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Biopolishing of Cellulosic Fabrics: A Study on Low-Stress Mechanical Properties, Microstructure, and Dye Uptake

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Abstract: This study evaluated the effect of biopolishing using cellulase enzymes on the low stress mechanical properties, microstructure, and dye uptake of different cellulosic fabrics (cotton, modal, and cotton denim). The tactile features were studied via the Kawabata evaluation system (KES) and showed increments in tensile elongation, tensile resilience and surface properties leading to wearing comfort, whereas the enhanced shear and bending properties of yarns in the fabrics became stronger and more rigid. The surface morphology of the cellulosic fabrics was evaluated by scanning electron microscopy (SEM), and the results indicated that the surface of the treated fabrics became smooth and polished. Biotreatment enhanced comfort, luster, and smoothness without affecting other important properties of the fabrics. The effect of biopolishing before and after the dyeing process has been studied. This was the first study to compare cotton, modal, and denim fabrics for the evaluation of low-stress mechanical properties by employing biopolishing.

Keywords: Biopolishing, Cellulosic fabrics, Low stress mechanical properties, Surface morphology, Cellulase

Introduction

Fabrics are subjected to a series of processes referred to as textile finishing before they are converted into garments [1]. In fact, finishing is a prerequisite for all kinds of fabrics made from diverse types of fibers. The various mechanical and chemical processes that are carried out include calendering, raising, shearing, anti-shrinking, crease-resisting, waterproofing or water repellent finish [2]. Cotton is the most commonly used natural fiber in the apparel industry due to its comfort and hydrophilic properties. Like every other natural fiber, cotton has various shortcomings, such as poor luster, tendencies to shrink and crease badly, and poor drape quality [3,4]. These properties can be improved by the finishing process. Hence, the finishing processes employed will depend on the end use requirement of a particular garment [5]. The finishing operations differ according to the properties to be imparted to the material. It includes improving the whiteness and luster by bleaching, improving the feel, softness, and fullness by mercerizing and imparting special properties to fabric such as a flame-retardant finish and anti-soil finish [6].

The textile industry uses large amounts of chemicals, water, and energy, which leads to direct and indirect effects on the environment [6,7]. The textile finishing industry accounts for the high consumption of natural and synthetic finishing agents, such as starch, polyvinyl acetates, fluorocarbons, nanoforms of different metals, and inorganic salts [8,9]. The use of enzymes in textile finishing (e.g., amylases for starch desizing) has been known since 1910, and they are indispensable in modern pretreatment operations in the textile industry [1]. Advancements in the field of science have led to the development of novel enzymes that are being widely adopted in the textile processing and garment industry for improving fabric properties [10]. Enzymatic improvement of the properties of cellulosic fabrics is generally carried out by cellulase enzymes. Cellulase enzyme activity is comprised of three enzyme complexes, i.e., endoglucanases, cellobiohydrolases, and cellobiases, which work in a synergistic way [11-13]. Endoglucanases act on β-1,4-glycosidic linkages and hydrolyze the cellulose chain randomly, cellobiohydrolases act on the ends of the cellulose chain and release cellobiose units, and cellobiase hydrolyzes cellobiose to glucose units. The slow kinetics of the cellulase enzyme on cotton fabrics make them suitable candidates for the biopolishing of fabrics without impacting fabric quality [13].

Enzymatic treatment of cellulosic fabrics by cellulase

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*Corresponding author: manoj.kumar13@icar.gov.in enzymes can be subdivided into two major phases. In the first phase, the enzyme acts on the cellulosic part and results in increased softness, pilling reduction, improved fabric appearance and surface structure (biopolishing) [14,15]. In the second phase, this biopolishing treatment results in the replacement of the stone-wash step in the case of denim fabrics. Enzymatic surface modification is reported to have a soft, smooth, elegant look for cellulosic fabrics, thereby improving the quality [13,16]. In this way, it is possible to convert low-grade fabric qualities to top-quality fabric. The demand profile in today's fashion headline "soft-smooth-high class" can be fully met with the help of this enzyme technology. Cellulase enzymes are currently being marketed for textile finishing and differ in behavior in terms of their origin and the specific composition of the cellulase enzyme complex. The treatment of woven and knitted fabrics with cellulase enzyme can seriously damage textile fabrics if parameters, such as pH, temperature, and time of treatment, are not controlled properly. Therefore, knowing the optimum conditions for improving the quality of fabric by enzyme treatment is also very important. Enzyme technology is also of interest in the chemically demanding pretreatment of cellulosic fabrics. Modal fiber is a distinct regenerated cellulosic fiber that has a better wet modulus than normal viscose rayon and a minimum value of tenacity in the wet stage at 5 % elongation [17]. Currently, modal fibers are gaining market value in the apparel industry.

Yang *et al.* [18] reported the effect of yarn structures on cellulase treatment on denim fabrics. It was reported that after cellulase treatment, the quantity of dissociable groups in cotton decreased, while reducing groups, such as -CHO, increased [19]. It has been reported that the nature and composition of cellulase plays a vital role in determining the hand value of regenerated cellulosic materials such as modal materials [20]. Therefore, it is understood that the inherent structure of cellulose influences the final outcome of cellulase treatment. To our knowledge, no research has compared the low stress mechanical properties and other related physical changes in different cellulosic fabrics or structures after cellulase treatment. In this study, cellulosic fabrics were treated with cellulase enzyme to study their effect on weight loss, tensile strength, surface morphology by SEM, comfort properties by KES, dye uptake, and the depth of color using a computerized color matching system. This is the first comparative study to evaluate the low-stress mechanical properties of three different cellulosic fabrics by employing cellulase enzymes.

Experimental

Materials

Three different cellulosic fabrics were used in this study. Plain grey cotton fabric was procured from a local fabric market. Modal and denim fabrics in grey form were generously supplied by the Institute of Chemical Technology, Mumbai. Various specifications of these fabrics are mentioned in Table 1. The fabrics were enzymatically desized using 0.6 g/*l* of amylase and 0.5 g/*l* of wetting agent at 70 °C for 45 min. The fabrics were then scoured and bleached using the one bath method in an autoclave using 2.5 g/*l* of sodium hydroxide, 8 g/*l* of hydrogen peroxide (35 % w/w), 1 g/*l* of peroxide stabilizer, and 0.3 g/*l* of nonionic wetting agent at 120 °C for 45 min with a 1:15 material-to-liquor ratio (MLR). The fabrics were thoroughly washed with hot water and cold water and dried in a hot air oven.

