

# A comparative study on acidic and neutral enzyme on various properties before and after reactive dyeing

Rajib Saha<sup>1</sup>, Md. Jahidul Islam<sup>2</sup>, Mahade Hasan Ikram<sup>2</sup>, Asha Akter Ete<sup>2</sup>

<sup>1</sup>Assistant Professor, Department of Textile Engineering, Southeast University, 251/A & 252, Tejgaon Industrial Area, Dhaka-1208, Bangladesh

<sup>2</sup>Student, Department of Textile Engineering, Southeast University, 251/A & 252, Tejgaon Industrial Area, Dhaka-1208, Bangladesh

**Abstract** — Enzymatic wash is vital for removing the fuzz fibers which are responsible for forming pilling over the surface. The present work was focused mainly with the assessment of the performance among various enzymes: BPA (acidic), Hyzyme C 1085(acidic), Superzyme LDB (acidic), Supracel NOTR (neutral) and N9 (neutral). The properties of cotton fabrics before and after the enzymatic and reactive-dyeing treatment were investigated, including the color strength, color fastnesses, difference of color and gsm, whiteness, reflectance, pilling as well as price. The results showed that application of immobilized enzymes in a vertical bioreactor enhanced the formation of pilling and color difference found minimum for BPA (acidic). On the other hand, fastness rating found almost same for all enzymes. Considering the outcome-data, performance of acidic enzyme is better than neutral one and best one is BPA (acidic).

**Keywords** — Enzyme wash, Acidic, Neutral, Fastness property, Reactive dyeing, Cotton fabric.

## I. INTRODUCTION

Cellulase has the ability to degrade cellulosic fuzz-fibres of cotton, flax and vis-cose by hydrolysing the repeating unit of  $\beta$ -1,4-glycosidic bonds in cellulose molecules [1, 2] and has been used for enzymatic treatments of different cellulosic textiles. During the bio-scouring of cotton fabrics, the combined use of cellulase and pectinase might enhance the efficacy of impurity removal and impart satisfactory wettability to fabric without severe environmental pollutions [3]. Enzymes have been increasingly employed in the textiles industry over the past decade. They have been used for desizing, scouring, polishing, washing, degumming, and bleaching, as well as for decolouring of dye-house wastewater. As enzymes show a large variety of side chains of the outer amino acids and a large 3-d protein structure, it can be expected that an enzyme which can interact with virtually any chemical agent can always be found [4]. The scouring process represents the first step in the processing of natural fibres. It has the aim of removing dirt and impurities and preparing the fibres

for further processing. It may also be performed again at a later stage, often with the aim of increasing fabric wettability. Traditionally, scouring is performed with inorganic compounds such as NaOH. However, several enzymes have also been used in the scouring process over the years. Cellulase and pectinase are among the most promising enzymes for scouring. They have been employed either together or separately and have proved effective for cotton scouring. Both enzymes improve cotton absorbency. They have been shown to impart adequate absorbency to the fibres with short treatment time [5]. Treatment with pectinase, lipase, and protease, applied individually, provide a slight improvement in the water wettability and strength retention properties of cotton [6]. The combination of pectinase and cellulase significantly improves the wetting outcome of the scouring process. Pectinase, however, improves the water absorbency more than either lipase or protease [7]. Cellulase, via a hydrolysis reaction, removes the readily accessible surface fibrils yielding a softer fabric hand [8].

The present study analyzed the impacts of the various enzyme activities before and after reactive dyeing on cotton fabrics. In addition, the assessment of the modification of enzyme treated cotton fabric will also be discussed.

## II. Experimental

### Materials

The 100% cotton knit fabric was taken from Micro Fibre Knit Composite LTD. Five different enzymes i.e. BPA (Acidic), Hyzyme C 1085(Acidic), Superzyme LDB (Acidic), Supracel NOTR (Neutral) and N9 (Neutral). All types of pretreatment and dyeing chemicals are also supplied by that factory. Other chemicals used in this work were analytical grade.

### Scouring and bleaching

For conventional pretreatment the fabric was scoured and bleached for 50 min. at 98 °C with a liquor ratio of 1:10 and pH 11 in a bath containing 0.5 g/l wetting agent, 0.5 g/l sequestering agent, 0.5

g/l anti-creasing agent, 0.22 g/l stabilizer, 1.8 g/l caustic soda and 1.75 g/l hydrogen peroxide. After that peroxide killer of 0.5 g/l and acetic acid of 0.75 g/l is used for peroxide killing at 55°C for 60 min following cold wash.

