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TRESEARCH

MICROSTRUCTURAL CHANGES IN SECONDARY CELLULOSE DURING ENZYMATIC BIOPOLISHING

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ABSTRACT

In order to restore the primary texture and improve the properties of fabric made from secondary cellulose, enzymatic biopolishing using cellulase was applied. Microstructural changes in secondary cellulose during the biopolishing process, resulting from the breaking of certain chemical bonds and removal of impurities, were identified using FTIR spectroscopy. It was shown that enzymatic treatment increases the degree of amorphousness of the cellulose matrix and enhances its functional and technological properties during chemical finishing processes. The advantages of the biopolymer modification method were confirmed by comparing surface density, tensile strength, abrasion resistance, and air permeability of fabrics made from secondary cellulose, traditionally treated with soap-alkaline solutions, and enzymatically treated.

KEYWORDS: secondary cellulose, biopolishing, enzyme, microstructure, strength

1. INTRODUCTION

In recent years, the textile industry has become a promising market due to the growing demand from consumers for everyday yet innovative products [1, 2]. This extensive sector is considered one of the key economic fields in developing countries [3]. Textile manufacturing involves a series of chemical processes, primarily aimed at removing impurities, dyeing, and achieving the desired finishing effects [4]. Unfortunately, chemical treatments generate a significant amount of waste, especially during desizing, as well as through the use of bleaching agents and synthetic dyes, which contribute substantially to environmental pollution [5].

In recent decades, there has been a clear trend toward producing eco-friendly and hygienic textile products by developing efficient, cost-effective, and environmentally sustainable technologies, as well as using appropriate reagents [6]. Among these environmentally friendly agents are enzymes, which are widely used in textile manufacturing for removing sizing agents, cleaning, bleaching, dyeing, and final fabric finishing [7].

One of the final finishing processes, aimed at removing microfibrils and fuzzy fibers from the fabric surface after dyeing, is biopolishing [8]. This process plays a crucial role in preventing fibrillation and improves appearance, color brightness, tactile feel, water absorbency, and fiber crystallinity [9, 10]. It has been found that acidic cellulase derived from *C. globosum* can effectively perform biopolishing on cotton fabrics [11]. Another study reported that biopolishing using acidic cellulases is typically carried out at pH 4.5–5.5 and 55 °C, with the process lasting about 40 minutes [12]. Similarly, acidic cellulase obtained from *Trichoderma* species is used for bio-finishing of cotton fabrics at pH 5.5 and 55 °C, with a treatment time of 1 hour [13].

Modification, especially enzymatic, has a significant impact on the chemical structure and morphology of textile materials. Consequently, it affects their physical and mechanical properties, which are critical for selecting optimal processing conditions and predicting fabric performance. While substantial research has been devoted to the modification of primary cellulose, including enzymatic biopolishing, the biopolishing of secondary cellulose is of particular importance. It improves fabric texture and properties, making them closer to those of primary cellulose [14]. FTIR spectroscopy is applied to detect microstructural changes in cellulose fibers, such as hydrogen bonding alterations, impurity removal, and changes in glycosidic linkages, providing insight into the effect of enzymatic biopolishing [15].

The aim of this study is to investigate the microstructural transformations in secondary cellulose during enzymatic biopolishing using FTIR spectroscopy and to assess the resulting physical-mechanical properties. The research also

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highlights innovative aspects of enzymatic modification aimed at enhancing material performance and improving the sustainability of textile processing.

2. MATERIALS AND METHODS

The primary object of this study was a fabric made from secondary cellulose and viscose fibers obtained from technological waste of textile and garment-knitting enterprises. The warp of the fabric consists of 100% recycled cotton cellulose, while the weft is composed of 50% cellulose and 50% viscose fibers. The material was produced on an STB-175 weaving machine (Russia) with a linear density of 25×2 tex. A 3/1 twill weave is used. The fabric density is 200 threads in the warp and 220 threads in the weft.

For the biopolishing procedure, the weight of the untreated fabric sample was initially recorded. Biopolishing was carried out at various initial cellulase concentrations ranging from 0.5 to 5 g/l, temperatures of 30–60 °C, and durations of 30–180 minutes [14]. Based on the analysis of the results, the optimal treatment conditions were selected: enzyme concentration 2 g/l, fabric-to-liquor ratio 1:20, and incubation in a controlled thermostat for 2 hours at 60 °C. After the treatment, the fabric was taken out of the solution, rinsed first with running water, then with distilled water, and finally dried until it reached a constant weight.

