

Effect of Dyeing Techniques on Strength Abrasion of Reactive-Dyed DMDHEU-Finished Cotton Sheeting

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Cotton sheeting (200 g/m²) samples dyed with monochlorotriazinyl dye, CI Reactive Red 31, by pad-dry-cure (PDC) and pad-dry-steam (PDS) techniques were subjected to catalyst and DMDHEU-finishing treatments. Dyeing by PDC technique, wherein urea and alkali are used along with the dye at high temperature, led to the formation of cellulose carbamate, which, in turn, resulted in higher water imbibition and better retention of strength abrasion properties on PDC dyed DMDHEU-finished fabrics, especially at wash and wear level of resilience, than on the corresponding fabrics dyed by the PDS technique. However, cellulose carbamate did not help in imparting any additional resilience properties either at the dyed or dyed DMDHEU-finished stage.

Keywords: Cotton sheeting, Dimethyloldihydroxyethyleneurea, Dyeing, Finishing, Monochlorotriazinyl dye, Strength abrasion

1 Introduction

Resin finishing of cotton textiles improves the resilience properties markedly with concurrent losses in the strength abrasion properties. To minimize this loss, much work¹⁻⁴ has been done to assess the role of pretreatments, such as scouring, bleaching, mercerizing and their varying sequences, on strength abrasion retention of resin-finished fabrics. However, most of these studies are on undyed fabrics even though, at the plant level, 70-80% of the fabric produced is either dyed or printed.

The use of reactive dyes, especially those based on chlorotriazine reactive system, has gained wide popularity for dyeing cotton because of their high reactivity and tinctorial power, comparative cheapness and good fastness characteristics. Resin finishing of these dyed fabrics could lead to some changes in tone, depth and fastness characteristics in addition to the inevitable loss in strength abrasion properties. Braun and Rieker⁵ reported the use of factorial trial procedure and regression analysis for optimizing resin finishing on reactive dyed cotton fabric. Their main aim was to optimize the resin finishing process to have a minimum colour shift on the dyed finished fabric. Recently, the effect of resin finishing and soaping treatments on fastness and colour shift of sheeting dyed with two monochlorotriazinyl (MCT) dyes by pad-dry-cure (PDC) and pad-dry-steam (PDS) techniques has been reported elsewhere⁶. In the present study, evaluation of the influence of these

dyeing techniques on resilience and strength abrasion retention of sheeting dyed with CI Reactive Red 31 and further finished with dimethyloldihydroxyethyleneurea (DMDHEU) is attempted.

2 Materials and Methods

2.1 Materials

Mill-bleached mercerized sheeting (grey particulars: count, 17s × 9s; reeds × picks per inch, 81 × 31 and weight, 202 g/m²) having barium activity No. 140, drop absorbancy < 1 s and cuprammonium fluidity of 3.4 poise⁻¹ was used for dyeing and finishing experiments. The chemical processing of this fabric was carried out in the mill and the results are reported elsewhere⁷. One commercially available MCT dye (CI Reactive Red 31) was used for dyeing experiments. Dimethyloldihydroxyethyleneurea (DMDHEU) having 44% solid content was used for finishing experiments. All other chemicals used were of laboratory reagent grade.

2.2 Methods

2.2.1 Dyeing

For dyeing, the fabric was padded at 85% wet pickup with a solution containing 20 g/l dye, 150 g/l urea, 16 g/l sodium carbonate and 10 g/l resist salt (sodium salt of metanitrobenzene sulphonic acid, a mild oxidizing agent) for PDC technique and 20 g/l urea, 15 g/l sodium carbonate and 10 g/l resist salt for PDS technique. The padded fa-

abric was dried at 80°C for 5 min and the fixation of the dye was carried out by curing in a hot air oven at 150°C for 5 min in the case of PDC technique and by steaming in a Benz continuous steaming range with saturated steam at 102-105°C for 4 min in the case of PDS technique. The dyed fabrics were thoroughly washed with cold and hot water (60°C) and then again with hot water (85°C) for 20 min followed by rinsing with cold water and then line-dried.

