Effects of Enzyme Concentration in Bio-polishing on Cotton Knit Fabric

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Abstract

In this work, exhaust method was followed on undyed and dyed single jersey cotton knit fabric by Lava Cell NBG enzyme with a varied enzyme concentration keeping all other parameters constant and thereafter different properties like handle, weight loss, absorbency and pilling of treated samples were assessed. Additionally the fastness properties for dyed fabric were evaluated before and after enzyme treatment. The enzyme used in this work was acid stable cellulase enzyme that also worked at neutral condition effectively and compatible with some other enzymes and dyeing processes. The tested samples were treated with various enzyme concentrations like 0.5%, 1%, 1.5 %, 2% and 3% at 600 c temperature for 30 minutes with M:L 1:20 at neutral bath condition with a medium mechanical agitation and thereafter enzyme deactivation was carried out at an elevated temperature. The hand feel of tested sample was optimum with 2% enzyme concentration and weight loss tendency was increased with the increase of concentration % and considerable weight loss was noticed up to 1.5 % conc. range and it was severe with 3% concentration both for undyed and dyed samples. Enzyme treatment after dyeing lowered the pilling resistance properties of dyed materials but escalated color fastness properties. However, the tested samples treated with 1% and 1.5% concentration showed acceptable values in all cases especially from a minimum fiber damage perspective.

Keywords: *Bio-polishing, cellulase enzyme, cotton fabric, weight loss, pilling test, absorbency, color fastness to wash*

1. Introduction

The current growing environmental issues always put demands to discover environmental friendly processes and products. The enzymatic treatment on textile substrates is a kind of novel innovation that has reduced environmental pollution amazingly as the enzymes used in such processes are readily bio-degradable.

Now a day's enzymes have found diverse application areas in textile processing. Most popular uses are on denim fabrics in stone washing and on cellulosic textile materials in surface modification to improve their appearance and handle. The process of treating with cellulase enzymes is termed as bio-polishing which is normally carried on cotton goods (T. Hobberg, and S. Thumm, Report-1999). Bio-polishing eliminate projecting fibers and slubs from fabric surface and thus significantly reduces pilling, softens fabric hand and provides smoother surface appearance.

Bio- polishing has also shown its potential actions on regenerated cellulose fabrics especially on lyocell. The disadvantages of bio-polishing are the formation of fiber dust, and in worst case, loss of weight and tear strength (S. K. Laga, A. I. Wasif, and K. Shah, Report-2010).

Apart from bio-polishing, enzymes also have some effective applications in textile pretreatments. The use of amylase enzyme is first introduced in desizing cotton fabric to hydrolyse starch and this is still used extensively (K. J. V. Vardhini, and N. Selyakumar, Report 2009). In scouring, pectinase enzyme is used to break down the pectin in the cotton and thus assists in the removal of waxes, oils and other impurities. After bleaching process with hydrogen peroxide, catalase enzyme can be used in removing residual peroxide which can reduce water and energy consumption. In combined scouring and bleaching process, two enzymes like amyloglucosidase and glucode oxidase can be used in a single bath to obtain the similar effects of conventional combined scouring- bleaching process (K. J. V. Vardhini, and N. Selyakumar, Report 2009).

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1.1 Factors Influence Catalytic Action of Enzymes

Enzymes perform catalytic action on cotton and hydrolyse the celluloses which are strongly influenced by some factors like concentration, pH, temperature, time and mechanical agitation.

1.1.1 Effect of pH

The working condition for a typical enzyme at an optimal ph solely depends on its origin. Trichoderma based enzymes like acid cellulases work best at ph 4.5 -6, while enzymes from Humicola like neutral cellulases are more active at ph 6-6.5 (M. Ueda, H. Koo, and T. Wakida, "Cellulase Treatment of Cotton Fabrics, Part II: Inhibitory Effect of Surfactants on Cellulase Catalytic Reaction", Textile Research Journal, Vol. 64, No. 10, pp. 615-618, American Association of Textile Chemists and Colorists, Report-1999).

