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Effects Optimization of Bio-Polishing Industrial Process Parameters

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Abstract

The purpose of this study is to select an appropriate commercial neutral cellulase enzyme for denim garments bio-polishing to achieve specific industrial goals. A full factorial experimental design was used to evaluate the effect of factors considered in the optimization of the bio-polishing process (fabric ID: x1, treatment time: x2, treatment temperature: x3, enzyme concentration: x4, storage time: x5, inactivation step: x6 and enzyme type: x7). Experiments were conducted using laboratory washing machine. Subjective evaluation was performed at a pilot and at an industrial scale. Tear, tensile strength and subjective evaluation concerning hand feel, fabric color, indigo dye pocket back staining and fuzziness extent were evaluated. Results showed that x6 and x7 had significant effects on the fabric tear and tensile strength loss. In the optimization, the great dependence between observed and predicted tear strength and tensile strength loss, the correlation coefficient of the models ($R^2 > 0.85$) and the important value of F-ratio proved the validity of the models. Results showed that denim leg panels treated with the enzyme Lava-Cell NSZ presented a minimum loss of tear and tensile strength. A low-temperature and time enzymatic bio-polishing process was developed at industrial scale.

Keywords

Bio-Polishing, Cellulase, Tensile Strength, Optimization, Fabric Hand Feel

1. Introduction

Nowadays the world moves more and more to ecofriendly processes and products. Enzymatic treatments have been attempted to replace harsh chemical treatments with green and sustainable biotreatments [1]. As mentioned by several researchers [2] [3] [4] enzymes are the better sustainable alternative to the harsh toxic chemicals used in the textile wet processing. Then, enzymes operate in moderate conditions (Temperature, pH) and have specific actions. So, the enzyme use reduces water and energy consumption. In the first step, reduction of water consumption minimizes the waste water. In second step, specific actions minimize the effect on mechanical properties of garments. The use of enzyme treatments increases productivity and give less dust.

In conventional process sodium hypochlorite, potassium permanganate, pumice stone were used to achieve fading aspect. The abrasion effect can be caused from the garments clashing against each other and the wall of the washer drum. Disadvantage of these method are as follows:

- Pumice stones cause large amount of back-staining.
- Pumice stones are required in very large amount.
- They cause considerable wear and tear of machine.
- Use of toxic chemical products.

The continuous research on new enzymes and formulations are going hand in hand with the innovation and sustainability strategies of leading fashion brands and laundries. Catarina Costa *et al.* [5] noted that a property that remains of extreme importance is smoothness. Human skin is very sensitive to soft-touch being a crucial factor in offering comfort in clothing.

Bio-polishing is one of use of biotechnology. It controlled the hydrolysis of cellulosic fiber by the cellulose group of enzymes in order to improve surface appearance. Textile products requirements have increased due to changes in the buyer's expectations and their awareness of quality and environment. It removes the weakened fibers and surface pills and significantly reduces pilling, resulting in smoother appearance and improved softness [6]. Cellulase is a complex enzyme system composed of endoglucanase (1,4-β-D-glucon 4-glucano-hydrolase), cellobiohydrolases (1,4-β-D-glucan cellobiohydrolase) and cellobiases (β-D-glucohydrolases) [7] [8]. Commercial cellulases for bio-polishing originate from *Trichoderma reesei* and *Humicola insolens* [9] [10]. Cellulose is a linear, unbranched polymer, and it has highly oriented molecular structure with intermolecular chain bonding. Initially, endoglucanase randomly attacks cellulose chains and cleaves the β-1,4-linkage between the glucose units, creating new cellulosic chain ends. Exoglucanase splits at the reducing and non-reducing ends of cellulose with a release of cellobiose. Finally, β-glucosidase hydrolysis cellobiose into glucose [11]. All these enzyme components act in a synergistic fashion during the degradation of cellulose. There are two major commercial classifications of cellulase enzymes based on optimum ranges. Acid cellulases exhibit the most activity within the pH range: 4.5 - 5.5. While neutral cellulases are more effective in the 5.5 - 8.0 pH range [12] [13].

Literature provides several studies about the effect of bio-polishing on knitted fabrics [14]-[18]. Many studies discussed the effects of cellulases on denim fabrics during laboratory bio-polishing. Though the effects of cellulase hydrolysis remain a surface phenomenon, changes in many physical aspects as well as mechanical properties of fabrics take place during denim bio-polishing. The major drawback associated with the use of cellulase is the loss of mass and the reduction in the fabric tensile strength [19] [20] [21]. Statistically, significant differences were observed between untreated and treated fabrics with regard to breaking strength. Cellulase enzyme treated cotton fabrics exhibited about 12% - 18% strength loss [22]. Weight loss values ranging from 1.7% to 19.7% with proportionate loss in the breaking strength have been reported in the bio-polished fabrics with acid fungal cellulase (*Trichoderma reesei*) [23]. A maximum of 10% loss in fabric strength is considered acceptable [12] [24]. Although neutral cellulase enzymes require longer process, they are less aggressive compared to acid ones [25]. The cellulose amorphous region is generally responsible to form the fuzz and pilling in cotton fabric. Hence, a single type of cellulase preferentially endoglucanase may probably be sufficient for degradation of amorphous cellulose with minimum weight loss [21] [26].

On the other hand, during bio-polishing treatment, the removed indigo can back stain the reverse side of the fabric if acid cellulases are used, while neutral enzymes yield very low dye redeposition [27]. Indeed, cellulase enzymes will cleave to cellulose chains, producing a reducing end and a nonreducing end for each broken (1-4)-β-D glycosidic bond. Reducing ends produced might induce indigo back staining [27]. Indigo dye has a higher affinity for acid cellulases than neutral cellulases (which gives lower staining levels). In fact, acid cellulases with a higher content of neutral amino acids show more affinity for indigo dyes [26]. Besides, reproducibility of the cellulase treatment is relatively poor compared to other chemical or mechanical textile processes [16].

In the present study, the effect of four cellulases on denim fabric tear and tensile strength were analyzed. Desirability optimization method was applied in order to choose the best tool giving minimum tear and tensile strength with acceptable surface aspect and having the best price with less temperature and processing time. Aspect is given by subjective evaluation carried out by industry experts to support the selection of the proposed enzyme. Earlier cited literature does not show any industrial results of such study, and therefore do not reflect the facts exactly. For this reason, all the phases of this study will be specifically carried out under common working conditions prevailing in the textile industry. In our case results were checked in all scales (laboratory, pilot process). This work gives for the first time to textile industry one process containing desizing, scouring, stone treatment and bio-polishing with lowest degradation.

2. Materials and Methods

2.1. Materials

Four indigo dyed denim fabrics were used in this investigation. Fabrics were referred to as Fabric I., Fabric II., Fabric III., and Fabric IV (see **Table 1**). All the substrates (fabrics) were sized by the same sizing recipe. Leg panels were used

Table 1. Main characteristics of the tested denim fabric samples.

Fabric ID		Fabric I.	Fabric III.	Fabric II.	Fabric IV.
Composition		100% cotton	98% cotton + 2% elastane	93% cotton + 5% polyester + 2% elastane	100% cotton
Weave design		3/1 twill, warp faced			
Mass, g/m², (ASTM D 3776),		375	351	365	420
Tensile strength, N (ASTM D5034)	Warp	1098 (±2.1)	1190 (±2.5)	1009 (±1.9)	1190 (±3.2)
	Weft	547 (±1.6)	538 (±3.4)	450 (±2.6)	557 (±0.9)
Tear strength, cN, (ASTM D 1424)	Warp	7180 (±3.9)	7240 (±4.6)	7020 (±3.8)	7150 (±3.8)
	Weft	4060 (±3.9)	3970 (±2.9)	3880 (±4.1)	4080 (±4.3)

for tests in laboratory scale, five pants manufactured from each fabric were used in pilot scale and 100 pants are tested in industrial scale.

2.2. Commercial Enzymes

Four commercials neutral cellulase enzymes: *Azu cell* (Dystar), *Hydros rti* (Garmon), *Lava cell NBG* (Dystar), *Lava cell NSZ* (Dystar) referred as A, B, C and D were used in this study (see Table 2). These neutral enzymes are suitable for defibrillation of fibers and the enzyme system used in this case is endoglucanase. According to enzymes' technical sheets, proposed pH and temperature of application correspond to maximum enzyme efficiency.

