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Dyeing of Environmentaly Friendly Pretreated Cotton Fabric

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1. Introduction

Raw cotton fibres have to go through several chemical processes to obtain properties suitable for further dyeing and use. With scouring, non-cellulose substances (wax, pectin, proteins, hemicelluloses…) that surround the fibre cellulose core are removed, and as a result, fibres become hydrophilic. Conventional scouring processes of cotton are conducted at temperatures up to 130 °C in a very alkaline medium (pH 10–12) with sodium hydroxide. Since a non-specific reagent is used in the treatment, it attacks impurities but it also causes damages to the cellulose portion of the fibres. Several auxiliary agents, such as wetting agents, emulsifiers and sequestering agents, which improve the efficiency of scouring and reduce the damage of fibres, are also added to the scouring bath.

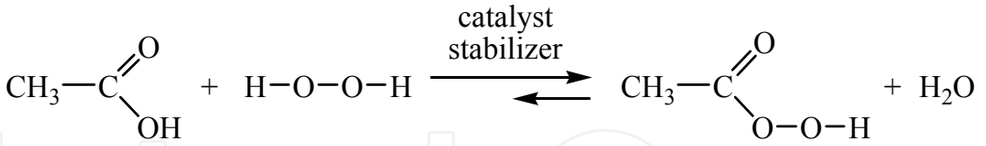
Scouring is regularly followed by a bleaching process, which removes the natural pigments of cotton fibres. Cellulose fibres are most frequently bleached with hydrogen peroxide resulting in high and uniform degrees of whiteness. The water absorbency also increases, however, during the decomposition of hydrogen peroxide, radicals that can damage the fibres are formed. For this reason, organic and inorganic stabilizers and sequestering agents are added to the treatment bath.

Hydrogen peroxide is not ecologically disputable. The large amount of water used to rinse and neutralize the alkaline scoured and peroxide bleached textiles is ecologically disputable. Namely, the bleaching process is conducted in an alkaline bath at pH 10 to 12 and at temperatures up to 120 °C. Due to high working temperature, a large amount of energy is consumed. Auxiliary chemicals added into the bath increase the TOC and COD values of effluents. Upon neutralization of highly alkaline waste baths, large amounts of salts are produced. Consequently, the textile industry is considered one of the biggest water, energy and chemical consumers (Alaton et al., 2006; Warke & Chandrate, 2003).

To comply with more and more rigorous environmental regulations and to save water and energy, biotechnology and several types of enzymes have entered the textile sector. Many review (Jayani et al., 2005; Galante et al. 2003) and scientific (Gummandi & Panda, 2003; Buchert et al., 2000) papers describe the use of different enzymes for textile finishing. Pectinases are an efficient alternative to sodium hydroxide in the removal of non-cellulose substances from the cotton fibre surface (Preša & Tavčer, 2008a; 2008b). This process occurs at moderate temperatures in a slightly acidic (Calafell & Garriga, 2004; Li & Hardin, 1998;

Sawada et al., 1998, Buschle-Diller et al., 1998) or alkaline (Etters, 1999; Durden et al., 2001; Lenting & Warmoeskerken, 2004) medium and is dependent on the type of pectinase. Pectin acts as a sort of cement or matrix that stabilizes the primary cell wall of cotton fibres. Pectinases decompose insoluble pectin into smaller particles thereby destabilizing the structure in the outer layers. The weakened outer layers can be removed in a subsequent wash process to such extent that following finishing processes and dyeing can be easily preformed (Etters, 1999; Losconzi et al., 2004; Cavaco-Paulo & Gubitz, 2003). It has been established that agitation of the treatment bath is very important for pectinase to function efficiently (Anis & Eren, 2002; Li & Hardin, 1998) and that selected sequestering agents can improve their effectiveness (Losonczi, 2005). Since enzymes act selectively, no damage to fibres occur during treatment. Also, it has been observed that after enzymatic treatment, baths are less polluted than baths after scouring with sodium hydroxide (Losonczi et al., 2005; Choe et al., 2004).