**Enzyme wash**

The enzyme wash was done by using 0.25% each enzyme (BPA(Acidic), Hyzyme C 1085(Acidic), Superzyme LDB(Acidic), Supracel NOTR(Neutral)

Process	Chemical Name	Dosing
Scouring & Bleaching 98°C×50min pH: 11	LLF-13(Wetting Agent)	0.50 g/l
	SQ-114(Sequestering agent)	0.50 g/l
	CBA(Anti-creasing agent)	0.50 g/l
	RS-200(Stabilizer)	0.22 g/l
	Caustic soda(NaOH)	1.80 g/l
Peroxide Killing 55°C×60min	H <sub>2</sub> O <sub>2</sub> (50%)	1.75 g/l
	N.Acid(Acetic Acid)	0.75 g/l
Killing	OEM(Peroxide Killing)	0.50 g/l

and N9(Neutral)) with 0.5 g/l wetting agent, 0.5 g/l sequestering agent and 0.5 g/l anti-creasing agent at 50°C for 60 min. with pH 4.5 and a liquor ratio of 1:10.

**Dyeing**

The enzyme washed samples were dyed with ORANGE 2RX (Reactive Dye), RED 3BX(Reactive Dye) and RED 2B(Reactive Dye) with 50 g/l glauber salt, 18 g/l soda ash and other chemicals. Here, temperature was 60°C for 60 minutes with maintaining pH of 10.5. After that neutralization and soaping wash was carried out.

Process	Chemical name	Dosing
Dyeing (60°×60min) pH:10.5	SQ-117(Sequestering agent)	0.50 g/l
	CL-225(Levelling agent)	1.00 g/l
	CBA(Anticreasing agent)	0.50 g/l
	ORANGE 2RX(Reactive Dye)	0.284%
	RED 3BX(Reactive Dye)	0.06 %
	RED 2B(Reactive Dye)	1.64 %
	Glauber salt	50 g/l
Neutralization (55°C×5min)	Soda ash	18 g/l
	N.ACID(Acetic Acid)	0.20 g/l
Soaping (50°C×5min)	R-536(Fixing agent)	0.50 g/l
	N ACID(Acetic acid)	0.80 g/l
Soaping (50°C×5min)	AWP-5045(Soaping agent)	1.00 g/l

**Color strength**

The color strength of the dyed fabric was determined using a Color-Eye 7000A spectrophotometer (GretagMacbeth, USA) according to the Kubelka– Munk method, and the result was calculated using following equation.

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

Where, K and S represent the coefficients of absorption and scattering, respectively, and R denotes the reflectivity of the fabric at the wavelength of maximum dye absorption [9].

**Color fastness measurement**

The rubbing fastness of the dyed cotton fabric was tested according to AATCC Test Method 8-2007 using a rubbing fastness tester (Wenzhou Darong Company, China). Colour fastness to wash was determined according to ISO 105 C06 method (Gyrowash, England), Color fastness to perspiration was determined according to ISO-105 E04, colour fastness to water was measured according to ISO105-C01, colour fastness to rubbing was determined according to ISO 105X12 (James Heal Crockmeter, UK) and color fastness to saliva was measured according to GB/T 18886 .

**Measurement of Grams per Square Meter (g/m<sup>2</sup>)**

GSM was measured by GSM cutter in gray, scoured-bleached, after enzyme and for dyed fabric of all types of enzyme wash.

**Whiteness Index, Reflectance % and Color difference (ΔE)**

Whiteness Index, Reflectance percentage of grey, scoured-bleached, enzyme wash fabric and dyed fabric was measured by spectrophotometer (datacolor 600, Taiwan) and difference of colour was also determined by that machine.

**Pilling**

According to ISO-12945-1 method pilling of different enzymed fabric was measured with the help of Opti pill, UK.

**III. Results and discussion**

**GSM**

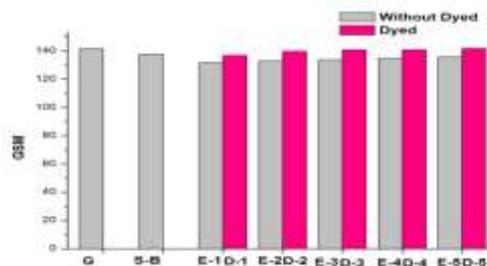


Fig.1: Effect of enzyme on GSM

Here G- Grey, S-B-Scour-bleach, E-1=BPA D-1=Dyed after BPA enzyme wash

Only scour-bleached sample for BPA, which represented the top most position. On the other hand, N9 decreased only by 2 gm compare to pre-treated one. Here, it is significantly clear that the loss of weight was always more than pre-treated sample from the pre-treating enzyme washed sample and that difference was 4 for all types of enzyme.

