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# Enzyme washing of indigo and sulphur dyed denim

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## Abstract

A wide ranging finish effects have been given to denim fabrics in industrial scale to make it more fashionable and functional. Among the numerous wet and dry treatments given, worn out looks of various kinds have received the maximum attention. In this context, herein, two denim fabrics, one dyed with vat indigo and the other with sulphur black were enzyme washed. The enzymes used were acid cellulase and neutral cellulase and the treatments were done at varying concentrations, time and mechanical agitation. The trials so taken were based on design of experiments, and the effect was analysed in terms of decrease in colour depth and weight loss. Back staining of fabric during the wash was also checked and acid enzyme despite higher colour removal, caused significant staining in sulphur dyed quality. Attempt was made to contain this back staining by suitable treatment. The application conditions of cellulases on the denim qualities were also optimized for fading effect.

**Keywords:** Acid cellulase, Back staining, Colour strength, Denim, Fading, Neutral cellulase

## Introduction

Denim as a fabric, has received the widest acceptance among all textile products. It has had an incredible influence on consumers both socially and culturally (Paul 2015). This twill cotton fabric usually has warp threads indigo dyed while weft remains plain white. The warp faced fabric therefore looks blue on one side and white on reverse. The warp being ring dyed, creates denim's fading characteristics, which is unique compared to every other textile material. With changing times, many new variants of denim came into existence. Denim washing is one of the key areas in getting the faded look. Earlier stone washing used to be done to achieve soft feel and the desired appearance. During washing, the pumice stone, scraps off the dye particles from the yarn surface in the denim fabric. Due to the ring dyeing of denim fabric and heavy abrasion during stone washing process, the faded effect is achieved. Oxidative bleaching agent with or without the addition of stones have also been used to get fading effect. Difficulty in removing residual pumice from fabric, damage to equipment and clogging of machine drainage passage due to particulate material proved to be major drawbacks with the technique (Heikinheimo et al. 2000). Later on use of enzymes became a sustainable option to get the worn out look in denim. The enzymes used are cellulase enzymes, specifically acting on the cellulose part, mainly on the surface of the fabric. This gives the desired look

and at the same time, removes hairiness from surface thus giving a smooth and soft feel. Enzymes are substrate specific in their action, unaffected by the others. They are primarily used in textiles to promote hydrolysis of the required substrate (Patra 2003).

A fair amount of experimental research on enzyme wash, has also been carried out in last two decades or so. Denim fabric has been treated with *Trichoderma Reesei* cellulases and compared for stone washing denim fabrics (Heikinheimo et al. 2000). The redeposition of indigo after treating with cellulase has been examined to certain extent (Andreas et al. 2000). Campos et al. (2000) investigated the indigo-cellulase interactions for cellulases from different fungal origins. However differing from all these, in the present work, two denim fabrics of similar weave and dyed with different classes of dyes are treated with acid cellulase and neutral cellulase. The laboratory scale treatments were done varying their concentration and time. Standard steel balls were also used in the bath to enhance the rigours of treatment. The levels of the process parameters were changed based on design of experiment. Since washing of denim is sometimes accompanied by back staining, treatment with a commercial anti back staining agent was also done. Out of the various dyes used for denim, indigo vat and sulphur colours are the more common ones and hence they were taken for the study.

## Methods

### Materials

Grey denim fabrics of two different colours were taken for the study. The fabrics of 2/1 twill with 7 s warp count and 10 s weft count had following specifications.

Dark blue indigo dyed: epi 49; ppi 49; gsm 210.

Sulphur black dyed: epi 50; ppi 51; gsm 220.

The enzymes used for the denim washing are from Novozymes with specifications as given below (Table 1).

For desizing of the sized warp yarn Invazyme ADC of Huntsman was used while acetic acid used was of L R grade. A bleached plain woven cotton fabric with 40 s yarn was used for back staining.

### Enzymatic fading

To start with, the standard test for dye on fibre was done to conform vat dye and sulphur colour on the two fabrics. Then, in both the denim qualities, fabric was first desized with 5 g/l of the amylase enzyme at 60–65 °C for 60 min. The pH was maintained at 5–6 using

**Table 1 Enzyme specifications**

Name	Type	Strength (ECU <sup>a</sup> /g)	Recommended conditions	
			pH	Temperature (°C)
Cellusoft Conc. L	Acid cellulase	1500	4.5–5.5	40–50
Neutrancell CLD	Neutral cellulase	2400	6.5–7.5	25–35
Denimax core 1380S	Anti backstaining	–	6.5–7.5	40–55

<sup>a</sup> ECU—amount of enzyme producing one n mole of reducing sugar as glucose in 1 s

acetic acid and MLR was kept 1:30. After desizing, the enzyme was deactivated by raising the temperature of water bath to 80 °C for 10 min. Then fabric was rinsed with warm water followed by cold water. It was then dried in an oven and checked for size removal with Iodine drop test. After passing the test, fabric was taken for further treatments.

The two denim qualities were treated with the cellulase enzymes under recommended conditions. These enzymes in ready to use form, were applied in different concentrations with varying parameters like time and mechanical agitation. The two denim fabrics as mentioned were of different colours and enzymatic fading action was supposed to differ from fabric to fabric. The enzymes were applied at concentrations of 0.5, 1.25 and 2% and treatment time was 30, 45 and 60 min for acid enzyme while it was 60, 75 and 90 min for the neutral cellulase. Desized samples were given the treatment in Infra red dyeing machine with required number of steel balls. An MLR of 1:30 was maintained while pH of the bath was adjusted with acetic acid. After the specified time of treatment, hot and cold wash were given followed by soaping with Lissapol D 1 g/l at 80 °C for 15 min. Then hot and cold wash were given and finally samples were dried.

