.95	2.15	1.61	157	73.15	16.64
4	60	-	5	150	5	88.86	106.18	109.38	107.54	2.19	1.49	169	73.62	16.79
5	50	-	5	160	5	88.27	93.14	110.73	106.95	2.09	1.5	170	73.5	17.42
6	50	-	5	150	4	88.86	100.91	108.36	109.76	2.33	1.61	158	73.61	16.43
7	50	-	4	150	5	85.34	109.9	112.43	110.65	2.28	1.63	159	73.17	17.13
8	-	0.5	4	-	-	95.25	97.32	109.27	104.29	2.63	1.7	126	75.19	16.12
9	-	0.5	5	-	-	91.06	96.27	100.56	100.74	2.65	1.71	135	75.33	17.68
10	-	1.0	4	-	-	75.3	95.26	104.07	97.34	2.75	1.75	141	75.53	17.53
11	-	1.0	5	-	-	77.35	95.71	108.59	111.69	2.73	1.76	131	74.86	17.13
12	-	1.5	4	-	-	78.71	92.95	105.65	97.78	2.81	1.79	126	74.37	17.27

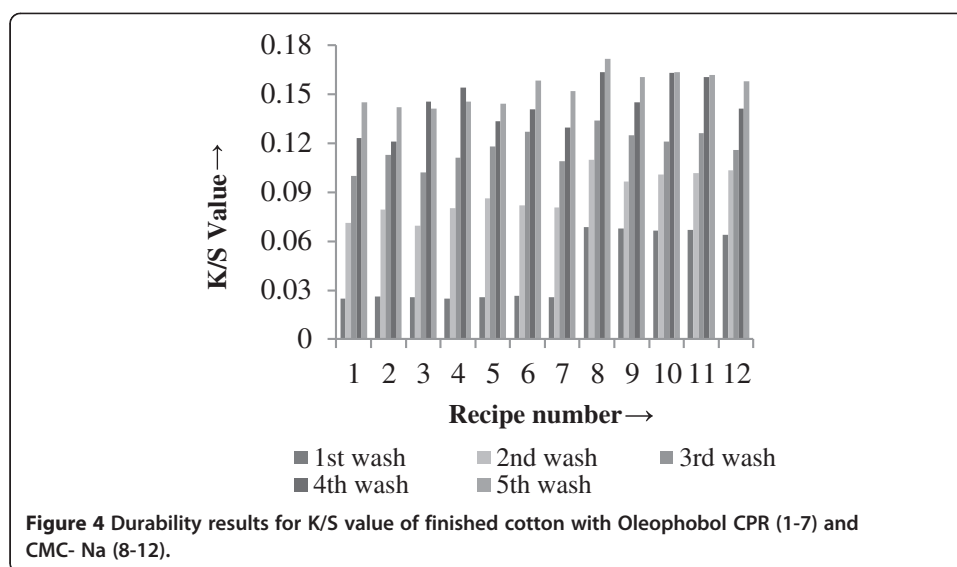
\*AP: Air permeability; TCRA: Total Crease recovery angle; WI: Whiteness index; UN: Untreated fabric.

**Table 7 Behavior of penetration of soils in finished cotton**

Finishing agent	Soil	Initial soil on finished cotton	After external pressure (1 minute)	After drying (2 hrs)
Oleophobol CPR	Mustard oil			
Oleophobol CPR	Hot Coffee			
CMC-Na salt	Mustard oil			
CMC-Na salt	Coffee			

In case of Oleophobol CPR, all selected conditions had shown soil release rating close to 3.5-4 and significantly retained more soil that is evident from K/S value which increased from 0.025 up to 0.15 with every cycle of test performed. Oleophobol CPR at 60 and 50 gpl showed a rating of 3.5- 4 with better durability at 60 gpl showing lower K/S value of 0.14 than that for 0.15 at 50 gpl at the end of all 5 cycles. Therefore, better level of soil release property was obtained after multiple launderings with higher concentration of fluoropolymer. CMC-Na finished fabric showed clear visible stains at





the end of 5<sup>th</sup> soiled-aged-laundered cycle. All conditions selected for durability study showed that rating decreased to 3.5 and K/S value increased from 0.06 to 0.17 by the end of 5<sup>th</sup> cycle. CMC-Na salt (1-1.5 gpl) showed better soil release rating and K/S value may be due to its removal from cotton after every laundry cycle but higher concentration still had much of it intact on cotton. However, all conditions showed low ratings and increased K/S value by the end of 5<sup>th</sup> cycle.

