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RESEARCH PAPER

Cellulase immobilized on kaolin as a potential approach to improve the quality of knitted fabric

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Abstract

Biopolishing is a textile process that uses cellulases to improve the pilling resistance of fabrics. Although the process improves the pilling resistance, softness and color brightness of fabrics, it causes a significant loss of tensile strength in treated fabrics. The present work studied the use of cellulase immobilized on kaolin by adsorption and covalent bonding in biopolishing to get around this problem. The cellulase immobilization has been reported as promising alternative to overcome the inconvenient of biopolishing, but it has been very poorly explored. The results showed that cellulase immobilized by both covalent bonding and adsorption methods provided to the knitted fabric similar or superior pilling resistance to free cellulase, but with greater tensile strength. Immobilization also allowed for efficient recovery and reuse of the enzyme. The present work is a relevant contribution to the literature, since, as far as we know, it is the first work that shows it is possible to minimize the loss of tensile strength and also reuse the immobilized enzyme, giving a better-quality product and also contribution to reducing the cost of the polishing step.

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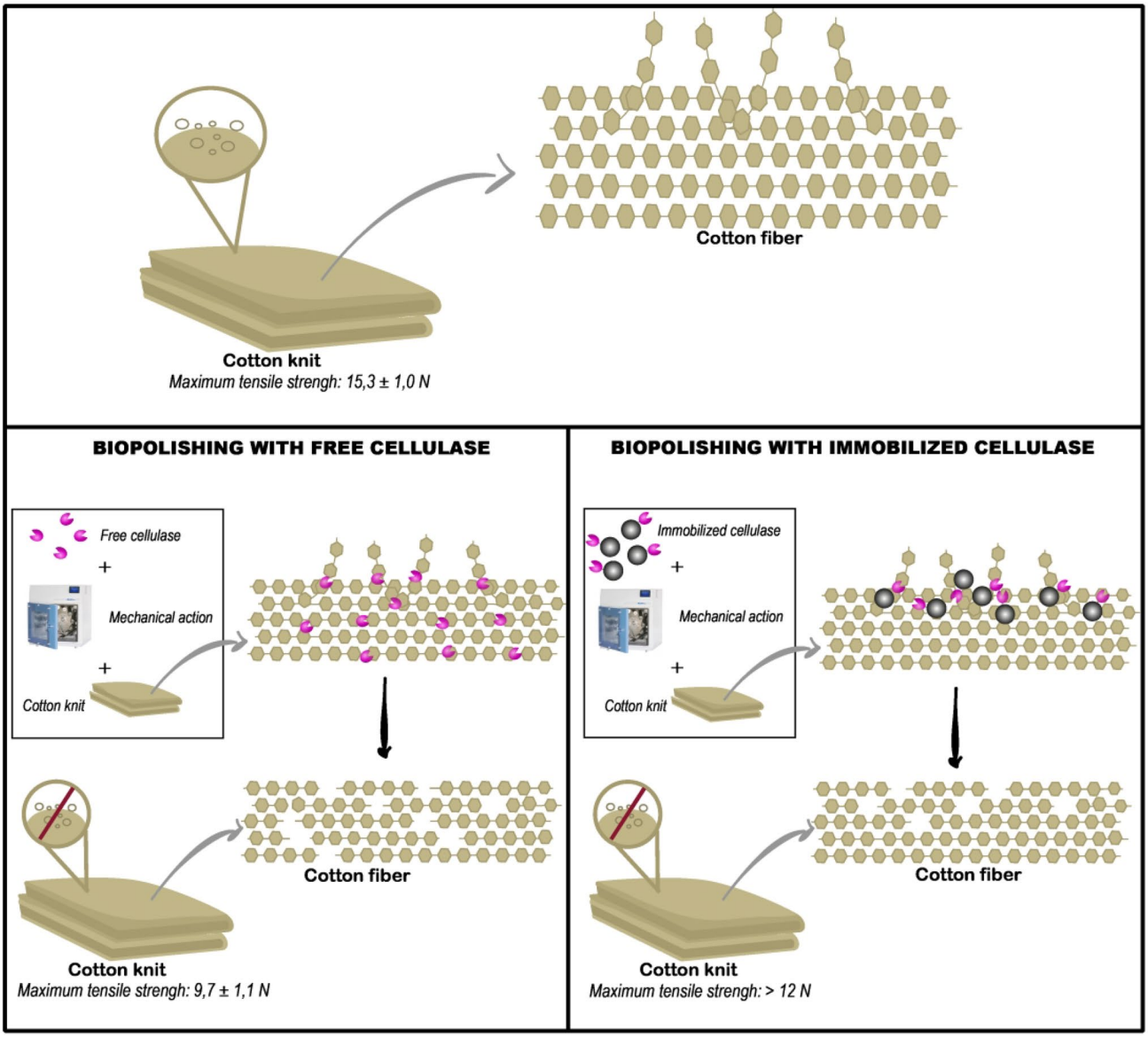
Graphical abstract



Keywords Biopolishing · Cellulase immobilization · Enzyme reuse · Textile · Kaolin

Abbreviations

BPc	Biopolishing with buffer solution only
BPk	Biopolishing with buffer solution and kaolin
BPe	Biopolishing with free cellulase
BPads	Biopolishing with cellulase immobilized on kaolin by adsorption
BPcov	Biopolishing with cellulase immobilized on kaolin by covalent bonding

Introduction

Enzymes are a sustainable alternative for textile industries to produce the same or sometimes even better-quality products with less chemical, water, and energy consumption and with less problematic waste generation than traditional processes [1–3]. Therefore, their use in some processes of textile production can help both industry and the environment [4].