This new generation of enzyme is used at neutral pH and cold temperature. It can be combined with bio-scouring, desizing and with dyeing process. The use of new generation saves water and energy.

All enzymes can be used at temperature of 30˚C and at pH equal to 7 (Table 2).

2.3. Denim Garments Treatments

Desized, scoured and stoned denim pants were subject to bio-polishing during the 6th step (see more details in Table 3) using different cellulase enzymes. The enzyme is then inactivated. A storage time of wet bio-polished samples can be predicted due to the unavailability of the machines, especially in the industrial scale.

Laboratory tests and pilot tests were carried out using a laboratory washing machine type OMI LCF30, and industrial tests were carried out using TONELLO 420LW1 machine.

2.4. Experimental Design

Seven factors selected as influential parameters were analyzed to evaluate their effects on selected fabrics' tear and tensile strength. We established using MINITAB® 17.1.0 statistical software a mixed-level factorial design of experiments

Table 2. Enzymes and their application characteristics.

Enzyme properties	Enzyme			
Enzyme ID	A: Azucel NE/2	B: Hidros rti	C: Lava cell NBG	D: Lava Cell NSZ
Optimum pH	5.5 - 8	5.5 - 7	5.5 - 8	6.5 - 7.5
Optimum temperature, ˚C	30 - 65	25 - 50	30 - 60	30 - 50

Table 3. The conditions of fabrics washing.

Step	Treatment	Liquor ratio	Time, Min.	Temperature, (˚C)	Product	Product dose
1	Rinsing	1/10	2	30	Water	-
2	Desizing	1/10	5	40	Desizing agent	1.25 g/L
3	Rinsing	1/10	2	30	Water	
4	Pumice stone	1/10	35	30	Pumice stones	0.5 kg/kg (fabric)
5	Rinsing	1/10	2	30	Water	-
6	Bio-polishing♠	1/10				
7	Rinsing	1/10	2	30	Water	-
8	Inactivation	1/10	5	40	Na_2CO_3	1 g/L
9	Rinsing	1/10	2	30	Water	-
10	Spin	-	2	-	-	-
11	Drying	-	50	60	-	-

♠See details of bio-polishing step in Table 4.

to study simultaneously the effects of these factors on the measured properties and to determine the optimum application conditions. Factorial design was employed to achieve the best overall optimization of processes [28] [29].

In the current study, factors ranged from x1 to x7 with their corresponding levels are obtained based on primary tests, suppliers' recommendations, and enzymes data sheets (see Table 4). 384 leg panels were tested in laboratory washing machine. Proposed dosage varies according to fabric composition, mechanical action of the washing machine and mainly according to the desired effects.

Storage time is the time during which the leg panels are held wet after bio-polishing waiting available machine for rinsing/inactivation. To avoid the atmospheric laboratory conditions' effects, the leg panel is kept in hermetically sealed bags.

2.5. Tensile Strength

The tensile strength (breaking force) of samples was determined using the US Standard Grab test method, ASTM D 5034-09 (2017): Standard Test Method for Breaking Strength and Elongation of Textile Fabrics [30].

The tensile strength loss (%) of the samples was measured using Equation (1):

Table 4. Factors and levels related to bio-polishing treatment.

Coded input	Uncoded input parameters	Levels			
		1	2	3	4
x(1)	Fabric ID	I.	II.	I.	-
x(2)	Treatment time, min	5	15	-	-
x(3)	Treatment temperature, ˚C	30	50	-	-
x(4)	Enzyme concentration, mL/L	0.7	1.5	-	-
x(5)	Storage time, day	0	1	-	-
x(6)	Inactivation step	Not applied (N)	Applied (A)	-	-
x(7)	Enzyme ID	A	B	C	D

Dosed volume of the enzyme (mL/L).

$$\text{TSL} = (\text{TSI} - \text{TSF})/(\text{TSI}) \times 100 . \quad (1)$$

TSL: Tensile strength loss;

TSI: Tensile strength of fabric before bio-polishing;

TSF: Tensile strength of fabric after bio-polishing.

The fabric before bio-finishing is rinsed, desized, stone washed, rinsed and dried.

The fabric before bio-finishing is rinsed, sized, stone washed, bio-finished, rinsed, inactivated/not, rinsed, spun, and dried.

The fabric is desized with the enzyme Lava sperse Ktg with a concentration of 0.5 g/L during 10 min at a temperature of 40˚C. Then stonned with a fair mixture of used and new pumice stones (200%) at a temperature of 40˚C for 40 min. Finally, it dried at temperature of 60˚C during 50 min.

An average of three values is measured and outliers are rejected.

2.6. Tear Strength

Tear strength was evaluated according to ASTM D 1424-1996: Standard Test Method for Tearing Strength of Fabrics by Falling-Pendulum Type (Elmendorf) Apparatus. The tear strength is the force required to propagate a single-rip tear starting from a cut in a fabric. The tear strength loss (%) was calculated using Equation (2).

$$\text{TrSL} = (\text{TrSI} - \text{TrSF})/(\text{TrSI}) \times 100 . \quad (2)$$

TrSL: Tear strength loss;

TrSI: Tear strength of fabric before bio-polishing;

TrSF: Tear strength of fabric after bio-polishing.

An average of three values is measured and outliers are rejected.

Different tests were performed in textile standard conditions according to ISO 139:2005. All samples were preconditioned for 24 hours before tensile and tear strength measurements.

2.7. Subjective Evaluation

Five experts in fashion and quality inspection evaluated the finished pants to assess the fabric hand feel, the fabric color, the indigo dye pocket back staining and the fuzziness extent. Pocket back staining is used as an indication of the degree of back staining on the right side of the denim fabric [31]. The members examined each specimen independently and then rated them on a scale from 1 to 5. Following the recommendations of the AATCC evaluation procedure 5-2006: Guidelines for the subjective evaluation of fabric hand, the evaluation is carried out under the same lighting conditions, the evaluators avoided activities before the testing and were relaxed. The evaluators were unaware of sample bio-polishing conditions. An average of five notes is measured from individual marks. This method was used previously to evaluate the stone washing effect on denim fabrics by a panel of five randomly selected persons [26]. The cited properties were assessed by comparing the original sample (before bio-finishing) with bio-polished samples and rated using the following procedure (**Table 5**).

Table 5. Rating procedure.

(a)

Rate	Pocket back staining	Fastness properties
5	No stain	Excellent
4	Very slightly stained	Good
3	Slightly stained	Fair
2	Moderately stained	Poor
1	Strongly stained	Very poor

(b)

Rate	Color change	Fastness properties
5	Equal	Excellent
4	Slight	Good
3	Noticeable	Fair
2	Considerable	Poor
1	Much	Very poor

(c)

Rate	Visual appearance	Fuzziness properties
5	No fuzziness	Excellent
4	Slight fuzziness	Good
3	Moderate fuzziness	Fair
2	Severe fuzziness	Poor
1	Very severe fuzziness	Very poor

(d)

Rate	Hand feel	Hand properties
5	Soft	Excellent
4	Pleasant	Good
3	Comfortable	Fair
2	Empty feeling	Poor
1	Hard	Very poor

3. Results

The aim of this research was to decide whether to maintain using enzyme A or to replace it by another one (denoted B, C or D) which allows a minimum loss in fabric strength, low fuzziness extent, an acceptable color change, a minimum indigo dye back staining, and a good hand feel.

3.1. Fabric Strength Loss

Table 6 comprises the average tear strength loss and tensile strength loss through the 384 tests.

3.2. Effect of Variables on Tear Strength and Tensile Strength Loss

Analysis of variance, ANOVA, was used to determine the significance of each variable and their interactions on the response. In this section, we will only discuss the case of tensile strength loss in weft direction as example. The remaining responses will be treated with the same way. Table 7 presents variance analysis used to determine the effect of the bio-polishing treatment on the tensile strength loss in weft direction. Residual plots, diagnostic statistic for unusual observations and the model summary statistics were analyzed to determine if the regression model chosen provided a good fit to the experimental data obtained. From Table 7, the p-values for the variables *i.e.*, fabric ID; treatment time; storage time; inactivation step and enzyme type had statistically significant effect on weft direction tensile strength loss (p-value < 0.05). Of the nine two-way interactions, only interaction of fabric ID and inactivation step was statistically significant on tensile strength loss since its p-value was less than the level of significance (at $p = 0.05$). Three-way inter-action given by the model had a significant effect ($p < 0.05$) on tensile strength loss in weft direction.

