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Enhancing hydrophilicity of bioscoured flax fabric by emulsification post-treatment

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ABSTRACT

Bioscouring of flax fabrics was carried out using combinations of different pectinase preparations and different surfactants having varying ionic natures. In order to attain satisfactory fabric hydrophilicity, the enzymatic scouring step was necessary to be followed by emulsification post-treatment, in which the temperature of the bioscouring liquor was raised to 90 °C for 2 min. Flax fabrics subjected to such combined treatments (bioscouring followed by emulsification post-treatment) were evaluated by measuring their physico-chemical properties, morphological features and metrological parameters and comparing them with the corresponding properties recorded for the grey flax fabric. The measurements showed high improvement in the water absorbency and percent moisture regain of the treated fabrics. The physico-chemical properties, morphological structure and metrological parameters of the treated flax fabrics were found to be dependent on both the efficiency of the enzyme preparation and the ionic nature of the used surfactant.

1. Introduction

Cellulosic fibers are treated to become more absorbent and easily wetted with water or other aqueous solutions, in a process known as scouring. The success of the subsequent wet processing operations like bleaching and dyeing depends on the efficiency of the scouring process (Hiseh, Thompson, & Miller, 1996). The constituents of cellulosic fibers mainly responsible for their hydrophobicity are localized in the cuticle of the primary wall, where the pectin acts as cementing material including waxes (Sakai, Sakamoto, Hallaert, & Vandamme, 1993). The major goal of any scouring process is to improve the water absorbency of cellulosic fibers by removing such water repellent components from the fibers, which facilitates uniform dyeing and finishing. Efficient scouring process should result in considerable removal of both pectic and waxy substances.

Traditionally, for cotton fabrics for example, this preparation process is carried out in an aqueous alkaline medium at the boil. Alkaline scouring has large energy requirements, consumes large quantities of alkali and requires an extensive rinsing process that loads the washing effluent with environmentally harmful chemicals (Traore & Buschle-Diller, 2000). 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 its physico-mechanical properties. For these reasons, during the last two decades, several attempts have been made to replace the conventional alkaline scouring of cotton with enzymatic systems working at milder conditions, in order to improve the process output and to reduce its environmental impact. Different individual enzymes and their mixtures were studied (Achwal, 1992; Anis & Eren, 2002; Agrawal, Nierstrasz, & Warmoeskerken, 2008; Aly, Moustafa, & Hebeish, 2004; Buchert, Pere, Pualakka, & Nousiainen, 2000; Cavaco-Paulo, 1998; Csiszar, 1998; Calafell & Garriga, 2004; Choe, Nam, Kook, Chung, & Cavaco-Paulo, 2004; Cavaco-Paulo, Almeida, & Bishop, 1996; Csiszar et al., 2001a; Csiszar, Urbanszki, & Szakacs, 2001b; Durden, Etters, Sarkar, Henderson, & Hill, 2001; Degani, Gepstein, & Dosoretz, 2002; Etters, 1999; Hartzell & Durrant, 2000; Hashem, 2007; Klug-Santner et al., 2006; Kalantzi, Mamma, & Paul, 2008; Li & Hardin, 1998b; Li & Hardin, 1997; Lin & Hiseh, 2001; Robner, 1993; Sahin & Gursay, 2005; Sangwatanaro & Choonukulpong, 2003; Tzanov, Calafell, Guenitz, & Cavaco-Paulo, 2001; Wang, Fan, Gao, & Chen, 2006; Wang, Fan, Hua, & Chen, 2007a; Wang, Fan, Hua, Gao, & Chen, 2007b).