Enzymes can be used for various wet processing operations, from cleaning preparations to finishing processes [5–7]. Biopolishing is a finishing process that combines

mechanical agitation and cellulase action to reduce the pilling of cellulosic fabrics (natural and regenerated) [8–10]. Pilling is a term used for small tangles of fibers in the form of a ball that is attached to the fabric surface. The pills are formed during wearing and washing by the entanglement of protruding microfibers that come off from the yarn surface [11]. Pilling is a problem that not only affects the appearance and touch of fabrics but also reduces their useful life [12].

Besides improving the resistance to pilling, the biopolishing process also provides a softer, smoother handle and better color brightness to fabrics [13, 14]. Although biopolishing improves the quality of cellulosic fabrics, it inevitably causes significant tensile strength loss to treated fabrics [15, 16]. This problem has been attributed to the hydrolysis of cellulose in the interior of the cotton fibers since cellulase can easily diffuse into them [17–19]. To minimize this problem, one of the proposed approaches is the immobilization of the enzyme on supports. Immobilization may limit the diffusion of the enzyme, particularly inhibiting its penetration into cotton fibers [17, 18, 20, 21]. Thus, it is expected that the loss of tensile strength will reduce, because the enzymatic hydrolysis will be limited to the surface of the cotton fibers preserving the cellulose molecules in the interior of the cotton fibers [17, 18, 20, 21].

The immobilization may offer another important advantage: the possibility of reusing the biocatalyst [20]. The reuse of immobilized enzymes is very important, because it can compensate for the costs associated with immobilization and thereby make the technique economically viable.

Yu et al. [19, 22, 23], and Sankarraj and Nallathambi [17] showed that immobilized cellulase improved the surface properties of the fabrics causing lower tensile strength loss than free cellulase. Although these researchers have obtained important results for the biopolishing process, they have not evaluated the reuse of immobilized cellulase. Kumar et al. [24] showed that immobilized cellulase could be used for biopolishing for successive cycles efficiently, but the immobilized enzyme did not minimize the tensile strength loss.

So far, there are no papers in the literature that have proved the minimization of tensile strength loss of treated fabrics and efficient reuse of immobilized cellulase at the same time. Moreover, the supports used (Eudragit, ConcanavalinA layered with calcium alginate bead and epoxy resin) are organic or/and synthetic, expensive, not mechanically resistant to high agitation conditions (as required in biopolishing process), which makes them not adequate for industrial applications.

In this context, the present investigation aims to evaluate the effects of free and immobilized cellulase on the physical and mechanical properties of cotton knitted fabric, as well as, the reusability of immobilized cellulase. The physical and mechanical properties of cotton knitted fabric such as pilling resistance, mass variation, tensile strength, and white index have been analyzed for free and immobilized enzymes. The cellulase was immobilized on kaolin, an inorganic, natural, low-cost material, that has not yet been used in biopolishing processes.

Materials and methods

Materials

The commercial enzyme preparation Biokey AKM (rich in endoglucanases) was kindly donated by Akmey Brazil (Indaial, Santa Catarina, Brazil). The enzyme preparation was submitted to a dialysis process with phosphate buffer solution, pH 6.0, for 7 days at room temperature (approximately 25 ºC). The resulting enzyme solution was lyophilized and stored under refrigeration (4 ºC).

Kaolin (Saca B) (Imerys—Pará, Brazil), 3-aminopropyltriethoxysilane (APTES) (Sigma Aldrich), glutaraldehyde (GA, 25% w/v solution in water) (Vetec), dibasic sodium dihydrogen phosphate (P.A) (Vetec), and monobasic anhydrous potassium phosphate (P.A) (Vetec) were used for enzyme immobilization. Sodium carboxymethylcellulose (CMC) (Sigma Aldrich), acid 3,5-dinitrosalicylic (DNS, P.A) (Vetec), citric acid monohydrate (PA) (Vetec), potassium and sodium tartrate (P.A) (Dinamica), glucose D (+) anhydrous dextrose (P.A) (Vetec), and sodium hydroxide (P.A) (Lafan) were used for enzymatic assays.

A grey knitted fabric (150 g/m^2, 100% cotton, and made from open-end yarn) was used as a textile substrate. Before the experiments, the knitted fabric was scoured and bleached according to ABNT NBR 13218-1994 [25] using a solution containing sodium silicate (5 g/L), magnesium sulfate (5 g/L), hydrogen peroxide (2% v/v), and emulsifier Prote-Pon WRR 14-BR (Prox do Brazil) (1% v/v) with pH adjusted to 11 by adding caustic soda solution (50% w/v). The process was performed at 90 ºC, for 60 min, and 1:10 (1 g of knitted fabric for 10 mL of solution) fabric-to-liquor ratio, using a rotary drum machine (MTP-HT, Mathis). At the end of the process, the knitted fabric was neutralized with an acetic acid solution (2% v/v), using the same bath ratio used previously, at room temperature for 10 min. Finally, the knitted fabric was rinsed twice with water, dried at ambient conditions, and stored.

