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Process optimization for bioscouring of cotton and lycra cotton weft knits by Box and Behnken design

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ABSTRACT

The aim of the study was to discover the effect of experimental parameters like pH, enzyme dosage and temperature, on the removal of natural and accidental impurities from cotton and lycra cotton weft knitted fabrics, to make them suitable for further processing like dyeing and finishing. The optimal experimental conditions and their effects have been ascertained by response surface methodology using the Box–Behnken model. The optimum values were found to be pH 8.5, enzyme dosage 0.4% on weight of fabric and temperature 55 °C. The r^2 values and the *F* values indicate that the effect of all the parameters taken together is significant. Based on the results of the study it can be understood that the bioscouring process performed with these optimal values is suitable for pretreatment of cotton and lycra cotton weft knitted fabrics.

1. Introduction

Textile materials possess a variety of impurities. Some are natural and some may be added for better spinning ability or accidentally acquired during handling. All such impurities are to be removed before dyeing or printing. The processes which are used for the removal of these impurities are called preparatory processes and are classified into two categories: cleaning processes (scouring) where bulk of the foreign matter or impurities are removed by physical or chemical means and whitening processes (bleaching) where trace coloring matters are destroyed chemically or the whiteness of the material is improved optically (Hossain & Uddin, 2011).

The surface layers of cotton contain non-cellulosic components like lipids, waxes, pectins, organic acids, protein/nitrogenous matters, non-cellulosic polysaccharides and other unidentified substances. Waxes are very complex substances composed of high molecular weight alcohols and fatty acids in free or esterified form. They are chemically linked to cellulose or pectin or alternatively to residual proteins (Losonczi, 2004). Waxes and pectin are mainly responsible for the non-absorbent characteristics of raw cotton, which hinder the wet processing treatments (Ismal, 2008). All types of impurities and contaminants, which are hydrophobic in nature, should be removed from the fabrics after the scouring process as they interfere with further aqueous chemical processes such as bleaching, dyeing and finishing (Ramasamy & Kandasamy, 2004).

Currently, enzymes are becoming increasingly important in sustainable technology and green chemistry. Enzymes are complex protein ferments secreted by living organisms, capable of catalyzing chemical reactions of nature efficiently, without leaving any pollutants behind. Enzymes, being natural products (proteins), are easily and completely bio-degradable (http://www.aidic.it/IBIC2008/webpapers/2Opwis.pdf; Singh & Goel, 2004). Many enzymes have been used for the cotton scouring process but pectate lyase enzyme is most suitable for the bioscouring process due to two reasons. First, pectinases favor acidic conditions for efficient hydrolysis of pectate, whereas pectate lyases have alkaline pH optima. The methyl esters in pectin are rapidly hydrolyzed under alkaline conditions and optimal temperature to facilitate scouring of cotton Secondly, pectic substances in cotton fiber are in the form of pectate which are the favored substrate of pectate lyase (http://www.jbc.org/content/280/10/9431.long). Further, the enzyme pectate lyase helps to degrade pectin and remove wax from raw cotton, at lower temperatures than the conventional scouring process. This reduces the number of rinsing baths and resources needed, resulting in savings of 990 kg carbon dioxide (CO_2) per ton of material (http://www.novozymes.com/en/news/news-archive/Pages/Enzymes-cut-industrial-emissions-resource-use—New-study.aspx).

Pectate lyase enzyme belongs to the family of lyases, specifically those carbon–oxygen lyases acting on polysaccharides. The systematic name of this enzyme class is (1,4)-alpha-D-galacturonan lyase

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(http://en.wikipedia.org/wiki/Pectate_lyase; Rodríguez, Orchard, & Seymour, 2002). Lyases catalyze the addition of groups to double bonds or the formation of double bonds through the removal of groups. The catalysis of the reaction is as follows: pectate = pectate + a pectate oligosaccharide with 4-(4-deoxy-alpha-D-gluc-4-enuronosyl)-D-galacturonate end.

This reaction is the eliminative cleavage of pectate to give oligosaccharide with 4-deoxy-alpha-D-gluc-4-enuronosyl groups at their non-reducing ends (Jenkins, Shevchik, Pattat, & Pickersgill, 2004); thereby degrading the pectin–wax interface between the wax and the primary cotton wall.