Pectinases appear to be the most suitable enzymes for cotton bioscouring, being capable of depolymerising the pectin, breaking it down to lower molecular weight, water soluble oligomers

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(McNeil, Darvill, Fry, & Albertsheim, 1984), and thereby improving the absorbency of the fabric without causing cellulose destruction. Pectinase is a multi-component enzymatic preparation, having several enzymatic activities and differing in substrate preference, reaction mechanism, and action pattern. Jayani, Saxena, and Gupta (2005) made a review on pectinolytic enzymes which includes their classification. The depolymerising enzymes are divided into polygalacturonases (PG), and lyases (PL) (or trans-eliminases). Pectin lyase (EC 4.2.2.10) catalyzes cleavage of α-1,4-glycosidic linkage in pectin. Pectate lyase (EC 4.2.2.2) catalyzes cleavage of α-1,4-glycosidic linkage in pectic acid. These two enzymes cleave linkages by β-elimination and generate products with 4,5-unsaturated residues at the non-reducing end. Polygalacturonase (EC 3.2.1.15) catalyzes cleavage of α-1,4-glycosidic linkage in pectic acid by hydrolysis (Sakai et al., 1993). Lyases have optimal pH around 8.5 and (except for pectin lyase) require divalent cations (Collmer, Ried, & Mount, 1988). Hydrolases have optimal pH 6.0 or lower and do not require divalent cations. Several pectinases that have pectate lyase activity are commercially available and could be used successfully in cotton bioscouring (Anis & Eren, 2002; Etters, 1999).

Although many research work has been carried out on bioscouring of cotton fabrics, very few attempts have been made to try enzymatic treatment for other cellulosic fabrics like flax (Abdel-Halim, Opwis, Knittel, & Schollmeyer, 2007; Ibrahim, El-Hossamy, Hashem, Refai, & Eid, 2008; Lipp-Symonowicz, Tańska, Wołukanis, & Wrzosek, 2004; Sharma, Whiteside, & Kernaghan, 2005).

Flax has provided high quality fibers used in linen. Despite the fact that cotton has preempted flax as natural cellulosic fiber for textiles, linen is valued for its distinctive appearance and comfort and still maintains a share of the luxury textiles market. In addition to 100% flax products, blends with other fibers periodically appear as part of the fashion trends. Particularly, moisture management and air permeability are improved with increasing amounts of flax in cotton blends. In contrast to cotton, flax fiber is found in the stem of the plant. It is classified with other fibers such as hemp, ramie and jute, as bast fibers. Cotton and bast fibers are constructed from fibrils which are chemically composed of cellulose and certain quantities of non-cellulosic impurities. The impurities in cotton fall into protein, pectic substances, waxes, mineral and coloring matter. Bast fibers, with larger amounts of impurities than cotton, contain in addition, lignin and hemicelluloses. These additional impurities contribute very important properties to these fibers and must be taken into account in the subsequent treatments (such as scouring and bleaching) to which the fiber may be subjected (Koch, 1994).

The aim of the present study is to treat flax fabric enzymatically in the presence of different surfactants having varying ionic nature and to examine the effect of such combined treatments on the physico-chemical properties, morphological structure and metrological parameters of the biotreated flax fabric.

2. Materials and methods

2.1. Flax substrate

The substrate used was plain weave flax fabric 175 g/m^2, from the stock of Institute of Natural Fibers & Medicinal Plants, Poznan, Poland.

2.2. Enzymes used in bioscouring

Four enzyme preparations were used for bioscouring of flax fabric, three of them (Texazym SCW, Texazyme SER-3 conc. and Texazym DLG) were supplied by Inotex Company. The fourth enzyme Scourzym L was supplied by Novozymes. Texamyl BL, bacterial alpha-amylase was supplied by Inotex Company and was added individually with each enzyme preparation in the treatment bath as an enzymatic desizing agent.

2.3. Surfactants added to the bioscouring bath

Four surfactants of different ionic nature (anionic and non-ionic) were tried individually with each enzyme. Texazym A (anionic) and Texazym T (non-ionic) were supplied by Inotex Company. Sulfolen 148 (anionic) was supplied by ROTTA GmbH and Surfynol 104 (non-ionic) was supplied by Air Products.

