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One-step process for bio-scouring and peracetic acid bleaching of cotton fabric

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A B S T R A C T

In this study, we investigated the utilization of peracetic acid (PAA) formed *in situ* from reaction of tetraacetylethylenediamine (TAED) with sodium perborate to affecting simultaneous desizing and bleaching whereas cellulase, pectinase and their mixture were utilized to affect bio-scouring. In this regards, two parallel studies were designed, the first were, separate desizing either with PAA or ammonium persulphate followed by bio-scouring and PAA bleaching in one step. The second sets of experiments were involved one-step process for desizing, bio-scouring and PAA bleaching. Residual starch, fabric wettability, residual wax content retained tensile strength elongation at break and fabric whiteness index were taken as a measure of the extent of cotton desizing, scouring and bleaching. Results obtained show that, cotton fabric bleached with PAA and either cellulase or pectinase enzyme shows excellent wettability and acceptable whiteness index (WI). This indicated that the combination between PAA and either cellulase or pectinase enzyme did not detract from the effectiveness these enzyme towards bio-scouring or the effectiveness of PAA towards low temperature bleaching of cotton fabric. The optimum bleaching recipe consists of utilizing a bath containing 25 g/L, TAED; 15 g/L, sodium perborate; 2 g/L, pectinase and 5 g/L Egyptol® (non-ionic wetting agent) and the treatment was carried out at 60 °C for 90 min. Desizing with PAA prior to bio-scouring and bleaching enhances the WI to 73.5 the retained tensile strength remain at 85.5%. One-step process for desizing, bio-scouring and PAA was also feasible, which involved treatment the loomstate cotton fabric with the same recipe. Although, the fabric bleached with one-step process shows lower WI compared with two-step processes, but its wettability was excellent and no detectable residual starch was found.

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% (Karmakar, 1999).

Cotton is composed almost entirely of cellulose (90–96% based on weight of fibres). The impurities in cotton fibre range from 4% to 10%. The overall composition of raw cotton fibres depends on its type, origin, fibres maturity, weathering and agricultural conditions (Brushwood, 2003; Carr, 1995; Lewin & Sello, 1984; Marsh, Barker, Kerr, & Butler, 1950). The yellowish or brown coloration of the cotton fibre is related to the protoplasmic residues of protein and the flavones pigments of cotton flowers (Brushwood, 2003; Carr, 1995). Apart from these, the loom-state fabric is also contaminated with processing lubricants, such as machine oils, tars, and greases from harvesting, ginning, spinning and weaving (or knitting) (Karmakar, 1999).

Waxy materials and pectins are responsible for the hydrophobic properties of the raw cotton (Freytag & Dinze, 1983). Therefore, purification of cotton in the fiber, yarn and fabric forms through removal of such impurities is a must prior to dyeing, finishing and utilization. With the exception of pigments (natural colouring matters) which removed by bleaching using certain oxidants, all other impurities are removed by alkali treatment in a process known as scouring. The latter, in common practice involves boiling the cotton in sodium hydroxide (2–5%) for about 1 h (Elliot & Whittlestone, 1994; Karmakar, 1999; Lewin & Sello, 1984).

From the ecological point of view, scouring is a heavy burden since; it consumes large quantities of alkali and requires an extensive rinsing process that loads the washing effluent with environmentally harmful chemicals. On the other hand cellulose is susceptible to oxidation damage under these treatment conditions (Buschle-Diller, El Mogahzy, Inglesby, & Zeronian, 1998), which might result in decreased tensile strength of the fabrics. Alkaline scouring may also cause fabric shrinkage and changes in physico-mechanical properties of fabrics, e.g. their handle (Segal & Wakelyn, 1988).

Several attempts have been made to replace the conventional alkaline boiling with enzymatic systems working at milder conditions (Wang, Fan, Hua, Gao, & Chen, 2007; Yonghua & Hardin,

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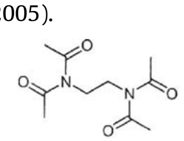
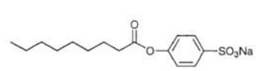
1997). Pectinases appear to be the most suitable enzymes for this purpose, being capable of depolymerising the pectin, breaking it down to low molecular water-soluble oligomers (Calafell & Garriga, 2004; Etters, Husain, & Lange, 1999; Ranveer, Saxena, & Gupta, 2005), and thereby improving the absorbency of the textile material, without causing cellulose destruction. Cellulases evaluated for the same application provoked weight, and strength losses (Robner, 1993; Yonghua & Hardin, 1997).

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; pectin esterases, polygalacturonases and pectin lyases. 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 surfactants and by mechanical action. Scouring with pectinases is carried out either in acid or alkaline media, depending on the type of pectinases at 50–60 °C and the time of treatment is 30–60 min (Gadre, Van Driessche, Van Beeumen, & Bhat, 2003; Klug-Santner et al., 2006; Losonczi, Csiszar, Szakacs, & Kaarela, 2004; Losonczi, Csiszár, Szakács, & Bezúr, 2005).