Cotton plays an important role in the world economy and the consumption ratio in India and the world is high. It may be noted that the worldwide consumption and growth of spandex (lycra) is 30–40% a year and when combined with cotton it has many advantages of comfort, stretch and recovery (Senthilkumar, Anbumani, & Hayavadana, 2011). Thereby, the present investigation was made on bioscouring of cotton and lycra cotton weft knitted fabrics with pectate lyase enzyme. The pectate lyase (EC 4.2.2.2) used for the experiment, is a brown liquid of strength 375 APSU/g (Alkaline Pectinase Standard Units), supplied by Novozymes, Denmark with CAS No.9015-75-2. This process was optimized by Box–Behnken design and finally correlated with process variables such as pH, enzyme dosage and temperature.

2. Materials and methods

100% cotton yarn of 30s count was used to knit single jersey fabrics using the Camber machine. 40 denier lycra yarn was used along with the 100% cotton yarn to knit the lycra cotton single jersey fabrics (lycra 4% and cotton 96%). The gray 100% cotton fabric was first wetted with the 0.5 grams per liter (gpl) wetting oil (Kierlon Jet) and 0.5 gpl lubricant (Primasol Jet) for 15 min in the soft flow machine. Pectate lyase enzyme for scouring was added, after the pH of the bath was adjusted to 8.5–9 with the help of soda ash and acetic acid. The temperature was raised to 55 °C and the fabric was rotated in the soft flow machine for 30 min. The temperature was increased to 75 °C to deactivate the enzyme. Any increase from optimum temperature will cause denaturation of the enzyme leading to change in structure of enzyme and loss of activity. The optimum temperature for pectate lyase is 50–55 °C. Hence the temperature of 75 °C was chosen to deactivate the enzyme (http://www.worthington-biochem.com/introbiochem/enzymes.pdf; http://faculty.ksu.edu.sa/52876/Undergraduate%20lecture%20slides/Protein%20structure.pdf). The bath was drained and the fabric was subjected to a cold wash for 10 min at room temperature.

In the case of lycra cotton, the gray fabric was passed through a heat setting machine to set the lycra for the processing treatment. The fabric was worked for 30 min at 80 °C with 0.5 gpl demineralizing agent (Altranol ELB), to remove the oils from the lycra and make it suitable for pretreatment. The bath was drained and the pretreatment processes following the demineralizing process was similar to that of 100% cotton fabric pretreatment.

2.1. Design of experiment

In order to ascertain optimum process conditions and to evaluate the effect of the enzyme on the properties of 100% cotton and lycra cotton weft knits, an experimental design was used. The response surface design, Box and Behnken model, with three levels of the variables and 15 different conditions was chosen for the study. The Minitab 14 software was used for the analysis of the data obtained. A multiple linear regression analysis was used to determine the relationship between these variables as represented by the response surface equation.

Table 1
Variables and their levels in experimental plan.

Variables	Bioscouring		
	Coded level		
	−1	0	+1
pH	8	8.5	9
Enzyme dosage (pectate lyase enzyme)	0.3	0.4	0.5
Temperature	45	55	65
Time	30 min		
MLR (material liquor ratio)	1:10		

$$Y = B_0 + B_1X_1 + B_2X_2 + B_3X_3 + B_4X_1X_1 + B_5X_2X_2 + B_6X_3X_3 + B_7X_1X_2 + B_8X_1X_3 + B_9X_2X_3$$

where Y is the response from each experiment. B_0 is a constant, B_1–B_9 are coefficients of each monomial and X_1, X_2 and X_3 are the variables. A negative coefficient of a variable in the response surface equation indicates that the particular property or response decreases with an increase in that variable and a positive coefficient indicates that the same property increases with the increase of the variable analyzed (Tyagi, Singh, Gupta, Goyal, & Salhotra, 2003). In this study, the parameters analyzed namely weight loss and absorbency is the response Y for 100% cotton and lycra cotton fabrics; pH, enzyme dosage and temperature are the variables X_1, X_2 and X_3 respectively.

2.2. Box and Behnken design

To optimize the bioscouring process, the Box and Behnken design was utilized during the investigation. The range of process parameters were taken in three different levels at equal intervals and coded as (−1, 0, +1) namely pH (8, 8.5, 9), enzyme dosage (0.3%, 0.4%, 0.5%) and temperature (45 °C, 55 °C, 65 °C) as given in Table 1. The variables (pH, enzyme dosage and temperature) and their levels (−1, 0, +1) were chosen after many trials and based on the scouring efficiency. These variables, in three levels, were used in different combinations according to the Box and Behnken 3 level design as shown in Table 2.

