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Bioscouring and bleaching process of cotton fabrics – an opportunity of saving water and energy

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Bio-processing was accompanied by a significant lower demand of energy, water, chemicals, time and costs. This study attempted to introduce the bio-processes in the conventional scouring and bleaching with peracetic acid (PAA) of the cotton fabrics. The scouring with two types of pectinases, acting under acidic and alkaline conditions, respectively, was as efficient as the chemical process in terms of obtained adequate water absorbency of the fabrics. Bleaching with PAA can substitute bleaching with hydrogen peroxide when medium degree of whiteness is demanded. The bioscouring and bleaching with PAA processes cause no damage to fibres and this is one of the benefits of such processes. Wastewaters are not charged with harmful chemicals. The consumption of water and energy is the lowest at combined scouring/bleaching treatments. Consequently, at these processes arises the lowest amount of effluents and the produced wastewater is biodegradable.

Keywords: enzymes; pectinases; bio-processes; peracetic acid; eco-friendly characteristics

Introduction

The progress of industrial biotechnology in the last twenty years, especially in molecular biology, protein engineering and fermentation technology, enhanced the development of new uses of enzymes in the food industry, textile industry, detergents, paper and leather industry, organic chemical synthesis, etc. The use of enzymes in the textile industry is an example of white industrial biotechnology, which allows the development of environmentally friendly technologies in fibre processing, improves the final product quality and it requires less energy. Only about 75 of 7000 known enzymes are commonly used in textile industry processes (Quandt & Kuhl, 2001). The principal enzymes applied in textile industry are hydrolases and oxidoreductases. Amylases, pectinases, cellulases, proteases, catalases, peroxidases and lactases are the enzymes that can replace aggressive chemicals into every step of textile wet processing, bleaching, dyeing to finishing and effluent treatment (Cavaco-Paulo & Gübitz, 2003).

Amylases were the only enzymes applied in textile processing until the 1980s. These enzymes are still used to remove starch-based sizes from fabrics after weaving. The potential of proteolytic enzymes was assessed for the removal of wool fibre scales, resulting in improved anti-felting behaviour. Cellulases have been applied to enzymatically remove fibrils and fuzz fibres and have also successfully been introduced to the cotton textile industry. Catalases have been used to remove H_2O_2 after bleaching, reducing water consumption. Pectinases are used as agents in cotton scouring and in biopreparation of bast fibres such as flax, ramie and jute (Holme, 2004).

All known enzymes are large protein molecules made up of long-chain amino acids which are produced by living cells in plants, animals and microbes. Some enzymes require small non-protein molecules, known as cofactors, in order to function as catalysts (Jenkins, 2003). Enzymes-catalysed reactions are several times faster than chemically catalysed ones. Enzymes catalyse a reaction under mild reaction conditions. These naturally occurring enzymes are quite often not readily available in sufficient quantities for industrial use, but the number of proteins being produced using recombinant techniques is exponentially increasing. Screening approaches are being performed to rapidly identify enzymes with potential industrial application (Korf et al., 2005). For this purpose, different expression hosts (*Esherichia coli*, *Bacillus* sp., *Sacharomyces cerevisae*, *Pichia pastoris*) have been developed to express heterologous proteins (Chelikani, Reeves, Rajbhandary, & Khorana, 2006; Silbersack et al., 2006; Li et al., 2007).

One of the most negative environmental impacts of textile production is the traditional processes used to prepare cotton fibre, yarn or fabric. In the conventional preparatory process, concentrated sodium hydroxide solution and hydrogen peroxide (HP) are applied for removing the impurities from raw cotton. On the other

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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 (AS) may also cause fabric shrinkage and changes in physico-mechanical properties of fabrics, e.g. their handle (Etters, Husain, & Lange, 1999). The disadvantages of scouring with sodium hydroxide have motivated textile industry to introduce more enhanced biological agents which would be as effective in removing non-cellulose substances as sodium hydroxide but would not have damaging effects on cellulose and would be less water and energy consuming. Favourable effects of scouring have been obtained with the enzymes pectinases (Buchert, Pere, Puolaka, & Pertti, 2000; Csiszar et al., 2001), which catalyse the hydrolysis of pectin substances. The potential advantages that make the enzyme scouring commercially appealing are a higher quality of the fibres, less wastewaters with lower biological oxygen demand (BOD5), chemical oxygen demand (COD), total organic carbon (TOC), lower process time, cotton weight loss and harshness to handle, economy of energy and compatibility with other procedures, equipment and materials (Cavaco-Paulo & Gübitz, 2003).