**Experimental design**

To study the individual and interactive effects of the concentration of enzymes, treatment time and mechanical agitation and optimize the results, Box Behnken design was used. These three variables were chosen as they were expected to potentially affect the fading process. The limits of each variable were chosen based on the preliminary investigations. Colour strength, weight loss percentage and back staining values were taken as response factors. The three levels of the variables and the set of experimental combinations for all the three types of enzymes are shown in following tables (Tables 2, 3).

The other parameters namely temperature and pH maintained for the acid cellulase was 40–50 °C and 4.5–5.5, while it was 25–35 °C and pH of 6.5–7.5 for neutral enzyme. All the treatments were carried out at 45 rpm in Infra Red dyeing machine with digital programming system. The pH was maintained with acetic acid while temperature was monitored every 10 min. The contour plots obtained from the values in the observation tables were used for interpretation. Further, ridge analysis was used for optimization of values in the experimental design region.

**Test methods**

The extent of fading was determined by measuring the colour strength of the treated samples in Premier Colorscan SS 5100A spectrophotometer. The measurement was done using the formula

**Table 2 Experimental conditions for the enzyme fading**

Variables	Coded levels for different enzymes					
	Acid enzyme			Neutral enzyme		
	- 1	0	+ 1	- 1	0	+ 1
Concentration (%)	0.5	1.25	2	0.5	1.25	2
Time (min)	30	45	60	60	75	90
Mechanical agitation (number of steel balls)	0	5	10	0	5	10

**Table 3 Experimental design based on coded levels**

Sample no.	Time	Enzyme conc.	Mechanical agitation
E1	- 1	- 1	0
E2	1	- 1	0
E3	- 1	1	0
E4	1	1	0
E5	- 1	0	- 1
E6	1	0	- 1
E7	- 1	0	1
E8	1	0	1
E9	0	- 1	- 1
E10	0	1	- 1
E11	0	- 1	1
E12	0	1	1
E13	0	0	0
E14	0	0	0
E15	0	0	0

$$\text{Colour strength, } K/S = (1 - R)^2/2R$$

where *R* reflectance.

The effect of enzyme on cellulose was tested by finding the % age weight loss as per ASTM D 3776. Since enzyme action on denim often causes undesirable back staining, the intensity of the staining was also measured in terms of K/S. To check the impact of treatments on stiffness of the denim qualities, bending length was measured in Shirley Stiffness tester as per BS 3356 standard test method.

**Results and discussion**

**Effect of acid cellulase on dark blue indigo dyed denim fabric**

The impact of process parameters namely, enzyme concentration, time and mechanical agitation using Cellusoft Conc. L was studied on the K/S value of dark blue indigo and sulphur black dyed denim fabric using Box-Behnken design. The values so obtained for the vat dyed fabric were,

Multiple R	0.965
Squared multiple R	0.931
Adjusted squared multiple R	0.805
Standard error of estimate	0.459

The response model evaluated in this study is significant, with coefficient of determination *R*<sup>2</sup> of 0.931 at a confidence level of 95%. The response surface equation obtained for the K/S value is as follows:

$$\begin{aligned} \frac{K}{S} \text{ value} = & 18.725 - 0.470X - 0.332Y - 1.164Z \\ & + 0.185X^2 - 0.054Y^2 + 0.294Z^2 \\ & - 0.205XY - 0.11YZ + -0.072XZ \end{aligned}$$

where, *X* concentration, *Y* treatment time and *Z* mechanical agitation.

The model is very significant, as is evident from its  $F$  value ( $F_{\text{model}} = 7.442$ ) and very low probability value ( $p = 0.020$ ) obtained from analysis of variance (Table 4).

The trend in colour fading under actual experimental conditions are shown by the K/S value in Table 5, while the contour plots for the interactive effects are in Fig. 1a–c.

It was observed that colour strength of denim fabric decreased significantly after they were exposed to enzyme treatment particularly at higher concentrations of the enzyme. A portion of the primary wall of cotton is always in contact with enzyme during washing and hence at the contact point, surface of fibers are decomposed by the aqueous solution of the enzyme. Consequently, enzyme washed denim become duller and color is faded. As a result, the indigo colored warp yarn in denim fabric tends to restore its original white color, because warp yarns are surface dyed. The results show that increasing the enzyme concentration from 0.5 to 2% (owf) has effect on change in color strength.

Figure 1a, c also indicate that K/S value decreased with increasing treatment time. This is likely due to the fact that longer enzyme treatment time will prolong degradation of cellulose and the time for further abrasion. With regards to the effect of mechanical agitation, it is clear from Fig. 1b, c that the K/S value decreases with increase in mechanical agitation caused by number of steel balls. In fact, increasing steel balls leads to higher