As mentioned by TIGALANA, DAN, analysis of variance (ANOVA) was used to deter-mine the adequacy of the model using F-value and p-value, the significance of the model was determined by evaluating the F-value expressed as the square to residual error ratio of the mean model. The model F-value is the ratio of mean square for the individual term to the mean square for the residual. If the determined F-value is found to be greater than that of the tabulated F-value, then the model is a strong experimental data predictor [32].

The R-sq value equal to 77.21 was acceptable, an indication that the model

Table 6. Average tear and tensile strength loss obtained from the design of experiments.

Fabric properties		Fabric I.	Fabric II.	Fabric III.
Tensile strength loss, %	Warp	21.87	19.73	18.46
	Weft	16.77	11.36	10.80
Tear strength loss, %	Warp	19.51	18.63	15.96
	Weft	13.45	14.01	9.87

Table 7. Variance analysis for tensile strength loss in weft direction (%) after bio-polishing.

Source	DF	Adj SS	Adj MS	F-Value	p-Value
Model	28	40963.2	1463.0	42.95	0.000
Linear	10	33074.1	3307.4	97.11	0.000
x(1)	2	14028.2	7014.1	205.93	0.000
x(2)	1	29.3	29.3	0.86	0.354
x(3)	1	16.6	16.6	0.49	0.486
x(4)	1	64.8	64.8	1.9	0.169
x(5)	1	222.2	222.2	6.52	0.011
x(6)	1	17203.3	17203.3	505.09	0.000
x(7)	3	1509.7	503.2	14.77	0.000
2-way interactions	12	6623.8	552.0	16.21	0.000
x(1) × x(2)	2	119.5	59.8	1.75	0.175
x(1) × x(4)	2	24.3	12.1	0.36	0.701
x(1) × x(6)	2	6369.2	3184.6	93.5	0.000
x(2) × x(4)	1	5.1	5.1	0.15	0.699
x(2) × x(5)	1	1.7	1.7	0.05	0.821
x(2) × x(6)	1	4.9	4.9	0.14	0.705
x(3) × x(4)	1	85.6	85.6	2.51	0.114
x(3) × x(5)	1	0.3	0.3	0.01	0.923
x(4) × x(5)	1	13.2	13.2	0.39	0.535
3-Way Interactions	6	1265.4	210.9	6.19	0.000
x(1) × x(2) × x(4)	2	656.1	328.1	9.63	0.000
x(1) × x(2) × x(6)	2	287.8	143.9	4.22	0.015
x(2) × x(4) × x(5)	1	154.0	154.0	4.52	0.034
x(3) × x(4) × x(5)	1	167.5	167.5	4.92	0.027
R-sq = 77.21%, R-sq (adj) = 75.41					

DF: The total degrees of freedom; Adj SS: Adjusted sums of squares; Adj MS: Adjusted mean squares; p-value ≤ α: The differences between some of the means are statistically significant; p-value > α: The differences between the means are not statistically significant.

used explained 77.21% of the variance in tensile strength loss (%) after bio-polishing. According to Aklilu Azanaw *et al.* the R^2 (R-sq value) values found for optimum absorbency, weight loss and tensile strength are 72.43%, 90.27% and 99.66% respectively [33]. This is depicted in **Figure 1** where most of the data points fell closer to the fitted regression line, which further showed that the model chosen fits experimental data extremely well.

Residuals versus fits plot [34] shown in **Figure 2** was used to check whether the model used met the model assumptions (*i.e.*, residuals are randomly distributed and have a constant variance). As it is shown, the points fell randomly on both sides of 0 and there was no recognizable pattern in the points which was an indication that the model satisfied all the model assumptions. This further validated the fitted model indicating that there was no abnormal occurrence during

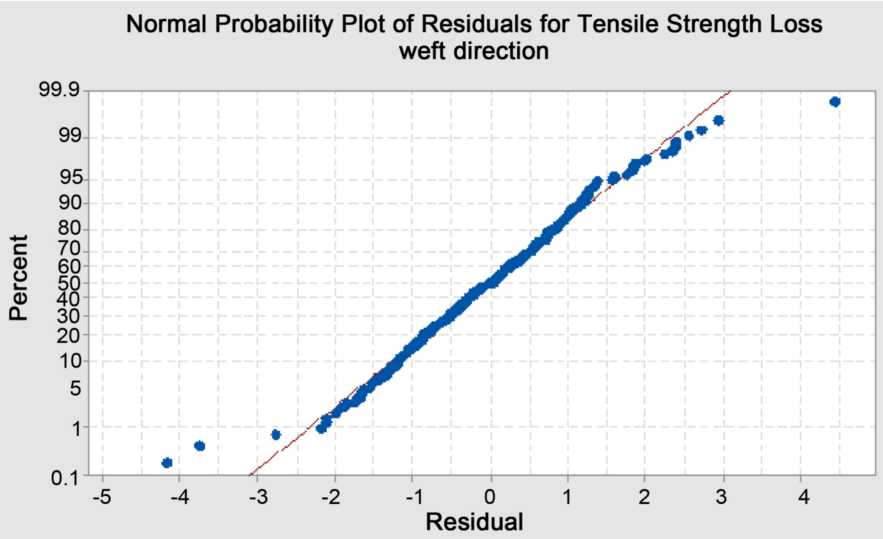

Figure 1. Normal plot of residuals for tensile strength loss in weft direction (%).

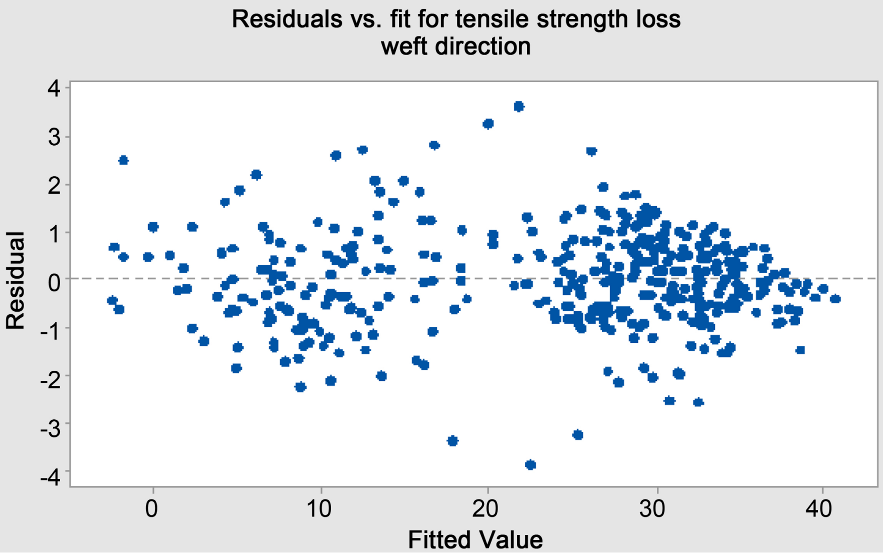

Figure 2. Residuals vs fit for tensile strength loss.

data collection, and residuals had equal variance [32].

Residual versus order plot (**Figure 3**) was used to analyze the order in which experimental data was collected and to verify the assumption that residuals were independent from each other. From the graph, no pattern or trend was observed when the residuals were displayed in time order of data collection [35]. Moreover, the residuals randomly fell around the center line, indicating that the residuals were independent one another, thus were not correlated [36]. Therefore, the model chosen to fit experimental data was satisfactory and fulfilled all model assumptions.

Graphs of fitted mean of variables (**Figures 4(a)-(d)**) were used to determine the effect of in-dependent variables on the response. Similarly, the effect of interaction between independent variables on the response was depicted in **Figures 5(a)-(d)**. **Figures 4(a)-(d)**, plotted in Minitab software, show the main effects of each process parameter on the fabric strength loss.

Because there were many ways interaction terms that were significant, the main effect cannot be considered without including the interaction effects. Only the two significant two-way interactions could be examined from **Figure 5**.

3.3. Experimental Optimization

For experimental optimization, we used the desirability functions shown in **Figure 6** in which we considered the target Y_{target}, the different acceptance intervals [Y_{min}, Y_{max}], the importance of every property Y_i in the definition of the global quality and the requirement [37].