Enzyme immobilization

The immobilization of cellulase on kaolin by covalent bonding and by adsorption was carried out according to the methodology previously described by Lima et al. [26, 27], respectively. The immobilization process was carried out by adding 10% (w/v) of natural kaolin (for immobilization by adsorption) or functionalized and activated kaolin (for

immobilization by covalent bonding) to phosphate buffer solution (50 mM phosphate buffer pH 7.0) containing 100 mg/mL of cellulase. This mixture was then stirred for 24 h, at 25 ºC and 150 rpm. Subsequently, the kaolin particles were recovered by centrifugation (3 min and 3130×*g*), washed, suspended in buffer solution, and stored at 4 ºC.

Biopolishing

The cotton knitted fabric was treated with free and immobilized (covalent bonding and adsorption) cellulases using a laboratory beaker dyeing apparatus (HT ALT-B, Mathis). The process conditions used were 1:30 fabric-to-liquor ratio, pH 5 (acetate buffer, 50 mM), 50 ºC, and 90 min. The free and immobilized cellulases were applied with the same CMCase activity. Ten steel balls (diameter 0.6 cm) were added to each beaker to enhance the mechanical agitation (according to ABNT NBR ISO 105-C06 [28]). After the cellulase treatments, the samples were rinsed three times with acetate buffer (pH 5, 50 mM). Then, it was performed the deactivation of residual cellulases using 1 mM sodium hydroxide at 100 ºC for 15 min. Finally, the samples were rinsed with water and dried at 60 ºC for 2 h.

To evaluate the reusability of immobilized enzymes, the solutions (bath) used during the biopolishing and the solution resulting from samples washing were centrifuged to recovery the immobilized enzymes. After the centrifugation, the particles were resuspended keeping the 1:30 fabric-to-liquor ratio and used in the biopolishing of new samples.

Two control assays were performed under the same conditions but without cellulase: only buffer solution (BPc) was used in the first control assay and buffer solution with kaolin (BPk) was used in the second.

Dyeing procedure

After the biopolishing, the treated and untreated knitted fabrics were dyed using a reactive red dye (Tiafix AF/B—Aupicor Química). The procedure was performed using the laboratory beaker dyeing apparatus (HT ALT-B, Mathis) with a 1:50 fabric-to-liquor ratio following the procedure represented in Fig. 1. At the beginning of the dyeing process, the samples were added to a solution containing 1.5 g/L of dispersant Ladiquest 2005, 0.6% (in relation to knitted fabric sample weight) red dye Tiafix AF/B, and 80 g/L sodium chloride. After 30 min, soda ash (sodium carbonate), at 6.7 g/L concentration, was added in two steps and the process was carried out for another 60 min. At the end, the dyeing bath was discarded and the samples were washed according to the procedure described in Fig. 2. In the first step of washing, 0.75 g/L of dispersant Verolan NVR and water were used. In the last step, the water pH was adjusted for 6.5 adding acetic acid. After washing, the samples were dried at 60 ºC for 2 h. The dyeing assays were performed in duplicate.

Analysis

Mass variation

Mass variation of the samples was determined by the difference in mass of samples before and after biopolishing according to Eq. 1. Before the analysis, the samples were dried at 60 °C for 2 h and cooled in a desiccator. The average mass variation was recorded by considering eight specimens for each treatment:

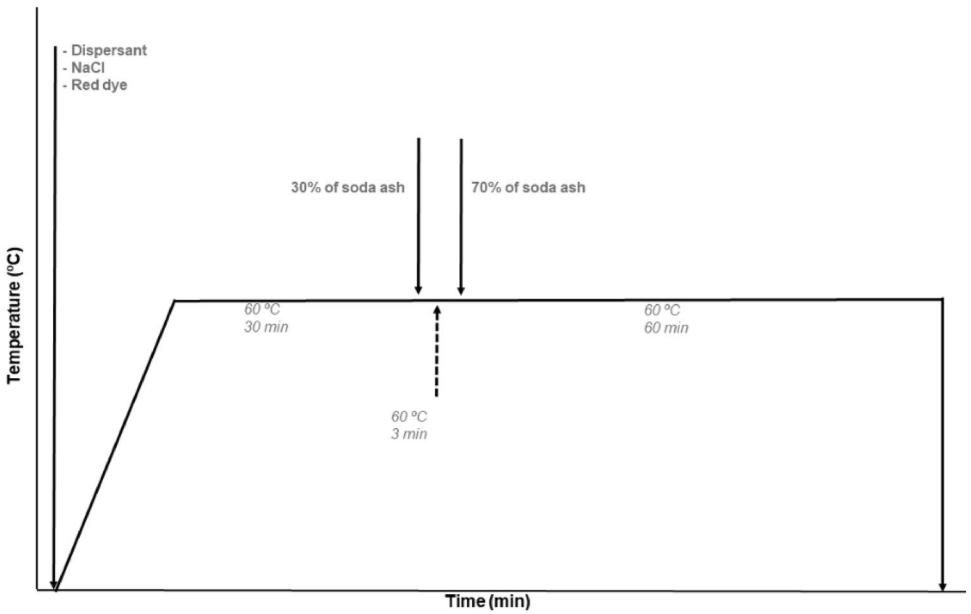


Fig. 1 Dyeing procedure for cotton knitted using a reactive dye and 1:50 fabric-to-liquor ratio

