STUDY AND OPTIMIZATION OF COTTON BIOBLEACHING METHODOLOGY

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ABSTRACT

The textile industry plays an important role in the economic scenarios of several countries. However, it stands out for the considerable environmental impact associated with its processes. In addition to significant water and energy consumption, textile processes generate substantial effluents containing dyes and chemicals. Therefore, the present study aims to develop a process for desizing, scouring, and bleaching cotton fabrics using an enzymatic one-bath, aiming to replace aggressive conventional methods with more sustainable alternatives. The enzymes α-amylase, amyloglucosidase, pectinase, and glucose oxidase were combined with titanium oxide (TiO_2) and photoactivation to study a more environmentally friendly process. Biodesizing proved to be effective in removing starch, producing enough glucose for the biobleaching step. Bioscouring, especially with longer treatment times, was comparable to conventional scouring, effectively removing pectin. In biobleaching, the photoactivation of hydrogen peroxide with TiO_2 increased the degree of whiteness of the fabrics, presenting itself as a promising alternative for bleaching the cotton. Based on studies that will be carried out and new conditions duly optimized, this approach can become a viable and sustainable alternative to conventional methods of bleaching cotton fabric.

Keywords: Biobleaching. Enzymatic processes. Cotton. Photoactivation.

1 INTRODUCTION

Cotton represents one of the most important and widely used natural fibers globally among textile substrates. The fiber is highlighted for its characteristics, such as excellent breathability and moisture absorption, and its significant resistance, contributing to the durability of products made with this material.[1]

To be adequately used, cotton must go through some processes. The preparation of cotton fabric includes the processes of desizing (removal of gum, generally starch-based), scouring (elimination of non-cellulosic impurities, such as pectins, greases, waxes, and proteins), and bleaching (elimination of natural pigments from the fibers), allowing the fabric to absorb dye solutions and chemicals evenly. However, for this to occur, highly alkaline chemicals (pH greater than 10.0) are used at high temperatures (above 90 °C), which remove impurities from the cotton and impact the cellulose, resulting in a notable loss of fabric strength and mass. Furthermore, these steps require an extensive rinsing process, resulting in the production of effluents containing substances harmful to the environment, in addition to high water consumption.[1]

Enzymes can be used in different stages of wet processing to generate more moderate processes, from preparation for dyeing to finishing processes. Enzymes are natural and biodegradable catalysts that catalyze chemical reactions under moderate pH, temperature, and pressure conditions, often avoiding the use of chemicals that are harmful to the environment. Furthermore, due to the high specificity of the enzymes, cellulose fibers are preserved, and treatments have minimal impact on the mechanical resistance of the fabrics.[2]

It is necessary to employ different enzymes to achieve the desired characteristics during fabric preparation. In the biodesizing step, α-amylase and amyloglucosidase are used, being enzymes capable of hydrolyzing starch. During bioscouring, enzymes such as pectinases, cellulases, proteases, xylanases, and lipases can be used. In the biobleaching step, enzymes such as peroxidase, laccase, glucose oxidase, and hemicellulase can be used. Although these processes are commonly performed separately, a more interesting approach is to conduct all these procedures using an enzymatic one-bath. In this sense, a promising alternative consists of coupling enzymatic reactions, using enzymes whose products can serve as substrates for other enzymes until the fabric achieves the desired characteristics.[3]

Therefore, the present project aims to develop a one-pot process for desizing, scouring, and bleaching cotton fabric by combining enzymes in an enzymatic one-bath and studying more moderate forms of activation of hydrogen peroxide produced *in situ*. Thus, the aim is to optimize time, save water and chemicals, and reduce the environmental impacts generated by the bleaching processes. The aim is to obtain a cotton fabric with a satisfactory degree of whiteness, good physical and mechanical properties, and high wettability so that the desired finish can be given to the product efficiently.

2 MATERIAL & METHODS

Based on the methodology described by Mojsov[1], the enzymatic treatment baths were prepared in a 1:20 ratio, that is, for every 1 unit of fabric mass (in grams), 20 units of bath volume were used (in milliliters). The cotton used in each test consists of a plain weave with an area of 4 cm^2 and a mass of approximately 50 mg.

