ICTSE-2022
BUTEX

A Qualitative Testing Method for Assessing Enzymatic Biopolishing Effect on Textile Substrate

Shohag Chandra Das[1], Mohammad Abbas Uddin[1]*
[1]Department of Dyes and Chemical Engineering, Bangladesh University Of Textiles, Dhaka, Bangladesh.

Abstract

A qualitative testing methodology has been developed to assess the enzymatic biopolishing effect on textile substrates. The system divided the assessment into five grades: α-Alpha, βγ-Beta, γ-Gamma, Θ-Theta and δ-Delta to be meant as Not Satisfactory, Satisfactory, Good, Very Good and Bad, respectively, depending on the amount of surface fibre removed after treatment. A stereo microscope has been used to analyse the surface of folded fabric and single yarn. Two different fabrics (scoured and bleached single jersey and black rib fabric) have been biopolished with three different commercial enzymes - Acid cellulase enzyme, Biopolish enzyme -880 and Azypolish-AC enzyme in a similar condition and finally graded according to this developed grading system. The grading system is an easier and potential way of assessing effect of biopolishing on textile materials.

Keywords— Biopolishing, cellulase enzyme, test method, textiles, bioprocessing, green chemistry

1. Introduction

Textiles are subjected to various physical, chemical and biological treatments for various purposes, particularly to impart certain functionalities [1]. The protruding fibres present on the surface of yarn or fabric make the fabric less absorbable and cause a fuzzy appearance, hairiness, pilling, bad hand feel, poor colour, etc. [2]. The introduction of enzymes in treating textile material was developed in the 19th century [3]–[5]. Among them, most of them are hydrolase enzymes. Biopolishing is a biological method of reducing protruding fibres from the surface of the fabric or yarn to make the product more aesthetic and functional, which is environmentally friendly [6] and different cellulase enzymes are used for the process, which can be aerobic, anaerobic, mesophilic and thermophilic [7]. The treatment can be carried out before, during, or after dyeing with a view to different objectives [3], [8]. Still, if carried out before dyeing, it greatly impacts the dyeability of the fabric [9].

Although singeing is an alternative to biopolishing, the quality of the outcome is not praiseworthy and can reduce the strength of the fabric or yarn. In biopolishing, a specific type of cellulase enzyme attacks the protruding fibres and breaks the hairy fibres from the main body of the yarn/fabric surface [10]. The dimension of the enzyme is very tiny, about 60Å and as a result, it has been easier for the enzyme molecule to penetrate the fibre [11]. The enzymes can be produced from different bacteria sources like *Trichoderma reesei [12], Trichoderma viride, Aspergilluas niger [13], Hypocrea jecorina [14], Acanthophysium sp. KMF001 [15], Alkalothermophilic Thermomonospora sp. [16], Humicola insolens, Bacillus licheniformis KM999221 [17]*, etc.

The basic mechanism is that the cellulase enzyme attacks the β-(1, 4) glycosidic linkage of the cellulose and breaks it down by three possible cellulase enzymatic system - endoglucanases (EC 3.2.1.4), exoglucanases (EC 3.2.1.91) and β-glucosidases/cellobiases (EC 3.2.1.21) [1], [4], [10], [18]. Endoglucanases hydrolyse the β-1,4-glycosidic linkages of the cellulose chain, randomly producing new cellulosic chain ends; exoglucanases attack on the reducing and non-reducing ends of the cellulose chain producing cellobiose units and cellobiase hydrolyzes cellobiose to glucose units [16], [19], [20]. However, this process can affect both the surfaces of the yarn and the chemical structure of fibre, thus affecting the tensile strength [21], [22].

There are different ways to check the performance of the biopolishing effect on fabric, such as visual assessment, hand feel assessment, weight loss % (ranging from 1.7% to 19.7%) [23], reflectance value and whiteness degree [24], pilling test, abrasion weight loss, water absorbance tests [25], hairiness amount [26]. However, all these methods are mostly subjective, depend on the judgment of the assessor and are not specific toward biopolishing effects on fabric after treatment.

Therefore, to assess the biolpolishing effect on textiles, in this study, a qualitative grading system has been developed, dividing the value into five (5) grades depending on the surface analysis/view. Both fabric and individual yarn have been assessed for the evaluation system. Finally, the system has been used to evaluate ready to dye fabric (RFD) and dyed cotton fabric treated with three different acid cellulase enzymes.