These desirability functions d_i can be classified into three types: desirability function to minimize, to maximize and to reach a property target value. Thus, when we want to minimize a property Y_i, such as tensile strength loss and tear strength loss, we use the desirability function shown in **Figure 6**. Where d_i is

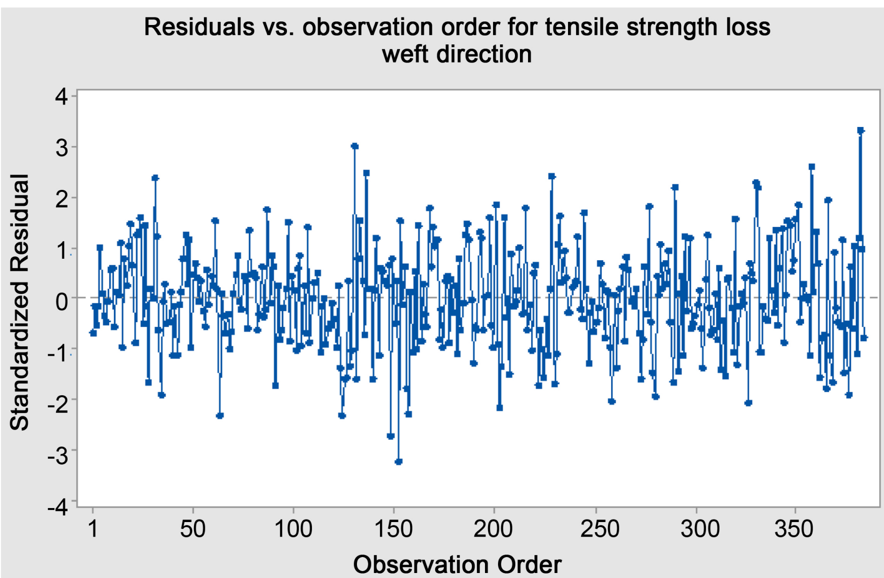

Figure 3. Residuals vs observations order for tensile strength loss percentage.

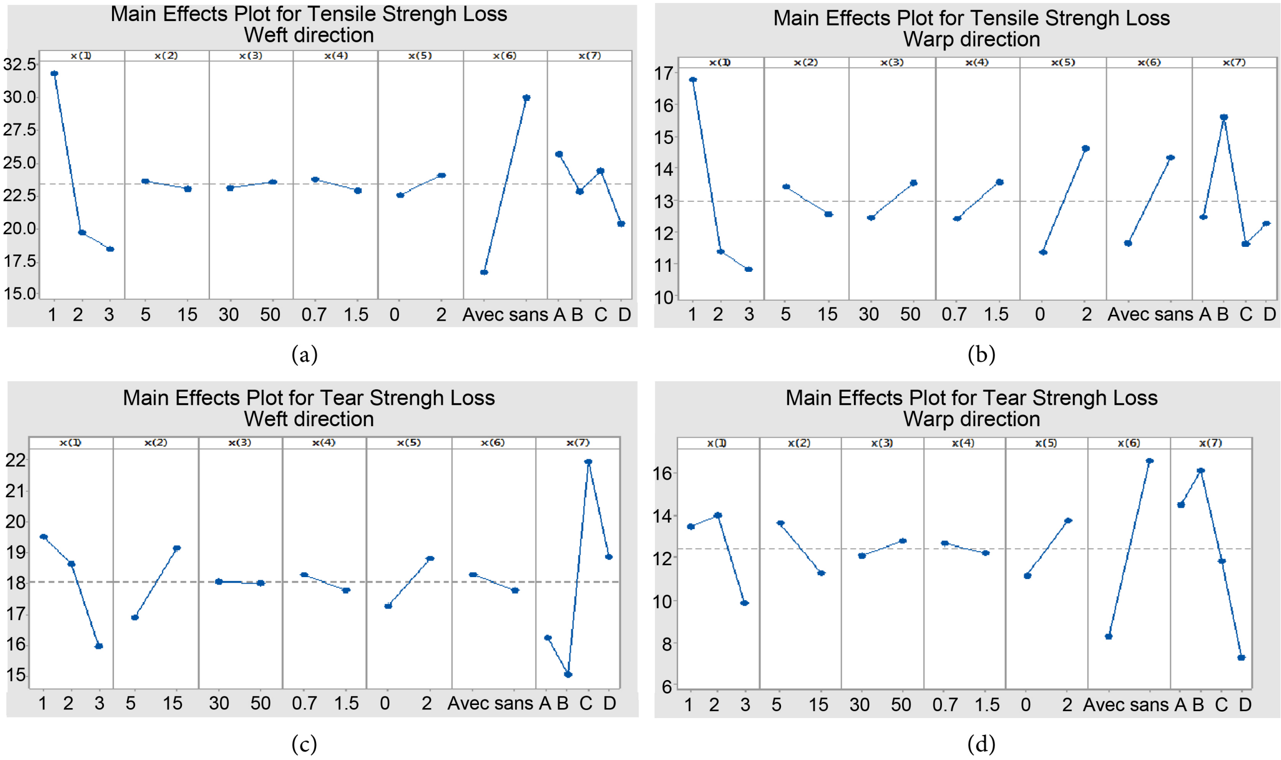

Figure 4. Main effect plots for tensile and tear strength loss.

calculated in Equation (3).

$$d_i = \begin{cases} 1 & \text{if } Y_i < Y_{\text{target}} \\ \dfrac{(Y_i - Y_{\max})^t}{Y_{\text{target}} - Y_{\max}} & \text{if } Y_{\text{target}} \le Y_i \le Y_{\max} \\ 0 & \text{if } Y_i \ge Y_{\max} \end{cases} \quad (3)$$

The manufacturer's requirement is represented in this function by the t value that is proportional to the requirement. We consider that if $t = 1$, the consumer is fairly required. On the other hand, if $t \gg 1$, we estimate that the manufacturer is very much required and finally when $t \ll 1$, we can consider that the manufacturer is very little required [38].

Every transformation d_i gives the equivalent of a satisfaction degree calculated by the desirability function of the property while considering the fixed target and the acceptance intervals. For each property affecting the global quality, we calculated the satisfaction degree d_i and attributed a relative weight to indicate the property's importance. We grouped these different satisfaction degrees by using the Derringer and Suich desirability function defined by Equation (4).

Where d_i the individual property's desirability function Y_i, $i \in [1, \cdots, m]$, w_i the weight of the property Y_i in the d_G desirability function (considered constant and equal to 1), $w = \sum w_i$ and m is the number of properties. The compromise between the properties is better when d_G increases, it becomes perfect when d_G is equal to 1 33.