2.2.2 DMDHEU Finishing

Dyed fabrics were finished with 40, 80, 120 and 160 g/l DMDHEU by using magnesium chloride hexahydrate as a catalyst. For 40 and 80 g/l DMDHEU, 15 g/l catalyst and for 120 and 160 g/l DMDHEU, 20 g/l catalyst was used. The dyed fabrics were padded with the finishing solution at 80% wet pickup, dried at 80°C for 5 min and then cured at 150°C for 5 min. Catalyst control treatment was also carried out by using a solution of 20 g/l magnesium chloride hexahydrate. The cured fabrics were washed thoroughly with cold and hot water and soaped (5 g/l soap and 2 g/l sodium carbonate) at 60°C for 30 min followed by thorough hot and cold water rinses and finally line-dried.

2.2.3 Test Methods

Water imbibition values were determined by centrifuging the water-soaked sample [15 min each in hot (80°C) and cold water (25°C)] at 7500 rpm for 20 min on a Remi research centrifuge and by taking the weights before and after bone-drying. The nitrogen content of the samples was determined by Kjeldahl's method. Durable press (DP) rating was assessed after machine wash (41 ± 2°C for 12 min) and line-drying (AATCC

No. 124-1982). Conditioned and wet wrinkle recovery angles (WRA) were determined by using a Monsanto tester (AATCC Test Method No. 66-1976). Tensile strength and elongation at break were determined by the ravelled strip method as per ASTM D-1682-54. Tear strength was determined on the Elemendorf tear strength tester (ASTM D-1424-67). Flex abrasion was determined by the ravelled strip method on a Stoll's universal wear tester using 1 lb head load and 4 lb tension load (ASTM D-1175-71). Accelerator weight loss was determined by using a Carborundum universal liner No. 320 as liner at a rotor speed of 2000 rpm for 2 min (AATCC Test Method No. 74-1974). IR absorption spectra were recorded on a Perkin-Elmer double beam spectrophotometer (Type 377) by using the KBr disc technique. For scanning electron microscopic (SEM) examination, the samples were mounted on a specimen holder and coated with gold platinum in a sputter. The coated samples were then examined in Hitachi scanning electron microscope (Model No. 510) at suitable accelerating voltage.

3 Results and Discussion

3.1 Nitrogen Fixed and Water Imbibition

The value for nitrogen fixed (Table 1) is higher for sheeting dyed with CI Reactive Red 31 by PDC technique (0.32%) than for the sheeting dyed by PDS technique (0.10%). This could be attributed to the use of a high amount of urea (150 g/l) and the curing conditions (150°C/5 min) employed. It is reported⁸ that in the presence of alkali, urea combines at high temperature with cellulose to form cellulose carbamate. This is evident from Fig. 1 wherein differential IR spectra of bleached-mercerized sheeting against (i) sheeting

Table 1 - Data on Resilience and Related Properties of Sheeting Dyed with 2% CI Reactive Red 31 by PDC and PDS Techniques and Finished with DMDHEU

Sl. No.	Sample	Finishing		N ₂ (% owf)	Water imbibition %	DP rating	WRA (W+F) ^a		Total resilience (R) score
		DMDHEU g/l	MgCl ₂ ·6H ₂ O g/l				Conditioned	Wet	
				PDC /PDS	PDC/PDS	PDC/PDS	PDC/PDS	PDC/PDS	PDC/PDS
1	Dyed control (C)	—	—	0.32/0.10	33.4/33.1	2.0/2.1	196/199	180/186	15.3/19.3
2	Dyed and catalyst treated (Fo)	—	20	—/0.09	33.2/32.9	2.1/2.0	190/200	184/196	15.6/22.0
3	Dyed and finished (F1)	40	15	0.48/0.26	25.7/24.8	2.7/2.9	225/224	211/206	42.3/42.4
4	Dyed and finished (F2)	80	15	0.69/0.46	23.0/22.3	3.0/3.2	245/242	227/224	57.4/57.4
5	Dyed and finished (F3)	120	20	0.89/0.67	20.9/18.9	3.2/3.4	257/258	246/242	69.7/70.7
6	Dyed and finished (F4)	160	20	1.12/0.92	20.2/18.2	3.6/3.7	272/268	248/248	79.4/79.0