The first studies of enzyme treatment for scouring, that is cleaning of cotton fibres were carried out by researchers (Röβner, 1995; Schacht, Kesting, & Schollmeyer, 1995). Many other researchers followed their path. On the basis of their studies, they concluded that a simple procedure with pectinases in the presence of non-ionic surfactant is sufficient to attain good absorbency (Buchert et al., 2000; Galante & Formantici, 2003; Traore & Buschle-Diller, 2000).

The next step, following scouring in textile processing, is the bleaching process. The greyness of cotton is due to the natural pigments and matter present in the fibres. Cellulose fibres are most frequently bleached by HP resulting in high and uniform degrees of whiteness. Hydrogen peroxide is not ecologically disputable. Namely, the bleaching process is conducted in an alkaline bath at pH 10–12 and at temperatures up to 120 °C. Due to high working temperature, a large amount of energy is consumed. Auxiliary chemicals added into the bath increase the TOC and COD values of effluents. Consequently, the textile industry is considered one of the biggest water, energy and chemical consumers (Alaton, Insel, Eremektar, Babuna, & Orhon, 2006). Bleaching with peracetic acid (PAA) is an alternative to bleaching with HP (Hickman, 2002; Tavčer, 2010). Results of dyeing of cotton fabrics with a bifunctional reactive dye were significantly improved when the fabric after bleaching with HP was treated with catalase for the elimination of HP residues from the fabrics (Amorim, Gasques, Jürgen, & Scharf, 2002).

The main purpose of this research was to compare the properties of bioscouring and PAA-bleached cotton fabrics, with conventionally treated fabrics (alkaline scoured and bleached with HP). The whiteness degree, tenacity at maximum load and water absorbency of the treated cotton fabrics were evaluated. After the treatments, the pH, TOC, COD and BOD5 values of the remaining baths were measured. The amount of water and heating energy used during the treatments and rinsing was measured.

Materials and methods

Materials

- Desized cotton fabric, 120 g/m^2.
- Enzymes–alkaline pectinase Bioprep 3000L from Novozymes, Denmark.
- Enzyme–acid pectinase Viscozyme 120L from Novozymes, Denmark.
- Textile auxiliaries agents: Cottoblanc HTD-N (anionic wetting and dispersing agent, alkansulphonate with chelator) was supplied from CHT, Germany; Felosan RG-N (non-ionic wetting agent) was supplied from CHT, Germany; Locanit S (ionic-nonionic dispersing agent) was supplied from Cognis, Germany; Lawotan RWS (nonionic wetting agent) was supplied from CHT, Germany.
- H_2O_2 35% and PAA as a 15% equilibrium solution in the commercial bleaching agent Persan S15 were obtained from Belinka, Slovenia.
- NaOH, Na_2CO_3 and acetic acid were obtained from Sigma Aldrich.

Treatment methods

The desized cotton fabric was scoured according to three different procedures using sodium hydroxide, alkaline pectinases or acid pectinases. The scoured fabrics were bleached with two bleaching agents: HP and Persan S15. The abbreviation of the treatments and treated samples with treatment description are presented in Table 1.

Alkaline scouring (AS) was carried out in a bath containing 2 g/L of Cottoblanc HTD-N and 4 g/L of NaOH at 95 °C for 40 min. Traditional bleaching with HP was carried out in a bath containing 7 g/L of H_2O_2 (35%), 2 g/L Cottoblanc HTD-N, 4 g/L of NaOH (100%) at 95 °C for 45 min. After alkaline scouring and peroxide bleaching, the fabrics were neutralized with a neutralizing bath containing acetic acid rinsed in cold water.

Table 1. Abbreviations of the treatments and treated samples with treatment descriptions.