Commercial acid cellulase enzyme was purchased from M/s Zytex Industries, Mumbai. This enzyme was used for the treatment of the fabrics without further purification. Acetic acid, sodium chloride, sodium hydroxide, hydrogen peroxide, sodium carbonate, and sodium acetate were of analytical reagent grade. The reactive dye "Novacron Reactive Red FN-2BL (CI Reactive Red 271), a heterofunctional reactive dye supplied by M/s Huntsman Pvt Ltd., was used for the dyeing experiments.

Method of Treatment of Fabrics with Cellulase Enzyme

Cellulase enzyme treatment was carried out on all three fabrics (cotton, modal, and denim) in a Launder-o-meter (M/s RB Electronics, Mumbai, India). Containers in the Launder-o-meter were revolved at a speed of 50 rpm. Cotton, modal, and denim fabrics were treated with cellulase enzyme (1 %, 2 %) in 0.2 M acetate buffer at pH 4.5, 20:1 MLR, and 55 °C for 60 min. After treatment, the fabrics were thoroughly rinsed with deionized water to remove abraded or digested fibers, followed by heat deactivation of the remaining cellulase enzyme on fabrics for 10 min at 80 °C. Afterwards, the fabrics were dried in a hot air oven at 105 °C. Each experiment was replicated three times. The treated fabrics were analysed for weight loss, tensile strength, low mechanical properties by KES and chromaticity analysis using a

Table 1. Specifications of the cellulosic fabrics used in the study

Nature of fiber	Composition (%)	Weaving pattern	Fabric mass (g/m^2)	No. of ends per inch (EPI)	No. of picks per inch (PPI)	Warp count (Ne)	Weft count (Ne)
Cotton	100	Plain	128	79	73	30	30
Cotton denim	100	Twill	355	90	35	24	16
Modal	100	Plain	86	95	91	50	50

computerized color matching system.

Determination of Weight Loss (*WL*)

WL(%) was determined as the difference in weight of samples before (w_1) and after (w_2) enzyme treatments by using the following equation:

$$WL(\%) = \frac{w_1 - w_2}{w_1} \times 100$$

Evaluation of Tensile Strength

The changes in the tensile strength of the control and enzyme-treated cotton, modal, and denim fabrics were evaluated using the Instron® Universal Tensile Strength Tester via the strip method (ASTM D1682-64). The average of five specimens (10 cm length and 2.5 cm width) was accounted for in the tensile test measurement.

Low-Stress Mechanical Properties

The low-stress mechanical properties (bending, compression, tensile, shear, and surface properties) of the cellulase enzyme-treated and control fabrics were evaluated by KES supplied by KATO TECH Co. Ltd., Japan. Various data on the fabrics as measured using this system are useful to estimate the cause of the change in relation to fabric fatigue.

Surface Morphology

The surface microstructures of the cotton, modal and denim fabrics (treated and untreated) were observed using SEM (Philips XL-30). The samples were coated with a thin layer of gold to obtain conductivity using a sputter coater and scanned under SEM with an accelerating voltage of 10 kV.

Dyeing of Cellulosic Fabrics

The cellulosic fabrics were dyed with reactive dye using a laboratory IR beaker dyeing machine (M/s RB Electronics, Mumbai, India) using the isothermal method. The dye bath was prepared using water with an MLR of 1:15, and the required amount of dye (3 % by weight of the material) was added to the beaker. Wetted out and squeezed cellulosic fabric was introduced into the beaker. Then, the beaker was tightly closed and kept on the rotating shaft of the dyeing machine. The temperature of the bath was increased to 60 °C and maintained for 30 min. Then, 15 g/*l* of sodium carbonate was added to facilitate the reaction between the dye and fiber. The process continued for an additional 30 min. Afterwards, the temperature was reduced to 40 °C, and the dyed fabric was removed from the beaker. Then, it was subjected to soaping treatment using nonionic detergent followed by hot and cold washes.

Evaluation of Color Strength and Fastness Properties

The uniformity of the dyeing was observed subjectively. The colorimetric values (L^*, a^*, b^*, and *K*/*S*) of the dyed samples were evaluated using a Premier Colorscan computer color matching system (M/s Premier Colorscan Instruments Pvt. Ltd., Mumbai) at D65 illuminate/10 ° observers according to AATCC Evaluation Procedure 6. The *K*/*S* value, which is the indication of relative color intensity, was calculated using the Kubelka-Munk equation:

$$K/S = \frac{(1-R)^2}{2R}$$

where *R* is the reflectance value, *K* is the coefficient of absorption, and *S* is the coefficient of scattering.

For washing fastness testing, the dyed fabric in contact with adjacent cotton fabrics was mechanically agitated in a Launder-o-meter with 5 g/*l* of sodium carbonate and 2 g/*l* of standard detergent for 30 min at 60 °C while maintaining the MLR at 1:50 and then rinsed and dried. The change in color/shade of the specimen and the staining of the adjacent fabric(s) was assessed with reference to the original fabric using a greyscale.

A schematic diagram of the methodology is presented in Figure 1.

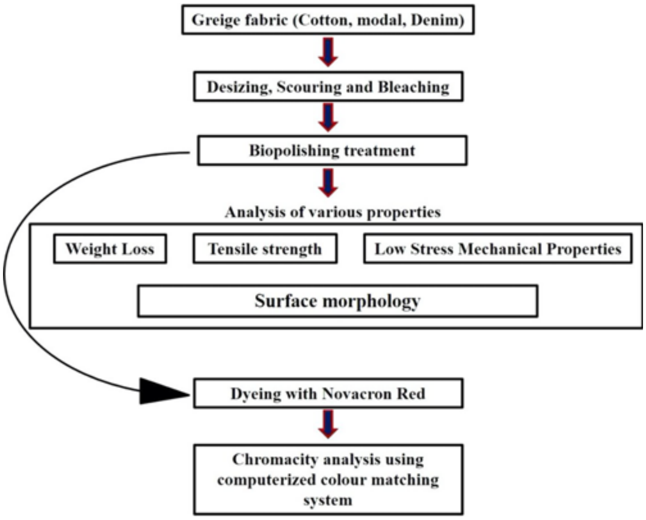

Figure 1. Schematic diagram of the methodology followed for the study. Greige fabric was subjected to pretreatment (desizing, scouring, and bleaching), and then cellulase enzyme treatment was performed at different levels (1 % and 2 %). The biopolished fabrics were then analysed for weight loss, tensile strength, low stress mechanical properties, and surface morphology by scanning electron microscopy. The dye uptake properties of the biopolished fabrics were evaluated after dyeing the fabrics with Novacron red. Color strength was evaluated using a computerized color matching system.