2.Materials and methods

2.1. Materials

2.1.1 Fabric: 100% cotton fabric (woven or knitted) cut into a square shape for 14 gm.

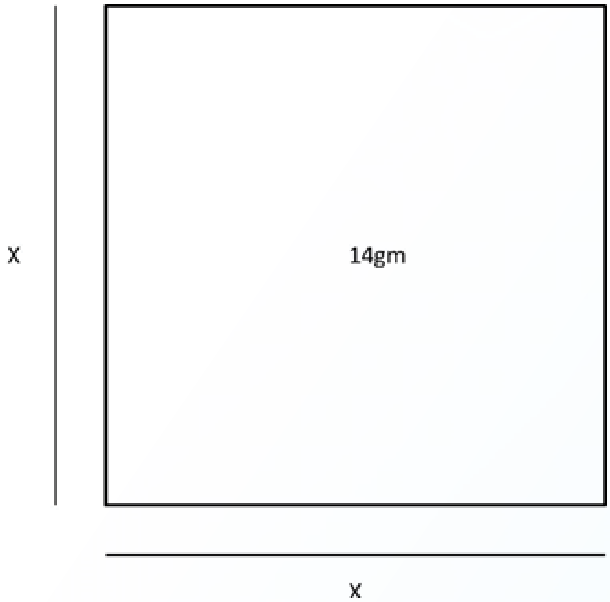


Figure 1: Standard fabric size for biopolishing

2.1.1.1. Fabric labeling

Table 1: Labeling the required fabric for different enzymes and percentages.

Fabric	Enzyme (owf%)	ENZA cellulose enzyme	Biopolish enzyme - 880	Azypolish-AC enzyme
Scoured and bleached ready for dyeing single jersey fabric (RFD S/J)	STD	Untreated fabric		
	0.5%	A1	B1	C1
	1.0%	A2	B2	C2
	1.5%	A3	B3	C3
Dyed rib fabric	STD	Untreated fabric		
	1.0%	D1	E1	
	1.5%	D2	E2	
	2.0%	D3	E3	
	3.0%	D4	-	

2.1.2 Liquor: Deionised water at room temperature

2.1.3 Enzyme: Required % on the weight of the fabric.

2.1.4 Acetic acid: Amount to maintain required acidic pH.

2.2. Machine

2.2.1 Sample dyeing machine

- Rotation speed of (40±2) rpm,
- Stainless steel container (capacity 200±10 ml),
- Stainless steel ball (dia = 0.6 cm, weight = 1 gm),

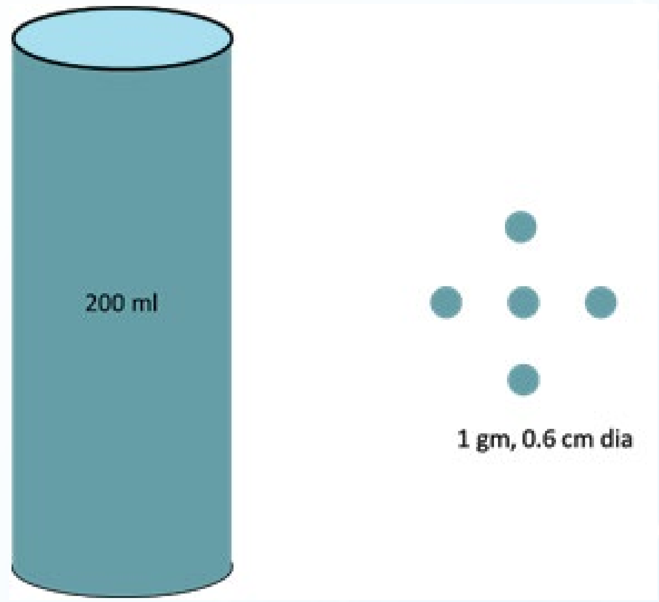


Figure 2: Steel container and steel ball

2.2.2 Microscope

Stereo microscope Euromax (upto 5.5 times magnification with 0.5 magnification gap).

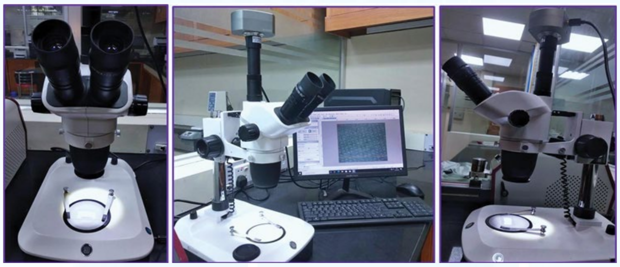


Figure 3: Stereo microscope

2.3. Method

2.3.1. Nomenclature

Table 2: The nomenclature of the testing method

BUTEXDCE2022C01	
BUTEX	The developer institute of the testing method
DCE	The laboratory where the methodology has been developed
2022	The year when this standard is developed
A	For physical testing method
B	For chemical testing method
C	For biological testing method
01	Serial number for the year

2.3.2. Recipe preparation

At first, the water (140 ml) is kept in a container and acetic acid is added with a dropper to maintain the pH of 5.5± 0.2.Then the required percentages of enzyme (0.5%, 1.0%,1.5%, 2.0% and 3.0%) are added to the liquor. Finally, the fabric is added to the liquor in the roll form.