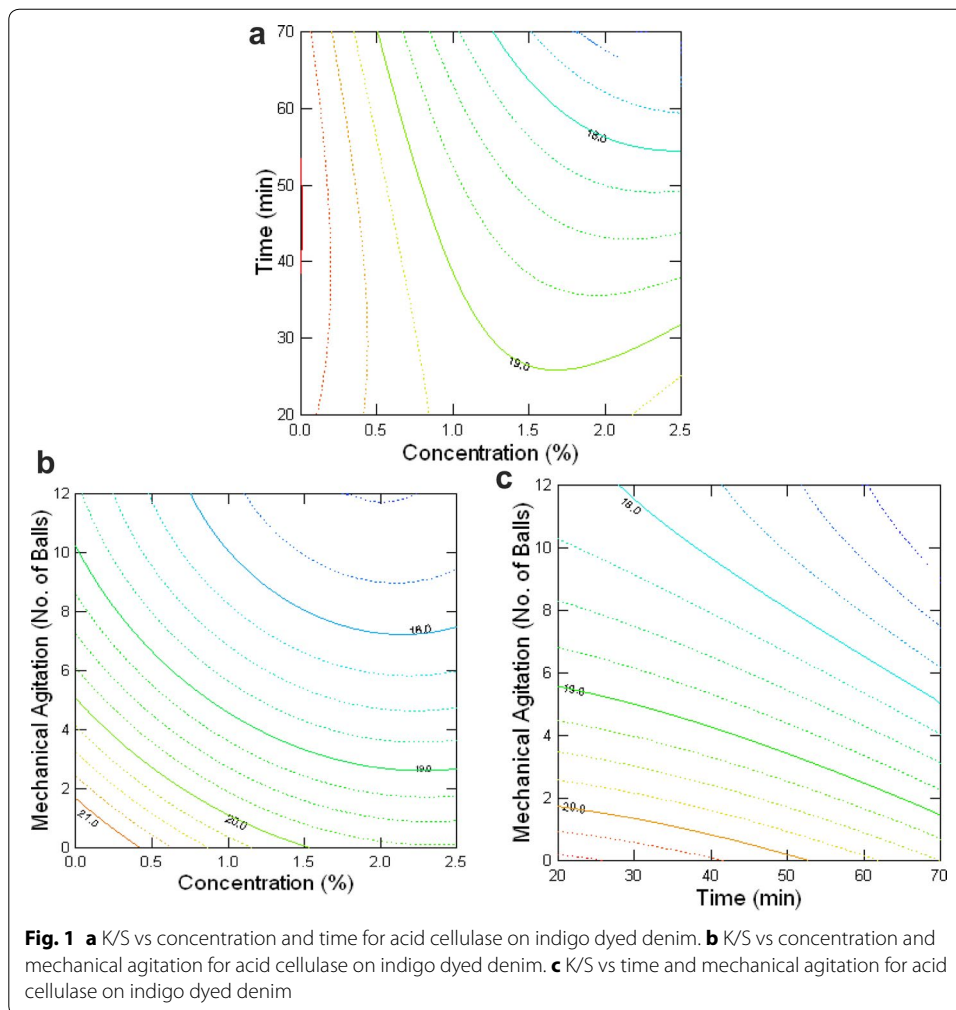
**Table 4 Analysis of variance table**

Source	df	Sum of squares	Mean squares	F-ratio	p value
Regression	9	14.128	1.570	7.442	0.020
Residual error	5	1.055	0.211		
Total error	14	15.182			

**Table 5 Experimental conditions and results for the effect of acid cellulase on dark blue INDIGO DYED denim**

Concentration (%)	Time (minutes)	Mechanical agitation (No. of steel balls)	K/S value	Weight loss (%)
0	0	0	22.32 <sup>a</sup>	–
0.5	30	5	19.38	2.94
2	30	5	19.17	3.01
0.5	60	5	18.94	3.72
2	60	5	17.91	4.55
0.5	45	0	20.79	3.03
2	45	0	19.39	4.52
0.5	45	10	18.87	5.16
2	45	10	17.75	5.35
1.25	30	0	20.63	3.04
1.25	60	0	20.17	4.75
1.25	30	10	17.78	3.59
1.25	60	10	17.27	5.09
1.25	45	5	18.99	4.44
1.25	45	5	18.38	4.32
1.25	45	5	18.79	4.52

<sup>a</sup> K/S value of desized denim fabric is 22.32



removal of dye particles along with protruding surface fibers. Hence more prominent fading effects are observed i.e. K/S value decreases.

**Optimum conditions for the K/S value**

With the help of ridge analysis method of optimization of process variables, optimum fading with Cellusoft Conc. L enzyme is found to be at concentration 1.25–1.55%, time about 45–52 min and with 5–8 steel balls at 95% confidence level.

**Effect of acid cellulase on sulphur black dyed denim fabric**

Similarly the effect of Cellusoft Conc. L treatment parameters when studied on the K/S value of sulphur black dyed denim fabric, the values obtained are

Multiple R	0.954
Squared multiple R	0.910
Adjusted squared multiple R	0.747
Standard error of estimate	1.202

