IMPROVEMENT OF COTTON DYEING BY SIMULTANEOUS TREATMENT WITH CELLULASES

MAGDALENA FOGORASI, MIHAELA DOCHIA and MICHAELA DINA STANESCU

Aurel Vlaicu University, Elena Dragoi Str. 1-3, Arad, Romania
✉ *Corresponding author: M. D. Stanescu, stanescu@uav.ro*

In memoriam Acad. Bogdan C. Simionescu
(1948-2024)

The textile industry generates pollution due to the chemicals necessary in the different steps of textile processing. The dyeing step produces polluted wastewaters containing dyes, as well as other chemicals. Thus, solutions for remediation of these wastewaters have been searched. However, a better approach is to increase dye fixation on textile materials. Reactive dyes provide a strong bond to the textile material. This work studies the dyeing of knitted cotton fabric with a reactive dye. To improve the dye fixation, the process was performed in the presence of two commercial cellulases. Enzymatic processes are ecological and "green". Due to their hydrolytic effects, cellulases break down internal fiber bonds, enhancing the access of the dye to the reaction sites, thus improving dye fixation, as well as the color fastness to washing. The properties of the dyed cotton, *i.e.* weight loss and the color attributes, were also determined.

Keywords: bio-finishing, cotton knitted fabric, reactive dye, commercial cellulases, color measurement, color fastness to washing

INTRODUCTION

A set of proposals known as the "Green Deal", aiming to effectively decrease greenhouse gas emissions, reducing them by 55% until 2030, compared with 1990, was adopted by the European Commission (EC) on July 14th, 2021.[1] Its purpose is to make Europe the first climate-neutral continent by 2050.

The textile industry is one of the most polluting economic activities, generating pollutants across all the operations included in the flowchart, from raw material to the final product. The dyeing stage has been identified as the most hazardous step of the process because of the high volume of water required and of the substantial amount of chemicals used.[2]

Synthetic dyes used in textile dyeing are one of the pollutants.[3] The presence of unused dyes in the wastewaters results in diminished water transparency, inhibiting chlorophyll synthesis and subsequently disrupting the entire aquatic ecosystem. In addition, the dyes exhibit toxicity to aquatic organisms, which can be transferred to humans through the food chain. Due to bioaccumulation, synthetic dyes and their decomposition products may cause cancer and mutagenic transformations in humans.[4,5] The dye content in effluents was evaluated at 10-15%, explaining the concern about such hazard.[6] Attempts to use natural dyes instead of synthetic were not very successful. Several problems, such as availability, reproducibility, fixation, *etc*., were noticed.[7] The requirement of using metallic salts (mordants) for the fixation of natural dyes also generates toxicity.[8]

Research attention has been mostly directed to the remediation of wastewaters resulting from dyeing by using different physical, chemical or biological procedures.[9-14] However, a better approach would be the prevention of the presence of dyes in the wastewaters.

Therefore, procedures have been developed for enhancing the dye fixation on the textile materials, consequently diminishing as much as possible the content of dyes in the wastewaters. One direction for improving the dye fixation was the synthesis of reactive dyes. These dyes are linked to the textile materials by covalent bonds, formed by either nucleophilic substitution or

addition of the functional groups of the materials, such as hydroxyl (cotton) or amino (wool) to the reactive part of the dye.[15]

Another direction for achieving a better fixation was the improvement of the surface reactivity of the materials by different treatments with biopolymers, such as chitosan,[7,16-19] which may also be used for dye encapsulation.[20] The treatment of cotton with synthetic copolymers is another solution for better results in dyeing.[21,22] Before the dyeing process, the polymer may be linked to the reactive dye, leading to better dyeing bath exhaustion and fixation.[23] The addition of cationic starch may also improve cotton dyeing.[24] The cotton-dye affinity may also be modified by carbamation with urea[25] or by a layer-by-layer deposition of C_6-fluorocarbon resin, a hyper-branched polymer in a hydrocarbon matrix.[26] Treatments with plasma have also been examined.[27,28] The combination of cationization with plasma treatments improves the dyeing process.[29] A less polluting procedure for improving the cotton affinity to dyes may be enzymatic treatment. Thus, previously enzymatically pretreated cotton was dyed with natural dyes. A mixture of enzymes (protease, α-amylase, lipase and esterase) has been used for this process.[30] According to the results enzymatic treatment provides rapid dye adsorption.[31]

The present work outlines the results of dyeing cotton with reactive dyes in the presence of commercial cellulases. These experiments aimed to evidence the influence of enzyme addition on the dyeing process. The expected results should consist in a smoother surface and in no significant influence on the color attributes and color strength compared to the reference (conventional dyeing). According to our knowledge, such a procedure was generally avoided, the reason being the supposed deactivation of the enzyme by the dye.[32] Literature mentioned a paper studying the results of cellulase treatment concomitantly with the dyeing process, in a one bath procedure, confirming the severe deactivation of the enzyme by the reagents (alkali) necessary for the dye fixation.[33] Thus, in the present paper, the one bath approach (enzyme and dye) was investigated, the enzyme treatment being carried out prior to the alkali addition for dye fixation.

