# Development of brightness on handmade woolen carpets

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A process for the achievement of brightness on handmade carpet made from coarse wool fibre considering chlorine based oxidation of wool has been developed. Box-Behnken design of experiment is adopted for three factors (sulphuric acid, sodium hypochlorite and sodium hydroxide concentrations) at three levels for computation of responses like whiteness index, brightness index, and wear and abrasion loss. The optimized conditions are determined by multiple regression analysis aided by computer generated ANOVA analysis of raw data and contour graphs corresponding to various response surface models. The optimum conditions for process parameters are found to be 10 g/L sulphuric acid, 4.5g/L sodium hypochlorite and 10g/L sodium hydroxide. The handle, softness, whiteness, lustre and the anti-shrinkage properties of treated wool fibre improve in comparison to those of original fibre.

Keywords: Abrasion properties, Brightness index, Carpet, Whiteness index, Wool

## **1** Introduction

Handmade carpets command an important place in the carpet sector as they provide a means of earnings to millions of people attached to the industry in various capacities. The carpet types in the handmade sector include hand knotted, hand tufted and rod aided hand knotted (Tibetan/ Indo-Nepali) carpets. In general, the handmade carpets are wool focused from material point of view, since wool possesses several advantageous properties. Various types of natural and synthetic fibres are also used as alternative raw materials to gain economic and other benefits. Although wool possesses many desirable properties, yet it is not free from faults. The most troublesome part originates from the frictional anisotropy shown by the fibre, which in turn, gives rise to a property called felting shrinkage. The scales on the surface of the wool fibres are all aligned in the same direction with their free ends pointing towards the tip resembling the appearance of the scales on fish skin. This structural arrangement results in a difference in the coefficient of friction when wool fibre is subjected to abrasion against adjacent fibres with scale and against scale directions. This is known as directional frictional effect (DFE) which is the root cause of felting shrinkage<sup>1</sup>.

The strong entanglement between the filaments due to felting shrinkage poses many problems during processing of woolen carpets. The felting problem in

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woollen fabrics and garments is mitigated by shrink resist treatments with oxidizing agents, such as nascent chlorine (Hercosett process), dichloro isocyanuric acid, permonosulphuric acid, etc. followed by application of polymer coat. The oxidizing agents modify the morphology of the filaments by etching the surface and the polymer coat further reduces frictional difference. Other shrink resist treatments include use of enzymes and electrical discharge with plasma and  $UV^{2-6}$ . However, no information on shrink resist treatments for woollen carpets is documented in the literature, although the carpet industry employs some esoteric techniques to address the problem.

A methodical research on chemical processing of carpets to develop so called bright cool wool technology for woolen handmade floor coverings has hardly been conducted till date<sup>7</sup>. Handmade woolen carpet sector is age-old one and it mainly focuses on art and craft aspects without involving adequate supportive scientific information. The present research work has, therefore, been undertaken to fill up the gap between traditional practices and scientific reasoning in chemical processing of handmade woolen carpets.

The finishing of woolen carpet is generally carried out by mechanical as well as chemical treatments. The mechanical finishing comprises trimming of piles and hemming of edges which are generally carried out after chemical treatment. Traditionally, the chemical treatments are given by application of dilute solutions of sulphuric acid, bleaching powder and caustic soda in tandem to improve the functional properties of carpets,

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such as sheen, glaze, or brightness. These treatments also improve properties like felting, handle, etc. but the reason for poor sheen/glaze/brightness is not clearly understood. Hence, optimization of the chemical process and a proper interpretation of the cause-effect relationships is the need of the hour. The present investigation is therefore aimed at establishing the effects of input chemical on output responses, like whiteness, brightness, and wear and abrasion loss, in order to narrow down the gap in understanding. A better understanding of wool and its chemical processing would improve unit value realization and benefit the sector immensely.