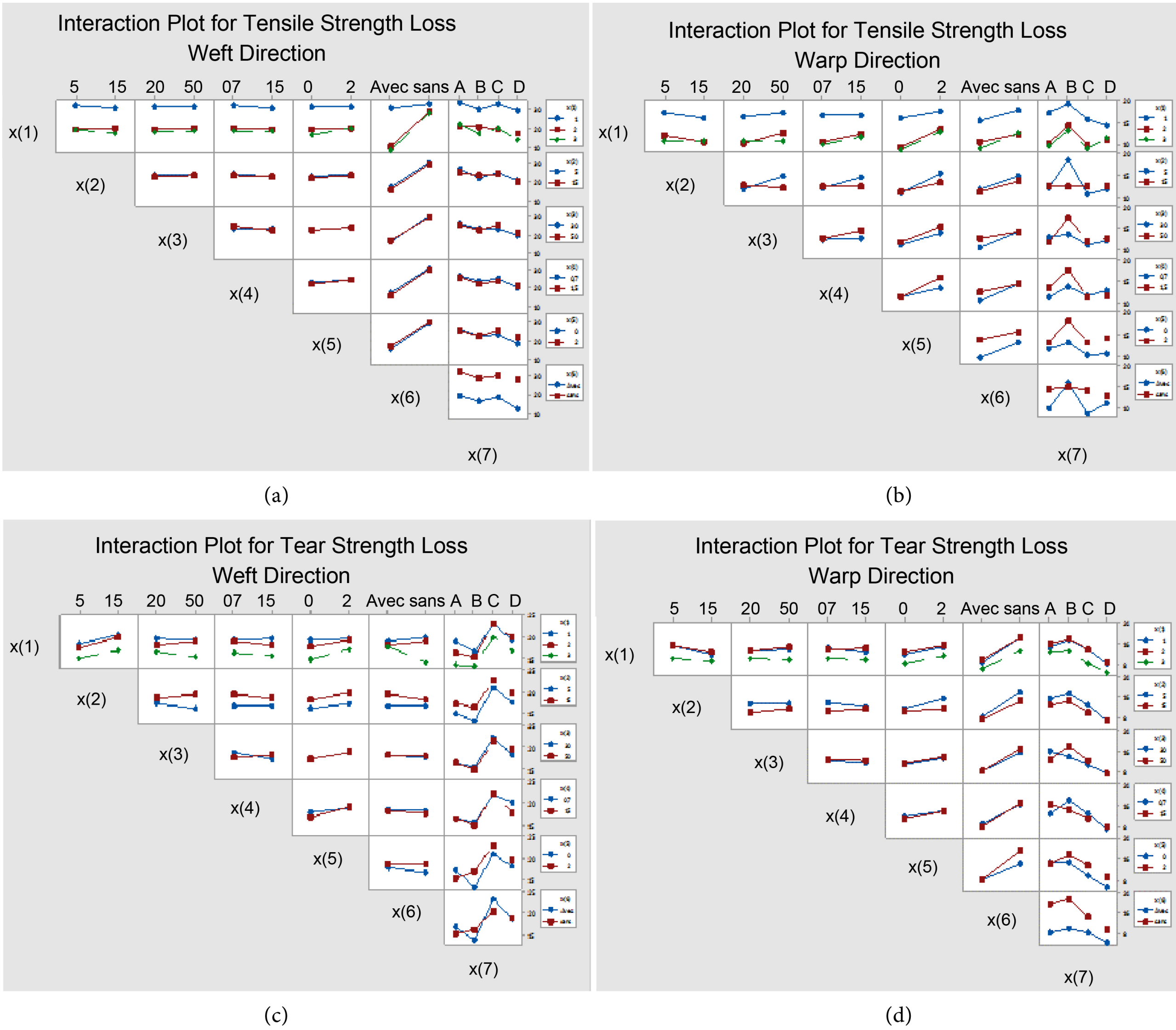

Figure 5. Interaction plot for fabric strength loss.

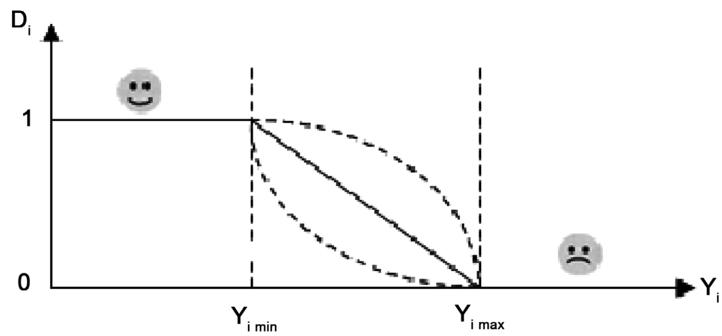

Figure 6. Desirability function to minimize.

MINITAB® 17.1.0 statistical software package generated regression equations which demonstrated the empirical relationship between the response values and parameters varied. The behaviors of the treated fabrics properties as function of the studied inputs can be explained according to the multilinear regression equations, which were necessary for multiple response optimization using desirability functions.

$$d_G = \sqrt[w]{d_1^{w_1} \times d_2^{w_2} \times \cdots \times d_m^{w_m}} \quad (4)$$

3.4. Laboratory Confirmation Tests

Laboratory confirmation is an important step after optimization and before the pilot tests. In fact, fabrics face very high fluctuation in the tensile and tear strength after stone washing. Therefore, 15 leg panels from each fabric are tested. Fabrics I. II. III. are treated with the enzyme D applied according to the optimum process parameters presented in Table 8. Results were compared to those of the same samples (15 leg panels) treated with enzyme A, applied at 40˚C for 10 min. with 1.3 mL/L enzyme, and followed directly by inactivation step (industrial standard process). Fabric IV was used here to confirm our results.

Performance indicators help textile companies in selecting processes and products [38].

The performance of the enzyme D (EP) is calculated according to Equation (5).

$$\text{PFED} = (\text{STED D} - \text{STED})/(\text{STEA}) \times 100 \quad (5)$$

PFED: Enzyme D performance;

STED: Strength of fabric treated with enzyme D;

STEA: Strength of fabric treated with enzyme A.

Through this equation, the performance in terms of tear or tensile strength is obtained using enzyme D and compared with that given by enzyme A.

3.5. Pilot Results

Enzyme D is applied according to the optimum process parameters presented in Table 8. Results were compared to those of the pants treated with enzyme A, applied at 40˚C for 10 min. with a concentration of 1.3 mL/L, and followed directly by inactivation step (Table 3).

4. Discussion

4.1. Fabric Strength Loss

Table 6 shows that warp yarns are more affected by bio-polishing treatment than

Table 8. Optimized bio-polishing parameters.

Fabric ID	Treatment time, min	Treatment temperature, ˚C	Enzymes concentration, mL/L	Storage time, day	Inactivation step	Enzyme type
Fabric I.	10	30	1	0	Applied	D
Fabric II.	10	40	1	0	Applied	D
Fabric III.	10	40	0.7	0	Applied	D

weft ones. The maximum tensile strength loss is 28.1% in warp direction, and 17.2% in direction way for the three fabrics. The maximum tear strength loss is 20.2% in warp way, and 15.4% in weft way through the three fabrics. The tensile and the tear strength loss seem to be important compared to previous results of Karmakar [12]. The fabric I. made from cotton fibers is the most damaged by the bio-polishing treatment. The fabric III displays lower strength loss. In fact, it is understandable that cellulase enzymes do not attack polyester. Alternatively, cellulase enzymes cannot attack polyester.

4.2. Effect of Variables on Tear Strength and Tensile Strength Loss

In the main effect plots, if the line for a particular parameter is nearly horizontal, then the parameter has no significant effect. On the other hand, a parameter for which the line has the highest inclination will have the most significant effect [39].

It can be seen from **Figures 4(a)-(d)** that fabric ID, enzyme type and inactivation step affect the fabric strength to a greater extent. Indeed, cellulase enzymes affect various spun yarns and various fabric constructions in different degrees [40] [41]. On the other hand, cellulose hydrolysis depends on cellulase activity, cellulase composition, and cellulose substrate [26].

Of the seven variables, treatment temperature had an insignificant effect on the tested responses. This result is favorable for cellulase application at industrial scale where high temperatures are undesirable. Besides, enzyme concentration has a negligible effect on the fabric strength. This result can be explained by the fact that with the concentration range tested [0.7 mL/L - 1.5 mL/L] and with the liquor ratio used (1:10), the localized concentration of effective cellulase on the protruding fibers is higher than the concentration of cellulase in the yarns, which are therefore not affected.

In our work, bio-polishing was carried out on desized, scoured and stone washed denim fabrics. These treatments caused the important percentage of tensile and tear strength loss. The aim of the work is to compare commercial neutral enzymes for industrial goals. Then, fabrics undergo the same treatments and at the last step different enzymes are used for bio-polishing.

As mentioned by Choudhury [2], cellulases hydrolyze the microfibrils protruding from the surface of the yarn because they are most susceptible to enzymatic attack. In this paper enzyme concentration is relatively low and cannot causes an important percentage of tensile and tear loss. Results (**Figure 4(a)**) demonstrate a little bit variation.

Finally, reducing storage time of bio-polished fabrics and inactivation step may decrease the fabric damage. This result was reported by Buschle *et al.* [42], who found that cotton and linen fabrics show a significant reduction in tensile energy after 24 hours of cellulase treatment.

Figure 5 confirms the presence of two-way interactions determined previously as example in **Table 7**. Interaction plot indicates weak interactions between two factors when there are two semi-parallel lines. The greater the departure of the lines from the parallel state, the higher the degree of interaction and more this degree is raised. A significant interaction connects enzyme type (x7) and fabric ID (x1). The interaction shows that the relationship between the fabric strength loss and the enzyme type depends on the fabric ID *i.e.*, yarns, construction, and composition.

The regression and variance analysis obtained using "stepwise option" show adequate regression models (high coefficient of determination R^2 > 85%) and highly meaningful at 95% significance level. Indeed, compared with the published works dealing with the significance of the coefficient of regression [43] [44] and they have been considered as highly significant (the R^2 values ranged from 68% to 72%).