EXPERIMENTAL

Materials

Knitted cotton (100%, Interlock structure, weight per unit area 180 g/m^2), previously scoured and bleached, was used in the study. A reactive triazinic dye, namely, Procion blue HEGN, produced by DyStar, was selected. The cellulases used were a neutral cellulase IndiAge Neutra L and an acid cellulase IndiAge 2XL, both supplied by Genencor, Denmark. IndiAge 2XL was designed for biofinishing of cotton, the optimal parameters for application being pH 4.5-5.5 and temperature 55-60 °C. The IndiAge Neutra L, usually applied for finishing denim products, has the following optimal values: pH 6-8 and temperature 45-55 °C.[34]

Methods

Dye structure

The dye structure was confirmed by the FT-IR spectra obtained on a Bruker Vertex 70 spectrophotometer (Bremen, Germany), equipped with an ATR cell, in the 600-4000 cm^{-1} wavelength range, with a resolution of 4 cm^{-1}. Acquisition of data, normalization, and the baseline correction were achieved with OPUS 6.5 software.

Cotton dyeing

The batch dyeing procedure was used. The dyeing was performed simultaneously with the enzymatic treatment, by bath exhaustion. Blank experiments, without enzymes, were carried out for comparison. For dye fixation, the necessary alkaline pH was obtained by the addition of Na_2CO_3 and NaOH. The dyeing process was accomplished under the following conditions: 1% o.w.f. (*on weight of fabric*) dye, 40 g/L NaCl, 5 g/L Na_2CO_3, 2 g/L NaOH, 1:20 fabric to liquor ratio, following the dyeing prescription for triazinic reactive dyes.[35] The dyeing process was performed as follows:

- *Step I*: cotton, dye, water and enzyme were introduced in the dyeing bath and maintained, with stirring, for 20 min, at 40 °C. During this period, the enzyme acts on the cotton surface, and the dye is adsorbed.
- *Step II*: the temperature was elevated to the optimal value for the enzymatic activity during 10 min – 50 °C for IndiAge Neutra L and 55 °C for IndiAge 2XL, respectively. The bath was kept at this optimal temperature for t minutes (t_1 = 20, t_2 = 30 and t_3 = 45).
- *Step III*: an increase in temperature at 80 °C was performed, with the addition in stages of NaCl, Na_2CO_3 and NaOH, facilitating the reaction of the dye with the cotton substrate and the deactivation of the enzyme (Fig. 1). The dyeing bath was maintained at this temperature for 30 minutes.

After dyeing, the cotton was removed from the dyeing bath, washed with warm and cold water, and soaped for 30 minutes with Cotoblanc NSR for 30 minutes at a 1:30 fabric to liquor ratio, and further rinsed with warm and cold water, and then air-dried.

For comparison, blank experiments, without enzyme, were performed for each enzymatic experiment, respecting temperature and time conditions.

Dyed cotton properties

To evidence the influence of the enzyme on the dyeing process, several characterization tests have been performed. The weight loss due to enzyme treatment was determined using a Mettler Toledo high-precision balance. The color attributes: brightness, chroma (purity or intensity of color), and hue were investigated by using a Datacolor 500 spectrophotometer, equipped with Datacolor Tools 2.0 software based on the CIELAB equation. The color fastness to washing was evaluated under domestic and commercial laundering conditions, according to ISO 105-C08:2010.[36]

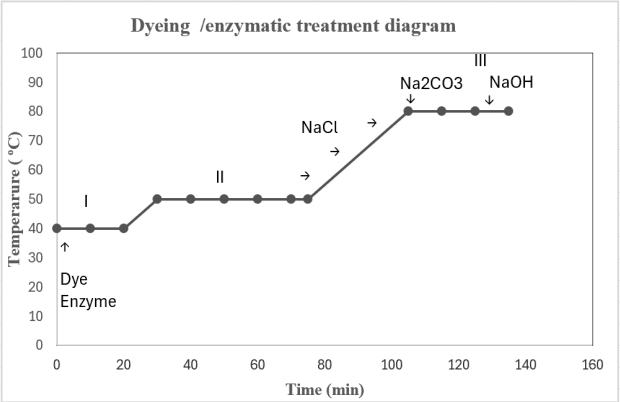

Figure 1: Diagram of cotton dyeing

RESULTS AND DISCUSSION

Chemical structure of the dye and its fixation on cotton

A reactive dye was chosen due to its higher stability, as it is fixed by covalent bonds to the textile material.[37] The dye used belongs to the class of reactive triazine derivatives. Such dyes have a complex linear structure and link to cotton materials through covalent bonds formed by the nucleophilic substitution of the chlorine atoms on the triazine ring[38,39] (Fig. 2).

The dye used is Procion blue HEGN, according to Color Index CI Reactive blue 198 (see Fig. 3). The dye chromophore is a conjugated triphenodioxazine ring.[40,41] It contains two chlorine atoms, linked to two triazine moieties, which the reactive oxygen from cellulose may substitute (Fig. 3).