2.4. Enzymatic scouring

According to the specification sheets provided by the enzymes suppliers, the mode of action and optimum treatment pH of the four enzyme preparations can be summarized as follows. Texazym SCW is a commercial (non-ionic) preparation used for pretreatment of bast fibers at optimum working pH of 8–9.5 (soda ash). Texazym SCW catalyzes decomposition of pectin, hemicellulose and lignin with very low impact on cellulosic fibers. Texazyme SER-3 conc. is a commercial (non-ionic) preparation suitable for pretreatment of bast fibers in the stage of tow, roving, wovens or knittings with very low impact on cellulosic fibers. Its optimum working pH is 7.5–9.5 (soda ash) and it catalyzes decomposition of pectin, hemicellulose and lignin. Texazym DLG is a commercial (non-ionic) preparation applied optimally at pH 3.5–6 (acetic acid). It catalyzes decomposition of hemicellulose and partially lignin and can affect cellulosic fibers. Scourzym L is an alkaline pectinase used for bioscouring of cotton, linen, hemp and their blends at optimum working pH of 8–9 (soda ash). It removes pectin and other impurities from cellulosic fibers without degrading the cellulose itself.

For enzymatic scouring of flax fabric, each enzyme was applied in a concentration of 0.5% based on weight of fabric (owf) in combination with 0.5% (owf) Texamyl BL and 0.1% (owf) surfactant. Each enzyme was tried individually with each of the four mentioned surfactants. All experiments were carried out in duplicates in stopper-flasks using a material (flax fabric) to liquor ratio of 1:20. The enzymatic scouring was run for 2 h and the temperature was kept at 60 °C throughout the reaction duration using a thermostatic shaking water bath.

2.5. Emulsification post-treatment

At the end of bioscouring duration, the temperature of the reaction medium was raised to 90 °C for 2 min. This post-treatment aimed at melting and emulsifying the naturally occurring waxes through the action of surfactants existing already in the bioscouring liquor, this is in addition to deactivate the enzymes effect at this high temperature. After the post-treatment, the fabric was washed thoroughly with hot water, rinsed with cold water and then air dried.

2.6. Testing and analysis

2.6.1. Water absorbency (wettability)

Water absorbency of flax fabric was measured quantitatively by means of the drop test (JIS, 1990), before and after the enzymatic scouring to evaluate the bioscouring efficiency. Air dried samples were climatized at 65% relative humidity for at least 12 h. The time interval (s) between the first contact of the water drop with the fabric surface and its disappearance into the bulk of the fabric was counted as the wetting time. For each sample, the wetting time was counted in at least 10 different areas and the average wetting time was calculated. Wetting time of 1 s or less gives an indication that the fabric has adequate absorbency (Hiseh & Cram, 1999).