For comparison, another fabric sample was subjected to traditional scouring in a soda-alkaline solution at a temperature of 90 – 95 °C for 20 – 30 minutes.

The microstructure of the samples was analyzed using a Fourier Transform Infrared (FT-IR) Spectrophotometer, Nicolet iN10 (Thermo Fisher Scientific, USA), within a scanning range of 500–4000 cm^{-1}.

The physical and mechanical properties of the treated fabrics were assessed in accordance with GOST 29298-92: *"Household cotton and blended fabrics. General technical specifications"*. Tests were conducted at the certified center "CENTEXUZ" of the Tashkent Institute of Textile and Light Industry, under standard conditions: temperature 20 ± 3 °C and relative humidity 65 ± 5%. All measuring instruments used in the certification center were manufactured in Japan.

To reduce error, the result of each test was taken as the average value of at least three samples, with deviation not exceeding 5% of the mean.

The surface density (M, g/m^2) of the fabric was determined by weighing the sample and calculating it using the formula:

$$M = \frac{m}{L \cdot B}$$

Where: *m* – mass of the fabric sample, g

L – length of the sample, mm

B – width of the sample, mm

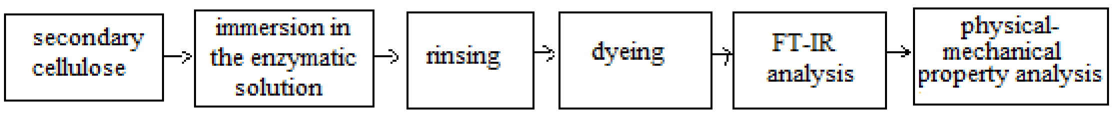
Abrasion resistance of the fabrics was tested using the MT – 194 abrasion testing machine. A circular fabric sample with a diameter of (85 ± 2) mm was placed between the abrasive disks of the device under a pressure of 1 kgf/cm^2. In the contact plane between the sample and the abrasive surface, planetary motion was applied. As soon as a hole appeared on the surface of the specimen, the device stopped automatically, and the abrasion resistance was recorded by the counter as the number of abrasion cycles.

Air permeability was measured using the AR – 360SM device. During the test, a flow of air was passed through the fabric sample by a built-in fan, while the hydrostatic pressure in the supply and suction chambers was measured. Using a reference table, the air permeability of the tested sample was determined and expressed in $cm^3/cm^2 \cdot s$.

Tensile strength was measuring using the AG–1 testing machine, designed for determining the breaking characteristics of fabrics, yarns, and other textile products. For tensile testing, samples were cut in both the warp and weft directions in the form of strips measuring 300 × 50 mm.

Each parameter under study was determined as the mean value of at least three measurements, whose deviations did not exceed 5% of the mean.

The biomodification of secondary cellulose and the testing of its properties can be represented by the following scheme:



The mass of the initial raw fabric sample was preliminarily weighed. An enzymatic solution was prepared using the required amount of cellulase enzyme. The fabric sample was immersed in the solution at a liquor ratio of 1:10 and

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incubated for 2 hours at a specified temperature in a thermostatic bath. After treatment, the fabric sample was removed from the solution, rinsed first with running water and then with distilled water, and dried to a constant mass. The bio-treated sample was subsequently dyed with a reactive dye. Finally, FT-IR analysis and the physico-mechanical properties of the finished samples were determined.

3. RESULTS AND DISCUSSION

To determine the microstructure of the biopolymer-modified fabrics made from secondary cellulose, FTIR (Fourier-transform infrared) spectroscopy was carried out. The FTIR spectrum of the enzymatically treated sample was compared with that of the original (untreated) secondary fabric and the fabric treated using the conventional alkaline method.

The FTIR spectrum of the untreated fabric sample is characterized by: a wide absorption range at 3336 and 3264 cm^{-1} , corresponding to stretching vibrations (ν) of hydroxyl groups (ν) ν_{O-H}, absorption in the range 2931 – 2849 cm^{-1}, corresponding to ν_{C-H}; stretching vibrations; a peak at 1660 cm^{-1}, which may be attributed to the presence of carbonyl – containing impurities such as residual lignin or proteins; distinct bands in the region 1200 – 1000 cm^{-1}, corresponding to ν_{C-O}, δ_{C-O} vibrations within the polysaccharide matrix; a characteristic peak at approximately 895 cm^{-1}, assigned to β – glycosidic linkages (β – 1,4) in cellulose (Figure 1).