Peracetic acid (PAA) is environmentally safe alternative bleaching agent. European textile mills are now using peracetic acid in TCF (totally chlorine free) bleaching (Guersy & Dayioglu, 2000; Scarborough & Mathews, 2000; Wurster, 1992). For use as bleaching agents for cotton fabrics, PAA can be generated by reaction of H_2O_2 with bleach activator. Bleach activators are peracid precursors, which generate peracids *in situ* in an alkaline hydrogen peroxide solution. The most widely used bleach activators are tetraacetylethylenediamine (TAED) and nonanoyloxybenzene sulphonate (NOBS) structures I and II, respectively (Krizman, Kovac, & Tavcer, 2005; Lim, Lee, Hinks, & Hauser, 2005; Prabaharan & Almeida, 2004). The application of these bleach activators in textile preparation has been reported as an attempt to minimise the problems caused by the conventional peroxide bleaching. It is claimed that NOBS gives better bleaching performance than TAED and the poor water solubility of TAED limits its application in textile bleaching (Evans, Smith, & Cai, 2001; Krizman et al., 2005; Lim et al., 2005).



(I) Tetraacetylethylenediamine (TAED)



(II) Nonanoyloxybenzene sulphonate (NOBS)

Sodium perborate (H_2O_2 precursors) hydrolyzed in aqueous solution and produce H_2O_2 which, in turn, dissociated to perhydroxyl anion. The latter reacts with TAED or NOBS to form peracetic acid and diacetyl ethylene diamine (DAED). At pH above 5, PAA peracetic acid form active oxygen, this acts as a bleaching agent (Guersy & Dayioglu, 2000; Krizman et al., 2005; Lim et al., 2005; Prabaharan & Almeida, 2004; Scarborough & Mathews, 2000).

PAA is most effective as a bleaching agent for cotton in the following conditions: pH, 6–7, bleaching temperature 50–80 °C and bleaching time of 20–60 min depending on the temperature. This results in lower energy and water consumption both during bleaching and rinsing of the fabric. Neutralization of the fibre after bleaching is not required, unlike bleaching with hydrogen peroxide, where large amounts of alkali must be removed before dyeing. There is also much less damage to the cotton fibre when PAA is used (Guersy & Dayioglu, 2000; Krizman et al., 2005; Lim et al., 2005; Prabaharan & Almeida, 2004; Scarborough & Mathews, 2000).

With the above in mind and as bio-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 treatments could be joined into one-step process. We assumed that, both PAA and pectinases hold their activities upon combination.

More specifically, the present work is undertaken with a view to develop a novel one-step process for bio-scouring and PAA bleaching of cotton fabrics. To achieve the goal, cotton fabrics were subjected to different pretreatment parameters and formulation with a view to determine the best formulations along with optimum conditions that can be applied to cotton fabrics and provoked higher fabric performance. The efficiency of one-step process for bio-scouring with acidic or alkaline pectinases and PAA bleaching was evaluated by monitoring the treated cotton fabric for whiteness index, residual starch and wax content as well as tensile strength and elongation at break.

2. Experimental

2.1. Materials

Greige 100% woven cotton fabric (100 g/m^2) was kindly supplied by Misr Company for Spinning and Weaving, Mehala El-Kubra, Egypt. Chemical analysis carried out on the greige fabric showed that the fabric warps were sized with starch-based sizing agent. Scourzym® L (Alkaline Pectinase with an activity 375 apsu/g) was kindly supplied by Novo-Nordisk A/S, Copenhagen, Denmark [1 (apsu/g) unit being the amount of enzyme which catalyses the decrement in viscosity of 1% (w/v) pectin solution by 20% in 5 min at pH 3.4 and 25 °C] (Hickman, 2002). Cellulase (BioPrep®, Novoenzymes A/S, Denmark). Tetraacetylethylenediamine (TAED) and sod-perborate were purchased from Henkel AG, Germany. Egyptol®, non-ionic wetting agent based on ethylene oxide condensate (Starch and Yeast Co., Alexandria, Egypt) was of technical grade. Ammonium persulphate (APS) and all other chemicals and reagents were of laboratory grade.

2.2. One-step process for desizing, bio-scouring and PAA bleaching

Exhaustion technique was employed to carry out desizing, bio-scouring and PAA bleaching for greige cotton fabric. The experimental technique was adopted as follows: a swatch from greige cotton fabric was immersed in aqueous solution containing 2 g/L, enzymes (pectinase and/or cellulase) along with 5 g/L Egyptol® (non-ionic wetting agent). Different concentrations from TEAD (5–25 g/L) and sodium perborate (5–25 g/L) were subsequently added. Material to liquor ratio (LR) was adjusted at 30. The bath temperature was then gradually raised to 60 °C for 1 h. The samples were then washed several times with boiling water to deactivate the enzyme, followed by thoroughly washing, tumble drying and conditioning.