1.1.2 Effect of Temperature

Temperature also plays a vital role in enzymatic reaction as at low temperatures, the reaction rate is slower than the desired, but an elevated temperature can deactivate enzyme. In very high temperature due to enough energy formation, enzymes lose its catalytic ability as its molecular alignments alter (M. Ueda, H. Koo, and T. Wakida, "Cellulase Treatment of Cotton Fabrics, Part II: Inhibitory Effect of Surfactants on Cellulase Catalytic Reaction", Textile Research Journal, Vol. 64, No. 10, pp. 615-618, American Association of Textile Chemists and Colorists, Report-1999).

1.1.3 Effect of Concentration

Enzyme concentration needs to be ideal to get optimal effects keeping others adverse effects minimum. As the increase in concentration of cellulase enzyme advances weight loss significantly, causes an increase in strength loss and also fabric thickness is reduced (S. K. Laga, A. I. Wasif, and K. Shah, Report-2010).

1.1.4 Effect of M: L Ratio

Material to liquor ratio also shows substantial effect on bio-polishing. As the liquor ratio increases the bath concentration of cellulase enzyme decreases and the fabric weight loss decreases. The dilution affects substantially enzymatic activity (S. K. Laga, A. I. Wasif, and K. Shah, Report-2010).

1.1.5 Effect of Agitation

Mechanical agitation is another important factor to consider in measuring enzyme's performance on substrates. De-pilling of fabrics depends on the amount of cellulose adsorption on materials to remove hydrolysis by-products and expose new fiber areas to attack Fabric treated in jet and winch machine using same type of enzyme and similar process conditions show different level of effects due to the difference in agitation provided by these machines.(J. M. Cortez, J. Ellis, and D. P. Bishop) To keep the tensile strength loss minimum in cellulose treatment on fabrics agitation is considered as an important factor (K. J. V. Vardhini, and N. Selyakumar, Report 2009).

1.1.6 Effect of Time

Treatment time also has a greater influence on cotton materials in enzymatic treatment. Fiber damage increases dramatically for a longer treatment time with cellulose enzymes (A. Cavaco-Paulo Report -1998)Longer treatment times with no agitation shows more adverse effects which affects the internal structure of the materials, whereas short treatment times with vigorous agitation also deteriorate the fiber surface properties (K. J. V. Vardhini, and N. Selyakumar, Report 2009). In general, increase in treatment time results in superior weight loss and strength loss and these phenomenon are due to slight decrease in degree of polymerization (A. Cavaco-Paulo Report-1998, K. J. V. Vardhini, and N. Selyakumar, Report 2009).

1.2 Effect on Fabric Properties

In bio-polishing, acid cellulase imparts a number of benefits on cotton goods like improvement in pill resistance, cooler feel, brightness of dyed fabric and softness. Apart from these good things, materials also have to allow some adverse effects like loss in weight and strength (K. J. V. Vardhini, and N. Selyakumar, Report 2009). Moreover the overall appearance and handle of cotton knits also improve by treatment with cellulaes as such treatment removes surface hairs from fabric (S. K. Laga, A. I. Wasif, and K. Shah, Report-2010).Cellulase enzyme treatment significantly improves the fabric smoothness and less friction happens during wear N. (Ozdil, E. Ozdooan, and T. Oktem, Report-2003) Dimensional stability of cotton woven and knit fabrics also improve in cellulase treatment and offer less resistance to bending and stretching (J. M. Cortez, J. Ellis, and D. P. Bishop, Report 2002, R. Mori, T. Haga, and T. Takagishi, Reprt-1999).

