Development of New Eco-Friendly Options for Cotton Wet Processing

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ABSTRACT: A new approach was used to search for the optimal conditions for enzymatic scouring with an alkaline pectinase and to investigate the feasibility of performed combined bioscouring and H_2O_2 bleaching and combined bioscouring and reactive dyeing of unscoured cotton fabrics. The possibility of conducting enzymatic desizing, bioscouring, and H_2O_2 bleaching of starch-sized cotton fabrics in a single bath was also examined. The results indicated that changes in the parameters of the bioscouring process, the types and concentrations of the treating bath components, and the sequence of the treatment and addition had pronounced effects on certain properties of the treated cotton substrates (e.g., the residual size, weight loss, wettability, yellowness and whiteness, and dyeability with reactive dyes). The optimal conditions for efficient bioscouring alone and in combination with H_2O_2 bleaching, reactive dyeing, and enzymatic desizing and H_2O_2 bleaching were determined. © 2004 Wiley Periodicals, Inc. J Appl Polym Sci 93: 1825–1836, 2004

Key words: additives; dyes/pigments; enzymes; fibers; processing

INTRODUCTION

The pretreatment of cotton-containing fabrics consists of one or more extractions, desizing and/or alkaline scouring, and bleaching.[1–3] An improper or inadequate pretreatment negative affects subsequent wet processes, such as dyeing, printing, and finishing.[4] In addition, current pretreatment processes, using harsh chemicals and severe conditions, are also problematic from an environmental point of view because of the high COD, BOD, pH, and salt content in textile effluents and high air pollution due to high energy consumption.[5]

Recent research activities and developments in biotechnology have led to technical feasibility for the enzymatic breakdown of natural impurities and noncellulosic materials in cotton (8–12%), such as hemicellulose, pectins, waxes, and proteins;[6–9] enzymatic desizing of starch size at high temperatures for short times;[10–13] and the smoothing and softening of cellulosic products via biopolishing.[12,14]

This work was undertaken (1) to determine the proper conditions for the enzymatic scouring of cotton fabrics, (2) to combine bioscouring and H_2O_2 bleaching and bioscouring and reactive dyeing of unscoured cotton fabrics, and (3) to study the technical feasibility of enzymatic desizing, bioscouring, and H_2O_2 bleaching of starch-sized cotton fabric in a single bath.

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EXPERIMENTAL

Materials

The specifications for the cotton fabrics used in this work are given in Table I.

Two commercial-grade enzymes, BioPrep 3000L (an alkaline pectinase with an activity of 3000 APSU/g) and Aquazym 240L (an α-amylase with an activity of 240 KNU/g), were kindly supplied by Novo Nordisk.

Concentrated Kierlon Jet B (a nonionic surfactant; BASF), Leophen M (a nonionic surfactant and defoaming agent; BASF), Sandozin MRN [a nonionic, silicone- and solvent-free wetting, washing, and cleaning agent based on poly(glycol ether) derivative; Clariant], Sandozin EH (an anionic, low-foaming agent based on an aliphatic ester; Clariant), Hostapal CV-ET [a nonionic wetting agent and detergent based on alkyl aryl poly(glycol ether); Clariant], Dekol SN (a chelating agent based on polyacrylate; BASF), peroxide stabilizer H-ET (an anionic stabilizer based on organic products; Clariant), and Hostalux CBA (an anionic optical brightener for cellulosic fibers, suitable primarily for exhaust methods; Clariant) were technical-grade.

Remazol B. Red 3BS (a heterobifunctional reactive dye), Levafix R. Blue E-FR (a homobifunctional reac-

TABLE I
Specifications of the Experimental Fabrics

Substrate	Weave	Count	Weight/area (g/m^2)	Starch-size add-on (%)	Tensile stength (K g) warp direction	Wettability (s)
I	Plain	46	156	None	87	None[a]
II	Twill	28	205	None	108	None
III	Plain	25	100	8	39	None

[a] None (>120 s).

tive dye), and Levafix B. Red E-2RN (a monofunctional reactive dye) were kindly supplied by DyStar.

Sodium hydroxide, sodium carbonate, sodium dihydrogen phosphate, disodium hydrogen phosphate, sodium sulfate, calcium chloride (anhydrous), acetic acid, and hydrogen peroxide (35%) were reagent-grade.

Methods

Trial scheme

Unless otherwise indicated, this study included the trails listed in Table II.

Optical brightening

Portions of selected samples were treated with the following formulation: Hostalux CBA (0.5 g/L) and concentrated Kierlon Jet B (2 g/L) at pH 6 with a material-to-liquor ratio (LR) of 20/1 at 45°C for 30 min.

Postreactive dyeing

Portions of selected samples were dyed with Remazol B Red 3BS (2% owf) according to the standard method recommended by DyStar.

Testing

Residual starch percentage [violet scale (VS)]

The residual starch contents were assessed with the Tegewa scale method.[15]

Weight loss (WL)

WL was expressed as a percentage with respect to the initial dry weight.

Absorbency (AB)

The times required for a drop of water to be absorbed into the fabric are called the AB values (AATCC Test Method 39-1980).

Whiteness index (WI) and yellowness index (YI)

WI and YI were evaluated with a Color-Eye 3100 spectrophotometer from SDL Inter.[16]

Percentage of retained strength (warp)

The breaking strength was determined by the strip method according to ASTM D 1682-64:

$$\text{Retained strength}(\%) = [(T_1 - T_2)/T_1] \times 100 \quad (1)$$

TABLE II
Wet-Processing Trails

Trail	Substrate	Particular trail[a]	Related Figure	Related Tables
1	I and II	Enzymatic scouring	1	III–VIII
2	I and II	Enzymatic scouring and bleaching		
		One-bath, two-step bioscouring/H_2O_2 bleaching	2	IX–XII
		One-bath, one-step bioscouring/H_2O_2 bleaching	3	XIII
3	I and II	Enzymatic scouring and reactive dyeing		
		Option 1 (at 90°C for 15 min)	4	XIV
		Option 2 (at 70°C for 30 min)	5	
4	III	Enzymatic desizing, scouring, and bleaching		
		One-bath, two-step	6	XV
		All in one	7	

[a] With Rotadyer D cups at a rotation speed of 40 rpm.

