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Enzyme assisted coloring of cotton fabrics using reactive dyes

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ABSTRACT

Cellulase (enzyme) mediated reactive dyeing of cotton fabric was carried out by hydrolyzing the chitosan (biopolymer) as an alternative method of eliminating the salt for exhaustion. The overall fastness rating is compared to the conventional reactive dyeing process & the spectral values (CIELAB) & K/S value were also found to be higher than normal reactive dyeing. Significant reduction in effluent load was found & the above technique can be used for improving the substantivity of cotton for reactive dyes & increasing the efficacy of dye-fibre interaction.

KEYWORDS

Cellulase;
Color fastness;
Effluent;
Reactive dyes;
Oxidizing;
Reducing agents.

INTRODUCTION

In current practice, cellulosic fibres are predominantly dyed with reactive dyes in the presence of a considerable amount of salt and fixed under alkaline conditions. However, dye fixation efficiency on cellulosic fibres is generally low (varying from 50 -90%), thus leading to a low utilization ratio of reactive dyes[1,2]. Thus, in turn, results in a highly colored dye effluent, which is unfavorable on environmental grounds. Furthermore, the high concentrations (40 -100 g/l) of electrolyte and alkali (5-20 g/l) required in cellulose fibre dyeing may pose additional effluent problems. With the ever increasing popularity of reactive dyes, environmental problems arising from the reactive dyeing of cellulose fibres have also become increasingly aggravating[3].

Based on these considerations, it would be ideal if efficient exhaustion and fixation of reactive dyes onto fibres could occur under neutral to slightly acidic conditions in the absence of salt [4]. Many studies have been devoted to improving the substantivity of cotton for reactive dyes, thus diminishing or eliminating the amount of electrolyte required and increasing the efficiency of the dye – fibre reaction[5]. However, none to the date has achieved significant commercial success, since all suffer from one or more disadvantages, such as significantly reduced light fastness, unsatisfactory dye fixation efficiency or poor wet fastness, marked change in hue, limited suitability of the treatments for different kinds of reactive dyes, unpleasant odors released during application, and

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during dyeing[6,7]. The objective of our study is to develop a new technique that minimizes the problems cited above.

In the last few years various patents were reported on coloration of protein fibres using laccase. However, dyeing of cellulose fabrics has not been reported so far, except for a patent application that claims in general the dyeing of the whole range of natural and synthetic fibres without specific examples for cellulosic fibres[8]. The main difficulty consists in the lack of substantivity of the enzymatically generated dyes towards the cellulose substrate. Hence this research attempted to develop a cellulase –mediated dyeing process for cotton fabric.

EXPERIMENTAL MATERIAL

Scoured 100% cotton fabric was used as the substrate throughout. The dyes, chemicals and enzymes used are listed in TABLE 1.

Dyeing procedure

Enzymatic dyeing

The scoured cellulose fabric was given mild bleaching treatment (or) mild oxidizing treatment by exhaust process using, 3% H_2O_2 on weight of the fabric for 90min at 100º C. The fabric was further treated in the presence of formic acid taken 3% on weight of the fabric for 30 min at room temperature. In the same bath, fabric was further treated with chitosan obtained through enzymatic hydrolysis. The resultant treated fabric was then stabilized by the addition of oxalic acid 3% on weight of the fabric for 30min at room temperature and subjected to washing. Finally, cotton fabric was dyed using 2% Reactive (Red MB) cold brand dyes. After dyeing the dyed material was subjected to 2% soap solution & 1% sodium carbonate for 30min at 50-60° C.

TABLE 1 : Chemicals used and function

Chemical Name	Function
Sodium Carbonate	Fixing Agent
TRO	Non ionic wetting agent
H_2O_2	Bleaching Agent
Sodium silicate	Stabilizer
Chitosan (Chitosan E)	Biopolymer
Acetic acid	pH Controlling Agent
Cellulase	Enzyme
Formic acid	Oxidising Agent
Oxalic acid	Reducing Agent
Soap solution	After Treatment
Reactive dye (Red MB) (Cold Brand)	Fabric Dyeing

Enzymatic hydrolysis of biopolymer

Chitosan (2g) was completely dissolved in 100ml of water with 2% acetic acid and the pH was adjusted to 4.7 with a dilute solution of NaOH. The fabric was treated with 2% enzyme (cellulase) and the reaction mixture was kept in a water bath (shaker) at 50° C and 130 rpm. After an hour, the solution was taken out and boiled for 10min to deactivate the enzyme. The hydrolyzed product was filtered to remove precipitates using 10% NaOH.