# 2 Materials and Methods

#### 2.1 Carpet

Carpets (2 feet x 3 feet) used in this investigation, were prepared by a highly experienced artisan. All carpets were hand knotted made from 100% scoured but undyed wool yarns with a knot density of 54 knots/inch<sup>2</sup> and pile height of 12 mm. The wool selected for the investigation has an average diameter of 37.4 microns and a fibre length of 64.5 mm (Hauter). Wool was spun in woolen system to produce 3.9 Nm count yarns. Finishing treatment on carpets comprised mechanical trimming of piles and hemming of edges.

#### 2.2 Chemicals

Unless stated otherwise all reagents, such as sodium hydroxide flakes (E. Merck) and sulphuric acid (98% w/w, sp. gravity 1.84; Qualigens,) conformed to AR grade. Sodium hypochlorite solution (Qualigens, av.Cl<sub>2</sub> 4% w/v) conformed to LR grade.

## 2.3 Methods

The following process sequence was followed for treatment of each sample:

- Step I Impregnation with aqueous sulphuric acid for 10 minutes at 30°C
- Step II Washing with fresh water with continuous scrapping
- Step III Impregnation with sodium hypochlorite solution for 10 minutes at 30°C
- Step IV Washing with fresh water with continuous scrapping
- Step V Impregnation with sodium hydroxide solution for 10 minutes at 30°C
- Step VI Washing with fresh water with continuous scrapping
- Step VII Neutralization with acetic acid, if necessary, and confirmation by checking pH
- Step VIII Drying

Knot density and pile height of carpets were determined according to IS specifications<sup>8-9</sup>. The average wool diameter was determined with the help of Sirolan Laser Scan and the average fibre length according to IWTO method, using WIRA Fibre Diagram Machine (WIRA Instrumentation, UK). Whiteness Index (HunterLab), and brightness data were recorded at 10° observer angle by using Minolta spectrophotometer,(model CM 2500d), equipped with Jaypak 4802 colour matching software. Illuminant D65 was used as light source and the test method ISO brightness R 457was followed for estimation of optical properties<sup>10-12</sup>. Whiteness Index was measured using the following formula by spectrophotometer:

Whiteness index<sup>13</sup>(WI <sub>Hunter Lab</sub>) =L-3b  $L_{hunter} = 100(Y)^{1/2}$   $b_{hunter} = 70(Y' -Z')/(Y)^{1/2}$   $X' = X/X_w$   $Y' = Y/Y_w$  $Z' = Z/Z_w$ 

*X*, *Y*, *Z* are tri-stimulus values of sample,  $X_{w,}, Y_{w}, Z_{w}$  are tri-stimulus values of MgO reference tile.

ISO brightness R 457 is defined as Intrinsic radiance (reflectance) factor measured with a reflectometer having characteristics described in ISO 2469, equipped with a filter or corresponding function having an effective wavelength of 457 nm and a half bandwidth of 44 nm, and adjusted so that the UV content of irradiation incident upon the test piece corresponds to that of the CIE illuminant C. It has been measured using Minolta spectrophotometer.

Carpet wear and abrasion loss was evaluated using carpet wear and abrasion tester (SDL International, U.K.) according to test method IWS TM  $283^{14}$ , by recording the weight loss in mg after subjecting the carpet to abrasion for 1000 cycles. In all experiments, the carpets were treated as per the process sequence summarized in Section 2.3. The dried carpets were conditioned at  $20^{\circ}$ C and  $65 \pm 2\%$  RH and then subjected to evaluation.

# 2.4 Design of Experiment

Box-Behnken design of experiments (DOE) was adopted for quantitative estimation of the effect of different factors on various responses, especially on brightness of carpet. The independent variable and their levels were decided considering the findings of preliminary trials and on the basis of the feedback received from the industry regarding their average values used in traditional process. Sulphuric acid, sodium hypochlorite and sodium hydroxide were taken as the independent variables and their levels are given in Table 1. Multiple regression analysis of experimental data was carried out by using statistical