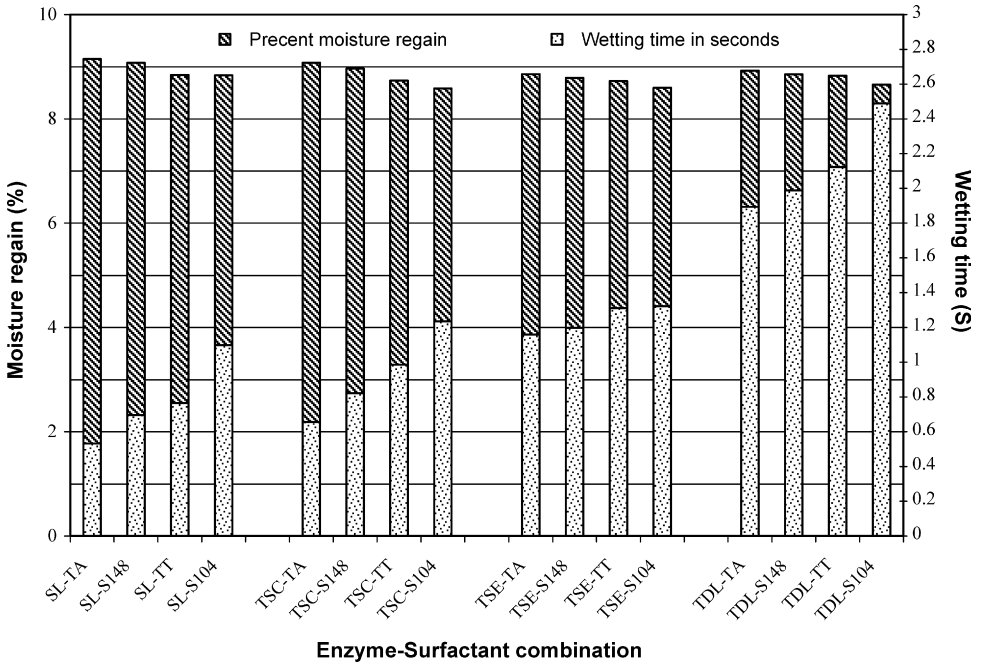

Fig. 1. Wettability and hygroscopicity of flax fabrics treated with different enzyme–surfactant combinations.

2.6.2. Hygroscopicity

Hygroscopicity of grey and bioscoured flax fabrics (in duplicates) were estimated according to Polish Standard (PN-80/P-04635, 1990). Air dried samples were climatized at 65% relative humidity for at least 24 h and then weighed to get the mass of wet sample (M_w). The mass of dry sample (M_d) was determined by drying the wet samples in an air-circulated oven at 105 °C for 16 h, followed by cooling in a desiccator and weighing. The humidity percent was calculated using the following equation:

$$\text{humidity } (\%) = \frac{M_w - M_d}{M_d} \times 100$$

2.6.3. Determination of pectin and lignin contents

Samples of grey and bioscoured flax fabrics (in duplicates) were subjected to wax removal via extraction with toluene/ethanol mixture (38/62) for 24 h in a soxhlet apparatus. The pectin content was estimated in the wax free samples according to the method described by Jin and Masako (2001) and the lignin content was estimated according to the method described by Hortling, Turunen, and Sundquist (1992).

2.6.4. Metrological properties measurements

Samples of grey and bioscoured flax fabrics (in duplicates) were cut into strips (5 cm × 30 cm) and climatized at 65% relative humidity for at least 24 h. The tensile properties of the climatized samples were tested according to Polish standard (PN-EN ISO 13934-1, 2002).

2.6.5. Scanning electron microscopy

The morphological structure of grey and differently treated flax fabrics was evaluated by scanning electron microscopy (SEM). The photos were taken with the use of an S-3400N Hitachi Scanning Electron Microscope in high vacuum mode (secondary electron detector SE). Natural fibers as non-conductive materials were initially coated with gold in order to ensure proper parameters. Parameters used were views magnification of 500, stage height of 60 mm and accelerating voltage of 15 kV.

3. Results and discussions

3.1. Water absorbency and hygroscopicity

Fig. 1 shows the wetting time and percent moisture regain of flax fabrics bioscoured and post-treated with different enzyme–surfactant combinations. It was found that the absorbency and hygroscopicity of the flax fabrics depend to great extent on both the efficiency of the enzyme preparation and the ionic nature of the used surfactant. While ionic surfactants could decrease enzyme activity, they are known to have a better emulsifying ability than non-ionic surfactants which do not affect the enzyme activity (Sawada & Kajiwara, 2004). It is clear from Fig. 1 that regardless the enzyme used, using the anionic surfactant Texazym (A) resulted in the best fabric wettability and hygroscopicity, and the two properties improved following the ascending order Surfynol (104) < Texazym (T) < Sulfolen (148) < Texazym (A). Also regardless the surfactant used, using the enzyme preparation Scourzym L resulted in the best fabric wettability and hygroscopicity, and the two properties improved following the ascending order Texazym DLG < Texazym SER 3 < Texazym SCW < Scourzym L.

