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Mojsov Kiro¹Faculty of Technology, University of Stip, Republic of Macedonia
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Abstract: *Biotechnology in textiles is one of the revolutionary ways to promote textile field. Bio-processing were accompanied by a significant lower demand of energy, water, chemicals, time and costs. In the world are carried out investigations to replace conventional chemical textile processes by eco-friendly and economically attractive bioprocesses using enzymes. This paper will show the experimental results relating to optimization of the treatment processes with neutral cellulase enzyme to improve physical properties of handloom cotton fabric. Cellulase enzymes are known as effective in treatments of cotton fabric. For the use of neutral cellulase enzyme and evaluate in physical properties like weight loss, moisture content, strength loss, bending length and water absorption on cotton fabric was made optimization of the conditions of concentration, treatment time and temperature. From these results was chosen an optimum enzyme concentration-2.0% owf (on weight of fabric), treatment time-70 minutes and temperature-65 °C.*

Keywords: neutral cellulase, weight loss, moisture content, strength loss, bending length, water absorption.

EFEKTI ENZIMSKOG TRETMANA NA FIZIČKE OSOBINE RUČNO TKANI PAMUČNIH TKANINA

Apstrakt: *Biotehnologija u tekstila je jedna od revolucionarnih podsticanja tekstilnu polje. Bio-obrađa je praćena značajnog smanjenja potražnje energije, vode, hemikalija, vremena i troškova. U svetu se sprovode istrage da zamene konvencionalne hemijske procese tekstilnih po ekološkim i ekonomski atraktivnim bioprocima koristeći enzime. Ovaj rad će pokazati eksperimentalne rezultate koji se odnose na optimizaciji procesa tretmana sa neutralnim celulaza enzima za poboljšanje fizičke osobine ručno tkani pamučne tkanine. Celulaza enzimi su poznati kao efikasni u odrada pamuka. Za korišćenje neutralnog celulaza enzima i ocenjuju po fizičkim osobinama kao što su gubitak težine, sadržaj vlage, gubitak jačina, dužinu savijanja i upijanja vode na pamučne tkanine je napravljena optimizaciju uslovima koncentracije, vremena i temperature tretmana. Iz ovih rezultata izabrana je optimalna koncentracija enzima-2.0% ofw (o težini tkanine), vreme tretmana-70 minuta i temperatura-65 °C.*

Ključne reči: neutralna celulaza, gubitak težine, sadržaj vlage, gubitak jačina, dužinu savijanja, upijanja vode.

1. INTRODUCTION

The use of enzymes in textile processing is already established industrial technology. Enzyme treatments of textiles, typically cellulose materials such as cotton, viscose or lyocell fabrics, have widely been used in the textile industry since the 1980s. Enzymatic processing enables the textile industry to reduce production costs, to reduce the environmental impact of the overall process and to improve the quality and functionality of the final products. Enzymes are non-toxic and they allow mild conditions of temperature and pH. Today enzymatic treatment of cotton either in denim washing or in biostoning is standard technique in industry. The enzymatic treatment of textiles significantly improves some of their properties as well as increases their aesthetic values and comfort of use. Although the use of cellulases in cotton finishing has been increasing very rapidly, the cellulases used hitherto have mainly been crude mixtures causing unacceptable losses of fabric strength and weight. Furthermore, the unoptimized cellulase composition of commercial preparations and non-optimal dosage of the enzymes have lead to low reproducibility of the processes. Cellulase enzymes are highly effective in removing loose fibres from fabric surface, a process known as biopolishing.