Table 3: Recipe for the biopolishing

Reagents	Amount
Biopolishing	
Enzymes	Required percentage
Acetic acid	As required to maintain pH
M:L	1:10
Fabric	14gm
Liquor	140 ml
pH	5.8± 0.2
Time	30± 2 min
Temperature	58± 2°C
Enzyme inactivation	
Time	10± 1 min
Temperature	80± 2°C
Normal wash	
Temperature	Room
Drying	
Time	20 min
Temperature	120± 5°C

2.3.3. Biopolishing procedure

Biopolishing was carried out by the test specimen treated in the wash wheel at 60± 2°C for 30± 5 minutes at 40± 5 rpm. Then the temperature was raised to 80 ± 2°C for 10 ± 1 min to kill the enzymes. Then wash the sample with water and dry the fabric in the oven dryer for 20 minutes at 120 ± 5°C. Finally, the fabrics were conditioned under standard atmospheric conditions (25 ± 2°C and 65 ± 2%) for 24 hours before the assessment. Each sample was run in duplicate.

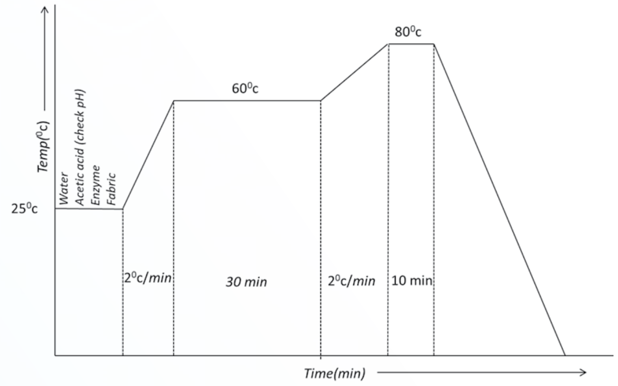


Figure 4: Process curve of biopolishing

2.3.4. Experimental design for surface analysis

2.3.4.1. Fabric view: The fabric is cut through the grain line into 3cm*3cm and folded into 1.5cm*3cm so that the front side is visible (not applicable for woven fabric). Take the folding fabric on the microscope slide (7.62 cm*2.54 cm) so that the edge is on the folding side. Attach both sides of the fabric on the slide with tape. Now capture the edge image with 3-5.5 times magnification so that protruding fibres are clearly visible.

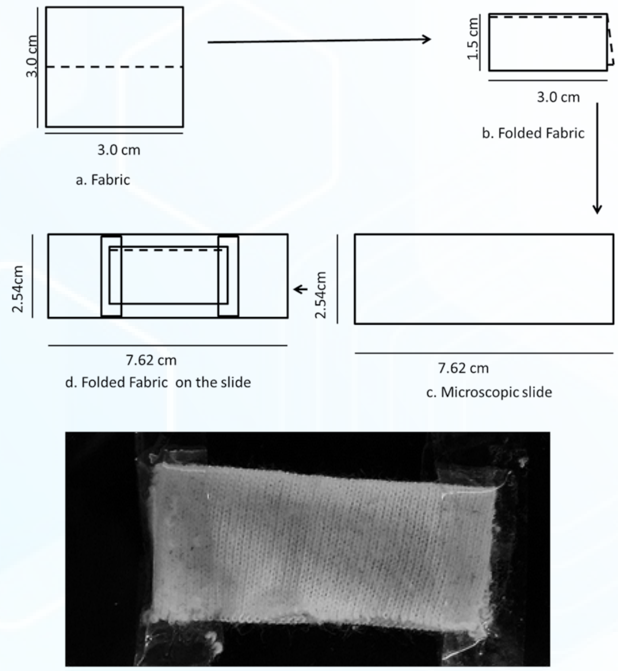


Figure 5: Experimental design for surface analysis (for fabric)

2.3.4.2. Yarn view: A single yarn from the fabric has been taken out and cut into 4 cm. Then, attach the yarn to the slide with the help of tape. Now capture the image with 3-5.5 times magnification.