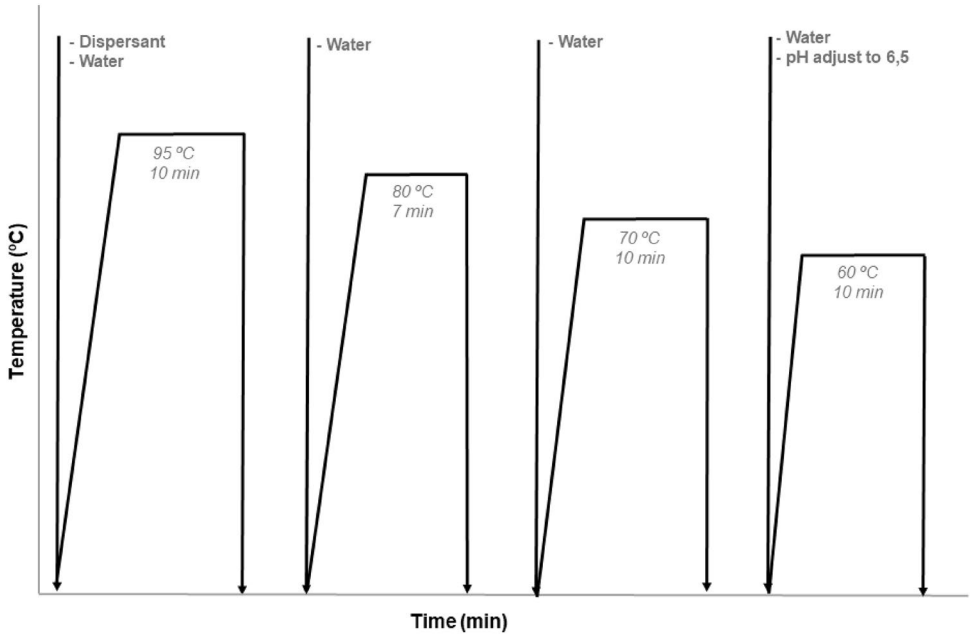


Fig. 2 Washing procedure after dyeing process of cotton knitted (1:50 fabric-to-liquor ratio)

$$\text{Mass variation } (\%) = \left(\frac{W_0 - W_1}{W_0}\right) \times 100 \quad (1)$$

where W_0 is the weight of the knitted fabric sample before biopolishing; W_1 is the weight of the knitted fabric sample after biopolishing.

Tensile strength

Tensile strength tests were performed according to the adapted ABNT NBR ISO 13934 [29] standard using a texturometer (TA.HD plus, Stable Micro Systems) with a load cell of 50 N. The size of the specimens was 25 mm wide and 100 mm long. The tensile strength of the samples was calculated as the mean value of ten specimens from the warp (machine) direction.

Whiteness index

The whiteness index of the samples was measured following the ASTM E313-15 [30] standard using the spectrophotometer (CM-3600a, Konica Minolta). Measurements were made in three different regions of each specimen. The whiteness index was recorded by considering six specimens for each treatment.

Pilling resistance

Pilling resistance was evaluated according to ASTM D4970 [31] standard (Standard for pilling tests) using a Martindale procedure (3000 rubbing/cycles). The degree of pilling was visually evaluated by comparing the sample with the photographic patterns and rated on a scale of 1–5, with 1 indicating severe pilling and 5 indicating no pilling.

Surface morphology

The surface morphology of the treated and untreated knitted fabric was analyzed using Scanning electron microscope (SEM) (JSM-5919LV, JEOL). Prior to analysis, the samples were coated with gold using a sputter coater.

Measurement of cellulase activity

Enzymatic activity of free and immobilized endoglucanase was determined according to the methodology previously described by Lima et al. [32] with some modifications. The assays were carried out with 900 µL of 4% CMC solution (prepared with 0.15 M citrate–phosphate buffer pH 5.0) and 100 µL of cellulase solution at 50 °C for 30 min. At the end, 1.5 mL of dinitrosalicylic acid (DNS) solution was added to each sample to stop the hydrolysis reaction and quantify the reducing sugars (DNS method [33]). The samples were heated in a boiling water bath for 5 min and cooled in an iced bath. Before absorbance measurements, the samples were diluted and centrifuged at 3130×*g* for 3 min. The absorbance of the samples was measured at 540 nm using a UV–Vis spectrophotometer (Cirrus 80, Femto) and it was converted to reducing sugars concentration through a calibration curve with glucose as standard. All assays were carried in triplicate and mean values are reported.

Dyeing properties of treated knitted fabric

The color evaluation of the dyed knitted fabric was carried out using a spectrophotometer (CM-3600a, Konica Minolta) according to the CIE system (CIELAB). CIELAB system relates visual color differences to colorimetric measurement. It expresses color as three values: L^*, a^*, and b^*. L^* represents the color's lightness and varies from 0 (perfect black) to 100 (perfect white). The value of a^* is a measure of the red–green character of the color (a^*, negative values indicate green shades, while positive values indicate red). The value of b^* gives the yellow–blue character (b^*, negative values indicate blue shades and positive values indicate yellow) [34]. From these three values, it is calculated the total color difference between a sample and a reference (ΔE^*). The higher ΔE^* value, the greater the color difference and, consequently, the more perceptible the difference to the human eye [35]. Each replicate was analyzed at four different points and mean values were reported. The control sample BPc was used as reference (standard).