Bleaching with peracetic acid (PAA) is an alternative to bleaching with hydrogen peroxide (Tavčer, 2010). It is a powerful oxidizing agent (redox potential: 1.81 eV) (Preša & Tavčer, 2009) with excellent antimicrobial and bleaching properties. It is efficient at low concentrations, temperatures and in neutral to slightly alkaline medium. Its products of decomposition are biologically degradable. In the past, it was prepared in situ from acetic acid anhydride and hydrogen peroxide (Rucker, 1989; Wurster, 1992). However, the risk of explosion during the synthesis reaction prevented affirmation of peracetic acid as a bleaching agent in industry. In recent years, peracetic acid has become interesting (Hickman, 2002; Križman et al., 2005; Križman Lavrič et al., 2007). Several commercial products are available as balanced mixtures of peracetic acid, acetic acid and hydrogen peroxide (Equation 1). They are stabilized with a minimum amount of sequestering agent. Today, peracetic acid products available in the market are safe, simple to use, and price-effective. Equation 2 shows the reaction that occurs when peracetic acid is used for bleaching.



(1)

$$CH_3COOOH + \text{impurities} \longrightarrow CH_3COOH + \text{oxidised impurities} \quad (2)$$

Both processes, scouring with pectinases and bleaching with peracetic acid, are conducted at temperatures of 50–60 °C for 40–60 minutes and pH 5–8. If both processes could be combined into one process, huge amounts of water, energy, time, and auxiliary agents can be saved. In a recent study (Preša & Tavčer, 2008b), it was confirmed using a viscosimetric method that pectinases retain their activity in the presence of peracetic acid and that combined processes are feasible.

The objective of our work was to compare the properties of enzymatically-scoured and peracetic acid-bleached cotton fabrics treated by two-bath and one-bath scouring/bleaching methods, with respect to conventionally-treated fabrics (alkaline scoured and bleached with hydrogen peroxide) with emphasis on their degree of whiteness and dyeability.

Dyeability of differently pretreated fabrics is very important for industry and was studied from many aspects before. The dyeing behaviour of cotton fabrics treated with different enzymes by using reactive, cationic and acid dyes showed that cationic and acid dyes were more sensitive to the enzymatic treatment used as reactive dyes (Canal et al., 2004). When dyed with bireactive dyes the dyeing showed excellent evenness and their capacity to cover differences in whiteness arising from different pretreatment processes was significant. On the contrary, the dyeings with acid and cationic dyes revealed, with large differences in dye exhaustions, that each enzymatic system produces different ionisable residues on the primary wall of the cotton fibre (Canal et al., 2004). The importance of charges and functional availability on the fibre surface after different bioscouring treatments for their dyeing behaviour was also exposed by Calafell and co-workers (Calafell et al., 2005).
The ability of reactive dyes to cover the differences in whiteness from different bioscouring and bleaching processes when dyed in dark shades was confirmed by several researchers (Fakin et al., 2008, Loszonci et al., 2004).
Our research will upgrade the above results with reactive dyeing of differently scoured and differently bleached cotton fibres in medium and light shades and estimate the processes from the ecological point of view.

2 Experimental

2.1 Materials

Desized cotton fabric, 100 g/m^2, was obtained from Tekstina, Slovenia. Acid pectinase Forylase KL (AP) was supplied from Cognis, Germany, and alkaline pectinase Bioprep 3000L (BP) from Novozymes, Denmark. Cotoblanc HTD-N (anionic wetting and dispersing agent, alkansulphonate with chelator) was supplied from CHT, Germany. H_2O_2 35% (HP) and peracetic acid (PAA) as a 15% equilibrium solution in the commercial bleaching agent Persan S15 were obtained from Belinka, Slovenia. Foryl JA (nonionic wetting agent) and Locanit S (ionic-nonionic dispersing agent) were obtained from Cognis, Germany and Lawotan RWS (nonionic wetting agent) was obtained from CHT, Germany. Sodium hydroxide was supplied from Šampionka, Slovenia, and acetic acid and sodium carbonate were supplied from Riedel-de Haen, Germany.

2.2 Treatment methods

The cotton fabric was scoured according to three different procedures using sodium hydroxide, acid pectinase or alkaline pectinase. The scoured fabrics were bleached with two bleaching agents: hydrogen peroxide and Persan S15. The abbreviation of processes and treatment conditions are displayed in Table 1. Enzymatic scouring and one-step treatments were performed 60 minutes at 55 °C, than the temperature of the bath was increased to 80 °C for 10 minutes to deactivate the enzymes. To activate peracetic acid in AP/PAA treatment, the pH was adjusted to 8 after 30 minutes. Demineralised water was used in all processes. The treatments were performed on the Jet JFL apparatus manufactured by Werner Mathis AG loaded with 50 g of fabric at a liquor ratio of 1:20. After all treatments, the bath was discharged and the jet was filled sequentially with fresh water heated to 80 °C, 60 °C and 25 °C to rinse the fabric. After alkaline scouring and peroxide bleaching, the fabrics were additionally neutralized with a neutralizing bath containing acetic acid and rinsed with cold water.