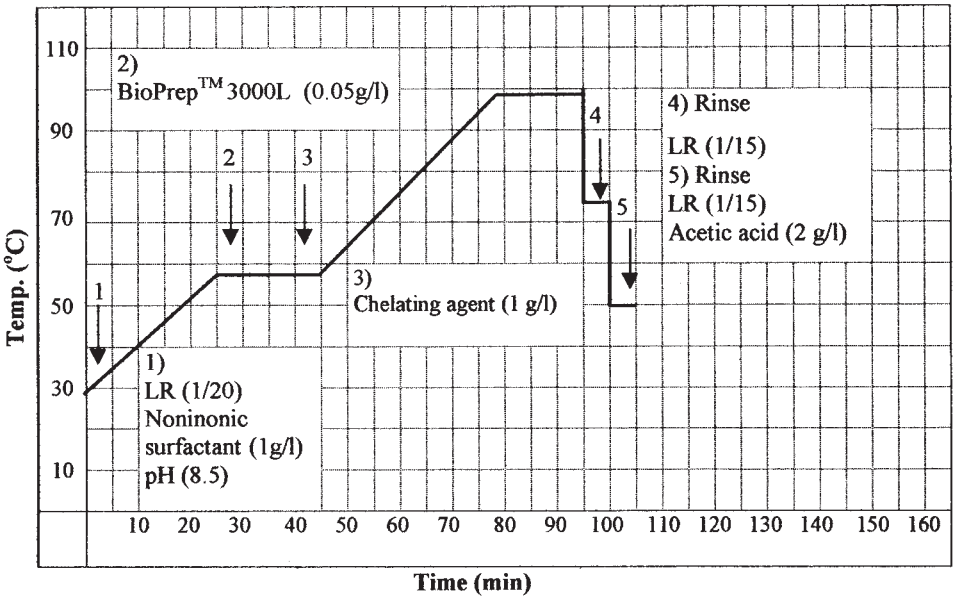

Figure 1 Enzymatic scouring cycle.

where T_1 is the tensile strength before the treatment and T_2 is the tensile strength after the treatment.

Color strength [light absorption coefficient (*K*)/light scattering coefficient (*S*)]

The color strength of dyed fabric samples was measured at the wavelength of the maximum absorbance with a Color-Eye 3100 spectrophotometer, and it was calculated with the Kubelka—Munk equation:[17]

$$K/S = (1 - R)^2/2R \quad (2)$$

where R is the reflectance of the dyed samples at the wavelength of the maximum absorption.

RESULTS AND DISCUSSION

In order to use green technology in textile wet processing, to produce high-quality textile products, and to minimize the environmental impact, we have focused on the development of new eco-friendly options for cotton wet processing, especially pretreatments. The obtained results, along with appropriate discussion, follow.

Factors affecting the biopreparation of unscoured cotton fabrics

LR

As far as changes in AB, YI and WI, and the extent of postreactive dyeing, expressed as K/S values, of bioscoured cotton fabrics (substrates I and II), and for a given set of bioscouring formulation conditions (Fig. 1), the data in Table III reveal that increasing LR up to 1/30 led to a slight improvement in the fabric wettability and water AB values, a reduction in YI, an increase in WI, and a marginal improvement in the K/S values of postdyed samples, regardless of the substrate. The slight improvement in the aforementioned properties could be explained in terms of the better mobility of the bulky enzyme molecules, the better swellability of the cellulose structure, and the

TABLE III
Effects of LR

	Substrate I				Substrate II			
LR	AB (s)	YI	WI	*K/S*	AB (s)	YI	WI	*K/S*
1:10	~2	20.54	14.72	9.11	~2	20.22	16.52	9.65
1:20	~1	19.65	18.79	9.35	~1	19.28	21.89	9.72
1:30	<1	19.22	18.90	9.48	<1	19.10	22.15	9.93

Formulation of step 1: BioPrep 3000L = 0.05 g/L; nonionic surfactant = 1 g/L; pH = 8; temperature = 60°C; 20 min; agitation rate = 40 rpm. Formulation of step 2: chelating agent = 1 g/L; temperature = 100°C; 15 min. Substrate I: light cotton; substrate II: heavy cotton.

TABLE IV
Effects of the Enzyme Dosage

Enzyme dosage (g/L)	Substrate I					Substrate II				
	AB (s)	YI	WI	RS (%)	*K/S*	AB (s)	YI	WI	RS (%)	*K/S*
0.000	6	21.65	15.75	99.1	8.66	9	20.24	18.41	99.6	8.73
0.025	2	20.46	16.39	98.4	9.10	4	19.55	20.32	99.0	9.21
0.05	~1	19.65	18.79	37.3	9.35	~1.3	19.28	21.89	98.2	9.72
0.1	<1	19.53	19.78	36.0	9.53	<1	19.17	22.06	97.3	10.13

Formulation of step 1: BioPrep 3000L = 0–0.1 g/L; nonionic surfactant = 1 g/L; LR = 1/20; pH = 8; temperature = 60°C; 20 min; agitation rate = 40 rpm. Formulation of step 2: chelating agent = 1 g/L; temperature 100°C; 15 min; RS = retained strength.

better availability of noncellulosic impurities (e.g., pectic substances, waxes, fats, and oils), which enhanced the extent of the enzymatic attack and the removal of both the enzymatic reaction products, along with other natural coloring matters with a slight improvement in *K/S* in the dye uptake.