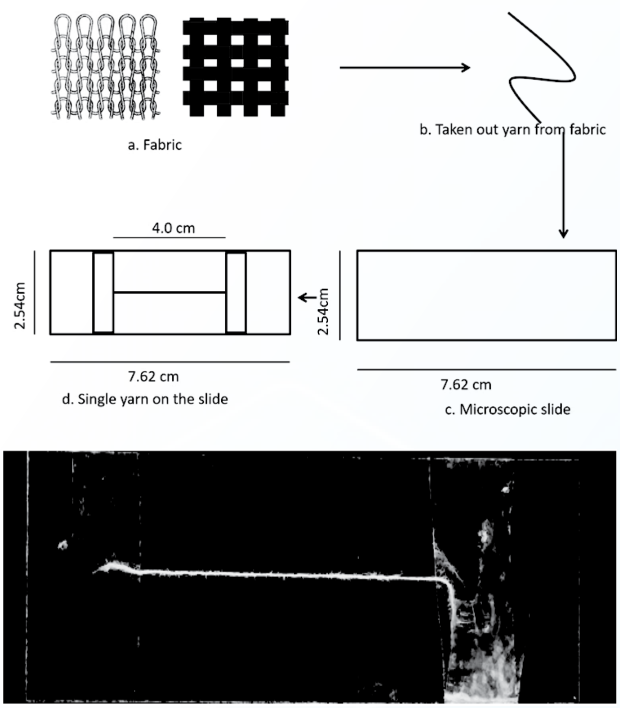


Figure 6: *Experimental design for surface analysis (for yarn)*

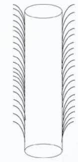
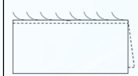
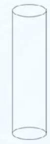
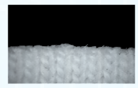
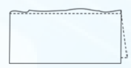
2.3.5. Evaluation/rating scale

Comparing the treated fabric with the untreated fabric for both fabric and yarn view these are the following assessing standards:

Table 4: Rating scale for the assessment

Rating	In words	Meaning	Description
α	Alpha	Not satisfactory	No loss of protruding fibres in comparison to untreated fabric
ß	Beta	Satisfactory	Approximately less than 50% loss of protruding fibres in comparison to untreated fabric
ϒ	Gamma	Good	Approximate loss of more than 50% protruding fibres in comparison to untreated fabric
Θ	Theta	Very Good	About 100% removal of protruding fibres in comparison to untreated fabric
δ	Delta	Not acceptable	Damage of the fabric structure with or without loss of protruding fibres

Table 5: Visual representation of the rating standards

Rating	Drawing		Visuals	
	Fabric	Single yarn	Fabric	Single yarn
α				
ß				
ϒ				
Θ				
δ				

3. Results and discussion

In Figure 7, A1, A2 and A3 show the effect of acid cellulase enzyme on RFD S/J cotton fabric compared to standard untreated fabric (STD) for both fabric and yarn view. A1 shows approximately less than 50% loss of protruding fibres compared to STD fabric, which makes it to be graded as ß; A2 showsapproximately more than 50% loss protruding fibres compared with STD fabric which makes it to be graded as ϒ and finally, A3 shows about to 100% removal of the protruding fibres comparing with untreated fabric. Still, some fibres are yet to make it ϒ grade. All the grading and interpretations are added on Table 6.

Similarly, in Figure 8, B1, B2 and B3 show the effect of Bio polish enzyme -880 on S/J RFD cotton fabric compared with standard untreated fabric (STD) for fabric and yarn view. Here B1 and B2 are graded as ß and ɤ in Table 6. But B3 is graded as Θ, which means 100% removal of the protruding fibres compared with STD fabric.

Finally, in Figure 9, C1,C2 and C3 show the effect of Azypolish-AC enzyme on S/J RFD cotton fabric compared with standard untreated fabric (STD) for both fabric and yarn view. Here C1 is graded as α, meaning no loss of protruding fibres compared with STD fabric; C2 is graded as ß, approximately less than 50% loss of protruding fibres compared with STD fabric and C3 is graded as ɤ, meaning approximately more than 50% loss protruding fibres comparing with untreated fabric. Among these three enzymes, Biopolish enzyme 880 shows comparatively better activity and Azypolish - AC shows comparatively worse (Table 6).

In the case of colored rib fabric Figure 10, only fabric surface analysis was considered, as taking out of yarn from the fabric is difficult.
In both D1 and D2, ß was the grade as approximately less than 50% removal of protruding fibres compared with STD fabric. D3 shows more than 50% removal of protruding fibres comparing with untreated fabric, which makes it ɤ grade. Finally, D4 shows the best result,which is about 100% removal of the protruding fibres compared with untreated fabric to be graded as Θ. Figure 11 shows a comparing view of STD and D4 fabric. It is clearly visible that the D4 shows a good result in that concentration of enzyme.

Similarly, E1 and E2 show ɤ meaning that more than 50% removal of protruding fibres compared with STD fabric and E3 contains about 100% removal of the protruding fibres graded as Θ.