On contrary, after dyeing the scenario became change. In figure-1, the difference between grey and scouring-bleaching enzyme washed sample were less than that of without dyed sample. The loss of gsm was maximum for BPA, that is 5 and for N9 there was no loss. For others enzyme it was minimal. On the other hand, pre-treated and pre-treating enzyme washed sample decreased by 1 for BPA and others gained and maximum 4 gm gained for N9. The reason behind it for incorporated dyes with the molecule of fibre.

**Whiteness Index**

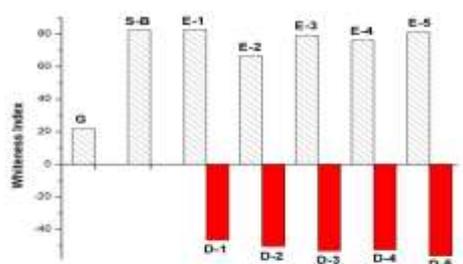


Fig.2: Effect of enzyme on Whiteness Index

From figure-2, it was visible that whiteness-index was 4 times greater than grey sample (21.78) for pre-treated (82.49) and pre-treating enzyme washed samples. And after enzyme washing the WI became slightly less than scour-bleached one. Here, the lowest position stood for superzyme LDB, which value was 66.37. On the other side, after dyeing WI turned negative in values. Here, minimum for BPA (-46.63) and maximum for N9 (-56.28).

**Reflectance %**

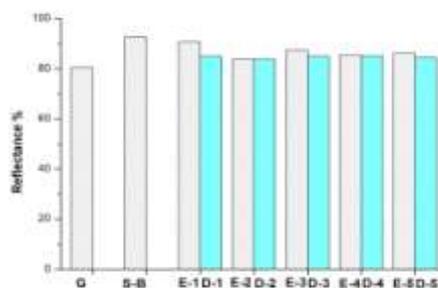


Fig. 3: Effect of enzyme on Reflectance %

In case of reflectance % as figure-3, the values raised for enzyme washed sample for all cases. Mostly gained for BPA to 10.18% and minimum for Hyzyme to 4.17%. But compare with pretreated samples that values became downfall. For Hyzyme it was maximum to 7.4% and for BPA it was minimum to 2.01%. The figure-3 also depicted after dyeing the reflectance% became fall compare to pretreated and enzyme washed sample but increased from grey one in case of BPA, the value was 10.18% more than grey, which was at pick position. Whereas it was 4.17% that of Hyzyme, which was at lowest place. But compare with pretreated sample, the lowest loss of reflectance% to 2.01% for BPA and to 7.4% for Hyzymes that took most decreased. Overall, considering all enzymes BPA accounted highest position in Reflectance % but lower than pretreated. The difference between enzyme and dyed one became minimal for all enzymes but it was 5.78% for BPA, that was maximum.

**Color fastness**

**a) Color fastness to rubbing**

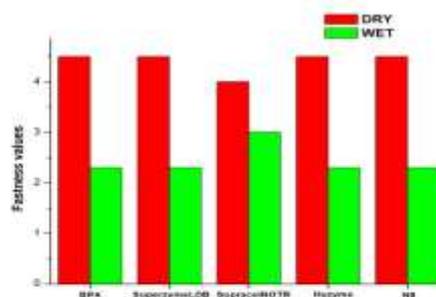


Fig.4: Effect of enzyme on Rubbing fastness

According to figure -4, for color fastness to rubbing, in case of dry for all enzymes the results were excellent (4-5) except Supracel NOTR which was very good (4). However, for wet, most of the test results were slightly fair (2-3) whereas for Supracel NOTR is stood moderate (3).

**b) Color fastness to wash**

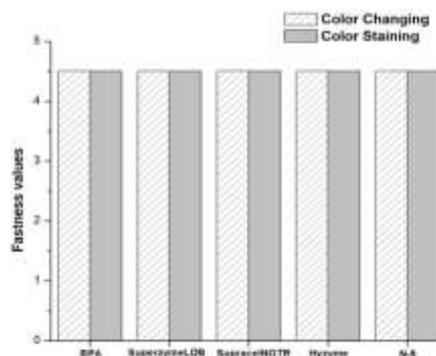


Fig.5: Effect of enzyme on Wash fastness.