Conventional dyeing procedure

The scoured cellulose fabric was given bleaching treatment by exhaust process using, 3% H_2O_2 on weight of the fabric for 90min at 100º C and the bleached fabric is worked for 10 min and the temperature is gradually raised to 60-80º C. Then the required quantity of previously dissolved exhausting agent is added in two portions in 20 min and worked for further 20 min. Fixing agent is added in two portions in 20 min and dyeing is continued for further 45 min at 80º C. After dyeing the material is taken out form the dye bath, squeezed well, washed, soaped at boil for 30 min, washed and dried.

Testing methods

The color fastness of the dyed fabrics was evaluated following established test procedures : ISO 105 C06 A2S:1994 - Color fastness to domestic and commercial washing (Grey Scale 1-5); ISO 105 X12:2001 -Color fastness to rubbing (Grey Scale 1-5); ISO 105 E04:1994 - Color fastness to perspiration (Grey Scale 1-5); AATCC-16 (Option 5): 2004 - Color fastness to artificial light: Xenon arc fading lamp test (Blue Scale 1-8); ISO 105 D01:1993 (Solvent Perchloroethylene) - Color fastness to dry cleaning (Grey Scale 1-5); IS 975:88 - Color fastness to sublimation

(Grey Scale 1-5).

The dyed samples were compared with white samples (standard) for the spectral values K/S, ΔL, Δa, Δb and ΔE (CIE 2000) determined using a Minolta 508 spectrophotometer with Macbeth Match View software (X-Rite, USA) in D65 daylight. The color difference (ΔE) was calculated according to Eqn 1:

$$\Delta E = (\Delta L^2 + \Delta a^2 + \Delta b^2)^{1/2} \quad (1)$$

The effluent load was analyzed according to the standards of the examination of water for pollution control and the waste water characteristics were determined. All the test results were compared with the conventionally dyed samples and evaluated.

RESULTS AND DISCUSSION

The cotton fabric was dyed using enzymatic assisted technique with the listed chemicals and procedure mentioned above. The color values of the dyed sample was assessed and compared with conventionally dyed fabric using the electrolyte and alkali. It was observed that the enzymatic dyed sample values are found to be better than the conventionally dyed samples.

Dyeing quality of enzymatic treated cotton

Color Fastness to Washing & Rubbing

Both enzymatic and conventionally dyed samples are subjected to washing fastness and assessed for grey scale rating and the results are listed in TABLE 2. From TABLE 2, it is observed that enzymatic assisted dyed sample showed higher fastness ratings to washing and rubbing than the conventionally dyed samples. This is mainly due to the permanent fixation of dye molecules leading to low surface degradation.

Color fastness to light

Both enzymatic and conventionally dyed samples are subjected to light fastness and assessed for grey scale rating and the results are listed in TABLE 3. It is observed from the TABLE 3 that light fastness of the conventional dyed sample is identical to those of the enzymatic dyed sample under conventional conditions with only a slight drop in light fastness value. The result generally leads to a 1-2 point drop in light fastness ratings of the enzymatic dyed sample. This may be due to the low concentration level of the dye on the fabric leading to slight dullness in the depth of shade.

TABLE 2 : Color fastness to washing & rubbing

Material	Observation	Normal dyeing	Enzymatic dyeing
Cotton Fabric	Change in color	2	3
	Staining on cotton	2	2-3
Cotton Fabric	Dry Rubbing	4-5	4-5
	Wet Rubbing	3	3-4

TABLE 3 : Color fastness to light

Material	Normal Dyeing	Enzymatic Dyeing
Cotton	1-2	1.0

Color fastness to perspiration, dry-cleaning & sublimation

Both enzymatic and conventionally dyed samples were subjected to perspiration, dry cleaning & sublimation fastness and the results are listed in TABLE 4. From TABLE 4, it is observed that that enzymatic dyed sample showed better fastness ratings to perspiration, dry cleaning and sublimation fastness than the conventionally dyed samples. The reason might be due to the improved resistance and bonding nature of the dye molecules between the fibre interstices and avoiding the degradation of color.

Effluent analysis

Textile effluents generally contribute to the pollution load and the determination of waste water characteristics is highly essential to be analyzed. Hence, both the enzymatic dyed and con-

TABLE 4 : Color fastness to perspiration, dry-cleaning & sublimation

Observation	Type	Normal Dyeing	Enzymatic Dyeing
Change in colour	Acid	2	3
	Alkali	2	3-4
Staining on cotton	Acid	2	2-3
	Alkali	2	2-3
Change in colour		2-3	3-4
Staining on cotton		4	3-4
Staining on Cotton		4-5	4-5
Staining on Polyester		4-5	4-5

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TABLE 5 : Effluent Analysis

Effluent characteristics with units	Effluent load values		
	Normal water	Normal dyeing	Enzymatic dyeing
P^H	7.40	10.30	2.30
TDS (ppm)	123	41495	8229
Total Hardness (ppm)	40	5000	3000
TSS (ppm)	8	886	92
COD (ppm)	16	7200	6800
BOD (ppm)	73	6	186
Color (hazan units)	1	26250	23000

ventionally dyed samples are subjected to the above analysis and compared with the standard values and the results are listed in TABLE 5. From TABLE 5, it is inferred that the enzymatic assisted dyeing process had highly reduced the effluent load than the conventional dyeing process. The results of enzymatic assisted dyeing indicates low pollution load except BOD value. Hence, the formulation is found to be more ecofriendly as compared to conventional method. However due to the reduction in water consumption, the oxygen required to decompose the organic matter in a sample of water is less which lead to the increase in the level of BOD.