2.3. Desizing of cotton fabric

Greige cotton fabric was desized either with PAA or ammonium persulphate prior to bio-scouring and PAA bleaching. Desizing with PAA was carried out by treating the fabric with aqueous solution containing 10 g/L TAED and 5 g/L sodium perborate along with 5 g/L Egyptol® at 70 °C for 30 min, using LR of 30. In case of using ammonium persulphate (APS) to affect desizing, the samples were treated with aqueous solution containing, 0.5 g/L, APS and 5 g/L, Egyptol® at 90 °C for 30 min, using LR of 30. After treatment time, both samples were washed with boiling water followed by washing with cold water, tumble drying and conditioning. The samples were then proceeds to bio-scouring and PAA bleaching.

2.4. Testing and analysis

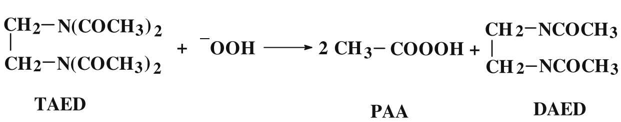
- Residual wax content of the sample was determined according to the reported method (Peter, 1955). A known weight of the fabric sample was Soxhlet-extracted using tetrachloro-ethylene solvent for 6 h. The percent loss in fabric weight after extraction represents the residual wax content of the fabric.
- Wettability was assessed in terms of drop disappearance time, measured by allowing a drop of water to fall on the sample and recording the time required for drop disappearance (AATCC Test Method 39-1980). The fabric shows wetting time less than 5 s is judged as good wetting property.
- Residual starch on fabric was determined according to AATCC reported method.
- Tensile strength and elongation at break were determined according to ASTM standard test method.
- The degree of whiteness was measured on the Datacolor Spectraflash SF600 Plus spectrophotometer by using the CIE method according to EN ISO 105-J02:1997(E) standard.

3. Results and discussion

Sodium perborate (H_2O_2 precursors) hydrolyzed in aqueous solution and produce H_2O_2 which, in turn, dissociated to perhydroxyl anion (Eqs. (1) and (2)). The latter reacts with TAED to form peracetic acid and diacetyl ethylene diamine (DAED) (Eq. (3)). At pH above 5, PAA spontaneously decomposes to give acetic acid and active oxygen which acts as a bleaching agent (Eq. (4)) (Evans et al., 2001; Krizman et al., 2005; Lim et al., 2005). The bleaching action of PAA is mainly the result of epoxidation of the double bonds present in unwanted coloured compounds.

$$Na_4(H_4B_2O_8) + 4H_2O \rightarrow 2Na_2[(BOH)_4] + H_2O_2 \quad (1)$$

$$H_2O_2 \leftrightarrow H^{\oplus} + OOH^- \overset{OH^-}{\rightarrow} H_2O + OOH^- \quad (2)$$



(3)

$$2CH_3COOH \overset{Temp.>40\ ^{\circ}C,\ pH>5}{\longrightarrow} 2CH_3COOH + O_2 \quad (4)$$

It is also envisaged from the above reaction scheme that, the magnitude of cotton bleaching would rely on TAED, sodium perborate concentration and the state of the fabric (i.e., desized or scoured).

In this study, we investigated the utilization of PAA formed *in situ* from reaction of TAED with sodium perborate to affecting simultaneous desizing and bleaching whereas cellulase, pectinase and their mixture were utilized to affect bio-scouring. Optimum conditions for PAA desizing and bleaching were investigated whereas the recommended conditions for bio-scouring with pectinases were supplied by the enzyme manufacturing. In this regards, two parallel studies were designed, the first were, separate desizing treatment either with PAA or ammonium persulphate followed by bio-scouring and PAA bleaching process in one step. The second sets of experiments were involved one-step process for desizing, bio-scouring and PAA bleaching. Residual starch, wettability, retained tensile strength elongation at break and fabric whiteness index were taken as a measure of the extent of cotton scouring and bleaching.

3.1. Preliminary investigation the effect of combination between PAA and scouring enzymes

Table 1 shows the properties of cotton fabric desized, bio-scoured and bleached in one-step process using formulation consists of PAA and either cellulase or pectinase enzymes along with non-ionic wetting agent. PAA was formed *in situ* by reaction of TAED and sodium perborate. The treated fabrics were monitored for WI, wettability, residual wax content, retained tensile strength (RT %) and elongation at break. Results obtained with untreated fabric (blank) are set-out in the same table for comparison.