Enzyme dosage

As far as changes in the aforementioned fabric properties as functions of the enzyme dosage, Table IV demonstrates that, for a given set of bioscouring conditions (Fig. 1), increasing the BioPrep 3000L dosage to 0.1 g/L improved the fabric wettability, that is, wax removal, and dyeability with the used reactive dye and reduced the yellowness of the fabric, regardless of the cotton fabric substrate; this could be ascribed to an increase in the extent of enzymatic hydrolysis and the removal of pectin along with the simultaneous removal of cotton waxes during the wax-emulsification step (at 100°C for 15 min) and some other colored impurities.[18]

However, differences in the extent of bioscouring and postreactive dyeing with different cotton substrates could be associated with differences in the fabric weight and construction, natural impurities and noncellulosic materials, the surface morphology, the extent of swellability, and the accessibility of these impurities to enzymatic attack and subsequent emulsification .[14,19]

Effect of pH

For a given set of bioscouring conditions (Fig. 1), Table V demonstrates that raising the pH of the bioscouring bath from 7 to 8.5 gradually increased the extent of pectin removal along with wax removal; that is, it increased the wettability and dyeability. it also gradually reduced YI and improved WI of bioscoured fabric samples (i.e., a gradual removal of natural and colored impurities). A further increase in pH had a positive impact on reducing YI and increasing WI without practically affecting the extent of wetting and postdyeing.

Within the range examined, the data suggest that pH 8–8.5 enhanced the activity and performance of the used pectinase enzyme, thereby giving rise to a higher extent of pectin removal, which was present in the cotton fiber cuticle, with a subsequent release of waxes and proteins with the help of the used nonionic surfactant and chelating agent at a temperature higher than the melting point of wax, thereby enhancing both water and dye uptake.[19,20]

Treatment time

Table VI shows that prolonging the enzymatic treatment time up to 30 min at 60°C and pH 8.5 improved the water AB values (wetting time < 1 s), WI values and color depth, and *K/S* values of bioscoured/postdyed fabric samples.

TABLE V
Effects of the pH of the Enzyme-Treatment Step

pH	Substrate I				Substrate II			
	AB (s)	YI	WI	*K/S*	AB (s)	YI	WI	*K/S*
7	~1.4	20.09	18.60	9.04	~1.5	19.82	20.56	9.28
8	~1.0	19.65	18.79	9.35	~1.3	19.28	21.89	9.72
8.5	<1	19.46	21.11	9.70	<1	18.13	23.28	9.90
9	<1	19.04	21.30	9.73	<1	17.72	23.85	9.98

Formulation of step 1: BioPrep 3000L = 0.05 g/L; nonionic surfactant = 1 g/L; temperature = 60°C; 20 min; agitation rate = 40 rpm. Formulation of step 2: chelating agent = 1 g/L; temperature = 100°C; 15 min.

TABLE VI
Effects of the Enzyme-Treatment-Step Time

Time (min)	Substrate I				Substrate II			
	AB (s)	YI	WI	*K/S*	AB (s)	YI	WI	*K/S*
10	~1.3	20.48	19.62	9.31	~1.6	19.23	21.67	9.56
20	<1	19.46	21.11	9.70	<1	18.13	23.28	9.90
30	<1	18.75	24.10	9.85	<1	17.48	26.64	10.20

Formulation of step 1: BioPrep 3000L = 0.05 g/L; nonionic surfactant = 1 g/L; temperature = 60°C; agitation rate = 40 rpm. Formulation of step 2: chelating agent = 1 g/L; temperature = 100°C; 15 min.

The data suggest that a sufficient enzymatic treatment time favored (1) the adsorption of pectinase enzyme onto the unscoured cotton substrate; (2) the attachment to the pectin (a powerful biological glue) site in the primary wall and the formation of an intermediate complex; (3) the catalysis of pectin hydrolysis, thereby interrupting the matrix structure of the wall; (4) the diffusion away both water-soluble products and other materials from the wall; and (5) the release of the pectinase enzyme to be readsorbed onto another pectin site until the enzyme was deactivated.[19–21] The net effect of the aforementioned mechanism of enzymatic hydrolysis of pectin was the interruption of the cuticle matrix; this led superior fabric AB values (<1 s).[20]

Treatment temperature

For a given set of bioscouring conditions (Fig. 1), Table VII shows that raising the bioscouring bath temperature up to 60°C at pH 8.5 for 20 min led to a noticeable improvement in both the water and dye uptake and an increase in the fabric whiteness, regardless of the cotton substrate. This was a direct result of (1) enhancing the enzyme activity, (2) increasing the swellability of the outer surface of the fiber, (3) enabling enzyme penetration through cracks or micropores in the cuticle and making contact with the pectic substances, and (4) accelerating the extent of the hydrolysis of pectin with the aid of the pectinase enzyme. This resulted in facilitating the removal of pectin, which functioned as a cementing and binding agent, and the partial or complete removal and breakdown of the continuity of the cuticle.[6]

A reduction in AB, WI, and *K/S* values as the temperature rose to 70°C for 20 min at pH 8.5 could be interpreted as a negative impact of a high temperature on the enzyme activity, which reduced the extent of attack and biohydrolysis of pectic substances and minimized the extent of removal of waxes, proteins, and other impurities located in the primary cell wall.