Color difference & color intensity

Color difference (ΔE) of the samples was analyzed in both the conventionally dyed and enzymatic dyed samples and they were also assessed for K/S values. The enzymatic dyed sample showed better values in color strength & depth of shade than the conventionally dyed sample as shown in TABLE 6. The color intensity of the enzymatic dyed sample is found to be increased resulting in darker shade than the conventional dyed sample.

Effect of dyeing temperature

TABLE 6 : Colour difference values

Material	Method	Normal dyeing	Enzymatic dyeing
Cotton	(ΔE)	0.4	0.8
Cotton	K/S	1.534	1.694

A high level of dye exhaustion on the treated fabric was achieved in the absence of salt and alkali at a temperature as low as that used in the conventional dyeing process, recommended by the dye manufacturer for the particular dye selected (normally at 60 -80º C). Further increases in temperature improved dye bath exhaustion, but only to a limited extent. However, higher temperatures (90 -100º C) are generally recommended for dyeing modified fabrics to obtain better penetration and fixation.

Effect of soaping treatment

The soaping was done to the dyed fabrics with 2% soap solution & 1% sodium carbonate for 30min at 50-60° C after reactive dyeing under acid to neutral conditions. A high level of dye fixation (91%) was achieved without any further discharge in subsequent soaping cycles. This may be explained by the alkaline conditions and high temperature during soaping, which facilitated the formation of Cell-O-. Such conditions further increase the reaction efficiency between cotton and unfixed dyestuff on the substrate and between cotton and the polymer's residual groups, leading to higher level of dye fixation. Hence, the results indicate that the alkaline soaping treatment allows dyeing under more flexible conditions of low temperature and low pH without compromising much on dye fixation levels. From Figure 1, it was observed that the absorption band λ of FTIR spectra is comparatively improved in enzymatic dyed sample compared to the conventional dyed sample. The overall absorbance of the enzymatically dyed cotton fabrics is higher in the region 1000 to 1600 cm-1 and more pronounced between 1400 and 1600 cm-1. This suggests large conjugation of aromatic rings through C-C, C-O-C,-N=C and NH- bonds. However, due to the low concentration of the dye on the fabric and overlapping with characteristic absorbance of cellulose, a well- defined absorption band for the colorant is difficult to detect.

CONCLUSION

100% cotton fabrics were dyed using normal reactive dyeing and enzyme mediated dyeing procedure and the results were compared for fastness, spectral values and effluent load. The overall fast-