Process	Conditions
AS - Alkaline scouring	3 g/l NaOH, 2 g/l Cotoblanc HTD-N, 95 °C, 40 minutes
AP - Scouring with acid pectinases	5 ml/l Forylase KL, 0.75 ml/l Foryl JA, 2 ml/l Locanit S and CH_3COOH to pH 5.5, 55 °C, 40 min.
BP - Scouring with alkaline pectinases	0.05 % Bioprep, 0.5 g/l Lawotan RWS, Na_2CO_3 to pH 8, 55 °C, 40 min.
HP - bleaching with hydrogen peroxide	7 g/l H_2O_2 35%, 1 g/l Cottoblanc HTD-N, 4 g/l NaOH 100%, 95 °C, 40 min
PAA - bleaching with peracetic acid	15 ml/l Persan S15, 55 ml/l Na_2CO_3 0.5 M, 0.1g/l Lawotan RWS, pH 8, 55 °C, 40 min
AP+PAA - one step scouring with acid pectinase and bleaching with peracetic acid	5 ml /l Forylase KL, 0.75 ml/l Foryl JA, 2 ml/l LocanitS, 15 ml/l Persan S15
BP+PAA - one step scouring with alkaline pectinase and bleaching with peracetic acid.	0.05 % Bioprep 3000L, 0.1 mL/L Lawotan RWS, 15 mL/L Persan S15, pH 8 with NaOH, 55 °C, 40 min.

Table 1. The abbreviation of processes and treatment conditions

2.3 Dyeing procedure

Dyeing the pre-treated fabrics was performed at 60 °C for 90 minutes with 0.5% and 2% Cibacron rot F-B. 30 g/L Na_2SO_4 and 8 g/L Na_2CO_3 was used for pale shade and 50 g/L Na_2SO_4 and 11 g/L Na_2CO_3 for medium shade. The weight of the dyed samples was 5 g. Finally, the cotton was soaped, washed and air dried. Dyeing was performed in closed beakers on an Launder-Ometer (Atlas).

2.4 Analytical methods

Prior to the measurements, fabrics were conditioned 24 hours at 20 °C and 65% relative humidity. The degree of whiteness and the colour values were measured on the Spectraflash SF600 Plus using the CIE method according to EN ISO 105-J02:1997(E) standard and EN ISO 105-J01:1997(E), respectively. Weight loss due to the pretreatments was determined by weighing the fabric samples before and after pretreatment and was expressed in percent. Water absorbency was measured according to DIN 53 924 (velocity of soaking water of textile fabrics, method for determining the rising height). Measurements of tenacity at maximum load were performed on Instron Tensile Tester Model 5567. The mean degree of polymerization (DP) was determined with the viscosimetric method in cuoxam.
Samples of remaining bleaching and scouring baths were collected after all treatments. Their ecological parameters (pH, total organic carbon (TOC), chemical oxygen demand (COD), biological oxygen demand (BOD_5)) were measured, the consumption of water and energy was estimated (Preša, 2007).

3. Results and discussion

3.1 Whiteness

The achieved degrees of whiteness (W) and tint values (TV) are presented in Table 2. The desized (untreated) sample (D) had a degree of whiteness of 11.1. After alkaline scouring,

the fibres swelled, became smoother and clean of non-cellulose impurities and the degree of whiteness increased to 19.5. However, after scouring with acidic and alkaline pectinases, both samples had lower degrees of whiteness relative to the desized sample, i.e. the sample treated with acidic pectinases (AP) had a whiteness degree of 8.2, and the sample treated with alkaline pectinases (BP) had a whiteness degree of 8.4. Negative TV values demonstrated that all scoured and desized samples had a red shade. After alkaline scouring, the red shade decreased, whereas after both bioscourings, the red shade increased.

The degree of whiteness of all scoured samples increased significantly after hydrogen peroxide bleaching. The differences in whiteness from previous scouring disappeared. Alkaline and bioscoured samples have a whiteness values above 84 and the red shade almost disappeared.

