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Process optimization for bioscouring of 100% cotton textiles using Box-Behnken design

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ABSTRACT

Conventional scouring processes of cotton are conducted at temperatures up to 100 °C in a highly alkaline medium (pH 10-12) with sodium hydroxide. This process is slowly being replaced by environmentally friendly and economical approach using enzymes which remove the non-cellulosic impurities without appreciable losses of strength. In this work, the paper presents the optimization of the bioscouring treatment on 100% cotton material using BEISOL PRO enzyme. For the bioscouring treatment a Box-Behnken Design experiment was used, with the independent variables such as enzyme concentration, treatment temperature and time. The test results such as weight loss, absorbency and tensile strength were also analyzed using the designed expert software 8.00 to predict the optimum process parameters. The optimum values were found to be enzyme concentration 2.5% on weight of fabric, temperature 75 °C and time 20 minute. Furthermore, the effects of bioscouring on bleaching were also investigated and compared with that of the conventionally scoured fabric. Based on the results of the study it can be understood that the bioscouring process performed much better as compared to conventional process and the process is eco-friendly and sustainable.

Keywords: Cotton Fabric, Enzyme, Alkaline Scouring, Bioscouring, Sustainable Process

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INTRODUCTION

Cotton fibre is a single biological cell with a multilayer structure. These layers are structurally and chemically different, and contain approximately 10% by weight of non-cellulosic substances such as lipids, waxes, pectin substances, organic acids, proteins/nitrogenous substances, non- cellulosic polysaccharides, and other unidentified compounds included within the outer layer of the fibre. In nature, these non-cellulosic materials create a physical hydrophobic barrier to protect the fibre from the environment throughout its development. In aqueous textile processing, the waxes and pectins impede wetting of fabric and wicking, thus obstructing effective treatments [1, 2].
Cotton yarn or fabric prior to dyeing or printing goes through a number of processes in textile processing stages and a very important process is scouring. During scouring, waxes and other hydrophobic materials are removed from the cotton fibres in order to obtain a sufficiently hydrophilic fabric [3, 4].

Conventionally, scouring is done in a hot aqueous solution of NaOH to remove hydrophobic components from the primary wall (e.g. pectin, protein and organic acids) and the cuticle (waxes and fats). However, alkaline scouring is a nonspecific process. The use of high concentrations of NaOH also requires neutralization of wastewater. Even though alkaline scouring is effective and the costs of NaOH are low, the scouring process is rather inefficient because it consumes large quantities of water and energy [5, 6]. Additionally, a growing number of consumers are also interested in clothes that are produced in an environmentally friendly way without the extensive use of harsh chemicals and hence it is clear that this process needs to be improved considerably to meet today's energy and environmental demands.

The use of enzymes in the textile industry is an example of white industrial biotechnology, which allows the development of environmentally friendly technologies in fibre processing and strategies to improve the final product quality. The consumption of energy and raw-materials, as well as increased awareness of environmental concerns related to the use and disposal of chemicals into landfills, water or release into the air during chemical processing of textiles are the principal reasons for the application of enzymes in finishing of textile materials.

Enzymatic scouring makes it possible to effectively scour fabric without negatively affecting the fabric or the environment [7, 8]. Hydrolysis by enzymes such as pectinases promotes efficient interruption of the matrix to achieve good water absorbency without the negative side effect of cellulose destruction. Enzymatic degradation of pectin accelerates the removal of waxy materials from the cotton primary wall, thus produces water wettable cotton [9, 10].

The objective of this present work is to optimize bioscouring process parameters and describe the results obtained using BEISOL PRO on 100% cotton material in order to obtain a less expensive bio-scouring technology that supports sustainable process and finally to make the comparative analysis in the performance of the enzyme during bioscouring with the conventional scouring method. The typical enzyme selected here was in the context of Ethiopia, as it is widely used in the country due to its availability.

MATERIALS AND METHODS

1.1. Fabric

Greige cotton was used for the experiment. The process trials were carried out on the fabric of plain weave 100% raw cotton fabric with 120 GSM.

1.2. Chemicals

For bioscouring a commercial grade enzymes namely BEISOL PRO was supplied by CHT India Pvt.Ltd. For conventional scouring sodium hydroxide was used. Auxiliary chemicals namely Felosan NFG (wetting agent) and Beixion NE (sequestering agent) were supplied by CHT India Pvt.Ltd.

1.3. Pretreatment Fabric

1.3.1. Desizing

The desizing was carried out in in Rota dyer at pH 7 at 60 °C for MLR 1:20 with enzyme concentration 2% (o.w.f) BEISOL T2090 and wetting agent Felosan NFG 1.5 % (o.w.f) and sequestering agent Beixion NE 2% (o.w.f). The process time for the de-sizing enzymes was prolonged to 30 minutes excluding the time required for heating at a heating rate 3°C /min.