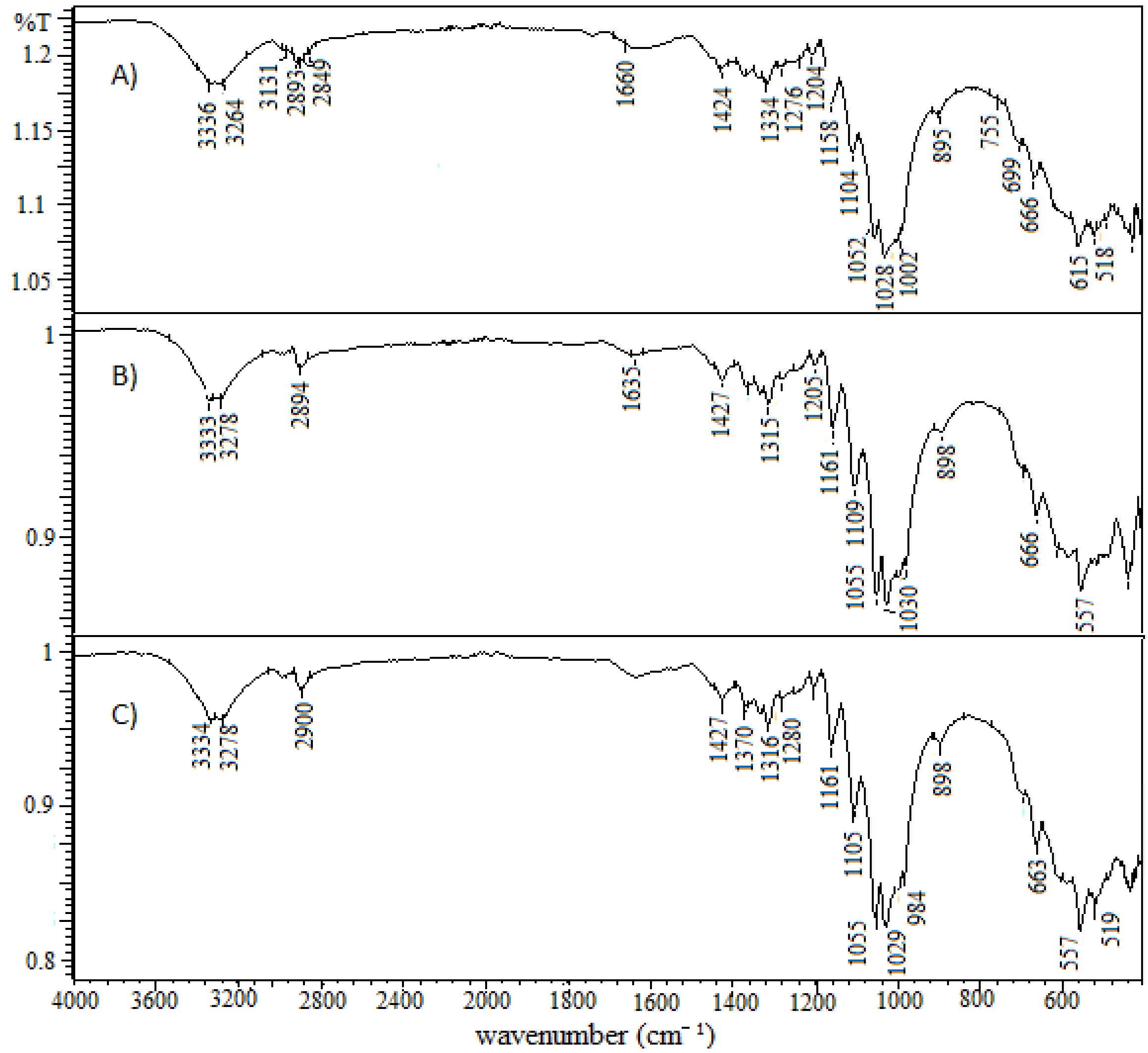

Figure 1 FTIR spectra of secondary cellulose fabric: A) untreated sample, B) after conventional treatment, C) after enzymatic treatment.