Results and discussion

Biopolishing with free and immobilized cellulase

According to Table 1, the pilling resistance was improved when the knitted fabric was treated with both free and immobilized cellulases. The greater pilling resistance of these samples can be attributed to the removal of protruding microfibrils by the enzyme. It can be observed through SEM images (Fig. 3), in which the control samples (BPc and BPk) showed a higher number of microfibrils on the surface than the samples treated with cellulase (BPe, BPads, and BPcov).

Samples treated with immobilized enzyme showed equal or superior pilling resistance to the samples treated with free cellulase, which suggests that immobilized cellulases may promote adequate microfibrils removal. Thus, the next step was to assess whether the immobilized cellulase was able to improve the tensile strength.

As can be observed in Table 1, compared with the control sample (BPc), the samples treated with free cellulase (BPe) showed about 36% reduction in tensile strength, while the samples treated with immobilized cellulase, BPads, and BPcov, showed about 20 and 12% reduction in tensile strength, respectively. During the assays, some samples treated with free cellulase showed some damages, such as holes in the fabric, which was not noted for samples treated with immobilized cellulase. These findings suggest the treatments using free cellulase caused more damage in the internal structure of the knitted fabric than treatments using immobilized cellulase, which corroborate the hypothesis of many researchers [17, 18, 20, 21].

Biopolishing also causes fabric weight loss, since the process removes microfibrils from the surface of cotton fibers. For Saravanan et al. [36], weight loss of about 1–2% appears to be enough to obtain a remarkable reduction in pilling tendency for knitted fabric. Commercially, weight loss of 3–6% is considered acceptable [21, 37, 38].

Also, according to Table 1, the samples treated with free cellulase (BPe) showed a 2.5% weight loss, a value commercially acceptable. In contrast, the samples BPk, BPads, and BPcov showed an increase in weight. This result can be attributed to the physical retention of kaolin particles in the micropores or empty spaces between the microfibrils of the cotton filaments [34], as can be observed in SEM images (Fig. 3). From SEM images, kaolin particles can be seen deposited on BPads, BPcov, and BPk samples, while no clay particles were observed in the BPe and BPc samples.

When comparing the samples treated with immobilized cellulase, it is observed that the BPcov sample showed a higher mass increasing than the BPads sample. The greater incorporation of kaolin particles observed in BPcov sample may be also attributed to chemical reactions between kaolin and cellulose, such as: (i) the condensation of silane groups of APTES (introduced into the surface of kaolin to obtain the covalent bonding with the cellulase) with hydroxyl groups

Table 1 Physical–mechanical properties of treated cotton knitted fabrics

Sample	Mass variation (%)	Maximum tensile strength (N)	Pilling resistance	Whiteness index
BPc	− 0.6 ± 0.1	15.3 ± 1.0	1.0	61 ± 1
BPk	+ 2.4 ± 0.04	14.9 ± 0.7	1.0	58 ± 1
BPe	− 2.5 ± 0.3	9.7 ± 1.1	3.0	61 ± 1
BPads-1st cycle	+ 0.9 ± 0.5	12.3 ± 0.8	3.0	59 ± 1
BPads-2nd cycle	+ 1.2 ± 0.4	14.3 ± 0.9	3.0	60 ± 1
BPads-3rd cycle	+ 1.7 ± 0.4	14.5 ± 0.4	2.5	60 ± 1
BPcov-1st cycle	+ 4.5 ± 0.3	13.5 ± 0.9	4.0	39 ± 2
BPcov-2nd cycle	+ 4.9 ± 0.4	13.6 ± 0.6	3.0	41 ± 2
BPcov-3rd cycle	+ 4.3 ± 0.4	13.9 ± 1.1	3.5	41 ± 2

