THE ECO-MODIFICATION OF TEXTILES USING ENZYMATIC PRETREATMENT AND NEW ORGANIC UV ABSORBERS

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Abstract:

Textile fabrics were subjected to bio-pretreatment using high-activity laccase from Cerrena unicolor for comparison to standard alkaline scouring and organic absorber of ultraviolet (UV) radiation based on 1,3,5-triazine derivatives. The basic aim of the study was the development of textiles made of natural cellulose fibers (mainly flax or its blends with cotton) to provide barrier properties against UV radiation. Controlled application of enzymatic pretreatment of woven fabrics made of natural cellulose fibers allows for an efficient removal of impurities from these fibers, resulting in the improvement of sorptive properties and good penetration of dyeing agents, UV organic absorbers and other chemical modifiers, into the textile structure. In this way, products with UV-protection properties (ultraviolet protection factor >40) are obtained. These innovative barrier materials can be used for outdoor textiles that protect professional people from harmful effects of UV radiation.

Keywords:

Laccase, linen fabric, barrier properties, organic absorbers UV; enzymatic pretreatment

1. Introduction

An important problem in the refinement and modification processes of textiles is their proper pretreatment before further technological processes.

The research carried out in previous years shows that the use of chemical pretreatment (alkaline treatment) or surface modification of textile products with low-temperature nonequilibrium plasma contributes significantly to improving their sorptive and adhesive properties. However, as it is known, the chemical modification of the surface of a textile product is not ecological (a large amount of alkaline wastewater with a high chemical oxygen demand [COD] value). On the other hand, physical modification of a fiber surface as a waste-free process is ecological, but often impossible to implement in industrial conditions.

Biotechnology, along with nanotechnology, is currently one of the most progressing science disciplines. Application of enzymes in the finishing process of textiles made of cellulose fibers is the subject of investigations [1-5].

Besides cellulose, natural cellulose fibers include noncellulosic substances such as waxes, fats, pectins, proteins, hemicelluloses, lignins, and minerals. The purpose of the pretreatment of textiles is to improve their wettability by the removal of noncellulosic components. Traditionally, purification of natural fibers is carried out in the textile industry when treating products in concentrated solutions of sodium hydroxide at an elevated temperature (alkaline scouring). Partial hydrolysis of waxes, fats, and pectins and partial extraction of hemicelluloses and proteins take place. Adverse effects of alkaline treatment include partial degradation of cellulose fibers and formation of highly alkaline wastewater. Flax fibers, apart from the abovementioned noncellulosic components, contain lignin – its presence due to incrustation on amorphous part of cellulose affects fiber rigidity [6, 7]. In raw flax fiber, lignin occurs in the primary cell wall and in the outer layer of the secondary wall and is an undesirable substance. Noncellulosic components render to raw flax fibers gray color, rigidity, and poor wettability; hence, they should be removed during a controlled pretreatment process. Fiber chemical composition can hold different quantities of chemical substances depending on plant type and conditions of cultivation and climate.

Based on other literature data [6], the chemical composition of cotton and flax fibers is as follows (Table 1).

In recent years, research has been developed in which the enzymatic treatment of natural fibers (purification of natural fibers) is becoming increasingly important in the preparation of textiles for further finishing processes. This method is environmentally friendly and energy saving [3]. The use of enzymatic treatment in the textile industry is primarily related to the improvement of the appearance and quality of products by removing short fibers from their surface or giving the fabric a fashionable look by bio-polishing, bio-stoning, stone washing, and removing from it before dyeing the residual hydrogen peroxide that remained after bleaching [5, 8].
### 2. Experimental

#### 2.1. Materials

**2.1.1. Textile fabrics**

The textile fabrics consisted of the following:

- **raw linen 60% and cotton 40% woven fabric (plain weave), mass per unit area 297 g/m$^2$**
- **raw linen woven fabric 100% (plain weave), mass per unit area 260 g/m$^2$.**

**2.1.2. Modifiers – organic UV absorbers**

Two organic UV absorbers based on 1,3,5-triazine derivatives – developed within the previous project are the subject of patents EP 2565187; Patent 225 494 [16, 17].