The FTIR spectrum of the sample after conventional treatment shows:

- A slight shift and narrowing of the ν_{O-H} absorption band (now at 3333 and 3278 cm^{-1}), indicating less disruption of hydrogen bonds;
- A band at 1635 cm^{-1}, suggesting the presence of residual impurities (e.g., lignin); sharper and more intense peaks in the region 1200 – 1000 cm^{-1}, indicating a partially crystalline structure;
- Preservation of the β – glycosidic peak at 898 cm^{-1}, confirming the structural integrity of cellulose chains.

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The FTIR spectrum of the sample after enzymatic treatment shows the following changes:

- A slight decrease in the intensity of the ν_{O-H} stretching bands (3334–3278 cm^{-1}), indicating partial disruption of hydrogen bonds;
- Disappearance of the band at 1660 cm^{-1}, indicating the removal of carbonyl impurities;
- Decreased intensity and flattening of bands in the 1200–1000 cm^{-1} region, indicating degradation or modification of glycosidic bonds;
- Preservation of the peak at 898 cm^{-1}, indicating the retention of the β-1,4-cellulose structure.

The conducted study showed that enzymatic treatment promotes the disruption of hydrogen bonds and the removal of copolymers (lignin, proteins), leading to the amorphization of the cellulose matrix. At the same time, traditional chemical treatment, although it removes some impurities, preserves a high degree of order and crystallinity of the structure. Thus, the enzymatic method is a selective and environmentally friendly approach to modifying the structure of secondary cellulose, facilitating more complete surface purification and depilling of the fabric.

The physical and mechanical properties of fabric made from secondary secondary cellulose were investigated. The following fabric samples were studied: 1 – Raw fabric, 2 – Fabric after traditional treatment, 3 – Fabric after enzymatic treatment.

When comparing the mechanical properties of fabric made from secondary cellulose after traditional preparation and bio – treatment, it can be noted that both finishing methods positively affected these characteristics. Contrary to expectations, an increase in the fabric's surface density, i.e., the mass of the fabric per 1 m², was observed as a result of the treatment (Figure 2).

This can be explained by an increase in the fabric's capillary properties, causing the fabric to absorb moisture from the air. It is possible that after treatment, the fabric becomes denser, which may be related to changes in the fiber structure and their mutual arrangement.

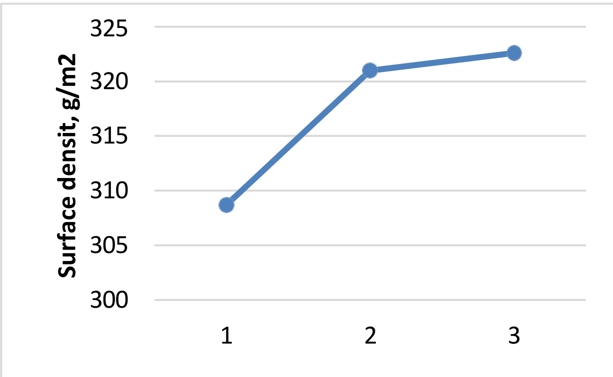
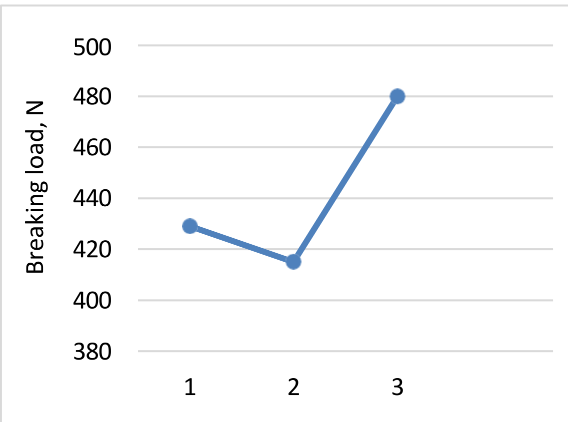


Figure 2. Surface density of fabric samples

A)



B)