To study the biodesizing step, enzymatic baths were prepared by adding the enzymes α-amylase (2 units/g of fabric) and amyloglucosidase (3 units/g of fabric). The final bath volume was completed with acetate buffer pH 5.0 containing the surfactant Berol® with a concentration of 1.5 g/L. The baths were placed in an orbital shaker/incubator at 60 °C and shaken at 115 rpm for 30, 60 and 120 minutes. All assays were conducted in duplicate.[1]

To ensure that the step was successful, the glucose produced by starch hydrolysis was quantified by reacting an aliquot of the enzymatic bath with a specific colorimetric reagent for this sugar. The color change was analyzed using a UV-Vis spectrophotometer, and the concentration was calculated using a calibration curve as a reference. Furthermore, the removal of starch from the treated fabric was verified by testing with an iodine/potassium iodide solution. The concentration of starch present in cotton was determined by the intensity of the blue color using the Tegewa scale, where the darker color (grade 1) indicates a high presence of starch, while the pale yellow color (grade 9) means that the starch was efficiently hydrolyzed. For comparison, this test was performed with raw cotton that was not treated. Fabric colors were obtained by analysis in a standard lamp remission spectrophotometer (Datacolor 500).[4]

After the biodesizing step, bioscouring was studied. To this end, enzyme baths were prepared by adding different amounts of the pectinase enzyme to the conditions already optimized from the previous step. Furthermore, the influence of time on this process was also verified. In this sense, 4 units of pectinase per gram of fabric were added to the bath, shaken at 115 rpm and 60 °C for 60 and 120 minutes. The same was done using 20 enzyme units per gram of fabric. For subsequent analysis, conventional scouring was performed, in which the fabric (which also underwent biodesizing) was treated with 3 g/L NaOH at 95 °C for 60 minutes. All assays were conducted in duplicate.[1,5]

To verify the efficiency of the bioscouring step, the removal of pectin from treated fabrics was evaluated through the reaction with methylene blue dye. For analysis, this test was also performed with raw cotton. After the tests, the K/S values of each sample were read at the maximum wavelength using a remission spectrophotometer with a standard lamp. In this way, the percentage of residual pectin in the fabrics was quantified using Equation 1, where K/S is the value of the enzymatically treated fabric sample, K/S_0 is the value of the conventionally treated sample, referenced as 0% of residual pectin, and K/S_{100} is the value of the raw fabric sample, referenced as 100% residual pectin.[6,7]

$$\%\ Residual\ pectin = \frac{(K/S\ - K/S_0)}{(K/S_{100} - K/S_0)} \times 100 \quad (1)$$

To study the subsequent stage, which consists of biobleaching combined with the activation of the hydrogen peroxide produced, some preliminary tests were carried out by adding 25 units of the enzyme glucose oxidase (GOx) per milliliter of bath. Furthermore, 5 and 10 g/L of TiO_2 were added to the baths, which were introduced into a cabin containing a UV lamp (254 nm) to photoactivate the hydrogen peroxide. A blank (without adding of TiO_2) was also irradiated by UV light for comparison. The tests were carried out at 35 °C for 60 minutes. The whiteness index of the samples, expressed in °Berger, was measured using a remission spectrophotometer with a standard lamp.[8]

3 RESULTS & DISCUSSION

The glucose produced during the biodesizing step was determined, obtaining a value of 8.6 ± 0.7 g/L. To obtain satisfactory bleaching, 4 g/L of glucose is necessary in the enzymatic bath during the pre-treatment step.[1] In this sense, the result obtained is promising so that the following steps can be carried out efficiently since the production of this amount of glucose not only meets the desired requirements for bleaching, but also establishes a solid basis for subsequent optimizations, indicating significant potential for successful process development.