- **UV absorber – direct (symbol A 22),** which like direct dyes has an affinity to fibers due to its linear structure (Figure 1).
- **UV absorber – reactive (symbol A8G),** which due to adequate chemical groups is able to react with cellulose hydroxyl groups (Figure 1).

Figure 1 shows the chemical structure of these absorbers.

![Figure 1. The structure of organic UV absorbers A8G and A22.](http://www.autexrj.com/)

#### 2.1.3. Enzyme

The white-rot fungus *C. unicolor* C-139 (culture collection of the Department of Biochemistry, Maria Curie-Skłodowska University, Lublin, Poland) was used in the tests. This strain particularly noteworthy in this respect are multi-enzymatic complexes containing cellulases, pectinas, as well as oxyreductases and hydrolases. Numerous published scientific papers prove the importance of the involvement of enzymatic processes in textile finishing technologies [8]. Vigneswaran and Jayapriya analyzed in their studies the effects of cellulase, xylanase, and pectinase on the physical properties of bast fibers – jute. The results obtained have shown that the use of enzymatic treatment has improved the degree of jute whiteness and significantly reduced the stiffness of the fibers [9].

The wide range of enzymes applied in the textile industry includes environmentally friendly laccase. Laccases (EC 1.10.3.2, $p$-diphenol oxidases) are the enzymes belonging to the oxidoreductase group, containing multiple copper atoms in their structure [10]. The ability of laccase to directly oxidize phenols and other aromatic compounds means that it is considered a very attractive enzyme for industrial and environmental purposes [11, 12]. In recent years, there is also a growing interest in laccase in the textile industry [13]. The range of potential applications involves not only the decolorization of textile wastewater but also bleaching processes, modification of fiber surfaces, and synthesis of new dyes [14].

Laccases are produced by fungi, higher plants, some bacteria, and even some insects. However, the largest amounts of the enzyme are secreted by wood-decaying *Basidiomycetes* (white-rot fungi), which include *Cerrena unicolor*. However, from a practical point of view, the crude enzyme used was not suitable for industrial applications. In order to improve the efficiency of the process, further treatment of the enzyme is needed to increase its durability and stability. The practical use of laccase from *C. unicolor* in the textile industry was the modification of fabrics made from flax fibers (bio-scouring). The applied enzymatic method caused efficient removal of lignin from flax fibers, provided a high level of water sorption capabilities in linen fabrics and enabled penetration of oxidizing whitening agents into the fiber structure [15]. Thus, laccase plays a significant role in the pretreatment of woven fabrics made of bast fibers.

The basic aim of the study was the development of textiles made of natural cellulose fibers (mainly flax or its blends with cotton) to provide barrier properties against ultraviolet (UV) radiation. Textile fabrics were subjected to bio-pretreatment using laccase from *C. unicolor* with a high activity for comparison to standard alkaline scouring. Thereafter, the pretreated textiles fabrics were modified with organic absorber of UV radiation based on 1,3,5-triazine derivatives.

| Cellulosic fibers | Cellulose (%) | Hemi-cellulose (%) | Extraction(%) | Lignin (%) | Water soluble (%) |
|-------------------|---------------|--------------------|--------------|------------|------------------|
| Cotton            | 82.7          | 5.7                | 6.3          | –          | 1.0              |
| Flax              | 64.1          | 16.7               | 1.5–3.3      | 2.0        | 3.9              |

**Table 1. The chemical composition of cotton and flax fibers**

http://www.autexrj.com/
is characterized by high laccase production yield without the use of additional inducers. The mycelium was maintained on 2% malt extract agar (MEA) at 4°C; after inoculation, the plates were incubated for 14 days at 28°C.