With peracetic acid bleaching, a high degree of whiteness was not achieved and the differences in whiteness from the previous scouring remained visible. The sample, which was alkaline scoured prior to bleaching (AS+PAA), had the highest degree of whiteness (72.7), whereas both bioscoured samples had lower degrees of whiteness (57.7 AP+PAA and 57.3 AP+PAA). The red shade was visible on all peracetic acid bleached samples and was more on bioscoured than on alkaline scoured fabrics, which suggests that bleaching with peracetic acid is not as effective as bleaching with hydrogen peroxide. This occurs because bleaching with peracetic acid proceeds at a low temperature and pH, where the impurities remaining after scouring could not be fully oxidised. Bioscoured fibres contained also more waxes and other impurities that hindered the successful oxidation with peracetic acid at mild conditions. Bleaching the alkaline scoured fabrics with peracetic acid is more effective since the impurities were removed from cotton fibres to a higher extent in the previous process and the pigments within fibres were more exposed to the oxidant's influence. This is confirmed by comparing data of the mass loss during treatments (Table 3).

The degrees of whiteness after a one-bath treatment (68.7 AP/PAA and 69.6 BP/PAA) were higher than those after two-bath bioscouring and bleaching with peracetic acid and close to the whiteness achieved after alkaline scouring and bleaching with peracetic acid. This can be explained by the fact that at the bleaching conditions of peracetic acid (55 °C, pH 8), hydrogen peroxide, which was present in the balanced mixture with peracetic acid, was not consumed, whereas at temperature 80 °C, which was the finite temperature of the one-bath process, hydrogen peroxide was activated, which further increased the degree of whiteness.

	Scouring		HP		PAA		Scouring/PAA	
	W	TV	W	TV	W	TV	W	TV
D	11.1	-9.1	-	-	-	-	-	-
AS	19.5	-7.6	84.12	-0.45	72.7	-0.95	-	-
AP	8.2	-9.73	85.59	-0.37	57.7	-2.05	68.7	-1.3
BP	8.4	-9.6	85.07	-0.36	57.3	-1.9	69.6	-1.2

Table 2. Whiteness (W) and tint values (TV).

3.2 Fabric properties

Table 3 represents the loss of weight, rising height in warp direction, tenacity at maximum load and degree of polymerization (DP) of differently pretreated cotton fabric samples.

The **loss of weight** demonstrates that scouring with NaOH is more intensive and removes more incrusts than enzymatic scouring. The loss of mass after alkaline scouring was ca. 1.3% and after enzymatic scouring, was less than 1%. During hydrogen peroxide bleaching, the loss of mass was greater in those samples, where the loss of mass was lower during scouring; alkaline scoured only 0.25%, acid pectinases scoured 1.2% and alkaline pectinases scoured 0.73%. This suggests that hydrogen peroxide bleaching removed a large portion of compounds, which remained on fibres after scouring. The total mass loss after scouring and hydrogen peroxide bleaching was similar for all samples.

Peracetic acid bleaching also removed a certain part of the noncellulosic substances, which remained on fibres after scouring, but the quantity was lower relative to hydrogen peroxide bleaching. Bleaching with peracetic acid did not equalize the differences in the loss of mass, which is in agreement with the whiteness results. We can conclude that high temperature and high pH are conditions that contribute decisively to the removal of non-cellulosic impurities. Specifically, waxes cannot be removed completely when all processes are conducted at low temperatures and neutral pH, as is the case for bioscouring and peracetic acid bleaching.

	Weight loss (%)	Rising height (cm)	Tenacity (cN/tex)	DP
D		0	18.47	2482
AS	1.27	2.9	18.45	2432
AP	0.30	2.7	16.96	2451
BP	0.89	2.5	17.95	2385
AS+HP	1.52	3.0	16.65	1774
AP+HP	1.51	3.0	17.12	1947
BP+HP	1.62	2.8	16.83	2004
AS+PAA	1.30	2.8	16.94	2278
AP+PAA	0.65	2.9	18.12	2318
BP+PAA	0.95	2.9	13.75	2399
AP/PAA	0.40	2.7	16.94	2438
BP/PAA	0.60	2.8	18.84	2300

Table 3. The loss of mass, rising height in warp direction, tenacity at maximum load, degree of polymerization (DP)

The remained substances influence on the water absorbency and consequently alkaline scoured samples had the highest absorbency. Bleaching improved the absorbency of the scoured fabrics, particularly of enzymatically scoured ones. However, the difference in rising height was so small, that all the samples could be considered absorbent.