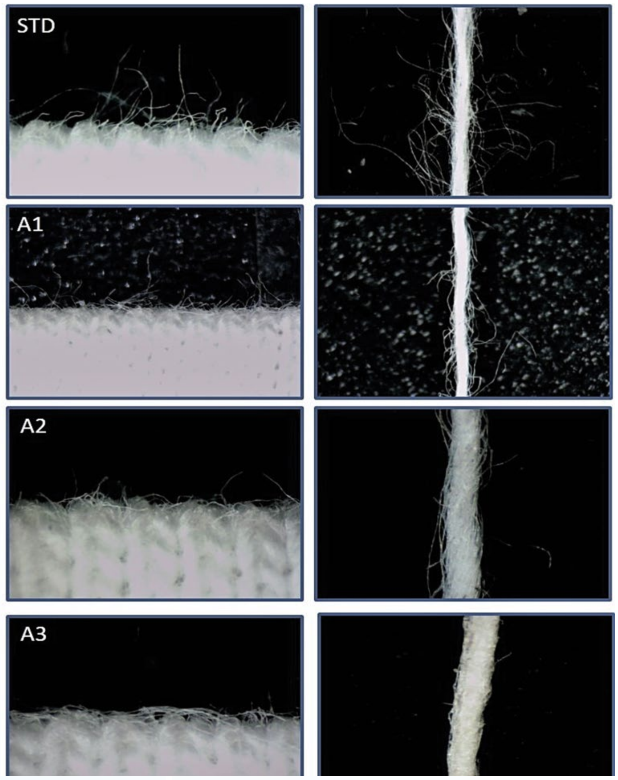


Figure 7: Surface view of samples treated with ENZ cellulase enzyme at different concentrations

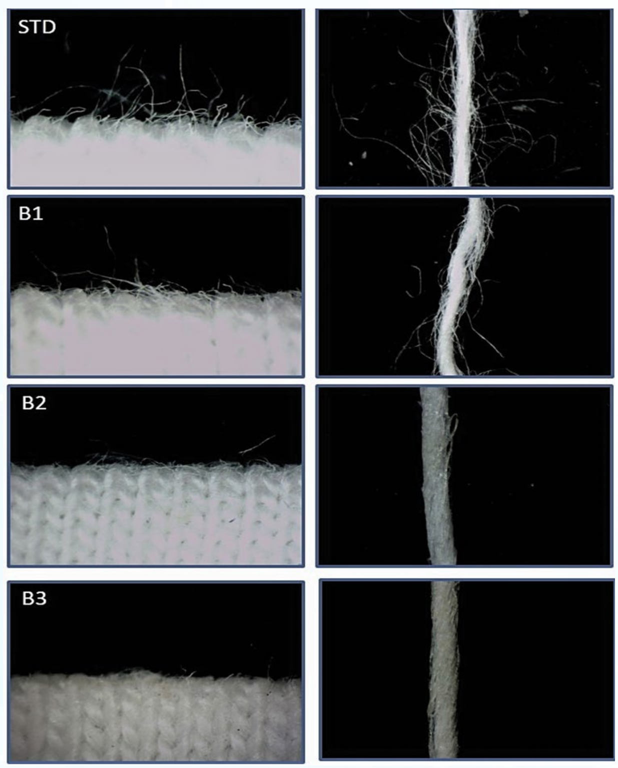


Figure 8: Surface view of samples treated with Biopolish enzyme-880 at different concentrations

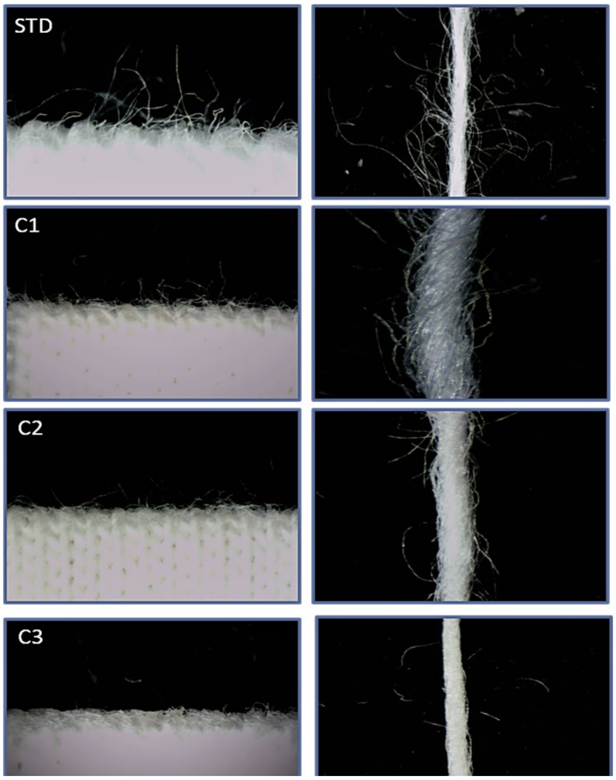


Figure 9: Surface view of samples treated with Azypolish-AC enzyme at different concentrations

