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STUDY ON THE INFLUENCE OF ULTRASOUND IN BIOSCOURING TREATMENT OF 50 % OF FLAX + 50 % OF COTTON FABRICS

DOCHIA Mihaela[1], PUSTIANU Monica[1, 2], GAVRILAŞ Simona[1, 3], TOMESCU Daniel[1], COPOLOVICI Dana Maria[3]

[1]"Aurel Vlaicu" University of Arad, Research Development Innovation in Technical and Natural Science Institute, Postal address, 310330, 2-4 Elena Dragoi Street, Arad, Romania, E-Mail: dochiamihaela@yahoo.com, tomescudaniel86@yahoo.com@yahoo.com

[2]"Aurel Vlaicu" University of Arad, Faculty of Engineering, Department of Automation, Industrial, Textile and Transportation Engineering, Postal address, 310330, 2-4 Elena Dragoi Street, Arad, Romania, E-Mail: pustianumonica@yahoo.com

[3]"Aurel Vlaicu" University of Arad, Faculty of Food Engineering, Department of Technical and Natural Sciences, Postal address, 310330, 2-4 Elena Dragoi Street, Arad, Romania, E-Mail: simona2213@yahoo.com, dana.copolovici@uav.ro

Corresponding author: Dochia, Mihaela, E-mail: dochiamihaela@yahoo.com

Abstract: *Study on the influence of ultrasound in Bioscouring treatment of 50 % of flax + 50 % of cotton fabric was made. The role of the Bioscouring treatment was the removing of natural cellulose attendants such as: pectin, hemicelluloses, waxes, extractable substances, etc.*
The cleaning treatment was carried out with a commercial enzymatic product called Beisol PRO (which consists of a mixture of enzymes pectinases), in water at a 20:1 liquid to fabric ratio and a temperature of 55 ^{0}C, in the precence of a complexing agent (2 g/L EDTA) and a washing agent (0.5 % Denimcol Wash RGN).
The effect of the enzyme mixture was intensified by ultrasound at a frequency of 45 kHz in an ultrasonic bath Elmasonic X-tra basic 2500 from Elma Company, Germany, leading to the improvement of the process and better properties for treated material.
The experiments were conducted after a central, rotatable second order compound program with two independent variables: enzyme concentration (concentrations between 1-3% o.w.f) and treatment time (15-55 minutes).
Treated samples were analyzed for weight loss, hydrophilicity, whiteness index, yellowness index, crystallinity, tensile strength, elongation at break, scanning electron microscopy analysis (SEM), CIELAB color system analysis of the samples dyed with alizarin and ruthenium red dyes.
The results showed that the bioscouring process performed in the presence of ultrasound has been effective at a lower enzyme concentration and a lower duration of the treatment, thereby reducing the costs and the possibility of degradation of the treated material.

Key words: *cotton/flax fabric, bioscouring treatment with ultrasound, weight loss, whiteness index, K/S color strength, crystallinity*

1. INTRODUCTION

Ultrasounds are elastic waves with frequencies between 16-106 kHz which can be used in the form of longitudinal waves to improve the mass and heat transfer. This intensification increases

with the increasing of frequency and power transmitter, having the effect of reducing treatment duration and the temperature below the usual ones. Also, by using ultrasound, the following effects are obtained: changing of layer and substrate limit of the interface material-solution with the increasing of rate diffusion; degassing of the solutions and materials occurs and a temporary fiber deformation, which ensures a deep penetration of chemical agents in the treated textile materials [1].

Flax and cotton fibers contain besides cellulose, other morphological impurities like: hemicelluloses, pectins, extractable substances, waxes, etc., which have to be removed to get a good wettability and a good whiteness degree of the fabrics. These impurities can be removed by classical scouring treatment where NaOH is used or enzymatic scouring process where specific enzymes like pectinases are involved. In addition, for both type of treatment, a complexing agent and a washing product are utilized. EDTA (Ethylenediaminetetraacetic acid) is the complexing agent most used because complexes with the majority of the metal ions and has a good stability in an alkaline medium. The washing product is used to emulsify the unsaponifiable materials. Nonionic surfactants are preferred because of their high emulsification capacity [2].

These treatments must be conducted in order to increase the percentage of the crystalline zone with the reducing of amorphous one, so that the treated material to have appropriate mechanical properties [3].

During chemical or biochemical treatments of textiles, various cellulose degradations may occur by conversion of alcohol groups into carbonyl or carboxyl groups by oxidation or hydrolysis reactions. These degradations can be expressed by: microscopic, chemical and viscosimetric methods, etc. [4].