It could be concluded that durability of Oleophobol CPR finish was maintained with multiple soiled-aged and laundered cycles, with soil release rating close to 4 as compared to CMC-Na salt finish whose rating decreased to 3.5 with visibly clear soil marks. Moreover, K/S value in durability results showed that by the end of all five cycles CMC-Na salt had K/S value close to 0.17 i.e., it had more soil retention as compared to that with Oleophobol CPR with K/S value close to 0.15 by the end; Oleophobol CPR showed better resistance against soiling throughout all five cycles studied.

#### **Physical properties of finished fabrics**

Physical properties were evaluated for only those selected combinations which showed excellent soil release rating and lower K/S value for both soil release chemicals and are summarized in Table 6 along with their combinations.

Oleophobol CPR finished cotton retained its tensile strength with only 10-15 (%) loss. There was no loss in its tear strength rather it showed slight improvement. This may be due to the nature of fluorocarbon polymers which caused slippage of the yarn during the tearing process. Concentration of 60 gpl and curing temperature of 160°C showed slight improvement in TCRA. Stiffness, whiteness index and air permeability of finished fabric remained almost unchanged. Therefore, it could be concluded that finishing with Oleophobol CPR didn't hinder much of original physical properties of cotton rather maintained or slightly improved its properties along with provision of better soil release as well as repellent properties.

Increase in concentration of CMC-Na salt produced negative effect on some of the physical properties of fabric, especially stiffness (Table 6). When CMC-Na salt concentration was increased, it caused loss in tensile strength upto 20-25 (%) for warp and 5-7 (%)

for weft. Tear strength was almost retained and showed slight improvement. The possible reason could be increase in add on (%) from the film formed by CMC-Na salt on cotton which caused yarns to jam, thereby increasing its tear strength. This increase in add on imparted extensive stiffness on cotton, even at low concentrations. TCRA of finished and unfinished were found to be poor and comparable; may be due to high stiffness of the fabric not allowing fabric to recover to its original state. Whiteness index and air permeability decreased slightly.

It can be concluded that CMC-Na imparted a very stiff handle to the fabric; it showed poor TCRA, parallel to that of unfinished cotton and in case of Oleophobol CPR finish it was slightly improved. Whiteness index was slightly better in case of CMC-NA salt than with Oleophobol CPR. Oleophobol CPR finished cotton had better tensile retention with warp strength loss of 10–15 (%) and weft strength loss of 5 (%) as compared to those with CMC-Na salt with 20–25 (%) warp strength loss and 5–7 (%) weft loss. Tear strength was retained or improved slightly in both finishes.

## Conclusions

In this study, the soil release performance of two soil release finishes were investigated using design of experiment and factors playing important role in finish performance were studied. Durability of finish and physical properties of selected combinations having excellent soil release performance were also evaluated. Both Oleophobol CPR and CMC-Na finished cotton resulted in excellent soil release rating at various combinations for mustard oil but both showed poor soil release for coffee. Oleophobol CPR also exhibited soil repellent property and required high curing temperature for crosslinking with cotton. It didn't impart stiffness to the cotton as compared to that with CMC-Na salt finish. Oleophobol CPR showed much better tensile strength retention as compared to that with CMC-NA salt. Both finishes showed better tear strength retention of cotton. TCRA was slightly improved with Oleophobol CPR. Whiteness index was comparatively better in CMC-Na salt finished cotton. Air permeability in both cases was slightly decreased. Durability tests showed that Oleophobol CPR maintained its soil release and repellent properties with multiple (five) soiling tests and had better soil release rating as compared to that with CMC-Na salt.

## Competing interests

The authors declare that they have no competing interests.

## Authors' contributions

GD carried out the work and drafted the manuscript. Both authors read and approved the final manuscript.

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