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Novel pre-treatment processes to promote linen-containing fabrics properties

N.A. Ibrahim [a,*], M. El-Hossamy [b], M.M. Hashem [a], R. Refai [a], B.M. Eid [a]

[a] Textile Research Division, National Research Center, El-Behouth Street, Cairo, Egypt
[b] Textile Dyeing Technology Division, Faculty of Applied Arts, Cairo, Egypt

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ABSTRACT

This study was undertaken to investigate the effect of plasma pre-treatment, followed by enzymatic treatment in the absence and presence of bleaching agent on the properties of linen and linen-containing fabrics. Different plasma gases (air, oxygen and nitrogen), enzymes (acid-cellulases, neutral-cellulase and alkaline-pectinase) as well as bleaching agents (peracetic acid and H_2O_2) were used. The changes in physico-mechanical properties, surface morphology and dyeing properties of the treated substrates have been investigated. The obtained results indicated that plasma pre-treatment followed by subsequent acid-cellulases/peracetic acid or alkaline-pectinase/H_2O_2 treatment result in: a dramatic improvement in hydrophilicity and wettability as well as in the degree of whiteness of the treated substrates, an improvement in reduction of surface roughness and extent of post-reactive dyeing, along with a weight loss and a drop in the tensile strength. The extent of surface modification as well as the changes in the above-mentioned properties are governed by the characteristics of the substrate, the plasma gas, the nature and dose of the used enzyme, as well as the type of bleaching agent and additive. The optimal treatment sequence for attaining better performance properties was O_2-plasma followed by alkaline-pectinase/H_2O_2 treatment in presence of PEG 400.

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1. Introduction

Flax is a multicellular, lustrous and very compact bast fiber with a high stiffness and excellent tensile properties. The main chemical constituents of flax fibers are α-cellulose, hemi-cellulose, lignin, pectin along with a small amounts of fats, waxes, nitrogenous compounds, residual ash and natural coloring matter (Fakin, Golob, & Kleinschek, 2006).

Linen is the yarn or the fabric made from flax fibers. Utilization of linen textiles for both the apparel and house-hold textile items are mainly attributed to their freshness, comfort, elegance, high tenacity, excellent tensile properties along with good absorbency and wicking ability (Sampaio, Shen, Bishop, Onionen, & Tazanov, 2005; Wong, Tao, Yuen, & Yueng, 2000). Blending or mixing of linen with other fibers such as cotton, viscose and polyester, is carried out for producing linen-containing textiles with desirable performance properties for certain end uses (Hashem & Ibrahim, 2002; Sampaio et al., 2005; Wong et al., 2000).

The main task of pre-treatment is to remove the hydrophobic impurities, natural or may be added for better spinnability and weavability, from the flax fibers for attaining better and uniform absorbency which is essential for subsequent wet processes. Conventional alkaline scouring is generally used for pre-treatment on an industrial scale. This process is harmful to both product and environment, therefore, there have been many trials to replace the harsh chemical processes, such as conventional scouring, with more acceptable ones taking into consideration both the economical and ecological aspects (Fakin, Golob, Kleinschek, & Marechal, 2006; Ibrahim, Abd Allah, Hassan, & Borham, 2005; Ibrahim, El-Hossamy, Morsy, & Eid 2004; Ibrahim, Fahmy, Hassan, & Mohamed, 2005). Plasma (El-Zawahry, Ibrahim, & Eid 2006; Sun & Stylios, 2004, 2005) and/or enzymatic treatments (Batog et al., 2006; Bhattacharya & Shah, 2004; Ibrahim et al., 2005; Kan, Yuen, Jiang, Tung, & Cheng, 2007; Wong et al., 2000) have been shown to be environmentally benign and promising alternatives for modifying, preparing and finishing treatments of textile materials (Batog et al., 2006; El-Zawahry et al. 2006; Sun & Stylios, 2004, 2005).

The present work is, therefore undertaken to investigate the impact of plasma pre-treatment on subsequent combined enzymatic treatment/peracetic acid or hydrogen peroxide bleaching of linen-containing fabrics as well as the changes in their properties.

2. Experimental

2.1. Materials

The specifications of linen-containing fabrics used throughout this work are shown in Table 1.

* Corresponding author.
E-mail addresses: nabibrahim@hotmail.com, nabibrahim49@yahoo.co.uk (N.A. Ibrahim).

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Table 1
Specification of the experimental fabrics

Substrate	Type	Weave	Mixing ratio	Wt/area (g/m^2)
I	Linen	Plain	100% Linen	207
II	Linen/cotton IV	Plain	50% Linen and 50% cotton	160
III	Linen/(cotton/polyester)	Plain	50% Linen warp, 50% cotton polyester (50/50) weft	250
IV	Linen/viscose	Plain	15% Linen and 85% viscose	114

Scourzyme® L (a specially developed alkaline-pectate lyase having an activity of 375 APSU/g), DeniMax BT (a neutral-cellulase having activity of 1100 DAU/g) and Cellusoft® L (an acid-cellulase with an activity of 750 EGU/g) were kindly supplied from Novozymes.

Hostapal® CV-ET (non-ionic wetting agent based on alkaryl polyglycol ether), Hostapal N 100 (Industrial cleaner, emulsifier, wetting agent and dispersion based on nonylphenol polyglycol ether), and Sirrix® 2 UD liq (organic special product acts as a sequestrant for metal ions) were kindly supplied from Clariant. Leucophor® PC (Sandoz, presently Clariant) (C.I. FBA 134) an anionic stilbene derivative with affinity for cellulosic textiles was used.

Tetra acetyl ethylene diamine (TAED), Hydrogen peroxide (35%), sodium carbonate, sodium sulfate, sodium dihydrogen phosphate and disodium dihydrogen phosphate, glucose, polyethylene glycol PEG 400, citric acid, 2,2′-bipyridine and acetic acid, used were of commercial grade.

C.I. Reamzol Red 239 (hetero-bifunctional reactive dye) was kindly supplied by DyStar.

2.2. Methods

2.2.1. Plasma treatment

The system used to study the atmospheric pressure dielectric barrier discharge APDBD consisted of two stainless steel plates, each 4 × 15 cm. The lower plate was covered by a dielectric plate of 1 mm thickness. The sample was placed between the two electrodes and separated from the upper electrode with a Teflon spacer of 2 mm thickness. The system was placed in a rectangular Pyrex glass enclosure into which the working gas was introduced to pass through the gap between the electrodes. The exhaust gas was carried via plastic tubing to the fuming cupboard. The electrodes were connected to the power supply. The plasma was created by using AC source power supply with frequency 20,000 Hz, 50 W and an output of 5 kV/20 mA. The plasma reactor system used is schematically shown in Fig. 1.

2.2.2. Combined biofinishing and bleaching with peracetic acid in one bath

APDBD-treated fabric samples and untreated samples were bleached and biopolished in one bath as follow: the bath contained TAED (8.75 g/L), hydrogen peroxide 35% (4.37 ml/L), acid-cellulases (0–3% owf) and non-ionic wetting agent (2 g/L) in the presence or absence of glucose, PEG 400 or 2,2′-bipyridine, using material-to-liquor ratio (1:20 or 1:40) at pH 5 for 1 h at 50 °C. Then the pH of the treated bath was adjusted to pH 10 and the temperature was gradually raised to 95 °C (2 °C/min) and kept for 20 min at these conditions to utilize the residual H_2O_2 as well as to inhibit the enzyme (Fig. 2). The treated samples were washed twice with hot and cold water and finally air dried.

2.2.3. Combined bioscouring and bleaching with hydrogen peroxide in one bath

APDBD-treated fabric samples and untreated samples were bioscoured and bleached in one bath. The process was carried out as follows: the bath contained Scourzyme® L (0–1.0% owf) or DeniMax BT (2% owf), hydrogen peroxide 35% (0–10 ml/L) and non-ionic wetting agent (1 g/L) at pH 8.5, LR (1:20 or 1:40) at 20 °C for 10 min then the temperature was raised gradually to 60 °C and kept for 15 min. Sodium carbonate (2 g/L), chelating agent (1 g/L) and emulsifier (1 g/L) were then added to the bath in the presence or absence of glucose (2 g/L), PEG 400 (2 g/L) or citric acid (0.5 g/L), and the temperature was raised gradually to 95 °C and kept for 45 min. The treated samples were rinsed and washed thoroughly and finally air dried (Fig. 3).