Results of Table 1 show that (i) the fabric WI obtained after PAA bleaching and those obtained after bleaching with mixture of PAA and cellulase or pectinase enzyme were approximately comparable. This indicated that, the combination between PAA and either cellulase or pectinase enzyme did not detract from the effectiveness of PAA towards low temperature bleaching of cotton fabrics. (ii) The wettability of the bleached fabric remains almost unchanged after bleaching the loomstate cotton fabric with PAA only without addition of cellulase or pectinase enzymes. However, addition of 2 g/L cellulase or pectinase enzyme to PAA bleaching bath improves the wettability of the fabric, where the wettability time decreased from above 180 s before treatment to only 4 s after treatment. The same holds true if mixture of cellulase and pectinase (2 g/L each) were added to PAA bath, where the wettability time decreased from above 180 s to only 4 s. Here too, the combination between PAA and either cellulase or pectinase enzyme did not detract from the effectiveness these enzyme towards bio-scouring of desized cotton fabric. The enhancement in the fabric wettability is indicative of removal of almost, if not all, waxes via bio-scouring under the influence of alkaline pectinase or cellulase enzymes followed by hot washing. This is confirmed by observing the change in wax content after enzymatic treatment. Results of Table 1 make it clear that, incorporation of either cellulase or mixture of them reduces the residual wax content to only 0.05%. It is also seen that, bleaching with PAA without the addition of cellulase or pectinase did not reduce the residual wax content of cotton fabric after treatment. (iii) The retained tensile strength decreased after PAA bleaching and shows a value of 93%. Addition of cellulase or pectinase enzymes in PAA bleaching bath further decreased the retained tensile strength to a value of 90% and 92%, respectively. Addition of mixture from cellulase and pectinase enzymes to PAA bleaching bath detract from the strength properties of the treated cotton fabric where the retained tensile strength decreased to 88%. (iv) A silent feature observed in Table 1 is ability of PAA to remove starch sizing agent from the loomstate cotton fabric after bleaching. This was observed whether cellulase or pectinase enzymes were added or not.

Result of Table 1 clarify also that, PAA bleaching in the acid medium does not induce breakdown of pectin. PAA also does not have any effect on the activity of alkaline pectinases or cellulase enzyme. These two enzymes induced partial degradation of pectin; and their combined action with PAA enhances not only the fabric whiteness but also improves the fabric wettability and induces desizing of the loomstate cotton fabric. Stated in other words, with all the combined processes, a sufficient quantity of pectin and wax was removed, so that the fabrics absorbed water well. A medium degree of whiteness was achieved and the fibres remained undamaged. Indeed these results form a new route for environmentally safe bio-scouring and bleaching of cotton based textile and deserve optimization of the process parameters.

Two treatment routes were investigated and optimized for desizing, bio-scouring and PAA bleaching of loomstate cotton fabric. In the first route, the loomstate cotton fabric was first desized with either PAA or ammonium persulphate in separate step then proceed to bio-scouring and PAA bleaching in one bath, i.e., two-step process form pretreatment and bleaching of loomstate cotton fabric. In the second route, the loomstate cotton fabric was desized, bio-scoured and bleaching in one-step process. Optimization of process parameters in each route is discussed below.

Table 1
One-step process for cotton desizing, bio-scouring and PAA bleaching – Effect of enzyme and PAA combination.

Constituent of the pretreatment bath	WI	Wett. (s)	Wax (%)	RT (%)	Elon. (%)	RS
Untreated cotton fabric (loomstate)	9.30	>180	0.37	100	29	+++
PAA without enzyme	43.71	>180	0.35	93	28	–
PAA and cellulase enzyme	45.16	4	0.07	90	28	–
PAA and pectinase enzyme	45.37	4	0.05	92	28	–
PAA and mixture from Cellulase/pectinase enzyme	44.67	4	0.05	87	29	–

WI, whiteness index; Wett., wettability; RS, residual starch content; RT, retained tensile strength, Elon., Elongation at Break.
Residual starch: Very high amount (+++), high amount (++), appreciated amount, (+), zero (–).
Conditions used: Pectinase, 2 g/L; cellulose, 2 g/L; TEAD, 5 g/L; sodium perborate, 5 g/L; Egyptol® (non-ionic wetting agent), 5 g/L; LR, 30; Temp., 50 °C; time, 90 min.

3.2. Two-step process for desizing, bio-scouring and PAA bleaching of loomstate cotton fabric: Effect of process parameters

3.2.1. Effect of TAED and sodium perorate concentrations

Loomstate cotton fabric was first desized either with ammonium persulphate or PAA as explained in the experimental parts then proceeds for bio-scouring and PAA bleaching in subsequent treatment. The idea for utilization of PAA to affect desizing of starch based sized cotton fabric was initiated from results in Table 1 discussed previously. The results indicated that, a medium improved in degree of whiteness together with the removal of appreciated amount from starch-based size were achieved when the loomstate cotton fabric was treated with a bath containing PAA formulation.