The proposed structure was confirmed by the FT-IR spectrum (Fig. 4), the peaks being assigned according to literature.[42] The dye has the following specific vibrations: a broad peak around 3411 cm^{-1}, which was assigned to N-H vibration, the peaks at 2956 cm^{-1} and 2930 cm^{-1}, assigned to the C-H symmetric stretching and C-H asymmetric stretching of CH_2 groups, respectively, the bands at 1603 cm^{-1} combine phenyl ring vibrations with stretching of the C=N group.

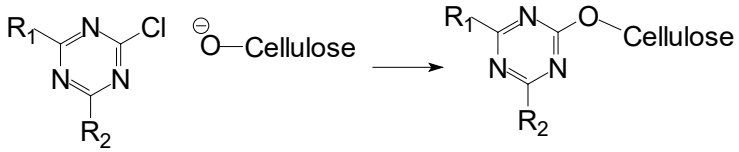

Figure 2: Fixation of reactive dye with the triazine moiety to cellulosic materials

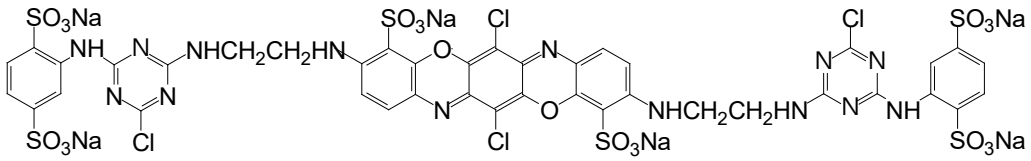

Figure 3: Chemical structure of CI Reactive blue 198

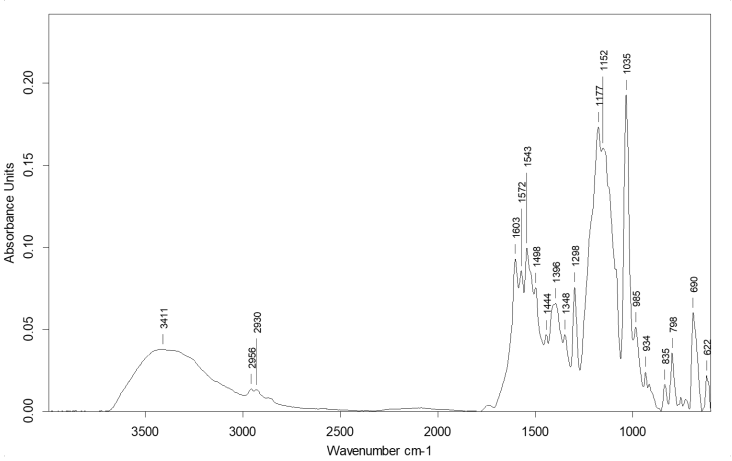

Figure 4: FTIR spectrum of CI Reactive blue 198

The bands at 1543 cm^{-1} and 1177 cm^{-1} are associated with the triphenodioxazine chromophore of the dye. The peak at 1152 cm^{-1} represents the symmetric vibrations of the sulfonate groups, while the peaks at 1035 cm^{-1} and 796 cm^{-1} account for the C-Cl bonds.

Role of cellulases in the dyeing process

The enzyme was applied concomitantly with the dye, being introduced into the dyeing bath at the beginning of the process.

Cellulases are enzymes that fragment cellulose.[43] These enzymes are largely used having numerous applications.[44,45] They improve the aspect of the material surface by polishing or softening, as well as by removing white specks (bundles of surface fibers that are not dyed).[46] Based on the positions in the chain where this fragmentation occurs, there are: exo-cellulases [cellobiohydrolase (EC 3.2.1.91) and cellodextrinase (EC 3.2.1.74)], acting at the ends of the polymer, endo-cellulase (EC 3.2.1.4), hydrolysing the amorphous part inside the chain, and β-glucosidases (EC 3.2.1.21), hydrolyzing cellobiose to glucose.[47,48] The cellulose fragmentation is performed by an acid-base catalysis[49,50] and depends on the binding of the enzyme to the cellulose substrate.[51,52]

The IndiAge enzymes used are depleted in exo-glucanase and enriched in endo-glucanase activities.[53,54] IndiAge enzymes have no binding domain, consequently being less adsorbed on the fiber surface.[52] These enzymes are genetically modified products.[55] IndiAge 2XL, an acid cellulase, was obtained from *Trichoderma reesei*,[52] and IndiAge Neutra from species of alkaliphilic *Streptomyces* sp. isolated from Lake Nakuru in Kenya.[56]

To investigate the enzyme influence on the dyeing process, different quantities of the commercial cellulases have been employed. For the acid cellulase, the experimental quantities tested were as follows: 0.7%, 1%, 1.2% and 1.4% o.w.f., while for the neutral cellulase, these were: 0.2%, 0.5%, 0.8% and 1.0% o.w.f.

The enzymes act during the period of dye adsorption by the cotton fibre. The cotton dye adsorption is a physical process due to the electron-rich sites of the dye (oxygen, nitrogen, sulphonic moieties, *etc*.) that may connect by hydrogen bonds to the cotton cellulose.[57]

One consequence of the enzyme treatment is the weight loss of the textile material, due to the water-soluble carbohydrate resulting from cellulose hydrolysis. The experimental results are presented in Figure 5. As expected, the weight loss increased with increasing treatment time. However, the values of the weight loss are small, ranging from 0.17 to 1.5% for IndiAge Neutra L, and 0.9 to 2.5% for IndiAge 2XL. These results confirm the reduced percentage of exo-cellulases in the composition of the employed commercial enzymes, enzymes which generate soluble carbohydrates.