Table 1 — Description of independent variables and corresponding values at each level								
Independent variables	-1	0	1					
Conc. of sulphuric acid $(X_l)$ , g/L	10 (0.204 N)	20 (0.408 N)	30 (0.612 N)					
Conc. of sodium hypochlorite $(X_2)$ , Av. Cl <sub>2</sub> in g/L	1.5	3.0	4.5					
Conc. of sodium hydroxide $(X_3)$ , g/L	10 (0.25N)	25 (0.625 N)	40 (1.0N)					

Table 2 — Independent variables as per Box-Behnken design of experiments

Exp. No	Independent variables						
	Sulphuric	Sodium hypochlorite	Sodium				
	acid, g/L	Av. Cl <sub>2</sub> , g/L	hydroxide, g/L				
	$(X_I)$	$(X_2)$	$(X_3)$				
1	30	3.0	10				
2	20	1.5	10				
3	30	1.5	25				
4	20	4.5	40				
5	10	3.0	40				
6	10	4.5	25				
7	10	1.5	25				
8	20	3.0	25				
9	20	3.0	25				
10	30	3.0	40				
11	10	3.0	10				
12	20	3.0	25				
13	20	1.5	40				
14	30	4.5	25				
15	20	4.5	10				

software DX7 as a statistical tool for computation of the empirical relation between various factors and their responses.

# **3** Results and Discussions

In the present study, the traditional process has been modified by replacing bleaching powder with sodium hypochlorite and attempts have been made to optimize the process parameters in quantitative terms. The brightness index (BI) of undyed silk carpet is found to be 36, whereas the same for undyed woollen carpet is found 31. Hence, BI of silk is set as the benchmark for the present study.

Three factors chosen for the studies are sulphuric acid  $(X_1, g/L)$ , sodium hypochlorite  $(X_2, Av. Cl_2 in g/L)$  and sodium hydroxide  $(X_3, g/L)$ at three concentration levels (Table 2). The general form of the second degree polynomial equation has been generated by statistical package to estimate the responses of the input variables, as shown below:

$$Y = b_0 + b_1 X_1 + b_2 X_2 + b_3 X_3 + b_{11} X_1^2 + b_{22} X_2^2 + b_{33} X_3^2 + b_{12} X_1 X_2 + b_{23} X_2 X_3 + b_{13} X_1 X_3 \qquad \dots (1)$$

A three factor Box-Behnken experimental design involving a total number of 15 experiments including three replicates at the centre point is used (Table 2). Whiteness index (WI), BI, and carpet wear & abrasion loss (WAL) have been chosen as the dependent variables. The specimen obtained by treatments on carpet samples according to the DOE (Table 2) are subjected to testing for various responses. The experimental data vis-à-vis predicted values of responses are summarized in Table 3.

Table 3 — Summary of computed values of responses WI, BI and WAL against the corresponding experimental values

Exp. No	$X_{I}$	$X_2$	X3	Whiteness index (HunterLab) $(Y_l)$		Brightness index $(R 457) (Y_2)$		Wear & abrasion loss, mg/1000 cycle ( $Y_3$ )	
				Expt.	Predicted	Expt.	Predicted	Expt.	Predicted
1	30	3	10	59.54	59.18	28.37	29.06	42.98	43.00
2	20	1.5	10	61.36	60.20	29.80	29.53	42.50	41.86
3	30	1.5	25	60.00	56.72	28.76	28.36	46.09	46.69
4	20	4.5	40	61.17	58.63	29.92	30.24	49.76	50.39
5	10	3	40	62.00	59.94	32.05	31.40	48.30	48.27
6	10	4.5	25	63.40	62.40	31.62	32.06	46.40	45.78
7	10	1.5	25	61.90	60.85	31.17	31.08	43.22	43.48
8	20	3	25	61.70	58.63	29.92	29.09	45.60	45.27
9	20	3	25	59.87	58.63	28.64	29.09	44.17	45.27
10	30	3	40	61.36	55.93	29.80	29.46	51.00	50.61
11	10	3	10	62.74	63.19	32.08	32.45	40.80	41.17
12	20	3	25	59.87	58.63	28.64	29.09	46.07	45.27
13	20	1.5	40	62.84	60.40	32.48	33.26	47.98	47.73
14	30	4.5	25	60.60	58.52	29.32	29.44	47.02	46.75
15	20	4.5	10	66.59	65.32	35.37	34.61	41.32	41.55