TAED is peracetic acid precursor and under the influence of sodium perborate (H_2O_2 precursors) perhydrolyze and liberate peracetic acid (PAA) (Guersy & Dayioglu, 2000; Krizman et al., 2005; Lim et al., 2005; Prabaharan & Almeida, 2004; Scarborough & Mathews, 2000). Hence, the effective concentration of liberated PAA required to achieve bleaching of desized cotton fabric will depend on the concentration of both TAED and sodium perborate.

Table 2 shows the effect of TAED concentration on the performance properties of desized cotton fabric. The results show that increasing TAED concentration is accompanied by increasing the whiteness index of the bleached fabric. This was observed irrespective to the method of desizing applied before bleaching but with certainty that the values was much higher with those fabric desized with PAA. The higher WI observed with that fabric desized with PAA is due to the medium bleaching effect occurred during desizing of the loomstate cotton fabric. This was not the case with other fabric desized with ammonium persulphate. Maximum WI was achieved at TAED concentration equal to 25 g/L. Higher concentrations exert practically marginal change. It is seen also from Table 2 that, the fabric wettability greatly improved after bleaching with PAA and cellulase enzyme, where the wettability time decreased to only 2 s. The concentration of TEAD has no influence on both wax content and wettability of the fabric after PAA bleaching without enzyme. It is further that, both retained tensile strength and elongation at break decreased as the concentration of TEAD increased. This was observed regardless the method of desizing employed before bleaching but with certainty that the value of retained tensile strength is much higher with those fabric per-desized with PAA.

Similar study was carried out on the effect of sodium perborate concentration on the performance properties of bleached cotton fabric. The results are set out in Table 3 and indicated that; 15 g/L sodium perborate achieved the highest WI. Higher sodium perborate exerts little effect on the WI of the bleached cotton fabric.

3.2.2. Effect of bleaching temperature and time

Table 3 shows the effect of temperature on the efficiency of one-step bio-scouring and PAA bleaching for cotton fabric carried out as per exhaustion method. It is seen from Table 3 that, raising the bleaching temperature from 25 °C to 60 °C is accompanied by

Table 2
Two-step process for cotton desizing, bio-scouring and PAA bleaching – Effect of TAED concentration.

Method of desizing	TAED conc. (g/L)	WI	Wett. (s)	RT (%)	Elon. (%)
Untreated fabric (blank)	–	9.0	>180	100	29
Cotton fabric desized with PAA before bio-scouring and PAA bleaching	5	50.90	2	97.0	26
	10	55.56	2	93.0	27
	15	58.60	2	89.1	26
	20	64.70	2	87.3	26
	25	72.80	2	85.3	27
	30	72.90	2	83.5	27
Cotton fabric desized with ammonium persulphate before bio-scouring and PAA bleaching	5	21.20	2	93.2	29
	10	27.50	2	89.3	28
	15	48.14	2	84.2	27
	20	55.10	2	81.5	27
	25	60.53	2	79.5	26
	30	60.50	2	78.3	25

WI, whiteness index; Wett., wettability; RT, retained tensile strength; Elon., elongation at break.
Desizing conditions: Desizing with PAA: TEAD, 10 g/L; sodium perborate, 15 g/L; Temp., 80 °C; time, 30 min. Desizing with ammonium persulphate (APS): APS, 0.5 g/L; Egyptol, 5 g/L; Temp., 80 °C; time, 60 min.
Bio-scouring and bleaching conditions: Sodium perborate, 15 g/L; pectinase enzyme concentration, 2 g/L; Egyptol® (non-ionic wetting agent), 5 g/L; LR, 30; Temp., 60 °C; time, 60 min.

Table 3
Two-step process for cotton desizing, bio-scouring and PAA bleaching – Effect of sodium perborate concentration.

Method of desizing	Sodium perborate conc. (g/L)	WI	Wett. (s)	RT (%)	Elon. (%)
Untreated fabric (blank)	–	9	>180	100	29
Cotton fabric desized with PAA before bio-scouring and PAA bleaching	5	59.6	2	91.0	28
	10	68.6	2	89.0	28
	15	72.5	2	85.3	28
	20	70.6	2	83.0	27
	25	68.6	2	76.0	27
Cotton fabric desized with ammonium persulphate before bio-scouring and PAA bleaching	5	42.59	2	86.0	27
	10	47.68	2	81.5	28
	15	60.50	2	79.50	27
	20	60.40	2	77.0	29
	25	60.47	2	75.0	29

WI, whiteness index; Wett., wettability; RT, retained tensile strength, Elon., elongation at break.
Desizing conditions: Desizing with PAA: TEAD, 10 g/L; sodium perborate, 15 g/L; Temp., 80 °C; time, 30 min. Desizing with ammonium persulphate (APS): APS, 0.5 g/L; Egyptol, 5 g/L; Temp., 80 °C; time, 60 min.
Bio-scouring and bleaching conditions: TAED, 25 g/L; pectinase enzyme concentration, 2 g/L; Egyptol® (non-ionic wetting agent), 5 g/L; LR, 30; Temp., 60 °C; time, 60 min.