The evaluation of the enzymatic scouring effect was performed monitoring the water absorbency and hygroscopicity of the treated samples. For a fabric to be bleached and dyed successfully, it should be highly water absorbent (the wetting time should be less than 1 s). In order to be comfortable for the wearer, the hygroscopicity of the fabric should be higher than 8%. Enzymatic scouring was carried out at the beginning of the work without applying the emulsification post-treatment and although all enzyme preparations were applied using the optimum conditions according to the supplier recommendations, there was no improvement in neither the wettability

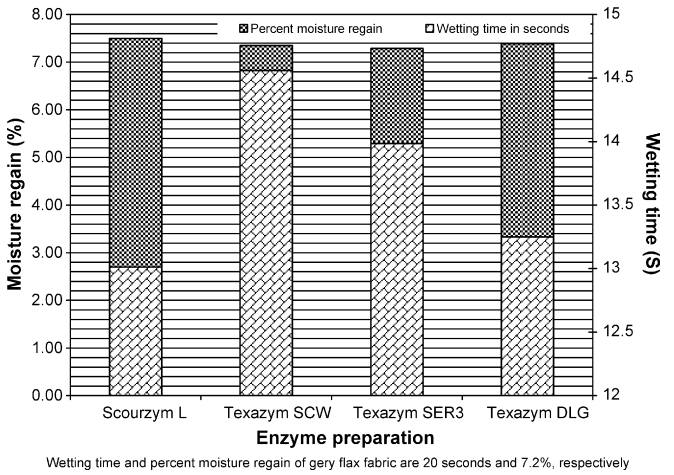

Fig. 2. Wettability and hygroscopicity of flax fabrics treated with different enzyme preparations.

nor the hygroscopicity of the flax fabric. Fig. 2 shows the wetting time and percent moisture regain of flax fabrics bioscoured using different enzyme preparations without surfactant post-treatment. It is clear from the figure that the treated flax fabrics did not show satisfactory improvement neither in wetting time nor in hygroscopicity. When the same bioscouring experiments were repeated but followed by emulsification post-treatment, the wettability and hygroscopicity improved to great extent (wetting time less than 1 s and more than 9% moisture regain). Surfactants with different ionic natures were added individually with each enzyme and at the end of the enzymatic scouring duration; the temperature was raised to 90 °C for 2 min. On the other hand, when the emulsification treatment with surfactant was carried out alone without bioscouring, the flax fabric showed neither improved wettability nor improved moisture regain, compared with flax fabrics which undergo bioscouring followed by emulsification post-treatment.

The poor water absorbency and hygroscopicity of flax fabrics when applying enzymatic scouring alone and their general improvement after applying the emulsification post-treatment give a good idea about the specific and selective action of enzymes. Simply, the enzyme preparations acted properly in degrading their substrates (pectin and/or lignin) but did not affect wax which is not one of their substrates. Since pectins are not the only non-cellulosic component responsible for flax hydrophobicity but waxes as well, it was necessary to emulsify the remaining waxes in a post-treatment step in order to get wetting time less than 1 s and moisture regain percent higher than 8%. Emulsification post-treatment becomes unnecessary if the flax fabric is intended to be bleached with H_2O_2, since wax emulsification and removal takes place normally under the conditions of the bleaching process (Abdel-Halim et al., 2007).

3.2. Pectin and lignin contents

The percent removal of pectin and lignin was found to be a function of both the composition and mode of action of the enzyme preparation used, and the ionic nature of the surfactant applied. Fig. 3 shows the pectin and lignin contents of flax fabrics bioscoured and post-treated with different enzyme–surfactant combinations. As a general remark, one can see that for the same enzyme, the pectin and lignin contents of flax fabrics treated with enzyme–anionic surfactant combination are higher than the corresponding values recorded for flax fabrics treated with enzyme–non-ionic surfactant combination. This observation supports the findings of Sawada and Kajiwara (2004), that ionic surfactants retard the enzyme activity more than do non-ionic surfactants.

Considering the four enzyme combinations with the anionic surfactant Texazym (A), it is clear that there is a trend in the enzymes efficiency in removing pectin and lignin, which follow the ascending order Texazym DLG < Texazym SER 3 < Texazym SCW < Scourzym L. Regarding the other surfactants–enzymes combinations, no clear trend in the pectin and lignin removal efficiency was observed.