Biosynthesis of laccase was carried out in a computer-controlled Biostat ED bioreactor (Sartorius, Germany) with the total working volume of 15 l. The Lindeberg–Holm medium was prepared according to Janusz [18] with glucose and asparagine as the main source of carbon and nitrogen. The culture was performed at a constant temperature of 28°C for 10 days. During the process, the medium was aerated at 0.12 Nm³/h and stirred using a paddle stirrer at 200 rpm. The bioreactor was equipped with a set of sensors to control pH and oxygen electrodes, pressure and temperature indicators, and a special rotary filter for continuous, sterile separation of biomass and fermentation broth containing laccase [19]. Cultivations were carried out in a repeated fed-batch mode with a periodic addition of the substrate and simultaneous collection of the culture media with the enzyme. The culture broth with a crude enzyme was separated from the residual mycelium by filtration. Subsequently, the obtained filtrate with laccase was concentrated and/or purified by ultrafiltration using a Vivaflow 50 PES membrane (Sartorius) with 30 kDa cutoff. The process was carried out until the volume of the retentate with the highly active enzyme was reduced tenfold.

The methods of up- and downstream processing of laccase (enzymatic complex) from C. unicolor were used to achieve highly concentrated product with a high laccase activity of 45 U/ml.

2.2.2. Traditional alkaline or enzymatic pretreatment

Traditional alkali-scouring pretreatment of cellulosic woven fabrics was performed using a Ugolini laboratory dyeing apparatus at the ratio of 10:1 in a bath containing sodium hydroxide (2 g/l), sodium carbonate (4 g/l), and sequestering and wetting agent (1.0 g/l). The process was carried out at 98°C for 60 min followed by rinsing at 80, 60, and 40°C for 10 min.

Linen and linen/cotton woven fabrics were subjected to pretreatment in the Ugolini laboratory dyeing apparatus using different amounts of enzymatic complex with a high laccase activity produced by C. unicolor. The enzymatic pretreatment of woven fabrics was performed in baths of varying laccase concentrations of 2.5 and 5.0 U/g of the fabric in the optimal treatment conditions: pH 5.3 (phosphate buffer), temperature 60°C, time 60 min, and 10:1 liquid ratio. The enzymes were inactivated in the water bath at 80°C for 5 min.

2.2.3. Methods of evaluation of woven fabrics after pretreatment

Water sorption capability of tested sample of linen woven fabric was examined according to the method developed at the Łukasiewicz – Textile Research Institute, Lodz, determining liquid sorption using the SORP-3 instrument. Details are given in [15].

The analysis of surface microstructure of the fabrics after pretreatment – alkaline scouring or enzymatic complex – was performed using an EVO 40 scanning electron microscope (SEM; Zeiss, Germany). Investigations were performed in the Poznan University of Technology, Faculty of Chemical Technology, Institute of Chemical Technology and Engineering.

Selected samples of linen and linen/cotton woven fabrics after alkaline scouring or enzymatic complex pretreatment were dyed with a reactive dye at a concentration of 0.5 wt.%. The dyeing bath contained an organic UV absorber – 0.5 wt.% and the following auxiliaries: sodium chloride (2 x 20 g/l) and sodium carbonate (20 g/l).

2.2.1. Enzyme assays

Laccase activity was determined in the culture liquid measuring the oxidation of 300 μM 2,2’-azino-bis(3-ethylthiazoline-6-sulfonate) (ABTS) buffered with 50 mM citrate phosphate (pH 4.5, ε 420=36 1/(mMxcm) [19]. All spectrophotometric measurements were carried out using a UV/VIS T80+ spectrophotometer (PG Instruments Ltd.) at the wavelength λ_max of 420 nm. Enzyme activities were expressed in units (U) defined as 1 μmol of product formed per minute.

Figure 2. Diagram of the dyeing process of linen or linen/cotton woven fabrics with reactive dye and organic UV absorbers

2.1.5. Chemical agents

The chemical agents used were as follows:

- sodium chloride – NaCl and
- sodium carbonate – Na₂CO₃

2.2. Methods

2.2.1. Dyes

The reactive dye used was Synozol Blue KBR (CI Reactive Blue 221, CAS No. 93051-41-3; Kyung-In Synthetic Corp.).