4.3. Experimental Optimization

The optimization results presented in **Table 8** show that a low-temperature enzymatic bio-polishing process was developed using cellulase enzyme type D. In fact, cellulase enzyme D has provided the minimum loss in tear and in tensile strength for the three tested fabrics. Inactivation step must be applied to avoid fabric damage and storage time should be reduced for the same reason. Treatment time could be maintained to 10 min. Nevertheless, enzyme concentration and treatment temperature vary from one fabric to another.

4.4. Laboratory Confirmation Tests

Figure 7 and **Figure 8** show that the tensile and the tear strength of the tested leg panels are higher after being exposed to cellulase enzymes D, also for fabric 4. This trend confirms our previous results. The performance indicator used encouraged garments' manufacturers to test enzyme D at a pilot and at industrial scale since it reaches 26.9% (see **Figure 8(a)**). The tensile strength has been decreased after bio-polishing and this was consistent with other previous studies [9] [26]. Although the tear and the tensile strength are acceptable in warp direction [30], it is critical in weft direction, especially when the tear strength reaches 1800 cN using enzyme A [45].

4.5. Pilot Results

Subjective evaluation of the hand feeling shows that the samples treated with enzyme D have, in general, a more pleasant feeling than those bio-polished with enzyme A (darker grey cells). It is observed that enzyme D exhibits comparable activity in reducing fuzziness of the fabric to that of enzyme A. This is due to efficient enzyme treatment confirming effective removal of protruding loose fibers. This result may be further supported by scanning electron microscopy or yarn hairiness measurement. Enzyme D gives less dye redeposition then enzyme A. In fact, enzyme D may present a lower affinity to indigo and weaker binding to cotton cellulose [31] [46]. However, samples treated with enzyme D exhibit

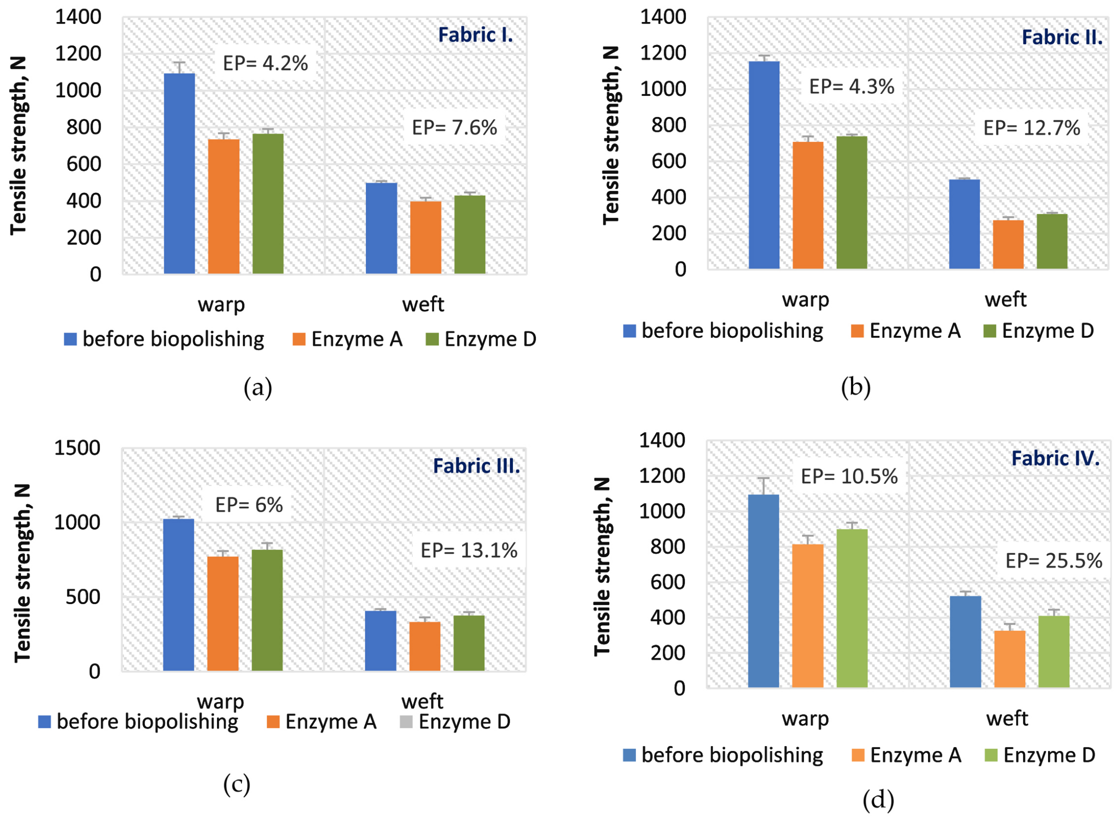

Figure 7. Tensile strength of fabrics treated with enzyme A and D.

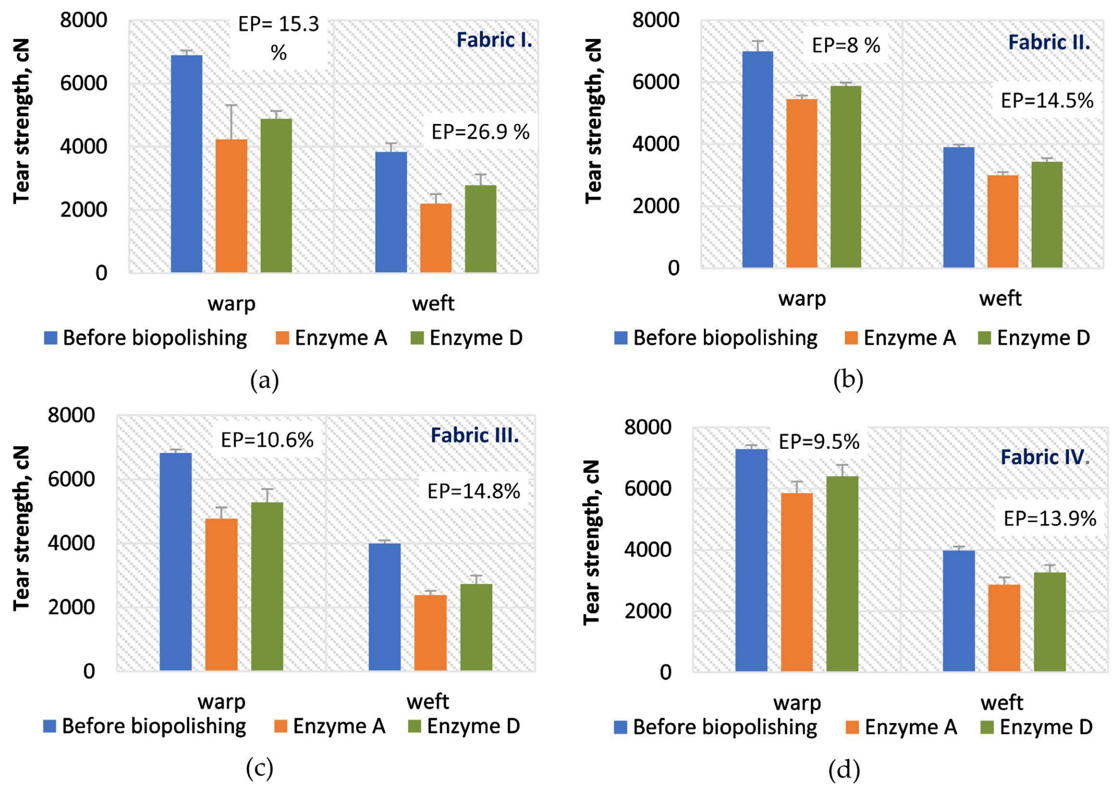

Figure 8. Tear strength of fabrics treated with enzyme A and D.

noticeable color change compared to samples treated with enzyme A which exhibit slight color change (**Table 9**).

To conclude, enzyme D applied at the optimum conditions shown in **Table 8** is not appreciated by all experts, especially if we focus on the fabric color change. Therefore, a total subjective evaluation of the effect of temperature and enzyme concentration is carried out in a pilot scale to ameliorate experts' rate values before industrial tests.

Table 10 shows the total performance of enzymes A and D evaluated by industry experts. In this total evaluation, experts use general expressions such as good or bad or a numerical rate 1 or 5 to classify the total quality of finished pants [47]. It is noticed that the expert's note depends on fabric type, enzyme type, enzyme concentration, treatment temperature, and desired effects. In fact, cellulase enzyme can cause non-homogeneous surface removal of indigo dye trapped inside the fibers creating a desired fading and worn look. Bio-finished pants were accepted by the experts within the enzyme D concentration range 0.7 mL/L - 1 mL/L at a temperature 30˚C - 40˚C.