PDC - Pad-dry-cure; PDS - Pad-dry-steam; owf - on the weight of fabric.

dyed with CI Reactive Red 31 (20 g/l) and alkali (15 g/l Na_2CO_3) without urea by PDC technique, (ii) sheeting dyed with CI Reactive Red 31 (20 g/l), alkali (15 g/l Na_2CO_3) and urea (150 g/l) by PDC technique, and (iii) bleached sheeting treated with urea (150 g/l) and alkali (15 g/l Na_2CO_3) by PDC technique are shown. The figure shows that the development of the band at 1715 cm^{-1} , representing the carbamate radical, occurs in spectra B and C wherein urea is used in the treatment baths. This is also reflected by the higher water imbibition values (Table 1) for PDC-dyed samples than for PDS-dyed samples, especially after finishing with DMDHEU. Similar higher values for

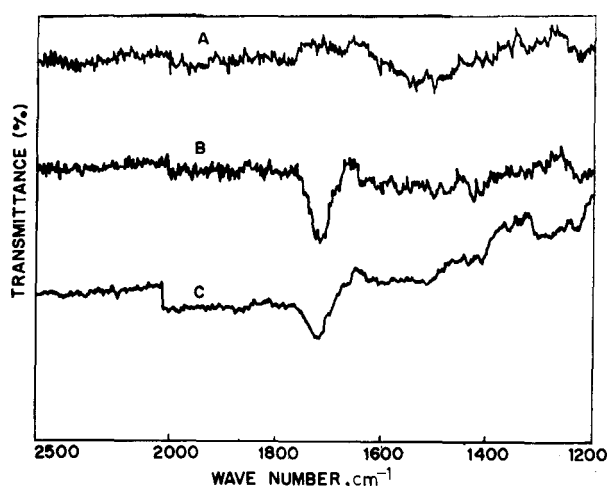


Fig. 1 – Differential IR spectra of bleached-mercerized sheeting against (i) sheeting dyed with CI Reactive Red 31 (20 g/l) and alkali (15 g/l Na_2CO_3) without urea by PDC technique – A, (ii) sheeting dyed with CI Reactive Red 31 (20 g/l), alkali (15 g/l Na_2CO_3) and urea (150 g/l) in the dye bath – B, and (iii) bleached sheeting treated with urea (150 g/l) and alkali (15 g/l Na_2CO_3) by PDC technique – C

moisture regain have been reported⁹ for cotton treated with urea.

3.2 Resilience Properties

Finishing of dyed fabrics with 40-160 g/l DMDHEU improves their resilience properties (Table 1). The properties are improved to the wash and wear level on finishing with 80 g/l DMDHEU, and on finishing with 160 g/l DMDHEU, durable press (DP) level of resilience is attained. The improvements in DP rating and conditioned and wet WRA, observed on all the dyed finished samples which were dyed by either PDC or PDS technique, are comparable. This is rather surprising since the PDC-dyed samples possess higher nitrogen fixed, due to the formation of cellulose carbamate, than the samples dyed by PDS technique and this could not contribute to any significant additional improvement in their resilience properties.

3.3 Strength Abrasion Properties

The strength abrasion retention values of sheeting dyed with CI Reactive Red 31 by PDC and PDS techniques and subjected to finishing treatments show progressive loss with the increasing amount of DMDHEU applied (Table 2). Even the catalyst control treatment leads to some loss in strength and a significant reduction in flex abrasion. The reductions in tensile and tear strengths of DMDHEU-finished samples are in the expected range of 40-50% while those in flex abrasion are quite high. There is a clear difference in the retention values of finished samples dyed by PDC and PDS techniques. Thus, in almost all the tests, except the accelerator weight loss test, PDC-dyed samples show higher retention than PDS-dyed samples. In accelerator weight loss test, may be