Cotton is still the most important of all textile fibres, accounting for 36% of textile fibre production. It has many desirable fibre properties making it an important fibre for textile applications. Cotton has high moisture regain, moderate strength, and is stronger when wet. Of the synthetic fibres, polyester is by far the most widely used fibre. Today it accounts for 30% of textile fibre production and its production rate is still expected to grow [1]. Cotton fibre has a fibrillar structure and it consists of a primary wall, a secondary wall and a lumen. In addition to cellulose, cotton also contains lipids, proteins, pectins, waxes, organic acids and noncellulosic polysaccharides constituting up to 10% of the total fibre weight [2]. Handloom cotton has some shortcomings, like higher maintenance costs for washing and ironing,

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partly for rough texture and less durability [3]. Fortunately, in the present time, commercially viable alternative methods for preparing and finishing cotton fibre substrates based on the use of enzymes have emerged. Such methods will ensure the supremacy of cotton over other fibers for decades to come [4].

The chemical composition of cellulose is simple, consisting of anhydroglucose units joined by β -1,4-glycosidic bonds to form linear polymeric chains (Figure 1). The physical properties of the cotton fibre as a textile material, as well as its chemical behaviour and reactivity, are determined by arrangements of the cellulose molecules with respect to each other and to the fibre axis.

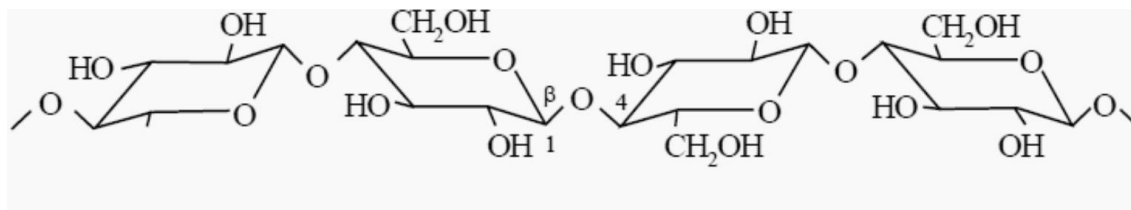


Figure 1.- Structure of cellulose

In nature cellulose is degraded by both fungi (*Aspergillus* and *Trichoderma*) and bacteria (*Bacillus*). These organisms produce cellulases that specifically degrade cellulose, yielding shorter chain cellulose polymers and glucose which are metabolised by these organisms. Typically, the fungal and some bacterial cellulolytic enzyme systems consist of several enzymes acting at the ends (exoglucanases, also called cellobiohydrolases) or in the middle (endoglucanases) of the cellulose chains [5]. *Trichoderma reesei* is one of the most important industrially used strains for cellulase production. According to current knowledge, its cellulolytic system is composed of two cellobiohydrolases and at least six endoglucanases and two β -glucosidases. β -glucosidases act on short, soluble oligosaccharides to produce primarily glucose [6].

For optimizing the cellulase enzyme activity are important process variables such as, concentration, treatment time and temperature. In this study were optimized conditions for the use of neutral cellulase enzyme and evaluate the changes in physical properties like weight loss, moisture content, strength loss, bending length and water absorption on handloom cotton fabric.

2. EXPERIMENTAL PART

2.1. Materials

- Commercial textile enzyme (Neutral Cellulase Powder Tep-15) from Dong Guan Proamine Chemicals Co. Ltd, Dongguan, China.
- Pure white handloom cotton fabric was provided from trade market.

2.2. Enzyme treatment

Enzyme treatment was carried out in Launderometer and consisted of commercial neutral enzyme.

2.3. Weight loss

The weight loss due to pretreatment was determined by weighing of fabric samples before and after treatment and was expressed in percent. These fabric samples were conditioned for 24 hours at room temperature in a desiccator and then weighed each sample separately, before and after the enzyme treatment. The weight loss (%) was calculated using the following formula:

$$\text{Weight loss (\%)} = \frac{(W_1 - W_2) \times 100}{W_1}$$

where:

- W_1 weight of the fabric before enzyme treatment,
- W_2 weight of the fabric after enzyme treatment.

2.4. Moisture content

The moisture content of cotton fabric was determined using IS 199 : 1989 test method.