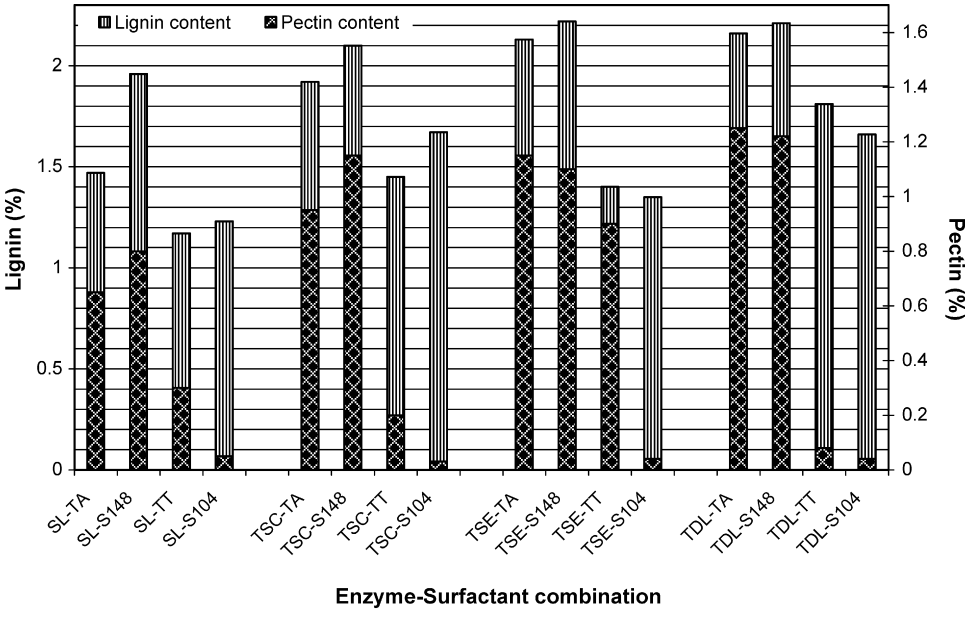

Fig. 3. Pectin and lignin content of flax fabrics treated with different enzyme–surfactant combinations.

Table 1
Tensile properties of flax fabrics treated with different enzyme preparations.

Flax sample	Enzyme–surfactant treatment combination	Force (N)	Loss in tensile strength (%)	elongation at break (%)
Gray	–	1022.20	0	12.29
1	Scourzym L–Texazym (A)	951.56	6.9105	12.73
2	Texazym SCW–Texazym (A)	917.32	10.2602	14.76
3	Texazym SER–Texazym (A)	826.35	19.1596	10.84
4	Texazym DLG–Texazym (A)	599.67	41.3353	11.44

3.3. Tensile properties

Referring to the results of water absorbency and hygroscopicity (Fig. 1), it is observed that upon using each of the four surfactants with one of the enzymes under investigation, the use of the anionic surfactant Texazym (A); resulted in highly improved fabric absorbency and hygroscopicity. Based on this observation, and for tensile properties assessment, Texazym (A) was chosen for individual treatment with each of the four enzyme preparations in bioscouring of four larger flax samples to examine the effect of each enzyme preparation on the tensile properties of the treated fabrics. Fig. 1 and Table 1 illustrate that flax fabric treated with the bioscouring combination Scourzym L–Texazym (A) showed improved absorbency and hygroscopicity in addition to minimal loss in tensile properties. The loss in tensile properties increased in the case of bioscouring using the other Enzyme–Texazym (A) combinations and this loss follows the ascending order Scourzym L–Texazym (A) < Texazym SCW–Texazym (A) < Texazym SER–Texazym (A) < Texazym DLG–Texazym (A). Referring to Fig. 3, it is obvious that the efficiency of the above mentioned Enzyme–Texazym (A) combinations in removing pectin and lignin showed an opposite trend and follows the descending order Scourzym L–Texazym (A) > Texazym SCW–Texazym > Texazym SER–Texazym (A) > Texazym DLG–Texazym (A). This trend suggests that the loss in tensile strength can be ascribed to increased lignin and pectin removal together with increased hemicellulose removal and/or cellulose degradation.