Table 2 – Data on Strength Abrasion Properties of Sheetting Dyed with 2% CI Reactive Red 31 by PDC and PDS Techniques and Finished with DMDHEU

Sl. No.	Sample	Tensile strength (kg/2.5 cm) (W+F) % Rtn.	Elongation at break (%) (W+F) % Rtn.	Tear strength (g) (W+F) % Rtn.	Flex abrasion (cycle) (W+F) % Rtn.	Accelerator wt loss %	Total strength abrasion (S) score
		PDC/PDS	PDC/PDS	PDC/PDS	PDC/PDS	PDC/PDS	PDC/PDS
1	Dyed control (C)	100/100 (69.6/68.7) ^a	100/100 (27.0)/(30.0)	100/100 (10131/10448)	100/100 (1558/1505)	(4.6/3.9)	145.4/146.1
2	Dyed and catalyst treated (Fo)	93/83	91/96	74/61	54/58	(6.7/5.7)	110.8/102.3
3	Dyed and finished (F1)	89/86	87/89	76/64	69/48	(8.9/8.1)	109.3/98.3
4	Dyed and finished (F2)	79/71	80/80	54/54	62/45	(14.3/12.9)	89.2/77.6
5	Dyed and finished (F3)	71/67	73/70	50/46	45/28	(15.3/15.7)	70.2/61.9
6	Dyed and finished (F4)	57/65	63/72	47/38	26/18	(17.4/16.9)	53.3/53.8

^aValues within the parentheses are the actual (W + F) values obtained for the controls.

the double curing operations undergone by the PDC-dyed DMDHEU-finished samples would have contributed to greater embrittlement of the surface fibres, which would have brought about the greater weight loss on these samples during the high-speed abrasive action occurring during the accelerotor weight loss test.

3.4 Performance Profile Chart

To get a clear perspective on the influence of dyeing techniques on the strength abrasion retention of dyed DMDHEU-finished fabrics, a performance profile chart (Fig. 2), similar to the one used in an earlier study¹⁰, was used. It included three resilience properties, viz. conditioned WRA, wet WRA and DP rating, and five strength abrasion properties, viz. tensile strength, elongation at break, tear strength, flex abrasion and accelerotor weight loss. Using this chart, scores for resilience and strength abrasion properties were read from the common rating scale. Scores for resilience properties were added up to give the total resilience (R) score. Similarly, the total strength-abrasion (S) score was obtained by adding up the individual score for each strength abrasion test.

3.5 Overall Performance Profile

To evaluate the role of PDC and PDS dyeing techniques on the performance of dyed

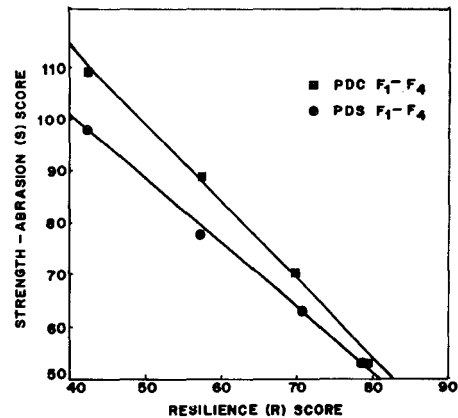


Fig. 3—Performance evaluation of dyeing technique (PDC vs PDS) on dyed DMDHEU-finished sheeting

Table 3—Strength-Abrasion (S) Scores at Wash and Wear and Durable Press Levels of Resilience for MCT Dyed Sheetting Finished with DMDHEU

Sample	Strength-abrasion 'S' score at resilience (R) score of		Total 'S' score	Slope Tan ϕ
	R = 50 Wash and wear	R = 80 Durable press		
PDC F1-F4	99	54	153	1.50
PDS F1-F4	88.3	52	140.3	1.21

RESILIENCE				STRENGTH-ABRASION % RETENTION (W + F)				
RATING SCALE	DP RATING	WRA (W + F) COND.	WET	TENSILE STRENGTH	ELONGATION AT BREAK	TEAR STRENGTH	FLEX ABRASION	ACCELERATOR WEIGHT
40	5.0	300	300	100	100	100	100	100
30	4.0	270	270	80	80	80	100	100
20	3.0	240	240	60	100	60	66.7	100
10	2.0	210	210	40	60	40	33.3	90
0	1.0	180	180	20	20	20	0	80