The efficiency of the bioscouring process can be determined by the following analyses: weight loss, hydrophilicity, whiteness index, yellowness index, crystallinity, tensile strength and elongation at break, scanning electron microscopy analysis (SEM), CIELAB color system analysis of the samples dyed with alizarin and ruthenium red dyes.

2. EXPERIMANTAL PART

For conventional alkaline scouring and enzymatic treatments, samples of 50 % of flax + 50 % of cotton material with the following characteristics were used: 120 ± 3 cm width, 220 ± 10 g/m^2, for warp direction 100 % of cotton yarn with Nm 14 and 50 % of flax + 50 % of cotton yarn with Nm 14 for weft direction.

Prior to the alkaline and bioscouring treatments, the samples were washed at 100 °C using a AATCC standardized Lander-Ömeter, model M228-AA from SDL Atlas Company - USA, followed by: drying, conditioning and weighing [5].

All enzymatic experiments were carried out with a commercial enzymatic product called Beisol PRO (which consists of a mixture of enzymes pectinases), in water at a 20:1 liquid to fabric ratio and a temperature of 55 ^{0}C, in the precence of 2 g/L EDTA as a complexing agent and 0.5 % Denimcol Wash RGN as a surfactant. A central, rotatable second order compound program with two independent variables: enzyme concentration (concentrations between 1-3 % o.w.f) and treatment time (15-55 minutes) was used. The effect of the enzyme mixture was intensified by ultrasound at a frequency of 45 kHz in an ultrasonic bath Elmasonic X-tra basic 2500 from Elma Company, Germany. After bioscouring treatment, the samples were washed with hot water at 70°C, cold water and dried at room temperature [6]. For comparison a classic alkaline treatment was done with 10 g/L sodium hydroxide, 5 g/L sodium carbonate, 1 g/L sodium bisulfite, 2 g/L sodium silicate and 2 g/L Sulfolen 148 (S-148, alkyl polyglicol ether) as a wetting agent.

After drying and conditioning of the treated samples till constant weight, the following analyses were done: weight loss, hydrophilicity, crystallinity, tensile strength and elongation at

break, whiteness index, yellowness index, CIELAB color system analysis of the samples dyed with alizarin and ruthenium red dyes and scanning electron microscopy analysis (SEM).

The weight loss was determined by gravimetric method. The samples were dried at 105 °C in an oven from Caloris Group, Romania till constant weight. The weight loss was calculated using the following equation:

$$\% \text{ weight loss} = (W1-W2)\ x100/W1 \quad (1)$$

where, W1 and W2 are the weights of dried samples fabric before and after the treatments.

The hydrophilicity of the treated samples was determined according to AATTCC Test Method 79-2007.

Because during the treatment, fibers degradation may appear by oxidation or hydrolysis reactions, XRD analysis and tensile strength of the material were made.

The cellulose crystallinity procent of the samples was determined by X-ray diffraction with a MiniFlex 600 diffractometer (Rigaku Corporation, Japan), operating at 40 kV, 15 mA, with CuKα monochromatic radiation, and using SC-70 detector and the automatic 6 position sample changer, ASC-6. The scan range was 5°-45°, with step width 0.05°, scan speed 1.2° / min. at room temperature and humidity. The PDXL2 Version 2.4.2.0 containing powder diffraction analysis package, PDXL Comprehensive Analysis, was used to analyze and calculate the cristallinity procent based on the diffraction intensity of the crystalline and amorphous phases.

The tensile strength and elongation at break was determined by using a testing machine 5KT from Tinius Olsen – United States with an interface running on a connected PC by Horizon software. The ASTM D 5035 – 06 "*Standard Test Method for Breaking Force and Elongation of Textile Fabrics (Strip Method)*" was used [7].

Whiteness and yellowness degree were determined on a Datacolor 500 spectrophotometer. For an opaque view each sample was folded twice to give four plies and the whiteness was measured four times at different points. The Hunter Whiteness Index and DIN 6170 Yellowness Index were calculated automatically by Datacolor Tools 2.0 software.

The degree of pectins removal was determined by ruthenium red dyeing method. The reflectance (R %) at 540 nm was measured on Datacolor 500 spectrophotometer and K/S values were calculated. The dyeing method with alizarin dye was used to determine the amount of calcium removed from the treated fabric.

The surface morphologyies of the cotton/flax fabrics were explored by scanning electron microscopy (SEM). The samples were placed on a specimen support, then coated with Au using an auto fine coater (JFC-1200, JEOL Co., Japan). Observations were conducted using a SEM (LYRA 3, Tescan, Czech Republic) at 2 or 10 kV, respectively.

