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New development for combined bioscouring and bleaching of cotton-based fabrics

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ABSTRACT

A thorough investigation into conditions appropriate for effecting combined eco-friendly bioscouring and/or bleaching of cotton-based fabrics was undertaken. Fabrics used include cotton, grey mercerized cotton, cotton/polyester blend 50/50 and cotton/polyester blend 35/65. The four cotton-based fabric were subjected to bioscouring by single use of alkaline pectinase enzymes or by using binary mixtures of alkaline pectinase and cellulase enzymes under a variety of conditions. Results of bioscouring show that, the bioscoured substrates exhibit fabrics performances which are comparable with these of the conventional alkali scouring. It has been also found that, incorporation of ethylenediaminetetraacetic acid (EDTA) in the bioscouring with mixture from alkaline pectinase and cellulase improves the performance of the bioscoured fabrics. Addition of β -cyclodextrin to the bioscouring solution using alkaline pectinase in admixtures with cellulase acts in favor of technical properties and performance of the bioscoured fabrics. Concurrent bioscouring and bleaching by in situ formed peracetic acid using tetraacetylethylenediamine (TAED) and H₂O₂ was also investigated. The results reveal unequivocally that the environmentally sound technology brought about by current development is by far the best. The new development involves a single-stage process for full purification/preparation of cotton textiles. The new development at its optimal comprises treatment of the fabric with an aqueous formulation consisting of alkaline pectinase enzyme (2 g/L), TAED (15 g/L), H₂O₂ (5 g/L), nonionic wetting agent (0.5 g/L) and sodium silicate (2 g/L). The treatment is carried out at 60 °C for 60 min. Beside the advantages of the new development with respect to major technical fabric properties, it is eco-friendly and reproducible. This advocates the new development for mill trials.

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1. Introduction

Cotton cellulose has excellent properties such as higher water absorbency and moisture, being comfortable to wear and easily to dye. For these reasons, the apparel industry is predominantly cotton based, and the share of cotton in total fibre consumption is about 50% (Carr, 1995; Karmakar, 1999).

Cotton is composed almost entirely of cellulose (88.0–96% based on weight of fibres (owf)). The impurities in cotton fibre range from 4 to 12% and include protein (1.0–1.9%), wax (0.4–1.2%), ash (inorganic salts) (0.7–1.6%), pectin (0.4–1.2%) and others (resins, pigments, hemi-cellulose) (0.5–0.8%) (Lewin & Mark 2007, chap. 3; Segal & Wakelyn, 1988).

With the exception of natural coloring matters that may be removed by bleaching using certain oxidants, many other impurities are removed by alkali treatment in scouring stage. The latter in

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common practice involves boiling the cotton in sodium hydroxide (2–5%) for 30–60 min, which means that a higher energy is consumed. Because of the high pH values, the treatment should be followed by intensive rinsing and neutralizing, which means that large amount of water is also consumed. Moreover, the aggressive scouring treatment conditions frequently damage the fibre (Hashem, 2007; Lewin & Sello, 1984).

The aforementioned disadvantages of scouring with sodium hydroxide have motivated the textile industry to introduce more enhanced biological agents, which would be as effective in removing non-cellulose substances but would not have damaging effects and would be less energy and water consuming. Pectinases or pectinolytic enzymes are the enzymes that catalyze the hydrolysis of pectin substances. Three main types of enzymes are used to break down pectin substances namely, pectin esterases, polygalacturonases and pectin lyases (Hashem, 2007). After breaking down and removing pectin, which binds, as a natural binder, non-cellulose substances with the fibre cellulose core, other non-cellulose substances can be removed from cotton by using hot washing with surfactant.

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Over the last two decades, chlorine-containing bleaching compounds have been withdrawn from the market and their usage become limited. This is due to the formation of highly toxic chlorinated organic by-products (AOX) during the bleaching process as well as effluents discharged therefrom. Moreover, the legal regulations have stipulated very limiting values for (AOX) in the textile effluent. Nowadays textile industries are obliged to bleach without using chlorine-containing compounds. Peracetic acid is environmentally safe alternative bleaching agent, since it decomposes to acetic acid and oxygen. The decomposition products are biodegradable and do not form any toxic by-products (Boesinga et al., 1999; Krizman, Kovac, & Tavcer 2005; Reinhardt, 2006).

Peracetic acid as an industrial chemical agent is easily available and can be safely introduced into an existing process design. An important advantage of peracetic acid, which contain –COOH groups, is that they are fast acting, non foaming, water soluble liquids and break down to innocuous decomposition products that are environmentally acceptable (Guersoy & Dayioglu, 2000; Hashem, 1999).

Recent research has shown that tetraacetylethylenediamine (TAED) activated peroxide system has potential in bleaching of cotton-based textiles with improved bleaching effectiveness under mild conditions. Both TAED and reaction by-product (diacetylethylenediamine) as well as peracetic acid are nontoxic and biodegrade to give carbon dioxide, water, nitrate and ammonia as end product. Therefore, TAED provides totally environment friendly bleaching agent (Hashem, El-Bisi, & Hebeish, 2003).

As scouring with pectinases and bleaching with PAA are carried out at the same temperature, a similar pH value and a similar time, it is so feasible that, both processes could be joined into a single combined process. In this way, consumption of water, energy, time and auxiliary agents would be lowered. Prior to combining both processes, we wanted to find out whether pectinases retained their activity in the presence of PAA.

The present work was essentially aimed at:

(i) Discovering the proper conditions for the enzymatic scouring of cotton and cotton/polyester fabrics. (ii) comparing the alkaline scouring of cotton and cotton/polyester fabrics with enzymatic scouring of the latter using pectinases and mixtures of pectinase and cellulase enzymes, (iii) studying the influence of EDTA and β -cyclodextrin independently when used at different concentrations as a pretreatment for bioscouring on the efficiency of the latter and (iv) investigating the effects of EDTA and β -cyclodextrin on the efficiency of bioscouring using alkaline pectinase enzyme in single use or in admixtures with cellulase enzyme. (v) The optimization of bleaching process parameters including TAED and H₂O₂ concentrations, pH, temperature and time and; vi) comparing the single use of bleaching process using TAED and H₂O₂ with the combined use of TAED, H₂O₂ and alkaline pectinase in one bath. Quality of bleached cotton-based fabric was measured in terms of weight loss, whiteness index, tensile strength, fabric absorbency and carboxyl content.

2. Experimental

2.1. Materials

Four different types of cotton-based fabrics were used. These comprised grey 100% cotton fabric ($160 \, \text{g/m}^2$), grey mercerized cotton fabric ($160 \, \text{g/m}^2$), grey cotton/polyester (50/50) blended fabric ($160 \, \text{g/m}^2$) and grey cotton/polyester (35/65) blended fabric ($24 \, \text{g/m}^2$). These four cotton-based fabrics will be referred to as substrates I–IV for convenience. All substrates (fabrics) were sized using the same sizing recipe based on starch. The cotton/polyester

blend fabrics were sized using a size base consisting of starch along with PVA or CMC. They were supplied by EL-Nasr Company for Spinning, Weaving and Dyeing, Mehala EL-Kubra, Egypt.

Tetraacetylethylenediamine (TAED), sodium phosphate dibasic, sodium hydrogen phosphate, sodium hydroxide and hydrogen peroxide (50%), were of laboratory grade chemicals. Sodium silicate and Egyptol® (nonionic wetting agent based on ethylene oxide condensate) were of commercial grade chemicals.

Three commercial grade enzymes, Aquazyme 240 L (α -amylase with an activity 240 KNU/g), Scourzym L (an alkaline Pectinase with an activity 375 APSU/g) and Cellusoft Conc. L (a cellulase with an activity 1500 EGU/g) were kindly supplied by Novo-Nordisk A/S, Copenhagen, Denmark.