1.3.2. Bioscouring

BEISOL PRO enzyme was used for the scouring process. The scouring process experiments were carried out in Rota dyer at pH 9 with MLR 1:20 with wetting agent Felosan NFG 1.5% (o.w.f) and sequestering agent Beixion NE 2% (o.w.f) for various enzyme concentration, temperature and time intervals.

1.3.3. Conventional alkaline scouring

Cotton fabric was boiled in an aqueous solution containing 4% (o.w.f) NaOH and wetting agent Felosan NFG 1.5% (o.w.f) and sequestering agent Beixion NE 2% (o.w.f) for 60 min using MLR 1:20 in Rota dyer. After the alkaline treatment, the fabric was washed two times with boiling water, twice with cold water and finally air dried.

1.3.4. Design of Experiment

To optimize the bioscouring process, the Box-Behnken design was utilized during the investigation. The range of independent process parameters were taken in three different levels at equal intervals such as time (10, 15, and 20) minute, enzyme concentration (1%, 2.5%, and 4%) and temperature (30 ◦C, 65 ◦C, 100◦C). The variables (time, enzyme concentration and temperature) and their levels (low, medium, high) were chosen based on supplier's recommendation and according to the Box-Behnken design statistical tools as shown in Table 1. The samples were subjected to bioscouring as per the 17 trials suggested. After each scouring process, the fabric was dried and tested for percentage weight loss, tensile strength loss and absorbency to evaluate the efficiency of the process to obtain the optimum parameters by adopting systematic statistical approach.

Table 1: Treatment conditions and the responses observed

Run	A:Enzyme Dosage (% o.w.f)	B:Temperature (°C)	C:Time (min)	Absorbency (Sec.)	Weight Loss (%)	Tensile strength loss (%)
1	2.50	65.00	15.00	14.25	1.45	1.98
2	4.00	30.00	15.00	21.58	1.41	2.31
3	2.50	65.00	15.00	14.11	1.41	2.09
4	4.00	65.00	20.00	3.85	2.02	3.90
5	1.00	100.00	15.00	5.21	1.93	3.86
6	2.50	65.00	15.00	14.23	1.51	2.57
7	1.00	65.00	20.00	10.00	1.81	3.45
8	4.00	65.00	10.00	12.00	1.73	2.80
9	1.00	65.00	10.00	15.00	1.41	2.31
10	4.00	100.00	15.00	2.42	2.31	3.96
11	2.50	65.00	15.00	10.91	1.41	2.21
12	2.50	30.00	10.00	20.21	1.38	1.59
13	2.50	100.00	10.00	2.14	2.36	4.09
14	2.50	30.00	20.00	10.25	1.79	2.90
15	1.00	30.00	15.00	26.00	1.35	1.52
16	2.50	65.00	15.00	19.00	1.45	2.11
17	2.50	100.00	20.00	1.00	2.41	4.12

2.3.5. Testing

2.3.5.1. Fabric Water Absorbency

Standard drop penetration test was carried out for the absorbency. The absorbency test standard is AATCC-79-2000. A drop of water allowed falling from a fixed height on to taut fabric and the time required for specular reflection of the water drop to disappear is measured and recorded as wetting time.

2.3.5.2. Weight loss

Under this test dry weight of the fabric sample was measured before and after the scouring process and the difference was taken as the weight loss. This would be taken as a value of the degree of scouring which gives the amount of unwanted matters removed. The performance of scouring was evaluated by calculating the weight loss% (Wt. %) as follows:

$$Wt.\ \% = \frac{W_1 - W_2}{W_1} \times 100$$

Where, $W_1$and W_2 is the weights of the fabric before and after scouring respectively.

2.3.5.3. Loss in tensile strength

Tensile strength and elongation were measured as per ASTM D 5034. A sample of 16x2.5cm was taken for the test. The tensile strength of the fabric was determined by cloth tensile strength tester. Four readings for every sample were taken and the average was calculated. The performance of scouring was evaluated by calculating the tensile strength (TS) loss% as follows:

$$TS\ Loss\ (\%) = \frac{T_1 - T_2}{T_1} \times 100$$

Where T_1 is tensile strength-before treatment and T_2 is tensile strength after treatment,

RESULTS AND DISCUSSION

The most important parameters checked after scouring of 100% cotton woven fabrics are absorbency, weight loss and tensile strength loss. The design experiment and results of the 17 trials are presented in Table 1. The response for absorbency, weight loss, and tensile strength loss are given in Fig. 1, Fig. 2, and Fig. 3, respectively.