The enzymatic treatment facilitates the dyeing process by the dissolution of the hydrogen bonds from cellulose polymer chains, increasing the accessibility of the dye to the textile substrate.[58] The fragmentation by hydrolysis of the cellulose polymer, due to the endo-Glucanase activity of the applied enzymes, generates more reactive glycosidic OH, facilitating the dye fixation.[59] Thus, the enzyme treatment results in better fixation of the dye to the cotton substrate. The properties of the dyed cotton support this conclusion.

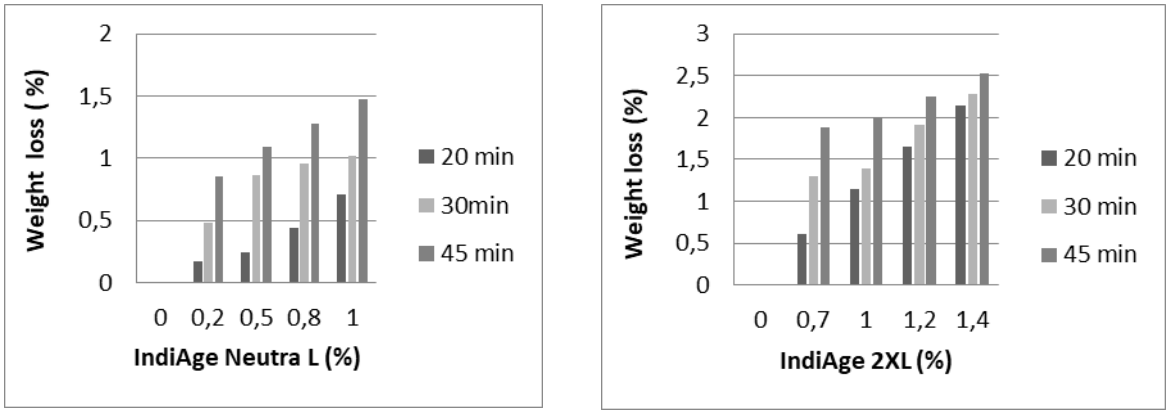

Figure 5: Weight loss of samples treated with (a) IndiAge Neutra L, and (b) IndiAge 2XL

Color measurements

The effect of the enzymatic treatment on the material's behaviour during dyeing was examined. The color attributes – brightness, chroma, and hue – were calculated and plotted in the CIE Lab color diagram (Figs. 6 and 7).

Furthermore, an important index reflecting the surface color depth is the K/S value. The determination of K/S values was performed using the Kubelka–Munk equation:[60]

$$K/S = (1-R)^2/2R \quad (1)$$

where R = reflectance at the dye's maximum wavelength of absorption; K = absorption; S = scattering coefficients of the dyed cotton sample.

The resulted K/S (color strength) and ΔE (color difference), which measure the color accuracy, are presented in Tables 1 and 2, respectively. For further analysis, among all the samples subjected to the simultaneous enzymatic and dyeing procedures, we considered optimal those with K/S values similar to or higher than the reference sample values. Also, the color difference should fall within the tolerance limit, considered to range from 0 to 2.[61]

Among all tested enzyme concentrations (for 20 min treatment time), the sample subjected to 0.2% IndiAge Neutra L yields an enhanced K/S compared to the reference, along with a lower color difference (ΔE = 1.014). Considering a 30-minute treatment period and all evaluated enzyme concentrations, the samples treated with 0.2% and 1.0% IndiAge Neutra L exhibit a lower colour difference (ΔE< = 1) and a stronger color [K/S] than the reference. When examining all the enzyme concentrations and the 45-minute treatment period, the sample that was exposed to 0.2% IndiAge Neutra L produced a lower color difference (ΔE = 0.310) and a decreased K/S in comparison to the reference. Although the samples treated with superior enzyme concentration yielded better color strength, the color difference exceeded the upper limit for ΔE and consequently are not recommended.

Thus, the proper parameters for Step II of the dyeing process with IndiAge Neutra L are as follows: 0.2% enzyme concentration for 20 or 30 minutes at 50 °C, as well as 1.0% enzyme for 30 minutes at 50 °C (see Table 1).

Concerning the color properties of the samples dyed in the presence of IndiAge Neutra L (Fig. 6), the following observations can be made:

- After 20 minutes of treatment, modifications in lightness, hue, and chroma were observed related to the untreated sample. There was a shift towards the blue region with chroma enhancement, except for the experiments with 1.0% enzyme o.w.f. This sample is lighter and less rich in chroma when compared to the reference, which aligns with the K/S value.
- The values calculated for 30 minutes and the color diagram a^*b^* reveal a blue displacement for all samples, with no significant changes in chroma. The color difference ΔE = 1.637 for the sample treated with 0.8% o.w.f. can be ascribed to the lightness enhancement.
- For the treatment time of 45 minutes, a notable decrease in lightness for the experiments with 0.5, 0.8 and 1.0% enzyme was observed, also reflected in a visible increase of the color depth. Changes in chroma were also noticed. These samples are more saturated in blue. Accordingly, the

color differences are outside the tolerance limit.