Multiple regression analysis on the experimental data furnished the following second order polynomial Eqs (2), (3) and (4), which are used to compute the predicted values of WI ( $Y_1$ ), BI ( $Y_2$ ) and WAL ( $Y_3$ ) respectively, for a particular set of experimental conditions. The responses  $Y_{I_1}$   $Y_2$  and  $Y_3$  have been derived from the Tables 4-6. The polynomial equations are given below:

 $Y_{I} = 65.555 -0.0964 X_{I} - 1.045X_{2} - 0.150X_{3} + 0.00$  $426X_{I}X_{2} - 0.0766X_{2}X_{3} - 0.00292X_{I}^{2} + 0.5722X_{2}^{2} + 0.00543X_{3}^{2}$ ... (2)

 $Y_{2}=34.590 - 0.16638 X_{1} - 0.7344X_{2} - 0.1429X_{3} + 0.001833 X_{1}X_{2} + 0.002433X_{1}X_{3} - 0.09X_{2}X_{3} - 0.000833X_{1}^{2} + 0.5485X_{2}^{2} + 0.007074X_{3}^{2} \dots (3)$ 

 $Y_{3} = 38.41 + 0.0389X_{1} + 0.2902X_{2} + 0.1073X_{3} - 0.0375X_{1}$   $X_{2} + 0.000866X_{1}X_{3} + 0.03288X_{2}X_{3} + 0.00391X_{1}^{2} + 0.005$  $X_{2}^{2} + 0.00043884X_{3}^{2}$ 

#### 3.1 Whiteness index

The multiple regression equation for whiteness index is given by the quadratic Eq. (2). The contour graph (Fig. 1) derived from the response surface obtained by plotting  $X_1$  vs  $X_3$  shows that the isometric lines of WI follow parabolic paths. The perpendicular drawn at a given point on  $X_1$ -axis intersects each isometric lineat two points. The junction points approach each other with diminishing values of  $X_1$  and eventually converge at a threshold value Also, the WI

[Response $(Y_I)$ whiteness index; ANNOVA for response surface reduced quadratic model; Analysis of variance table (Partial sum of squares-Type III)]							
Source	Sum of squares	Df	Mean square	F Value	p-value	Prob > F	
Model	39.32	8	4.92	4.96	0.0333	Significant	
$X_I$ -sulphuric acid	9.12	1	9.12	9.20	0.0230	Significant	
$X_2$ -sodium hypochlorite	4.00	1	4.00	4.04	0.0911	Significant	
$X_3$ -sodium hydroxide	1.02	1	1.02	1.03	0.3489	Not significant	
$X_1X_3$	1.64	1	1.64	1.65	0.2458	Not significant	
$X_2X_3$	11.90	1	11.90	12.01	0.0134	Significant	
$X_I^2$	0.32	1	0.32	0.32	0.5928	Not significant	
$X_2^2$	6.12	1	6.12	6.18	0.0474	Significant	
$X_{3}^{2}$	5.52	1	5.52	5.57	0.0563	Significant	
Residual	5.94	6	0.99				
Lack of fit	3.71	4	0.93	0.83	0.6101	Not significant	
Pure error	2.23	2	1.12				
Cor total	45.27	14		-			
R-squared	0.8687						

Table 4 — Analysis of variance for whiteness index

Table 5 — Analysis of variance for brightness index

[Response (Y<sub>2</sub>) brightness index; ANNOVA for response surface reduced quadratic model; Analysis of variance table (Partial sum of squares-Type III)]