The results of the starch removal tests also corroborate the efficiency in the biodesizing step. Figure 1 illustrates the colors of the fabrics obtained after the tests with the iodine/potassium iodide solution. The color obtained is shown (a) for an enzymatically treated cotton and (b) for raw cotton. For starch hydrolysis to be successful, it is necessary to reach grade 6 on the Tegewa scale.[4] Therefore, when comparing, it is possible to classify the enzymatically treated fabrics between grades 7 and 8, indicating that starch removal occurred efficiently and will not compromise the next pre-treatment steps. Furthermore, it was also possible to verify that the raw fabric has a high amount of starch, as expected, considering that it did not undergo any pre-treatment, classifying it as grade 1 on the scale. It is worth mentioning that only one image was presented for the enzymatic tests carried out, as there was no significant change in the color of the fabric due to the different times used.

Figure 1 Color of the (a) enzymatically treated cotton and (b) raw cotton after starch removal tests.

The tests to evaluate the efficiency of the bioscouring step were carried out with a methylene blue solution, as this dye interacts with the carboxylate anion present in cotton pectin. In other words, the bluer the fabric is after the tests, the greater the amount of pectin remaining in the fabric, which is reflected in the K/S value obtained.[9] Table 1 presents the percentages of residual pectin after the respective enzymatic treatments.

Table 1 Percentage of residual pectin after tests with methylene blue dye.

Enzyme ratio (units/g of fabric)	Time (min)	Residual pectin (%)
4	60	53 ± 4
	120	7,9 ± 0,8
20	60	5,0 ± 0,4
	120	0

Based on the values in the table, it is possible to observe that time has a more significant impact when removing pectin from cotton fabric. According to Joshi and collaborators[5], between 60 and 120 minutes, the efficiency in the process of removing cellulosic impurities is quite pronounced, which can also be observed in this study. This can be verified by finding a value of residual pectin after 120 minutes comparable to the value found after 60 minutes using an amount of enzyme five times greater. Another fact corroborating this discussion is that the enzymatic treatment with more enzyme (20 units/g of fabric) after 120 minutes is as efficient as conventional scouring since the fabric does not present residual pectin. This way, these conditions can be better optimized so as not to compromise the bleaching of the fabric in the next step.

Table 2 presents the whiteness index obtained after the different treatments regarding the fabric bleaching tests. From the results obtained, it can be inferred that the GOx enzyme effectively catalyzes glucose (produced in the biodesizing step), forming hydrogen peroxide *in situ*. However, given that this enzyme works under moderate temperature (35 °C) and pH (around 5.0), the hydrogen peroxide produced is not active in the enzyme bath and, therefore, is not as efficient in bleaching the cotton.[1] By adding TiO_2 and irradiating the bath with UV light, an increase in the degree of whiteness was observed, indicating that TiO_2 may function as a moderate alternative to produce hydroxyl radicals (from hydrogen peroxide), which are more efficient oxidizing agents in bleaching fabric. In this way, new studies will be carried out and the conditions will be optimized to improve the efficiency of the process and achieve the degree of whiteness that the industry requires.

Table 2 Whiteness index obtained in the biobleaching step combined with photoactivation of hydrogen peroxide.

Sample	Whiteness index (°Berger)
Raw cotton	11,5
GOx	16,2
GOx + UV	17,8
GOx + UV + TiO_2 5 g/L	25,9
GOx + UV + TiO_2 10 g/L	28,4

4 CONCLUSION

This study developed a process for desizing, scouring, and bleaching cotton fabrics using an enzymatic one-bath, demonstrating that the enzymes α-amylase, amyloglucosidase, pectinase, and glucose oxidase, combined with TiO_2 and photoactivation, can be a promising alternative to conventional methods, which are more aggressive and environmentally harmful. The biodesizing step proved to be efficient in removing starch, with the treated fabrics reaching the necessary grade on the Tegewa scale and producing a satisfactory amount of glucose to serve as a substrate in the biobleaching step. Bioscouring, especially with a longer treatment time, has proven comparable to conventional scouring, effectively removing pectin. In biobleaching, the addition of TiO_2 and photoactivation of hydrogen peroxide significantly increased the degree of whiteness, indicating that the proposed approach has a promising potential to optimize cotton fabric preparation processes, minimizing the use of aggressive chemicals, saving water and energy, and providing satisfactory results in terms of whiteness and mechanical properties.

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