2.2.4. Dyeing of fabric samples

Portion of treaded fabric samples were dyed using reactive dye (C.I. Remazol Red 239) according to the conventional exhaustion method. Reactive dyeing bath solution containing 2% dye (owf), 40 g/L sodium sulfate and 15 g/L sodium carbonate was used. The dyeing process was performed at 70 °C for 45 min using a material-to-liquor ratio 1:30. After dyeing, the dyed samples were thor-

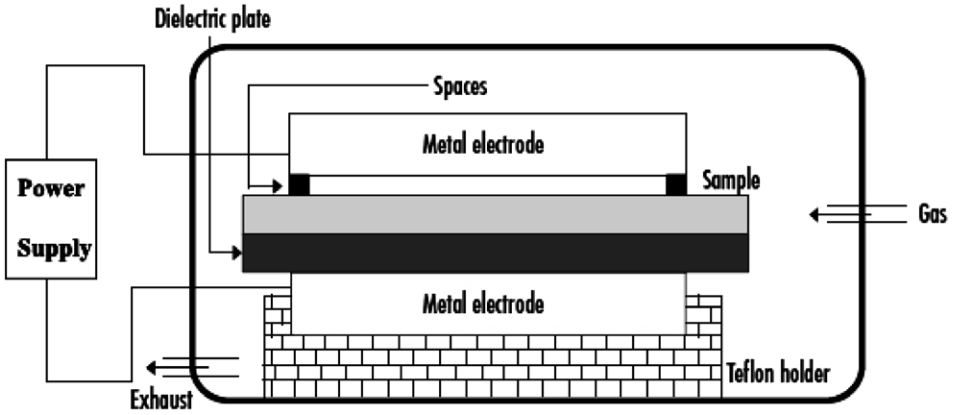


Fig. 1. A schematic diagram of the dielectric barrier discharge plasma system.

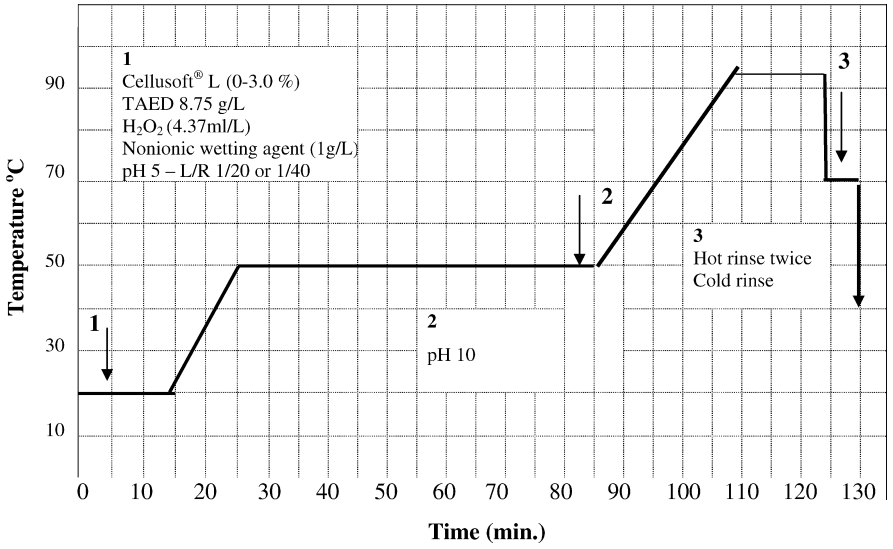


Fig. 2. One bath biopolishing and peracetic acid bleaching.

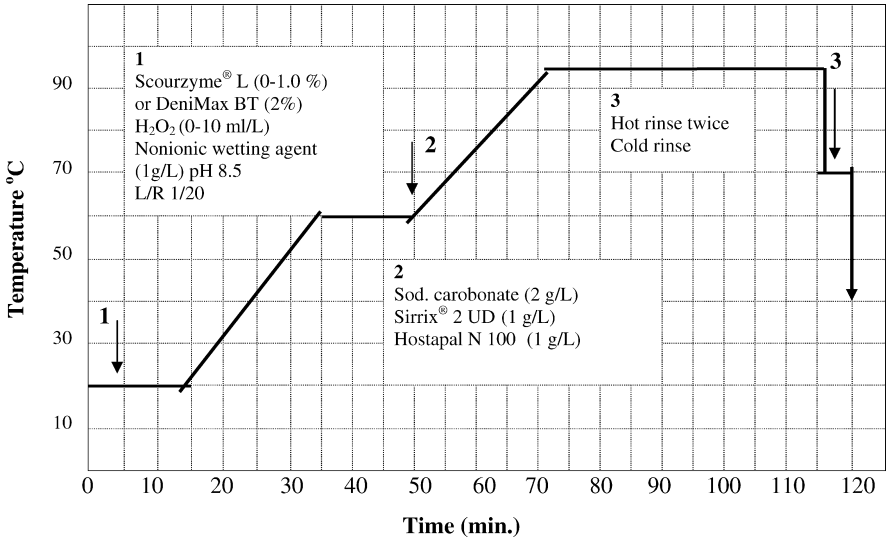


Fig. 3. One bath bioscouring and hydrogen peroxide bleaching.

oughly washed, in the presence of 2 g/L non-ionic detergent, at 90 °C for 10 min, rinsed with cold water and finally air dried.

2.2.5. Optical brightening

Portion of treaded fabric samples were optically brightened using Leucophor® PC (0.5% owf), and acetic acid (1 ml) at 100 °C for 30 min using a material-to-liquor ratio 1: 20.

2.3. Testing

The wettability was assisted using AATCC Test Method 39-1980. Weight loss of fabric was determined by the following equation:

$$\text{Weight loss}(\%) = \frac{W_b - W_a}{W_b} \times 100$$

where W_a is the weight after treatment and W_b is the weight before treatment. Tensile strength was determined using Instron Corporation 4435 Series IX Automated Materials Testing System, USA. According to D 5034-1995. Roughness of the treated and untreated samples was measured using roughness measuring instrument, SE 1700α, Japan, according to JIS 94 Standard. The whiteness and yellowness index were evaluated by using Colour-Eye® 3100 spectrophotometer supplied by SDL Inter, England, according to the Standard Test Method ASTM E313. Dyeability of the treated and untreated fabric samples was determined by measuring *K*/*S* values (*K*, absorption coefficient; *S*, scattering coefficient) at wavelength of maximum absorbance for the used dyes, with Colour-Eye® 3100 Spectrophotometer supplied by SDL Inter, England. A scanning electron microscope (SEM) examination was carried out for the treated and untreated, control, linen fabric samples by mounting the samples on stub with double stick adhesive tape and coated with gold in a S150A Sputler Coater Unit (Edwards, UK). The gold film thickness was 150 Å. The samples were then viewed in a JEAOL JXA-840A electron probe microanalyser.

3. Results and discussion

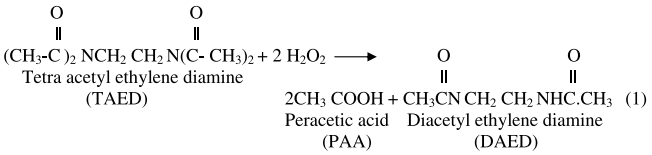
Since the main task of the present study is concerned with the development and promotion of eco-friendly preparation methods

for linen and linen-containing fabrics, a wide range of factors such as type of plasma, type of enzyme, type of bleaching agent, type of additives, type of substrate as well as sequence of treatment have been studied. Results obtained along with their appropriate discussion follow.

3.1. Effects of plasma and subsequent combined acid-cellulases/peracetic acid treatment

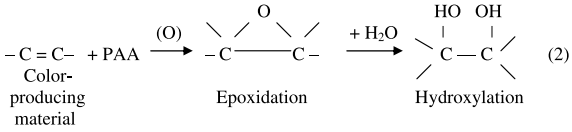
3.1.1. Air-plasma pre-treatment

For a given set of plasma pre-treatment and subsequent acid-cellulases treatment in the absence and presence of the in situ prepared peracetic acid (Scarborough & Mathews, 2000; Steiner, 1995), Table 2 demonstrates that (i) pre-treatment of the used substrates with air-plasma followed by acid-cellulases treatment results in a remarkable improvement in the wetting time (from >120 s down to <1 s), as a direct consequence of plasma etching, creation of hydrophilic active groups such as –COOH groups along with the removal of hydrophobic non-cellulosic impurities thereby increasing the hydrophilicity of the treated surface regardless of the used substrate (Wong et al., 2000); (ii) the decrease in the surface roughness of the treated fabric samples may be attributed to partial breakage of surface bio-polymer molecules during the plasma exposure along with the subsequent biopolishing effect of the acid-cellulases enzyme thus making the surface more smooth (Hashem & Ibrahim, 2002; Wong et al., 2000), and the extent of improvement is determined by the nature of the substrate, i.e. weight, fabric construction, surface area, chemical composition, crystalline/amorphous region ratio, cellulose content, non-cellulosic impurities as well as extent of modification (Sun & Stylios, 2005); (iii) the extent of improvement in the surface roughness follows the decreasing order: linen/viscose > linen/(polyester/cotton) > linen cotton > linen; (iv) plasma pre-treatment followed by enzymatic treatment is accompanied by a decrease in yellowness index along with an increase in the whiteness index values regardless of the used substrate, which reflects the positive impact of the subsequent treatments via partial oxidation/removal of yellow and colored impurities, and the extent of the improvement is governed by the type of substrate, i.e. linen viscose > linen/(polyester/cotton) > linen cotton > >linen (Wong, Tao, Yuen, & Yeung, 1999); (v) inclusion of TAED (8.75 g/L) and H_2O_2 (4.38 ml/L) in the enzymatic bath for in situ formation of peracetic acid according to the following reaction (Scarborough & Mathews, 2000; Steiner, 1995):