The data collected for the samples treated with IndiAge 2XL were presented in Table 2 and Figure 7. Within all tested enzyme concentrations and a 20-minute treatment duration, the sample treated with 1.2% IndiAge 2XL presents a similar color strength [K/S] with the reference, alongside an insignificant color difference (ΔE = 0.25). Even if the samples treated for 30 minutes with all enzyme amounts reached a higher color strength compared to the reference, they are not considered optimal because the color differences ΔE are outside the tolerance limit. Among the enzyme concentrations analysed during the 45-minute treatment period, the sample treated with 1.0% and 1.2% IndiAge 2XL exhibited lower color difference (ΔE < 1) and color strength relative to the reference sample. Thus, for Step II of the one bath dyeing with IndiAge 2XL, the optimal parameters are: 1.2% enzyme for 20-minute and 1% and 1.2% enzyme for 45 min, at 55 °C.

Regarding the color attributes, for the samples dyed in the presence of IndiAge 2XL (Fig. 7), the following observations have to be mentioned:

- For a 20 min treatment time, a decrease in chroma and an increase in lightness could be noticed. Generally, the dominant wavelength is situated in the blue region for all enzyme concentrations. The lower surface dye uptake and less saturated color of the samples, compared to the standard one, is also confirmed by the K/S values.
- For 30 minutes, the color diagram a*b* reveals a blue displacement for all the samples, and an increase of chroma in blue also appears. A decrease in lightness was also observed, which generates a considerable color difference between the samples and the standard. These results are in agreement with the measured K/S values (see Table 2).
- For 45 minutes, differences in the chroma and hue value were measured for all the enzyme-treated samples. No significant changes in lightness have occurred, and the color diagram a*b* reveals a green displacement for all the samples and, generally, a decrease in chroma.

Color fastness to washing

The color fastness to washing was evaluated according to ISO 105-C08:2010,[62] for the samples that showed the best results in terms of color differences and color strength. The results are presented in Tables 3 and 4.

Except for the sample treated for 45 minutes with 0.2% IndiAge Neutra L, the samples dyed in the presence of IndiAge Neutra L have superior washing color fastness compared to the reference. Also, according to the experimental data, the combined dyeing and enzymatic treatment with IndiAge 2XL provides a better washing color fastness compared to the reference.

We may conclude that the optimal enzymatic treatments in the dyeing bath provide better dye fixation on cotton, reducing the dye pollution generated by washing the products made from enzyme-treated materials, compared with untreated ones.

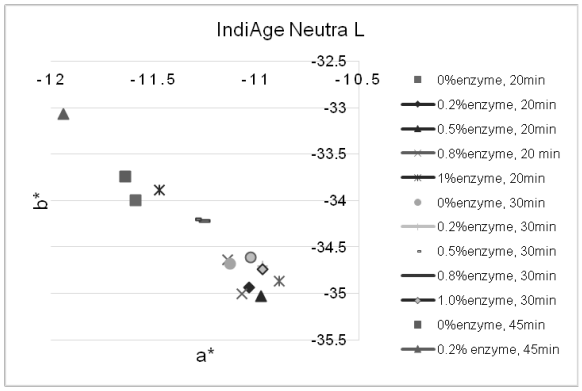

Figure 6: a*b* diagram for dyed cotton samples treated with IndiAge Neutra L

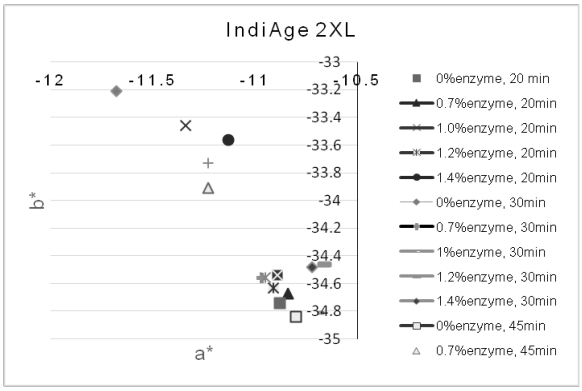

Figure 7: a*b* diagram for dyed cotton samples treated with IndiAge 2XL

Table 1
ΔE and K/S values for dyed samples treated with IndiAge Neutra L

Treatment time (min)	Enzyme (%)	ΔE	K/S
20	0	-	4.5448
	0.2	1.014	4.8851
	0.5	2.486	4.9383
	0.8	1.924	4.7894
	1	1.322	4.2143
30	0	-	4.7867
	0.2	0.151	4.9133
	0.5	1.409	4.4237
	0.8	3.913	4.2479
	1	0.589	4.9853
45	0	-	3.9383
	0.2	0.310	3.7459
	0.5	2.799	4.8718
	0.8	2.664	4.9304
	1	2.211	4.844