The dye used in the aqueous solution was characterized by a maximum wavelength λ_max of 604 nm, at which maximum absorption was measured.
The dyeing temperature was 60°C, and the whole process took 100 min. The dyeing process, carried out in the Ugolini laboratory dyeing apparatus at a 1:10 bath ratio, proceeded according to the diagram shown in Figure 2. After dyeing, the samples were rinsed with water at 60°C for 10 min and then with cold running water.

The measurement of absorbance was performed using a Jasco UV–VIS spectrophotometer V600 (Jasco, Japan). The degree of dye exhaustion \( E \) in the bath was calculated from formula (1) after prior analysis of the calibration curves for each dye.

\[
E = \frac{A_0 - A_1}{A_0} \times 100\% \tag{1}
\]

where \( A_0 \) is the parameter that characterizes the amount of dye in the bath before dyeing (m\(^{-1}\)) and \( A_1 \) the parameter that characterizes the amount of dye in the bath after dyeing (m\(^{-1}\)).

The concentration of dye on the fiber during and after the dyeing process was assessed indirectly by determination of the \( K/S \) coefficient using the Kubelka–Munk formula (2), proportional to the dye concentration on the fiber.

\[
\frac{K}{S} = \frac{(1 - R)^2}{2R} = k \cdot c_w \tag{2}
\]

where \( K \) is the light absorption coefficient, \( S \) the light-scattering coefficient, \( R \) the light remission rate, \( c_w \) the dye concentration on the fiber, and \( k \) the coefficient of proportionality.

The dyed fabric samples were dried, and the coefficient of remission (\( R \)) was measured. Measurement of the remission coefficient with calculations of the value of relative dye intensity \( (K/S) \) was performed on a Spectraflash 850 spectrophotometer (Datacolor International, USA) in accordance with PN-EN ISO 105 J01: 2002.

2.2.5. Evaluation of COD

The parameter that characterized wastewater in terms of environmental nuisance, COD (mg O\(_2\)/dm\(^3\)), was assessed by the dichromate method according to Polish Standard PN-74/C-04578, corresponding to German Standards DIN EN 1899-1 and DIN 38409-H 41 [20].

3. Results and discussion

3.1. Enzymatic activity

The obtained maximum activity of 2.5 U/ml was higher than that during conventional batch fermentation [21]. Additionally, the use of specially constructed spin filter inside the bioreactor made it possible to collect culture liquid without biomass. As a result, the further purification process was shortened by the step of biomass filtration.

Thanks to the application of a membrane with the cutoff of 30 kDa, a more specific enzymatic product containing highly active laccase was obtained.

The laccase from \( C. \) unicolor was the most active at pH of the bath equal to 5.3; therefore, the phosphate buffer was used. Moreover, COD of the enzymatic bath was very low (10 mg×O\(_2\)/dm\(^3\)) compared to the OD of the enzymatic bath with citrate buffer applied (2250 mg×O\(_2\)/dm\(^3\)). The detailed analysis of wastewater obtained after modification of the fabrics will be discussed later (Table 4).

3.2. Liquid sorption capability of tested sample of linen woven fabric

Water sorption by fibers was examined according to the method developed at the Textile Research Institute, Lodz, by the SORP-3 instrument. Upon analysis of kinetic curves of \( H_2O \) sorption (Figure 3), it has been stated that woven fabrics made of flax fibers after enzymatic pretreatment are characterized with almost the same sorption values when compared to woven fabrics after alkaline boiling off.

The time of water absorption after alkaline treatment is shorter than the time of laccase treatment.

3.3. Analysis of microstructure using SEM

In the first stage, the researchers focused on developing an ecological method of textile fabric preparation by pretreatment

![Figure 3. Kinetic curves of H\(_2\)O sorption on linen woven fabric after alkali or laccase enzyme treatment.](image-url)
with an enzymatic complex in order to efficiently and permanently incorporate organic UV absorbers. To compare the effect of pretreatment with alkaline scouring or enzymatic complex, the evaluation of the surface microstructure of the tested woven fabrics with the linen content was performed using SEM.