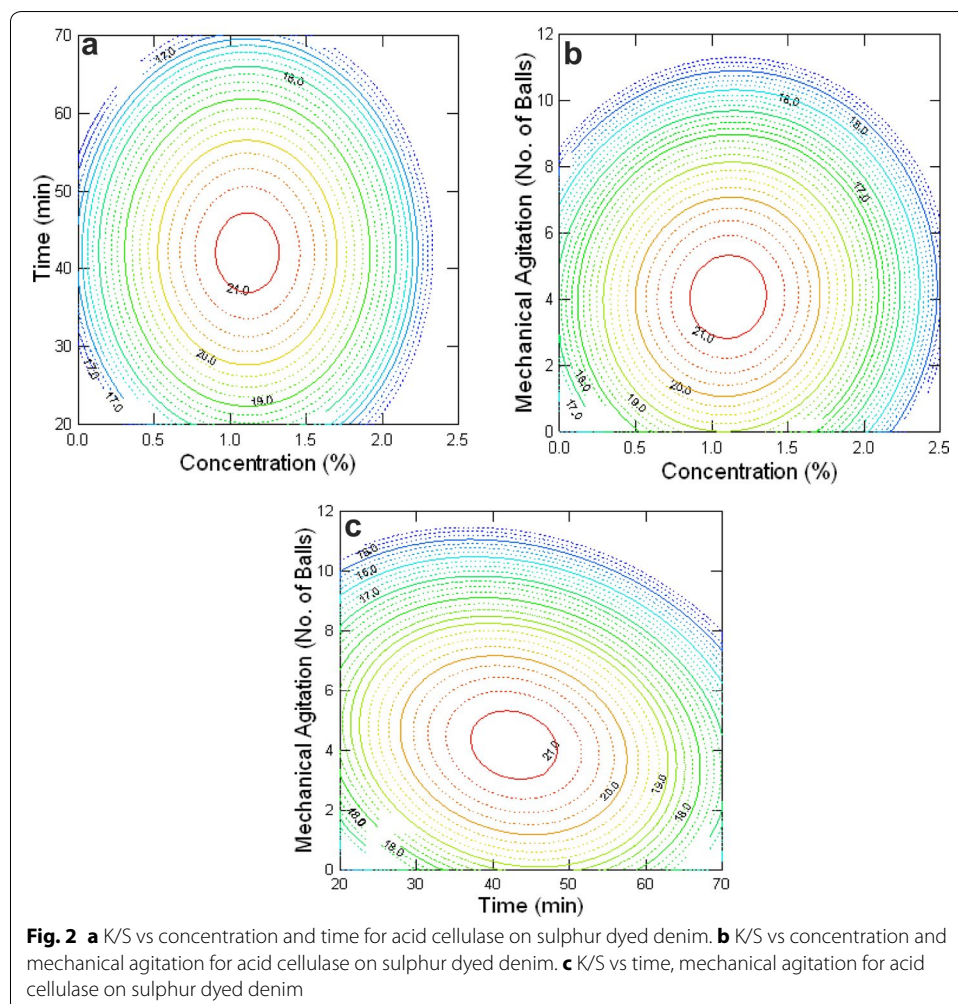
The response model evaluated in this study is significant, with a coefficient of determination  $R^2$  of 0.910 at a confidence level of 95%. The response surface equation obtained for the K/S value is as follows:

$$\begin{aligned} \frac{K}{S} \text{ value} = & 21.046 - 0.683X - 0.486Y - 1.205Z \\ & - 1.856X^2 - 1.234Y^2 - 3.349Z^2 \\ & - 0.022XY - 0.665YZ + 0.18XZ \end{aligned}$$

where  $X$  concentration,  $Y$  treatment time and  $Z$  mechanical agitation.

This model is quite significant, as per its  $F$  value ( $F_{\text{model}} = 5.597$ ) and very low probability value ( $p = 0.036$ ).

It can be seen from Fig. 2a, b that the color strength of denim fabric decreased significantly after they were exposed to cellulase treatment particularly at concentrations of 1–2%. During washing, fiber surfaces are hydrolyzed by the catalysis of the cellulase and the treated fabric becomes duller and color is faded. The hair-like cotton fibrils are likely to be degraded first and partly detached from the main fiber chain and sulphur dye bonds are broken from the yarn surface. Rotating fabric inside the machine with





steel balls, hydrolyzes more bonds due to mechanical friction and restores original white color. The longer treatment time also accelerates this reaction (Fig. 2c). The fading in each of the trials is as indicated in Table 6.

**Optimum conditions for the K/S value**

Using ridge analysis method of optimization, optimum fading with Cellusoft Conc. L enzyme is obtained at concentration of 1.25–1.33%, time about 45–48 min and with 5–9 steel balls.

**Effect of neutral cellulase enzyme on dark blue indigo dyed denim fabric**

The correlation aspects for treatment of above fabric with Neutrancell CLD are as follows.

Multiple R	0.960
Squared multiple R	0.921
Adjusted squared multiple R	0.779
Standard error of estimate	0.474

The response model evaluated in this study is significant, with a coefficient of determination R<sup>2</sup> of 0.921 at a confidence level of 95%. The response surface equation obtained for the K/S value is as follows

$$\begin{aligned} \frac{K}{S} \text{ value} = & 19.514 - 0.337X - 0.624Y - 0.729Z \\ & - 0.052X^2 + 0.343Y^2 - 0.935Z^2 \\ & - 0.288XY + 0.385YZ + 0.093ZX \end{aligned}$$

where X concentration, Y treatment time and Z mechanical agitation.

**Table 6 Experimental conditions and results for the effect of acid cellulase on sulphur black dyed denim fabric**

Concentration (%)	Time (minutes)	Mechanical agitation (No. of steel balls)	K/S value	Weight loss (%)
0	0	0	26.88 <sup>a</sup>	–
0.5	30	5	19.38	2
2	30	5	17.06	2.49
0.5	60	5	18.89	2.94
2	60	5	16.48	3.19
0.5	45	0	16.67	2.56
2	45	0	15.95	2.73
0.5	45	10	15.37	3.5
2	45	10	15.36	4.5
1.25	30	0	18.43	2.07
1.25	60	0	18.36	2.4
1.25	30	10	15.89	3.37
1.25	60	10	13.15	4.5
1.25	45	5	21.60	2.44
1.25	45	5	20.97	3.06
1.25	45	5	20.55	2.1