Table 4
Two-step process for cotton desizing, bio-scouring and PAA bleaching – Effect of bleaching temperature.

Method of desizing	Temp. (°C)	WI	Wett. (s)	RT (%)	Elon. (%)
Untreated fabric (blank)	–	9.0	>180	100	29.0
Cotton fabric desized with PAA before bio-scouring and PAA bleaching	25	46.04	2	90.1	30.9
	40	52.40	2	87.5	25.6
	60	72.30	2	85.4	28.0
	80	72.43	2	82.0	26.5
Cotton fabric desized with ammonium persulphate before bio-scouring and PAA bleaching	25	15.19	2	88.6	26.3
	40	32.27	2	83.9	27.3
	60	60.40	2	79.5	28.0
	80	60.50	2	77.0	20.0

WI, whiteness index; Wett., wettability; RT, retained tensile strength, Elon., elongation at break.
Desizing conditions: Desizing with PAA: TEAD, 10 g/L; sodium perborate, 15 g/L; Temp., 80 °C; time, 30 min. Desizing with ammonium persulphate (APS): APS, 0.5 g/L; Egyptol, 5 g/L; Temp., 80 °C; time, 60 min.
Bio-scouring and bleaching conditions: TAED, 20 g/L; sodium perborate, 15 g/L; pectinase enzyme concentration, 2 g/L; Egyptol® (non-ionic wetting agent), 5 g/L; LR, 30; time, 60 min.

Table 5
Two-step process for cotton desizing, bio-scouring and PAA bleaching – Effect of bleaching time.

Method of desizing	Time (min)	WI	Wett. (s)	RT (%)	Elon. (%)
Untreated fabric (blank)	–	9.0	>180	100	29
Cotton fabric desized with PAA before bio-scouring and PAA bleaching	30	57.4	2	90.6	28.5
	60	72.3	2	88.5	28.0
	90	73.6	2	85.5	29.17
	120	73.5	2	81.8	26.83
Cotton fabric desized with ammonium persulphate before bio-scouring and PAA bleaching	30	36.5	2	87.4	23.5
	60	60.1	2	80.1	26.0
	90	60.9	2	79.9	28.5
	120	60.9	2	79.2	27.0

WI, whiteness index; Wett., wettability; RT, retained tensile strength, Elon., elongation at break.
Desizing conditions: Desizing with PAA: TEAD, 10 g/L; sodium perborate, 15 g/L; Temp., 80 °C; time, 30 min. Desizing with ammonium persulphate (APS): APS, 0.5 g/L; Egyptol, 5 g/L; Temp., 80 °C; time, 60 min.
Bio-scouring and bleaching conditions: TAED, 25 g/L; sodium perborate, 15 g/L; pectinase enzyme concentration, 2 g/L; Egyptol® (non-ionic wetting agent), 5 g/L; LR, 30; Temp., 60 °C.

increasing the PAA bleaching efficiency. This was evidenced by increasing the WI of the bleached fabric. This could be associated with the favorable effect of the temperature on swelling of cotton fabric, diffusion of PAA, enhancement of reactant mobility and their collision with cotton impurities. Further increase in the bleaching temperature has practically no influence on the WI of the bleached fabric. The retained tensile strength was gradually decreased as the temperature increase. Obviously, 60 °C represents the optimal bleaching temperature of desized cotton fabric where the maximum WI and acceptable retained tensile strength were obtained.

Table 5 shows the effect of bleaching time on the performance propertied of bleached cotton fabric. Results of Table 5 imply that the WI gradually increases and attains a maximum value after 90 min. Further increase in the bleaching time has practically marginal effect. Although both retained tensile strength and elongation at break gradually decrease as the bleaching time increased, they rather acquired an acceptable value after 90 min bleaching.

Results in Table 5 evidenced that, 90 min duration presents the most appropriate time for bleaching of desized cotton fabric using PAA and pectinase enzyme. This is the case whether the loomstate cotton fabrics were pre-desized with PAA or ammonium persulphate. Longer time provides better opportunity for better contact among reactants and, therefore, higher extent of bleaching reaction.

A close examination of results in Tables 2–5 would reveals that desized cotton fabric can be successfully enzymatic scoured and PAA bleached in one bath using exhaustion technique. The optimum bleaching recipe consists of 25 g/L, TAED; 15 g/L, sodium perborate; 2 g/L, pectinase and 5 g/L Egyptol® (non-ionic wetting agent) the treatment was carried out at 60 °C for 90 min. Desizing with PAA prior to bio-scouring and bleaching enhances the WI and the bleached fabric shows higher retained tensile strength and elongation at break compared with those fabric desizing using ammonium persulphate.