(1)

results in: a slight decrease in the surface roughness, a dramatic increase in WI along with a significant decrease in YI values of the treated fabric samples as a direct consequence of destroying one or more of the double bonds in the conjugated chains of the color-producing organic materials, by rupturing the conjugated chain, and/or by oxidation of one of the other moieties as follows (Far, Smith, & Stechen 1992; Hashem, 2001; Hickman, 2002; Presa & Tavcer, 2008):



(2)

and (vi) the extent of post-reactive dyeing, expressed as *K*/*S* values is determined by the extent of purification and modification of the treated substrates, availability and accessibility of the dyeing active sites, extent of dye penetration and diffusion as well as interaction, affinity of the treated substrate components to the used reactive dye as well as the variation in both the WI and YI values of these substrates.

3.1.2. Enzyme dose and material-to-liquor ratio (LR)

Table 3 shows that (i) increasing the acid-cellulases dose, from 0% up to 3% owf, in the subsequent enzymatic/peracetic acid treatment step brings about an improvement in the reduction of the treated fabric roughness, as well as in extent of bleaching along with an increase in the extent of dye uptake without any variation in extent of wetting, regardless of the used substrate; (ii) the enhancement in the aforementioned properties reflects the positive effects of acid-cellulases on catalyzing the removal of the surface loose fibrils in addition to the removal of more cementing materials from the multicellular flax fibers pre-treated with the plasma thereby increasing the smoothness, partial removal of lignin along with hemicelluloses thereby attaining better whiteness index, as well as efficient removal of hydrophobic impurities and hydrolysis of excessive fibrillar matter on the yarn surfaces which facilitates the availability of more accessible dyeing sites, i.e. hydroxyl groups, thereby improving the dye uptake (Bhattacharya

Table 2
Effect of air-plasma treatment and subsequent enzymatic treatment in the absence and presence of peracetic acid on linen-containing fabrics properties

Treatment conditions	Type of substrate	Wt (s)	Ra (μm)	WI	YI	*K*/*S*
Untreated (control)	Linen 100%	>120	29.22	−55.34	–	–
	Linen/cotton	>120	27.52	−23.24	–	–
	Linen/(polyester/cotton)	>120	25.08	11.67	–	–
	Linen/viscose	>120	21.83	11.35	–	–
Air-plasma treatment followed by acid-cellulases	Linen 100%	<1	25.94	32.35	–	–
	Linen/cotton	<1	25.02	−5.37	–	–
	Linen/(polyester/cotton)	<1	19.54	21.11	18.50	–
	Linen/viscose	<1	18.47	22.04	17.76	–
Air-plasma treatment followed by acid-cellulases/peracetic acid	Linen 100%	<1	23.41	28.48	13.59	9.15
	Linen/cotton	<1	21.63	35.09	11.27	6.65
	Linen/(polyester/cotton)	<1	18.64	40.65	8.95	5.18
	Linen/viscose	<1	18.20	52.68	5.56	8.52

Plasma treatment: Gas (air), APDBD plasma; power supply with frequency 20,000 Hz, 50 W and an output of 5 kV/20 mA, for 30 s. *Enzymatic treatment:* Acid-cellulases (3% owf), TAED (8.75 g/L); H_2O_2 (4.38 ml/L); non-ionic wetting agent (2 g/L); pH (5); LR (1/40); temperature (50 °C); time (60 min). Wt, wetting time; Ra, roughness; WI, whiteness index; YI, yellowness index; *K*/*S*, color strength of dyed samples (λ = 540).

Table 3
Effect of enzyme dose and material-to-liquor ratio of subsequent enzymatic treatment on linen-containing fabrics properties

Substrate	Enzyme dose (%)	L/R	Wt (s)	Ra (μm)	WI	YI	*K*/*S*
Linen 100%	0	1:40	<1	23.89	25.16	14.24	8.75
	3	1:40	<1	23.41	28.48	13.59	9.15
	3	1:20	<1	21.73	31.24	12.79	9.35
Linen/cotton	0	1:40	<1	22.09	33.25	11.85	5.37
	3	1:40	<1	21.63	35.09	11.27	6.65
	3	1:20	<1	20.33	36.88	11.05	6.86
Linen/(cotton/polyester)	0	1:40	<1	19.22	36.76	9.45	4.83
	3	1:40	<1	18.64	40.65	8.95	5.18
	3	1:20	<1	18.10	41.96	8.66	5.60
Linen/viscose	0	1:40	<1	19.15	48.23	6.23	8.15
	3	1:40	<1	18.20	52.68	5.56	8.52
	3	1:20	<1	17.38	53.92	5.22	9.03

Plasma treatment: Gas (air), APDBD plasma; power supply with frequency 20,000 Hz, 50 W and an output of 5 kV/20 mA, for 30 s. TAED (8.75 g/L); H_2O_2 (4.38 ml/L); non-ionic wetting agent (2 g/L); pH 5; temperature, 50 °C; time, 60 min. Wet, wetting time; Ra, roughness; WI, whiteness index; YI, yellowness index; *K*/*S*, color strength of dyed samples (λ = 540).

& Shah, 2004; Diller, Zeronian, Pan, & Yoon, 1994; Hashem & Ibrahim, 2002); (iii) the lower the material-to-liquor ratio, the better will be the performance properties of the treated fabrics as a direct consequence of increasing the extent of enzymatic attack thereby leading to extensive surface peeling and interfibrillar splitting, greater removal of non-cellulosic as well as creating more available and accessible active surfaces and (iv) the extent of improvement in the above-mentioned properties is governed by the plain weave structure, accessibility to both the plasma and subsequent combined acid-cellulases/PAA treatment, along with its chemical composition and components as discussed before.

3.1.3. Additives

Table 4 shows the effect of including certain additives namely 2,2′-bipyridine, PEG 400, and glucose in the subsequent enzymatic treatment/PAA-bleaching combined bath on the performance properties of the treated substrates. For a given set of treatment conditions, it is clear that: (i) wettability of the treated substrate was excellent (<1 s) in the absence or presence of nominated additives, (ii) the increase in surface roughness values is determined by the kind of additive and it follows the decreasing order: glucose > 2,2′-bipyridine > none > PEG 400, (iii) the enhancement in whiteness degree follows the decreasing order: 2,2′-bipyridine > PEG 400 > glucose > none, irrespective of the used substrate, which may be attributed to the positive role of 2,2′-bipyridine as an efficient chelating agent thereby facilitating the dissociation for peracetic acid as well as improving its bleaching efficiency (Hashem, 2001), (iv) the lower degree of whiteness achieved by adding glucose can be discussed in terms of its reducing power thereby accelerating the decomposition of peracetic acid and (v) post-dyeing of the treated substrates with the nominated reactive dye, expressed as *K*/*S* values, is affected by the type of additive and could be ranked as follows: glucose > PEG 400 > none > 2,2′-bipyridine, reflecting the differences among the used additives in enhancing: the extent of modification of the treated substrates, even penetration and distribution of dye molecules within the fabric structure, availability and accessibility of the fabric active sites as well as the extent of interaction and fixation via covalent bond.