Treatment/sample abbreviation	Treatment description
AS	Alkaline scouring
BS-ALP	Bioscouring with alkaline pectinases
BS-ACP	Bioscouring with acid pectinases
BL-HP	Bleaching with hydrogen peroxide
BL-PAA	Bleaching with peracetic acid
BS-ALP/BL-PAA	One-step bioscouring with alkaline pectinase and bleaching with peracetic acid
BS-ACP/BL-PAA	One-step bioscouring with acid pectinase and bleaching with peracetic acid

Enzymatic scouring and one-step treatments were performed with two types of pectinases: an alkaline pectinase Bioprep 3000L 0.055% owb (on weight of bath), Na_2CO_3 to pH 8, for 60 min at 55 °C, in the presence of 0.5 mL/L Lawotan RWS (nonionic wetting agent) and an acid pectinase Viscozyme 120L 3 g/L, CH_3COOH to pH 5, for 60 min at 55 °C, in the presence of 1 g/L Felosan RG-N (nonionic wetting agent), 2 mL/L Locanit S (ionic–nonionic dispersing agent), then the temperature of the bath was increased up to 80 °C about 10 min to deactivate the enzymes. To activate PAA in BS-ACP/BL-PAA treatment, the pH was adjusted to 8 after 30 min. Demineralized water was used in all processes. The treatments were performed on the apparatus by Mathis Labomat Typ BFA12 loaded with 50 g of fabric at a liquor ratio of 1:20. After each treatment, the samples were washed in hot water, rinsed twice in cold water and air dried. The ingredients and treatment conditions are presented in Tables 2 and 3.

Analytical methods

Prior to the measurements, samples were conditioned for 24 h at 20 °C and 65% relative humidity.

Table 3. Data conditions for treatments.

Treatment	Treatment conditions
AS	95 °C, 40 min
BS-ALP	55 °C, 60 min; 80 °C, 10 min
BS-ACP	55 °C, 60 min; 80 °C, 10 min
AS + BL-HP	95 °C, 45 min
AS + BL-PAA	55 °C, 40 min
BS-ALP/BL-PAA	55 °C, 60 min
BS-ACP/BL-PAA	55 °C, 60 min

The degree of whiteness was measured with a spectrophotometer Spectraflash SF600 Plus (Datacolor, Switzerland) using the CIE method according to EN ISO 105-J02:1997(E) standard.

Weight loss – The weight loss due to the pretreatments was determined by weighing the fabric samples before and after pretreatment and was expressed in per cent.

The water absorbency was measured according to DIN 53 924 (the velocity of the soaking water for textile fabrics, the method for determining the rising height).

Measurements of *tenacity at a maximum load* were performed on an Instron Universal Tensile Tester Model

Table 2. Ingredients of treatments.

	Treatment						
Ingredient	AS	BS-ALP	BS-ACP	BL-HP	BL-PAA	BS-ALP/BL-PAA	BS-ACP/BL-PAA
0.055% owb[a] Bioprep 3000L	−	+	−	−	−	+	−
3 g/LViscozyme 120L	−	−	+	−	−	−	+
2 g/L Cottoblanc HTD-N	+	−	−	+	−	−	−
1 g/L Felosan RG-N	−	−	+	−	−	−	+
2 mL/L Locanit S	−	−	+	−	−	−	+
0.1 mL/L Lawotan RWS	−	−	−	−	+	+	−
0.5 mL/L Lawotan RWS	−	+	−	−	−	−	−
15 mL/L Persan S15	−	−	−	−	+	+	+
7 g/L H_2O_2 35%	−	−	−	+	−	−	−
4 g/L NaOH 100%	+	−	−	+	−	−	−
2 g/L Na_2CO_3 100%	−	+	−	−	+	−	−

Notes: + the ingredient is included in the treatment bath.
– the ingredient is not included in the treatment bath.
[a]owb (on weight of bath).