Table 2
ΔE and K/S values for dyed samples treated with IndiAge 2XL

Treatment time (min)	Enzyme (%)	ΔE	K/S
20	0	-	5.004
	0.7	0.73	4.73
	1	2.21	4.298
	1.2	0.25	4.995
	1.4	2.02	4.317
30	0	-	4.125
	0.7	2.62	4.849
	1	3.06	4.921
	1.2	3.01	4.898
	1.4	2.48	4.692
45	0	-	5.092
	0.7	1.32	4.785
	1	0.64	4.882
	1.2	0.62	4.921
	1.4	2.13	4.406

Table 3
Color fastness to washing for samples treated with IndiAge Neutra L

Enzyme (%)	Time (min)	ΔE	Color fastness to washing
0	20	1.72	4
0.2	20	1.09	4/5
0	30	1.02	4/5
0.2	30	1.05	4/5
1.0	30	0.99	4/5
0	45	1.44	4
0.2	45	2.12	3/4

Table 4
Color fastness to washing for samples treated with IndiAge 2XL

Enzyme (%)	Time (min)	ΔE	Color fastness to washing
0	20	2.18	3/4
1.2	20	1.17	4/5
0	45	1.57	4
1.0	45	1.75	4
1.2	45	1.86	4

CONCLUSION

The experimental data confirm the potential of performing dyeing and enzymatic treatment in a single-step process, thereby enhancing efficiency in time and reducing water and energy consumption. The enzymatic treatment has to be performed before the dye fixation step, when alkali is added and the enzymes are inactivated. By breaking the hydrogen bonds between the neighboring cellulose polymer chains, cellulases increase the contact surface of the dye with the textile material, leading to better dye fixation.

The weight loss of the textile material due to the enzymes used is low, confirming the small content of exo-cellulases in the composition of the two commercial enzymes. Also, differences in lightness, hue and chroma were noticed. The changes can be ascribed to the varying degrees of fuzziness between the control and treated samples, resulting in an increased level of reflection. Except for a few cases, the color of the treated samples fits in the established tolerance limits. The type and the duration of the enzymatic treatment are important, the optimal conditions being specified for each of the two commercial enzymes.

The improved values of color fastness to washing, when the optimal conditions of enzymatic treatments were applied, confirm the superior dye fixation in these experiments. The application of enzymatic treatment to the dyeing bath gave positive results in the described optimal conditions, being a sustainable procedure for dyeing of cotton with reactive dyes.

REFERENCES

[1] European Commission, Delivering the European Green Deal (europa.eu) https://ec.europa.eu/info/strategy/priorities2019-2024/european-green-deal/delivering-european-green-deal_en

[2] E. Conde Miranda, P. Gabriel Prado and C. León-Velarde, *Text. Leather Rev.*, **7**, 88 (2024), https://doi.org/10.31881/TLR.2023.165

[3] J. Sharma, S. Sharma and V. Soni, *Reg. Stud. Mar. Sci.*, **45**, 101802 (2021), https://doi.org/10.1016/j.rsma.2021.101802

[4] S. Mehra, M. Singh and P. Chadha, *Toxicol. Int.*, **28**, 165 (2021), https://doi.org10.18311/ti/2021/v28i2/26798

[5] S. Dutta, S. Adhikary, S. Bhattacharya, D. Roy, S. Chatterjee *et al.*, *J. Environ. Manage.*, **353**, 120103 (2024), https://doi.org/10.1016/j.jenvman.2024.120103

[6] M. Yusufm, in "Textiles and Clothing: Environmental Concerns and Solutions", edited by M. Shabbir, Scrivener Publishing LLC, 2019, pp. 11–26, https://doi.org/10.1002/9781119526599.ch2

[7] S. S. Affat, *UTsci*, **8**, 130 (2021), https://jsci.utq.edu.iq/index.php/main/article/view/790

[8] M. Verma, N. Gahlot, S. S. J. Singh and N. M. Rose, *Cellulose Chem. Technol.*, **55**, 343 (2021), https://doi.org/10.35812/CelluloseChemTechnol.2021.55.33

[9] S. A. S. Chatha, M. Asgher and H. M. N. Iqbal, *Environ. Sci. Pollut. Res.*, **24**, 14005 (2017), https://doi.org/10.1007/s11356-017-8998-1

[10] P. Singh, B. Mohan, V. Madaan, R. Ranga, P. Kumari *et al.*, *Environ. Sci. Pollut. Res.*, **29**, 69294 (2022), https://doi.org/10.1007/s11356-022-22550-7

[11] A. E. John, D. Mishra, S. T. Salammal and M. A. Khan, *Sustain. Water Resour. Manag.*, **10**, 105 (2024), https://doi.org/10.1007/s40899-024-01074-7

[12] M. Shabir, M. Yasin, M. Hussain, I. Shafiq, P. Akhter *et al.*, *J. Ind. Eng. Chem.*, **112**, 1 (2022), https://doi.org/10.1016/j.jiec.2022.05.013

[13] B. M Adesanmi, Y.-T. Hung, H. H Paul and C. R Huhnke, *GSCARR*, **10**, 126 (2022), https://doi.org/10.5281/zenodo.6331586