1.4. Absorbency

The Model F-value of 22.00 implies the model is significant and there is only 0.02% chance that a "Model F-Value" this large could occur due to noise. Values of "Prob > F" less than 0.05 indicate model terms are significant. In this case B, C, C^2 are significant model terms. Values greater than 0.1000 indicate the model terms are not significant.

Table 2: ANOVA for Response Surface Quadratic Model for absorbency

Source	Sum of Squares	DF	Mean Square	F Value	Prob > F	
Model	692.27	9	76.92	22.00	0.0002	Significant
A	19.10	1	19.10	5.46	0.0521	
B	500.39	1	500.39	143.14	< 0.0001	
C	73.51	1	73.51	21.03	0.0025	
A^2	0.88	1	0.88	0.25	0.6310	
B^2	7.73	1	7.73	2.21	0.1806	
C^2	65.53	1	65.53	18.74	0.0034	
AB	1.40	1	1.40	0.40	0.5464	
AC	2.48	1	2.48	0.71	0.4274	
BC	19.45	1	19.45	5.56	0.0504	
Residual	24.47	7	3.50			
Lack of Fit	14.25	3	4.75	1.86	0.2774	not significant
Pure Error	10.23	4	2.56			
Cor Total	716.75	16				

Final Equation in Terms of Coded Factors:
Absorbency =$+13.7-1.54\,A-7.91B-3.03C+0.46A^2-1.36B^2-3.94C^2-0.59\,A\,B-0.79AC+2.2\,BC$

In Fig.1 (a), the response surface plot shows that the shortest absorbency time was seen when enzyme concentration and time were in the high levels. It shows that the absorbency time was 2.42 s when the enzyme concentration of 4% (o.w.f) and temperature 100°C were used with constant time of 15 minute. This result is the best when compared with all other combinations of the levels of the variables, as shown in Table 1 and it also proves that the high levels are the ideal conditions for the optimization process as the absorbency time required is around 5s. The shortest absorbency time seen indicates that the scoured fabric was highly absorbent.

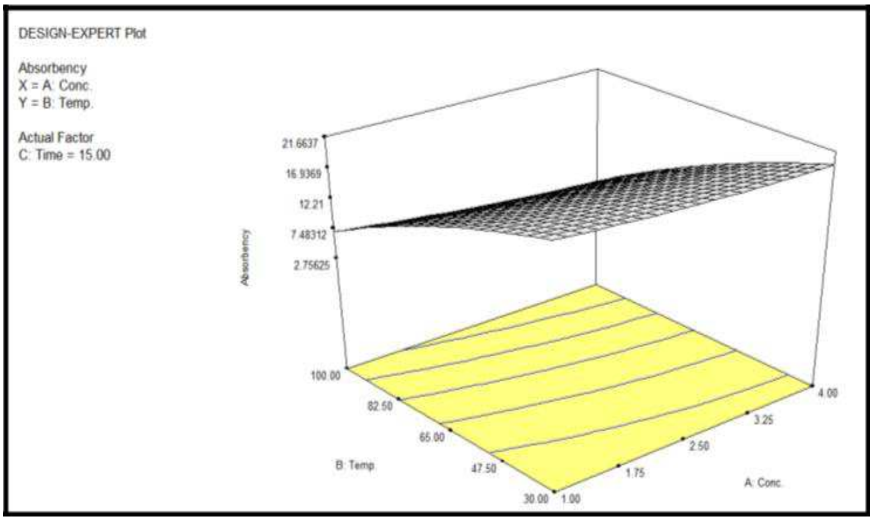

Figure 1(a): Effect of enzyme concentration and temperature on absorbency of cotton fabric at constant time of 15 minute

In Fig.1 (b), the response surface plot shows that the shortest absorbency time was also found to be when enzyme concentration and time were in the high levels. It shows that the absorbency time was 3.85 s (as indicated Table 1) was possible when the enzyme concentration of 4% (o.w.f) and time of 20 minute were used with constant of temperature 65°C.The standard acceptable absorbency time is up to 5s, it can be said that the scoured fabric was quite absorbent.