Statistical Analysis

The data obtained from weight loss, tensile strength, and low stress mechanical properties were examined using the

PROC GLM (General Linear Model) of SAS 9.4 (SAS Institute, Cary, USA). The effect of enzyme treatment on the cotton, modal, and denim fabrics was analyzed by one-way ANOVA. Furthermore, significant differences between means were assessed by Tukey's honest significant difference (HSD) test at $p<0.05$.

Results and Discussion

Weight Loss

Cellulase enzymes are extensively exploited industrially for textile applications. The cellulose-cellulase system is a very good example of the solid-liquid interaction of materials. During this interaction, the cellulase enzyme acts on cellulosic polymeric chains and depolymerizes the cellulosic polymer. This results in a reduction in the weight of the cellulosic material after cellulase treatment. Table 2 compares the percentage weight loss in cellulosic materials after acid cellulase treatment under different conditions. In the absence of enzymes, there was no significant change in the weight of the material after the treatment. However, after cellulase enzyme treatment, a significant reduction in the weight of cellulosic materials was found irrespective of the nature of the material. Treatment with cellulase leads to weight loss due to digestion of the protruding cellulosic fibers in all three fabrics [21-24]. An illustration showing the mechanism of action of cellulase enzyme in digesting the protruding and pilling fibers from the cellulosic fabric is presented in Figure 2.

The enzymatic digestion of cellulosic substances depends on the fabric used, incubation time, temperature, and many other parameters. The reduction in weight was much more

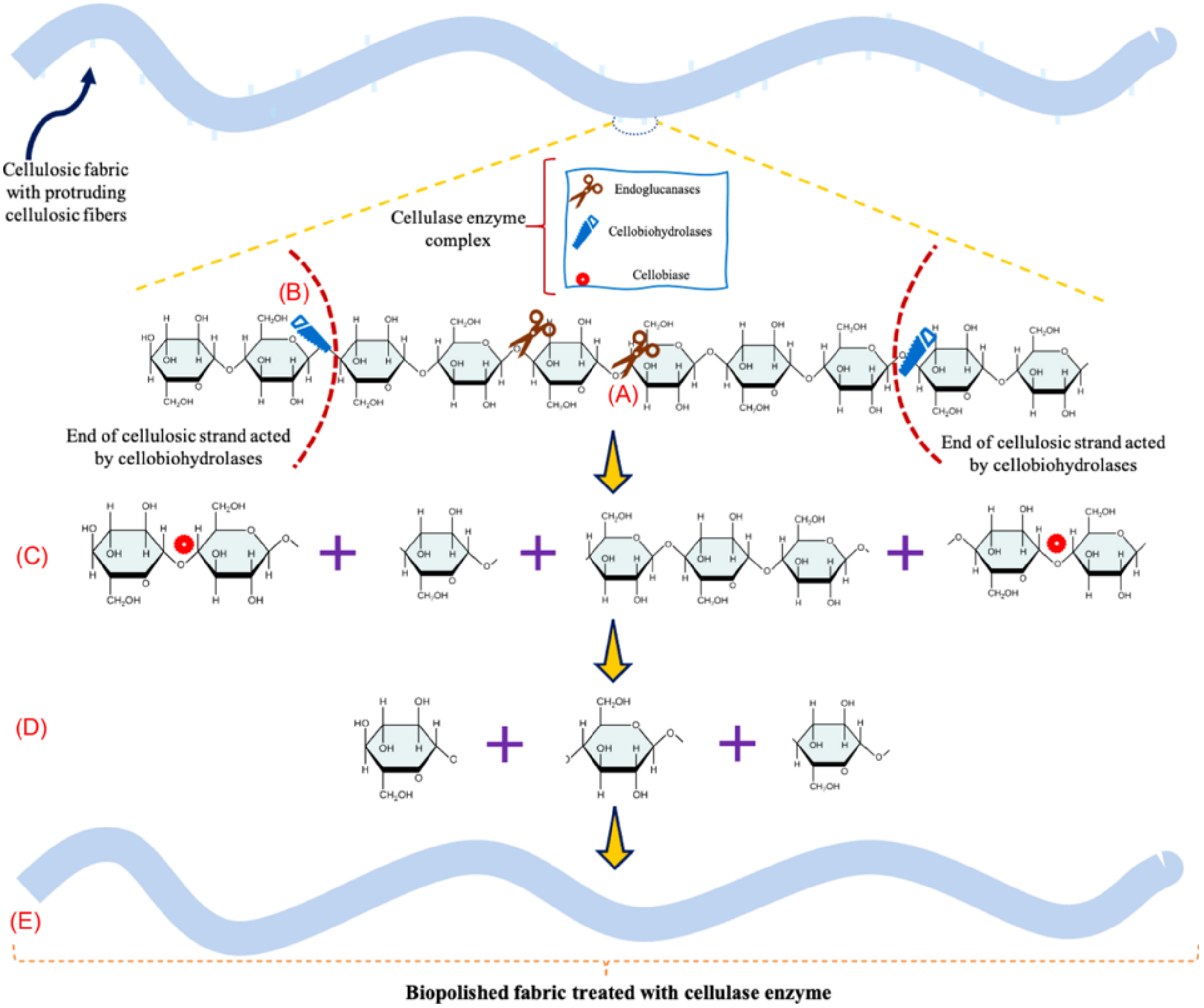

Figure 2. Mechanism of action of cellulase on cellulosic fabrics for improving their properties; (A) application of endoglucanase on the β-1,4-glycosidic bonds on the interior of the cellulosic strand, (B) release of cellobiose by the action of cellobiohydrolases (exoglucanases), (C) hydrolysis of cellobiose into glucose units by cellobiase, (D) hydrolysis product of protruding cellulosic strands, and (E) bio-polished fabric.