5567 (Instron, UK), following the standard test method ASTM D5034 – 09(2013) under standard atmospheric condition (65 ± 2% relative humidity and 21 ± 1 °C temperature) as directed in ASTM D1776(2004). Sample size had a width of 100 mm and gauge length 200 mm with strain rate of 300 mm/min. From the value of breaking load (N), the tenacity values (cN/10^{-6} kg m^{-1}) are calculated by the following formula:

$$\text{Fabric Tenacity } (\text{cN}/10^{-6}\ \text{kg m}^{-1}) = \frac{\text{Breaking load (N)}}{\text{Specimen width (mm)} \times \text{Fabric area dencity } \left(\frac{\text{g}}{\text{m}^2}\right)} \times 100$$

The mean degree of polymerization (DP) was determined with the viscosimetric method in Cuoxam [$Cu(NH_3)_4$] ($(OH)_2$) according to IS 244:1984. Cuprammonium hydroxide (Cuoxam) solution is one of the most important cellulose solvents used for the DP measurements.

Samples of remaining bleaching and scouring baths were collected after all treatments. Their ecological parameters such as pH, TOC, COD and BOD5 were measured. The amount of water and heating energy used during the treatments and rinsing was measured.

The pH was measured using a pH meter MA5740 (Iskra, Slovenia).

The total organic carbon (TOC) was measured with a TOC-5000A (Shimadzu, Japan) according to ISO 8245:1999.

The chemical oxygen demand (COD) was performed according to SIST ISO 6060:1996, *biological oxygen demand* (BOD5) according to SIST ISO 5815-1:2003, and *biological degradation* as a ratio of BOD5 and COD.

Results and discussion

Fabric properties. Whiteness

The whiteness values, the loss of weight, tenacity at maximum load, rising height and DP of various sample treatments are presented in Table 4. The alkaline-scoured sample (AS) had a whiteness of 21.3, the bioscoured sample with alkaline pectinases (BS-ALP) had 10.6 and the bioscoured sample with acid pectinases (BS-ACP) had 10.1. After the alkaline scouring, the fibres swelled, became smoother and clean of non-cellulose impurities and the degree of whiteness increased. AS is more intensive and removes some of the coloured substances from the fibre that the bioscouring does not. The whiteness degree increased significantly after bleaching with HP, and the differences in whiteness from the previous scouring disappeared. The highest whiteness (85.9) was obtained on the bleached with HP and the bioscoured sample with acid pectinases (BS-ACP/BL-HP). Not much less (85.4) was obtained on the bleached with HP and the bioscoured sample with alkaline pectinases (BS-ALP/BL-HP), while lower whiteness (84.3) was obtained on the bleached with HP and alkaline-scoured sample (AS + BL-HP). The whiteness degree decreased significantly after bleaching with PAA (74.5, 58.3 and 57.6 with AS, BS-ACP and BS-ALP sample, respectively). This occurs because bleaching with PAA is conducted at a low temperature and pH, where the impurities remaining after scouring could not be fully oxidized. Bioscoured fibres, which were not treated at high temperature and high pH, contained more waxes and other impurities that hindered the successful oxidation with PAA. Similar results were obtained by Križman, Kovač, and Tavčer (2005).

Weight loss

The loss of weight demonstrates that alkaline scouring is more intensive than enzymatic scouring. The loss of weight after alkaline scouring was 1.58% and after bioscouring was 0.94 and 0.37% with alkaline pectinases and acid pectinases, respectively. The total weight loss after scouring and HP bleaching was similar for all samples. This suggests that HP bleaching removed a large portion of compounds, which remained on fibres after scouring. Peracetic acid bleaching also removed a certain part of the non-cellulosic substances, which remained on fibres after scouring, but the quantity was lower relative to HP bleaching. We can conclude that the high temperature and high pH are conditions that contribute decisively to the removal of non-cellulosic impurities. Specifically, waxes cannot be removed completely when all processes are conducted at mild conditions, as is the case for bioscouring and PAA bleaching. Similar results were obtained by Tzanov, Calafell, Guebitz, and Cavaco-Paulo (2001).