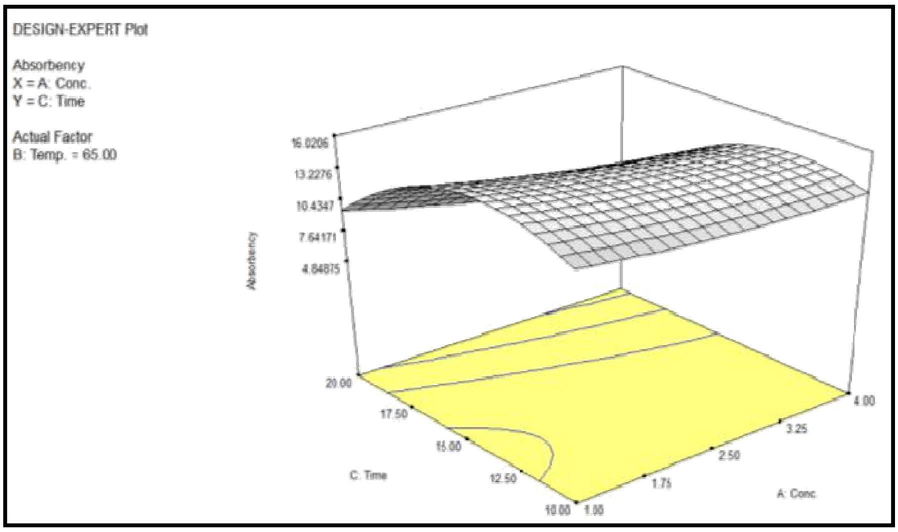

Figure 1(b): Effect of enzyme concentration and time on absorbency at constant temperature of 65°C

In Fig.1 (c), similarly the shortest absorbency time was seen when the maximum temperature and time were used. It showed that the absorbency time was 1s (as indicated in Table 1) when time of 20 minute and temperature 100 °C were used with constant enzyme concentration of 2.5% (o.w.f). The standard absorbency time expected being less than 5 s, the shortest absorbency time observed here of 1s indicates that the scoured fabric was highly absorbent.

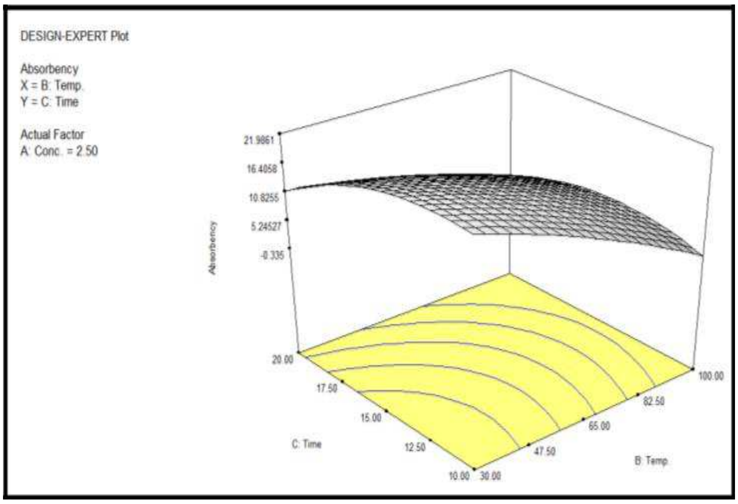

Fig.1(c): Effect of temperature and time on absorbency of cotton fabric at constant concentration of enzyme of 2.5 % (o.w.f.)

1.5. Weight Loss

The Model F-value of 100.82 implies the model is significant and there is only a 0.01% chance that a "Model F-Value" this large could occur due to noise. In this case A, B, C, B^2, C^2, AB, BC are significant model terms. The "Lack of Fit F-value" of 1.67 implies the Lack of Fit is not significant relative to the pure error.

Final Equation in Terms of Coded Factors:

Weight loss $=+1.44+0.12A+0.39B+0.1C+0.032A^2+0.27B^2+0.27C^2+0.080AB-0.028AC-0.090BC$

Table 3: ANOVA for Weight loss

Source	Sum of Squares	DF	Mean Square	F Value	Prob > F	
Model	2.20	9	0.24	100.82	< 0.0001	Significant
A	0.12	1	0.12	48.59	0.0002	
B	1.19	1	1.19	489.86	< 0.0001	
C	0.17	1	0.17	68.29	< 0.0001	
A^2	0.004	1	0.004	1.75	0.2270	
B^2	0.32	1	0.32	130.82	< 0.0001	
C^2	0.30	1	0.30	123.77	< 0.0001	
AB	0.03	1	0.03	10.58	0.0140	
AC	0.003	1	0.003	1.25	0.3005	
BC	0.03	1	0.03	13.38	0.0081	
Residual	0.02	7	0.002			
Lack of Fit	0.01	3	0.003	1.67	0.3090	not significant
Pure Error	0.00752	4	0.002			
Cor Total	2.213388	16				

From the response surface plots in Fig. 2(a), it can be observed that the weight loss increased with increase in concentration of enzyme and temperature. Results from Table.1 show that the weight loss percentage was 2.31% when the enzyme concentration of 4% on weight of fabric and temperature of 100 °C were used with constant time of 15 minute. Bioscouring under optimal conditions removes the natural waxes, pectins, fats and other impurities resulting in weight loss which is comparatively higher when compared with all other combinations of the levels of the variables. It is thus necessary that proper balance between control overweight loss and absorbency is obtained.