<sup>a</sup> K/S value of desized fabric is 26.88

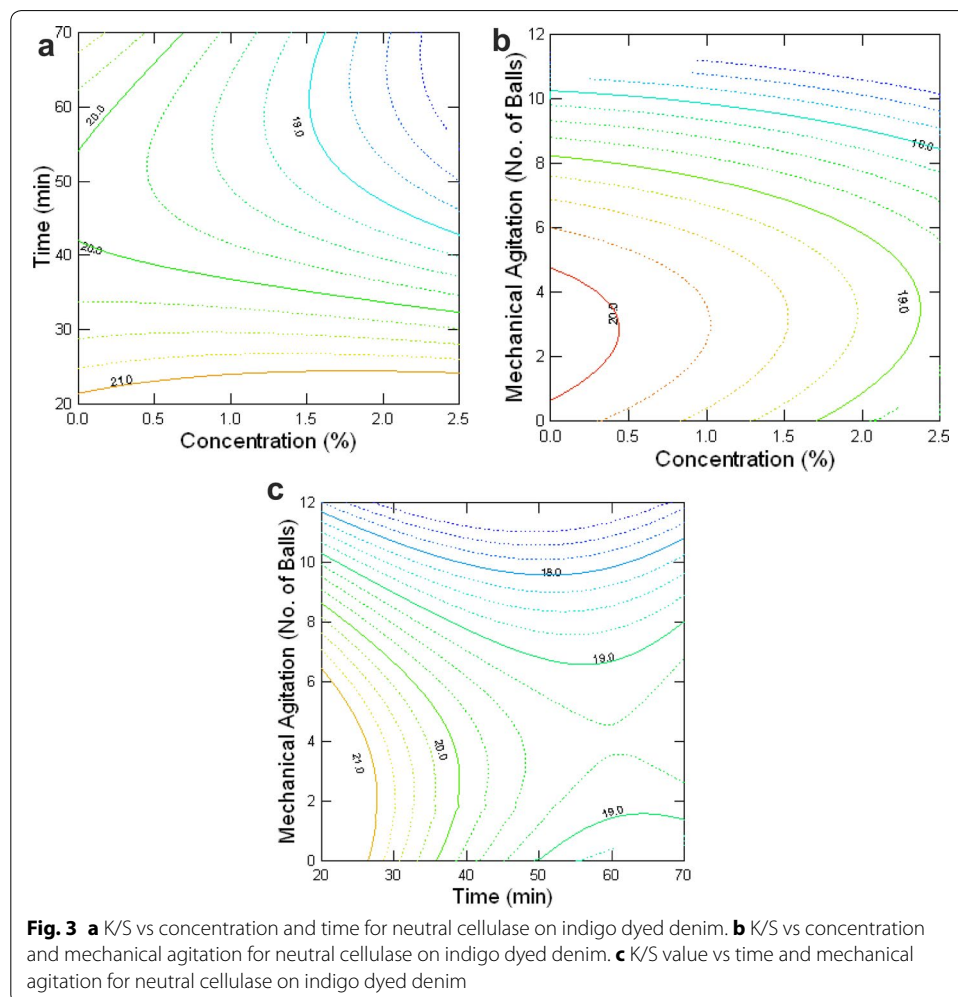


The computed F ratio (6.487) and p value 0.027 (less than 0.05) shows that the model is significant.

In Fig. 3b, c plots, the interactive effect of variables indicates reduction in the color strength value with the increase in the level of mechanical agitation. This may be due to the fact that by increasing the number of steel balls applied, the abrasion of the fabric surface will increase which creates more accessible sites for enzyme attack. The combined effect of enzyme and mechanical action facilitates removal of indigo dye trapped in cellulose fibre (Cavaco-Paulo et al. 1999). Also, the fading increased with increasing treatment time as understood from Fig. 3c, for reasons similar to that in acid enzymes. With regards to the effect of concentration of enzyme, as expected the K/S value decreases with increasing enzyme concentration. However the fading is less compared to that with acid enzyme as observed from the values in Table 7.

**Optimum conditions for the K/S value**

Using ridge analysis method, optimum fading with Neutrancell CLD enzyme can be achieved at concentration of 1.25–1.33%, time 75–76 min and with 5–10 steel balls at 95% confidence level.



**Table 7 Experimental conditions and measured values for neutral cellulase for dark blue indigo dyed denim fabric**

Concentration (%)	Time (minutes)	Mechanical agitation (No. of steel balls)	K/S value	Weight loss (%)
0	0	0	22.32 <sup>a</sup>	–
0.5	60	5	20.68	0.47
2	60	5	20.48	1.41
0.5	90	5	19.70	0.57
2	90	5	18.35	2.23
0.5	75	0	19.35	0.45
2	75	0	18.59	1.32
0.5	75	10	18.27	1.49
2	75	10	17.88	2.94
1.25	60	0	20.70	0.53
1.25	90	0	19.07	1.00
1.25	60	10	18.00	0.94
1.25	90	10	17.82	1.97
1.25	75	5	19.88	0.80
1.25	75	5	19.12	0.81
1.25	75	5	19.52	0.74

<sup>a</sup> K/S value of desized fabric is 22.32

**Effect of neutral cellulase on sulphur black dyed denim fabric**

The effect of enzyme Neutrancell CLD in terms of concentration, process time and mechanical agitation on the K/S value of sulphur black dyed denim fabric was studied as per experimental design and values obtained are shown.

Multiple R	0.988
Squared multiple R	0.977
Adjusted squared multiple R	0.936
Standard error of estimate	0.533

The response model evaluated in this study is significant, with a coefficient of determination R<sup>2</sup> of 0.977 at a confidence level of 95%. The response surface equation obtained for K/S value is as follows

$$\begin{aligned} \frac{K}{S} \text{ value} = & 19.143 - 0.310X - 1.378Y - 1.785Z \\ & - 0.661X^2 - 1.1001Y^2 - 1.739Z^2 \\ & + 0.355XY - 0.947YZ - 0.094ZX \end{aligned}$$

where X concentration, Y treatment time and Z mechanical agitation.

From the analysis of variance, the computed F-ratio and p value are 23.563 and 0.001 (less than 0.05) respectively which shows that the model is significant.