2.2. Enzymatic desizing

Loomstate fabrics were padded in an aqueous solution containing commercial (α -amylase) enzyme; 1 g/L at 40 °C, and pH 5.5 in acetate buffer and Egyptol® (0.5 g/L) and sodium chloride (0.5 g/L) for 10 min using material to liquor ratio 1:50. Fabric so treated was squeezed to 100% wet pick up then batched for 24 h at ambient conditions. The desized fabric obtained was washed twice with hot water then washed with cold water and dried at ambient conditions.

2.3. Alkaline pectinase scouring

Desized fabrics were scoured through harnessing alkaline pectinase enzyme in 0.05 M phosphate buffer containing 0.5 g/L nonionic wetting agent at pH 8.5 and temperature 55 °C using a material to liquor ratio 1:50. The whole operation of this bioscouring was carried out using mechanical washing machine. Four concentrations of alkaline pectinase were used in the treatments viz, 0.5, 1, 2 and 3 g/L. After the treatments the temperature was raised to 100 °C for 10 min to stop the enzyme action. The fabrics were then washed with hot water and cold water and finally dried at ambient conditions.

2.4. Combined cellulase/alkaline pectinase scouring

This method comprised two steps, the first step was pectinase treatment as described above, and the second step involved cellulase which treatment was done at 55 °C for 60 min. The treating solution in second step was prepared using acetate buffer containing Egyptol® (0.5~g/L) at pH 4.8 and material to liquor ratio 1:50 in a mechanical washing machine. Five balls were added for each gram of fabric tested. The fabric was loaded once the temperature reached 55 °C. After the treatment, the temperature was raised to 100~°C for 10~min to stop the enzyme action. The fabrics were washed with hot water then with cold water and finally dried at ambient conditions.

Alkaline pectinase and cellulase were used with three different combinations of enzymes:

- 1.5 g/L alkaline pectinase and 0.5 g/L cellulase.
- 1 g/L alkaline pectinase and 1 g/L cellulase.
- 0.5 g/L alkaline pectinase and 1.5 g/L cellulase.

2.5. Treatment with EDTA

Desized fabrics were treated with aqueous solution containing $1-4\,\mathrm{g/L}$ of EDTA and Egyptol® $(0.5\,\mathrm{g/L})$ at $50\,^\circ\mathrm{C}$ for $15\,\mathrm{min}$ using material to liquor ratio 1:50. The desized samples were then washed twice with hot water followed by washing with cold water and finally dried at ambient conditions.

2.6. Treatment of cotton-based fabrics with β -cyclodextrin

Desized fabrics were treated with aqueous solution containing 2.5–10 g/L of β -cyclodextrin and Egyptol® (15 g/L) at 85 °C for 15 min using material to liquor ratio 1:50. At this end the desized samples were washed twice with hot water then with cold water and dried at ambient conditions.

2.7. Bleaching

The bleaching process was performed using the exhaustion method by making use of *in situ* formation of peracetic acid. In this technique, bioscoured cotton and bioscoured cotton/polyester blend fabrics were treated with an aqueous solution containing different concentrations of tetraacetylethylenediamine (TAED) (5–20 g/L) and hydrogen peroxide (H_2O_2) (3–9 g/L). Sodium silicate (2 g/L) and Egyptol® (0.5 g/L) were added to the solution. A material to liquor ratio of 1:50 was used and the pH of the bath was adjusted at 8 using (2 g/L) sodium hydroxide aqueous solution was then added drop-wise with continuous stirring. The bleaching process was performed at different temperatures (40–70 °C) for different periods of time (30–120 min). The bleached sample was washed several times with hot water then with cold water and finally dried at ambient conditions.

2.8. Combined bioscouring and bleaching processes

In this process enzymatically desized cotton and cotton/polyester blend fabric were treated with an aqueous solution containing different concentrations of alkaline pectinase enzyme; (0.5–3 g/L), tetraacetylethylenediamine TAED (15 g/L), H₂O₂ (5 g/L), sodium silicate (2 g/L) and Egyptol® (0.5 g/L). The process was carried out at 55 °C and pH 8 for 30–120 min. A material to liquor ratio of 1:50 was used. Temperature was then raised to 100 °C and kept at this temperature for 10 min to stop the action of alkaline pectinase enzyme. The bleached samples were washed several times with hot water then with cold water and finally dried at ambient conditions.

2.9. Testing and analysis

All fabrics were conditioned in 65% RH and 30 $^{\circ}$ C for 24 h before testing.

Loss in fabric weight was expressed as the percentage with respect to the initial weight of the fabric.

Water absorbency was monitored according to an AATCC Test Method 39-1980 (Evaluation of Wettability) (AATCC, 1980). The time (in seconds) between the contact of water drop with the fabric and the disappearance of the water drop into the fabric called

wetting time. The shorter the wetting time, the better the fabric absorbency.

Tensile strength was determined by the strip method according to ASTM, Standard Test Method "Breaking Load and Elongation of Textile Fabric", D-1682-94 (1994).

Whiteness was evaluated with a Color-Eye 3100 Spectrophotometer from SDL Inter.

Carboxyl content was determined according to reported method (Hashem & Smith, 2003; Hauser, Smith, & Hashem, 2004).

3. Results and discussion

3.1. Optimization of bioscouring

3.1.1. Effect of pectinase concentration

The four substrates under investigation were treated with alkaline pectinase enzyme at different conditions and the technical properties of the resultant bioscoured substrates were determined; these properties are summarized in Table 1. Results of Table 1 signify that with all substrates, increasing the alkaline pectinase concentration from 0.5 to 2 g/L acts in favor of loss in fabric weight, however, type and nature of the substrate determine the magnitude of the loss in fabric weight. As the latter is regarded to include all impurities (pectins, waxes, coloring matter, residual size, etc.), the enhancement in loss in fabric weight is indicative of removal of almost, if not all, impurities via bioscouring under the influence of alkaline pectinase enzyme. Further increase in the concentration of alkaline pectinase up to 4 g/L causes marginal increase in the loss in fabric weight as compared with an enzyme concentration of 2 g/L, meanwhile differences in loss in fabric weight values among the four substrates persist. It is as well to emphasize that, substrates I and II which are based on 100% cotton possess higher losses in fabric weight than substrates III and IV which are based on cotton/polyester blended.

Table 1 shows also that the whiteness index marginally increases as the concentration of alkaline pectinase enzyme increases up to $2\,\mathrm{g/L}$. Beyond this concentration, the enhancement in whiteness index is practically negligible. This trend is substantiated by the results of yellowness index; the yellowness index decreases by increasing the alkaline pectinase concentration up to $2\,\mathrm{g/L}$ then remains almost intact by further increasing the enzyme concentration to $4\,\mathrm{g/L}$.

This means that, maximum purification from impurities of the four substrates could be achieved at 2 g/L alkaline pectinase; in accordance with previous reports which ascribed this to an increase in the extent of enzymatic hydrolysis, leading to removal of pectin, waxes and other colored impurities.

Water absorbency of the fabric is much affected by its treatment with alkaline pectinase enzyme even upon using the latter

Table 1Effect of alkaline pectinase concentration on some technical properties of cotton-based fabrics.

Cellulase conc. (%)	Wigh	t loss (S	%)		Whiteness inde	x			Wat (sec	ter ab	sorbe	ency	Retained	d tensile s	trength (%)
	a	b	С	d	a	b	c	d	a	b	С	d	a	b	С	d
Enzymatic desizing	6.17	1.20	3.36	1.55	0.32 (24.32)*	-9.26 (28.93)	11.66 (20.59)	32.09 (15.65)	16	35	15	30	100	100	100	100
0.5	1.9	1.4	1.45	1.05	6.4 (22.41)	-8.11 (27.01)	16.13 (11.37)	34.35 (13.61)	9	11	5	5	87.17	85.89	93.18	94.28
1.0	2.64	2.20	1.72	1.54	9.81 (21.57)	-7.65(26.72)	18.59 (13.89)	35.01 (12.98)	7	5	4	4	83.33	83.33	88.60	93.57
2.0	3.10	2.75	2.35	1.95	11.49 (18.61)	-5.76 (24.28)	21.32 (15.11)	37.72 (11.45)	5	4	3	3	83.33	83.33	82.95	92.85
4.0	3.40	2.95	2.21	2.04	11.51 (18.45)	-5.63 (24.01)	37.80 (11.43)	4	4	2	2	83.33	82.65	78.40	92.85	

Where (a) desized cotton fabric (substrate I), (b) desized mercerized cotton fabric (substrate II), (c) desized cotton/polyester blend 50/50 (substrate III) and (d) desized cotton/polyester blend 35/65 (substrate IV).