[14] N. El Messaoudi, M. El Khomri, A. El Mouden, A. Bouich, A. Jada *et al.*, *Biomass Convers. Bioref.*, **14**, 11739 (2022), https://doi.org/10.1007/s13399-022-03604-9.hal-04298171

[15] D. M. Lewis in "Handbook of Textile and Industrial Dyeing Principles, Processes and Types of Dyes", edited by M. Clark, Woodhead Publishing Series in Textiles, Woodhead Publishing, 2011, vol. 1, pp. 303-364, https://doi.org/10.1533/9780857093974.2.301

[16] M. Abd El-Moneim Ramadan, S. Samy, M. Abdulhady and A. A. Hebeish, in "Natural Dyes"

edited by E. P. Akcakoca Kumbasar, InTech, 2011, https://doi.org/10.5772/20097
[17] A. Rehman, K. Iqbal, F. Azam, F. Safdar, M. Ashraf *et al*., *J. Text. Inst*., **112**, 1208 (2020), https://doi.org/10.1080/00405000.2020.1805952
[18] B. Ashenafi, H. Berhane, H. Gashawbeza, M. Tesfaye and A. Dessie, *J. Text. Eng. Fashion Technol*., **6**, 37 (2020), https://doi.org/10.15406/jteft.2020.06.00224
[19] M. Verma, S. S. J. Singh and N. M. Rose, *Cellulose Chem. Technol*., **56**, 165 (2022), https://doi.org/10.35812/CelluloseChemTechnol.2022.56.16
[20] E. M. Costa, S. Silva, M. Veiga, P. Baptista, F. K. Tavaria *et al*., *Carbohyd. Polym*., **251**, 117 (2021), https://doi:10.1016/j.carbpol.2020.117120
[21] L. Tsimpouki, K. Papapetros, C. A. Anastasopoulos, L. Sygellou, A. Soto-Beobide *et al*., *Cellulose*, **30**, 6031 (2023), https://doi.org/10.1007/s10570-023-05220-w
[22] T. Niu, X. Wang, C. Wu, D. Sun, X. Zhang *et al*., *ACS Omega*, **5**, 15409 (2020), https://doi.org/10.1021/acsomega.0c01530
[23] Z. Zhang, H. Wang and J. Sun, *J. Appl. Polym. Sci*., **138**, e50455 (2021), https://doi.org/10.1002/app.50455
[24] S. Ali, M. A. Mughal, U. Shoukat, M. A. Baloch and S. H. Kim, *Carbohyd. Polym*., **117**, 271 (2015), http://dx.doi.org/10.1016/j.carbpol.2014.09.064
[25] N. Muhammed and N. Govindan, *J. Nat. Fibers*, **19**, 1402 (2020), https://doi.org/10.1080/15440478.2020.1776659
[26] S. S. Ugur, *Coatings*, **13**, 1129 (2023), https://doi.org/10.3390/coatings13061129
[27] A. G. Demir, F. R. Oliveira, T. Gulumser and A. P. Souto, in *Procs. 18th World Textile Conference* (AUTEX 2018), Istanbul, June 20–22, 2018, *IOP Conf. Ser.: Mater. Sci. Eng*., **460** 012026, https://doi.org/10.1088/1757-899X/460/1/012026
[28] M. H. Ullah, H. Akther, M. M. Rahman, A. B. M. Foisal, M. M. Hasan *et al*., *Heliyon*, **7**, e07893 (2021), https://doi.org/10.1016/j.heliyon.2021.e07893
[29] J. Correia, K. Mathur, M. Bourham, F. Ribeiro Olivera, R. De Cassia Siqueira Curto Valle *et al*., *Cellulose*, **28**, 9971 (2021), https://doi.org/10.1007/s10570-021-04143-8
[30] P. S. Vankar and R. Shanker, *Desalination*, **230**, 62 (2008), https://doi.org/10.1016/j.desal.2007.11.016
[31] P. S. Vankar, R. Shanker and A. Verma, *J. Clean. Prod*., **15**, 1441 (2007), https://doi.org/10.1016/j.jclepro.2006.05.004
[32] S. Vieira Besegatto, F. Nunes Costa, M. S. Pascoal Damas, B. L. Colombi, A. C. De Rossi *et al*., *Ind. Biotechnol*., **14**, 298 (2018), https://doi.org/10.1089/ind.2018.0018
[33] P. Wang, Y. Hong, Y. Ding, L. Cui, J. Yuan *et al*., *Fibres Text. East Eur*., **23**, 109 (2015)
[34] Genencor Company, "Genencor Textile Product List", 2000
[35] A. Grigoriu, I. Alexandrescu, R. Bidalach, M. Cernat and M. Dobrovăţ, "Indrumar teoretic şi practic pentru vopsirea materialelor textile", Ed. Certex, 1994, pp. 122-124
[36] ISO 105-C08 Textiles-Tests for colour fastness Part C08: Colour fastness to domestic and commercial laundering using a non-phosphate reference detergent incorporating a low-temperature bleach activator, (2010)
[37] J. Koh, in "Handbook of Textile and Industrial Dyeing Principles, Processes and Types of Dyes", edited by M. Clark, Woodhead Publishing, vol. 2, 2011, pp, 129-146, https://doi.org/10.1533/9780857094919.1.129
[38] O. A. Stamm, *J. Soc. Dyers Colour*. **80**, 416 (1964), https://doi.org/10.1111/j.1478-408.1964.tb02603.x
[39] J. Cui, L. Jieqiong, C. W. Xia, Z. Jianwei, H. Xincheng *et al*., *Dyes Pigments*, **204**, 110392 (2022), https://doi.org/10.1016/j.dyepig.2022.110392
[40] V. C. Almeida, W. F. Costa, J. Nozaki and C. C. Oliveira, *Anal. Sci*., **22**, 445 (2006), https://doi.org/10.2116/analsci.22.445
[41] S. A. Khan, S. B. Khan, L. U. Khan, A. Farooq, K. Akhtar *et al*., in "Handbook of Materials Characterization", edited by S. K. Sharma, Springer, Cham, 2018, pp. 317-344, https://doi.org/10.1007/978-3-319-92955-2_9
[42] W. A. Khattak, M. Ul-Islam, M. W. Ullah, S. Khan and J. K. Park, in "Polysaccharides Bioactivity and Biotechnology", edited by K. Ramawat and J. M. Mérillon, Springer, Cham, 2015, pp. 535-579, https://doi.org/10.1007/978-3-319-16298-0_55
[43] R. Ranjan, R. Rai, S. B. Bhatt and P. Dhar, *Biochem. Eng. J*., **198**, 109020 (2023), https://doi.org/10.1016/j.bej.2023.109020
[44] V. J. Rodrigues and A. A. Odaneth, in "Current Status and Future Scope of Microbial Cellulases", edited by D. K. Tuli and A. Kuila, Elsevier, 2021, pp 189-209, https://doi.org/10.1016/B978-0-12-821882-2.00007-7]
[45] M.-A. Rousselle, N. R. Bertoniere, P. S. Howley, J. Pere and J. Buchert, *Text. Res. J*., **73**, 921 (2003), https://doi.org/10.1177/004051750307301013
[46] N. Bhardwaj, B. Kumar, K. Agrawal and P. Verma, *BIOB*, **8**, 95 (2021), https://doi.org/10.1186/s40643-021-00447-6
[47] U. Ejaz, M. Sohail and A. Ghanemi, *Biomimetics*, **6**, 44 (2021), https://doi.org/10.3390/biomimetics6030044
[48] S. B. A. Hamid, M. M. Islam and R. Das, *Cellulose*, **22**, 2157 (2015), https://doi.org/10.1007/s10570-015-0672-5
[49] M. D. Stanescu, *Cellulose Chem. Technol*., **57**, 1 (2023), https://doi.org/10.35812/CelluloseChemTechnol.2023.57.01
[50] Q. Liu, M. Madadi, S. Al Azad, C. Sun, E. Zhang *et al*., *Bioresour. Technol*., **416**, 131756 (2025), https://doi:10.1016/j.biortech.2024.131756