3.4. Morphological structure

Morphological changes in the flax fabrics treated with the anionic surfactant Texazym (A) combined individually with each of the four enzyme preparations, Scourzym L, Texazym SCW, Texazym SER and Texazym DLG (bioscouring followed by emulsification post-treatment) have been studied in comparison with the grey flax fabric using scanning electron microscopy. Fig. 4 shows the surface morphology of the untreated flax fabric. The micrograph shows large amounts of impurities and flaking compounds on the fiber surface. These are the non-cellulosic cementing materials binding the fiber bundles together, resulting in good tensile properties but on the other hand, the presence of large amounts of these impurities makes the fiber surface highly hydrophopic. Enzymatic treatment with Scourzym L–Texazym (A) combination (Fig. 5) removes nearly all the non-cellulosic compounds from the fiber surface and produces a typical smooth, clean and highly hydrophilic surface (see Fig. 1). No fiber damage was noticed in Fig. 5 and that is why the flax fabric treated with Scourzym L–Texazym (A) combination showed little loss in tensile strength (see Table 1). Figs. 6 and 7 represent flax fabrics treated with Texazym SCW–Texazym and Texazym SER–Texazym (A) combinations, respectively. The micrographs (Figs. 6 and 7) showed that the two combinations were not highly efficient in removing all non-cellulosic materials from the fiber surface and in spite of that, the recorded losses in tensile properties of flax fabrics treated with these two combinations were higher than the loss resulting from treating flax fabric with Scourzym

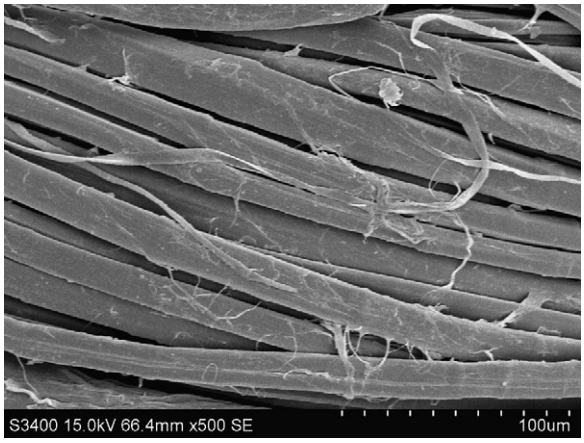

Fig. 5. SEM micrograph of flax fabric treated with Scourzym L–Texazym (A) combination.

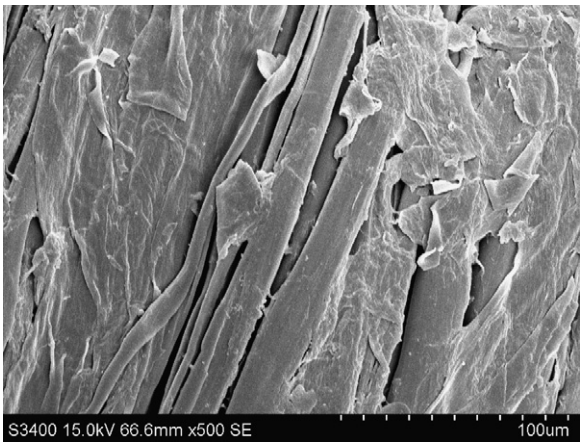

Fig. 4. SEM micrograph of grey flax fabric.

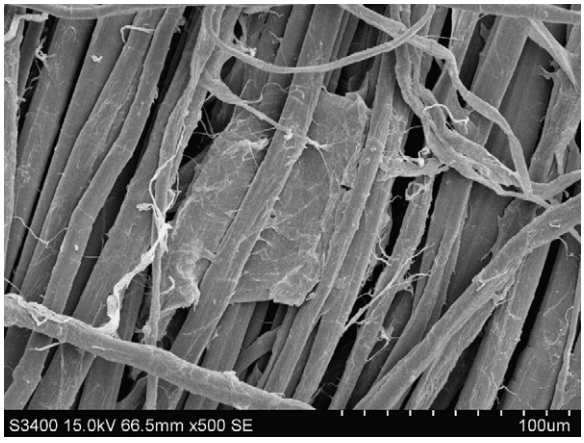

Fig. 6. SEM micrograph of flax fabric treated with Texazym SCW–Texazym (A) combination.