pronounced in modal fabric (6.47 %) than in cotton materials. This may be due to the presence of the many accessible regions present in the modal fiber, which is a regenerated fiber. In the modal fiber, much amorphous cellulose II is present, while in cellulose I, much more crystalline cellulose is present. The literature also supports that cellulase action is much more severe in mercerized cotton than in unmercerized cotton [25,26]. Similar results have been also reported for viscose rayon fabric. When we compared cotton plain woven fabric with denim fabric, weight loss was higher in the plain-woven fabric. This may be because plain woven fabric is much more accessible to enzymes than twill fabric. Another hypothesis is that it may be due to less fuzziness in the denim cotton structure than in the plain weave structure. Increasing the enzyme dosage resulted in an increase in the weight loss regardless of the substrate used, i.e., in all three cases, cotton, modal and denim fabric, as observed in Table 2. This is the reason why there is more of the substrate, i.e., cellulose is exposed to the cellulase enzyme.

Tensile Strength Loss

A major limitation of cellulase treatment of cellulosic material to improve the fabric comfort properties is the drastic reduction in tensile strength of treated fabrics. Depolymerization of cellulose negatively influences the tensile strength of treated materials. For all fabrics, the tensile strength loss in the warp direction was found to be higher than that in the weft direction. This may be because of the higher weave density in the warp direction. Increasing the enzyme concentration from 1 to 2 % resulted in a significant ($p<0.05$) decrease in tensile strength regardless of the type of fabrics, i.e., cotton, modal, and denim (Table 2). This is because digestion of cellulosic fibrils leads to fragility of fabric yarns and, as a result, a reduction in the strength of the yarns of the fabric. Similar findings were reported by Sankarraj and Nallathambi [27] and Ulson de *et al.* [28].

Effect of Biopolishing Using Cellulase on the Low-Stress Mechanical Properties of Fabrics

Fabric handle is a very important characteristic required for fabrics to be used in the garment industry. It is directly related to the comfort of fabrics. The KES for fabrics was used in this study for the objective evaluation of low stress mechanical properties, such as tensile, shearing, bending, compression, and surface properties, which are used for expressing fabric comfort in apparel industries. The low-stress mechanical properties of cotton, modal and denim fabrics were measured by KES for fabrics, and the results are depicted in Table 3.

Tensile Properties

The linearity (LT) of the stress-strain curve is indicative of wearing comfort, and LT=1 indicates that the curve is a straight line. As shown in Table 3, the LT values of cotton fabric ranged from 0.823-0.903. There was a significant ($p<0.05$) decrease in the LT of cotton fabric treated with cellulase enzyme compared with the control fabric. However, increasing the concentration of enzyme from 1 to 2 % caused no significant change in LT values. In the case of denim and modal fabric, the LT values ranged from 0.730-0.739 and 0.753-0.777, respectively. Treatment of denim and cotton fabric with different dosages of enzyme showed

Table 2. Effect of enzymatic treatment on weight loss (WL) in percent and loss in tensile strength (TS) in percent in the different fabrics (at 4.8 pH, 60 minutes and 20:1 MLR)

Property	Enzyme concentration (%)	Fabric			Mean (E)
		Cotton	Denim	Modal	
Weight loss (%)	0	0.08^{e}	0.07^{e}	0.08^{e}	0.07^{c}
	1	4.87^{b}	2.34^{d}	5.30^{b}	4.17^{b}
	2	6.21^{a}	3.37^{c}	6.47^{a}	5.35^{a}
	Mean (F)	3.72^{a}	1.93^{b}	3.95^{a}	
	SEM	SE (F)=0.0776; SE (E)=0.0776; SE (F×E)=0.1345			
Tensile strength loss (%) (warp)	1	23.76^{c}	18.21^{d}	10.79^{e}	17.58^{b}
	2	31.92^{a}	19.15^{d}	27.88^{b}	26.31^{a}
	Mean (F)	27.83^{a}	18.68^{b}	19.34^{b}	
	SEM	SE (F)=0.3187; SE (E)=0.3187; SE (F×E)=0.5521			
Tensile strength loss (%) (weft)	1	11.07^{c}	5.19^{d}	11.93^{c}	9.39^{b}
	2	19.97^{b}	10.10^{c}	27.27^{a}	19.11^{a}
	Mean (F)	15.51^{b}	7.65^{c}	19.60^{a}	
	SEM	SE (F)=0.1969; SE (E)=0.1969; SE (F×E)=0.3409			

Means with same superscript letter are not significantly different (significance assessed at $p<0.05$); SEM=standard error of mean. Where MLR is material to liquid ratio.

Table 3. Effect of cellulase enzyme treatment on the low-stress mechanical properties of the three fabrics