IS 199 : 1989 – Textiles - Estimation of moisture, total size or finish, ash and fatty matter in grey and finished cotton textile materials [TXD 5: Chemical Methods of Test].

From the sample under test draw at least 2 test specimens each weighing approximately 3 g. The specimen was weighed accurately in a dry and cleaned petridish and it was placed in the drying oven and the specimen was dried at $105\pm 3\%$ to constant mass, and the oven-dry mass of the test specimen was determined. The mass may usually be regarded as constant if the loss between two successive weighing, taken at an interval of 20 minutes, does not exceed by 0,1 percent of the first of the two values. Similarly other test specimens were tested. The moisture content (%) in the test specimen was calculated by taking the mean of three measurements by the following formula:

$$\text{Moisture content (\%)} = \frac{(a-b)\times 100}{a}$$

where:

- a original mass in (g) of the test specimen,
- b oven-dry mass in (g) of the test specimen.

2.5. Strength loss

The strength loss due to pretreatment was determined by measuring of the tensile strength of fabric samples before and after enzyme treatment on the Tensile Strength Tester and was expressed in percent. The strength loss (%) was calculated using the following formula:

$$\text{Strength loss (\%)} = \frac{(S1-S2)\times 100}{S1}$$

where:

- $S1$ breaking strength of the fabric before enzyme treatment,
- $S2$ breaking strength of the fabric after enzyme treatment.

2.6. Bending length

The bending length of the cotton fabric due to pretreatment was determined on the Cloth Stiffness Tester using IS 6490 : 1971 test method.

IS 6490 : 1971 – Method for Determination of Stiffness of Fabrics - Cantilever Test [TXD 1: Physical Methods of Tests]

Stiffness is resistance of fabric to bending. This standard prescribes a method for determination of stiffness of fabrics made from any textile fibre or a blend of two or more textile fibres. The principle employed is to measure a particular length of the fabric specimen of specified dimensions which when used as a cantilever bends to a constant angle under its own weight.

The samples were cut rectangular test specimens of 25×200 mm size from both warpway and weftway direction preferably with the help of a template from different portions of the sample under test. The lengthwise direction of specimens shall be parallel to the warp or weft direction for which the stiffness was determined. The bending length (cm) for warpway and weftway test specimens was calculated by taking the mean of three measurements for each test specimen by the following formula:

$$\text{Bending length (cm)} = \frac{L}{2}$$

where:

- L the mean length of over-hanging portion in centimetres.

2.6. Water absorption

The water absorption of the cotton fabric was determined using BS 3449 : 1990 test method by measuring the total amount of water that a fabric will absorb.

BS 3449 : 1990 – Method for resistance of fabrics to water absorption (Static immersion test).

In the test weighed samples of the cotton fabric were immersed in water for a given length of time, taken out and the excess water was removed by shaking. Then, they were weighed again and the weight of water absorbed was calculated as a percentage of the dry weight of the fabric. The test specimens of 80×80 mm were cut at 45° to the

warpway and then were weighed and immersed in water at $20\pm 1^{\circ}\text{C}$ to a depth of 10 cm. The samples were left in that position for 20 minutes. After removing were transferred to airtight containers and then reweighed. The water absorption (%) was calculated by taking the mean of three measurements for each test specimen by the following formula:

$$\text{Water absorption (\%)} = \frac{(\text{mass of water absorbed}) \times 100}{\text{original mass}}$$

2.7. Statistical analysis

One-way analysis of variance and Least Significant Difference (LSD) comparison test were used to statistically interpret mean differences in mean values if any, at 95 % accuracy level.