Source	Sum of squares	Df	Mean square	F value	p-value	Prob > F
Model	47.79	9	5.31	6.70	0.0248	Significant
$X_I$ -sulphuric acid	14.23	1	14.23	17.96	0.0082	Significant
$X_2$ -sodium hypochlorite	2.02	1	2.02	2.55	0.1712	Not Significant
$X_3$ -sodium hydroxide	0.23	1	0.23	0.30	0.6097	Not Significant
$X_1 X_2$	3.025E-003	1	3.025E-003	3.818E-003	0.9531	Not Significant
$X_1X_3$	0.53	1	0.53	0.67	0.4494	Not Significant
$X_2X_3$	16.52	1	16.52	20.86	0.0060	Significant
$X_{I}^{2}$	0.026	1	0.026	0.032	0.8643	Not Significant
$X_2^{\ 2}$	5.62	1	5.62	7.10	0.0446	Significant
$X_{3}^{2}$	9.35	1	9.35	11.81	0.0185	Significant
Residual	3.96	5	0.79			
Lack of fit	2.87	3	0.96	1.75	0.3836	Not significant
Pure error	1.09	2	0.55			
Cor total	51.76	14				
R-squared	0.9235					

[Response $(T_3)$ wear and abrasion loss; ANNOVA for response surface reduced quadratic model; Analysis of variance table (Partial sum of squares-Type III)]							
Source	Sum of squares	Df	Mean square	F Value	p-value	Prob > F	
Model	123.98	9	13.78	16.96	0.0031	Significant	
$X_I$ -sulphuric acid	8.76	1	8.76	10.78	0.0219	Significant	
$X_2$ -sodium hypochlorite	2.77	1	2.77	3.41	0.1239	Not significant	
$X_3$ -sodium hydroxide	108.34	1	108.34	133.42	< 0.0001	Significant	
$X_1 X_2$	1.27	1	1.27	1.56	0.2671	Not significant	
$X_1 X_3$	0.068	1	0.068	0.083	0.7845	Not significant	
$X_{2}X_{3}$	2.19	1	2.19	2.70	0.1614	Not significant	
$X_{I}^{2}$	0.57	1	0.57	0.70	0.4421	Not significant	
$X_2^2$	4.673E-004	1	4.673E-004	5.755E-004	0.9818	Not significant	
$X_{3}^{2}$	0.036	1	0.036	0.044	0.8415	Not significant	
Residual	4.06	5	0.81			-	
Lack of fit	2.10	3	0.70	0.72	0.6276	Not significant	
Pure error	1.96	2	0.98			C C	
Cor total	128.04	14					
R-squared	0.9683						





Fig. 1 — Effect of sulphuric acid and sodium hydroxide concentration WI at 3 g/L available chlorine of sodium hypochlorite

passes through a minimum with increase in alkali concentration  $(X_3)$  at any given acidconcetration. On the contrary, the WI decreases with increase inacid concentrationin the domains 10:30 (Fig. 1). The predicted values closely match with the experimental values only when higher concentrations of NaOCl are used in the intermediate stage and alkali extractionis done atlow alkali concentration (Table 3). This is understandable because at higher hypochlorite concentrationsthe thioester linkage between lipid layer and protein, located on cuticle cellsjust beneath the surface, is rupturedmore efficiently. Controlled alkali treatment dissolves the lipid, namely 18-MEA of wool but higher alkali concentrations cause yellowing of the fibre at the cost of WI. The highest value of WI is predicted at the point (10, 10).



Fig. 2 — Effect of sodium hypochlorite and sodium hydroxide on brightness index at sulphuric acid oncentration of 20 g/L

#### 3.2 Brightness Index

The same arguments hold good for the BI data which show that the BI increases with increase in both NaOCl and NaOH concentrations (Fig. 2). The lowest predicted value of BI lies at the central point ( $X_2$ = 3,  $X_3$ = 25) and the highest value at the point (4.5,10) within the domain 1.50: 4.5 for NaOCl and 10:40 for NaOH. Starting from the point (4.5,10), the BI diminishes along the diagonal line, passes through a minimum at the central point (3, 25) and again increases till the point (1.5, 40) is reached. The multiple R<sup>2</sup> value stands at 0.9235 which indicates that interactive effect of hypochlorite and alkali on BI is significant.