Conditions used: [Epyptol®], 0.5 g/L; temperature, 55 °C; time, 60 min; pH; 8.5; MLR; 1:50.

^{*} values between brackets represent the yellowness index.

at a concentration of 0.5 g/L. Higher enzyme concentration works for inducing better absorbency; the latter increases by increasing the alkaline pectinase concentration from 0.5 to 4 g/L. This, indeed, reflects the effectiveness of current biotreatment in removing hydrophobic impurities, notably, waxes and pectin along with other impurities irrespective of the substrate used. However substrates III and IV display higher values of water absorbency than those of substrates I and II.

As the substrates III and IV are cotton/polyester blends, current data suggest that the alkaline pectinase enzyme hydrolyzes the polyester surface and converts the polyester from a hydrophobic component to hydrophilic component in the blend fabric. However, the onset of biotreatment as a whole on the cotton and polyester components of the blend cannot be ruled out.

The retained strength (%) decreases after any of the substrates in question is treated with alkaline pectinase at a concentration of 1 g/L; an average value of retained strength for substrates I and II amounts to about 83%. This is against a value of 90% for substrates III and IV. These values remain almost the same when the biotreatment is conducted using higher alkaline pectinase concentrations (up to 4 g/L). It is understandable that the higher retained strength observed with substrates III and IV is due to the polyester component of the blend. The polyester is much less affected by the biotreatment than does the cotton component. The impurities are also confined to the cotton component. Removal of such impurities from cotton would open the cotton structure, weaken the forces binding together the individual fibres in yarns bundle, destroying the cuticle and primary cell wall of the cotton fibre and allowing protruding fibres. As a result retained strength of cotton is reduced.

3.1.2. Bioscouring using binary mixtures from alkaline pectinase and cellulase

The effect of alkaline pectinase enzyme alone and in admixtures with cellulase enzyme on the performance of the bioscoured cotton-based fabrics (substrates I–IV) is examined. The results obtained are set out in Table 2.

It is seen from Table 2 that, the fabric performance of bioscoured fabrics using cellulase enzyme alone falls short with respect to all measured properties in, particular, retained tensile strength and water absorbency as compared with their corresponding properties using alkaline pectinase alone. For instance, substrates I and IV exhibit retained strength of 65.38 and 76.42%, respectively, when cellulase was used; this is against values of 83.33 and 92.85% when alkaline pectinase was used. Similarly, cellulase-bioscoured substrates I and IV required 11 s and 15 s to achieve water absorbency while the corresponding alkaline pectinase-bioscoured substrates required 5 s and 3 s.

The use of alkaline pectinase/cellulase mixture causes higher loss in fabric weight than when the two enzymes are used individually, indicating synergetic effect. This is rather observed at all ratios of the two enzymes in their mixture. It is certain, however, that

the loss in fabric weight is higher the higher ratio of cellulase enzyme in the binary mixture. On the other hand, though there are no clear-cut relations between other properties and ratios of the enzymes in the mixture, yet higher ratios of cellulase tend to bring about improved water absorbency and elongation at break; meanwhile the retained strength, whiteness and yellowness index decrease.

Based on the foregoing it may be concluded that the performance of the bioscoured cotton-based fabrics is a manifestation of :(a) type and nature of the fabric, (b) kind and nature of the enzymatic system used, (c) properties measured for assessment of the fabric performance, (d) interrelationship of measured properties and their dependence on each other, (e) interaction of the enzymatic system with the substrate and (f) mode and uniformity of the enzymatic action.

Worthy mentioning is the significant adverse effect of cellulase enzyme alone and in admixture on retained strength of all-cotton fabrics and cotton/polyester (35/65) blend fabric. While the decrease in retained strength of all-cotton fabrics is logically due to the hydrolytic attack of the cellulase enzyme on the cotton cellulose, the decrease in case of this blend fabric suggests that cellulase attacks the surfaces and accessible portions of the polyester component in the blend and, in so doing, decreases the contribution of the polyester in the tensile strength. It seems also that such attack brings about modified polyester with higher water absorbency which together with that of the cotton component constitutes the greatest water absorbency of the blend.

3.1.3. Bioscouring versus conventional alkaline scouring

A comparison is made between the bioscouring using either alkaline pectinase enzyme (method 1) alone or in admixture with cellulase enzyme (method 2) and the conventional scouring using sodium hydroxide (method 3). Fabrics used are represented by the four substrates in question. Efficiencies of the conventional scouring method and the two bioscouring methods are realized through fabric performance which, in turn, is brought into focus through determination of major technical fabric properties. The latter include loss in fabric weight, whiteness index, water absorbency, retained tensile strength and elongation at break. The results are shown in Table 3.

Results of Table 3 make it evident that fabrics scoured as per the conventional method acquire much higher losses in fabric weight than those scoured according to the two bioscouring methods. The loss in weight follows the order: conventional scouring method using NaOH (method 3) > Bioscouring method using a mixture of alkaline pectinase and cellulase (method 2) > Bioscouring method using alkaline pectinase (method 1).

It is believed that the success of the conventional scouring in removing all impurities (except the coloring matter) accounts for the above order. Bioscouring by alkaline pectinase under the conditions used would be able to remove all the waxes as it does in

Table 2Combined effect of alkaline pectinase and cellulase enzymes on some physical and mechanical properties of cotton-based fabrics.

Pec./cellu. ratio	Wigh	t loss (%	%)		Whiteness inde	х			Wat	er abso	rbency	(sec)	Retaine	ed tensile	e strengt	h (%)
	a	b	С	d	a	b	c	d	a	b	с	d	a	b	С	d
2:0	3.10	2.75	2.35	1.95	11.49 (18.61)*	-5.76 (24.28)	21.32 (15.11)	37.72 (11.45)	5	4	3	3	83.33	83.33	82.95	92.85
1.50:0.5	5.20	4.85	4.24	3.38	11.71 (18.01)	-5.36 (23.71)	22.01 (14.89)	38.11 (10.89)	4	3	2	2	82.05	81.69	81.25	75.71
1.0:1.0	6.16	4.57	6.82	4.18	12.81 (17.33)	-4.46 (22.66)	23.11 (13.41)	38.61 (10.05)	3	2	2	2	82.00	75.04	79.54	75.41
0.5:1.5	7.23	5.42	7.01	4.48	9.13 (22.91)	-8.31 (27.71)	18.01 (17.45)	33.61 (14.72)	5	5	4	4	88.9	60.25	81.25	75
0.0:2.0	2.11	2.63	1.90	1.41	7.11 (23.45)	-9.01 (28.61)	16.38 (19.22)	33.01 (14.03)	11	14	13	15	65.38	56.41	83.44	76.42

Where (a) desized cotton fabric (substrate I), (b) desized mercerized cotton fabric (substrate II), (c) desized cotton/polyester blend 50/50 (substrate III) and (d) desized cotton/polyester blend 35/65 (substrate IV).

Conditions used: [Epyptol®], 0.5 g/L; temperature, 55 °C; time, 60 min; pH, 8.5; MLR, 1:50.

^{*} Values between brackets represent the yellowness index.

Table 3Comparison of conventional alkaline scouring with bioscouring of different cotton-based fabrics.