The samples were subjected to bioscouring as per the 15 trials suggested by the Box and Behnken design. After each scouring process the fabric was dried and conditioned for 24 h in standard testing environment. The samples were tested for weight loss and absorbency to evaluate the efficiency of the process.

The absorbency test was carried out as per the AATCC Test Method 79, 2007. The conditioned fabric was spread over an embroidery hoop without wrinkles and kept 9.5 mm below a buret positioned to deliver 15–25 drops of water per milliliter. A drop of distilled or deionized water was allowed to fall on the cloth. The stopwatch was started immediately and the time required for the drop of water to lose its specular reflection and appear as a dull wet spot, was noted. The mean value of ten readings was taken and the shorter the average time the more absorbent the fabric.

The extent of chemical and enzymatic hydrolysis can be measured by weight loss. Pretreatment removes the impurities, pectins and waxes, and causes enzymatic hydrolysis of cuticle components resulting in loss in weight (Degani, Gepstein, & Dosoretz, 2002). After conditioning the samples for 24 h in standard testing atmosphere, the mass of the sample was recorded using an electronic balance. The weight of the fabric after each pretreatment process was measured according to the ASTM D 3776 small swatch option.

Table 2
Treatment conditions and the responses observed and predicted after bioscouring 100% cotton weft knits with pectate lyase enzyme.

Sample	Coded levels			Variables			Weight loss		Absorbency	
				pH	Enzyme dosage	Temperature	Predicted value	Observed value	Predicted value	Observed value
1	1	0	1	9	0.4	65	3.337	3.49	5.375	5
2	1	1	0	9	0.5	55	3.132	3.17	7	8
3	−1	1	0	8	0.5	55	3.067	3.54	5.75	6
4	0	−1	1	8.5	0.3	65	3.11	3.43	4.375	5
5	0	1	−1	8.5	0.5	45	3.28	2.96	7.625	7
6	1	0	−1	9	0.4	45	2.727	3.01	8.375	8
7	−1	0	1	8	0.4	65	2.872	2.59	5.625	6
8	0	0	0	8.5	0.4	55	2.173	2.17	3	3
9	0	1	1	8.5	0.5	65	3.44	3.25	4.625	4
10	1	−1	0	9	0.3	55	2.752	2.28	5.25	5
11	−1	0	−1	8	0.4	45	2.732	2.58	8.625	9
12	0	−1	−1	8.5	0.3	45	2.52	2.71	7.375	8
13	−1	−1	0	8	0.3	55	2.357	2.32	7	6
14	0	0	0	8.5	0.4	55	2.173	2.18	3	3
15	0	0	0	8.5	0.4	55	2.173	2.17	3	3

The mean value of ten readings was taken and the weight loss of the samples was calculated using the formula,

$$\%WL = \left[\frac{W1 - W2}{W1}\right] * 100$$

where $\%WL$ = percentage weight loss, $W1$ = initial weight of the fabric, and $W2$ = weight of fabric after the scouring process.

3. Results and discussion

3.1. 100% cotton weft knitted fabric

The most important parameters checked after scouring of 100% cotton knitted fabrics are absorbency and weight loss percentage. The observed and predicted results of the 15 trials are presented in Table 2. The response surface plots for weight loss and absorbency are given in Fig. 1. The response surface equations for weight loss (percentage) and absorbency (time in seconds) are given below:

$$\begin{aligned}\text{Weight loss} = {} & (2.1733) + (0.115X_1) + (0.2725X_2) + (0.1875X_3) \\ & + (0.24208X_1X_1) + (0.41208X_2X_2) + (0.50208X_3X_3) \\ & + (-0.0825X_1X_2) + (0.1175X_1X_3) + (-0.1075X_2X_3)\end{aligned}$$

$$\begin{aligned}\text{Absorbency} = {} & (3) + (-0.125X_1) + (0.125X_2) + (-1.5X_3) \\ & + (2.125X_1X_1) + (1.125X_2X_2) + (1.875X_3X_3) \\ & + (0.75X_1X_2) + (0X_1X_3) + (0X_2X_3)\end{aligned}$$

The correlation coefficient between the observed and the calculated values obtained from the above equations for weight loss and absorbency are 0.861 and 0.960 respectively. The goodness of fit of the model for weight loss and absorbency were 85.2% and 92.3% respectively. The *F* value of 5.37 for weight loss and 6.63 for absorbency, show that the effect of all the parameters taken together was significant at 5% level. This shows that the observed values are in agreement with the response surface equation.