#### 3.3 Wear and Abrasion Loss

Wear and abrasion loss (WAL) of carpet sample shows significant relation among all factors as revealed by multiple R squared value which stands at 0.9683. The effect of H<sub>2</sub>SO<sub>4</sub> and NaOCl on WAL is shown in Fig. 3 which indicates that the extent of weight loss due to wear and abrasion lies between 40.8mg/1000 cycles and 51.0 mg/1000 cycles within the given domains. The predicted weight loss is minimum at the origin (1.50,10) which continuously increases along either axes. The maximum weight loss is attained at the point (4.50, 30). The reactive species present in NaOCl solution is a function of  $pH^{15}$ ; the concentration of elemental chlorine increases only at sufficiently low pH (<2). Thus, the higher the acid concentration more is the liberation of chlorine which, as mentioned earlier, culminates with rupture of the lipid-protein bond on the cuticle surface. Such surface modification paves inroads for penetration of chemicals (alkali) into the inner layers of cuticle cells, resulting in loss of fibre strength and enhanced WAL.

Figure 4 shows the effect of NaOCl and NaOH on WAL of carpet. In this case, the weight loss is marginally affected due to increase inhypochlorite concentration alone. This is because hypochlorite disrupts the surface lipid layer and subsequent treatment with alkali at low concentration does not cause much damageto the exposed protein layer. However, the weight loss tends toincrease with increase in alkali concentration, presumably due to susceptibility of wool to alkaline degradation. The shading or fibre loss due to abrasionis more pronounced when concentrations of both NaOCl and NaOH are high. The predicted weight loss is minimum at (3, 10) and maximum at (4.5, 40) within the given domain.

#### **3.4 Statistical Analysis**

The summary of results obtained by ANOVA analysis of various factors and WI as response is presented in Table 5. The analysis reveals that the factors  $X_1$  (sulphuric acid),  $X_2$  (sodium hypochlorite), interaction terms of  $X_2X_3$  (product of factors sodium hypochlorite and sodium hydroxide), and quadratic terms  $X_2^2$  (hypochlorite squared) and  $X_3^2$  (sodium hydroxide squared) are statistically significant. In general, the fitment of model represented by Eq. (2) is satisfactory since the multiple degree of determination ( $\mathbb{R}^2$  value) is 0.87.

The results of ANOVA analysis on the effect of various factors on BI is presented in Table 5. It is observed that amongst all the factors the effect of only sulphuric acid  $(X_I)$  is highly significant. The negative coefficient of  $X_I$  in Eq. (3) suggests that sulphuric

acid has a negative contribution towards BI which is statistically significant at 99% confidence level. The interaction terms  $X_2X_3$  as well as the quadratic terms  $X_2^2$  and  $X_3^2$  are also statistically significant. In general, the fitment of model represented by Eq. (3) is satisfactory since the multiple degree of determination (R<sup>2</sup> value) is 0.92.

The effect on WAL of carpet was examined by ANOVA analysis of various input variables and their responses (Table 6). The multiple regression Eq. (4) indicates that each factor has positive contribution towards WAL. The effect of factor ( $X_1$ ) is statistically significant at 98% and that of factor ( $X_3$ ) at almost 100% confidence levels. The contribution of remaining factors is not significant. The multiple degree of determination ( $\mathbb{R}^2$  value) is 0.97, indicating that the fitment of model represented by Eq. (4) is highly satisfactory in general.



Fig. 3 — Effect of sulphuric acid and sodium hypochlorite concentration on wear and abrasion loss at sodium hydroxide concentration of 25 g/L



Fig. 4 — Effect of sodium hypochlorite and sodium hydroxide on wear and abrasion loss at sulphuric acid of 20 g/L

The effect of sodium hypochlorite and sodium hydroxide on WAL of carpet is shown in Fig.4. The wear and abrasion loss of carpet progressively increases with increase in concentration of eachof the variables. The interactive action of the two input variables shows a synergistic effect. The predicted weight loss is minimum at (3, 10) and maximum at (3, 40). It is concluded that sodium hypochlorite and caustic soda have significant effect on shading or fibre loss due to abrasion of woolen carpet.