The examination of images (Figure 4) showed that the alkaline treatment of the linen woven fabric resulted in polishing/smoothing of single fibers, which, in turn, caused their better compacting. Morphological analysis showed that a single fiber diameter of 10 µm was the same as in the case of fiber in raw linen woven fabric.

The treatment of linen woven fabrics with the laccase from *C. unicolor* revealed in the morphology of individual fibers a violation of their structure, which may indicate the removal of noncellulosic constituents from the lignin–cellulose fiber matrix of flax. However, it is difficult to clearly determine the level of their removal (Figure 4).

On the other hand, the use of enzymatic complex for pretreatment of linen/cotton fabric caused relatively larger changes in its structure. Images of single fibers showed significant changes on their surface, which probably can be the effect of the enzymic activity. The observed changes are proportional to the amount of enzyme used. Microscopic images also showed “sticking” of individual fibers. These observations coincide with those of Patra and Madhu [7], who have shown that the enzymic activity is less aggressive and does not cause cellulose degradation, and results of the treatment applied are comparable to the results of conventional alkaline etching (Figure 5).

### 3.4. The dyeing properties of linen woven fabrics after modifications

Activity of the enzymatic complex was also evaluated on the basis of mass loss of the treated fabrics, as well as a comparative assessment of sorptive properties of linen and cotton fibers presented in the studies on dyeability of these products.

The mass loss of the samples of linen fabric after treatment with the enzyme was about 0.4–0.7% and after alkaline treatment was 2–2.5%, while for the samples of fabric from a blend of linen and cotton fibers after enzymatic treatment, it was 0.8–1.2% and after alkaline treatment, it was 2.1–2.5%.

The degree of dye exhaustion from the bath and the obtained UV-protection properties as a result of the modification—incorporation of the organic UV absorber – A8 or A22 – into the structure of the textile product by the method of exhaustion from the dyeing bath were analyzed.

The wavelength was determined for maximum absorbance of the Synozol Blue KBR reactive dye in visible light, and a standard curve was drawn for it (Figures 6 and 7, respectively).

The effect of high activity of laccase from *C. unicolor* is the enhancement of dye bath penetration into the fiber that apart from the dyestuff contains the organic UV absorber (A8 or A22). Figures 8 and 10 present the dependence of exhaustion rate (*E*, %) of reactive dye from a bath on the dyeing time of linen/

![Figure 4. Scanning electron micrographs: (a) raw linen woven, (b) linen woven after alkaline treatment, (c) linen woven after enzymatic treatment of 2.5 U/g fabric, and (d) linen woven after enzymatic treatment of 5.0 U/g fabric.](http://www.autexrj.com/)
A comparative analysis of the results confirms that sorptive properties of the products containing flax fibers after the pretreatment with the enzyme are comparable to those obtained after traditional boiling in strongly alkaline solutions of caustic soda. On the other hand, analysis of the relative color intensity of fabrics showed higher $K/S$ values for both linen/cotton woven fabric and linen woven fabric after enzymatic scouring.

3.5. Evaluation of barrier properties after application of organic UV absorbers

The effective application of organic UV absorbers to cellulose fibers during the dyeing process using the bath exhaustion method is confirmed by the values of UPF parameter obtained and the transmittance of modified fabrics, which are summarized in Tables 2 and 3.
3.6. COD load of wastewater after modification

Analysis of COD for wastewater after a two-step modification of fabrics from linen and cotton fibers (results presented in Table 4) showed that after pretreatment with the enzyme of laccase, dyeing wastewater had a much lower value of COD than in the case of alkaline treatment.