3. RESULTS AND DISCUSSION

3.1. Effect of concentration of enzyme on handloom cotton fabric

A set of trials were performed to find an optimum enzyme concentration for treatment on handloom cotton fabric. With varying concentrations of 0.4 to 2.8% owf (on weight of fabric) of commercial textile enzyme (Neutral Cellulase Powder Tep-15) at optimum pH 6.5 ± 0.2 , were treated the handloom cotton samples in Launderometer at temperature $50\pm 2^{\circ}\text{C}$ for 50 minutes. The minimum necessary enzyme concentration is 0.4% owf. The effect of concentration of enzyme on handloom cotton fabric is shown in *Table 1*. The results in *Table 1* indicate that on increasing the concentration of enzyme of 0.4 to 2.0% owf, was observed the large increase in the physical parameters, but on increasing the concentration of enzyme of 2.4 to 2.8% owf, slight or negligible increase. The significant increase in weight loss (1.51-3.64%), moisture content (2.02-4.02%), strength loss in both warp (0.41-1.07%) and weft (0.49-1.19%) and water absorption (34.58-70.02%) was observed on increasing the concentration of enzyme of 0.4-2.0% owf. The significant reduction in bending length in both warp (1.31-1.01 cm) and weft (1.28-0.95 cm) direction was observed on increasing the concentration of enzyme of 0.4-2.0% owf. From these results was chosen an optimum enzyme concentration of 2.0% owf.

Table 1.- Effect of concentration of neutral cellulase enzyme on handloom cotton fabric

Physical parameters	Enzyme concentration, % owf						
	0.4	0.8	1.2	1.6	2.0	2.4	2.8
Weight loss, %	1.51e	2.01d	2.39c	2.67b	3.64a	3.68a	3.71a
Moisture content, %	2.02e	2.76d	3.04c	3.59b	4.02a	4.11a	4.15a
Strength loss warp, %	0.41d	0.46d	0.59c	0.88b	1.07a	1.12a	1.15a
Strength loss weft, %	0.49c	0.55c	0.61c	0.93b	1.19a	1.23a	1.25a
Bending length warp, cm	1.31a	1.27a	1.16a	1.09b	1.01b	0.98b	0.97b
Bending length weft, cm	1.28a	1.23a	1.14a	1.06b	0.95b	0.93b	0.92b
Water absorption, %	34.58e	42.13d	50.36c	65.87b	70.02a	71.11a	71.25a

Results are the means of three repetitions. Different letters beside the mean of a compound denote a significant difference at the 95% significance level ($p < 0.05$).

3.2. Effect of treatment time of enzyme on handloom cotton fabric

A set of trials were performed to find an optimum treatment time on handloom cotton fabric with neutral cellulase enzyme. With different intervals of 30 to 80 minutes at optimum pH 6.5 ± 0.2 and concentration of 2.0% owf were treated the handloom cotton samples in Launderometer at temperature $50\pm 2^{\circ}\text{C}$. The effect of treatment time of enzyme on

handloom cotton fabric is shown in *Table 2*. The results in *Table 2* indicate that on increasing the treatment time of 30 to 70 minutes was found significant difference in the physical parameters, but on increasing the treatment time of 80 minutes, slight or negligible increase. The significant increase in weight loss (0.98-4.05%), moisture content (1.09-4.63%) and water absorption (43.57-78.05%) was found on increasing the treatment time of 30 to 70 minutes, and slight or negligible increase strength loss in both warp (0.36-0.47%) and weft (0.54-0.71). Tundall and Raleigh [7] have indicated that depending on the treatment time, a some weight loss and strength loss is to be expected after the enzyme treatment.

The reduction in bending length in both warp (1.21- 0.91cm) and weft (1.15-0.84 cm) direction was found on increasing the treatment time of 30 to 70 minutes. From these results was chosen an optimum treatment time of 70 minutes.