3.1.4. Plasma gas

As far as the changes in the linen-containing properties as a function of pre-treatment plasma gas, the data in Table 5 signify that: (i) pre-treatment with plasma enhances the hydrophilicity of the treated substrates, regardless of the used plasma gas and keeping the subsequent treatment constant as a direct consequence of modifying of the treated fabrics surfaces, (ii) the roughness of the treated substrates follows the decreasing order: O_2-plasma > N_2-plas-

Table 4
Effect of subsequent treatment components on linen-containing fabrics properties

Substrate	Additives (2 g/L)	Wt (s)	Ra (μm)	WI	YI	*K*/*S*
Linen 100%	Without	<1	21.73	31.24	12.79	9.35
	2,2′-Bipyridine	<1	23.05	35.23	10.22	9.12
	PEG 400	<1	21.52	34.79	10.82	9.58
	Glucose	<1	25.22	33.05	11.07	9.80
Linen/cotton	Without	<1	20.33	36.88	11.05	6.86
	2,2′-Bipyridine	<1	21.12	40.02	9.11	6.55
	PEG 400	<1	20.01	38.39	10.73	6.98
	Glucose	<1	22.51	37.76	9.77	7.10
Linen/(cotton/polyester)	Without	<1	18.10	41.96	8.66	5.60
	2,2'-Bipyridine	<1	20.52	45.82	6.04	5.50
	PEG 400	<1	17.68	44.22	7.55	5.82
	Glucose	<1	21.21	42.52	8.05	5.98
Linen/viscose	Without	<1	17.38	53.92	5.22	9.03
	2,2′-Bipyridine	<1	18.51	57.06	5.03	8.80
	PEG 400	<1	17.05	55.28	5.31	9.39
	Glucose	<1	19.01	54.64	5.44	9.62

Plasma treatment: Gas (air), APDBD plasma; power supply with frequency 20,000 Hz, 50 W and an output of 5 kV/20 mA, for 30 s. *Enzymatic treatment:* Acid-cellulases (3% owf), TAED (8.75 g/L); H_2O_2 (4.38 ml/L); non-ionic wetting agent (2 g/L), pH (5); LR (1/20); temperature (50 °C), time (60 min). Wet, wetting time; Ra, roughness; WI, whiteness index; YI, yellowness index; *K*/*S*, color strength of dyed samples (λ = 540).

Table 5
Effect of using different gases in the plasma pre-treatment

Type of gas	Type of fabrics	Wt (s)	Ra (μm)	WI	YI	*K*/*S*
Without	Linen 100%	2.90	21.68	27.84	15.16	9.02
	Linen/cotton	3.27	19.81	35.76	11.63	6.57
	Linen/(polyester/cotton)	1.47	17.50	40.60	9.01	5.66
	Linen/viscose	1.00	16.82	46.45	6.95	7.25
N_2	Linen 100%	<1	22.78	28.39	15.79	9.83
	Linen/cotton	<1	22.06	36.88	11.05	7.38
	Linen/(polyester/cotton)	<1	17.89	41.96	8.66	6.05
	Linen/viscose	<1	17.71	47.76	6.77	9.76
O_2	Linen 100%	<1	23.70	38.21	10.79	9.72
	Linen/cotton	<1	22.87	44.88	7.05	7.18
	Linen/(polyester/cotton)	<1	19.42	49.90	4.82	5.94
	Linen/viscose	<1	18.79	57.06	3.98	9.55
Air	Linen 100%	<1	21.82	34.79	10.82	9.58
	Linen/cotton	<1	20.01	38.39	10.73	6.98
	Linen/(polyester/cotton)	<1	17.68	44.22	7.55	5.82
	Linen/viscose	<1	17.05	55.28	5.31	9.39

Plasma treatment: Gas (air), APDBD plasma; power supply with frequency 20,000 Hz, 50 W and an output of 5 kV/20 mA, for 30 s. *Enzymatic treatment:* Acid-cellulases (3% owf), TAED (8.75 g/L), H_2O_2 (4.38 ml/L), PEG 400 (2 g/L), non-ionic wetting agent (2 g/L), pH (5), LR (1/20), temperature (50 °C), time (60 min). Wet, wetting time; Ra, roughness; WI, whiteness index; YI, yellowness index; *K*/*S*, color strength of dyed samples (λ = 540).

ma > air-plasma > none, which may be attributed to the etching action of O_2- and N_2-plasma thereby leading to a roughening effect on the fiber surface compared with untreated counter parts (Sun & Stylios, 2005), (iii) the extent of improvement in the degree of whiteness of the plasma-treated substrates increased significantly in the order of: none < N_2-plasma < air-plasma < O_2-plasma pre-treatment, which reflects the differences among the used plasma gases in: physical and chemical modification of fabric surface, providing a more reactive surfaces, enhancing the extent of removal of hydrophobic and colored impurities as well as in enhancing the efficiency of subsequent biopolishing/PAA-bleaching step and (iv) the variation in WI and YI and *K*/*S* values is determined by the characteristics of the treated substrate and its amenability and successibility to the suggested treatment regime.

3.2. Effect of plasma pre-treatment and subsequent bioscouring/H_2O_2-bleaching

3.2.1. O_2-plasma pre-treatment

The changes in some performance properties of linen-containing fabrics treated with O_2-plasma followed by bioscouring and H_2O_2-bleaching in one step are shown in Table 6. For a given set of pre- and subsequent treatments conditions, it can be observed that: both O_2-plasma pre-treatment followed by subsequent alkaline-pectinase in the absence or presence of H_2O_2 as well as combined bioscouring/H_2O_2-bleaching result in a significant improvement in the wetting time from >120 s down to<1 s, as well as remarkable improvement in the whiteness index of the treated substrates, (ii) the enhancement in the wettability properties is a direct consequence of improving the surface properties by the O_2-plasma as well as degradation and removal of pectin and pectinous gums thereby facilitating the removal of the hydrophobic, non-cellulosic materials (Ibrahim et al., 2004, 2005), (iii) the improvement in the WI values of the treated substrates is a direct consequence of the oxidation effect of O_2-plasma as well as the generation of the perhydroxyl anions $(HO_2)^-$, during the bioscouring/H_2O_2 step which are the active species in the bleaching thereby enhancing the extent of oxidative decoloration of the colored substances according to the following reactions (Choudhury, 2006; Ibrahim et al., 2004, 2005):

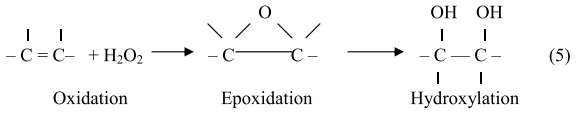
$$H_2O_2 \underset{H^+}{\overset{OH^-}{\rightleftharpoons}} H^+ + HO_2^- \quad (3)$$

$$HO_2^- + H_2O_2 \rightarrow \cdot OH + \cdot O_2^- + H_2O \quad (4)$$

Table 6
Effect of O_2-plasma treatment and subsequent bioscouring in the absence and presence of H_2O_2 on linen-containing fabrics properties

Treatment	Type of substrate	H_2O_2 (ml/L)	Properties			
			Wt (s)	WI_b	WI_a	*K*/*S*
Untreated (blank)	Linen 100%	–	>120	−55.34	–	–
	Linen/cotton	–	>120	−23.24	–	–
	Linen/(polyester/cotton	–	>120	11.67	–	–
	Linen/viscose	–	>120	11.35	–	–
Plasma pre-treatment	Linen 100%	0	<1	−37.99	–	–
followed by		5	<1	26.12	35.77	9.44
enzymatic		10	<1	29.40	40.20	9.53
scouring +	Linen/cotton	0	<1	−8.96	–	–
H_2O_2-bleaching		5	<1	31.10	41.29	6.71
		10	<1	35.77	48.09	6.93
	Linen/(polyester/	0	<1	11.95	–	–
	cotton)	5	<1	36.25	47.20	6.04
		10	<1	38.66	50.00	5.82
	Linen/viscose	0	<1	13.28	–	–
		5	<1	47.12	68.94	5.68
		10	<1	49.97	71.24	5.24
Enzymatic scouring	Linen 100%	10	<1	25.34	37.02	8.43
+ H_2O_2 bleaching	Linen/cotton	10	<1	27.94	33.47	6.54
	Linen/(polyester/cotton	10	<1	33.17	51.93	5.09
	Linen/viscose	10	<1	45.91	67.03	5.88

Plasma treatment: Gas (oxygen); APDBD plasma; power supply with frequency 20,000 Hz, 50 W and an output of 5 kV/20 mA, for 30 s. *Enzymatic treatment:* Step1: Scourzyme® L, 0.5% owf; H_2O_2, 0, 5 and 10 ml/L; non-ionic wetting agent, 2 g/L; pH 8.5; LR ,1/40; temperature, 60 °C; time, 15 min. Step 2: chelating agent, 2 g/L; emulsifier, 1 g/L; sodium carbonate, 2 g/L; temperature, 95 °C; time, 45 min. Wet, wetting time; WI_b, whiteness index before optical brightening; WI_a, whiteness index after optical brightening; *K*/*S*, color strength of dyed samples before optical brightening.