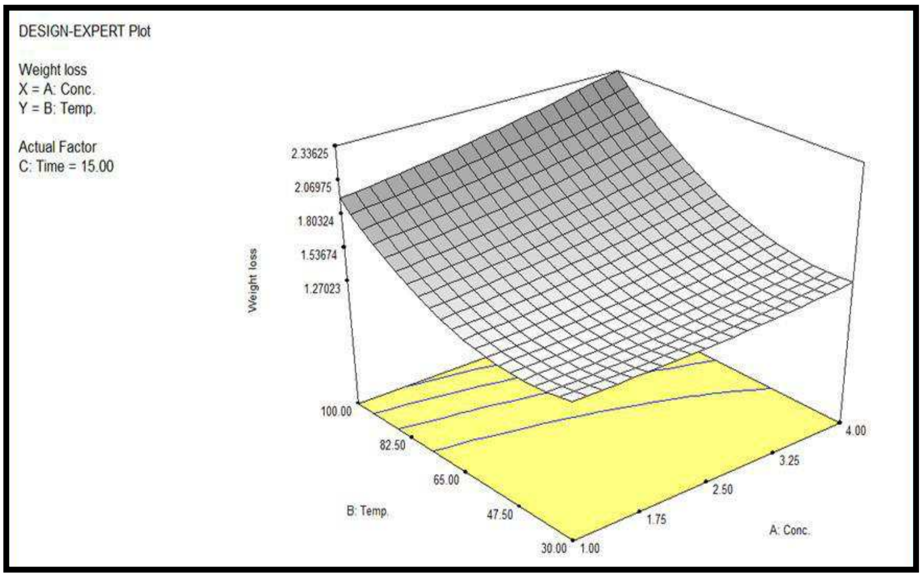

Figure 2(a): Effect of enzyme concentration and temperature on weight loss at constant time of 15 minute

From the response surface plot in Fig. 2(b), it can be observed that the weight loss was highest at the highest level of enzyme concentration and time. It shows that the weight loss percentage was 2.02% when the enzyme concentration of 4% (o.w.f) and time of 20 minute were used at constant temperature of 65^{o}C.

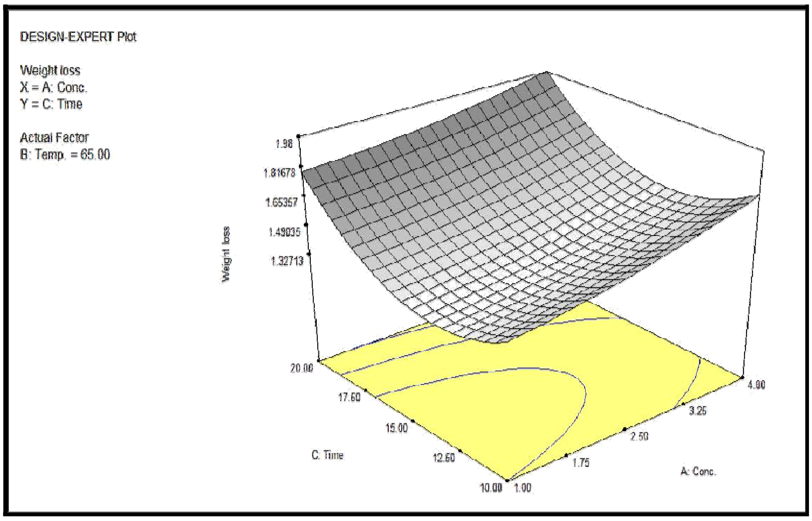

Figure 2(b): Effect of enzyme concentration and time on weight loss of cotton fabric at constant temperature of 65 ^{o}C

From the response surface plot in Fig. 2(c), it can be observed that the weight loss was highest at the highest level of time and temperature. It shows that the weight loss percentage was 2.41% when time of 20 minute and temperature of 100^{o}C were used at constant enzyme concentration of 2.5% (o.w.f).

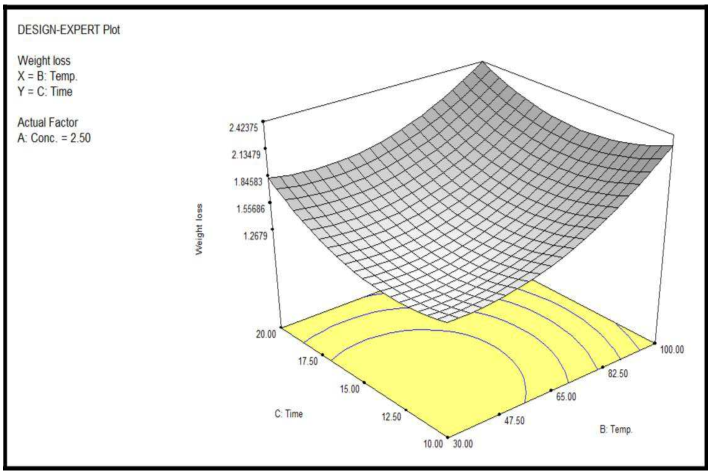

Figure 2(c): Effect of temperature and time on weight loss of cotton fabric at a constant concentration of enzyme 2.5 % (o.w.f.)