Fig. 2—Performance profile chart for evaluating resilience and strength-abrasion properties of dyed sheeting finished with DMDHEU

DMDHEU-finished sheeting, total 'S' scores were plotted against total 'R' scores obtained for all the finished samples. The profiles thus obtained, i.e. overall performance profile of the PDC and PDS techniques, are shown in Fig. 3. From these profiles, the 'S' scores obtained at 'R' score of 50 and 80 and their slope values were read. These values along with their added up value and slopes are given in Table 3. The 'R' score of 50 represents conditioned WRA of 230°, wet WRA of 220° and DP rating of 3.0, i.e. near wash and wear level of resilience. The 'R' score of 80 represents conditioned WRA of 270°, wet WRA of 255° and DP rating of 3.5, i.e. near durable press level of resilience. Fig. 3 shows that at the initial stages of resilience improvement, 'S' scores for PDC technique are significantly higher than those for PDS technique; however, the slope, i.e. the rate of fall of 'S' score per unit increase in 'R' score, is higher for PDC than for PDS. Hence, the profiles for both the techniques meet at an 'R' score of 80, i.e. near durable press level of resilience. At wash and wear and near about levels of resilience, dyeing of cotton sheeting with MCT dye by PDC technique is definitely more advantageous than by PDS technique. At durable press level, however, this

advantage no longer exists. The higher retentions in almost all the tests except the accelerator weight loss contribute to this behaviour. As discussed above, the cellulose carbamate formed on PDC-dyed samples may prevent the excessive collapse of the cellulose fibre during the curing operation and also minimize the thermohydrolytic damage due to the latent acid catalyst used for DMDHEU finishing. This is evident from the fact that the total 'S' score of catalyst-treated PDC-dy-

ed sample is 110.8 as against 102.3 for the PDS-dyed sample (Table 2).

3.6 Scanning Electron Microscopic (SEM) Studies

SEM micrographs of fabrics dyed with MCT dye by the PDC and PDS techniques as such and after finishing with DMDHEU were taken at a number of places and the most representative ones are shown in Fig. 4. Dyeing with MCT dye either by PDC (Fig. 4c) or PDS (Fig. 4b) tech-