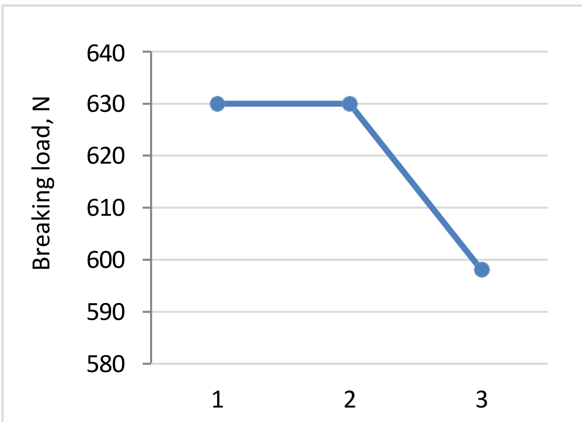


Figure 3 Breaking load of fabric samples, N: (A) warp direction, (B) weft direction

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The breaking load or tensile strength of the fabric depends on the strength of the fibers, as well as the structure and geometric dimensions of the material. It turns out that biomodification affects the warp and weft fibers differently (Figure 3).

The breaking load in the warp direction after cellulose treatment is higher than after traditional treatment and even higher than that of the raw fabric. At the same time, alkaline treatment led to a 4% decrease in the breaking load in the warp direction. Conversely, the fabric strength in the weft direction decreases by approximately 5% after enzymatic treatment. Apparently, the composition of the threads influences the change in strength. The warp threads consist entirely of secondary cellulose, while the weft threads are composed half of cellulose and half of viscose, which explains why the weft strength of the raw fabric is higher. Enzymatic treatment somewhat reduces the strength of viscose, which accounts for this result.

Both traditional and, to a greater extent, enzymatic treatments improve the fabric's abrasion resistance (Figure 4) and air permeability (Figure 5).

As shown by the data, traditional treatment increases the fabric's abrasion resistance by 20 – 25%, while enzymatic treatment increases it by 25 – 30%, making such fabrics more durable under operating conditions. This is likely due to the removal of surface roughness in the form of pills and fibers, which reduces the friction coefficient between the abrasive tester and the fabric surface, promoting smoother sliding of the disk and increasing the number of rotation cycles.

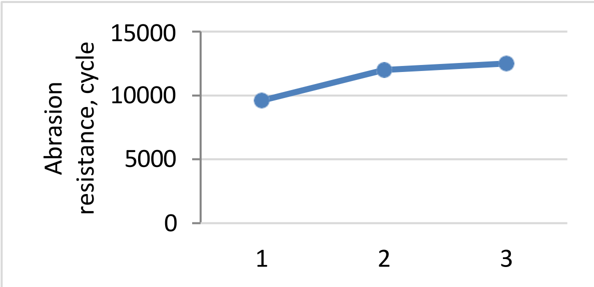

Figure 4 Abrasion resistance of fabric sample

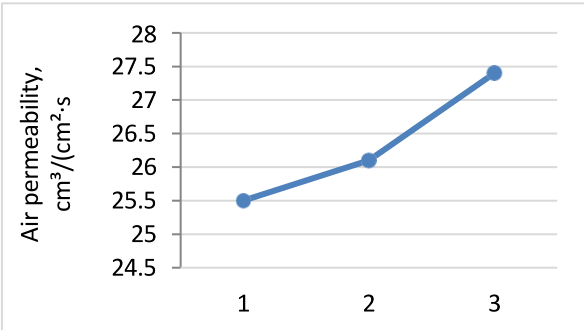

Figure 5. Air permeability of fabric samples

One of the important physical characteristics of fabric is its air permeability. Air permeability is defined as the ability of the fabric to allow airflow when a pressure difference is created on both sides of the tested sample. Air permeability depends on the fabric density, as well as the quantity, distribution, and diameter of macropores within the material. For the raw fabric sample, the air permeability value is 25.5 $cm^3/cm^2 \cdot s$. After traditional scouring and enzymatic treatment, this value increases by 2.3 – 7.4%. This is explained by the increased spacing between fibers due to the removal of the starch-based sizing layer, as well as pills and small fibers in the case of enzyme application.

CONCLUSIONS.

Enzymatic modification of textile materials, in particular biopolishing using cellulase, represents an effective and environmentally friendly alternative to traditional chemical treatments. This method contributes to the restoration of the primary texture of fabrics made from secondary cellulose and enhances their functional and mechanical properties by altering the microstructure: reducing the number of intermolecular hydrogen bonds, increasing the amorphous fraction of the cellulose matrix, and more completely removing associated impurities. The results confirm that enzymatic treatment is a selective approach that enables sustainable textile recycling and provides opportunities for further optimization of technological processes.

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TRESEARCH

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