4.6. Industrial Results

Cellulases are sensitive to mechanical action and completely different performances can be obtained using the same enzyme, same dosage, same time and same temperature, in a different washing machine. All these factors should be considered when designing the right washing protocol, especially when the agitation rate and the textile load increase the apparent activity of cellulases and the extent of cellulose hydrolysis 15.

Table 9. Subjective assessment of performance of enzyme in bio-polishing of denim pants treated under optimum conditions.

Expert rating of properties	Enzyme A				Enzyme D			
	Hand feel	Color changes	Fuzziness	Back-Staining	Hand feel	Color changes	Fuzziness	Back-staining
Fabric I.	2	4	4	2	4	3	4	5
Fabric II.	2	4	4	3	4	3	4	4
Fabric III.	1	3	3.5	3	4	2	4	4

Table 10. Total subjective assessment of enzyme performance in bio-polishing of denim pants function of enzyme concentration and temperature.

Enzyme type	Enzyme D						Enzyme A
Enzyme concentration, mL/L	0.5		0.7		1		1.3
Temperature, ˚C	30	40	30	40	30	40	40
Fabric I.	3.25	3.00	4.00	4.00	4.00	4.00	3.00
Fabric II.	2.00	3.00	4.00	4.00	4.00	3.75	3.25
Fabric III.	2.00	3.00	4.00	3.5	3.25	3.00	2.50

A standard process is applied with the enzyme D consisting of: a concentration of 0.7 mL/L for 10 min. at 30˚C. Enzyme A is applied with a concentration of 1.3 mL/L for 10 min. at 40˚C.

It is noticed from the total subjective evaluation that experts appreciated enzyme D. Enzyme D can replace the enzyme A according to experts' notes (4.25/5 against 3.75/5). Besides, the use of the enzyme D allows a decrease of process temperature from 40˚C to 30˚C and a decrease of enzyme concentration from 1.3 mL/L to 0.7 mL/L. Moreover, it removes the weakened fuzz fibers from denim fabrics resulting in softer, smoother feel with a slight change on denim shade and low back staining.

5. Conclusions

To summary, in this paper, the effect of commercial cellulase enzymes and the optimization of the bio-polishing process of denim pants were discussed. The success of bio-polishing in industry depended on five important aspects: minimum loss of fabric strength, minimum extent of fuzzing, acceptable color changes, an ideal fabric hand feel and no back-staining problems. The first aspect required the efficient enzyme's activity which can be ensured by accurate control of parameters. The other aspects can be ensured by a subjective control of laboratory swatches, pilot and industrials products.

Bio-polishing was carried out on desized, scoured and stone washed denim fabrics using four types of commercial enzymes. The influence of bio-polishing process parameters: enzyme concentration, treatment time and temperature, inactivation step and storage time were investigated simultaneously on the fabric tear and tensile strength loss using factorial experimental design in laboratory scale. Enzyme D (Lava Cell NSZ) was selected using desirability function and an optimized process is suggested. Laboratory confirmation tests were carried out to validate our results. For pilot and industrial tests, a subjective evaluation of the hand feel, the color change, the extent of fuzziness and the back-staining show that the samples (pants) treated with the proposed enzyme D have, in general, more pleasant properties than those of enzyme A. Using selected enzyme D, the manufacturer was able to gain 10˚C in the bio-polishing process, to reduce the concentration of the enzyme in bio-polishing recipe to 0.5 mL/L and 12% in the price of bio-polishing process.

For the first time the bio-polishing treatment was carried out at a temperature of 30˚C which gives a huge reduction in terms of energy and very good mechanical garment properties. Yarn hairiness and flexural rigidity measurement, color fastness properties and color measurements of the bio polished denim garments will be the subject of our future work.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

References

[1] Ibrahim, N.A., Allam, E.A., El-Hossamy, M.B. and El-Zairy, W.M. (2008) Enzymatic Modification of Cotton/Wool and Viscose/Wool Blended Fabrics. *Journal of Natural Fibers*, **5**, 154-169. https://doi.org/10.1080/15440470801929648

[2] Roy Choudhury, A.K. (2014) Sustainable Textile Wet Processing: Applications of Enzymes. In: Muthu, S., Ed., *Roadmap to Sustainable Textiles and Clothing. Textile Science and Clothing Technology*, Springer, Singapore, 203-238. https://doi.org/10.1007/978-981-287-065-0_7

[3] Araújo, R., Casal, M. and Cavaco-Paulo, A. (2008) Application of Enzymes for Textile Fibres Lothing. *Biocatalysis and Biotransformation*, **26**, 332-349. https://doi.org/10.1080/10242420802390457

[4] Mojsov, K. (2011) Application of Enzymes in Textile Industry: A Review. *II International Congress* "*Engineering, Ecology and Materials in the Processing Industry*", Jahorina, 9-11 March, 230-239.

[5] Costa, C., Azoia, N.G., Silva, C. and Marques, E.F. (2020) Textile Industry in a Changing World: Challenges of Sustainable Development. *U.Porto Journal of Engineering*, **6**, 86-97. https://doi.org/10.24840/2183-6493_006.002_0008

[6] Guo, Y., Sun, Z., Guo, X., Zhou, Y., Jiang, L., Chen, S. and Ma, J. (2019) Study on Enzyme Washing Process of Hemp Organic Cotton Blended Fabric. *International Journal of Clothing Science and Technology*, **31**, 58-64. https://doi.org/10.1108/IJCST-08-2017-0117

[7] Sajith, S., Priji, P., Sreedevi, S. and Benjamin, S. (2016) An Overview on Fungal Cellulases with an Industrial Perspective. *Journal of Nutrition & Food Sciences*, **6**, Article ID: 1000461. https://doi.org/10.4172/2155-9600.1000461

[8] Ali, H., Hashem, M., Shaker, N., Ramadan, M., El-Sadek, B. and Hady, M.A. (2012) Cellulase Enzyme in Bio-Finishing of Cotton-Based Fabrics: Effects of Process Parameters. *Research Journal of Textile and Apparel*, **16**, 57-65. https://doi.org/10.1108/RJTA-16-03-2012-B006

[9] Noreen, H., Anjum, Z.M., Ali, S. and Hussain, T. (2014) Optimization of Bio-Polishing of Polyester/Cotton Blended Fabrics with Cellulases Prepared from *Aspergillus niger*. *Indian Journal of Biotechnology*, **13**, 108-113.

[10] Uddin, M.G. (2016) Effect of Biopolishing on Dye Ability of Cotton Fabric—A Review. *Trends in Green Chemistry*, **2**, 1-5. https://doi.org/10.21767/2471-9889.100011

[11] Sankarraj, N. and Nallathambi, G. (2017) Effect of Biopolishing on Structural Degradation and Physical Properties of Cellulose. *Journal of the Serbian Chemical Society*, **82**, 567-578. https://doi.org/10.2298/JSC161123031S

[12] Karmakar, S.R. (1999) Application of Biotechnology in the Pre-Treatment Processes of Textiles. In: Karmakar, S.R., Ed., *Textile Science and Technology*, Vol. 12, Elsevier, Amsterdam, 418-440. https://doi.org/10.1016/S0920-4083(99)80016-4

[13] Sharma, A., Tewari, R., Rana, S. and Soni, S. (2016) Cellulases: Classification, Methods of Determination and Industrial Applications. *Applied Biochemistry and Biotechnology*, **179**, 1346-1380. https://doi.org/10.1007/s12010-016-2070-3

[14] Bahtiyari, M.I., Duran, K., Körlü, A. and Perincek, S. (2010) Usage of Commercial Cellulases in Bio-Polishing of Viscose Fabrics. *Textile and Apparel*, **20**, 57-64.

[15] Jevšnik, S., Stjepanovič, Z., Heikinheimo, L. and Gotlih, K. (2011) Effect of Enzyme Treatments on Interlock Knitted Fabric. *International Journal of Clothing Science and Technology*, **23**, 61-73. https://doi.org/10.1108/09556221111096741

[16] Jevšnik, S., Fakin, D., Heikinheimo, L. and Stjepanovi, Z. (2012) Changes in a Knitted Fabric's Surface Properties Due to Enzyme Treatments. *Fibers and Polymers*, **13**, 371-379. https://doi.org/10.1007/s12221-012-0371-x

[17] Mangat, A., Hes L. and Bajzik, V. (2017) Effect of Bio-Polishing on Warm-Cool Feeling of Knitted Fabric: A Subjective and an Objective Evaluation. *Autex Research Journal*, **17**, 95-102. https://doi.org/10.1515/aut-2016-0001

[18] Hassan, T.M. and Zaghlol, T.M. (2019) Influence of Cellulase Enzyme on Some Properties of Knitted Children's Wear. *International Design Journal*, **9**, 183-187.