3. RESULTS AND DISCUSSIONS

Table 1 presents the results obtained for the samples subjected to the alkaline and bioscouring treatments for different conditions.

Table 1: *The results obtained for the samples subjected to the alkaline and bioscouring treatments for different conditions*

No.	Enzyme [% o.w.f]	Time [min]	Weight loss [%]	Hydrophilicity [s]	Crystallinity	Tensile strength [N]	Elongation at break [%]	Whiteness index	Yellowness index	k/s Ruthenium red	k/s Alizarin
1	1.30	21.00	1.26	0.68	67	965	9	57.25	16.12	1.06	0.65
2	2.70	21.00	1.02	0.79	67	960	9	57.11	16.34	1.02	0.64
3	1.30	49.00	0.34	0.85	63	949	9	56.52	16.78	1.03	0.64
4	2.70	49.00	1.31	0.74	70	1035	8	57.02	16.17	1.06	0.65
5	1.00	35.00	0.36	0.76	66	975	8	55.97	16.79	1.07	0.66
6	3.00	35.00	0.90	0.82	65	957	9	57.55	16.09	1.05	0.65
7	2.00	15.00	0.26	0.79	64	950	9	56.18	16.82	1.11	0.67
8	2.00	55.00	0.80	0.74	67	980	9	57.52	15.94	1.02	0.65
9	2.00	35.00	1.28	0.79	73	1057	8	56.58	16.48	0.91	0.62
10	2.00	35.00	1.37	0.79	68	987	9	55.85	17.02	0.91	0.61
11	2.00	35.00	1.06	0.73	71	1048	8	56.33	16.83	0.88	0.60
12	2.00	35.00	1.38	0.77	72	1053	8	56.17	16.86	0.96	0.63
13	2.00	35.00	1.26	0.73	71	1050	8	56.28	16.64	0.97	0.64
AT	Alkaline	60.00	4.71	0.41	68	962	8	58.21	16.11	0.79	0.63
M	Control	-	-	-	62	874	9	48.00	18.42	1.31	0.72

Enzymatic treated samples present a weight loss lower than alkaline treated sample because the bioscouring treatment is less aggressive. It can be noticed that in the case of the enzymatic treatments the smallest weight loss occurs in the sample 7 at 2 % (o.w.f.) enzyme concentration and a treatment time of 15 min. The higher weight losses occur especially for samples 9 to13 where enzyme concentration was 2 % (o.w.f.) and treatment time 35 minutes.

Hydrophilicity values obtained from enzymatic treated samples are similar with the hydrophilicity of alkaline treated sample, which shows a good and effective treatment without significant weight loss. For all treatments, the weight loss values are in agreement with data from the literature which mentions values between 5-10 % for alkaline treatment and below 5 % for enzymatical ones [8]. Also, a hydrophilicity lower than 1 second is cosidered very good.

The crystallinity (Cr) value was obtained from the ratio between the peak area of the crystalline plane located for 2θ in the range of 22.46° and 22.86° and the total area. X-ray diffractograms for untreated flax/cotton fabric (1), enzymatically treated sample - 2 % o.w.f. and 35 minutes (2) and alkaline treated sample (3) are presented in Fig. 1.