(5)

(iv) increasing the H_2O_2 concentration, from 0 up to 5 ml/L, in the bioscouring bath brings about a sharp increase in the WI values of the treated substrates and the extent of decrement is determined by the nature and characteristics of the used substrates and can be ranked as follows: linen/viscose>linen/(polyester/cotton) > linen

cotton > linen, (v) further increase in H_2O_2 concentration, up to 10 ml/L, improves the extent of oxidation and removal of the colored matter thereby upgrading the degree of whiteness, (vi) post-reactive dyeing of the pre-bleached substrates, expressed as *K*/*S* values, is governed by both the extent of bleaching as well as the type of substrate and follows the decreasing orders: in the presence of H_2O_2 > in the absence, and linen > linen cotton > linen/viscose > linen/(polyester/cotton), respectively and (vii) optical brightening of the bleached substrates results in an outstanding improvement in the whiteness index values which reflects the positive impact of the used optical whitening agent on compensating the yellowish cast and at the same time improving the lightness of the treated substrate (Choudhury, 2006; Ibrahim et al., 2004).

3.2.2. Material-to-liquor ratio (LR)

Table 7 shows the effect of using different LRs on the studied properties of the treated fabric samples. Obviously, decreasing the LR from 1/40 to 1/20 is accompanied by a noticeable increase in the WI values, before and after optical brightening, in the depth of shade, i.e. *K*/*S* values, of bleached post-dyed fabric samples with no change in wettability. The enhancement in the WI and *K*/*S* values reflects the positive effect of maximizing the ingredients concentrations, enzyme/H_2O_2 concentration, in the subsequent enzymatic treatment and the dye or the optical brightener in post-dyeing or optical brightening, thereby giving rise to higher extent of modification and cleaning of the treated substrates making them more amenable and accessible to subsequent dyeing or optical brightening. On the other hand, the variation in the aforementioned properties values, keeping other parameters fixed is determined by the nature of the substrate as discussed earlier.

3.2.3. Enzyme dose

For a given set of pre-treatment and subsequent treatment conditions, Table 8 shows that increasing the alkaline-pectinase dose up to 0.5% owf results in a noticeable increase in WI before and after optical brightening as well as in *K*/*S* values of bleached post-dyed fabric samples, regardless of the use substrate. The enhancement in the above-mentioned properties reflects the positive role O_2-plasma in modifying the fabric surface as well as in partial oxidation and removal of some hydrophobic impurities, followed by the extra action of both the alkaline-pectinase, which has the ability to hydrolyze and transform the water-insoluble polygalacturonic acid into water-soluble oligomers thereby enhancing the release of other hydrophobic non-cellulosic impurities away from the wall, as well as facilitating and increasing the extent of H_2O_2-bleaching via creation of more available active-surface area for further modification (Bhattacharya & Shah, 2004; Ibrahim et al.,2004, 2005). Further increase in enzyme dose, i.e. up to 1% owf, has a slight positive effect on the aforementioned properties.

Table 7
Effect of material-to-liquor ratio in subsequent bioscouring/H_2O_2 treatment on the linen-containing fabrics properties

Substrate	L:R	Properties			
		Wt (s)	WI_b	WI_a	*K*/*S*
100% Linen	1:20	<1	36.50	49.28	9.53
	1:40	<1	31.40	42.40	9.15
Linen/cotton	1:20	<1	39.88	51.63	7.42
	1:40	<1	35.77	48.09	6.93
Linen/(cotton/polyester	1:20	<1	43.09	61.54	6.16
	1:40	<1	38.66	50.00	5.56
Linen/viscose	1:20	<1	52.68	75.23	6.89
	1:40	<1	49.97	71.24	6.39

Plasma treatment: Gas (oxygen); APDBD plasma; power supply with frequency 20,000 Hz, 50 W and an output of 5 kV/20 mA, for 30 s. *Enzymatic treatment:* Step1: Scourzyme® L, 0.5% owf; H_2O_2, 10 ml/L; non-ionic wetting agent, 2 g/L; pH, 8.5; temperature, 60 °C; time, 15 min. Step 2: chelating agent, 2 g/L; emulsifier, 1 g/L, sodium carbonate, 2 g/L; temperature, 95 °C; time, 45 min. Wet, wetting time; WI_b, whiteness index before optical brightening; WI_a, whiteness index after optical brightening; *K*/*S*, color strength of dyed samples before optical brightening.

Table 8
Effect of enzyme dose in subsequent bioscouring/H_2O_2-bleaching on the linen-containing fabrics properties

Enzyme concentration (% owf)	Type of substrate	Properties			
		Wt (s)	WI_b	WI_a	*K*/*S*
0	Linen 100%	<1	32.02	43.10	8.95
	Linen/cotton	<1	35.29	47.60	6.72
	Linen/(polyester/cotton)	<1	38.00	51.33	5.43
	Linen/viscose	<1	46.29	66.00	6.05
0.5	Linen 100%	<1	36.50	49.28	9.53
	Linen/cotton	<1	39.88	51.63	7.42
	Linen/(polyester/cotton)	<1	43.09	61.54	6.16
	Linen/viscose	<1	52.68	68.42	6.89
1.0	Linen 100%	<1	37.22	50.05	9.89
	Linen/cotton	<1	40.43	57.80	7.79
	Linen/(polyester/cotton)	<1	44.75	63.85	6.46
	Linen/viscose	<1	54.50	71.00	7.15

Plasma treatment: Gas (oxygen); APDBD plasma; power supply with frequency 20,000 Hz, 50 W and an output of 5 kV/20 mA, for 30 s. *Enzymatic treatment:* Step1: H_2O_2, 10 ml/L; non-ionic wetting agent, 2 g/L; pH 8.5; LR, 1/20; temperature, 60 °C; time, 15 min. Step 2: chelating agent, 2 g/L; emulsifier, 1 g/L, sodium carbonate, 2 g/L; temperature, 95 °C; time, 45 min. Wet, wetting time; WI_b, whiteness index before optical brightening; WI_a, whiteness index after optical brightening; *K*/*S*, color strength of dyed samples before optical brightening.

3.2.4. Type of additive

Table 9 shows the effect of inclusion of certain additives namely glucose (2 g/L), PEG 400 (2 g/L) and citric acid (0.5 g/L) in the bioscouring/H_2O_2-bleaching bath. For a given set of plasma pre-treatment and subsequent enzymatic treatment conditions, the obtained results disclose that: (i) inclusion of any of the aforementioned additives has practically no effect on the extent of wetting, (ii) the extent of improvement in the whiteness values is governed

Table 9
Effect of inclusion of certain additives in subsequent bioscouring/H_2O_2-bleaching on the linen-containing fabrics properties

Type of substrate	Type of additives	Properties			
		Wt (s)	WI_b	WI_a	*K*/*S*
Linen 100%	Without	<1	36.50	49.28	9.53
	Glucose (2.0 g/L)	<1	33.79	45.55	8.82
	PEG 400 (2.0 g/L)	<1	47.22	63.80	9.89
	Citric acid (0.5 g/L)	<1	45.08	60.83	9.65
Linen/cotton	Without	<1	39.88	51.63	7.42
	Glucose (2.0 g/L)	<1	36.50	47.18	7.20
	PEG 400 (2.0 g/L)	<1	52.62	68.22	7.94
	Citric acid (0.5 g/L)	<1	50.99	66.05	7.80
Linen/(cotton/polyester	Without	<1	43.09	61.54	6.16
	Glucose (2.0 g/L)	<1	39.15	50.45	5.85
	PEG 400 (2.0 g/L)	<1	57.36	73.38	6.54
	Citric acid (0.5 g/L)	<1	53.50	69.52	6.28
Linen/viscose	Without	<1	52.68	68.42	6.89
	Glucose (2.0 g/L)	<1	49.13	65.85	6.48
	PEG 400 (2.0 g/L)	<1	62.02	79.52	7.25
	Citric acid (0.5 g/L)	<1	58.57	74.89	6.90

Plasma treatment: Gas (oxygen); APDBD plasma; power supply with frequency 20,000 Hz, 50 W and an output of 5 kV/20 mA, for 30 s. *Enzymatic treatment:* Step1: Scourzyme® L, 0.5% owf; H_2O_2, 10 ml/L; non-ionic wetting agent, 2 g/L; pH 8.5; LR, 1/20; temperature, 60 °C; time, 15 min. Step 2: chelating agent, 2 g/L; emulsifier, 1 g/L; sodium carbonate, 2 g/L; temperature, 95 °C; time, 45 min. Wet, wetting time; WI_b, whiteness index before optical brightening; WI_a, whiteness index after optical brightening; *K*/*S*, color strength of dyed samples before optical brightening.

by the type of additive, i.e. PEG 400 > citric acid > without > glucose, as well as the nature of the substrate, i.e. linen/viscose > linen/(polyester/cotton) > linen cotton > linen, (iii) the extent of post-reactive dyeing follows the decreasing order: linen > linen cotton > linen/(polyester/cotton) > linen/viscose and PEG 400 > citric acid > without > glucose, (iv) the variation in both the WI and *K*/*S* values reflects the differences among the used additives in ability to: inhibit or activate the enzyme, enhance the extent of fabric modification, aid the removability of the hydrophobic contaminates, stabilize or activate the decomposition of H_2O_2 during the bioscouring/bleaching step, ability to swell the fabric structure and increase its active surface as well as availability of its active sites for subsequent treatment, i.e. post-dyeing or optical brightening, and (v) post-treatment of bleached substrate with the used optical brightener results in a remarkable improvement in their WI values.