[51] D. Dienes, A. Egyházi, K. Réczey, É. Polyánszky, S. Hernádi *et al*., in *Procs*. *8th International Conference on Biotechnology in the Pulp and Paper Industry*, June 4-8, 2001, Helsinki, pp 247-248
[52] S. Cheroni, B. Gatti, G. Margheritis, C. Formantici, L. Perrone *et al*., *Int*. *Biodeter*. *Biodegr*., **69**, 106 (2012), https://doi.org/10.1016/j.ibiod.2011.06.009
[53] A. Suurnäkki, M. Tenkanen, M. Siika-aho, M.-L. Paavola, L. Viikari *et al*., *Cellulose*, **7**, 189 (2000), https://doi.org/10.1023/A:1009280109519
[54] A. Dadwal, S. Sharma and T. Satyanarayana, *Front*. *Microbiol*., **11**, 1387 (2020), https://doi.org/10.3389/fmicb.2020.01387
[55] B. E. Jones, in "Halophilic Microorganisms", edited by A. Ventosa, Springer, 2004, pp. 275-284, https://doi.org/10.1007/978-3-662-07656-9_20
[56] C. V. Stead, *J*. *Chem*. *Technol*. *Biotechnol*., **38**, 55 (1987), https://doi.org/10.1002/jctb.280380106
[57] D. Ciolacu, J. Kovac and V. Kokol, *Carbohyd*. *Res*., **345**, 621 (2010), https://doi.org/10.1016/j.carres.2009.12.023
[58] M. Miljković, "Carbohydrates Synthesis, Mechanism and Stereoelectronic Effects", Springer, 2010, pp. 113-142, https://doi.org/10.1007/978-0-387-92265-2_5
[59] S. Landi, I. Rocha Segundo, E. Freitas, M. Vasilevskiy, J. Carneiro *et al*., *Solid State Commun*., **341**, 114573 (2022), https://doi.org/10.1016/j.ssc.2021.114573
[60] W. S. Mokrzycki and M. Tatol, *MGV*, **20**, 383 (2011)
[61] ISO 105-C08 Textiles-Tests for colour fastness Part C08: Colour fastness to domestic and commercial laundering using a non-phosphate reference detergent incorporating a low-temperature bleach activator, https://www.iso.org/standard/51574.html