Water absorbency

The remaining substances influence the water absorbency. All the treated samples revealed very good absorption properties. The highest rising height was measured on the traditionally alkaline-scoured (3.1 cm) and on the bleached with HP and alkaline-scoured sample (3.3 cm). Bleaching improved the absorbency of the scoured fabrics, particularly of enzymatically scoured ones. The rising height of the bioscoured samples was a bit lower but the differences among all the treated samples were not significant. Similar results were obtained by Tzanov et al. (2001).

Table 4. Whiteness, weight loss, tenacity at maximum load, rising height and DP of various sample treatments.

Treatment	Whiteness	Weight loss (%)	Tenacity at maximum load (cN/ 10^{-6} kg m^{-1})	Rising height (cm)	DP
Desized only cotton fabric sample	12.6		17.53	0	2473
AS	21.3	1.58	18.48	3.1	2421
BS-ALP	10.6	0.94	16.87	2.6	2374
BS-ACP	10.1	0.37	15.98	2.8	2442
AS + BL-HP	84.3	1.67	15.73	3.3	1763
BS-ALP + BL-HP	85.4	1.79	15.87	2.9	1993
BS-ACP + BL-HP	85.9	1.62	16.28	3.0	1932
AS + BL-PAA	74.5	1.43	15.89	2.9	2257
BS-ALP/BL-PAA	57.6	1.08	12.86	2.8	2375
BS-ACP/BL-PAA	58.3	0.73	17.25	2.9	2297

Tenacity at maximum load

There were no significant differences in the tenacity at maximum load between the desized only cotton fabric sample and differently treated samples. The highest value was measured on the alkaline-scoured sample (18.48 cN/10^{-6} kg m^{-1}). The high tenacity of the alkaline-scoured sample is a result of the contraction of the fabric exposed to a high process temperature. After the bleaching with PAA of the bioscoured sample with alkaline pectinases, the tenacity at maximum load decreased significantly (12.86 cN/10^{-6} kg m^{-1}).

Degree of polimerization

The results of DP demonstrate that bleaching with HP decreased the DP significantly of 1763–1993, while other processes preserved the DP values close to the starting value (2473).

Ecological parameters

The remaining concentrations in baths of different scouring and bleaching processes of cotton fabrics, TOC values, COD, BOD5, biological degradability (BOD5/COD) and the final pH values are presented in Table 5.

Traditional alkaline scouring and bleaching with HP are conducted in an alkaline environment. The final pH value was around 12. These baths should be neutralized prior to drainage into the sewage system. During the neutralization, salts that additionally load wastewaters are produced. The processes of bioscouring and bleaching with PAA occurred between pH 5 and 8, and the final pH values of the bioscouring bath were 5.8 and 7.9 with acid pectinases and alkaline pectinases, respectively. While bleaching with PAA and at both combined processes, the final pH value of the bath was near 6. Since none of these treatment processes requires a neutralization of the fabric, the treatment process can be shorter and less expensive. Additionally, the remaining baths do not require the neutralization step prior to drainage into the sewage system, which also reduces the cost of processes.

Bioscouring, traditional bleaching with HP and alkaline scouring baths had low TOC, COD and BOD5 values, and while bleaching with PAA and at both combined processes, baths had significantly higher values. The bioscouring bath with alkaline pectinases exhibited the lowest TOC, COD and BOD5 values, which were so low that they did not exceed the limit values (TOC 60 mg C/L, COD 200 mg O_2/L and BOD5 30 mg O_2/L) for direct drainage into the sewage system

Table 5. The ecological parameters – the remaining concentrations in baths of different scouring and bleaching processes of cotton fabrics, total organic carbon (TOC), chemical oxygen demand (COD), biological oxygen demand (BOD5), biological degradability (BOD5/COD) and the final pH values.

Treatment	pH	TOC (mg C/L)	COD (mg O_2/L)	BOD5 (mg O_2/L)	BOD5/COD
AS	12.0	650	2650	280	0.105
BS-ALP	7.9	55	150	27	0.180
BS-ACP	5.8	1850	4100	2120	0.517
BL-HP	12.0	235	/	1950	/
BL-PAA	6.0	1900	5800	2790	0.481
BS-ALP/BL-PAA	6.0	2050	5850	3000	0.512
BS-ACP/BL-PAA	6.0	3150	7800	3210	0.411

Table 6. The amount of water and energy required for the treatment of 1 kg of fabric at a liquor ratio 1:20 for different processes.