3.1.1. Weight loss

In Fig. 1, the response surface plots A, B and C reveal that the weight loss was lowest in the 0 level when compared to the (+1) and (−1) levels of the three variables. When the three variables pH, enzyme concentration and temperature are at the (0) level, the weight loss is 2.173% as shown in Table 2. Commercially a weight loss of 3–6% is considered acceptable and for higher production and better profit margins, it is necessary to control weight loss in knit processing (http://cdn.intechopen.com/pdfs/25013/InTech-Pre_treatment_of_textiles_prior_to_dyeing.pdf; Uddin, 2010). A low weight loss coupled with good absorbency shows that effective scouring has been accomplished.

3.1.2. Absorbency

In Fig. 1, the response surface plots D, E and F show that the lowest absorbency time was seen in the (0) level when compared to the (+1) and (−1) levels of the variables pH, enzyme concentration and temperature. Removal of wax from cotton is the critical factor for improved wettability. Bioscouring affects the pectin wax interface and renders wax extractable or emulsifiable increasing absorbency (http://www.textiletoday.com.bd/index.php?pid=magazine&id=54). Table 2 shows that the absorbency time was 3 s when the three variables pH, enzyme concentration and temperature were in the (0) levels, the standard absorbency time being less than 5 s (Phatthalung, Sae-be, Suesat, Suwanruji, & Soonsinpai, 2012). The lowest absorbency time was seen in the (0) levels of the variables indicating that the scoured fabric was highly absorbent.

3.2. Lycra cotton weft knitted fabric

The lycra cotton knitted fabrics after bioscouring were checked for absorbency and weight loss percentage. The results of the 15 trials, both the observed and predicted values, are presented in Table 3. The response surface plots for weight loss and absorbency of lycra cotton weft knitted fabric are given in Fig. 2. The response surface equations for weight loss (percentage) and absorbency (time in seconds) are given here.

$$\begin{aligned}\text{Weight loss} = {} & (3.55333) + (-0.00625X_1) + (0.625X_2) \\ & + (0.06125X_3) + (0.11833\,X_1X_1) + (0.12083X_2X_2) \\ & + (0.04833\,X_3X_3) + (-0.225\,X_1X_2) + (0.02\,X_1\,X_3) \\ & + (0.0225\,X_2X_3)\end{aligned}$$

$$\begin{aligned}\text{Absorbency} = {} & (1.3333) + (-0.25\,X_1) + (-0.375\,X_2) + (-0.375X_3) \\ & + (2.4583\,X_1X_1) + (2.2083\,X_2X_2) + (1.7083X_3X_3) \\ & + (0.5X_1X_2) + (-1X_1X_3) + (-0.25X_2X_3)\end{aligned}$$

The correlation coefficient between the observed and the calculated values obtained from the above equations for weight loss and absorbency are 0.994 and 0.977 respectively. The goodness of fit of the model for weight loss and absorbency were 99.0% and