The performed tests have clearly shown that it is possible to use the with high laccase activity for pretreatment, i.e.,...
Table 2. Values of UPF and transmittance for linen/cotton woven fabric subjected to various variants of pretreatment and application of organic UV absorber

| Amount of absorber 0.5% (linen/cotton fabric) | UPF | Transmittance |
|---------------------------------------------|-----|---------------|
| Raw linen/cotton                            | 27  | UVA = 2.43, UVB = 1.75, %T average = 2.09 |
| Laccasa 2.5 U/gtk + A22 0.5% (2 steps)       | >50 | UVA = 0.69, UVB = 0.17, %T average = 0.57 |
| Laccasa 2.5 U/gtk + A8G 0.5% (2 steps)       | >50 | UVA = 0.84, UVB = 0.25, %T average = 0.71 |
| Alkaline treatment + A8G 0.5% (2 steps)       | >50 | UVA = 0.50, UVB = 0.12, %T average = 0.41 |
| Alkaline treatment + A22 0.5% (2 steps)       | >50 | UVA = 1.33, UVB = 0.39, %T average = 1.12 |

UPF, ultraviolet protection factor; UV, ultraviolet.

Table 3. Values of UPF for linen fabric after enzymatic or alkali pretreatment and modification with organic UV absorbers in the amount of 0.5%

| Amount of absorber 0.5% (linen fabric) | UPF | Transmittance |
|---------------------------------------|-----|---------------|
| Raw linen                             | 28.03 | UVA = 4.56, UVB = 4.02, %T average = 4.29 |
| Laccasa 2.5 U/gtk + A8G 0.5% (2 steps) | >50 | UVA = 2.78, UVB = 1.95, %T average = 2.67 |
| Laccasa 2.5 U/gtk + A22 0.5% (2 steps) | >50 | UVA = 2.63, UVB = 1.12, %T average = 2.40 |
| Alkaline treatment + A8G 0.5% (2 steps) | >50 | UVA = 2.43, UVB = 1.10, %T average = 1.99 |
| Alkaline treatment + A22 0.5% (2 steps) | >50 | UVA = 2.65, UVB = 2.15, %T average = 2.41 |

UPF, ultraviolet protection factor; UV, ultraviolet.

Table 4. Final COD of wastewater after the modification of linen/cotton woven fabrics (two steps)

| COD (mg·O₂/dm³) |
|----------------|
| Citrate buffer pH = 5.3 | 2250 |
| Phosphate buffer pH = 5.3 | 10 |
| Phosphate buffer + laccase 5.0 U/gtk | 182 |
| Water bath + A8G + dye (2 steps) | 764 |
| Water bath + A22 + dye (2 steps) | 742 |
| Scouring laccase + A8G + dye (2 steps) | 762 |
| Scouring laccase + A22 + dye (2 steps) | 744 |
| Alkaline scouring + A8G + dye (2 steps) | 1420 |
| Alkaline scouring + A22 + dye (2 steps) | 1380 |

COD, chemical oxygen demand.
cleaning the linen and linen/cotton fabrics to improve their sorptive properties, and thus to effectively apply organic UV absorbers by the bath exhaustion method. This process is environmentally friendly and energy saving.

4. Conclusions

The following conclusions have been obtained:

- The applied methods of up- and downstream processing of laccase (enzymatic complex) from *C. unicolor* enabled to achieve highly concentrated product with a laccase activity of 45 U/ml.

- Controlled application of enzymatic pretreatment of woven fabrics made of natural cellulose fibers allows for an efficient removal of impurities from these fibers, resulting in the improvement of sorptive properties and good penetration of dyeing agents, UV organic absorbers.

- The modified materials from natural cellulose fibers containing flax fibers show very good barrier properties against UV radiation (UPF >50, transmittance in the whole UVA and UVB range <3%).

- Wastewater after two-step modification when using the enzymatic complex in the first step of pretreatment has a significantly lower COD value than that in the case of alkaline treatment.

- The use of enzymatic treatment is more energy efficient compared to the alkaline treatment, due to the significantly lower temperature of the enzymatic (60°C) process than the chemical (98°C) process.

- It is possible to use the initial pro-ecological bioprocessing of products from natural cellulose fibers before applying a new generation of organic UV absorbers. In this way, products with UV-protection properties can be obtained. These innovative barrier materials can be used for clothing parts textiles and technical that protect people exposed to harmful effects of UV radiation.

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