[19] Uddin, M.G. (2015) Effects of Bio-Polishing on the Quality of Cotton Fabrics Using Acid and Neutral Cellulases. *Textiles and Clothing Sustainability*, **1**, Article No. 9. https://doi.org/10.1186/s40689-015-0009-7

[20] Saleh, S.M., El-Sayed, I. and El-Shikh, A. (2012) Investigating the Impact of Enzymatic Treatment on Mechanical and Chemical Properties of Denim Fabrics. *Research Journal of Textile and Apparel*, **16**, 111-117. https://doi.org/10.1108/RJTA-16-02-2012-B012

[21] Özdil, N., Marmaralı, A. and Kretzschmar, S.D. (2007) Effect of Yarn Properties on Thermal Comfort of Knitted Fabrics. *International Journal of Thermal Science*, **46**, 1318-1322. https://doi.org/10.1016/j.ijthermalsci.2006.12.002

[22] Chikod, S. (1994) Effects of Bio-Finishing on Cotton and Cotton/Wool Blended Fabrics. Texas Tech University, Lubbock, 81 p.

[23] Saravanan, D., Sree Lakshmi, S.N., Senthil, R.K. and Vasanthi, N.S. (2013) Biopolishing of Cotton Fabric with Fungal Cellulase and Its Effect on the Morphology of Cotton Fibres. *Indian Journal of Fibre & Textile Research*, **38**, 156-160.

[24] Sarkar, M., Khalil, E. and Solaiman, M. (2014) Effect of Enzyme Washing Combined with Pumice Stone on the Physical, Mechanical and Color Properties of Denim Garments. *International Journal of Research in Advent Technology*, **2**, 65-68.

[25] Mir, S., Hossain, M., Biswas, P., Hossain, A. and Idris, M.A. (2014) Evaluation of Mechanical Properties of Denim Garments after Enzymatic Bio-Washing. *World Applied Sciences Journal*, **31**, 1661-1665.

[26] Anish, R., Rahman, M.S. and Rao, M. (2007) Application of Cellulases from an Alkalothermophilic *Thermomonospora* sp. in Biopolishing of Denims. *Biotechnology and Bioengineering*, **96**, 48-56. https://doi.org/10.1002/bit.21175

[27] Cavaco-Paulo, A., Morgado, J., Almeida, L. and Kilburn, D. (1998) Indigo Back Staining during Cellulase Washing. *Textile Research Journal*, **68**, 398-401. https://doi.org/10.1177/004051759806800602

[28] Elhalil, A., Tounsadi, H., Elmoubarki, R., Mahjoubi, F.Z., Farnane, M., Sadiq, M., Abdennouri, S., Qourzal, M. and Barka, N. (2016) Factorial Experimental Design for the Optimization of Catalytic Degradation of Malachite Green Dye in Aqueous Solution by Fenton Process. *Water Resources and Industry*, **15**, 41-48. https://doi.org/10.1016/j.wri.2016.07.002

[29] Kumar, L., Reddy, M., Managuli, R.S. and Pai, G. (2015) Full Factorial Design for Optimization, Development and Validation of HPLC Method to Determine Valsartan in Nanoparticles. *Saudi Pharmaceutical Journal*, **23**, 549-555. https://doi.org/10.1016/j.jsps.2015.02.001

[30] Repon, R., Islam, T., Sadia, H.T., Karim, R., Munir, M.U. and Jalil, M.A. (2021) Stretchable Denim Properties Dependency on Industrial Washing Techniques. *Fibres and Textiles*, **28**, 75-81.

[31] Shahid, M., Mohammad, F., Chen, G., Tang, R.-C. and Xing, T. (2016) Enzymatic Processing of Natural Fibres: White Biotechnology for Sustainable Development.

Green Chemistry, **18**, 2256-2281. https://doi.org/10.1039/C6GC00201C

[32] Tigalana, D. (2021) Optimization of Mercerizing Conditions for Enhanced Cotton Fabric. Moi University, Cheptiret, 96 p.

[33] Aklilu, A. and Ketema, A. (2022) Process Optimization for Biosingeing of 100% Cotton Terry Towel Fabric Using Box-Behnken Design. *Advances in Materials Science and Engineering*, **2022**, Article ID: 6920864. https://doi.org/10.1155/2022/6920864

[34] Emili, G-B. (2001) On the Misuse of Residuals in Ecology: Testing Regression Residuals vs. the Analysis of Covariance. *Journal of Animal Ecology*, **70**, 708-711. https://doi.org/10.1046/j.1365-2656.2001.00524.x

[35] Oduor, E.O., Ciera, L.W., Adolkar, V. and Pido, O. (2020) Effect of Conventional Degumming Methods on Eri Silk Fibers Produced in Kenya. *Research Journal of Textile and Apparel*, **24**, 163-182. https://doi.org/10.1108/RJTA-05-2019-0021

[36] Gupta, B.C. and Guttman, I. (2014) Statistics and Probability with Applications for Engineers and Scientists. John Wiley and Sons, Hoboken.

[37] Hadj Taieb, A., Msahli, S. and Sakli, F. (2008) Evaluation of the Consumer's Global Satisfaction of the Knits by Using the Desirability Functions. *Journal of Textile Institute*, **99**, 1-7. https://doi.org/10.1080/00405000701570575

[38] Ren, X. (2000) Development of Environmental Performance Indicators for Textile Process and Product. *Journal of Cleaner Production*, **8**, 473-481. https://doi.org/10.1016/S0959-6526(00)00017-2

[39] Cheriaa, R. and Baffoun, A. (2015) Effects of Cross Linkers Combination, for Three Dimensional Effects, on Denim Garment Properties. *Fibers and Polymers*, **16**, 1150-1155. https://doi.org/10.1007/s12221-015-1150-2

[40] Akaydin, M., Yilmaz, K. and Yimaz, N.D. (2015) Effect of Finishing on Performance Characteristics Of woven and Warp-Knitted Terry Fabrics. *Indian Journal of Fibre and Textile Research*, **40**, 301-307.

[41] Chinta, S.K., Landage, S.M. and Ketan, V. (2012) Effect of Biopolishing Treatment on Various Spun Yarn Knitted Fabrics. *Global Journal of Bio-Science & Biotechnology*, **1**, 287-295.

[42] Buschle, D.G., Zeronian, S.H., Pan, N. and Yoon, M.Y. (1994) Enzymatic Hydrolysis of Cotton, Linen, Ramie, and Viscose Rayon Fabrics. *Textile Research Journal*, **64**, 270-279.

[43] Abghari, R., ShaikhzadehNajar, S., Haghpanahi, M. and Latifi, M. (2004) Contributions of in-Plane Fabric Tensile Properties in Woven Fabric Bagging Behavior Using a New Developed Test Method. *International Journal of Clothing Science and Technology*, **16**, 418-433. https://doi.org/10.1108/09556220410554615

[44] Uçar, N., Realff, M.L., Radhakrishnaiah, P. and Uçar, M. (2002) Objective and Subjective Analysis of Knitted Fabric Bagging. *Textile Research Journal*, **72**, 977-982. https://doi.org/10.1177/004051750207201108

[45] Cheriaa, R. and Baffoun, A. (2015) Optimal Drying and Curing Process for Minimum Loss in Tear Strength of Distressed Look Garments. *Fibers and Polymers*, **16**, 1988-1994. https://doi.org/10.1007/s12221-015-5106-3

[46] Colomera, A. and Kuilderd, H. (2015) Biotechnological Washing of Denim Jeans. In: Roshan, P., Ed., *Denim Manufacture*, *Finishing and Applications*, Elsevier, Amsterdam, 357-403. https://doi.org/10.1016/B978-0-85709-843-6.00012-3

[47] Zeng, X. and Koehl, L. (2003) Representation of the Subjective Evaluation of the Fabric Hand Using Fuzzy Techniques. *International Journal of Intelligent Systems*, **18**, 355-366. https://doi.org/10.1002/int.10092