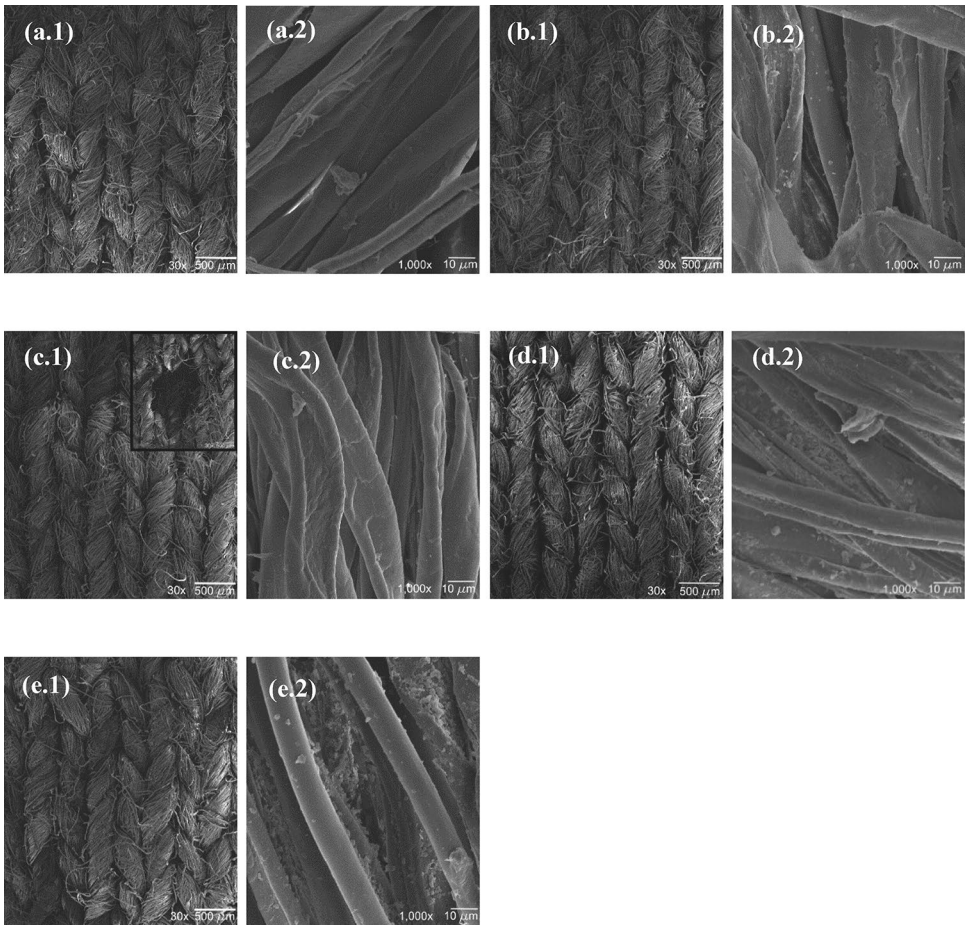


Fig. 3 Scanning electron microscope of **a** BPc. **b** BPk. **c** BPe. **d** BPads. **e** BPcov. The numbers 1 and 2 indicate the 30 and ×1000 magnification, respectively

of cellulose [39–42]; (ii) the acetylation reaction between the glutaraldehyde free aldehyde group (used to make the APTES amino groups reactive to cellulase) and the cellulose hydroxyl groups [43, 44].

According to the results, there were no significant differences in pilling resistance and tensile strength between BPk and BPc samples, indicating that kaolin did not influence the microfibrils removal and neither in mechanical properties of knitted fabric.

Due to the incorporation of the kaolin particles observed previously, the whiteness index was evaluated to verify the possible interference of these particles in the mesh brightness after biopolishing. According to the ASTM standard, the whiteness index is a number, calculated from colorimetric data, that express the degree of departure of an object color in relation to a preferred white (a standard) [30]. An adequate whiteness index is essential if high-quality white goods are being produced, or if the goods will be dyed with pale bright colors [34].

BPc, BPk, BPe, and BPads samples showed a similar whiteness index (Table 1). However, the BPcov sample showed a lower whiteness index, which indicates the yellowness of the knitted fabric. These results suggest that only functionalized kaolin particles had affected the whiteness of the knitted fabric samples. This problem can be explained by the color change (yellowing) of the kaolin particles after the glutaraldehyde activation step that intensified after the enzyme binding. The yellowish color of kaolin may be attributed to the formation of Schiff bases between silane-glutaraldehyde and glutaraldehyde-enzyme [45].

Reuse of immobilized cellulase

For biopolishing, cellulase immobilization is a strategy to minimize the problems caused by free cellulase. However, immobilization also allows the reuse of the enzyme, which is very important, since it is associated with the economic viability of the process. Immobilized enzymes have an additional cost over their free form due to the costs of the support and immobilization process. Thus, the reuse of the enzyme is very important to make the process with the immobilized enzyme economically viable.

According to the results from Table 1, the reuse of immobilized cellulase by adsorption and covalent bonding provided satisfactory results for pilling resistance and it did not affect the tensile strength of the samples. The decrease

in pilling resistance may be attributed to enzyme molecule loss due to kaolin particles incorporation on knitted fabric.

Kumar et al. [24] also reported a decrease in the pilling resistance of cotton fabrics when the immobilized cellulase on the epoxy resin was reused. However, in the above investigation, the enzyme reuse was the unique advantage provided by the immobilization, since the tensile strength of samples treated with free and immobilized cellulase was similar. Therefore, this research is very important, because, as far as we know, it is the first investigation that shows that it is possible to minimize the loss of tensile strength of fabrics and simultaneously promote the effective reuse of the enzyme.

This research shows that the characteristics of the substrate affect the reusability of immobilized enzymes. When the immobilized cellulase by covalent bonding and adsorption was used in biopolishing process (where the substrate is a piece of fabric, a solid substrate) about 84 and 80% of their initial activity after the third cycle was retained, respectively. In contrast, Lima [27] reported a superior activity retained (around 86% after the eighth hydrolysis cycle) using carboxymethylcellulose (CMC) as substrate. This difference may be attributed to the fact that CMC is a soluble substrate, which allows the immobilized enzyme to be recovered more efficiently, and therefore, greater activity is retained.

Dyeing properties of treated knitted fabric

Due to the incorporation of kaolin particles in the fabric, the samples were dyed to evaluate whether these particles interfere with the color properties of the dyed fabric. The presence of unwanted impurities can change the shade of a dyed fabric or hinder the absorption of solutions of dyes and chemicals causing uneven color. These problems are immediately visible and usually lead to rejection of the goods [34].

Table 2 shows the colorimetric coordinates of the dyed samples after biopolishing. All samples showed a color difference compared to the control sample (BPc). However, ΔE values less than 1.0 unit, as shown by the BPe and BPads samples, are unnoticeable to the human eye and it is an acceptable color match for the industries [34]. The results suggest there was no influence of the kaolin on the dyeing processes.

In contrast, the ΔE value presented by the BPcov sample represents a color difference noticeable for an experienced observer and may compromise the fabrics' acceptability [46]. Unexperienced observer notices color difference for ΔE values greater than 2.0 units [46]. The color difference of BPcov sample may be explained by the whiteness change observed for this sample. Therefore, the results suggest that functionalized kaolin particles had influenced on whiteness index and color dyeing properties of the knitted fabric.