Table 1 : Recipe of enzyme treatment (M.F. Hossain, Report-2009)

Recipe 1:	**Recipe 2:**	**Recipe 3:**	**Recipe 4:**	**Recipe 5:**
Conc. of enzyme: 0.5% Sequestering agent:1g/l Wetting agent:1g/l Temperature : 60°C pH : 6.5 Time:30 min M : L : 1:20 Mechanical Agitation: Medium Stirring	Conc. of enzyme: 1% Sequestering agent:1g/l Wetting agent:1g/l Temperature : 60°C pH : 6.5 Time:30 min M : L : 1:20 Mechanical Agitation: Medium Stirring	Conc. of enzyme: 1.5% Sequestering agent:1g/l Wetting agent:1g/l Temperature : 60°C pH : 6.5 Time:30 min M : L : 1:20 Mechanical Agitation: Medium Stirring	Conc. of enzyme: 2% Sequestering agent:1g/l Wetting agent:1g/l Temperature : 60°C pH : 6.5 Time:30 min M : L : 1:20 Mechanical Agitation: Medium Stirring	Conc. of enzyme: 3% Sequestering agent:1g/l Wetting agent:1g/l Temperature : 60°C pH : 6.5 Time:30 min M : L : 1:20 Mechanical Agitation: Medium Stirring

1.3 Dyeing and Enzyme Treatment

Enzyme treatment after dyeing always imparts different influences on dyeing properties. Depth of shade increases when enzyme treatment is given before dyeing and depth decreases if enzyme treatment is done after dyeing. Fastness properties are very good if sample is treated with enzyme after dyeing rather than before dyeing (S. K. Laga, A. I. Wasif, and K. Shah, Report-2010). Pilling resistance is good if the sample is enzyme treated before dyeing. Enzyme treatment after dyeing increases weight loss% due to removal of unfixed dyes from the fabric surface (C.W. Kan and K.P. Law, Report-2012).

2. Experimental

2.1 Materials

100% cotton knit single jersey fabric (scoured and bleached), gram per square meter (GSM) 150, Dyed (D) and Undyed (UD).

2.2 Methods

Case 1

In case 1, the scoured and bleached fabric samples were treated with 0.5%, 1%, 1.5%, 2%, 3% concentration of cellulase enzyme (Acid stable)-Lava cell NBG keeping other variables like temperature, pH, duration of treatment, material to liquor ratio, and nature of mechanical agitation constant with a standard recipe (M. F. Hossain, Report-2009).

Case 2

In second case, at first the scoured and bleached fabric samples were dyed with reactive dye (Hot brand) with a standard recipe (M. F. Hossain, Report-2009) and then the enzyme treatment was carried according to condition as it was followed in first case (M. F. Hossain, Report-2009).

Table 2: Recipe of dyeing (M.F. Hossain, Report-2009).

Reactive Red (Hot Brand)	: 1.5% owf
Sequestering Agent	: 1 cc/l
Wetting agent	: 1cc/l
Salt	: 60 g/l
Soda Ash	: 15 g/l
pH	: 10-11
Time	: 60 min.
Temperature	: 90 ^{0}C
M: L	: 1:20

2.3 Working Procedure

In case 1, at first the samples were weighted on a digital weight balance. Then the bath was set with the materials at room temperature and pH was checked. The bath temperature was raised to 55-60°C and run for 30 minutes. Materials were checked to evaluate the enzyme performance time to time and after satisfactory result the temperature was raised to 85°C and run for about 20 minutes to deactivate the enzyme. Then temperature was lowered down to 50-55°C and liquor was drained followed by a cold rinse of 5-10 minutes. After rinsing the materials were dried at 100 0C for 2 hours and conditioned properly before weight measurement.

In case 2, in first stage, the bath was set at room temperature with material, dye, sequestering agent wetting agent and salt for dyeing. Then run for 20 minutes and temperature of the bath was raised to 90 After 20 minutes 50% soda was added and remaining soda was added after 20 minutes of the first half addition and run for 20 minutes. Thus dyeing was continued for about 60 minutes. After 60 minutes of dyeing the material was removed and the liquor was drained. The material was rinsed with hot water at 50 o C for 10-15 minutes. Neutralization was carried out to lower down the pH to 5.5 -6.5 with acetic acid(1cc/l) at 50 o C. Soap wash, hot rinse and cold rinse were followed accordingly. After rinsing the materials were dried at 100 0C for 2 hours and conditioned properly before weight measurement.