Treatment	Wigh	t loss (%	ś)		Whiteness inde	ex			Wate	er abso	rbency	(sec)	Retained	d tensile	strength	(%)
	a	b	c	d	a	b	c	d	a	b	С	d	a	b	С	d
Desized	6.17	1.20	3.36	1.55	0.32 (24.32)*	-9.26 (28.93)	11.66 (20.59)	32.09 (15.65)	16	35	15	30	100	100	100	100
Alkaline NaOH Scouring	8.12	10.76	6.16	5.74	13.35 (16.81)	-4.32 (21.97)	25.35 (13.11)	36.62 (10.35)	3	4	4	4	93.71	93.58	92.04	96.44
Alk. Pec. 2 g/L Pec/cell. 1/1 (g/L)	3.10 6.16		2.35 6.82		11.49 (18.61) 12.81 (17.33)	, ,	` '	37.72 (11.45) 38.61 (10.05)	5 3	4 2	3 2	3 2	83.33 82.00	83.33 75.04	82.95 79.54	92.85 75.41

Conditions used: [Epyptol®], 0.5 g/L; temperature, 55 °C; time, 60 min; pH, 8.5; MLR, 1:50.

presence of cellulase and agitation. The higher water absorbency along with lower retained tensile strength observed with the bioscouring using the mixture of the two enzymes are in conformation with this. Mechanical agitation involved in bioscouring using the two enzymes enables the latter to work more efficiently.

Table 3 shows that the whiteness index and yellowness index are not much dependent upon the method of scouring employed in current work. On the other hand, these two properties differ much from one substrate to the other. They exhibit higher values with cotton/polyester blended fabrics than with all-cotton fabrics.

Meanwhile bioscouring falls short with respect to retained tensile strength as already indicated. Nevertheless for excellent water absorbency which is usually required in fast dyeing and finishing method, the bioscouring using a mixture of the two said enzymes becomes a good option.

3.1.4. Incorporation of EDTA in the bioscouring bath containing alkaline pectinase/cellulase mixture

Table 4 shows the effect on fabric performance of EDTA at different concentrations when used along with alkaline pectinase/cellulase mixture at a ratio of 1:1. Obviously, increasing the concentration of EDTA from 1 g/L to 2 g/L brings about increments in loss in fabric weight, water absorbency, whiteness index and elongation at break. At the same time the retained tensile strength and yellowness index decrease. Increasing EDTA concentration from 2 g/L to 4 g/L has practically no effect on the aforementioned properties. Hence, 2 g/L EDTA may be considered as an appropriate concentration for improved bioscouring of the four substrates under investigation using a mixture of alkaline pectinase/cellulase enzymes at a ratio 1:1.

Based on all what has been said above, presence of a chelating agent such as EDTA along with alkaline pectinase in single use or admixture with cellulase in the bioscouring solution accelerated the removal of impurities from cotton and cotton/polyester blend

fabrics. Furthermore removing calcium ions from calcium crossbridged in hemicelluloses by EDTA results in somewhat free accessible areas in the substrate where the enzyme is prompt to act in catalyzing the chain-cutting process. Thus, in this case the enzyme is capable of accessing the hemicelluloses macromolecules released during chelating process occurs at the same time.

3.1.5. Effect of β -cyclodextrin

Table 5 shows the effect of incorporation of different concentrations of β -cyclodextrin on the efficiency of enzymatic scouring using mixture of pectinase and cellulase enzymes. Such efficiency could be realized from the magnitudes of the values of technical properties of the so bioscoured fabrics. As is evident, increasing concentration of β -cyclodextrin from 2.5 g/L to 10 g/L causes increase in loss in fabric weight, fabric absorbency, whiteness index and elongation at break. On the other hand, the retained tensile strength and yellowness index decrease marginally.

The favorable effect of β -cyclodextrin especially on absorbency of water by the bioscoured fabrics could be explained as follows, the natural waxes present in the composition of desized fabrics are not likely removed during the enzymatic scouring using either alkaline pectinase or mixture of alkaline pectinase and cellulase. These waxes seem to undergo emulsification through incorporation into the cavities of β -cyclodextrin. Due to the high solubility of β -cyclodextrin by virtue of its outward pointed hydroxyl groups, waxes incorporated in β -cyclodextrin cavity becomes water soluble and amenable to removal from fabrics. In other words β -cyclodextrin has the ability to complex with waxes because of the hydrophobic nature of the interior of its cavity; in accordance with previous reports (Hashem, El-Bisi, & Hebeish, 2001).

3.2. Combined bioscouring and bleaching

As already stated, current work is targeted towards the establishment of innovative conditions for eco-friendly bleaching of cot-

Table 4Effect of EDTA concentrations on bioscouring using a mixture of alkaline pectinase and cellulase enzymes.

EDTA conc.	Wigh	t loss (%	%)		Whiteness inde	х			Wate	er abso	rbency	(sec)	Retained	l tensile s	trength (%	6)
(g/L)	a	b	с	d	a	b	с	d	a	b	c	d	a	b	С	d
Desized	6.17	1.20	3.36	1.55	0.32 (24.32)*	-9.26 (28.93)	11.66 (20.59)	32.09 (15.65)	16	35	15	30	100	100	100	100
0.0	6.16	4.99	4.82	4.18	12.81 (17.33)	-4.46 (22.66)	23.11 (13.14)	38.61 (10.05)	3	2	2	2	82.00	75.04	79.54	75.41
2	8.68	5.67	4.99	4.83	13.53 (16.34)	-4.20(22.01)	24.01 (11.21)	39.81 (9.37)	1	1	1	1	71.15	70.51	73.80	74.28
3	9.12	6.68	5.11	4.82	13.71 (16.22)	-3.91 (21.72)	24.21 (11.00)	40.13 (9.25)	1	1	1	1	67.90	69.87	73.80	74.28
4	9.25	6.91	5.19	4.90	13.79 (16.00)	-3.88 (21.74)	24.18 (10.91)	40.29 (9.11)	1	1	1	1	66.66	69.23	72.70	72.80

Where (a) desized cotton fabric (substrate I), (b) desized mercerized cotton fabric (substrate II), (c) desized cotton/polyester blend 50/50 (substrate III) and (d) desized cotton/polyester blend 35/65 (substrate IV).

^{*} Values between brackets represent the yellowness index.

^{0.0} concentrations of EDTA represent fabrics treated with a mixture of cellulase and alkaline pectinase at ratio (1:1) g/L.

Conditions used: [alkaline pectinase/cellulase enzymes at ratio (1:1)]; [Epyptol®], 0.5 g/L, temperature; 55 °C; time, 60 min; MLR, 1:50.

^{*} Values between brackets represent the vellowness index.

Table 5Effect of incorporation of β-cyclodextrin at different concentrations during bioscouring using mixture of alkaline pectinase/cellulase enzymes on technical properties of the so bioscouring data fabrics.

B-CD conc.	Wight	loss (%	()		Whiteness inde	х			Wate	er absoi	bency	(sec)	Retaine	d tensile	strengtl	n (%)
(g/L)	a	b	с	d	a	b	с	d	a	b	c	d	a	b	С	d
Desized	6.17	1.20	3.36	1.55	0.32 (24.32)*	-9.26 (28.93)	11.66 (20.59)	32.09 (15.65)	16	35	15	30	100	100	100	100
0.0	6.16	4.99	5.82	4.18	12.81 (17.33)	-4.46 (22.66)	23.11 (13.17)	38.61 (10.05)	3	2	2	2	82.00	75.04	79.54	75.41
5	7.53	6.48	7.20	4.91	13.03 (17.00)	-4.32(22.31)	23.41 (12.88)	38.66 (10.00)	2	4	3	3	76.92	73.07	79.54	72.85
7.5	7.66	6.91	7.63	5.25	13.61 (16.83)	-4.19(22.01)	23.91 (12.61)	38.91 (9.82)	2	3	2	2	70.51	71.79	78.40	69.28
10	7.81	7.35	8.17	5.42	13.95 (16.51)	-4.00 (21.91)	23.94 (12.50)	38.93 (9.90)	1	1	1	1	70.51	70.50	77.27	69.28

0.0 concentrations of β -cyclodextrin represent the fabrics treated by mixture of cellulase and alkaline pectinase enzymes in ratio (1:1) g/L.