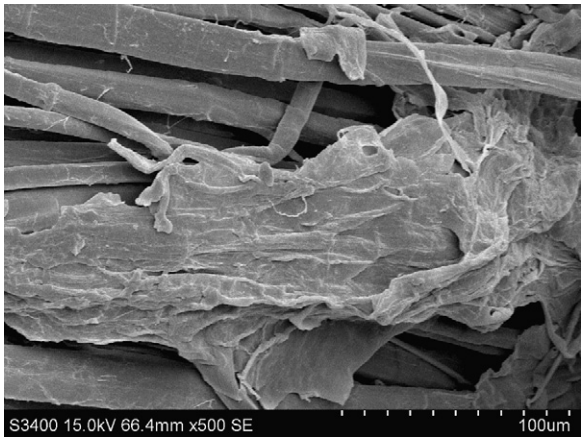

Fig. 7. SEM micrograph of flax fabric treated with Texazym SER–Texazym (A) combination.

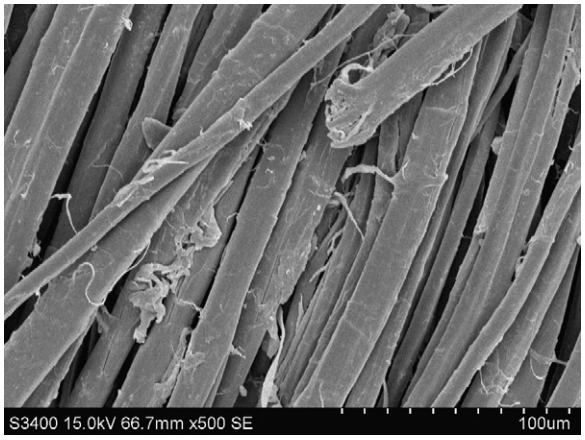

Fig. 8. SEM micrograph of flax fabric treated with Texazym DLG–Texazym (A) combination.

L–Texazym (A) combination (see Table 1). This can be explained in terms of partial cellulose degradation in case of treating flax fabrics with these two combinations. Fig. 8 represents flax fabric treated with Texazym DLG–Texazym (A) combination. The micrograph (Fig. 8) showed smooth and clean surface but the recorded loss in tensile strength reached about 41%. This extreme loss in tensile strength on treating flax fabric with Texazym DLG–Texazym (A) combination could be due to breaking up the outer layer of the fiber, allowing the enzyme preparation to reach and attack the cellulose molecules in the primary wall and accordingly producing significant loss in the fabric tensile strength.

4. Conclusion

In this study flax fabrics were treated enzymatically using sixteen combinations between four enzyme preparations and four different surfactants having varying ionic nature. The enzymatic scouring step was followed by emulsification post-treatment, through which the temperature of the bioscouring liquor was raised to 90 °C, where waxy components of flax fabrics are molten and emulsified by the action of the present surfactants. Flax fabrics subjected to such combined treatments were evaluated by measuring their physico-chemical properties, morphological structure and metrological parameters and comparing them with the corresponding properties recorded for the grey flax fabric. The measurements showed that treating flax fabrics with the combination Scourzym L–Texazym (A) resulted in flax fabric having wettability of 0.53 s and hygroscopicity of 9.15% whereas the same properties measured for grey flax sample were 20 s and 7.2%, respectively. The absorbency and hygroscopicity of the treated flax fabrics were found to be dependent on both the efficiency of the enzyme preparation and the ionic nature of the used surfactant. Best fabric hydrophilicity was attained on using the treatment combination Scourzym L–Texazym (A). Also flax fabric treated with this combination was found to show a little loss in tensile properties compared with flax fabrics treated with other enzyme–surfactant combinations. Scanning electron microscopy showed that enzymatic treatment with Scourzym L–Texazym (A) combination produces a typical smooth and clean surface without fiber damage.

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