From figure-5 it showed excellent fastness against wash for every washed sample, that was (4-5).

c) Color fastness to water

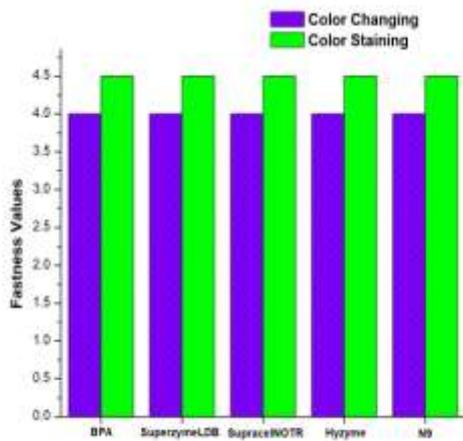


Fig.6: Effect of enzyme on color fastness to water.

d) Color fastness to saliva

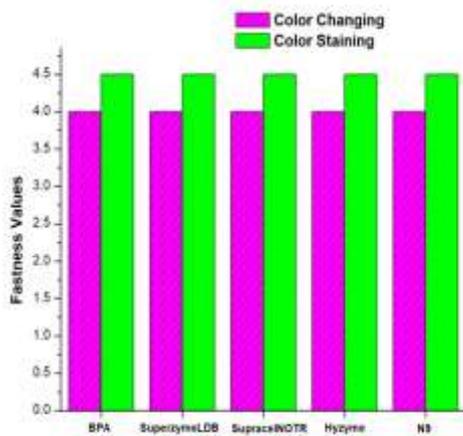


Fig.7: Effect of enzyme on color fastness to saliva.

e) Color Fastness to Perspiration

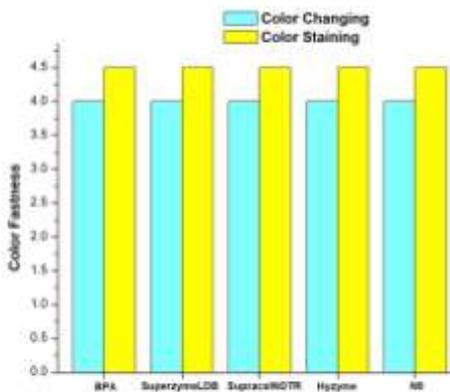


Fig.8: Effect of enzyme on color fastness to perspiration (Acid).

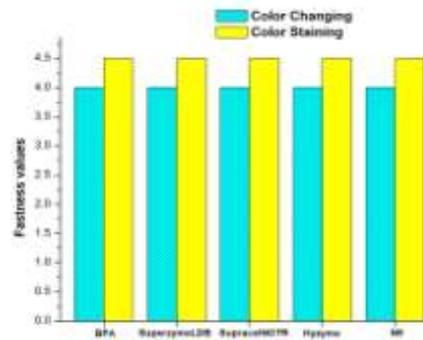


Fig.9: Effect of enzyme on color fastness to perspiration (Alkali)

For figure-6 & 7 changing in shade scale represented very good (4) and color staining scale it was excellent (4-5). Same trend was found for color fastness to perspiration (Acid & Alkali) which cleared in figure 8 and 9.

Color difference ( $\Delta E$ ) Values

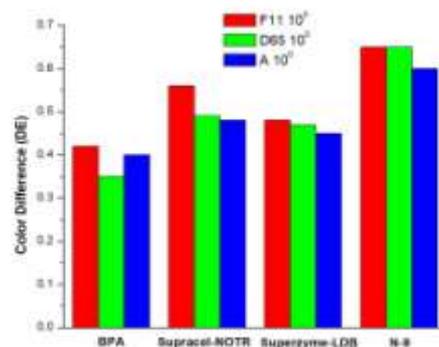


Fig.10: Effect of enzyme on color Difference

Figure-10 described about the color difference where Hyzyme was standard. The difference was highest for N9 ( $\Delta E=0.65$  at D65) and lowest for BPA ( $\Delta E=0.35$  at D65). Same outcomes for other source of light (F11 & A). But all values are less than 1 which is acceptable.

v) Color strength (K/S)



Fig.11: Effect of enzyme on color strength

There was not so variation among color strength (K/S) which values were taken after dyeing (enzymed). In figure-11 the lowest value was 9.2 for BPA and highest for Hyzyme was 9.7. Here, 500nm rating was considered.