Biopreparation steps

Table VIII shows the effects of biopreparation steps, that is, an alkaline pectinase treatment step at pH 8.5 and 60°C for 20 min in the presence of a nonionic wetting agent (for the removal of pectin) or the same step followed by a wax-emulsification step in the same bath at 100°C for 15 min in the presence of a chelating agent (for the removal of waxes). The combined application of an alkaline pectinase step and a wax-emulsification step in the same bath significantly improved the fabric AB values, the fabric whiteness, and the fabric dyeability in comparison with the values imparted by the alkaline pectinase step alone. This may be a direct reflection of the enhancement of pectin removal, which rendered waxes (fatty acids, alcohols, and esters) extractable and emulsifiable, and the removal of waxes and other impurities at 100°C for 15 min in the presence of a polyacrylate-based chelating agent, which interrupted the primary wall of cotton and facilitated the removal of noncellulosic impurities (e.g., pectin, waxes, and fats), making the cotton highly water-absorbent.[21]

TABLE VII
Effects of the Enzyme-Treatment-Step Temperature

Temperature (°C)	Substrate I				Substrate II			
	AB (s)	YI	WI	*K/S*	AB (s)	YI	WI	*K/S*
50	~1.2	20.97	15.57	8.85	~1.5	20.52	16.38	9.05
60	<1	19.46	21.11	9.70	<1	18.13	23.28	9.90
70	~1.2	20.63	16.60	9.25	~1.2	20.18	17.30	9.36

Formulation of step 1: BioPrep 3000L = 0.05 g/L; nonionic surfactant = 1 g/L; pH = 8.5; 20 min; agitation rate = 40 rpm. Formulation of step 2: chelating agent = 1 g/L; temperature = 100°C; 15 min.

TABLE VIII
Combined Bioscouring Steps

Property	Substrate I		Substrate II	
	B	B + E	B	B + E
AB (s)	7.08	<1	9.21	<1
YI	22.11	19.46	21.21	18.13
WI	14.03	21.11	15.55	23.28
K/S	8.71	9.70	8.79	9.90

B = BioPrep treatment step: BioPrep 3000L = 0.05 g/L; nonionic surfactant = 1 g/L; temperature = 60°C; 20 min; agitation rate = 40 rpm. B + E = BioPrep treatment step followed by a wax-emulsification step at 100°C for 15 min in the presence of a chelating agent (1 g/L).

Factors affecting the combination of biopreparation and H_2O_2 bleaching

To study the technical feasibility of combining bioscouring and H_2O_2 bleaching of unscoured cotton fabrics to remove noncellulosic impurities, such as pectin and waxes, in the outer coating of cellulosic fibers, that is, to improve water absorptivity, and to destroy colored impurities, that is, to produce a white substrate, we treated cotton fabric samples under different conditions (Figs. 2 and 3).

H_2O_2 concentration

The dependence of the extent of the improvement in the aforementioned properties upon the incorporation of H_2O_2 (2–10 mL/L, 35%) in the wax-emulsification step (Fig. 2) is presented in Table IX. For a given set of one-bath, two-step bioscouring and H_2O_2 bleaching, (1) increasing the H_2O_2 concentration up to 5 mL/L was accompanied by a gradual decrease in YI and a remarkable improvement in WI as a direct result of increasing the extent of generation of the perhydroxyl ion, which was the active species in bleaching, and thereby increasing the extent of oxidative decoloration of the natural colorants in the raw cotton;[3] (2) increasing the H_2O_2 concentration had practically no effect on the water absorptivity of treated cotton fabric, regardless of the used substrate; (3) the postoptical brightening of prebioscoured/H_2O_2-bleached fabric samples brought about a significant increase in the fabric surface whiteness and brightness, most likely because of the capability of the used optical brightener to remit light in the region of 450 nm (blue color) to neutralize the yellow component of the visible region present in cotton fabrics,[22,23] regardless of the H_2O_2 concentration and the cotton substrate; and (4) a further increase in the H_2O_2 concentration, that is, up to 10 mL/L, resulted in a slight reduction in YI values, a slight improvement in the whiteness index before brightening (WI_B), and with a marginal increase in the WI values of postbrightened cotton fabric samples.

Wax-emulsification/H_2O_2-bleaching step time

Table X shows that prolonging the wax-emulsification/H_2O_2-bleaching step up to 100°C, with H_2O_2 (5 mL/L), Na_2CO_3 (2 g/L), and Dekol SN (1 g/L), gradually and slightly reduced YI values, gradually and slightly increased WI_B values, and noticeably improved the whiteness index after optical brightening (WI_A) without affecting the water absorptivity of the treated cotton samples.

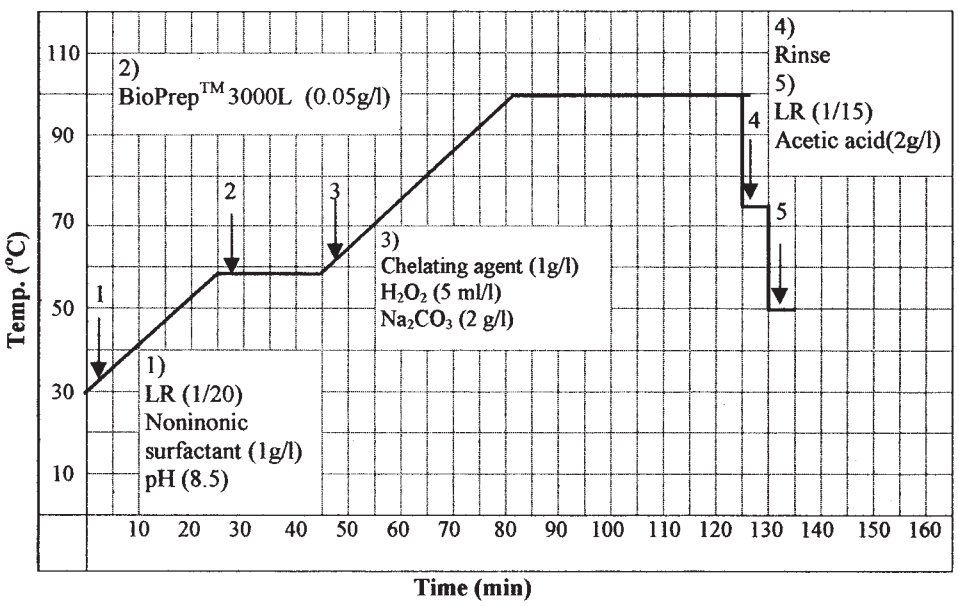

Figure 2 One-bath, two-step bioscouring and H_2O_2 bleaching.