3.2.5. Type of enzyme

Table 10 depicts the variation in wetting time, whiteness index as well as depth of obtained dyeing as a function of type of enzyme. The obtained results indicate that: (i) type of enzyme has no effect on wetting time, (ii) the enhancement in WI values as well as the variation in the *K*/*S* values of post-dyed samples follows the descending order: alkaline-pectinase > neutral-cellulase, irrespective of the used substrate and (iii) the changes in the above-mentioned properties reflect the differences between the two enzymes in: molecular size, functionality and accessibility of their active sites, specific catalytic activity, synergy with other ingredients, working conditions especially pH, in addition to the specific target and mode of action, i.e. breaking down the pectic and related compounds into water-soluble fragments thereby catalyzing hydrolysis and destroying the matrix structure of the wall as in the case of using pectinase enzyme or by destroying the cuticle structure via digesting the primary wall cellulose under the cuticle as in the case of using endoglucanases (EGs) which are rich cellulases enzyme, i.e. neutral-cellulase (Eid, 2001; Hebeish & Ibrahim 2007; Kan et al., 2007; Traore & Diller, 2000), and consequently the extent of modification of the treated substrate as well as its amenability to further modification, i.e. post-dyeing or optical brightening.

3.2.6. Nature of plasma gas

Table 11 shows the dependence of WI and *K*/*S* values on the plasma gas used in the pre-treatment step as well as the type of additive included in the subsequent bioscouring/H_2O_2-bleaching step. It is clear that (i) plasma pre-treatment followed by subsequent bioscouring/H_2O_2-bleaching in the absence or presence of the nominated additives results in a dramatic improvements in both the wetting time (>120) and whiteness index (−55.34) values of the blank (untreated) fabric samples, regardless of the used plasma gas, (ii) both the type of plasma gas as well as the type of additive have practically no effect on the wettablility of the treated linen fabric samples, (iii) the extent of improvement is determined by the nature of the plasma gas used in the pre-treatment step, i.e. O_2-plasma > N_2-plasma, as well as the type of additive, i.e. PEG 400 > citric acid > none in case of using O_2-plasma, and citric acid > PEG 400 > none in case of using N_2-plasma, (iv) the variation in the evaluated properties by using different plasma gases reflects the differences between them in: extent of physical and/or chemical modification of the substrate surface, creation of new active sites onto the fabric surface such as −COOH groups in case of using O_2-plasma or $-NH_2$ groups in case of using N_2-plasma, extent of removal of such non-cellulosic impurities and damaging or destroying the cuticle structure as well as extent of increasing the reactivity of the fabric surface to further chemical treatments (Hocker, 2002; Sun & Stylios, 2006), e.g. bleaching, dyeing, etc., which in turn affects both the extent of bioscouring/bleaching in addition to post-dyeing, and (v) the changes in both WI and *K*/*S* values by using different additives, keeping the plasma gas fixed, reflects their variations in: effectiveness, swelling power, ability to activate or stabilize H_2O_2 decomposition, compatibility with other ingredients especially the alkaline-pectinase, as well as the extent of modifying, not only the fabric surface, but also the inner part of the linen substrate for subsequent brightening or dyeing treatment.

Table 10
Effect of using different enzymes in subsequent enzymatic treatment/H_2O_2-bleaching on the properties of linen-containing fabrics

Type of substrate	Enzyme type	Properties			
		Wt (s)	WI_a	WI_b	*K*/*S*
100% Linen	Alkaline-pectinase	<1	36.50	49.28	9.53
	Neutral-cellulase	<1	30.00	40.03	8.70
Linen/cotton	Alkaline-pectinase	<1	39.88	51.63	7.42
	Neutral-cellulase	<1	31.95	42.85	6.65
Linen/(cotton/polyester	Alkaline-pectinase	<1	43.09	61.54	6.16
	Neutral-cellulase	<1	35.25	50.14	5.48
Linen/viscose	Alkaline-pectinase	<1	52.68	68.42	6.89
	Neutral-cellulase	<1	49.80	66.80	6.19

Plasma treatment: Gas (oxygen); APDBD plasma; power supply with frequency 20,000 Hz, 50 W and an output of 5 kV/20 mA, for 30 s. *Enzymatic treatment:* Step1: Scourzyme® L, 0.5% owf; H_2O_2, 10 ml/L; non-ionic wetting agent, 2 g/L; pH 8.5; LR, 1/20; temperature, 60 °C; time, 15 min. Or DeniMax® BT, 2.0% owf; H_2O_2, 10 ml/L; non-ionic wetting agent, 2 g/L; pH 7; LR, 1/20; temperature, 60 °C; time, 15 min. Step 2: chelating agent, 2 g/L; emulsifier, 1 g/L; sodium carbonate, 2 g/L; temperature, 95 °C; time, 45 min. Wet, wetting time; WI_b, whiteness index before optical brightening; WI_a, whiteness index after optical brightening; *K*/*S*, color strength of dyed samples before optical brightening.

Table 11
Effect of plasma gas followed by bioscouring/H_2O_2-bleaching in the presence or absence of different additives on linen-containing fabrics properties

Type of substrate	Type of additives	Type of plasma gas	Properties			
			Wt (s)	WI_b	WI_a	*K*/*S*
Linen 100%	Without	Oxygen	<1	36.50	49.28	9.53
		Nitrogen	<1	25.22	38.15	8.22
	PEG 400	Oxygen	<1	47.22	63.80	9.89
	(2.0 g/L)	Nitrogen	<1	38.65	40.06	9.54
	Citric acid	Oxygen	<1	45.oo	60.83	9.65
	(0.5 g/L)	Nitrogen	<1	40.76	42.37	8.51
Blank (untreated)	–	–	>120	−55.34	–	–

Plasma treatment: Gas (oxygen or nitrogen); APDBD plasma; power supply with frequency 20,000 Hz, 50 W and an output of 5 kV/20 mA, for 30 s. *Enzymatic treatment:* Step1: Scourzyme® L, 0.5% owf; H_2O_2, 10 ml/L; non-ionic wetting agent, 2 g/L; pH 8.5; LR, 1/20; temperature, 60 °C; time, 15 min. Step 2: chelating agent, 2 g/L; emulsifier, 1 g/L, sodium carbonate, 2 g/L; temperature, 95 °C; time, 45 min. Wet, wetting time; WI_b, whiteness index before optical brightening; WI_a, whiteness index after optical brightening; *K*/*S*, color strength of dyed samples before optical brightening.

3.3. Acid-cellulases/peracetic acid vs. alkaline-pectinase/H_2O_2

Finally, we investigated the changes in physico-mechanical, dyeing as well as surface properties of linen fabric samples pre-treated with O_2-plasma followed by subsequent treatment with the acid-cellulases enzyme in the presence of peracetic acid as a bleaching agent or with alkaline-pectinase in the presence of H_2O_2 as a bleaching agent. For a given set of pre-treatment and subsequent treatment conditions, it is clear that: (i) both of the two subsequent treatments result in a dramatic improvement in wetting time from >120 s down to >1 s, (ii) the extent of improvement in WI values follows the decreasing order alkaline-pectinase/H_2O_2 > acid-cellulases/peracetic acid > untreated (Fig. 4), (iii) the increase in *K*/*S* follows the same decreasing order (Fig. 5), which

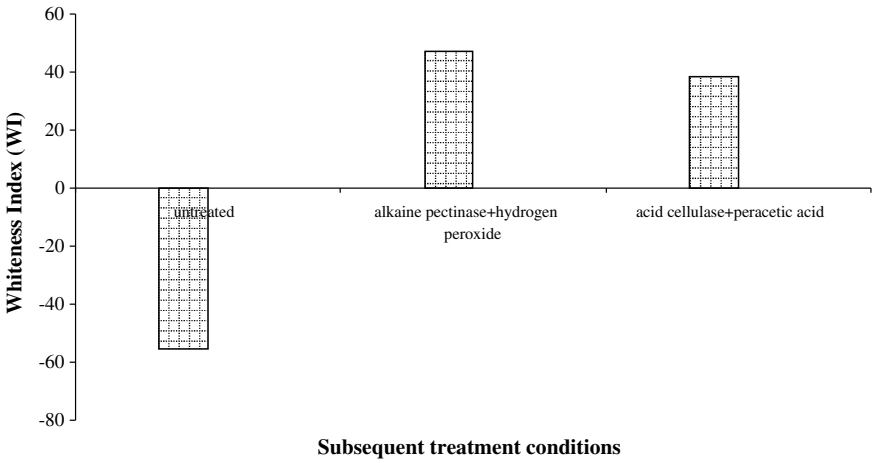


Fig. 4. Effect of subsequent treatment conditions on whiteness index of 100% linen fabric.