Considerable fading is observed for all the experimental samples as shown in Table 8. From contour plots (Fig. 4a–c), it can be seen that significant fading effect is obtained particularly at medium to higher concentrations on treatment with Neutrancell CLD enzyme. This is likely to be due to the fact that sulphur black dye being a dark shade have higher dye content on the fabric surface and during washing, along with the hydrolysis of

**Table 8 Experimental conditions and measured value for neutral cellulase for sulphur black dyed denim fabric**

Concentration (%)	Time (minute)	Mechanical agitation (No. of steel balls)	K/S value	Weight loss (%)
0	0	0	26.88 <sup>a</sup>	–
0.5	60	5	19.67	1.66
2	60	5	18.09	2.01
0.5	90	5	16.16	1.44
2	90	5	15.99	2.12
0.5	75	0	18.51	1.44
2	75	0	18.33	1.51
0.5	75	10	15.33	1.61
2	75	10	14.78	1.91
1.25	60	0	18.69	0.63
1.25	90	0	17.88	1.07
1.25	60	10	16.82	0.87
1.25	90	10	12.21	1.15
1.25	75	5	18.25	0.97
1.25	75	5	19.46	0.96
1.25	75	5	19.70	0.95

<sup>a</sup> K/S value of desized fabric is 26.88

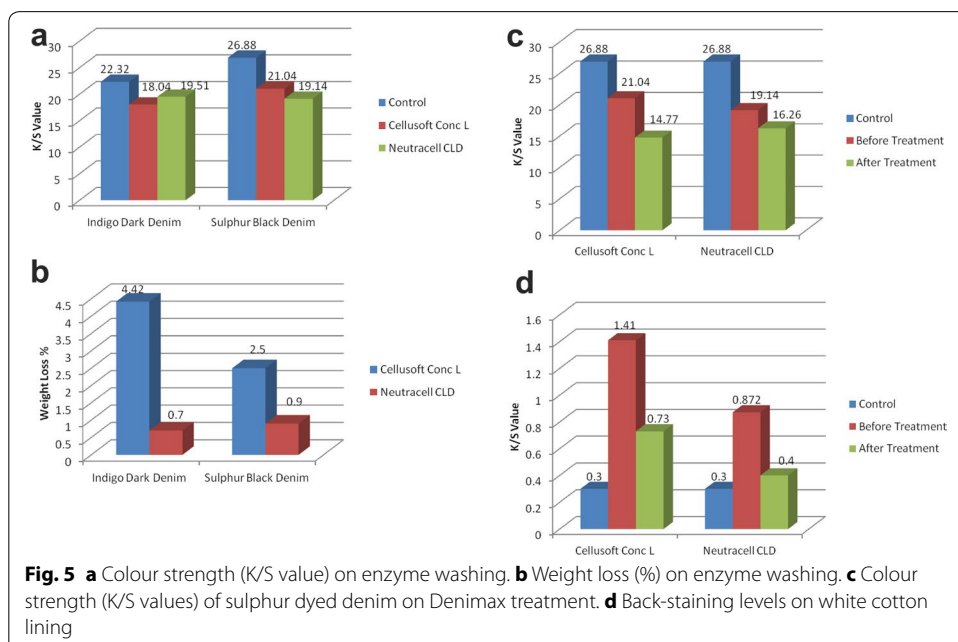
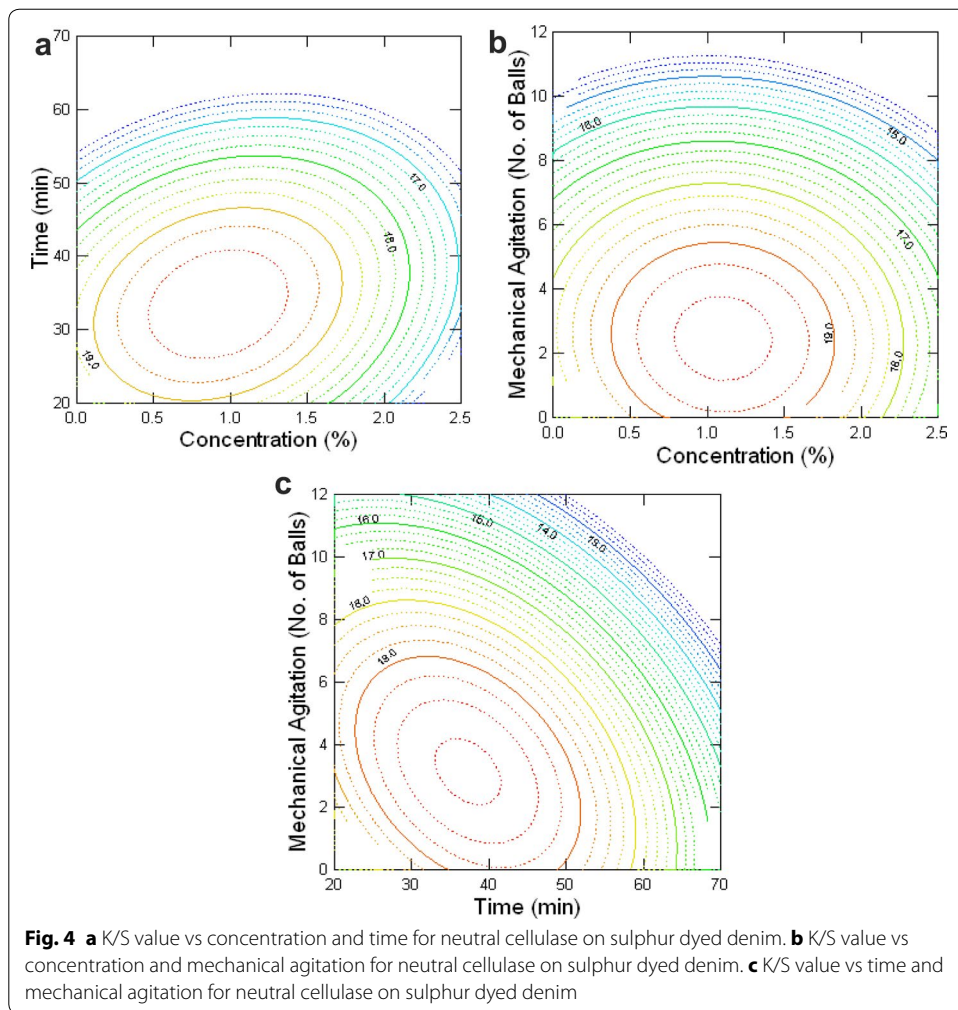
the surface fibre, sulphur dye-fibre bonds are also broken and higher amount of sulphur dye is removed from the fabric surface and faded look is obtained on the fabric surface. Higher mechanical agitation and longer treatment time assists this chemical reaction.