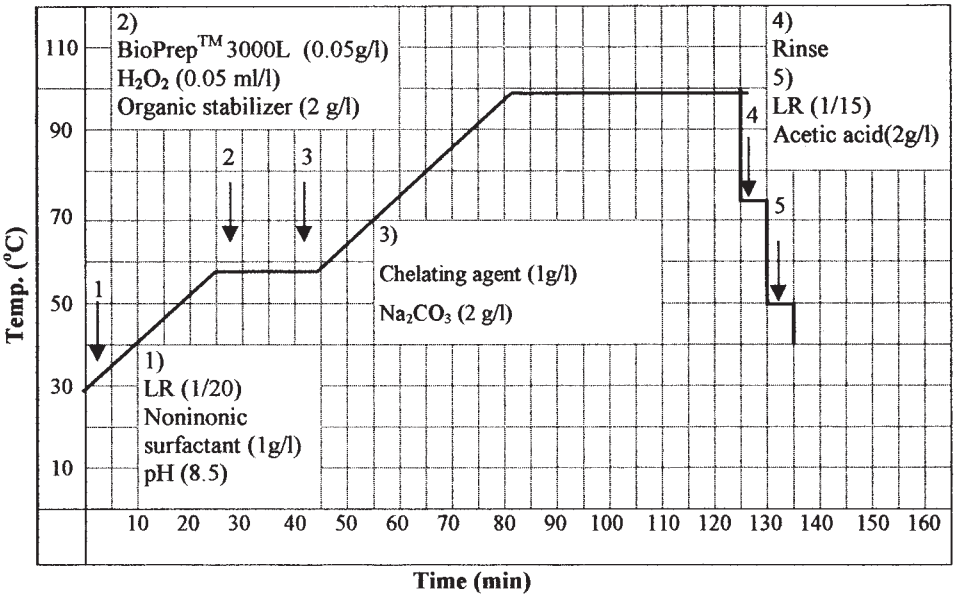

Figure 3 One-bath, one-step bioscouring and H_2O_2 bleaching.

Addition of the organic stabilizer

Table XI shows the effects of the incorporation of an organic stabilizer (peroxide stabilizer H-ET; 2 g/L) into the aforementioned wax-emulsification/H_2O_2 formulation on AB, YI, WI_B, and WI_A. For a given set of conditions, for one-bath, two-step bioscouring and H_2O_2 bleaching, the reduction of YI and the increase in WI followed this order: with the organic stabilizer > without the organic stabilizer. This could be associated with its positive impact on stabilizing the decomposition of H_2O_2 caused by trace transition-metal ions, particularly copper and iron, in the cellulosic fibers and/or solution.[3,24]

TABLE IX
Effects of the H_2O_2 Concentration

H_2O_2 concentration (mL/L)	Substrate I				Substrate II			
			WI				WI	
	AB (s)	YI[a]	WI_B	WI_A	AB (s)	YI[a]	WI_B	WI_A
0.0	<1	18.86	22.03	39.51	<1	17.30	24.36	43.50
2.5	<1	15.38	34.69	52.97	<1	13.99	37.77	65.14
5.0	<1	12.01	44.64	65.21	<1	10.69	47.23	78.01
10.0	<1	10.47	49.06	65.89	<1	9.96	50.39	79.12

Bioscouring conditions: BioPrep 3000L = 0.05 g/L; nonionic surfactant = 1 g/L; temperature = 60°C; 20 min; agitation rate = 40 rpm. Wax-emulsification/H_2O_2-bleaching conditions: H_2O_2 = 0–10 mL/L; chelating agent = l g/L; Na_2CO_3 = 2 g/L; 45 min; 100°C.

[a] Before optical brightening.

TABLE X
Effects of the Wax–Emulsification/H_2O_2-Bleaching Step Time

Time (min)	Substrate I				Substrate II			
			WI				WI	
	AB (s)	YI	WI_B	WI_A	AB (s)	YI	WI_B	WI_A
15	<1	13.15	40.99	54.41	<1	11.22	45.07	70.84
30	<1	12.99	42.28	60.84	<1	11.00	46.24	73.30
45	<1	12.01	44.64	65.21	<1	10.69	47.23	78.01

Bioscouring conditions: BioPrep 3000L = 0.05 g/L; nonionic surfactant = 1 g/L; temperature = 60°C; 20 min; agitation rate = 40 rpm. Wax-emulsification/H_2O_2-bleaching conditions: chelating agent = 1 g/L; Na_2CO_3 = 2 g/L; H_2O_2 = 5 mL/L; 100°C.