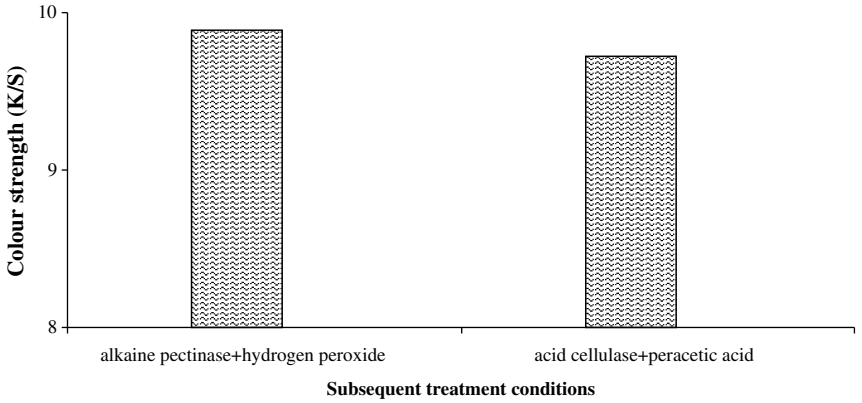


Fig. 5. Effect of subsequent treatment conditions on color strength of 100% linen fabric.

reflects the effectiveness of bioscouring/H_2O_2 system thereby enhancing the extent of linen modification and improving the availability and accessibility of pectic substrate, hydrophobic non-cellulosic compounds as well as colored matter impurity sites along with increasing the extent of penetration and diffusion of the H_2O_2 and allowing easy access of dye molecules i.e. better WI and *K*/*S* values, (iv) the loss in weight (Fig. 6) and the decrease in tensile strength (Fig. 7) as well as the decrease in surface roughness (Fig. 8) of the treated fabric samples are governed by the enzymatic treatment ingredients, i.e. acid-cellulases/peracetic acid > alkaline-pectinase/H_2O_2, which is mainly attributed to the aggressive action of the acid-cellulases on the treated substrate, resulting in the removal of protruded fibers, loss in weight, fiber damage as well as a reduction in surface roughness, and (v) the changes in the aforementioned properties by using acid-cellulase and alkaline-pectinase individually reflect the differences between them in: specific origin, activity, mechanism of action, specific target, degree of effectiveness, accesses to and/or into fiber, as well as compatibility with other ingredients in the subsequent treatment (Kan et al., 2007; Sampaio et al., 2005; Wong et al., 2000).

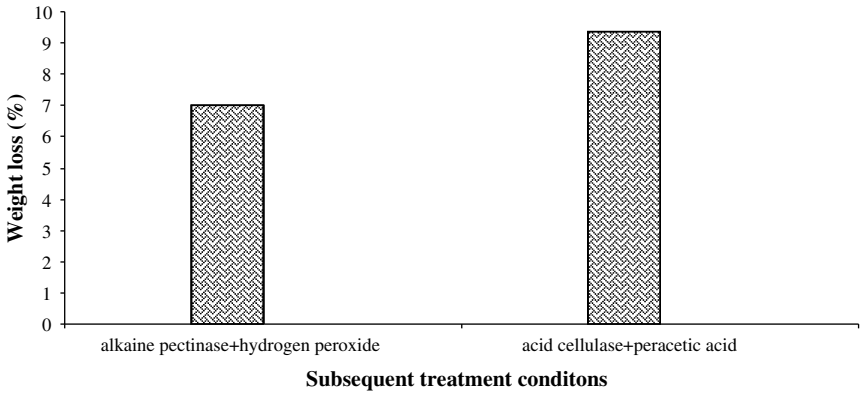


Fig. 6. Effect of subsequent treatment conditions on weight loss of 100% linen fabric.

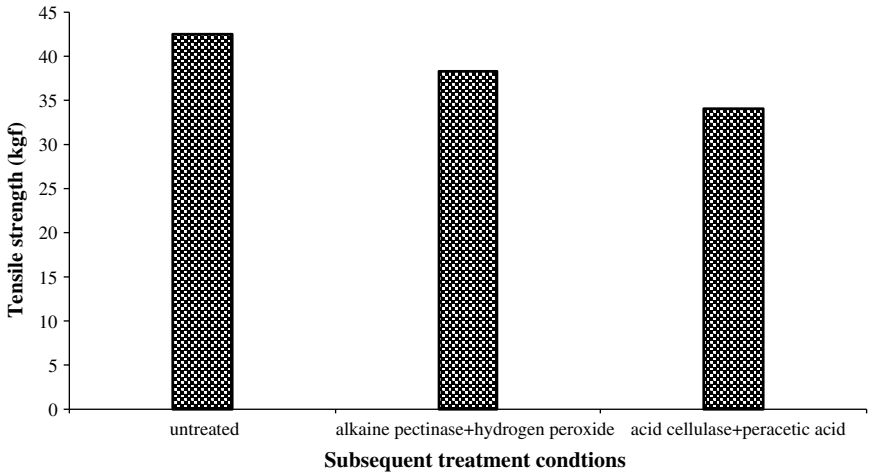


Fig. 7. Effect of subsequent treatment conditions on tensile strength of 100% linen fabric.

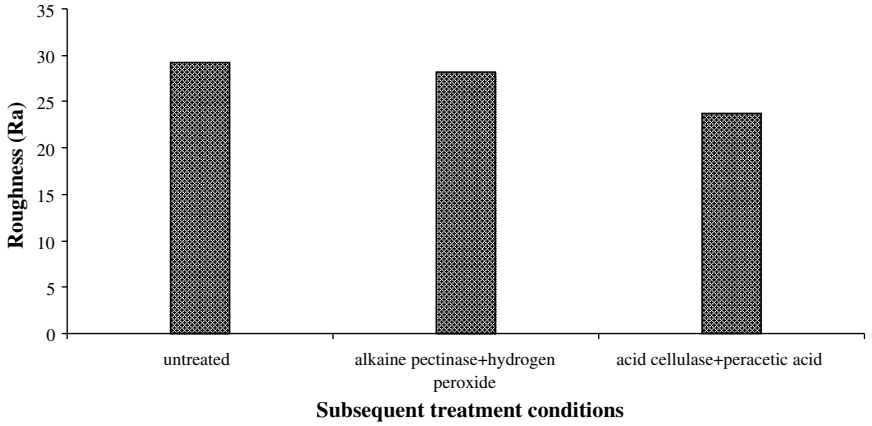


Fig. 8. Effect of subsequent treatment conditions on surface roughness of 100% linen fabric.

On the other hand the effect of treatment conditions on the surface topography of the linen substrate in comparison with the untreated one is examined by scanning electron microscopy (SEM, Figs. 9–11). Fig. 9 reveals a large number of deposits and particulate on the fiber surface that binds the fiber together and hides the nodes or cross marks of the flax fibers. Figs. 10 and 11 illustrate different degree of surface cleaning/smoothing effect depending on the treatment conditions. In the case of plasma pre-treatment followed by acid-cellulases/peracetic acid, the surface becomes smoother and the fiber appeared rounded in addition to removal of the fuzz and fibrils from the surface as well as the clear appearance of fiber nodes as shown in Fig 10a and c compared with those in Fig. 10b, most probably due to (i) biopolishing effect of acid-cellulases, (ii) removal of encrusting intercellular matters that caused loosening action on the fiber surface and (iii) ablation or etching of outer surface layer of the fiber caused by O_2-plasma. In the case of pre-treatment followed by alkaline-pectinase/H_2O_2, Fig. 11c shows a clean smooth and lustrous surface due to the hydrolysis of the pectic substance with the aid of the alkaline-pectinase resulting in the breakdown of the continuity of the cuticle.