Conditions used: [Epyptol[®]]; 15 g/L, temperature; 55 °C, time; 60 min, MLR; 1:50.

ton-containing textiles. Hence, α -amylase enzyme was used for desizing and alkaline pectinase enzyme was used for scouring. Conditions for bio-desizing and bioscouring have been thoroughly studied and presented in the foregoing chapter. The situation now necessitates optimization of eco-friendly bleaching conditions to enable development of a system for full purification (i.e. removal of non-cellulosic impurities and coloring matters) of the cotton-containing fabrics in one-stage process.

With the above in mind, optimal conditions for bleaching four cotton-based fabrics (substrates I–IV) are established using the *in situ* formation of peracetic acid. The so formed peracetic acid acts as on eco-friendly bleaching agents for cotton-containing textiles. Factors affecting such bleaching are presented below:

3.2.1. TAED concentration

Table 6 shows the effect of tetraacetylethylenediamine concentration on loss of fabric weight (WL%), whiteness index, tensile

strength, fabric absorbency and carboxyl content of four bleached cotton-based fabrics. The latter were subjected to a bleaching system involving *in situ* formation of peracetic acid. Peracetic acid is created via reaction of TAED and $\rm H_2O_2$ and constitutes the essential bleaching agent in current bleaching system.

It is clear that, WL% increases by increasing the concentration of TAED within the range studied (5–20 g/L) regardless of the substrate used. As will be shown latter the TAED concentration is related directly to the concentration of the generated peracetic acid. Then this can be looked upon as the reason behind the higher WL% observed at higher TAED concentration used. Fibre constituents of the fabric seems also to determine the magnitude of WL%; 100% cotton substrates (substrates I and II) display higher WL% particularly when relatively low TAED concentrations were used, most probably owing to removal of impurities which are mainly confirmed in the cotton component of the substrate. Higher concentration of TAED, e.g. 20 g/L eliminates differences in WL% due to fibre constituents of the fabric.

Table 6 shows also the variation in the whiteness index (WI) of the four substrates with TAED concentration. The results imply (a) that the WI increases by increasing the TAED concentration up to 15 g/L; beyond this concentration the increase in WI is very marginal, (b) that the 100% cotton substrates (substrates I and II) exhibit lower WI values than the other substrates containing polyester in cotton/polyester blend fabrics and (c) that mercerization prior to bioscoured cotton fabrics detracts from its WI as the latter exhibit the lowest values, in case of substrate II in comparison with WI values of other substrates, in particular, the bioscoured fabric (substrate I) from which it was prepared. Despite the further cotton purification by mercerization, the latter seems to change the light reflection properties of the bioscoured cotton fabrics through shrinkage and dimensional instability thereby decreasing the WI. The enhancement of WI by increasing the TAED concentration is a direct consequence of this on the amount of peracetic acid. It is understandable that peracetic acid is the main active bleaching agent in the bleaching system under investigation. It is further noted that

Table 6Effect of TAED concentration on the efficiency of bleaching process of cotton-based fabrics.

TAED conc. (g/L)	Wight	t loss (%	5)		Whiten	ness index	ĸ		Tens	sile str	ength ((kg.f)	Wate	er absoi	bency	(sec)	-СООН со	ontent (meq/100 g)
	a	b	С	d	a	b	С	d	a	b	с	d	a	b	С	d	a	b
5	1.08	1.65	0.95	0.80	41.08	30.15	45.39	58.73	64	64	73	127	2	2	1	1	31.36	25.52
10	1.36	1.73	1.24	1.11	45.13	38.62	47.76	61.27	64	64	71	126	1	1	1	1	32.71	28.48
15	2.05	1.97	1.50	1.99	57.33	53.25	60.93	67.03	62	63	71	125	<1	<1	<1	<1	37.42	35.90
20	2.11	1.99	1.67	1.98	57.55	53.22	60.34	67.27	58	63	70	120	<1	<1	<1	<1	38.58	36.11

Where (a) desized cotton fabric (substrate I), (b) desized mercerized cotton fabric (substrate II), (c) desized cotton/polyester blend 50/50 (substrate III) and (d) desized cotton/polyester blend 35/65 (substrate IV).

Conditions used: [H₂O₂]; 5 g/L, [Egyptol®], 0.5 g/L,[sodium silicate]; 2 g/L; temperature, 60 °C; time, 60 min; pH, 8; MLR; 1:50.

^{*} Values between brackets represent yellowness index values.

Table 7Effect of hydrogen peroxide concentration on the efficiency of bleaching process of bioscoured cotton-based fabrics.

H ₂ O ₂ Conc. (g/L)	Wight	loss (%))		Whiten	ess index			Tens	ile stre	ength (l	kg.f)	Abso	orbenc	y (sec)	-COOH	content (meq/100 g)
	a	a b c d 1.77 1.90 1.35 1.75			a	b	С	d	a	b	С	d	a	b	с	d	a	b
3	1.77	1.90	1.35	1.75	54.69	51.88	58.83	66.47	62	65	73	127	1	1	1	1	30.71	26.05
5	2.05	1.97	1.50	1.99	57.33	53.25	60.93	67.03	62	64	71	125	<1	<1	<1	<1	37.42	35.90
7	2.21	2.35	1.59	1.83	58.29	55.07	61.39	65.62	61	62	70	117	<1	<1	<1	<1	38.11	36.67
9	2.30	2.30	1.67	1.92	56.62	51.84	59.34	65.81	61	62	69	114	<1	<1	<1	<1	38.74	36.97

Conditions used: [TAED]; 15 g/L, [Egyptol®], 0.5 g/L, [sodium silicate]; 2 g/L; temperature, 60 °C; time, 60 min; pH; 8, MLR; 1:50.

substrates III and IV containing polyester in blend fabrics enjoy higher WI values than the 100% cotton (substrates I and II). Absence of impurities in polyester component is responsible for this.

Increasing the TAED concentration leaves the tensile strength of the said four substrates practically intact with the exception of bioscoured 100% cotton fabric which displays a decrease in its tensile strength upon using the highest TAED concentration within the range studied. This advocates the bleaching system under investigation though the values of WI are still lower than normally required in full bleached cotton textiles.

Fabric absorbency is excellent with all substrates. A water drop disappear from the surface of their substrates within one and two seconds upon using TAED at concentration of 5 g/L and lesser time than this when 10 g/L or above TAED were used.

Like cotton bleaching systems, 100% cotton fabrics (substrates I and II) treated with a system based on TAED and H_2O_2 to generate peracetic acid *in situ* exhibit values of carboxyl content ranging from ca. 25 to 38 meq/100 g cellulose. The value of carboxyl content relies on TAED and nature of the cotton fabrics.

3.2.2. Hydrogen peroxide concentration

Table 7 shows the effect of $\rm H_2O_2$ concentration on loss in fabric weight, whiteness index, tensile strength, fabric absorbency and carboxyl content of bleached cotton and cotton/polyester blend fabrics (substrates I–IV). It is clear that the WL% increases as the concentration of $\rm H_2O_2$ increases within the range studied (3–9 g/L $\rm H_2O_2$). This is observed irrespective of the substrate used. However, WL% values for 100% cotton fabrics (substrates I and II) are higher than those based on cotton/polyester blend fabrics (substrates III and IV). This is rather the trend observed above in the preceding section and could be explained on similar lines.