In second stage, the materials were treated with enzyme in a similar way as it was practiced in case 1 and weight measurement was done after this treatment properly.

3. Characterization

The following tests were done to assess the properties of bio-polished cotton knitted fabric:

a) Hand feel
b) Weight loss measurement
c) Absorbency test
d) Pilling [EN ISO 12945-1 (I.C.I. pilling box)]
e) Color fastness to wash [ISO 3] and rubbing (for dyed samples)
At least five samples were tested for a single test procedure and the average value was taken and put on the table in all cases.

4. Results and Analysis

4.1 Measurement of Hand Feel

After bio-polishing, the hand feel and luster of both dyed and undyed samples were assessed visually. It was observed that bio-polishing enhanced fabric surface smoothness and brightness in a similar way in both cases. When concentration was increased from 0.5% to 2%, smoothness also increased. But above 2% i.e. with 3% concentration fabric surface became rough. So, the optimum result was achieved with 2% concentration. It was due to the fact that when concentration was o.5% to 2%, the enzymatic action was occurred on the fiber surface but when concentration was 3% the enzymatic action was occurred interior of the material and thus fabric surface became rough.

4.2 Measurement of Weight Loss

Weight of un-dyed and dyed samples were measured before and after treatment by using a digital balance and weight loss% were calculated with a standard formula and in both cases weight loss percentage increased significantly with higher enzyme concentration.

Table 3: Measurement of weight loss with different enzyme concentration for undyed and dyed fabric sample.

Conc. of enzyme in %	Avg. wt loss after enzyme treatment in % (UD)	Avg. wt loss after enzyme treatment in % (D)
0.5	1.6	3.5
1	2.4	3.8
1.5	3.6	4.1
2	5	4.5
3	6.9	5.4

Table 4: Measurement of absorbency with different enzyme concentration for undyed and dyed fabric sample.

Conc. of enzyme in %	Avg. wt loss after enzyme treatment in % (UD)	Avg. wt loss after enzyme treatment in % (D)
0.5	1.6	3.5
1	2.4	3.8
1.5	3.6	4.1
2	5	4.5
3	6.9	5.4

It was noticed that the higher percentage weight loss was obtained at 3% concentration. In dyed samples weight loss percentage was higher comparing un-dyed fabrics up to 1.5% enzyme concentration and later on it was more or less same in both cases.

4. 3 Measurement of Absorbency

Absorbency measurement was carried out by dropping 0.1% direct red solution and time of absorption was calculated by stop watch. Enzyme treatment imparted and enhanced absorbency power of dyed and un-dyed fabric samples significantly and with 3% enzyme concentration maximum absorbency was noticed in both cases. It was also traced that the dyed samples had lower absorbency that un-dyed samples and moreover enzyme treatment could not alter this phenomenon.

Sample before enzyme treatment (UD) ? Avg. time to absorb one drop of solution was 1.20 sec.

Sample before enzyme treatment (D) ? Avg. time to absorb one drop of solution was 3.60 sec.

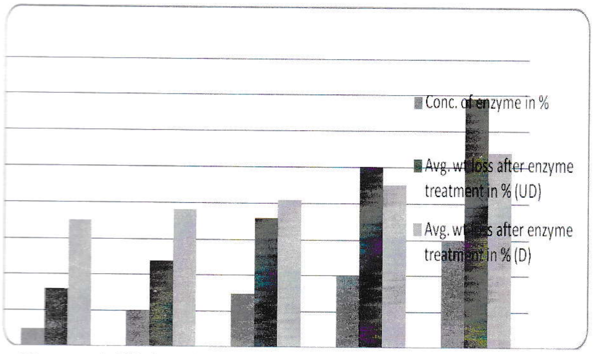

Figure - 1: Wt loss % at different enzyem concentration.