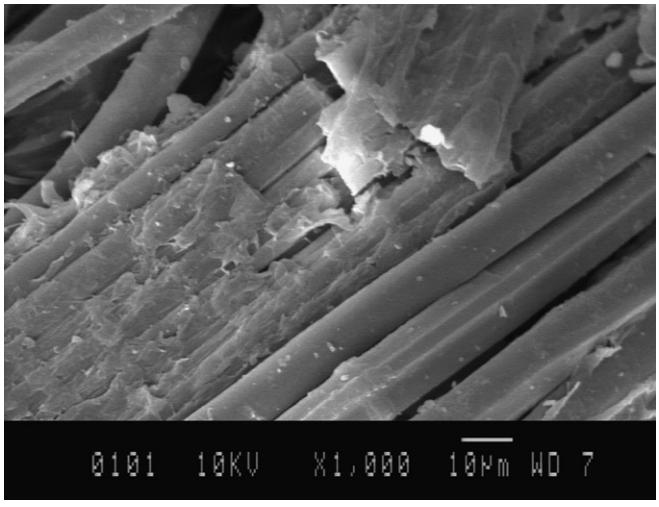
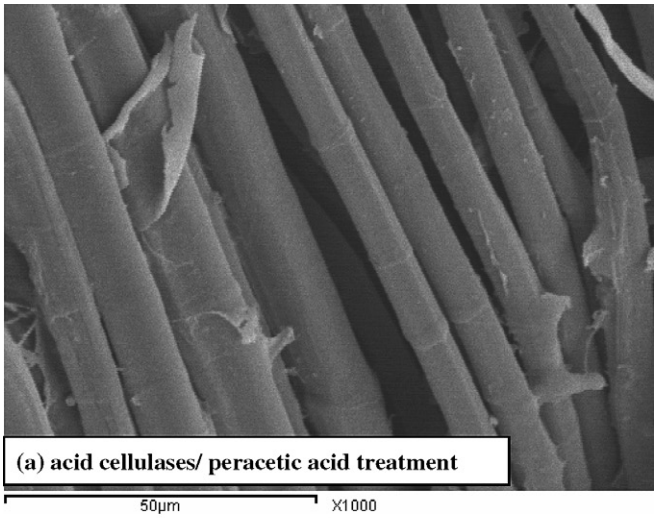
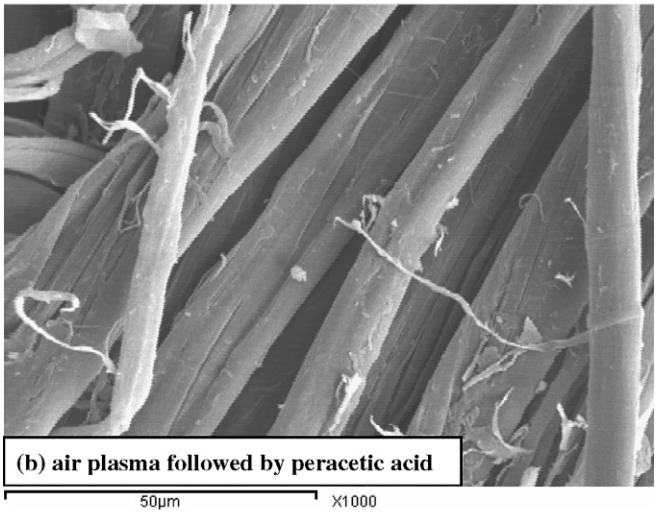


Fig. 9. SEM of 100% untreated linen fabric.

4. Conclusions

During this study, gray linen and linen-containing fabrics were pre-treated with plasma followed by combined enzymatic treatment and bleaching. This sequence of treatment results in changes in the surface, physical, mechanical and dyeing properties of the treated substrates, as a direct consequence of modifying the sur-





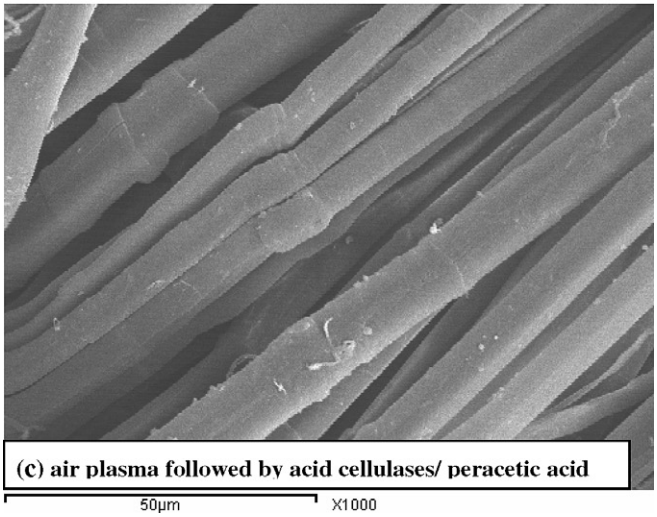
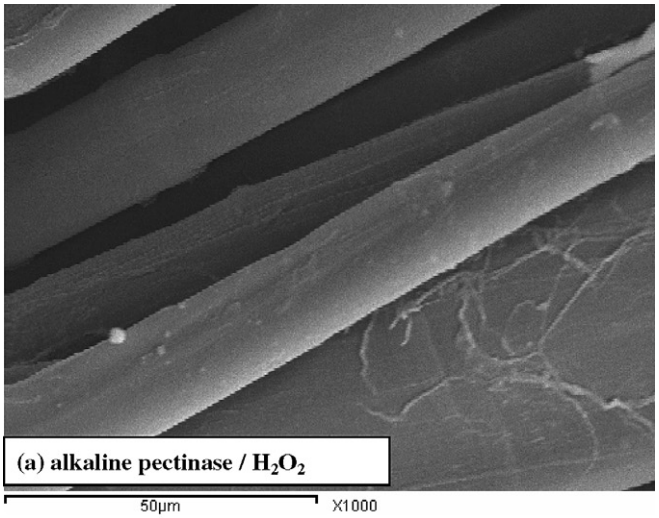
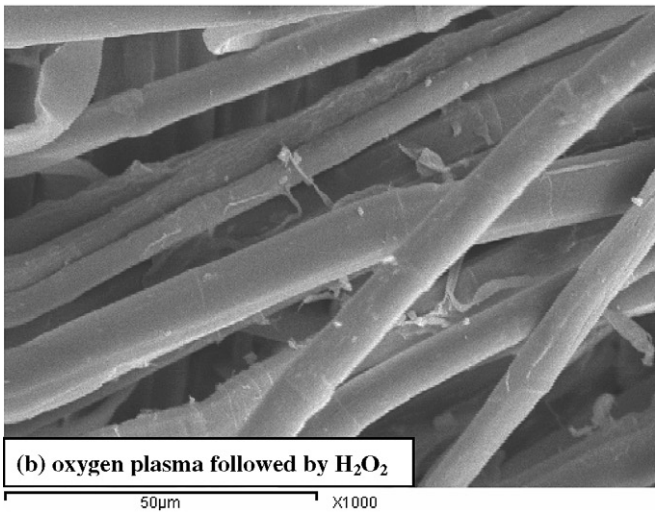


Fig. 10. SEM of air-plasma followed by biopolishing/peracetic acid bleaching for 100% linen fabric samples.





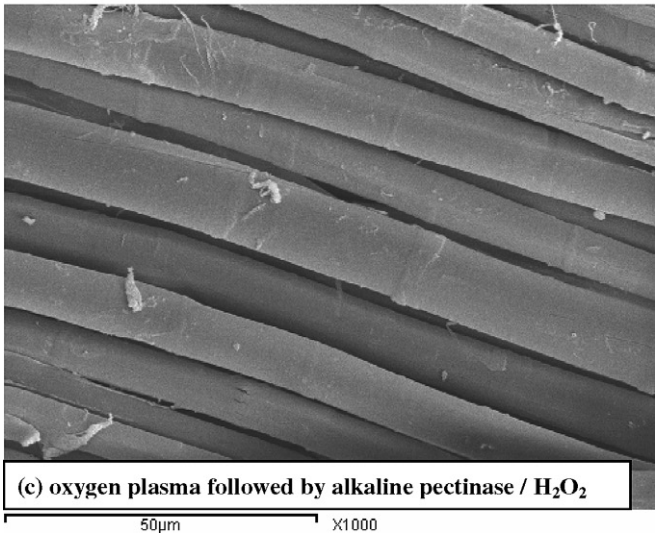


Fig. 11. SEM of oxygen plasma followed by bioscouring/H_2O_2-bleaching for 100% linen fabric samples.

face of the fiber by the plasma as well as the inner part of the fiber by the enzyme which in turn positively affect the extent of removal of hydrophobic non-cellulosic impurities as well as oxidized color substances at the fiber surface and in the fiber structure. The extent of improvement in whiteness index and post-dyeing with the used reactive dye as well as the retained weight and strength of the treated fabric samples follow the descending order: alkaline-pectinase/H_2O_2 > acid-cellulases/peracetic acid, keeping other parameters fixed. The reduction in surface roughness, the increase in weight loss as well as the drop in tensile strength are most proba-

bly due to: the specific action and extent of enzymatic attack, and can be ranked as follows: acid-cellulases > alkaline-pectinase, the initial fiber properties along with its amenability and accessibility to enzyme action.

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