Table 2.- Effect of treatment time of neutral cellulase enzyme on handloom cotton fabric

Physical parameters	Treatment time, min					
	30	40	50	60	70	80
Weight loss, %	0.98e	1.54d	2.87c	3.64b	4.05a	4.09a
Moisture content, %	1.09c	1.68c	3.24b	3.78b	4.63a	4.67a
Strength loss warp, %	0.36b	0.39b	0.42b	0.45a	0.47a	0.48a
Strength loss weft, %	0.54b	0.59b	0.63b	0.68a	0.71a	0.72a
Bending length warp, cm	1.21a	1.17a	1.11a	0.97b	0.91b	0.89b
Bending length weft, cm	1.15a	1.08a	1.03a	0.93b	0.84b	0.81b
Water absorption, %	43.57d	58.32c	69.31b	72.59b	78.05a	78.11a

Results are the means of three repetitions. Different letters beside the mean of a compound denote a significant difference at the 95% significance level ($p < 0.05$).

3.3. Effect of temperature of enzyme treatment on handloom cotton fabric

A set of trials were performed to find an optimum temperature for treatment on handloom cotton fabric with neutral cellulase enzyme. With different temperature levels of 45 to 70 °C at optimum pH 6.5±0.2, concentration 2.0% owf and treatment time 70 minutes were treated the handloom cotton samples in Launderometer. The effect of temperature of enzyme treatment on handloom cotton fabric is shown in *Table 3*. The results in *Table 3* indicate that with different temperature levels (45-65 °C) enzyme treatment on handloom cotton fabric had small difference in the physical parameters, but on increasing temperature of 80 °C, negligible increase.

The minimum increase in weight loss (4.32-6-4.85%) and strength loss in both warp (0.49-0.81%) and weft (0.68-0.98) was found on increasing temperature of 45-65 °C. Similar results on the weight loss (3-5%) were obtained by Shukla et al. [8]. The significant increase in moisture content of the handloom cotton fabric (3.94-5,47%) and water absorption (70.53-85.02%) was found on increasing temperature of 45-65 °C.

The minimum reduction in bending length in both warp (0.92- 0.75cm) and weft (0.87-0.71 cm) direction was found on increasing temperature of 45-65 °C. From these results was chosen an optimum temperature of 65 °C.

Table 3.- Effect of temperature of neutral cellulase enzyme on handloom cotton fabric

Physical parameters	Temperature, °C					
	45	50	55	60	65	70
Weight loss, %	4.32b	4.54b	4.69a	4.78a	4.85a	4.87a

Moisture content, %	3.94c	4.21c	4.59b	4.93b	5.47a	5.51a
Strength loss warp, %	0.49b	0.59b	0.67b	0.72a	0.81a	0.83a
Strength loss weft, %	0.68b	0.73b	0.81b	0.89a	0.98a	0.99a
Bending length warp, cm	0.92a	0.88a	0.83a	0.79b	0.75b	0.74b
Bending length weft, cm	0.87a	0.84a	0.79a	0.75b	0.71b	0.70b
Water absorption, %	70.53d	72.89c	75.01c	81.27b	85.02a	85.21a

Results are the means of three repetitions. Different letters beside the mean of a compound denote a significant difference at the 95% significance level ($p < 0.05$).

4. CONCLUSIONS

Textile processing is a growing industry that traditionally has used a lot of water, energy and harsh chemicals. Biotechnology offers the potential for new industrial processes that require less energy and are based on renewable raw materials. When all the benefits of using enzymes are taken into consideration, it's not surprising that the number of commercial applications for enzymes is increasing every year. New enzymes with high specific activity, increased reaction speed, and tolerance to more extreme temperatures and pH could result in development of continuous processes. Cellulase enzymes are important tools in the textile industry for processing cellulose fibres. The enzymatic pretreatment of the cotton fabrics are not aggressive to fibres and environment. From the present study it was concluded that the positive effects obtained of enzyme treatment on handloom cotton fabric were maximized by using the optimized enzymatic process variables. The optimum conditions of the experiments obtained were: enzyme concentration - 2.0% owf; treatment time - 70 minutes; temperature - 65 °C. Nowadays, the use of cellulases in the pretreatment process found much broad acceptance. This study is very useful for cotton manufacturers, textile finishers and consumers.

There is still considerable potential for new and improved enzyme applications in future textile processing.

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