TABLE XI
Effects of the Addition of the Organic Stabilizer

Properties	Substrate I		Substrate II	
	Without stabilizer	With stabilizer	Without stabilizer	With stabilizer
AB (s)	<1	<1	<1	<1
YI	13.30	12.02	11.22	10.10
WI_B	44.64	46.42	47.23	51.27
WI_A	65.21	76.95	78.01	80.24

Bioscouring conditions: BioPrep 3000L = 0.05 g/L; nonionic surfactant = 1 g/L; temperature = 60°C; 20 min; agitation rate = 40 rpm. Wax-emulsification/H_2O_2-bleaching conditions: chelating agent = 1 g/L; Na_2CO_3 = 2 g/L; H_2O_2 = 5 mL/L; organic stabilizer = 2 g/L; 45 min; 100°C.

Type of wetting agent

Table XII shows the effects of different wetting agents in the enzyme-treatment step on AB, YI, and WI (with and without an optical brightener) of bioscoured and H_2O_2-bleached cotton fabric samples. The improvement in the AB values and in the degree of whiteness of the bioscoured/H_2O_2-bleached cotton fabric depended on the presence and absence of the wetting agent and on its ionic nature; the improvement could be ordered as follows: nonionic wetting agent > anionic wetting agent > none.

The differences in the magnitudes of the AB and YI values with the surfactants under investigation were probably due to differences in the chemical composition, ionic nature, tendency to inhibit (with an anionic one) or activate the used enzyme, emulsification power, cloud point, compatibility with other ingredients in the treatment bath, and its positive or negative effect on enhancing the extent of saponification of fats and oils and the emulsification of unsaponifiable materials such as waxes and dirt.[3]

Effect of the sequence of H_2O_2 addition

Table XIII shows the effects of the incorporation of H_2O_2 (5 mL/L) and an organic stabilizer (2 g/L) in the enzyme-treatment step (Fig. 3) or wax-emulsification step (Fig. 2) on the AB, YI, and WI values of the treated fabric samples. The reduction of YI and increase in WI, as functions of the sequence of H_2O_2 addition, were ordered as follows, regardless of the used substrate and without any affect on the AB values: enzyme/H_2O_2 > dewaxing/H_2O_2. This could be attributed to the improvement in the extent of the destruction and oxidation of the colored impurities in the cellulosic fibers due to the prolonged time of exposure to H_2O_2 via its incorporation into the enzyme-treatment step, followed by the dewaxing step in the same bath.

Single-bath, two-step bioscouring and reactive dyeing

Table XIV lists the AB and *K/S* values for samples treated under various conditions (Figs. 4 and 5). For given bioscouring and reactive dyeing conditions, the results show the following:

1. The *K/S* values depended on the conditions of the dewaxing/reactive-dyeing step and could be ranked as follows, regardless of the substrate:

TABLE XII
Effects of the Wetting Agent Type

Wetting agent	Substrate I				Substrate-II			
			WI				WI	
	AB (s)	YI	WI_B	WI_A	AB (s)	YI	WI_B	WI_A
Hostapal CV-ET	<1	12.02	46.42	76.95	<1	9.25	51.27	78.61
Sandozin MRN liquid concentrate	<1	12.65	46.23	75.57	<1	10.23	52.00	78.15
Leophen M	<1	13.66	44.94	67.66	<1	10.51	55.30	72.60
Kierlon Jet B concentrate	<1	16.24	39.16	63.90	<1	10.89	56.30	71.20
Sandozin EH liquid	<1	12.56	46.23	75.57	<1	10.23	52.00	78.15
None	2	17.94	34.97	57.15	3	16.49	36.96	65.73

Bioscouring conditions: BioPrep 3000L = 0.05 g/L; nonionic surfactant = 1 g/L; temperature = 60°C; 20 min; agitation rate = 40 rpm; wax-emulsification/H_2O_2 bleaching conditions: chelating agent = 1 g/L; Na_2CO_3 = 2 g/L; H_2O_2 = 5 mL/L; organic stabilizer = 2 g/L; 45 min; 100°C.

TABLE XIII
Effects of the Sequence of H_2O_2 Addition

Property	Substrate I		Substrate II	
	Enzyme step[a]	Dewaxing step[b]	Enzyme step[a]	Dewaxing step[b]
AB (s)	<1	<1	<1	<1
YI	10.26	12.02	8.45	10.10
WI_B	49.65	46.42	53.85	51.37
WI_A	81.96	76.95	84.45	80.24

[a] Enzyme/H_2O_2 treatment step (Fig. 3).
[b] Dewaxing/H_2O_2 treatment step (Fig. 2).

option 2 > option 1. This could be explained in terms of a better extent of dye exhaustion and the efficiency of fixation for bioscoured and reactive-dyed fabric samples according to the conditions given in Figure 5.

2. The reduction in the *K/S* values with the conditions given in Figure 4 could be discussed in terms of increasing the tendency toward hydrolysis of the dye and thereby decreasing the extent of dye uptake and the dye–fiber interaction.[25]
3. The extent of dye fixation and the *K/S* values were governed by both the nature of the reactive dye, that is, the physicochemical properties, extent of diffusion and penetration within the substrate, fixation/hydrolysis ratio, and dye–fiber bond stability, and the nature of the cotton fabric substrate, as discussed earlier.[26,27]
4. No difference was found in the AB values of the bioprepared and dyed fabric samples with the aforementioned treatment options.

TABLE XIV
Effects of the Biopreparation/Reactive Dyeing Conditions

Reactive dye	Substrate I				Substrate II			
	Option 1[a]		Option 2[b]		Option 1[a]		Option 2[b]	
	AB (s)	*K/S*	AB (s)	*K/S*	AB (s)	*K/S*	AB (s)	*K/S*
Levafix B. Red E-2RN	2	14.64	2	14.93	2	16.81	2	17.33
Remazol B. Red 3BS	2	6.74	2	8.55	2	7.75	2	8.76
Levafix R. Blue E-FR	2	9.25	2	10.33	2	9.50	2	11.60

[a] Figure 4.
[b] Figure 5.