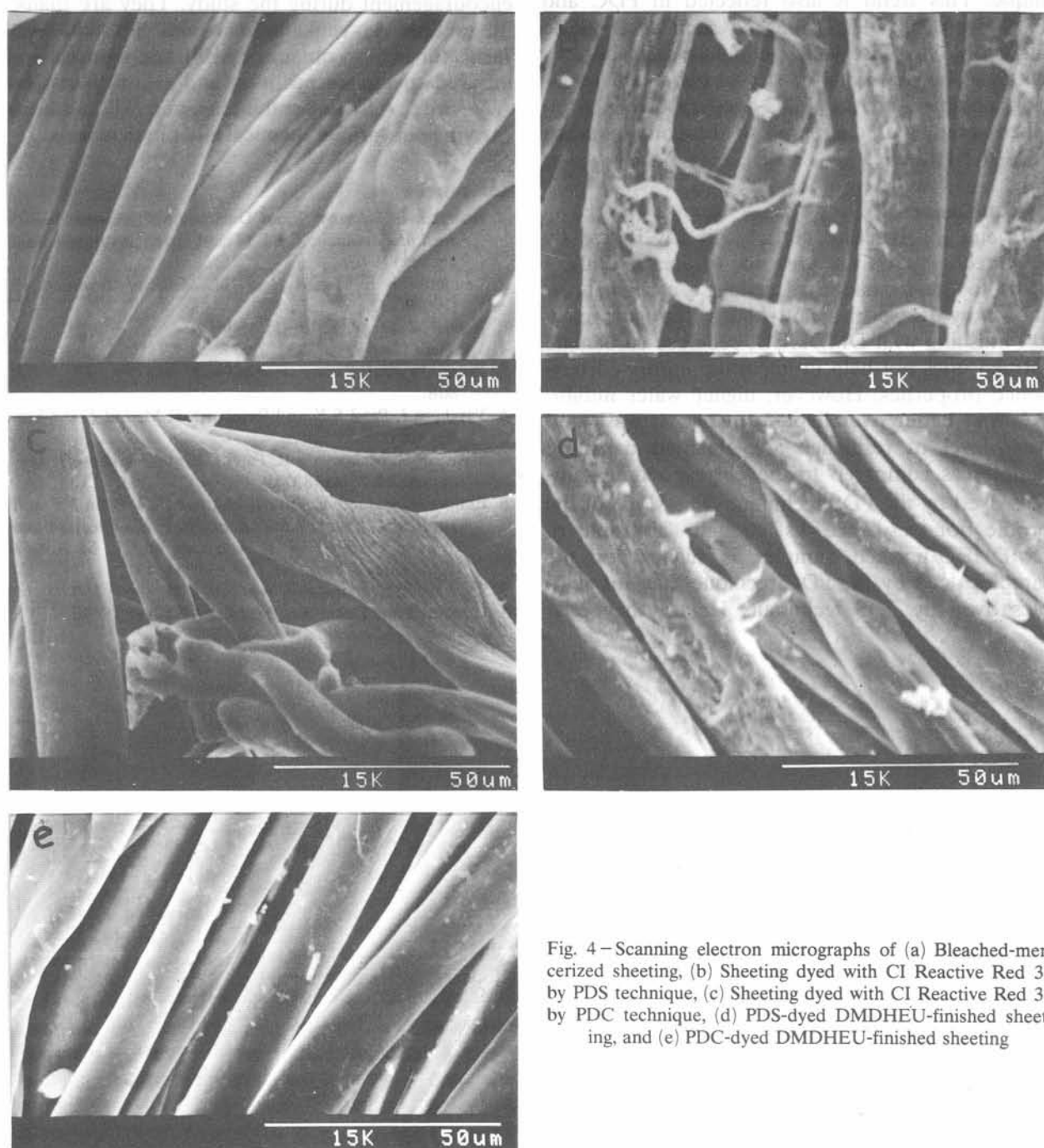


Fig. 4—Scanning electron micrographs of (a) Bleached-mercerized sheeting, (b) Sheetting dyed with CI Reactive Red 31 by PDS technique, (c) Sheetting dyed with CI Reactive Red 31 by PDC technique, (d) PDS-dyed DMDHEU-finished sheeting, and (e) PDC-dyed DMDHEU-finished sheeting

nique entailed some surface damage and more so on fabrics dyed by PDS technique. The difference between the two techniques, i.e. using 150 g/l urea at 150°C for 5 min for PDC technique as against 20 g/l urea at 102°C (saturated steam) for PDS technique, could have led to the formation of a protective coating by the melted urea on the fibre surface as well as the formation of cellulose carbamate during dyeing by the former technique and these, in turn, would have helped to reduce the fibre damage on samples dyed by this technique. This trend is also reflected in PDC and PDS dyed DMDHEU-finished samples (Figs 4e and 4d). This observation could be corroborated with the higher order of strength-abrasion retention obtained on the PDC-dyed DMDHEU-finished samples as compared to the corresponding PDS-dyed samples.

4 Conclusions

Dyeing of sheeting fabric with MCT dye by PDC technique in the presence of urea and alkali leads to the formation of cellulose carbamate as shown by IR spectra and higher nitrogen fixed. On finishing with DMDHEU, cellulose carbamate formed does not help in imparting improved resilience properties. However, higher water imbibition and better overall performance at wash and wear level of resilience are obtained on these samples as compared to the corresponding samples dyed by the PDS technique. The slope of the profile for resilience vs strength abrasion of DMDHEU-finished samples is higher for the samples dyed by the PDC technique than for samples dyed by PDS technique. Hence, they con-

verge at durable press level of resilience, and at this level, both the techniques give comparable performance.

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