Property	Enzyme concentration (%)	Fabric			Mean (E)
		Cotton	Denim	Modal	
LT	0	0.903^{a}	0.783^{b}	0.777^{b}	0.821^{a}
	1	0.843^{ab}	0.739^{b}	0.763^{b}	0.782^{ab}
	2	0.823^{ab}	0.73^{b}	0.753^{b}	0.770^{b}
	Mean (F)	0.857^{a}	0.752^{b}	0.764^{b}	
	SEM	SE (F)=0.0139; SE (E)=0.0139; SE (F×E)=0.0241			
WT (gf·cm/cm^2)	0	17.9^{ab}	18.48^{a}	15.2^{bcd}	17.196^{a}
	1	16.45^{abc}	17.8^{ab}	14.22^{cd}	16.158^{ab}
	2	15.28^{bcd}	17.76^{ab}	13.56^{d}	15.533^{b}
	Mean (F)	16.544^{b}	18.013^{a}	14.329^{c}	
	SEM	SE (F)=0.3227; SE (E)=0.3227; SE (F×E)=0.5589			
RT (%)	0	56.45^{b}	41.62^{c}	73.73^{a}	57.271^{a}
	1	59.92^{b}	39.35^{c}	78.27^{a}	59.182^{a}
	2	60.05^{b}	39.08^{c}	79.19^{a}	59.442^{a}
	Mean (F)	58.811^{b}	40.020^{c}	77.064^{a}	
	SEM	SE (F)=0.9061; SE (E)=0.9061; SE (F×E)=1.5694			
EMT (%)	0	2.77^{cd}	9.08^{b}	2.27^{d}	4.71^{b}
	1	3.46^{c}	9.58^{ab}	2.64^{cd}	5.23^{a}
	2	3.42^{c}	10.07^{a}	2.78^{cd}	5.42^{a}
	Mean (F)	3.21^{b}	9.58^{a}	2.56^{c}	
	SEM	SE (F)=0.1019; SE (E)=0.1019; SE (F×E)=0.1765			
G (gf/cm·deg)	0	2.07^{cd}	3.54^{a}	1.84^{d}	2.48^{a}
	1	2.51^{bc}	3.33^{a}	1.88^{d}	2.57^{a}
	2	2.59^{b}	3.51^{a}	1.87^{d}	2.66^{a}
	Mean (F)	2.39^{b}	3.46^{a}	1.87^{c}	
	SEM	SE (F)=0.0525; SE (E)=0.0526; SE (F×E)=0.0910			
2HG (gf/cm)	0	4.42^{b}	6.36^{a}	1.85^{c}	4.21^{b}
	1	5.91^{a}	6.38^{a}	1.78^{c}	4.69^{a}
	2	6.81^{a}	6.65^{a}	1.73^{c}	5.06^{a}
	Mean (F)	5.71^{b}	6.46^{a}	1.79^{c}	
	SEM	SE (F)=0.1139; SE (E)=0.1139; SE (F×E)=0.1974			
2HG5 (gf/cm)	0	5.78^{c}	12.62^{a}	1.87^{d}	6.76^{b}
	1	7.33^{b}	12.47^{a}	2.24^{d}	7.35^{ab}
	2	8.07^{b}	12.81^{a}	2.04^{d}	7.64^{a}
	Mean (F)	7.06^{b}	12.63^{a}	2.05^{c}	
	SEM	SE (F)=0.1629; SE (E)=0.1629; SE (F×E)=0.2822			
B (gf·cm^2/cm)	0	0.0367^{c}	0.5800^{ab}	0.0279^{c}	0.2156^{ab}
	1	0.0433^{c}	0.6267^{a}	0.0497^{c}	0.2400^{a}
	2	0.0367^{c}	0.5567^{b}	0.0307^{c}	0.2078^{b}
	Mean (F)	0.0389^{b}	0.5878^{a}	0.0367^{b}	
	SEM	SE (F)=0.0078; SE (E)=0.0078; SE (F×E)=0.0134			
2HB (gf·cm^2/cm)	0	0.0367^{b}	0.517^{a}	0.0132^{b}	0.1878^{a}
	1	0.0500^{b}	0.537^{a}	0.0212^{b}	0.2022^{a}
	2	0.0384^{b}	0.497^{a}	0.0137^{b}	0.1822^{a}
	Mean (F)	0.0422^{b}	0.5167^{a}	0.0133^{b}	
	SEM	SE (F)=0.0080; SE (E)=0.0080; SE (F×E)=0.0139			

Table 3. Continued

Property	Enzyme concentration (%)	Fabric			Mean (E)
		Cotton	Denim	Modal	
LC	0	0.527^{a}	0.283^{b}	0.539^{a}	0.4511^{a}
	1	0.537^{a}	0.299^{b}	0.542^{a}	0.4600^{a}
	2	0.517^{a}	0.317^{b}	0.567^{a}	0.4667^{a}
	Mean (F)	0.528^{a}	0.300^{b}	0.550^{a}	
	SEM	SE (F)=0.0124; SE (E)=0.0124; SE (F×E)=0.0215			
WC (g·cm/cm²)	0	0.058^{c}	0.430^{a}	0.035^{c}	0.176^{a}
	1	0.06^{c}	0.406^{a}	0.029^{c}	0.166^{ab}
	2	0.056^{c}	0.337^{b}	0.031^{c}	0.141^{b}
	Mean (F)	0.0589^{b}	0.3911^{a}	0.0322^{c}	
	SEM	SE (F)=0.0071; SE (E)=0.0071; SE (F×E)=0.0122			
RC (%)	0	31.99^{e}	38.00^{cde}	43.67^{bcd}	37.89^{c}
	1	35.76^{de}	42.74^{bcd}	49.81^{ab}	42.77^{b}
	2	45.85^{abc}	47.45^{ab}	53.32^{a}	48.87^{a}
	Mean (F)	37.87^{c}	42.73^{b}	48.93^{a}	
	SEM	SE (F)=0.9472; SE (E)=0.9472; SE (F×E)=1.6406			
MIU	0	0.183^{abc}	0.173^{abc}	0.163^{c}	0.173^{a}
	1	0.184^{abc}	0.197^{ab}	0.160^{c}	0.181^{a}
	2	0.186^{abc}	0.199^{a}	0.167^{bc}	0.186^{a}
	Mean (F)	0.187^{a}	0.190^{a}	0.163^{b}	
	SEM	SE (F)=0.0037; SE (E)=0.00367; SE (F×E)=0.0064			
MMD	0	0.020^{a}	0.017^{a}	0.011^{a}	0.017^{a}
	1	0.016^{a}	0.021^{a}	0.011^{a}	0.017^{a}
	2	0.018^{a}	0.018^{a}	0.012^{a}	0.017^{a}
	Mean (F)	0.020^{a}	0.020^{a}	0.010^{a}	
	SEM	SE (F)=0.000; SE (E)=0.000; SE (F×E)=0.000			
SMD (µm)	0	6.055^{a}	5.787^{a}	3.675^{b}	5.184^{a}
	1	6.504^{a}	5.870^{a}	3.749^{b}	5.419^{a}
	2	6.165^{a}	5.643^{a}	4.055^{b}	5.290^{a}
	Mean (F)	6.242^{a}	5.768^{b}	3.883^{c}	
	SEM	SE (F)=0.1029; SE (E)=0.1029; SE (F×E)=0.1782			

LT: Linearity, WT: tensile energy, RT: tensile resilience, EMT: tensile strain, G: shear rigidity, 2HG: hysteresis of shear force at 0.5 degrees, 2HG5: hysteresis of shear force at 5 degrees, B: bending rigidity, 2HB: hysteresis of bending movement, LC: linearity of compression, WC: compression energy, RC: compression resilience, MIU: coefficient of friction, MMD: mean deviation of MIU, SMD: geometrical roughness. Means with the same superscript letter are not significantly different (significance assessed at $p<0.05$); SEM=standard error of the mean.

negligible changes in the LT values. The lower values of LT give higher fabric extensibility in the initial strain range and impart comfort during wearing of the textile material. The decrease in LT was mainly due to the action of cellulase enzyme treatment as fiber became more elastic.