Treatment	Water consumption (L)	Vapour demand (kg/L bath)
AS	135	1.2
BS-ALP	85	0.7
BS-ACP	85	0.7
BL-HP	135	0.6
BL-PAA	80	0.2
AS + BL-HP	270	1.8
BS-ALP + BL-PAA	165	0.9
BS-ACP + BL-PAA	165	0.9
BS-ALP/BL-PAA	95	0.5
BS-ACP/BL-PAA	95	0.5

(Decree, 2011). The bioscouring bath with acidic pectinases had high TOC and COD values that were even higher than alkaline scouring. This occurs because the initial composition of the bath contained more auxiliary agents than the bath with alkaline pectinases, which contained only enzyme and wetting agent. We could not determine the real COD value after bleaching with HP, because a certain amount of the non-used HP remained in the bath.

The processes of bleaching with PAA had higher TOC and COD values, because PAA is an organic compound, which contributes to higher TOC and COD values. Peracetic acid is decomposed in the waste bath to acetic acid, which is not ecologically disputable. The BOD5 values are high with the bioscouring baths with acidic pectinases (2120 mg O_2/L) and the baths with PAA (between 2790 and 3210 mg O_2/L). The baths with alkaline pectinases exhibited the lowest BOD5 value (27 mg O_2/L). The baths with enzymes and PAA were biodegradable, and the bath after alkaline scouring was non-degradable.

Consumption of water and energy

The amount of water and energy required for the treatment of 1 kg of fabric at a liquor ratio 1:20 for different processes is presented in Table 6. The amount of water consumed for alkaline scouring and bleaching with HP was higher than the amount of water consumed for bioscouring and bleaching with PAA. After alkaline scouring and bleaching with HP, the fabric must be neutralized, while after bioscouring and bleaching with PAA is not required, because the pH value is only slightly acidic and is neutralized during the first rinsing. The process of bioscouring and bleaching with PAA consumed 165 L or only 61.1% of water relative to alkaline scouring and bleaching with HP (270 L). The combined process of bioscouring and bleaching with PAA consumed 95 L, i.e. only 57.5% in comparison with two-bath process (165 L), and only 35.2% in comparison with conventional pre-treatment process (270 L).

Conventional processes of scouring and bleaching were performed at temperatures of 95 °C, whereas bioscouring and bleaching with PAA were conducted at a temperature of 55 °C. Due to the lower temperature, less energy was required, which is presented in Table 6. The process of bioscouring consumed 0.7 kg/L or 58.3% of the steam, which was required during alkaline scouring (1.2 kg/L), and the process at bleaching with PAA consumed 0.2 kg/L or 33.3% of steam, which was required at bleaching with HP (0.6 kg/L). The combined process consumed 0.5 kg/L or 55.5% of the steam, which was required during two-bath process (0.9 kg/L), and only 27.7% of the steam required for alkaline scouring and bleaching with HP (1.8 kg/L).

Conclusions

Biopreparation of the cotton fibres is an enzyme-aided process by which the noncellulosic “impurities” (waxes, pectic supstances, proteins, lignin, etc.) are removed mainly by pectinases. Enzymes act under mild conditions (pH, temperature) with low consumption of water and energy relative to the conventional alkaline scouring with hot caustic soda, high consumption of water and energy, and chemical-intensive process. Conventional processes of scouring and bleaching were performed at temperatures near the boiling point, whereas bioscouring and bleaching with PAA were conducted at a temperature of 55 °C. Due to the lower temperature, less energy was required. Bleaching with PAA can substitute bleaching with HP when medium degree of whiteness is demanded. The bioscouring and bleaching with PAA processes cause no damage to fibres and this is one of the benefits of such processes. The consumption of water and energy is the lowest at combined scouring/bleaching treatments. Consequently, at these processes arises the lowest amount of effluents and the produced wastewater is biodegradable. Thus, bioscouring and bleaching with PAA provide an environmentally friendly alternative to the conventional process.

Disclosure statement

No potential conflict of interest was reported by the author.

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