1.6. Tensile Strength loss

The Model F-value of 31.60 implies the model is significant and there is only a 0.01% chance that a "Model F-Value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case A, B, C, A^2, B^2, C^2, BC are significant model terms. The "Lack of Fit F-value" of 0.72 implies the Lack of Fit is not significant relative to the pure error.

Table 4: ANOVA for Weight loss

Source	Sum of Squares	DF	Mean Square	F Value	Prob > F	
Model	12.84	9	1.43	31.60	<0.0001	Significant
A	0.42	1	0.42	9.27	0.0187	
B	7.43	1	7.43	164.62	<0.0001	
C	1.60	1	1.60	35.49	0.001	
A^2	0.46	1	0.46	10.17	0.02	
B^2	0.64	1	0.64	14.21	0.01	
C^2	1.48	1	1.48	32.78	0.0007	
AB	0.12	1	0.12	2.64	0.15	
AC	0.0004	1	0.0004	0.009	0.93	
BC	0.41	1	0.41	9.075	0.02	
Residual	0.32	7	0.04			
Lack of Fit	0.11	3	0.04	0.72	0.59	not significant
Pure Error	0.21	4	0.051			
Cor Total	13.15	16				

Final Equation in Terms of Coded Factors:

Tensile strength=$+2.19+0.23A+0.96B+0.45C+0.33A^2+0.39B^2+0.59C^2-0.17AB-0.01AC-0.32BC$

From the response surface plots in Fig. 3(a), it can be observed that the tensile strength loss was lowest at the lowest level of enzyme concentration and temperature and vice versa. It shows that the tensile strength loss percentage was 1.52% when enzyme concentration of 1% (o.w.f) and temperature 30 ^{o}C were used at constant time 15 minute. However, at this level the highest absorbency time of 26s was seen and this was not acceptable, since the standard absorbency time should be less than 5 s. In other worlds, one has to tolerate certain degree of strength loss to have basic criterion of absorbency met. I.e. absorbency time should be less than 5s.This required little more increase in temperature and time incase enzyme concentration kept same. Otherwise, higher concentration of enzyme can also do the job.

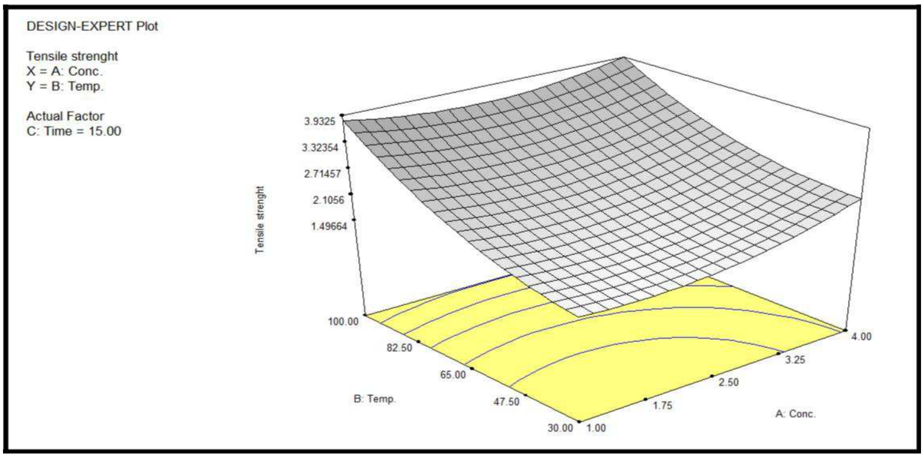

Figure 3(a): Effect of enzyme concentration and temperature on tensile strength of cotton fabric at constant time of 15 minutes

From the response surface plot in Fig. 3(b), it can be observed that the tensile strength loss was lowest at the lower level of enzyme concentration and time. Results in Table 3 show that the weight loss percentage was 1.98 % when enzyme concentration of 2.5% (o.w.f) and time of 15 minutes were used for constant temperature of 65^{o}C. In this case too absorbency time of 10.9s observed which was also unacceptable.