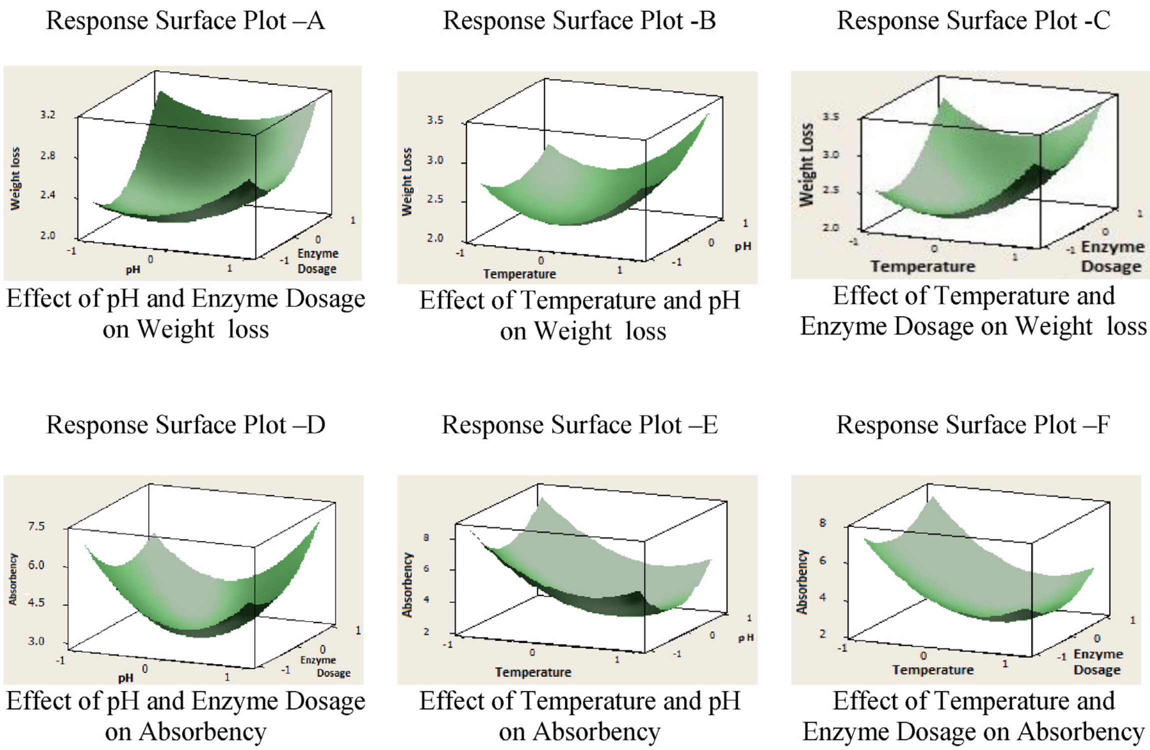

Fig. 1. Response surface plots for weight loss and absorbency of 100% cotton fabric after bioscouring with pectate lyase enzyme.

95.6% respectively. The *F* value of 52.39 for weight loss and 12.07 for absorbency, show that the effect of all the parameters taken together was significant at 1% level. This shows that the observed values are in agreement with the response surface equation.

3.2.1. *Weight loss*

From the response surface plots G, H and I in Fig. 2, it can be observed that the weight loss was lowest at the (0) level when compared to the (+1) and (−1) levels of the three variables pH, enzyme concentration and temperature. Bioscouring under optimal conditions removes the natural waxes, pectins, fats and other impurities resulting in weight loss which is comparatively lower than conventional alkaline scouring (http://textilelearner.blogspot.in/2012/07/application-of-enzyme-in-textile-wet.html). Table 3 shows that the weight loss percentage was 3.553, which is within the acceptable limits and is the lowest when compared with all other combinations of the levels of the variables. These results prove that the (0) levels are the ideal conditions for the optimization process.

3.2.2. *Absorbency*

The response surface plots J, K and L in Fig. 2, revealed that the absorbency time was the lowest in the (0) levels of the three variables pH, enzyme concentration and temperature, when compared to the (+1) and (−1) levels. Optimum conditions are essential for enzymes to work well and an increase or decrease from the optimum conditions may cause adverse actions or inactivate the enzyme (Blanchard, Graves, & Batiste, 2000). Bioscouring facilitates the removal of the pectic substances and waxes from the fabric resulting in better absorbency characteristics (Chinnadurai & Selvakumar, 2009). Table 3 shows the lowest absorbency time of 1.533 s at the (0) level of the three variables studied indicating that lycra cotton knitted fabric had very good absorbency after bioscouring.

Table 3
Treatment conditions and the responses observed and predicted after bioscouring lycra cotton weft knits with pectate lyase enzyme.