Tensile energy (WT) is defined as the ability of fabric to withstand an external load. It is the energy or work required to stress the specimen. The area under the force-elongation curve represents the work done in stretching the specimen. Among untreated fabrics, denim showed the highest value of WT (18.48 gf·cm/cm^2), indicating better toughness. Biopolishing of cotton, denim and modal fabric resulted in a significant ($p<0.05$) decrease in WT values. The WT of cotton fabric decreased from 17.9 to 15.28 gf·cm/cm^2, which was due to the enzymolysis of cellulose in the fabric. In the case of denim fabric, the WT values decreased from 18.48 to 17.76 gf·cm/cm^2, depicting an approximately 4 % decrease in WT. In contrast, in the modal fabric, the change in WT varied from 15.2 to 13.56 gf·cm/cm, depicting an approximately 11 % decrease in WT. Lower WT values of biopolished fabrics indicate low tensile strength and good handling value of the fabric. These results are also supported by the results obtained from the tensile strength test presented in Table 3.

Tensile resilience (RT) represents recovery after tensile deformation. A higher RT makes the fabric more elastic and improves its handling properties. Denim fabric was tougher than cotton and modal fabric due to its lower RT value. Enzyme treatment of denim fabric further reduced its RT from 41.62 to 39.08 %. The opposite trend was observed in the case of cotton and modal fabric, showing an increase in RT values after enzyme treatments. There was an approximately 6.4 % improvement in resilience in cotton fabric treated with 2 % cellulase enzyme over untreated fabric. The magnitude of improvement was still higher (7.4 %) in the case of modal fabric biopolished with 2 % cellulase enzyme.

EMT represents the tensile strain under strip biaxial extension. A high EMT value also denotes greater wearing comfort [29]. For untreated fabrics, the highest value of EMT was presented by denim fabric (9.08 %), followed by cotton (2.77 %) and modal fabric (2.27 %). This indicated the high wearing comfort of the denim followed by cotton and modal. Biopolishing of all fabrics caused significant ($p<0.05$) improvement in EMT over their untreated counterparts. The increases in EMT were 23.5 %, 10.9 % and 22.5 % for enzyme-treated cotton, denim and modal fabrics, respectively. The increased extensibility of biopolished fabrics was due to an increase in the crystalline region of the cellulose in the fabric. Similar findings have been reported earlier in durable press finished cotton fabric with cellulase enzyme [30] and in degumming of silk using enzymes [31].

Shear Properties

The shear rigidity (G) of a fabric depends on the mobility of cross threads at the intersection point. G is highly related to the fabric bending property. The lower the value of G is, the better the fabric handle will be. The hysteresis of the shear force at 0.5 degrees shear angle (2HG) and 5 degrees shear angle (2HG5) denotes the elastic recovery, i.e., the behavior of the removal of stress. Shear properties in conjunction with bending properties are thus a good indication of the ability of a fabric to drape. G, 2HG and 2HG5 values for various fabrics are shown in Table 3.

The results revealed that the highest (3.54 gf/cm·deg) and lowest (1.84 gf/cm·deg) values of G were exhibited by untreated denim and modal fabric, respectively. There was a significant ($p<0.05$) increase in the G value of enzyme-treated cotton fabrics with the highest increase represented by cotton fabric treated with 2 % cellulase enzyme. Additionally, the hysteresis of shear for cotton fabric increased after enzyme treatment. Cotton fabric treated with 2 % cellulase showed 54 % and 39.6 % increases in 2HG and 2HG5, respectively. This finding reflected the resistance of enzymatically treated cotton fabrics to shearing movement, resulting in harder material and thus poor comfort. This is due to the enzymatic removal of noncellulosic impurities, increasing the contact area and thus the friction between fibers [21,32]. It was found that cellulase enzyme treatment at 1 % and 2 % had no significant ($p>0.05$) effect on the shear properties of modal and denim fabrics.

Bending Properties

Bending rigidity (B) is a measure of the ease with which the fabric bends or is a measure of a fabric's ability to resist bending deformation, whereas hysteresis of bending movement (2HB) is a measure of recovery from bending deformation. Bending properties have a direct relationship and greater association with the fabric handle. The higher the value of B, the lower the fabric handle.

Table 3 shows the bending properties of untreated and enzymatically treated cotton, denim, and modal fabrics. The results indicated that the values of B and 2HB for denim fabric were greater than those for cotton and modal fabric. Treatment of denim with 1 % cellulase enzyme increased the values of B and 2HB from 0.5800 to 0.6267 gf·cm^2/cm and 0.517 to 0.537 gf·cm/cm, respectively. However, a further increase in enzyme concentration to 2 % decreased the B and 2HB values. A similar trend was observed in cotton and modal fabric; however, the change in B and 2HB values was insignificant ($p>0.05$). A high bending rigidity after enzymatic treatment indicates that the fibers became stronger and showed more resistance to bending motions [21]. The bending properties are also influenced greatly by the crystallinity and cross-sectional shape of the cellulose fiber.

Compression Properties

Compression properties provide a feeling of bulkiness and sponginess in the fabric. The compressibility is directly proportional to the thickness of the fabric. Higher thickness corresponds to better compressibility. The compression properties of fabrics, such as the linearity of the compression curve (LC), compression energy (WC), and compression resilience (RC), are shown in Table 3.

WC implies the fluffy feeling of the fabric. From Table 3, denim showed higher WC values than the other two fabrics. Low WC values of cotton and modal demonstrated their low fluffiness. The values of WC for denim ranged from 0.337 to 0.430 g·cm/cm^2, revealing the high compressibility of denim over cotton and modal fabric. Enzyme treatments loosened the denim fabric structure as WC decreased from 0.430-0.337 g·cm/cm^2 (at 2 % cellulase enzyme dosage), showing an approximately 21.6 % reduction in WC. In the case of cotton and modal fabric, no significant ($p>0.05$) change was observed in WC values after enzyme treatment.