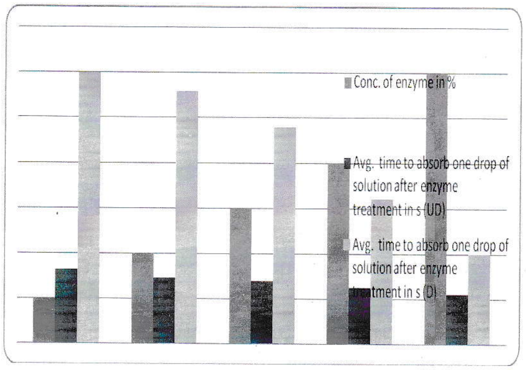

Figure - 2: Time to absorb one drop of solution in sec at different enzyme concentration.

4.4 Measurement of Pilling

Enzyme treatment significantly reduced pilling tendency in un-dyed and dyed fabric samples with increased enzyme concentration. But in dyed samples improvement was less noteworthy comparing undyed samples as the dyeing process interfere the performance of enzyme.

Sample before enzyme treatment (UD) - Avg. rating of pilling was 3.

Sample before enzyme treatment (D) - Avg. rating of pilling was 3.

Table 5: Measurement of pilling with different enzyme concentration for undyed and dyed fabric sample.

Conc. of enzyme in %	Avg. pilling rating (UD)	Avg. pilling rating (D)
0.5	3	3
1	4	3.5
1.5	4	3.5
2	4	3.5
3	5	4.5

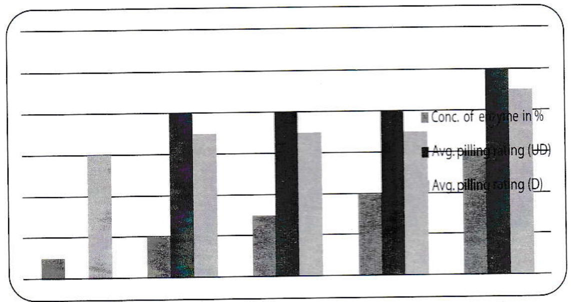

Figure- 3: Pilling at different enzyme concentration

4.5 Measurement of Color Fastness to Wash and Rubbing

Enzyme treatment on dyed samples deteriorated the depth of shade but enhanced fastness properties of dyed materials. Here it was observed that the fastness quality was constant for all concentration range both in wash fastness and rubbing fastness measurement.

Table 6: Measurement of color fastness to wash and rubbing with different enzyme concentration for dyed fabric

Conc. of enzyme in %	Avg. rating for color fastness to wash (D)		Avg. rating for color fastness to dry rubbing (D)		Avg. rating for color fastness rating to wet rubbing (D)	
	Color change	Staining	Color change	Staining	Color change	Staining
0.5	4/5	4	4/5	4	4/5	3/4
1	4/5	4	4/5	4	4/5	3/4
1.5	4/5	4	4/5	4	4/5	3/4
2	4/5	4	4/5	4	4/5	3/4
3	4/5	4	4/5	4	4/5	3/4

Sample before enzyme treatment (D)

- Avg. rating for color fastness to wash (Color change) was3/4
- Avg. rating for color fastness to wash (Staining) was 3
- Avg. rating for color fastness to dry rubbing (Color change) was3/4
- Avg. rating for color fastness to dry rubbing(Staining) was 3
- Avg. rating for color fastness to wet rubbing (Color change) was3/4
- Avg. rating for color fastness to wet rubbing(Staining) was 3

5. Conclusion

The hand feel of tested sample was optimum with 2% enzyme concentration in both undyed and dyed samples and there after it was deteriorated. Fabric's weight loss tendency was increased with the increase of conc. % and considerable weight loss was noticed up to 1.5 % conc. range. Weight loss was severe with 3% concentration and more significant with dyed samples. In pilling test we got a good result from 1 % to 2% concentration ranges and the very best result was obtained at 3% concentration range but dyed fabric samples showed lesser pilling resistance. In absorbency test, we found that the sample treated with 3% concentration had the maximum absorbency and absorbency power was always good with undyedenzyme treated samples. Color fastness to wash and rubbing improved on dyed samples after enzyme treatment.