Sample	Coded levels			Variables			Weight loss		Absorbency	
				pH	Enzyme dosage	Temperature	Predicted value	Observed value	Predicted value	Observed value
1	0	0	0	8.5	0.4	55	3.553	3.55	1.333	1
2	0	0	0	8.5	0.4	55	3.553	3.55	1.333	1
3	0	1	1	8.5	0.5	65	4.431	3.86	4.25	4
4	1	1	0	9	0.5	55	4.186	3.82	5.875	6
5	1	0	1	9	0.4	65	3.795	3.81	3.875	4
6	0	1	−1	8.5	0.5	45	4.264	3.72	5.5	5
7	0	0	0	8.5	0.4	55	3.553	3.56	1.333	2
8	−1	0	−1	8	0.4	45	3.685	3.67	5.125	5
9	0	−1	−1	8.5	0.3	45	3.059	3.63	5.75	6
10	−1	1	0	8	0.5	55	4.649	3.88	5.375	6
11	0	−1	1	8.5	0.3	65	3.136	3.68	5.5	6
12	1	0	−1	9	0.4	45	3.632	3.62	6.625	7
13	−1	−1	0	8	0.3	55	2.949	3.72	7.125	7
14	1	−1	0	9	0.3	55	3.386	3.75	5.625	5
15	−1	0	1	8	0.4	65	3.767	3.78	6.375	6

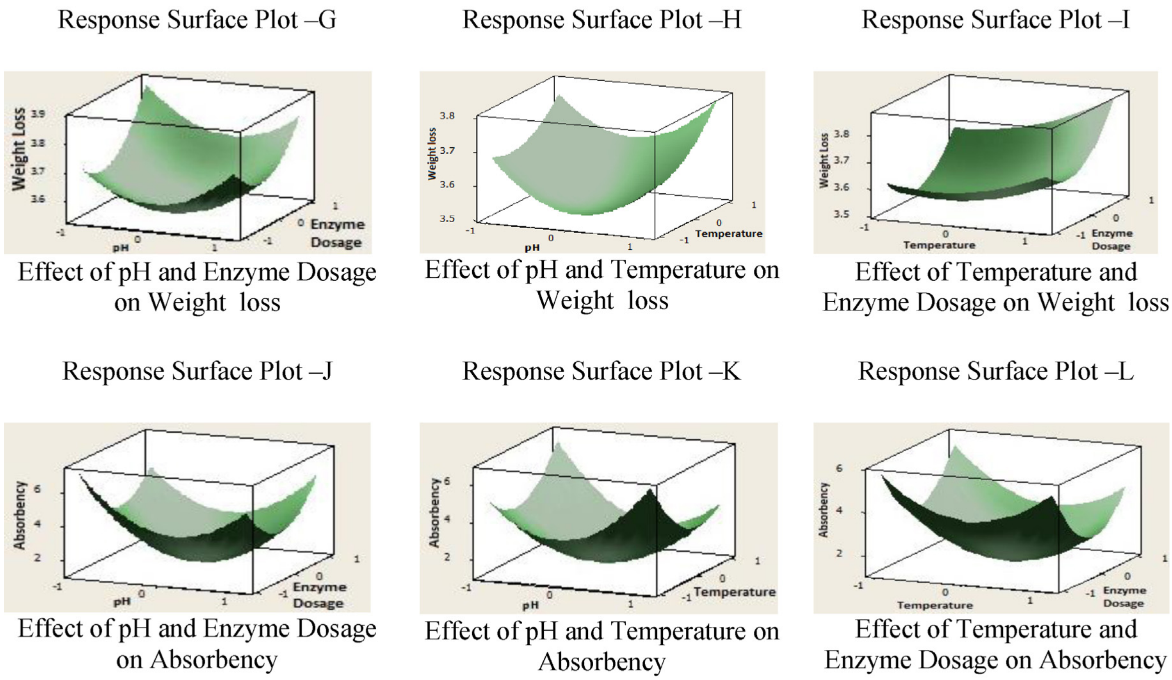

Fig. 2. Response surface plots for weight loss and absorbency of lycra cotton fabric after bioscouring with pectate lyase enzyme.

4. Conclusion

The ever growing cost of energy and the high amounts of polluted waste water resulting from the use of chemicals, coupled with rising consciousness toward sustainable technology and green chemistry, have paved the way for enzyme processing. From the observations stated above, the weight loss and absorbency were taken as the criteria for the optimization of pectate lyase enzyme for bioscouring cotton and lycra cotton weft knits and the best scouring efficiency was attributed to pH8.5, enzyme dosage 0.4% owf (on weight of fabric) and temperature 55 °C. The optimized conditions met the pretreatment requirements of good absorbency and weight loss within acceptable limits (3–6%) and were suitable for both 100% cotton and lycra cotton weft knitted fabrics. Since these conditions were statistically evaluated, they can be taken as the process conditions for bioscouring of cotton and lycra cotton weft knits.

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