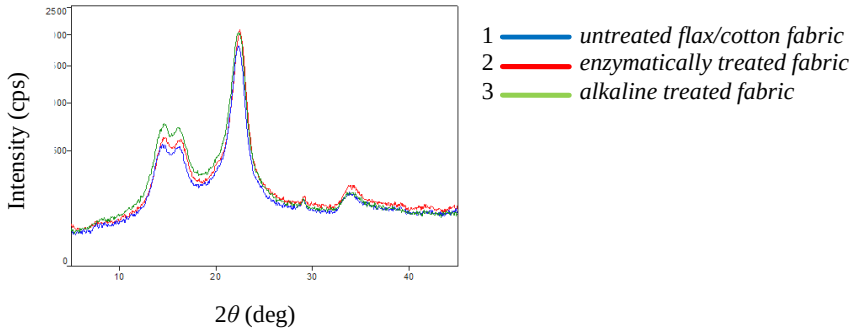


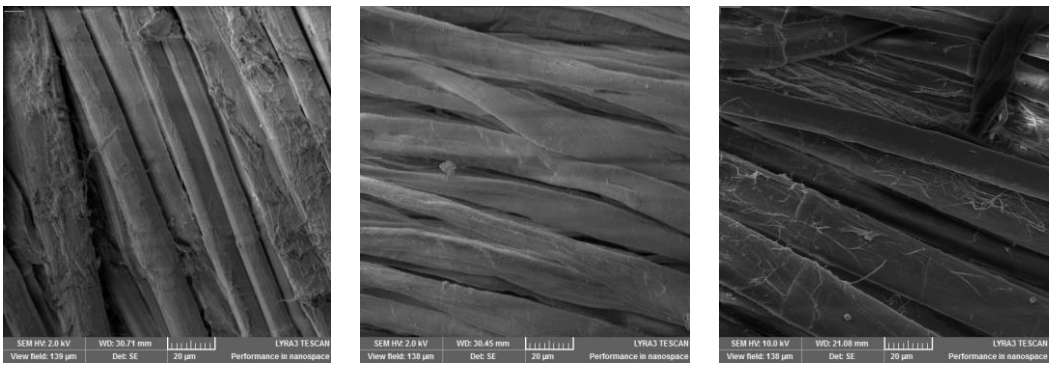
Fig. 1*: X-ray diffractograms for untreated flax/cotton fabric (1), enzymatically treated fabric- 2 % o.w.f. and 35 minutes (2) and alkaline treated fabric (3)*

As it can be seen from Table 1, for all treatments there was an increase in crystallinity compared to the control. With the increasing of crystallinity, the tensile strength of the fabric also increased. From figure 1 which presents a comparison between diffractograms obtained for the control, the best enzymatic treatment and alkaline treatment, it can be observed that enzymatic sample has the higher degree of crystallinity (73) followed by alkaline one (68) which means that the treatment was effective for removing of the non-cellulosic impurities.

A slightly whitening of the material appeared after the cleaning treatments. Small variations of Whiteness Index are oberved compared with the control. The whiteness index was increased with 17 % for the best enzymatic treatment and 21 % for classic alkaline treatment. Regarding the Yellowness Index this was decreasing by a percentage between 7 % and 13 %.

For the case of Ruthenium red dyeing method, the values obtained for color strength K/S of enzymatically treated samples compared to the control, a decrease between 15 % - 33 % is observed. For alkaline treatment a 40 % decreasing was registered. For alizarin dyeing method the K/S values present a decreasing between 7 -17 % for enzymatically treated samples and 12.5 % for alkaline treated sample.

The morphological changes of the treated samples were studied by scanning electron microscopy (SEM). Figure 2 presents the SEM micrograph of grey flax/cotton fabric (a), enzymatically treated sample at 2 % enzyme concentration and 35 minutes (b) and alkaline treated sample (c).



(a) (b) (c)

Fig. 2*: SEM micrographs for untreated fabric (a), sample treated with 2 % enzyme for 35 minutes (b), alkaline treated sample (c).*

The micrograph (a) shows some amounts of impurities and flaking compounds on the fiber surface for untreated sample. These represent the non-cellulosic materials binding the fiber bundles together and making the fiber surface highly hydrophobic. The enzymatic treatment (b) removed the impurities from the fiber surface leading to a smooth and clean surface. For the classical alkaline treatment (c) some peeling effect is observed due to the fibers degradation.

5. CONCLUSIONS

During the studies carried out it was found that:

- Enzymatic treated samples presented a weight loss lower than alkaline treated sample because the bioscouring treatment is less aggressive.
- Hydrophilicity values obtained for samples enzymatically treated are similar to that obtained for alkaline one, which showed a good and effective treatment without significant weight loss.

- It was noticed that in the case of the enzymatic treatments the smallest weight loss occurs in the sample 7 at 2 % (o.w.f.) enzyme concentration and a treatment time of 15 min. The higher weight losses occur especially for samples 9 to13 where enzyme concentration was 2 % (o.w.f.) and treatment time 35min. For all treatments, the weight loss values are in agreement with data from the literature which mentions weight loss values between 5-10 % for alkaline treatment and below 5 % for enzymatical ones. Also, all hydrophilicity values are lower than 1 second which demonstrates a very effective treatment.
- For all treatments was an increase in crystallinity compared to the control. With the increasing of crystallinity, the tensile strength of the fabric also increased.
- The whiteness index was increased with 17 % for the best enzymatic treatment and 21 % for classic alkaline treatment. Regarding the Yellowness Index this was decreasing by a percentage between 7 % and 13 %.
- For the case of Ruthenium red dyeing method, the values obtained for color strength K/S of enzymatically treated samples compared to the control, a decrease between 15 % - 33 % is observed. For alkaline treatment a 40 % decreasing was registered.
- For alizarin dyeing method the K/S values presented a decreasing between 7 -17 % for enzymatically treated samples and 12.5 % for alkaline treated sample.

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