### Pilling

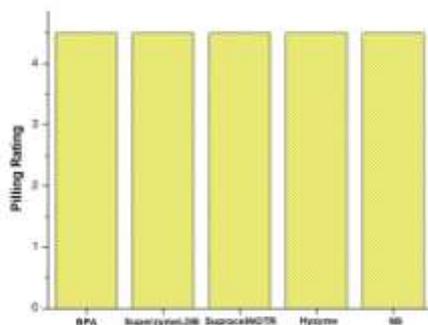


Fig.12: Effect of enzyme on pilling

Pilling rating as figure-12, for all enzyme treated fabric the rating came excellent (4/5).

### Price

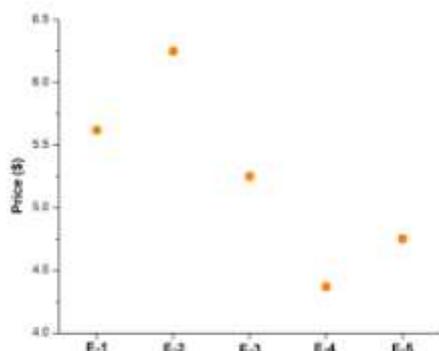


Fig.13: Price of enzymes in US\$

It is clear that LDB (6.25\$/l) represents top most price and Hyzyme (4.38\$/l) is the lowest.

## IV. Conclusion

Overall, considering all types of enzymes, BPA (Acidic), Superzyme LDB (acidic), Supracel NOTR (Neutral), Hyzymes C 1085(Acidic), N9 (Neutral), Reflectance% and whiteness Index is better than others for BPA. But loss of weight is minimum in case of N9. There is similar result for all enzymes. For color fastness to water, saliva, perspiration and wash, the grey scale rating between very good to excellent(4-4.5), but fluctuation found in case of rubbing, especially it was slightly fair to moderate (2.5-3). On the other side, dry test was almost excellent. There is no such variation in color strength (K/S) i.e., in between 9-10 for 500nm.

Same result is found after pilling test that was between 4 and 5. Accounting price, consuming highest rate for LDB where as Hyzyme is lowest. Variation of color with standard, worst result was for N9 and comparatively less variation was for BPA. But for all enzyme achieved acceptable results. That means  $\Delta E \leq 1$ .

In particular, BPA (acidic) is the best enzyme considering all sorts of data.

## REFERENCES

- [1] Ibrahim NA, EL-Badry K, Eid BM, Hassan TM. A new approach for biofinishing of cellulose-containing fabrics using acid cellulases. *Carbohydrate Polymers* 2011; 83(1):116-121.
- [2] Belghih H, Ellouz-Chaabouni S, Gargouri A. Biostoning of denims by *Penicillium occitanis* (Pol6) cellulases. *Journal of Biotechnology* 2001; 89(2-3): 257-262.
- [3] Karapinar E, Sariisik MO. Scouring of cotton with cellulases, pectinases and proteases. *Fibres and Textiles in Eastern Europe* 2004; 12(3): 79-82.
- [4] Tzanov T, Costa S, Guebitz GM, Cavaco-Paulo A (2001) Dyeing in catalase-treated bleaching baths. *Color Tech* 117, 1-5.
- [5] Li YH, Hardin IR (1997) Enzymatic scouring of cotton: effects on structure and properties. *Textile Chem Colorist* 29, 71-6.
- [6] Hartzell MM, Hsieh Y-L (1998) Enzymatic scouring to improve cotton fabric wettability. *Textile Res J* 68, 233-41.
- [7] Buchert J, Pere J, Puolakka A, Nousiainen P (2000) Scouring cotton with pectinases, proteases and lipases. *Textile Chem Colorist Am Dyestuff Rep* 32, 48-52.
- [8] Buschle-Diller G, Fanter C, Loth F (1999) Structural changes in hemp fibers as a result of enzymatic hydrolysis with mixed enzyme systems. *Textile Res J* 69, 244-51.
- [9] Senthilkumar M, Selvakumar N, Shamey R. The effect of humidity, fabric surface geometry and dye type on the colour of cotton fabrics dyed with a select range of anionic dyes. *Dyes and Pigments* 2011; 90(3): 225-232.