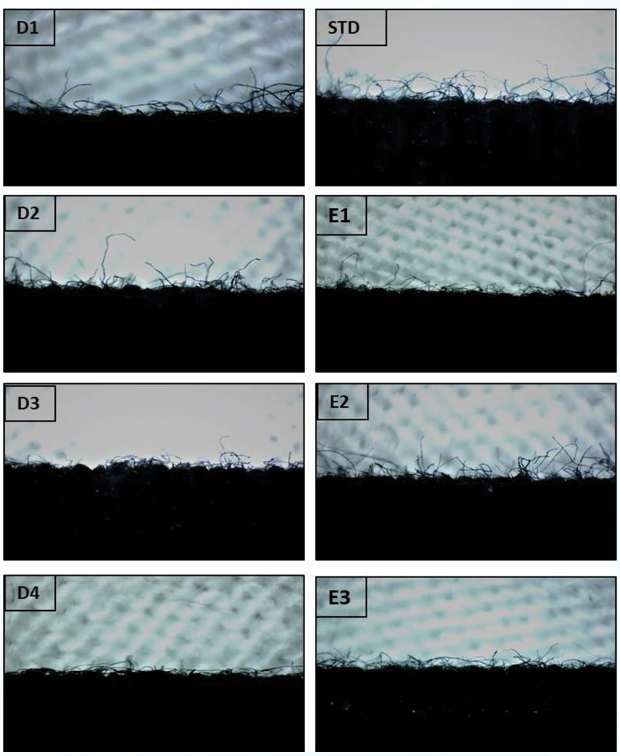


Figure 10: Surface view for samples treated with Acid cellulase and Bio polish enzyme -880 at different concentrations

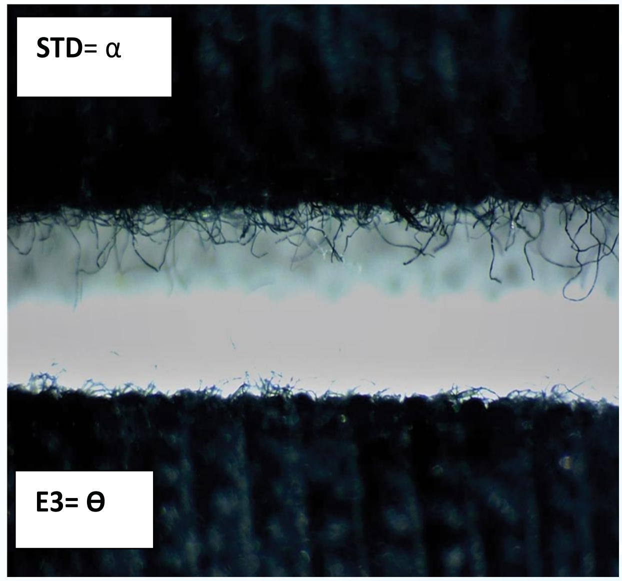


Figure 11: Comparative view of samples of STD and 3% ENZ cellulase enzyme

Depending on the grading onto the samples of three different enzymes, it can be concluded that the Acid cellulase enzyme works best at 3.0% concentration and about similar effect can be obtained by 2.0% of Bio polish enzyme -880. Finally, Azypolish-AC enzyme shows relatively little effect under these conditions. Maybe it works better under different conditions.

Table 6: Rating of all samples with the necessary interpretation

Sample	Rating	Interpretation
STD	-	Untreated fabric
A1	ß	Approximately less than 50% loss of protruding fibres in comparison to untreated fabric
A2	ɤ	Approximately more than 50% loss of protruding fibres comparing with STD fabric
A3	ɤ	Approximately more than 50% loss of protruding fibres comparing with STD fabric
B1	ß	Approximately less than 50% or equal to 50% loss of protruding fibres comparing with STD fabric