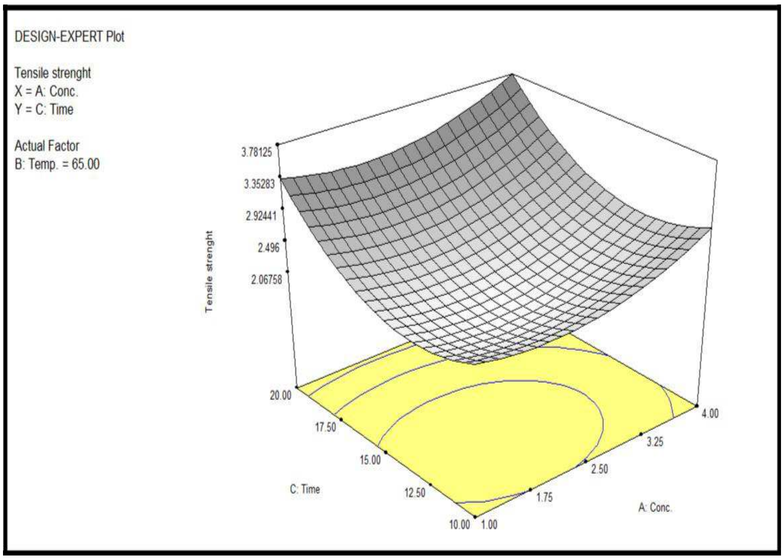

Figure 3(b): Effect of enzyme concentration and time on tensile strength of cotton fabric at constant temperature of 65^{o}C

From the response surface plot in Fig. 3(c), it can be observed that the tensile strength loss was lowest at the lower level of time and temperature. It shows that the weight loss percentage was 2.31% when time 15 minute and temperature 65 ^{o}C were used with constant enzyme concentration of 2.5% (o.w.f). However, the absorbency time was 15 s (Table 1) which was not acceptable. Hence, the other combinations of the levels of enzyme concentration, temperature and time giving absorbency time less than 5s may be acceptable as long as the tensile strength loss in such treated fabric less than 5%, if not lowest.

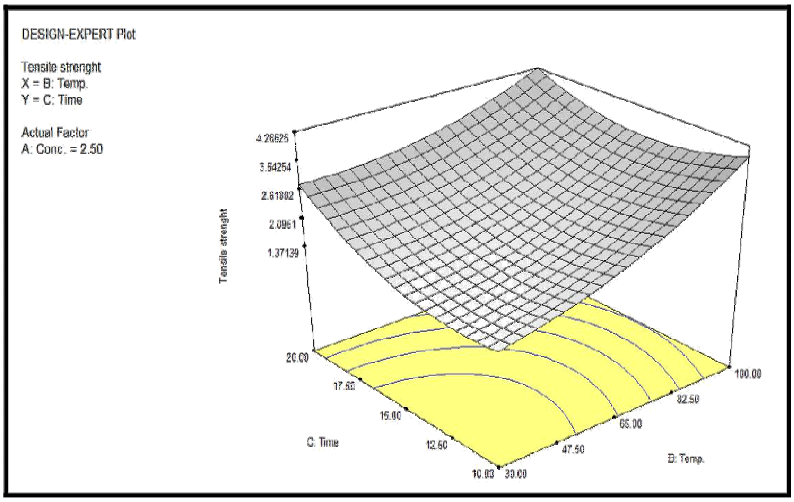

Fig. 3(c): Effect of temperature and time on tensile strength of cotton fabric at constant concentration of enzyme 2.5 % (o.w.f.)

1.7. Optimization

The process variables (time, enzyme concentration and temperature) had been optimized by using the Box-Behnken design experiment and their output values are executed by the designed expert software, as shown in Table 5.The optimum conditions were 9, enzyme concentration 2.5% (o.w.f), time 20 minute and temperature 75 ^{o}C. The output result of the designed expert software to achieve the desired parameters for predicted processes variables of solution is shown in Table 6.

Table 5: The optimum condition's variables (enzymes dosage and temperature) and their levels (Low, Medium, High)

Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
Temp	is target = 75.00	30	100	1	1	3
Time Minute	is target = 20.00	10	30	1	1	3
BEISOL PRO	is target = 2.50	1	4	1	1	3
Weight Loss	Maximize	1	22	1	1	3
Time of Absorbency	Minimize	1.35	2.41	1	1	3
Strength Loss	Minimize	1.52	4.12	1	1	3

Table 6: Predicted processes variables of solution

Number	Conc.	Temp. ◦C	Time Minute	Abs. Time sec.	Weight loss	Tensile strength
Predicted	2.5	75	20	4.98	1.96	3.44
Actual	2.5	75	20	3.36	1.83	2.78

The results obtained by actually conducting the experiments as per optimum, predicted parameters, shows that improved performance properties were obtained and they were in close agreement with predicted value.