 $\rm H_2O_2$ concentration has practically no effect on WI of all substrates I–IV but with the certainly that the WI values follow the order: substrate IV > substrate III > substrate I > substrate II. This indicate that the lowest $\rm H_2O_2$ concentration used (i.e., 3 g/L) along with 15 g/L TAED in alkaline solution are enough to effect generation of PAA in concentration capable of bleaching any of the substrates in question. Differences among the WI values obtained with different substrates could be associated with differences in fi-

bre-fabric constituents, fabric constructions, ability to shrink and dimensional instability.

Results of tensile strength obtained with the four substrates clarify the mildness of the treatment as no serious changes in values of tensile strength are observed with all substrates except substrate IV where the tensile strength decreases from 127 kg f to 117 and 114 kg f upon using 7–9 g/L H₂O₂, respectively.

Water absorbency values obtained with all substrates amount to 1 s and less than 1 s when 3 g/L $\rm H_2O_2$ and 5 g/L $\rm H_2O_2$ were used, respectively. At the same time substrates I and II display carboxyl content values ranging from Ca. 26 meq/100 g cellulose to Ca. 38.7 meq/100 g cellulose. This is rather in accordance with the results described in the preceding section.

3.2.3. pH of bleaching medium

Table 8 shows the effect of pH of bleaching system on major technical properties of cotton and cotton/polyester blend (namely, WL, WI, tensile strength, absorbency and carboxyl content). As in the case of hydrogen peroxide and hypochlorite bleaching, pH plays a significant role in peracetic acid bleaching. Two mechanisms are proposed for the bleaching action of peracetic acid or perhydroxy anion (along with carbonyl radical). The former reaction occurs in the acidic medium while the latter occurs in alkaline medium.

Results of Table 8 depict that, the loss in fabric weight increases sharply by increasing the pH of the bleaching solution from pH 6 to pH 8 then, remains almost constant when the pH increases further to higher pH's though a marginal increment in WL values is observed at pH 10. Results of whiteness index (WI) (Table 2) reveal that, optimal WI values are achieved at pH 8; blow or above pH 8 lower WI values are observed irrespective of the substrate used. The results are in full conformation with previous reports which ascribed this to liberation of perhydroxyl anions which are mainly responsible for bleaching, can only occur in neutral and alkaline medium (Guersoy & Dayioglu, 2000; Hashem, 1999; Hashem et al. 2003). pH's higher than pH 8 decrease the values of whiteness index. This behavior of peracetic acid in a strongly alkaline pH is similar to the effect of pH on hydrogen peroxide bleaching, if the pH is beyond 10.5, the hydrogen peroxide decomposes very rapidly

Table 8 Effect of pH on the efficiency of bleaching through *in situ* formation of peracetic acid of cotton-based fabrics.

pН	Wight	loss (%)			Whiten	ess index			Tens	ile strei	ngth (k	g.f)	Wate	r absorb	ency (se	ec)	-COOH co	ntent (meq/100 g)
	a	b	c	d	a	b	с	d	a	b	С	d	a	b	С	d	a	b
6	0.23	0.12	0.73	0.08	45.64	39.22	49.06	60.95	68	68	82	128	<1	<1	<1	<1	25.96	22.76
7	0.33	0.31	0.87	1.02	56.65	51.09	60.28	66.44	66	67	80	127	<1	<1	<1	<1	30.67	28.47
8	2.05	1.97	1.50	1.99	57.33	53.25	60.93	67.03	62	65	71	125	<1	<1	<1	<1	37.42	35.90
9	1.95	2.01	1.58	2.25	57.51	53.42	60.91	67.20	61	63	71	123	<1	<1	<1	<1	38.91	37.11
10	2.31	2.11	1.60	2.54	55.51	50.11	59.94	64.27	60	62	70	122	<1	<1	<1	<1	40.00	38.81

Where (a) desized cotton fabric (substrate I), (b) desized mercerized cotton fabric (substrate II), (c) desized cotton/polyester blend 50/50 (substrate III) and (d) desized cotton/polyester blend 35/65 (substrate IV).

Conditions used: [TAED]; 15 g/L, $[H_2O_2]$; 5 g/L; $[Egyptol^{®}]$, 0.5 g/L, $[sodium\ silicate]$; 2 g/L; temperature, 60 °C; time, 60 min; MLR; 1:50. Acetic pH was adjusted using 1 mol acetic acid whereas, alkaline pH was adjusted using 1 mol NaOH.

Table 9Effect of temperature on the efficiency of combined bioscouring and bleaching of cotton-based fabrics.

Temp. (°C)	Wight	loss (%))		Whiten	ess index			Tens	ile stre	ngth (l	kg.f)	Wate	er absor	bency (sec)	-COOH co	ontent (meq/100 g)
	a	a b c d			a	b	С	d	a	b	С	d	a	b	с	d	a	b
40	1.29	1.19	0.93	0.92	47.81	43.21	55.09	64.48	64	64	72	125	2	2	1	1	20.17	23.73
50	1.86	1.70	1.31	1.15	53.09	49.82	58.08	65.86	63	62	72	125	1	1	1	1	32.37	27.51
60	2.05	1.97	1.50	1.99	57.33	53.25	60.93	67.03	62	63	71	125	<1	<1	<1	<1	37.42	35.90
70	2.13	2.07	1.56	2.01	57.31	53.31	60.45	68.65	62	62	71	124	<1	<1	<1	<1	38.62	36.08

Conditions used: [TAED]; 15 g/L, [H₂O₂]; 5 g/L, [Egyptol®], 0.5 g/L,[sodium silicate]; 2 g/L; time, 60 min; pH, 8; MLR; 1:50.

and hence the liberated perhydroxy anions escape into the bleaching medium before they can bleach the fabric (Prabaharan, Ramesh, & Venkata, 2000). It is rather possible that the perhydroxy anions interact with each other to yield un-bleaching species.

Table 8 also shows that a high degree of values in whiteness index is accompanied by perceptible loss in tensile strength and increase in carboxyl content. This could be as a result of cellulose degradation and conversion of hydroxyl groups into carboxyl and aldehyde groups.

3.2.4. Bleaching temperature

Table 9 shows the effect of temperature of the bleaching process on loss in fabric weight, whiteness index, tensile strength, water absorbency and carboxyl content of cotton and cotton/polyester blend fabrics. As is evident increasing the temperature from 40 to 60 °C causes increment in loss of fabric weight, carboxyl content, whiteness index, and absorbency of the fabrics. On the other hand, the tensile strength decreases. This is observed irrespective of the substrate used. Raising the bleaching temperature further to 70 °C has practically no effect on all these properties though whiteness index tends to remain constant at temperature range of 60–70 °C.

Enhancement of bleaching by rising the temperature up to $60\,^{\circ}\mathrm{C}$ is a direct consequence of the favorable effect of temperature on both, the *in situ* formation of peracetic acid and the decomposition of the latter to perhydroxy anion and carbonyl radical; such decomposition has been elucidate in the preceding section. The tendency of the whiteness index to remain almost constant when bleaching temperature was elevated to $70\,^{\circ}\mathrm{C}$ is a manifestation of the fast rate of decomposition of peracetic acid, a matter which is accompanied by a very rapid generation of the said active species responsible for bleaching. At such very rapid rate of generation, these species (perhydroxy anions in particular) would escape into the bleaching medium before they could bleach.

3.2.5. Duration of bleaching

Table 10 shows the effect of time of the bleaching process on the aforementioned technical properties of cotton and cotton/polyester blend fabric. The results disclose that, prolonging the time of bleaching significantly favors the bleaching process as evidenced by enhancement in loss in fabric weight (removal of impurities), whiteness index and water absorbency. At the same time the adverse effect of bleaching time on the tensile strength and carboxyl

content is not striking. In a more specific sense, prolonging the duration of bleaching from 30 to 60 min is accompanied by increments in loss in fabric weight, whiteness index, water absorbency; meanwhile the fabrics under investigation undergo certain degradation as clarified by the results of tensile strength and carboxyl content. It is understandable that creation of carboxyl groups (and aldehyde groups) occurs under the action of the bleaching species on the cellulose hydroxyls and the cellulose chain molecules, in particular on 1-4- β -glucosidic linkages. It is as well to report that increasing the time of bleaching up to 120 min leaves the said technical properties practically intact. This is the case with all substrates examined.