RC reflects the ability of fabric to recover from compressional deformation. Higher RC values indicate better recovery properties. The concentration of enzyme significantly ($p<0.05$) affected the RC values of the fabrics (Table 3). Irrespective of the type of fabric, increasing the enzyme concentration resulted in increasing RC values. As shown by the results, biopolishing caused approximately 43.3 %, 24.9 %, and 22 % improvements in the RC values of cotton, denim, and modal fabrics, respectively.

The LC of fabric is mainly affected by the thickness of the fabric and compression characteristics of yarn. Enzyme treatment of all three types of fabrics showed no significant ($p > 0.05$) change in their LC values.

Surface Properties

The surface properties of the fabric have a close relationship with the fabric handle. They are of the most important parameters contributing to the smoothness of the fabric. The yarn structure, fabric geometry and applied finish greatly influence the surface features of fabric [33]. The surface properties of the fabrics in terms of the coefficient of friction (MIU), mean deviation of the MIU (MMD) and geometrical roughness (SMD) are presented in Table 3.

The lower the MIU and SMD are, the smoother the fabric will be. There was no significant ($p>0.05$) difference in the MIU of untreated and enzyme-treated fabrics except in denim. In denim fabrics, the value of MIU increased from 0.173 to 0.199, depicting a 15 % increase in MIU after enzyme treatment. Enzyme treatment was found to have no significant ($p>0.05$) effect on the cotton and modal fabrics. As expected, enzyme treatment caused a negligible change in the MMD values of all fabrics. Cotton fabric showed the highest SMD (6.055-6.504 μm), followed by denim (5.643-5.870 μm) and modal fabrics (3.675-4.055 μm). Enzyme-treated fabrics displayed higher SMD values than their control counterparts. However, the variation in values was insignificant ($p>0.05$).

Effect of Biopolishing on the Surface Morphology of Cellulosic Fabrics

SEM micrographs show the changes in the surface morphology of the cotton, modal, and denim fabrics with enzymatic treatment. Figures 3, 4, and 5 show the surface morphologies of the cotton, modal, and denim fabrics after cellulase enzyme treatment. The figure on the left shows untreated samples, and the figure on the right shows treated samples. The untreated cotton fiber can be characterized by the presence of parallel ridges and grooves. The hydrolytic reaction with 1 % cellulase enzyme caused some visible changes in its surface morphology. As shown in the figures, the adhered loose fibers in the fabric were efficiently removed by cellulase enzyme treatment, and as a result, individual fibers appeared finer and more polished in all three fabrics. This positive change in morphology was due to treatment with cellulase enzyme, which digested protruding fibers and undulating fibers [34].

Effect of Biopolishing on Dyeing Behavior Followed by Wash Fastness of Cellulosic Fabrics

Reactive dyes are popular for dyeing cellulosic fabrics due to their easier application, lower cost, durability, and brighter color. Reactive dyes are durable, as these dyes covalently link cellulose functional groups in amorphous form [35].

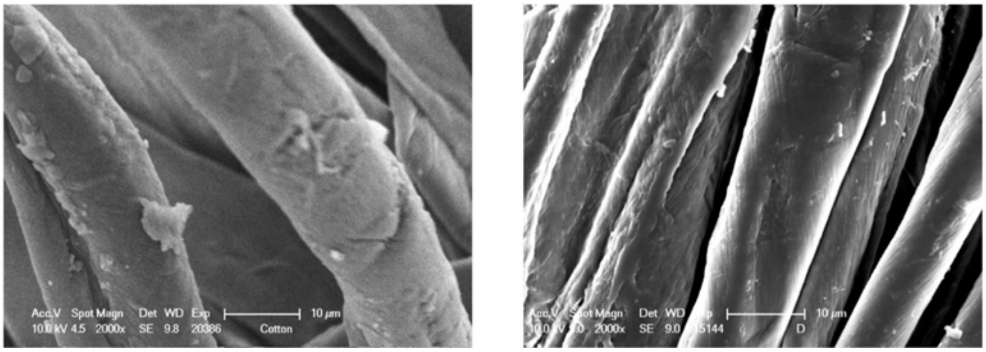

Figure 3. SEM images of untreated (left) and cellulase enzyme treated (right) cotton fabric.

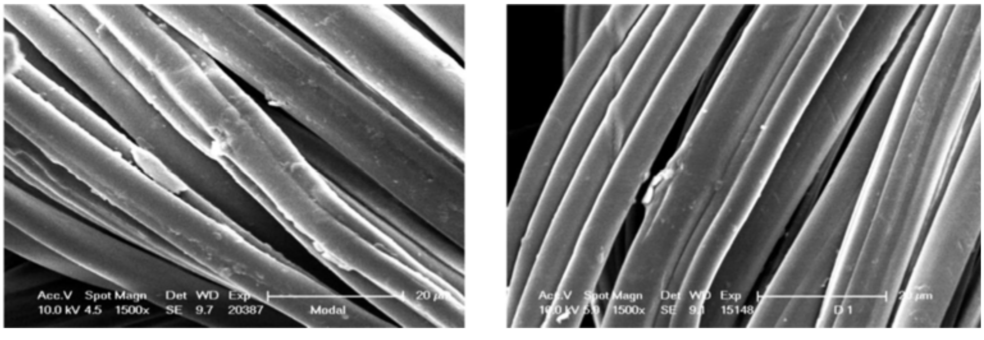

Figure 4. SEM images of untreated (left) and cellulase enzyme-treated (right) modal fabric.