1.8. Enzymatic scouring in comparison with alkaline scouring

1.8.1. Enzymatically scoured and conventionally scoured cotton fabric

Properties of cotton fabric treated gently with a BEISOL PRO enzyme (2.5 % o.w.f,) at optimum conditions of a pH 9 temperature of 75 ^{o}C for 20 minutes are given in Table 7 in terms of weight loss, tensile strength and absorbency. Similarly conventionally scoured cotton fabric with highly alkaline solution at boil was tested for the properties and they too are given in Table7.

Table 7: Properties of enzymatically scoured and conventionally scoured cotton fabric

Treatment Type	Properties				
	Weight loss (%),	**Absorbency. (sec.)**	**Tensile strength loss (%)**	**Whiteness index**	**Yellow index**
Alkali scoured	3.04	0.86	5.18	39.16	20.5
Bioscoured	1.83	3.36	2.78	36.49	21.23

Absorbency: The shortest absorbency time of the fabric treated with BEISOL PRO enzyme under optimized conditions was found to be 3.36 second (Table 7). The standard absorbency time being less than 5s, this was acceptable indicating that the scoured fabric was absorbent enough.

Weight loss (%): On analyzing the fabric scoured using the BEISOL PRO enzyme under the optimized conditions, the weight of the fabric reduced by up to 1.83% in comparison that of 3.04 % reduction in weight for conventional alkali scouring. There existed a significant difference between the different fabric treatments and lower percentage weight loss value suggested that there could be good saving in weight loss which can be up to1.21%. In this era, where the cotton fabric that is sold on weight basis of knitted fabric, a considerable advantage of reduced weight loss could be there for the processors.

Tensile strength and Elongation: The strength of enzyme treated fabric was greater than alkali treated fabric. Scouring with BEISOL PRO facilitates the removal of the cuticle components by partial hydrolysis. After bioscouring process, the cotton cellulose has remained cellulose structure intact with lower strength loss of only 2.4 % (Table 7). This proves that alkali scouring apart from reducing high amount of weight from the fabric sample; it also reduces the strength of the fabric due to the harsh treatment conditions. Furthermore, the fabric from the enzymatic scouring process shows softer handle and hence it requires much less softening agent in finishing process than that obtained through conventional process.

Water Saving: In bio- scouring process, only three baths are used before the actual dyeing starts, whereas in conventional process a minimum of 5 baths are used for intensive rinsing and neutralization before dyeing. Owing to the high sodium hydroxide concentration and its corrosive nature and the fact that the process is carried out at high pH range (12-14),makes essential not only thorough washing but treatment to effluent too. The bioscouring leads to saving of a total two bath which means 20%reduction in water consumption.

Energy Saving: The entire process takes place at a temperature of 75^{o}C as against 100 ^{o}C. The temperature difference is about 25^{o}C. In order to heat water from 75 ^{o}C to 100 ^{o}C the amount of heat energy required will be saved in addition to the energy saved in intensive washing after alkali treatment. (The specific heat of water is 4.186 Joule/ gram K. It requires 4.186 joules of energy to heat 1 gm. of water by 1 Kelvin).

Time Saving: The process of conventional scouring takes place in about 2 hour and 50 min for completion whereas the bio-scouring process will not take more than 75 min. Thus, for every batch dyed a time of 1 hour and 35 min per batch is possible. A conventional dyeing process takes place in about 7 hours. Whereas using bio- scouring the same dyeing can be completed in 5 hour and 25min. In terms of percentage, the time saving will be about 22%. Thus any process house can raise its production by 22% approximately by using the bio-scouring process.

Environmental benefits: Due to bioscouring effluent treatment cost will be greatly reduced. In case of caustic soda scouring at high alkalinity repeated washing as well as high concentration of alkali tremendously enhances total dissolved solid in effluent, thus increasing the ETP cost.

CONCLUSION

This investigation revealed that when tolerable limit of tensile strength loss and weight loss were taken to get maximum absorbency and optimizing of enzymatic scouring was done, the optimum conditions obtained were pH 9, enzyme concentration 2.5% (o.w.f), time 20 minute and temperature 75 ^{o}C. The optimized conditions met the pretreatment requirements for 100% cotton of good absorbency, tolerable loss in tensile strength, and weight loss within acceptable limits. Since these conditions were statistically evaluated, they can be taken as the process conditions for bioscouring of cotton. The traditional alkali scouring process ends up being high water, energy and time consuming (less productivity) and gives an increase effluent load, whereas enzymatic scouring, besides being environment friendly, removed pectin and waxes effectively to provide sufficient wettability for further treatment with a curtailed loss in tensile strength and weight as compared to that in the case of conventional scouring. The enzymatic scouring using BEISOL PRO enzyme would hence be a perfect alternative for the conventional scouring.

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