3.2.6. Combined of bioscouring and bleaching: pectinase concentration

Table 11 shows the effect of concentration of alkaline pectinase when used at different concentrations along with TAED and hydrogen peroxide on major technical properties of cotton-containing textiles. Alkaline pectinase together with in situ formed peracetic acid would affect concurrent bioscouring and bleaching. Hence, desized cotton and cotton blend fabrics were treated with 0.5-3 g/L alkaline pectinase enzyme, 5 g/L H₂O₂, 15 g/L TAED, 2 g/L sodium silicate and 0.5 g/L nonionic wetting agent. The bath was adjusted at pH 8 and temperature 55-60 °C and the combined bioscouring and bleaching process was allowed to proceed for 60 min. It is clear (Table 11) that increasing the concentration of alkaline pectinase from 0.5 g/L to 2 g/L is accompanied by increments in loss in fabric weight, whiteness index, water absorbency and carboxyl content. On the other hand, tensile strength decreases. Further increase in the concentration of alkaline pectinase causes marginal changes in these properties.

Results of Table 11 imply that alkaline pectinase works to effect bioscouring under conditions which are not only compatible with those of bleaching but also expedite bleaching simultaneously with bioscouring. Since the bleaching system used is environmentally friendly, this system in combination with bioscouring constitute a sound eco-friendly system for effecting scouring and bleaching of cotton-based textiles in a single-step operation.

3.2.7. Duration of the combined process

Table 12 show the effect of time on the efficiency of the combined process which comprises bioscouring using alkaline pectinase and bleaching using peracetic acid. Efficiency of the

Table 10Effect of time on the efficiency of combined bioscouring and bleaching of cotton-based fabrics.

Time (min)	Wight	loss (%)		Whiten	ess index			Tens	ile stre	ngth (kg.f)	Wate	er absor	bency (sec)	-СООН со	ntent (meq/100 g)
	a	b	С	d	a	b	с	d	a	b	с	d	a	b	c	d	a	b
30	1.04	1.58	1.68	1.88	49.83	46.48	56.78	65.08	67	66	80	127	1	1	1	1	21.79	23.56
60	2.05	1.97	1.50	1.99	57.33	53.25	60.93	67.03	62	63	71	125	<1	<1	<1	<1	37.42	35.90
90	2.34	2.21	1.68	2.10	57.51	54.90	63.11	69.04	60	65	69	124	<1	<1	<1	<1	38.58	36.81
120	2.46	2.25	1.67	2.00	59.19	55.92	62.75	69.27	60	63	67	122	<1	<1	<1	<1	39.62	37.99

Where (a) desized cotton fabric (substrate I), (b) desized mercerized cotton fabric (substrate II), (c) desized cotton/polyester blend 50/50 (substrate III) and (d) desized cotton/polyester blend 35/65 (substrate IV).

Conditions used: [TAED]; 15 g/L, [H₂O₂]; 5 g/L, [Egyptol®], 0.5 g/L,[sodium silicate]; 2 g/L; temperature; 60 min, pH; 8; MLR; 1:50.

Table 11Effect of pectinase concentration on the efficiency of combined bioscouring and bleaching of cotton-based fabrics.

A. Pec. conc.* (g/L)	Wight	t loss (%	5)		Whiten	ness inde	х		Tens	ile str	ength ((kg.f)	Wate	er abso	rbency	(sec)	-СООН со	ontent (meq/100 g)
	a	a b c d 1.46 1.77 0.65 1.93		a	b	С	d	a	b	c	d	a	b	c	d	a	b	
0.5	1.46	1.77	0.65	1.93	51.92	47.81	58.84	63.84	66	69	83	129	<1	<1	<1	<1	32.66	31.28
1	3.66	2.34	1.76	2.07	53.01	49.40	59.24	67.18	65	66	80	125	<1	<1	<1	<1	33.45	32.31
2	4.80	2.91	2.50	2.13	57.77	52.82	61.48	68.64	64	65	79	125	<1	<1	<1	<1	36.11	34.27
3	4.90	3.01	2.71	2.18	53.01	47.96	58.96	66.94	60	60	79	123	<1	<1	<1	<1	39.25	38.63

Conditions used: [TAED]; 15 g/L[H₂O₂]; 5 g/L, [Egyptol®], 0.5 g/L, [sodium silicate]; 2 g/L; temperature, 60 °C; time, 60 min; pH; 8; MLR; 1:50.

 Table 12

 Effect of time on the efficiency of combined bioscouring and bleaching of cotton-based fabrics.

Time (min)	Wight	loss (%)		Whiten	ess index	:		Tens	ile stre	ngth (kg.f)	Wate	er absor	bency (sec)	-СООН со	ntent (meq/100 g)
	a	b	c	d	a	b	с	d	a	b	с	d	a	b	с	d	a	b
30	3.68	2.28	1.84	1.91	52.24	49.44	57.25	64.66	66	66	80	126	<1	<1	<1	<1	38.63	35.41
60	4.80	2.91	2.50	2.07	57.71	52.82	61.48	68.64	64	65	79	125	<1	<1	<1	<1	36.11	34.27
90	5.10	3.07	2.71	2.14	57.75	53.11	61.57	68.69	63	64	78	123	<1	<1	<1	<1	38.51	35.11
120	5.11	3.21	2.69	2.21	57.92	53.59	61.32	68.78	62	63	76	122	<1	<1	<1	<1	39.83	36.84

Where (a) desized cotton fabric (substrate I), (b) desized mercerized cotton fabric (substrate II), (c) desized cotton/polyester blend 50/50 (substrate III) and (d) desized cotton/polyester blend 35/65 (substrate IV).

. Conditions used: [TAED]; 15 g/L, [H2O2]; 5 g/L, [alkaline pectinase enzyme]; 2 g/L, [Egyptol®], 0.5 g/L, [sodium silicate]; 2 g/L; temperature, 60 °C; time, 60 min; pH, 8; M/L; 1:50.

combined process is, indeed, a manifestation of the technical properties acquired by the cotton-based fabrics after the latter were subjected to the combined process. It is observed (Table 12) that increasing the time of treatment from 30 to 60 min accompanied by increments in loss in fabric weight, whiteness index, water

absorbency and carboxyl content. On the other hand tensile strength decreases slightly. Further increase the time of treatment causes marginal changes in these properties.

Based on results of Tables 11 and 12, it is possible to state that (a) alkaline pectinase is active in the presence of peracetic acid

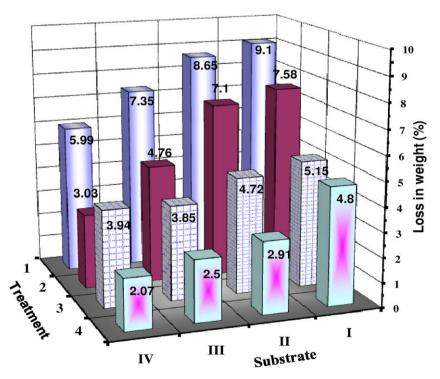


Fig. 1. Effect of different bleaching routes on total loss in fabric weight. Substrate I: desized cotton fabric. Substrate II: desized mercerized cotton fabric. Substrate III: desized cotton/polyester blend 50/50. Substrate IV: desized cotton/polyester blend 35/65. Treatment 1: scouring using NaOH then bleaching using H_2O_2 (conventional method). Treatment 2: scouring using NaOH then bleaching using TAED and H_2O_2 . Treatment 3: scouring using alkaline Pectinase then bleaching using TAED and H_2O_2 . Treatment 4: combined process of scouring and bleaching.

^{*} A. Pec. conc. = Alkaline pectinase concentration.