Sample	Rating	Interpretation
B2	ɤ	Approximately more than than 50% loss of protruding fibres comparing with STD fabric
B3	ϴ	About 100% loss of the protruding fibres comparing with STD fabric
C1	α	No loss of protruding fibres comparing with STD fabric
C2	ß	Approximately less than 50% loss of protruding fibres comparing with STD fabric
C3	ɤ	Approximately more than 50% loss of protruding fibres comparing with STD fabric
STD	α	No loss of protruding fibres
D1	ß	Approximately less than 50% /equal to 50% loss of protruding fibres comparing with STD fabric
D2	ß	Approximately less than 50% /equal to 50% loss of protruding fibres comparing with STD fabric
D3	ɤ	Approximately more than 50% loss of protruding fibres comparing with STD fabric
D4	ϴ	About 100% loss of the protruding fibres comparing with STD fabric
E1	ɤ	Approximately more than 50% loss of protruding fibres comparing with STD fabric
E2	ɤ	Approximately more than 50% loss of protruding fibres comparing with STD fabric
E3	ϴ	About 100% loss of the protruding fibres comparing with untreated fabric

This testing methodology is a qualitative grading system for assessing biopolishing effect. An expert's opinion or decision should be taken before the grading.

4. Conclusion

To sum up, a qualitative testing methodology has been developed for assessing enzymatic biopolishing effect on textile substrate.The system divided the analysis into 5 grade: α-Alpha, ß-Beta, ɤ-Gamma, ϴ-Theta and δ-Delta. Two different fabrics (RFD S/J and Black rib) have been treated with three different cellulase enzymes - Acid cellulase enzyme, Biopolish enzyme 880 and Azypolish AC in the similar condition and finally graded according to the grading system developed. The outcome of the study broadens a path for the textile testing method development.

This testing methodology has a great possibility of using in the textile industry for easier assessing of biopolishing effect. Further study can be taken using this methods to grade commercial textile samples.

Acknowledgment

The authors acknowledge the Dyes and Chemical Engineering (DCE) laboratory for all the chemical and instrumental supports.

Funding

The project was self-funded.

Conflict of interest

The authors state no conflict of interest.

Permission to reuse and copyright

All the Figures, tables and images are self-made and required citations were given to necessary places.

References

[1] M. Kumar et al., "Biopolishing of Cellulosic Fabrics: A Study on Low-Stress Mechanical Properties, Microstructure and Dye Uptake," Fibers Polym., vol. 22, no. 10, pp. 2803–2814, 2021, doi: 10.1007/s12221-021-0356-8.

[2] S. G. Mccloskey and J. M. Jump, "Bio-Polishing of Polyester and Polyester/Cotton Fabric," Text. Res. J., vol. 75, no. 6, pp. 480–484, 2005, doi:

10.1177/0040517505053846.
[3] M. L. Gulrajani, S. Agarwal and R. Agarwal, "Biopolishing of different varieties of tasar silk," Indian J. Fibre Text. Res., vol. 24, no. 3, pp. 226–228, 1999.
[4] N. Sankarraj and G. Nallathambi, "Effect of biopolishing on structural degradation and physical properties of cellulose," J. Serbian Chem. Soc., vol. 82, no. 5, pp. 567–578, 2017, doi: 10.2298/JSC161123031S.
[5] C. W. Kan and C. H. Au, "Effect of biopolishing and UV absorber treatment on the UV protection properties of cotton knitted fabrics," Carbohydr. Polym., vol. 101, no. 1, pp. 451–456, 2014, doi: 10.1016/j.carbpol.2013.09.044.
[6] S. A. Jabasingh and C. V. Nachiyar, "Process Optimization for the Biopolishing of Jute Fiberswith Cellulases from Aspergillus Nidulans AJ SU04," Int. J. Biosci. Biochem. Bioinforma., vol. 2, no. 1, pp. 12–16, 2012, doi: 10.7763/ijbbb.2012.v2.60.
[7] H. Ali, M. Hashem, N. Shaker, M. Ramadan, B. El-Sadek and M. A. Hady, "Cellulase Enzyme in Bio-finishing of Cotton-Based Fabrics: Effects of Process Parameters," Res. J. Text. Appar., vol. 16, no. 3, pp. 57–65, 2012, doi: 10.1108/RJTA-16-03-2012-B006.
[8] J. Liu, E. Otto, N. Lange, P. Husain, B. Condon and H. Lund, "Selecting cellulases for bio-polishing based on enzyme selectivity and process conditions," Text. Chem. Color. Am. Dyest. Report., vol. 32, no. 5, pp. 30–36, 2000.
[9] D. Saravanan, N. S. Vasanthi and T. Ramachandran, "A review on influential behaviour of biopolishing on dyeability and certain physico-mechanical properties of cotton fabrics," Carbohydr. Polym., vol. 76, no. 1, pp. 1–7, 2009, doi: 10.1016/j.carbpol.2008.10.019.
[10] H. Noreen, M. A. Zia, S. Ali and T. Hussain, "Optimization of bio-polishing of polyester/cotton blended fabrics with cellulases prepared from Aspergillus niger," Indian J. Biotechnol., vol. 13, no. 1, pp. 108–113, 2014.
[11] A. B. Kundu, B. S. Ghosh, A. K. Dutta, U. K. Ghosh, S. Sengupta and B. L. Ghosh, "Biopolishing of jute-cotton union fabric," Indian J. Fibre Text. Res., vol. 21, no. 2, pp. 127–130, 1996.
[12] L. Hao et al., "Utilizing cellulase as a hydrogen peroxide stabilizer to combine the biopolishing and bleaching procedures of cotton cellulose in one bath," Cellulose, vol. 21, no. 1, pp. 777–789, 2014, doi: 10.1007/s10570-013-0130-1.
[13] Y. Yu, J. Yuan, Q. Wang, X. Fan, P. Wang and L. Cui, "Noncovalent immobilization of cellulases using the reversibly soluble polymers for biopolishing of cotton fabric," Biotechnol. Appl. Biochem., vol. 62, no. 4, pp. 494–501, 2015, doi: 10.1002/bab.1289.
[14] L. Zilz, M. Rau, N. Budag, M. Scharf, A. Cavaco-Paulo and J. Andreaus, "Nonionic surfactants and dispersants for biopolishing and stonewashing with Hypocrea jecorina cellulases," Color. Technol., vol. 129, no. 1, pp. 49–54, 2013, doi: 10.1111/cote.12003.
[15] A. Hebeish, M. Hashem, N. Shaker, M. Ramadan, B. El-Sadek and M. A. Hady, "Effect of post- and pre-crosslinking of cotton fabrics on the efficiency of biofinishing with cellulase enzyme," Carbohydr. Polym., vol. 78, no. 4, pp. 953–960, 2009, doi: 10.1016/j.carbpol.2009.07.046.
[16] R. Anish, M. S. Rahman and M. Rao, "Application of cellulases from an alkalothermophilic Thermomonospora sp. in biopolishing of denims," Biotechnol. Bioeng., vol. 96, no. 1, pp. 48–56, 2007, doi: 10.1002/bit.21175.
[17] S. Gupta and V. Kelkar-Mane, "Use of uni-enzyme on cotton knitted fabric and its comparison with commercial formulation," Indian J. Fibre Text. Res., vol. 46, no. 4, pp. 349–353, 2021, doi: 10.56042/ijftr.v46i4.46218.
[18] M. Gias Uddin, "Effect of Biopolishing on Dye ability of Cotton Fabric - A Review," Trends Green Chem., vol. 2, no. 1, pp. 1–5, 2016, doi: 10.21767/2471-9889.100011.
[19] K. Mojsov, "Biopolishing Enzymes and their applications in textiles: a review," Tekst. Ind., vol. 61, no. 2, pp. 20–24, 2014.
[20] A. A. Ulson De Souza, F. C. S. Ferreira and S. M. A. Guelli, "Influence of pretreatment of cotton yarns prior to biopolishing," Carbohydr. Polym., vol. 93, no. 2, pp. 412–415, 2013, doi: 10.1016/j.carbpol.2012.12.023.
[21] N. Sankarraj and G. Nallathambi, "Enzymatic biopolishing of cotton fabric with free/immobilized cellulase," Carbohydr. Polym., vol. 191, pp. 95–102, 2018, doi: 10.1016/j.carbpol.2018.02.067.
[22] K. li Fu and D. nian Lu, "Effect of ethanediamine on bio-polishing of cotton fabrics with cellulase," Cellulose, vol. 20, no. 6, pp. 3093–3100, 2013, doi: 10.1007/s10570-013-0054-9.
[23] D. Saravanan, S. N. Sree Lakshmi, K. Senthil Raja and N. S. Vasanthi, "Biopolishing of cotton fabric with fungal cellulase and its effect on the morphology of cotton fibres," Indian J. Fibre Text. Res., vol. 38, no. 2, pp. 156–160, 2013.
[24] A. Esfandiari, E. Firouzi-Pouyaei and P. Aghaei-Meibodi, "Effect of enzymatic and mechanical treatment on combined desizing and bio-polishing of cotton fabrics," J. Text. Inst., vol. 105, no. 11, pp. 1193–1202, 2014, doi: 10.1080/00405000.2014.880222.
[25] S. Kalia and R. Sheoran, "Modification of ramie fibers using microwaveassisted grafting and cellulase enzyme-assisted biopolishing: A comparative study of morphology, thermal stability and crystallinity," Int. J. Polym. Anal. Charact., vol. 16, no. 5, pp. 307–318, 2011, doi: 10.1080/1023666X.2011.587946.
[26] S. Guide, "Standard Guide for Measuring Hairiness of Yarns by the Photo-Electric," Annu. B. ASTM Stand., vol. i, pp. 2–4, 2007.