3.3. One-step process for cotton desizing, bio-scouring and PAA bleaching – Effect of TAED and sodium perorate concentrations

Rather to a two-step process for bleaching of cotton fabric discussed above, one-step process for desizing, bio-scouring and PAA bleaching can therefore be adopted. The main objective of this part

Table 6
One-step process for cotton desizing, bio-scouring and PAA bleaching – Effect of TEAD concentration.

Type of enzyme used	TAED conc. (g/L)	WI	Wett. (s)	RT (%)	Elon. (%)	R.S
Untreated cotton fabric (blank)	–	9	>180	100	29	+++
PAA bleached without enzyme	5	16.15	>180	98	28	+
	10	37.45	>180	96	28	–
	15	41.88	>180	95	27	–
	20	42.86	>180	94	26	–
	25	45.84	>180	91	25	–
	30	45.85	>180	90	25	–
PAA with cellulase enzyme	5	17.64	2	96	28	+
	10	36.75	2	93	27	–
	15	44.73	2	91	27	–
	20	51.29	2	88	26	–
	25	61.12	1	85	25	–
	30	61.20	1	83	23	–
PAA with pectinase enzyme	5	18.38	2	91	29	+
	10	26.75	2	88	29	–
	15	39.46	2	85	27	–
	20	46.91	1	83	26	–
	25	59.78	1	78	25	–
	30	59.70	1	78	25	–

WI, whiteness index; Wett., wettability; RS, residual starch content; RT, retained tensile strength, Elon., elongation at break.
Residual starch: Very high amount (+++), high amount (++), appreciated amount, (+), zero (–).
Conditions used: Sodium perborate, 15 g/L; enzyme concentration, 2 g/L; Egyptol® (non-ionic wetting agent), 5 g/L; LR, 30; Temp., 60 °C; time, 60 min.

Table 7
One-step process for cotton desizing, bio-scouring and PAA bleaching – Effect of sodium perborate concentration.

Type of enzyme used	Sodium perborate conc. (g/L)	WI	Wett. (s)	RT (%)	Elon. (%)	RS
Untreated fabric (blank)	–	9	>180	100	29	+++
PAA bleached without enzyme	5	29.3	>180	97	27	+
	7	35.28	>180	97	26	−
	10	45.85	>180	95	26	−
	15	47.84	>180	91	25	−
	20	47.87	>180	90	24	−
PAA with cellulase enzyme	5	33.14	2	96	27	+
	7	40.55	2	94	27	−
	10	49.41	1	94	26	−
	15	61.12	1	85	24	−
	20	60.83	1	83	21	−
PAA with pectinase enzyme	5	31.45	2	91	28	+
	7	39.65	2	87	27	−
	10	43.20	1	83	23	−
	15	55.88	1	78	21	−
	20	55.16	1	78	21	−

WI, whiteness index; Wett., wettability; RS, residual starch content; RT, retained tensile strength, Elon., elongation at break.
Residual starch: Very high amount (+++), high amount (++), appreciated amount, (+), zero (−).
Conditions used: TAED, 25 g/L; enzyme concentration, 2 g/L; Egyptol® (non-ionic wetting agent), 5 g/L; LR, 30; Temp., 60 °C; time, 60 min.

is to point out reducing time and temperature of the bleaching process by using one-step process for desizing, bio-scouring and PAA bleaching instead of two steps namely, desizing followed by bio-scouring and PAA bleaching of cotton fabric.

The idea behind the developing one-step process for desizing, bio-scouring and PAA bleaching arose from result obtained previously in Table 1. These results indicated that, the ability of PAA to remove starch sizing agent from the loomstate cotton fabric after bleaching. This was observed whether cellulase or pectinase enzymes were added or not in the bleaching bath. Optimization of one-step process for desizing, bio-scouring and PAA bleaching required the studying of TAED, and sodium perborate concentrations along with bleaching temperature and bleaching time. The latter two parameters are investigated previously in two-step process. Therefore in this part attention will be paid to the effect of TAED and sodium perborate only.

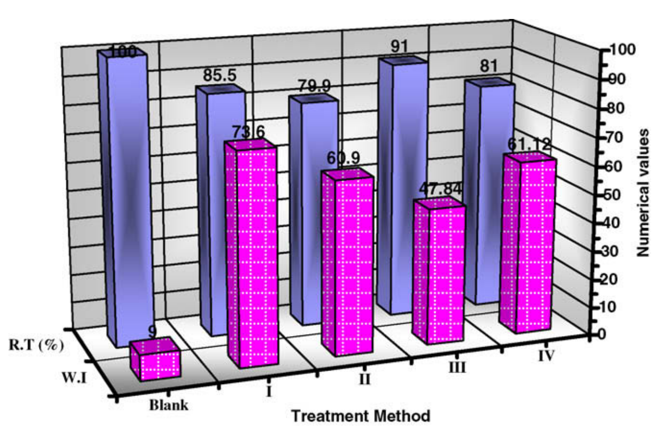


Fig. 1. Dependence of whiteness and retained tensile strength of cotton fabric on method of treatment. Method I: Desizing with PAA followed by bleaching with PAA and cellulase. Method II: Desizing with APS followed by bleaching with PAA and cellulase. Method III: Bleached with PAA only in one step. Method IV: Bleached with PAA and cellulase enzyme in one step.