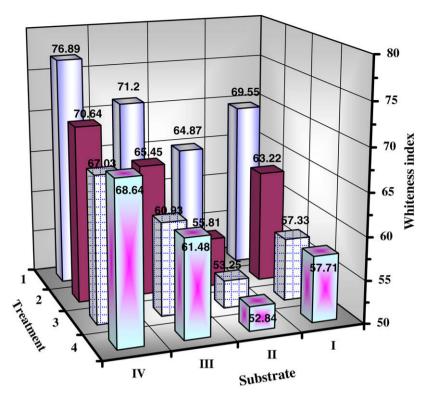


Fig. 2. Effect of different bleaching routes on the whiteness index of the fabrics. Substrate I: desized cotton fabric. Substrate II: desized mercerized cotton fabric. Substrate III: desized mercerized mercerized mercerized mercerized cotton fabric. Substrate III: desized mercerized merceriz

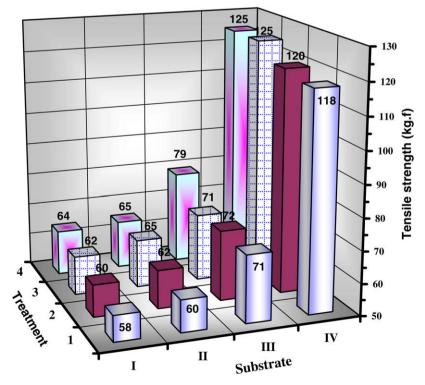


Fig. 3. Effect of different bleaching routes on the tensile strength of the fabrics. Substrate I: desized cotton fabric. Substrate II: desized mercerized cotton fabric. Substrate III: desized mercerized mercerized cotton fabric. Substrate III: desized mercerized merceri

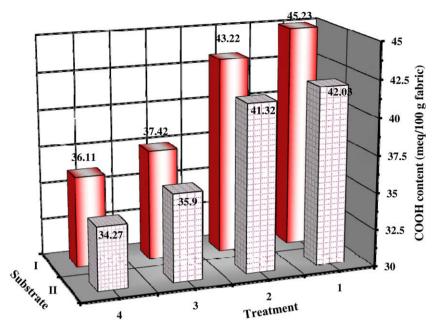


Fig. 4. Effect of different bleaching routes on the carboxyl content of the 100% cotton fabrics. Substrate I: desized cotton fabric, Substrate II: desized mercerized cotton fabric. Treatment 1: scouring using NaOH then bleaching using H_2O_2 (conventional method). Treatment 2: scouring using NaOH then bleaching using TAED and H_2O_2 . Treatment 3: scouring using alkaline Pectinase then bleaching using TAED and H_2O_2 . Treatment 4: combined process of scouring and bleaching.

formed through reaction of TAED with $\rm H_2O_2$. (b) Both alkaline pectinase and peracetic acid (formed during the treatment) contribute to removal of hydrophobic and other coloring substances from the cotton and cotton/polyester blend fabrics. (c) The highest whiteness index is obtained when the fabrics were treated with an aqueous preparatory formulation consisting of alkaline pectinase enzyme (2 g/L), TAED (15 g/L), $\rm H_2O_2$ (5 g/L), nonionic wetting agent (0.5 g/L) and sodium silicate (2 g/L). The treatment is carried out at 60 °C for 60 min. This is because of the convenient pH condition for alkaline pectinase and peracetic acid.

3.2.8. Combined bioscouring and bleaching process vis-à-vis conventional processes

In common practice, cotton-based fabrics are usually subjected to desizing, scouring and bleaching with a view to remove all natural impurities and sizes in order to prepare the fabrics for dyeing and/or finishing. The performance properties of cotton and cotton/polyester fabric pretreated by combined bioscouring and bleaching process vis-à-vis conventional and other processes were investigated. In this regards, four different pretreatment and bleaching routes were examined, these treatment are listed below:

Treatment 1: Scouring using NaOH then bleaching using H_2O_2 (conventional method).

Treatment 2: Scouring using NaOH then bleaching using TAED and H_2O_2 .

Treatment 3: Scouring using alkaline Pectinase then bleaching using TAED and H_2O_2 .

Treatment 4: Combined process of scouring and bleaching.

The results obtained are represented by Figs. 1–4.

At the first glance, results obtained reveal a number of interesting points. These are:

i. Fabrics processed as per treatment 1 display the highest values of whiteness index for all substrates examined within the range studied but this is accompanied by the highest molecular degradation of cellulose as evidenced by higher

losses in tensile strength along greater amounts of carboxyl content when treatment 1 is compared with the other three treatments in question.

- ii. Fabrics processed according to treatment 2 exhibits higher values of whiteness index than those processed as per treatment 3 or treatment 4. However fabrics prepared using treatment 2 suffers from high losses in tensile strength and greater amounts of carboxyl content as clarified by Figs. 1–4.
- iii. Fabrics processed as per treatment 4 acquire values of whiteness index which are higher than those brought about by treatments 3. Fabrics processed as per treatment 4 characterized by the lowest values with respect to loss in fabric weight, tensile strength and carboxyl content as compared with all other treatments used.

Based on the above, it is clear that treatment 4 which involves bioscouring using alkaline pectinase enzyme concurrently with bleaching by peracetic acid which is formed $in\ situ$ through reaction of TAED with H_2O_2 , is by far the best among the four treatments studied. Besides being an environmentally sound technology, treatment 4 provided a new development for purification and preparation of cotton-based fabrics.

4. Conclusion

Four cotton-based substrates in the fabric form were subjected to bioscouring by single use of alkaline pectinase enzymes or by using binary mixtures of alkaline pectinase and cellulase enzymes. Substrates under investigation comprised: (a) desized cotton fabric (substrate I), (b) desized mercerized cotton fabric (substrate II), (c) desized cotton/polyester (50/50) blend fabric (substrate III) and (d) desized cotton/polyester (35/65) blend fabric (substrate IV). The aforementioned cotton-containing fabrics (substrates I–IV) could be successfully bioscoured using 2 g/L alkaline pectinase at 55 °C for 60 min at pH 8.5 using phosphate buffer.

Results of bioscouring using different binary mixtures of alkaline pectinase and cellulase enzymes show that, the bioscoured substrates exhibit fabrics performances which are comparable

with these of the conventional alkali scouring. However, care should be taken when bioscouring is carried out using alkaline pectinase/cellulase mixture. Despite the superior water absorbency of bioscoured fabrics using the enzyme mixture, remarkable decrease in fabric tensile strength is observed. It has been also found that, incorporation of EDTA in the bioscouring solution along with mixture from alkaline pectinase and cellulase improves the performance of the bioscoured fabrics. Addition of β -cyclodextrin to the bioscouring solution using alkaline in admixtures with cellulase acts in favor of technical properties and performance of the bioscoured fabrics.

As scouring with pectinase and bleaching with PAA are carried out at the same temperature, a similar pH value and a similar time, it is so feasible that, both processes could be joined into a single combined process. In this way, consumption of water, energy, time and auxiliary agents would be lowered. The appropriate conditions for effecting combined eco-friendly scouring and bleaching of cotton-based fabrics were investigated. Fabrics used include desized cotton, desized grey mercerized, desized cotton/polyester blend 50/50 and desized cotton/polyester blend 35/65. After being desized by α -amylase enzyme, the fabrics were the target for bioscouring using alkaline pectinase enzyme, bleaching by in situ formed peracetic acid using TAED and H₂O₂ as well as concurrent bioscouring and bleaching. Results obtained declared that, The highest whiteness index is obtained when the fabrics were treated with an aqueous preparatory formulation consisting of alkaline pectinase enzyme (2 g/L), TAED (15 g/L), H₂O₂ (5 g/L), nonionic wetting agent (0.5 g/L) and sodium silicate (2 g/L). The treatment is carried out at 60 °C for 60 min. Beside the advantages of the new development with respect to major technical fabric properties compared with conventional scouring and bleaching methods, it is eco-friendly and reproducible. This advocates the new development for mill trials.

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