There were no higher differences in **tenacity at maximum load** between the de-sized and differently treated samples. On the other hand, the results of **DP** demonstrate that bleaching with hydrogen peroxide decreased the degree of polymerization significantly, while other processes preserved the DP values close to the starting value. The bioscouring and bleaching with peracetic acid in a one bath or two bath processes causes no damage to fibers and this is one of the benefits of such processes.

3.3 Dyeing

Colour strengths (expressed as K/S value) and colour differences, ΔE*, of the fabric samples dyed with 0.5% and 2% of Cibacron red F-B are presented in Tables 4 and 6, respectively. The lightness values, L*, croma, C*, and hue, h, are presented in Tables 5 and 7. A standard for colour difference calculation was in each set of processes the alkaline scoured sample.

	Scouring		HP		PAA		Scouring/PAA	
	K/S	ΔE*	K/S	ΔE*	K/S	ΔE*	K/S	ΔE*
D	4.02	-	-	-	-	-	-	-
AS	4.04	-[a]	2.61	-[b]	3.89	-[c]	-	-
AP	4.04	3.74	2.73	0.26	3.89	1.26	3.78	0.58
BP	4.23	3.04	2.72	0.22	4.10	0.85	3.97	0.30

a-standard for scoured fabrics, b-standard for HP bleached samples, c-standard for PAA bleached and one-bath treated fabrics

Table 4. Colour strengths (K/S) and colour differences (ΔE*) of the fabric samples dyed with 0.5 % of Cibacron red F-B.

K/S values of all samples dyed with one concentration of dye after only scouring are similar. No significant differences were observed between the colour depths of alkaline and enzymatic pretreatments. On the contrary, colour differences between alkaline scoured and enzymatic scoured samples dyed in light shades are significant (ΔE* is above 3). This is explained by differences in whiteness after scouring, which was not covered in light shade dyeing, whereas they were covered in dark shade dyeing to a greater extent (ΔE*≈1).

The colour strengths of the samples dyed after scouring and bleaching are very similar within a set of samples, but they differ between differently bleached samples. K/S values on all hydrogen peroxide bleached samples were lower than those obtained for all peracetic acid bleached samples, and this relationship exists on pale and medium-dyed samples. We can conclude that the differences in whiteness, which were visible on samples after scouring and bleaching, remained to a certain extent after dyeing. The lighter fabrics achieved lower K/S values relative to darker fabrics when dyed under same conditions.

ΔE* values reveal that bleaching with hydrogen peroxide enabled the achievement of equal pale and medium colours on alkaline and bioscoured samples (ΔE*<0.3), while the colour differences between alkaline and bioscoured samples were higher when the samples were bleached with peracetic acid and dyed (ΔE*≈1). The colour of one-bath pretreated and dyed fabrics was close to the colour of alkaline scoured, peracetic acid bleached and dyed sample (ΔE*<0.6), which confirms that hydrogen peroxide bleaching covered the differences in

colour arising from different scouring methods, while peracetic acid bleaching preserved those differences.

The colour evenness was excellent for all samples. The standard deviation of ten colour difference values ΔE^* was below 0.06 for all dyed samples. We can conclude that all presented types of pretreatment are appropriate for further dyeing with reactive dyes, but the initial colour of the material should be considered when the dyeing recipes are prepared.

	Scouring			HP			PAA			Scouring/PAA		
	L*	C*	h [°]	L*	C*	h [°]	L*	C*	h [°]	L*	C*	h [°]
D	57.4	44.4	351.8	-	-	-	-	-	-	-	-	-
AS	58.4	46.1	351.0	59.5	48.8	350.8	59.3	47.4	350.5	-	-	-
AP	57.4	43.6	351.6	59.0	49.4	350.7	58.9	46.2	350.6	59.3	46.9	350.2
BP	57.7	43.9	351.8	59.1	49.3	350.9	59.1	46.6	350.4	59.4	47.2	350.3

Table 5. Lightness values (L*), croma (C*) and hue (h) of the fabric samples dyed with 0.5 % of Cibacron red F-B.