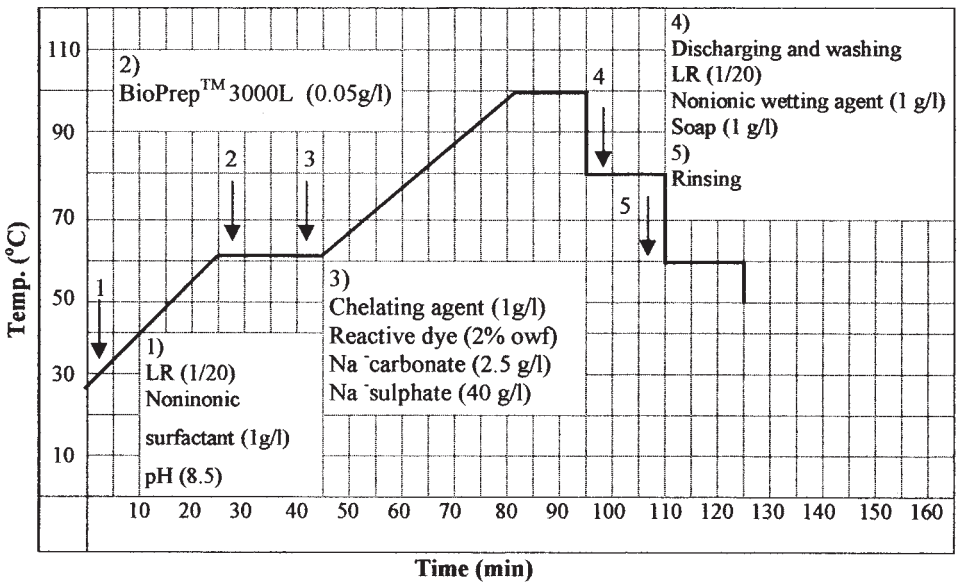

Figure 4 One-bath, two-step bioscouring followed by dewaxing and reactive dyeing (option 1).

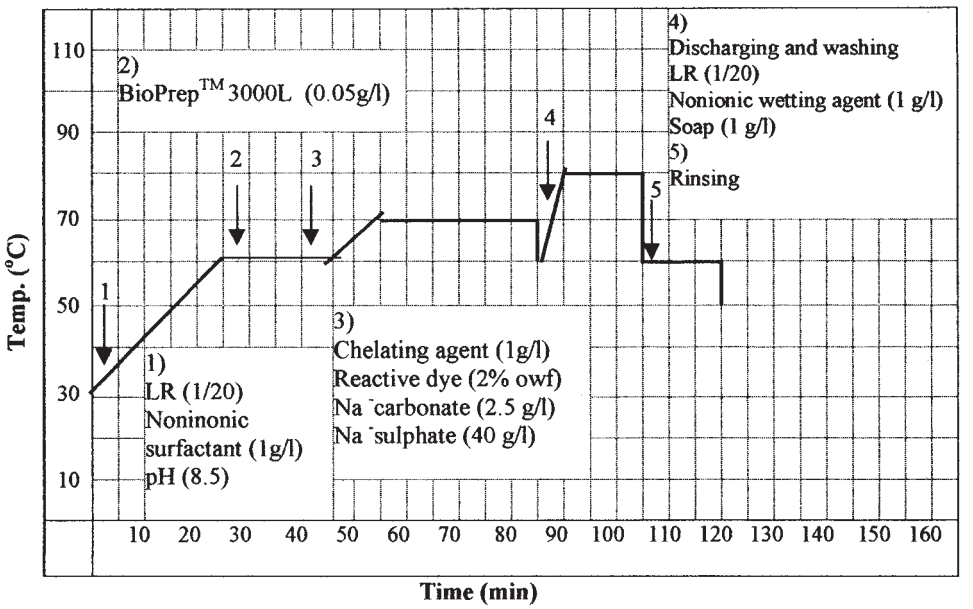

Figure 5 One-bath, two-step bioscouring followed by dewaxing and reactive dyeing (option 2).

Combination of enzymatic desizing, bioscouring, and H_2O_2 bleaching

Two approaches were used to determine the ability to run single-bath, two-step enzymatic desizing and biopreparation followed by H_2O_2 bleaching (Fig. 6) and single-bath, one-step simultaneous enzymatic desizing of starch-sized cotton fabric, bioscouring, and H_2O_2 bleaching (Fig. 7), and the impact of these two trials on the performance properties of the treated cotton fabric samples was examined (Table XV). For given pretreatment conditions, the incorporation of $CaCl_2$ (0.5 g/L) in the enzymatic-desizing/bioscouring step resulted in an improvement in the extent of starch removal, expressed as WL and VS, a reduction in YI and an increase in WI_B and WI_A of the pretreated fabric samples, which reflected the positive impact of $CaCl_2$ the activity and thermal stability of Aquazym 240L. This enhanced the extent of starch removal and the stability of H_2O_2 in the next step and improved the extent of the oxidization and removal of the colored impurities along with any residual starch size,[28,29] without adversely affecting the activity of BioPrep 3000L (AB $<$ 1 s). The change in the performance properties with different treatment conditions (Figs. 6 and 7) followed a decreasing order: one-bath and two-step in the presence of $CaCl_2$ $>$ single-bath and one-step in the presence of $CaCl_2$ $>$ one-bath and two-step in the absence of $CaCl_2$. This reflected the differences in these conditions in (1) the efficiency of size removal, pectin removal, dewaxing, and oxidation and decolorization of colored impurities and (2) the compatibility of different ingredients in the used formulations and their impact on the enzyme activity