Table 6 shows the effect of TAED concentration on whiteness index, retained tensile strength, elongation at break, fabric absorbency and residual starch content of the cotton fabric after one-step process for desizing, bio-scouring and PAA bleaching. Peracetic acid is created *in situ* via reaction of TAED and sodium perborate and constitutes the essential bleaching agent in current bleaching system.

The results imply (i) that the WI increases by increasing the TAED concentration up to 25 g/L; beyond this concentration the increase in WI is very marginal. This was observed whether the scouring enzymes were added or not. Addition of cellulase enzyme to the bleaching bath slightly improves the WI of cotton fabrics especially at higher TAED concentration. The enhancement of WI by increasing the TAED concentration is a direct consequence of this on the amount of peracetic acid. (ii) That the residual starch on cotton fabric was completely disappeared at TAED concentration equal 10 g/L. (iii) Although the fabric absorbency is remain practically intact (above 180 s) in absence of bio-scouring enzymes and with all TAED concentration used; it was excellent with all substrates treated with either cellulase or pectinase enzyme along with PAA and with all TAED concentrations used. (iv) The retained tensile strength gradually decreases as TAED concentration increases and such decrement is pronounced if cellulase or pectinase enzyme was added to affect the bio-scouring. At the same TAED concentration the values of retained tensile strength was lower with those fabric treated with PAA and pectinase enzyme compared with those treated with PAA and cellulase enzyme. It is clear from result in Table 6 that 25 g/L TAED represent the optimal concentration.

It is understandable that sodium perborate is hydrogen peroxide precursor and it hydrolyzed with water to yield H_2O_2. The latter per-hydrolyze TAED to yield PAA, which consider the main bleaching agent. Hence, the amount of PAA formed *in situ* and the fabric properties will also depend on perborate concentration.

Table 7 shows the effect of sodium perborate concentration on whiteness index, retained tensile strength, elongation at break, fabric absorbency and residual starch content of the cotton fabric after one-step process for desizing, bio-scouring and PAA bleaching. It is seen from Table 7 that, increasing sodium perborate concentration from 5 g/L to 15 g/L is accompanied by increasing in the WI of the bleached fabric. Further increase in sodium perborate concentration has no effect on the WI of the bleached fabric. It is further noted that, the retained tensile strength decreased as the concentration of perborate increased. The residual starch concentration disappeared after utilization of only 10 g/L perborate along with 25 g/L TAED. This result indicated that, low concentration from PAA formed *in situ* can affect complete desizing but not enough to give the fabric higher whiteness. Obviously, the maximum WI was attained on using 15 g/L sodium perborate.

Fig. 1 shows the dependence of whiteness and retained tensile strength of cotton fabric on method of treatment. It is evidence that the highest WI is obtained when the cotton fabric was desized with PAA followed by bio-scouring and bleaching (method I). The lowest WI was obtained when the loomstate cotton fabric was desized, bio-scoured and PAA bleached in one step (method III). It is understandable that, in one-step process much amount from PAA was consumed in affecting desizing and minor amount was left to affect bleaching. It is seen also from Fig. 1 that, the highest value of retained tensile strength was obtained in method III, whereas method I gave intermediate value compared with other pretreatment method.

4. Conclusion

The desized cotton fabrics bleached with PAA and either cellulase or pectinase enzyme show excellent wettability and acceptable WI. This indicated that the combination between PAA and either cellulase or pectinase enzyme did not detract from the activity of these enzymes towards bio-scouring cotton fabric or the effectiveness of PAA towards low temperature bleaching of cotton fabric. The optimum bleaching recipe consists of utilizing a bath containing 25 g/L, TAED; 2 g/L, sodium perborate; 0.15 ml/L, pectinase and 5 g/L Egyptol® (non-ionic wetting agent) and the treatment was carried out at 60 °C for 90 min. Desizing with PAA prior to bio-scouring and bleaching enhances the WI to 73.5 the retained tensile strength remain at 85.5%. Desizing using ammonium persulphate gave lower WI and retained tensile strength compared with desizing with PAA. Wettability of the bleached fabric depends on the addition of scouring enzyme, namely, cellulase or pectinase; fabric bleached with PAA only show very poor wettability. One-step process for desizing, bio-scouring and PAA was also feasible, by treating the loomstate cotton fabric with the same recipe. Although the fabric shows lower WI, but its wettability was excellent and no detectable residual starch was found.

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