	Scouring		HP		PAA		Scouring/PAA	
	K/S	ΔE*	K/S	ΔE*	K/S	ΔE*	K/S	ΔE*
D	13.24	-	-	-	-	-	-	-
AS	13.42	-[a]	9.72	-[b]	13.38	-[c]	-	-
AP	14.17	0.84	9.57	0.03	13.10	1.15	13.19	0.81
BP	13.33	1.27	9.55	0.03	13.26	1.00	14.25	0.65

a-standard for scoured fabrics, b-standard for HP bleached samples, c-standard for PAA bleached and one-bath treated fabrics

Table 6. Colour strengths (K/S) and colour differences (ΔE*) of the fabric samples dyed with 2 % of Cibacron red F-B and standard deviation in brackets.

	Scouring			HP			PAA			Scouring/PAA		
	L*	C*	h [°]	L*	C*	h [°]	L*	C*	h [°]	L*	C*	h [°]
D	44.7	54.6	356.0	-	-	-	-	-	-	-	-	-
AS	45.3	56.0	356.2	46.0	58.5	357.0	46.2	57.0	356.1	-	-	-
AP	44.6	54.9	356.3	46.2	58.5	356.9	45.7	56.0	355.8	45.7	56.4	355.9
BP	44.1	55.0	356.4	46.2	58.4	356.9	45.4	56.4	356.0	45.7	56.6	356.0

Table 7. Lightness values (L*) croma (C*) and hue (h) of the fabric samples dyed with 2% of Cibacron red F-B and standard deviation in brackets.

3.4 Ecological parameters

Conventional treatment of cotton fibres was conducted in an alkaline environment: final pH at alkaline scouring and at bleaching with hydrogen peroxide was around 12.5. Such alkaline baths should be neutralized prior to drainage into the sewage system. At neutralization, salts that additionally load wastewaters are produced.

While bleaching with peracetic acid and at both combined processes, the final pH value of the bath was near 6. Since neither of these processes requires neutralization of fibres, the treatment process can be shorter and less expensive.

While scouring with pectinases and bleaching with peracetic acid, the consumption of energy required to heat the bath was also lower. Conventional processes of scouring and bleaching were performed at temperatures near the boiling point, whereas bioscouring and bleaching with peracetic acid were conducted at a temperature of 55°C. Due to the lower temperature, less energy was required.

The consumption of water and energy is the lowest at combined scouring/bleaching treatments. Consequently, at these processes arises the lowest amount of effluents and the produced wastewater is biodegradable (Preša & Tavčer, 2009).

4. Conclusions

Enzymatically and alkaline scoured cotton fabrics have similar water absorbency, tenacity at maximum load and degree of polymerization. Because of lower loss of mass and lower whiteness of enzymatically scoured fabrics noticeable differences in colour occur between differently scoured samples dyed to light hues. The colour differences are overcame at medium and pale shades.

During bleaching with hydrogen peroxide the differences in whiteness arising from previous scouring processes disappeared. All samples obtained high whiteness values. At bleaching with peracetic acid the obtained whiteness values are lower and the differences

from previous scouring processes remained visible. This causes that the colour differences between differently scoured samples, which were bleached with hydrogen peroxide prior to dyeing, are not visually perceivable, while they remained visible on samples bleached with peracetic acid at light and medium shade dyeing.

At one-bath processes of scouring/bleaching with pectinases and peracetic acid the degree of whiteness of fabrics is higher than at two-step scouring and bleaching with peracetic acid, but lower than at bleaching with hydrogen peroxide. The colour of dyed fabrics is equal to alkaline scoured and peracetic acid bleached fabric.

The different shades obtained on differently pretreated fabrics are the consequence of their different initial whiteness values. All other parameters are very similar, the exhaustion rate, the fixation rate and the fastness properties (Preša, 2007). The bioscouring and bleaching with peracetic acid, especially the one-bath processes, are suitable treatments before dyeing with reactive dyes. Lower amount of water and energy is consumed than at conventionally pretreatment and the fibres are not damaged at all. For reproducible dyeing the initial colour of the fabric should be considered when preparing the dyeing recipes.

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The coloration of fibers and fabrics through dyeing is an integral part of textile manufacturing. This book discusses in detail several emerging topics on textile dyeing. "Textile Dyeing" will serve as an excellent addition to the libraries of both the novice and expert.

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