Conclusions

Biopolishing is a technique that uses cellulase enzyme to reduce the pilling resistance of cotton fabrics. However, the use of free cellulase causes a significant reduction in the tensile strength of the treated fabrics. To overcome this problem, the current work proposed the use of immobilized cellulase by adsorption and covalent bonding. The results showed that the cotton fabric treated with the immobilized cellulase showed greater tensile strength and similar or superior pilling resistance to those treated with the free cellulase. Therefore, the above results suggest that immobilized cellulase provided adequate removal of the microfibrils causing less damage to knitted fabrics. Such findings may be attributed to the restriction of the hydrolysis of the immobilized cellulase to the surface of the cotton fibers. In addition to minimizing the damage of the knitted fabric, the immobilization also allowed to recover and reuse of the enzyme efficiently. The reuse is a significant feature, because it is associated with the economic feasibility of the process with immobilized enzymes. Immobilized cellulase on kaolin by

Table 2 Colorimetric coordinates of the dyed samples after biopolishing

Sample	L^*	a^*	b^*	ΔE^*ab	Color
BPc	57.1	+43.3	− 7.9	–	
BPe	56.5 ± 0.01	+43.7 ± 0.5	− 7.9 ± 0.1	0.8 ± 0.3	
BPads	57.3 ± 0.1	+44.0 ± 0.01	− 8.3 ± 0.3	0.8 ± 0.001	
BPcov	58.8 ± 1.0	+42.6 ± 0.2	− 8.1 ± 0.1	1.9 ± 1.0	

adsorption showed greater potential for application in the biopolishing process, because it did not affect the whiteness index, as well as the dyeing properties of the knitted fabric. Moreover, immobilization by adsorption is simpler, cheaper, and faster than immobilization by covalent bonding, since it is unnecessary to functionalize and activate the support. Finally, this paper is a relevant contribution to biopolishing literature and to improve the process. Moreover, it showed another perspective to enzymes immobilization. Unlike most investigations that use immobilization intending to reduce the cost of the enzyme, in biopolishing the main objective of immobilization is to improve the quality of products.

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Declarations

Conflict of interest The authors declare that they have no conflict of interest.

References

1. Jegannathan KR, Nielsen PH (2013) Environmental assessment of enzyme use in industrial production—a literature review. J Clean Prod 42:228–240
2. Heikinheimo L (2002) *Trichoderma reesei* cellulases in processing of cotton. VTT Technical Research Centre of Finland, VTT Publications
3. Esfandiari A, Firouzi-Pouyaei E, Aghaei-Meibodi P (2014) Effect of enzymatic and mechanical treatment on combined desizing and bio-polishing of cotton fabrics. J Text Inst 105:1193–1202
4. Kirk O, Borchert TV, Fuglsang CC (2002) Industrial enzyme applications. Curr Opin Biotechnol 13:345–351
5. Aly A, Moustafa A, Hebeish A (2004) Bio-technological treatment of cellulosic textiles. J Clean Prod 12:697–705
6. Besegatto SV, Costa FN, Damas MSP, Colombi BL, De Rossi AC, de Aguiar CRL, Immich APS (2018) Enzyme treatment at different stages of textile processing: a review. Ind Biotechnol 14:298–307
7. Costa FN, Lima JS, Valério A, de Souza AA, de Oliveira D (2021) Utilization of montmorillonite in biostoning process as a strategy for effluent reuse. J Chem Technol Biotechnol 96:890–898
8. Galante YM, Formantici C (2003) Enzyme applications in detergency and in manufacturing industries. Curr Org Chem 7:1399–1422
9. Hauser PJ, Schindler W (2004) Finishing with enzymes: biofinishes for cellulose. Chemical finishing of textiles. Woodhead Publishing Ltd, Cambridge, England
10. Sheikh J, Bramhecha I (2019) Enzymes for green chemical processing of cotton. In: The impact and prospects of green chemistry for textile technology. Woodhead Publishing, Cambridge, UK
11. Ukponmwan J, Mukhopadhyay A, Chatterjee K (1998) Pilling. Text Prog 28:1–57
12. Chen X, Huang X (2004) Evaluating fabric pilling with light-projected image analysis. Text Res J 74:977–981
13. Andreaus J, Olekszyszen DN, Silveria MHL (2014) Processing of cellulosic textile materials with cellulase. In: Cellulose and other naturally occurring polymers. Research Signpost, Kerala, India, pp 11–19
14. Choudhury AR (2014) Sustainable textile wet processing: applications of enzymes. Roadmap sustainable textile and clothing. Springer, Singapore
15. Buschle-Diller G, Zeronian S, Pan N, Yoon M (1994) Enzymatic hydrolysis of cotton, linen, ramie, and viscose rayon fabrics. Text Res J 64:270–279
16. Uddin MG (2015) Effects of biopolishing on the quality of cotton fabrics using acid and neutral cellulases. Text Cloth Sustain 1:9
17. Sankarraj N, Nallathambi G (2018) Enzymatic biopolishing of cotton fabric with free/immobilized cellulase. Carbohydr Polym 191:95–102
18. Yu Y, Yuan J, Wang Q, Fan X, Ni X, Wang P, Cui L (2013) Cellulase immobilization onto the reversibly soluble methacrylate copolymer for denim washing. Carbohydr Polym 95:675–680
19. Yu Y, Yuan J, Wang Q, Fan X, Wang P, Cui L (2015) Noncovalent immobilization of cellulases using the reversibly soluble polymers for biopolishing of cotton fabric. Biotechnol Appl Biochem 62:494–501
20. Lenting H, Warmoeskerken M (2001) Guidelines to come to minimized tensile strength loss upon cellulase application. J Biotechnol 89:227–232
21. Šimić K, Soljačić I, Pušić T (2015) Application of cellulases in the process of finishing. Tekstilec 58:47–56
22. Yu Y, Yuan J, Wang Q, Fan X, Wang P, Cui L (2014) A promising approach for bio-finishing of cotton using immobilized acid-cellulase. Fibers Polym 15:932–937
23. Yu Y, Yuan J, Wang Q, Fan X, Wang P, Sun X (2013) Immobilization of cellulases on the reversibly soluble polymer E udragit S-100 for cotton treatment. Eng Life Sci 13:194–200
24. Kumar V, Meenakshisundaram S, Selvakumar N (2008) Conservation of cellulase enzyme in biopolishing application of cotton fabrics. J Text Inst 99:339–346
25. ABNT (1994) Textiles—determination of colour fastness to bleaching peroxide—method of test. ABNT, Sao Paulo
26. Lima JS, Costa FN, Bastistella MA, de Araújo PHH, de Oliveira D (2019) Functionalized kaolin as support for endoglucanase immobilization. Bioprocess Biosyst Eng 42:1165–1173
27. Lima JS, Boemo APSI, de Araújo PHH, de Oliveira D (2021) Immobilization of endoglucanase on kaolin by adsorption and covalent bonding. Bioprocess Biosyst Eng 44:1627–1637
28. ABNT (2009) ABNT NBR ISO 105–E06:2009—Textiles—tests for colour fastness Part E06: colour fastness to spotting: Alkali. ABNT, Sao Paulo
29. ABNT (2016) ABNT NBR ISO 13934-1:2016—Textiles—tensile properties of fabrics part 1: determination of maximum force and elongation at maximum force using the strip method. ABNT, Sao Paulo
30. ASTM (2015) ASTM E313-15—Standard practice for calculating yellowness and whiteness indices from instrumentally measured color coordinates. ASTM, West Conshohocken
31. ASTM (2007) ASTM D4970-07—Standard test method for pilling resistance and other related surface changes of textile fabrics: martindale tester. ASTM, West Conshohocken
32. Lima JS, Araújo PHH, Sayer C, Souza AAU, Viegas AC, de Oliveira D (2017) Cellulase immobilization on magnetic nanoparticles encapsulated in polymer nanospheres. Bioprocess Biosyst Eng 40:511–518
33. Miller G (1959) Determination of reducing sugar by DNS method. Anal Chem 31:426–428