Finally it could be said that the undyed tested samples treated with 1 % and 1.5 % concentration showed acceptable values in all testing procedures carried out in this work especially from a minimum fiber damage perspective and thus to obtain better performance for dyed cotton knit fabric it was recommended to carry out enzyme treatment with a concentration range of 1% to 1.5 % preferably before dyeing process.

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Appendix

Recipe calculation

Total liquor = Sample weight ×liquor ratio

Required concentration of enzyme = (Sample weight ×concentration %) /stock solution%

Required dyes = (Sample weight ×Shade %) / Stock solution%

Required chemical agent = (Total liquor ×recipe amount)/ (1000 × stock solution %)

Required adding water = Total liquor - Total chemical amount

Calculation for dyeing

Sample weight = 341 g

Total liquor = 341 × 20

= 6820 cc = 6.82 liter

Required dye = (341 × 1.5%) = 5.11 g

Required sequestering agent = (6.82 × 1 × 100) / 5 = 136.4 cc

Required wetting agent = (6.82 × 1 × 100) / 5 = 136.4 cc

Required salt = 6.82 × 60 = 409.2 g
Required soda = 6.82 × 15 = 102.3 g
Required adding wate r = 6820 cc-(136.4 cc + 136.4 cc) = 6547.2 cc = 6.55 liter

Calculation for enzyme treatment

Recipe 1
Sample weight =25 g
Total liquor =25×20=500 cc
Required concentration of enzyme = (25 ×.5%)/1% =12.5 cc
Required sequestering agent = (500×1×100)/ (1000×5) =10 cc
Required wetting agent = (500×1×100)/ (1000×5) =10 cc
Required water =500-(12.5+10+10) = 467.5 cc

Recipe 2
Sample weight =25 g
Total liquor =25×20=500 cc
Required concentration of enzyme = (25 ×1%)/1% =25 cc
Required sequestering agent = (500×1×100)/(1000×5)=10 cc
Required wetting agent = (500×1×100)/(1000×5)=10 cc
Required water =500-(25+10+10) = 455 cc

Recipe 3
Sample weight =25 g
Total liquor =25×20=500 cc
Required concentration of enzyme = (25 ×1.5%)/1% =37.5 cc
Required sequestering agent = (500×1×100)/ (1000×5) =10 cc
Required wetting agent = (500×1×100)/ (1000×5) =10 cc
Required water=500-(37.5+10+10) = 442.5 cc

Recipe 4
Sample weight =25 g
Total liquor=25×20 = 500 cc
Required concentration of enzyme = (25 ×2%)/1%=50 cc
Required sequestering agent= (500×1×100)/ (1000×5) =10 cc
Required wetting agent = (500×1×100)/ (1000×5) =10 cc
Required water=500-(50+10+10) = 430 cc

Recipe 5
Sample weight =25 gm
Total liquor=25×20=500 cc
Required concentration of enzyme = (25 ×3%)/1% =75 cc
Required sequestering agent = (500×1×100)/(1000×5) =10 cc
Required wetting agent = (500×1×100)/(1000×5)=10 cc
Required water =500-(75+10+10) =405 cc

Formula to calculate wt. loss%

$$\text{Wt. loss \%} = \frac{\text{Wt. of testd smple before bioplishing} - \text{Wt. of tested sample after biopolishing}}{\text{Wt. of tested sample before biopolishing}} \times 100$$