#### Optimum conditions for the K/S value

By applying ridge analysis method, optimum fading with Neutrancell CLD enzyme can be achieved at concentration in the range 1.25–1.28%, time about 75–82 min and with 5–9 steel balls at 95% confidence level.

#### Comparative analysis of action of enzymes on denim fabrics

To compare the effects of the two enzymes on the K/S value of the differently dyed varieties of denim fabrics, the K/S value of optimized samples are considered as shown in Fig. 5a. The fading effect of dark blue indigo and sulphur black dyed denims after enzymatic washing with Cellusoft Conc. L and Neutrancell CLD enzymes are shown in terms of K/S values. The enzymatic bio-washed denim samples showed an obvious decrease in K/S value as compared to the desized sample (control) due to the removal of surface dye particles along with the removal of short surface fibrils during treatment. The denim fabric treated with Cellusoft Conc. L enzyme showed higher fading effect compared to Neutrancell CLD enzyme, as the former is acidic in nature. Acid enzyme is more aggressive on the cotton cellulose (denim fabric) and causes more dye particles removal compared to neutral cellulase (Klahorst et al. 1994). The two cellulase enzymes differ in their amino acid compositions. The higher number of adsorption sites on acid enzyme is the likely cause for its stronger action (Campos et al. 2000). The result of weight loss (shown in Fig. 5b) also confirms the severity of Cellusoft Conc. L enzyme with greater weight loss %. Neutrancell CLD being neutral enzyme has milder action hence lower hydrolysis



level causing less weight loss % with lower reduction in the K/S value observed in indigo dyed denim samples.

Interestingly, the trend shown by sulphur dyed denim fabric is different from indigo dyed denim. This may be due to the reason that the dye removed from denim material after the treatment with cellulase cause back staining or re-deposition and re-coloration of black threads and black coloration of white threads, resulting in less contrast between black and white threads. Moreover, higher amount of dye is present on the surface of the fabric and hence more dye is released in the bath, thus resulting in heavy back staining. Cellusoft Conc. L being an acid enzyme shows higher level of back-staining as compared to neutral enzyme and results in less contrast or higher K/S value. Also sulphur dye particles have less affinity towards neutral cellulase as compared to acid cellulase. Thus, chances of back-staining are less and better contrast and higher reduction in K/S value is achieved with Neutrancell CLD.

#### **Further study on back-staining**

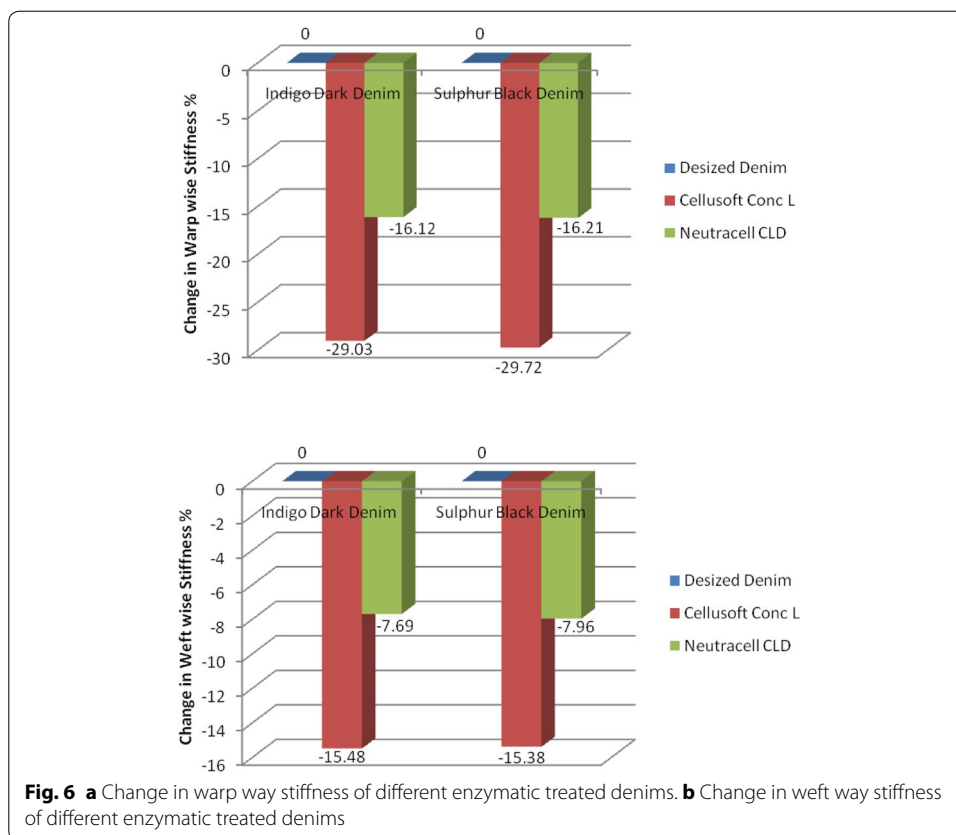
To study the effect of Denimax core-1380 on back-staining the K/S values of white cotton pocket material attached to both the denim fabrics was assessed with or without treatment of 1% of the stain regulating agent. In case of indigo dyed denim samples, no substantial difference was observed in the K/S values of white cotton pocket material, with or without the treatment. The fading effect of sulphur dyed denim samples with two different enzymes before and after Denimax core 1380 S treatment are shown in Fig. 5c and also for white cotton pocket lining fabric in Fig. 5d. The possible explanation for back staining in sulphur colour is as follows.