34. Broadbent AD (2001) Basic principles of textile coloration. Society of Dyers and Colourists, West Yorkshire
35. Pecho OE, Ghinea R, Alessandretti R, Pérez MM, Della Bona A (2016) Visual and instrumental shade matching using CIELAB and CIEDE2000 color difference formulas. Dent Mater 32:82–92
36. Saravanan D, Vasanthi N, Ramachandran T (2009) A review on influential behaviour of biopolishing on dyeability and certain physico-mechanical properties of cotton fabrics. Carbohydr Polym 76:1–7
37. Montazer M, Harifi T (2018) Introduction: textile finishing. In: Nanofinishing of textile materials. Woodhead Publishing, United States
38. Choudhury AKR (2017) Various ecofriendly finishes. In: Principles of textile finishing. Woodhead Publishing, Duxford, UK
39. Khanjanzadeh H, Behrooz R, Bahramifar N, Gindl-Altmutter W, Bacher M, Edler M, Griesser T (2018) Surface chemical functionalization of cellulose nanocrystals by 3-aminopropyltriethoxysilane. Int J Biol Macromol 106:1288–1296
40. Mondal M, Islam M, Ahmed F (2018) Modification of cotton fibre with functionalized silane coupling agents vinyltriethoxysilane and aminopropyltriethoxy-silane. J Text Sci Eng 8:1–8
41. Pujiasih S, Masykur A, Kusumaningsih T, Saputra OA (2018) Silylation and characterization of microcrystalline cellulose isolated from Indonesian native oil palm empty fruit bunch. Carbohydr Polym 184:74–81
42. Rafieian F, Mousavi M, Yu Q, Jonoobi M (2019) Amine functionalization of microcrystalline cellulose assisted by (3-chloropropyl) triethoxysilane. Int J Biol Macromol 130:280–287
43. Jeon JG, Kim HC, Palem RR, Kim J, Kang TJ (2019) Cross-linking of cellulose nanofiber films with glutaraldehyde for improved mechanical properties. Mater Lett 250:99–102
44. Wu T, Du Y, Yan N, Farnood R (2015) Cellulose fiber networks reinforced with glutaraldehyde–chitosan complexes. J Appl Polym Sci 132:42375
45. Hopwood D, Allen C, McCabe M (1970) The reactions between glutaraldehyde and various proteins. An investigation of their kinetics. Histochem J 2:137–150
46. Mokrzycki WS, Tatol M (2011) Colour difference ΔE-a survey. Mach Graph Vis 20:383–411

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