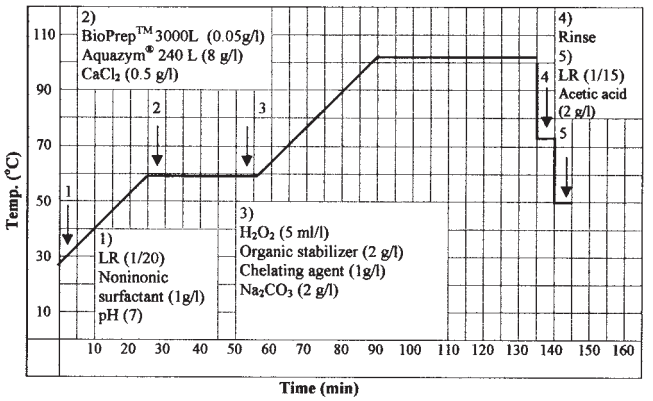

Figure 6 One-bath, two-step enzymatic desizing and bioscouring followed by H_2O_2 bleaching.

CONCLUSIONS

This article is focused on the development of new eco-friendly options for cotton wet processing with enzymes. The results led to the following conclusions. Increasing LR slightly improved the water absorption and WI and marginally improved the color strength of bioscoured, postdyed fabric samples. Increasing the BioPrep 3000L dosage up to 0.1 g/L improved the fabric wettability and dyeability and reduced YI, regardless of the cotton substrate. Raising the bioscouring bath pH up to 8.5 was accompanied by a gradual increase in the extent of pectin removal and wax removal; this enhanced both the wettability and dyeability of treated fabric samples and gradually improved WI. Bioscouring with an alkaline pectinase enzyme (0.05 g/L) at 60°C and pH 8.5 for 20 min was optimal

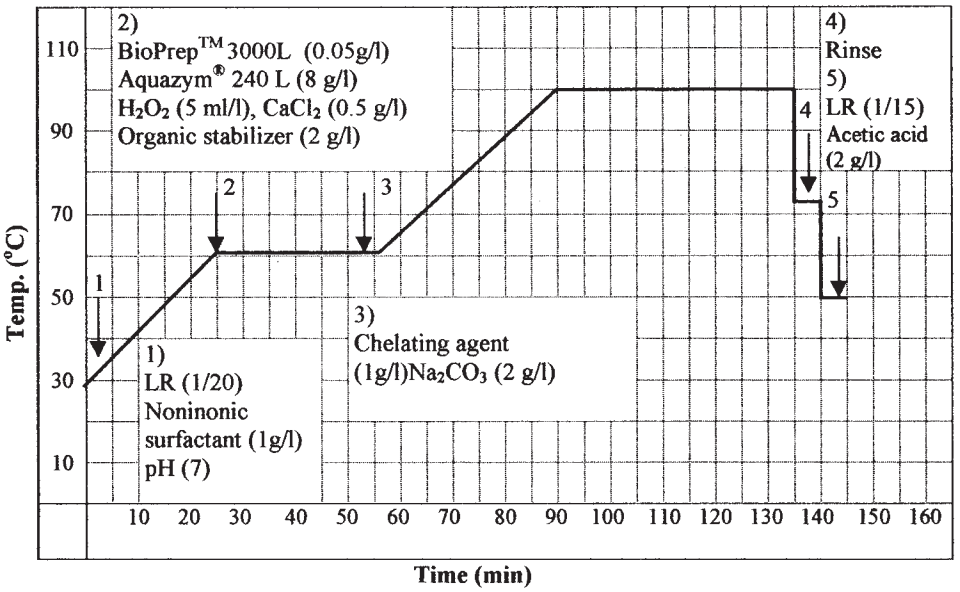

Figure 7 Single-bath, one-step enzymatic desizing, bioscouring, and H_2O_2 bleaching.

for better performance properties. The combined application of an alkaline pectinase enzyme step and a wax-emulsification step in the same bath enhanced the performance properties of the treated cotton fabric samples. The single-bath, two-step biopreparation and H_2O_2 bleaching of unscoured cotton substrates was feasible. The incorporation of H_2O_2 up to 5 mL/L significantly enhanced WI. Postoptical brightening of prebioscoured and H_2O_2-bleached cotton fabric samples significantly increased the fabric surface whiteness and brightness. Prolonging the wax-emulsification/H_2O_2-bleaching step up to 45 min at 100°C noticeably improved WI_A. The addition of an organic stabilizer (2 g/L) in the aforementioned step improved the extent of the oxidation and decolorization of colored impurities. The improvement in the performance properties of the treated fabric samples depended on the nature of the added surfactant: nonionic > anionic > none. The increase in WI, as a function of the sequence of H_2O_2 addition, followed a descending order: alkaline pectinase/H_2O_2 > wax emulsification/H_2O_2. The data indicated that it was feasible to conduct bioscouring and reactive dyeing in a single bath and two steps, and the extent of dye exhaustion and fixation was determined by the dewaxing and reactive-dyeing conditions and the nature of the reactive dye. The incorporation of Aquazym 240L, BioPrep 3000L, and H_2O_2 in the presence of other textile auxiliaries improved the extent of starch-size removal, the wettability, and the whiteness of treated fabric samples concurrently.

TABLE XV
Comparison of the Performance Properties of Pretreated Cotton Fabric Samples for Substrate III

Property	One bath, two steps (Fig. 1)	Single bath, one step (Fig. 5)
WL (%)	10.99 (9.89)	9.98
VS (shade)	7 (5–6)	6
YI	10.60 (12.38)	10.84
WI_B	43.86 (38.54)	41.66
WI_A	75.21 (51.52)	58.66
AB (s)	<1 (<1)	<1
RS (%)		

Values in parentheses indicate the absence of $CaCl_2$. RS = residual starch.

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