During enzyme washing, cellulose is degraded and sulphur dye is released. The cellulase enzymes are temporarily bound to the cellulose by means of an anchor. The surface fibre possibly gets split in terms of 1,4- $\beta$  glycoside composition (Kumar et al. 1997). After this process it is made available for further hydrolysis reaction. The cellulose is degraded hydrolytically by cellulases partially until it becomes glucose. The glucose to some extent is likely to reduce the sulphur dye, both on the fibre and in the treatment liquor (Akram 2010). This reduced form has limited affinity to cellulose fibre and thus soils the weft thread and the pocket lining. The acid cellulase enzymes have a strong effect on cellulose hydrolysis and create more glucose formation, resulting in increased back staining of the denim garment. Neutral cellulase as compared to acid cellulase, are less aggressive and hence there is less back staining.

It is clear from Fig. 5c, d that the enzymatic washing along with Denimax core 1380 S imparts significant reduction in K/S value (back-staining) of the white cotton fabric (pocket material) as well as denim fabric surface. The K/S value (back-staining) for white cotton lining fabric is found reduced up to 48.22% by adding Denimax enzyme along with Cellusoft Conc. L enzymes. This may be due to the fact that Denimax core 1380 S is formulated with special anti redeposition agents to ensure minimal back-staining of the fabric.

#### **Changes of fabric stiffness (bending length) after different enzymatic washing**

The change in the stiffness of the fabrics after enzymatic treatment for both warp and weft way are shown in Fig. 6a, b. The stiffness of both denim materials reduced compared



to respective desized control samples. This may be due to the fact that the cotton fibres are loosened by degradation and partly detachment of fibrils causing increased softness. Moreover the rubbing action due to steel balls has a great influence on the increased softness of the denim fabrics. Consequently, bending length was less and stiffness got reduced. It is clear from the figures that the Cellusoft Conc. L has great influence on the stiffness in both warp and weft directions. Stiffness value was reduced up to 29.72 and 15.48% in warp direction and weft direction respectively. Neutrancell CLD being milder in action, showed lower reduction in stiffness.

Moreover, the figures show that in the warp wise direction reduction in stiffness is higher as compared to weft direction. This could be attributed to the construction of fabrics which were 2 ends up warp face twill weave. As a result warp yarns face higher amount of friction than weft yarns. The figures also shows that trend of reduction in stiffness in both warp and weft way direction treated with both the enzymes are similar for indigo and sulphur dyed denims. This may be because the effect of enzymes on stiffness is independent of the dye applied to the fabric.

### Conclusions

The treatment of denim fabrics with cellulase enzymes gave effective results in terms of colour fading. The fading effect achieved was shown by the change in colour strength while the removal of surface fibres was clear from the loss in weight of the denim qualities. The effect was more in case of acid cellulase due to their stronger action than

neutral cellulose. However, in sulphur dyed fabric, the back staining was significant in case of acid enzyme, because of high colour removal and subsequently its redeposition. The application of anti-back staining agent was found to be quite useful in this case. Good softness was achieved in both the fabric qualities, shown in terms of decrease in bending length.

#### Authors' contributions

AKP, AM and NB carried out the enzyme studies on denim, participated in the sequence alignment and drafted the manuscript. All authors read and approved the final manuscript.

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#### Competing interests

The authors declare that they have no competing interests.

#### Ethics approval and consent to participate

Not applicable.

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#### References

- Akram, R. (2010) Anti back staining method for denim. Retrieved February 23, 2015, from <http://www.denimprocessing.blogspot.in/2010/01/anti-backstaining-method-for-denim.html>.
- Andreas, J., Campose, R., Gubitz, G., & Cavaco-Paulo, A. (2000). Influence of cellulases on indigo back staining. *Textile Research Journal*, 70(7), 628–632.
- Campos, R., Cavaco-Paulo, A., Andreas, J., & Gubitz, G. (2000). Indigo-cellulase interactions. *Textile Research Journal*, 70(6), 532–536.
- Cavaco-Paulo, A., Morgado, J., Andreas, J., & Kilburn, D. (1999). Interactions of cotton with CBD peptides. *Enzyme Microbial Technology*, 25, 639–643.
- Heikinheimo, L., Buchert, J., Miettinen-Oinonen, A., & Suminen, P. (2000). Treating of denim fabrics with *Trichoderma Reesei* cellulases. *Textile Research Journal*, 70(11), 969–973.
- Klahorst, S., Kumar, A., & Mullins, M. (1994). Optimizing the use of cellulose enzymes. *Textile Chemist & Colorist*, 26(2), 13–18.
- Kumar, A., Yoon, M., & Purtell, C. (1997). Optimizing the use of cellulose enzymes in finishing cellulosic fabrics. *Textile Chemist & Colorist*, 29(4), 37–42.
- Patra, A. K. (2003). Enzymes for wet-processing pretreatments. *Textile Asia*, 34(9), 46–51.
- Paul, R. (2015). Denim and jeans: an overview. In *Denim: manufacture, finishing and application*, (pp. 1–11). Woodhead Publishing, Cambridge.

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