The fitment of models represented by Eqs (2)-(4) for WI, BI and WAL & respectively, having been established, attention has been paid on defining the optimum levels of all factors in their respective domains to achieve maximum (or minimum) output responses, as desired. The predicted values of  $X_l$  and  $X_2$  for highest WI lies at the point  $X_1=10$ ,  $X_3=10$  (Fig. 3). Substituting these values of  $X_1$  and  $X_3$  along with those of  $X_{2(\text{max})}$  and  $X_{2(\text{min})}$  in the domain 1.5: 4.5 in Eq. (2), the predicted values of  $WI_{max}$  and  $WI_{min}$  stand at ~67.0 and 62.0 respectively. Substituting  $X_{2 \text{ max}}$  and  $X_{2}$ min for all possible combinations containing upper and lower extreme levels of  $X_1$  and  $X_3$  in Eqs (2), the predicted values indicate that the best possible combination for WI is  $X_1=10$ ,  $X_2=4.5$  and  $X_3=10$ , for which the predicted WI stands at 67. Similar exercise on BI data reveals that the predicted  $\mathrm{BI}_{max}\, lies$  at 36.2 with the same combination of  $X_1, X_2$  and  $X_3$ . However, the predicted value of WAL under this set of conditions is marginally higher (41.6 mg/1000 cycles) than the WAL<sub>min</sub> of 40.8 mg/1000 cycles over the entire range. It is concluded that the optimum level of the factors is given by the combination  $X_1=10$ ,  $X_2=4.5$ and  $X_3=10$ . The experimental values obtained for WI, BI and WAL under these conditions are 68.04, 36.86 and 41.83 respectively, which are in good agreement with the forecasts.

#### **3.5 Microscopic Studies**

Viewed under scanning electron microscope, the scales on the surface of untreated wool fibre appear as flat plates with sharp edges; the free edges being slightly protruded above the surface (Fig. 5). Some clusters of particulate matter are seen randomly adhered to the surface, which account for low lustre of the fibre. This is because the light falling on the particles is scattered in all directions, resulting in an enhanced diffused reflectance of the reflected rays. The clusters grow in size upon treatment with acid which cannot be removed by subsequent treatments with hypochlorite and sodium hydroxide either in



Fig. 5 — SEM images of (a) untreated wool fibre [showing deposits of particulate matter on the surface of scales] and (b) alkali trated fobre [showing deposited particles sticking onto the fibre surface, some morphological changes on the surface are observed]

isolation or in tandem. These particles could be the deposits of inorganic salts, which, in all probability, are not the integral parts of the fibre. As expected, the fibre obtained by treatment with all the input chemicals under optimized conditions shows roughening of surface with vertical scratch marks, indicating removal of the lipid layer. The open ends of scale edges are rendered somewhat blunt. The cluster of particles persists to remain on the surface and their chemical identity still remains to be deciphered.

## 4 Conclusion

A 3-factor 3-level Box-Behnken DOE was employed to examine the effect of selected factors on the responses. Multiple regression analysis of experimental data using statistical software DX7 furnished three second degree polynomial equations for WI, BI and WAL. The forecast for responses computed according to these models is found to be in good agreement with the experimental data. The forecast indicated that the optimum set of conditions is defined by  $X_I=10$ ,  $X_2=4.5$  and  $X_3=10$  for both WI<sub>max</sub> (67) and BI<sub>max</sub> (36.2). The predicted value of WAL under these conditions is slightly higher (41.6 mg/1000 cycles) than the predicted minimum (40.8 mg/1000 cycles) over the entire range. These values are well within the acceptable limit of WAL (70 mg/1000 cycles) for commercial carpets according to international standards. Hence, the optimized process condition may be useful in commercial chemical finishing of woolen handmade carpets.

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