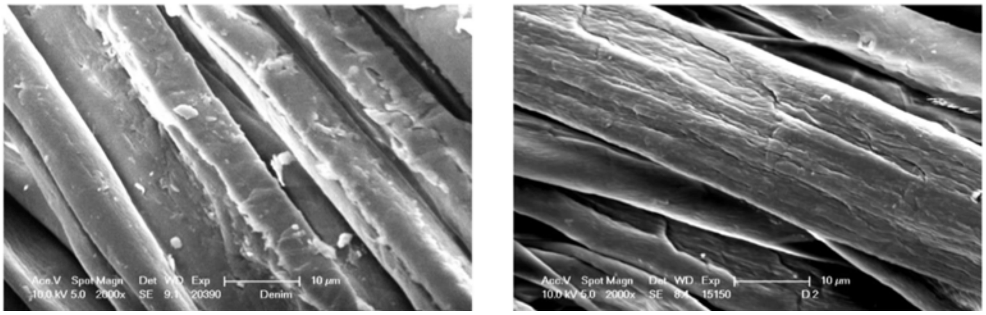

Figure 5. SEM images of untreated (left) and cellulase enzyme-treated (right) denim fabric.

Table 4. Effect of biopolishing on the dyeing behavior of cellulosic fabrics (cotton, modal and denim fabrics)

Sr. no.	Fabric sample	Condition for bio-polishing	*K*/*S* values 550 nm	L^*	a^*	b^*
1	Cotton fabric	Control (only buffer)	6.25	45.11	45.28	-5.45
		1 % Cellulase	7.55	44.22	44.36	-5.20
		2 % Cellulase	8.62	43.25	44.21	-5.05
2	Modal fabric	Control (only buffer)	9.54	40.44	46.23	-5.25
		1 % Cellulase	11.42	38.25	44.25	-4.27
		2 % Cellulase	12.47	37.86	44.12	-4.23
3	Denim fabric	Control (only buffer)	8.96	43.10	41.56	-5.37
		1 % Cellulase	12.49	39.85	40.28	-4.28
		2 % Cellulase	13.68	38.63	39.98	-4.16

Data are expressed as the means of triplicate samples with the standard deviation as zero.

After biotreatment with cellulase enzyme, reactive dyes resulted in enhanced color properties [36]. The results, presented in Table 4, indicate that increasing the concentration of enzyme led to an improved depth of color, and the highest *K*/*S* value was at 2 % cellulase enzyme. The increased *K*/*S* of the biopolished fabric may be due to the formation of more dye-absorbing surfaces and modification of the pore microstructure with simultaneous digestion of fibrillar material, thereby improving the extent of dye penetration into the treated fabric structure as well as fixation of dye molecules along with a decrease in the scattering coefficient, i.e., deeper shades [37]. The fabric was dyed and treated with 1 % and 2 % cellulase to determine the effect of treatment on the various color parameters, i.e., L^*, a^*, and b^* (Table 4). We found that the L^* value for all the treated samples marginally decreased, which suggests that the color partially gained strength upon treatment with the enzyme.

Table 5. Washing fastness of the dyed fabric after cellulase enzyme treatment

Sr. no.	Fabric sample	Condition for bio-polishing	Washing fastness: Change in shade	Washing fastness: Change in stain on cotton
1	Cotton fabric	Control (only buffer)	4	4
		1 % Cellulase	4	4
		2 % Cellulase	4	4
2	Modal fabric	Control (only buffer)	4	4
		1 % Cellulase	4	4
		2 % Cellulase	4	4
3	Denim fabric	Control (only buffer)	4	4
		1 % Cellulase	4	4
		2 % Cellulase	4	4

To determine the wash fastness properties of the dyed fabrics, the fabrics were assessed using the standard method, and the results are presented in Table 5. There was no change in the fastness properties of the dyed fabrics. The fastness properties of dyed material depend upon the nature of the dye fibers of the dyed material. Reactive dyes form covalent bonds that have very good stability compared to any other dye-fiber bond system. Since we have used hetero bifunctional dyes for the dyeing of cellulosic fabrics, they have two kinds of reactive systems that are stable under acidic and alkaline conditions. Pretreatment with cellulase did not influence the dye-fiber bond formation during the

Table 6. Color strength analysis before and after cellulase enzyme treatment of the dyed (3 % reactive dye) fabric

Sr. no.	Fabric sample	Condition for bio-polishing	*K/S* values	L^*	a^*	b^*
			550 nm			
1	Cotton fabric	Control (only buffer)	6.00	46.11	46.32	-6.02
		1 % Cellulase	4.96	47.65	43.36	-6.49
		2 % Cellulase	4.52	47.32	44.5	-6.25
2	Modal fabric	Control (only buffer)	9.14	41.44	47.6	-5.56
		1 % Cellulase	7.32	44.22	47.7	-4.91
		2 % Cellulase	7.04	44.64	47.4	-5.14
3	Denim fabric	Control (only buffer)	8.48	42.92	49.21	-5.49
		1 % Cellulase	7.91	42.75	46.5	-5.16
		2 % Cellulase	7.03	44.03	45.7	-5.85

Data are expressed as the means of triplicate samples with the standard deviation as zero.

dyeing process.

Effect of Biopolishing on Dyed Fabrics (Cotton, Modal, and Denim)

The dyed fabric was again treated with 1 % and 2 % cellulase enzymes to determine the effect of treatment on the color strength (*K*/*S*). We found that *K*/*S* decreased with increasing cellulase enzyme concentration. This may be due to further loosening of the fabric, leading to release of the reactive dye from the fabric. The decrease in the color strength occurred regardless of the substrate used, i.e., in all the fabrics, there was a loss in color strength due to enzyme treatment (Table 4). Fabric was dyed and treated with 1 % and 2 % cellulase to determine the effect of treatment on the various color parameters, i.e., L^*, a^*, b^*, chroma, hue, and color difference (Table 6). We found that the L^* values for all of the treated samples increased marginally, which suggested that some degree of color strength was lost upon treatment with the enzyme.

Conclusion

This study presented and compared the low stress mechanical properties and other related physical changes in different cellulosic fabrics or structures after cellulase enzymatic treatment. The application of commercial cellulase enzyme as a biopolishing agent for cellulosic fabrics leads to increased weight loss and decreased tensile strength and simultaneously improves the low stress mechanical properties studied by KES. SEM and KES analyses have shown that there is an overall increase in the smoothness and improved comfort properties of the fabrics. There was a loss in the color of dyed fabric upon enzyme treatment; therefore, the application of cellulase as a pretreatment agent before dyeing will improve the efficiency of the dyeing process. Hence, biopolishing is an excellent approach for enhancing comfort, luster, and smoothness without considerably affecting other important properties of the fabric.

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