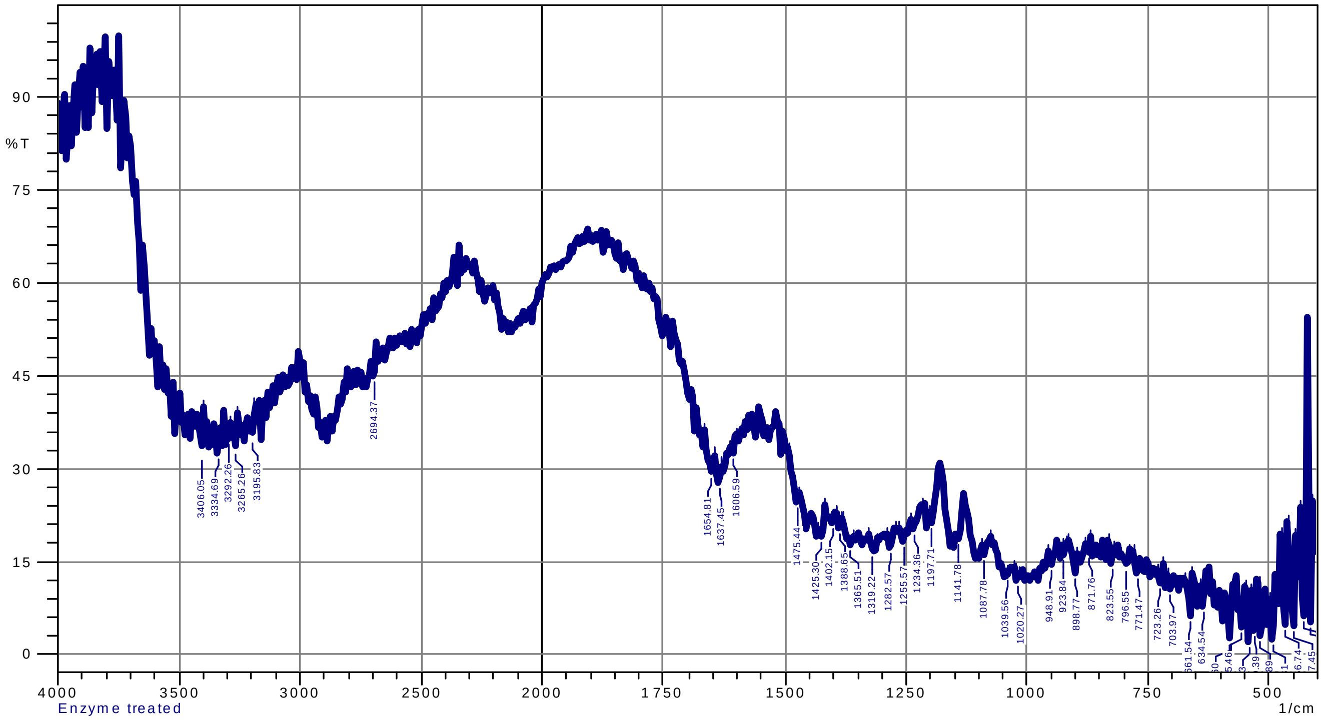

Figure 1 : FTIR Spectra of enzyme treated sample

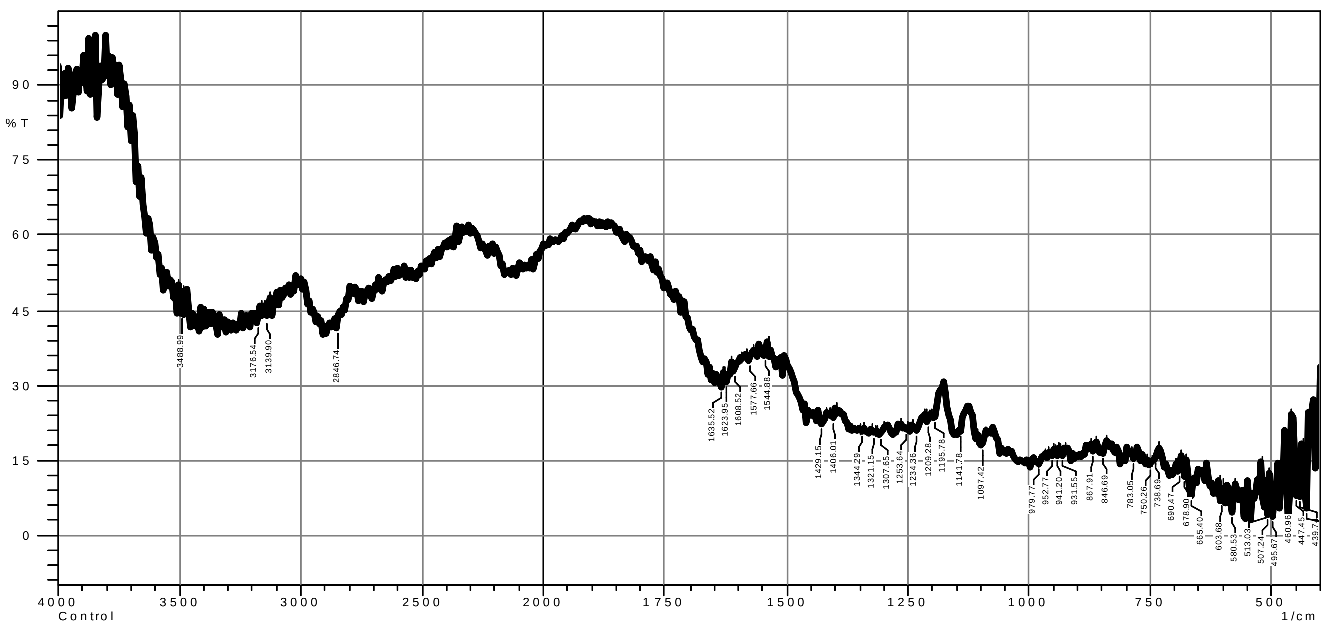

Figure 1 : FTIR Spectra of control sample

ness properties of the enzyme mediated dyed sample was found to be moderate to good except the light fastness which exhibited a marginal drop in the normal value. The effluent load was found to be significantly reduced in the enzyme mediated dyed sample except the BOD level. The spectral values (CIELAB) and the color strength (K/S value) of mediated dyed sample was found to be better than the normal dyed sample. As the usage of enzyme is more eco friendly and effective than using salt for reactive dyeing of cotton fabrics, efforts should be made to find more in the process optimization and improved performance of the results.

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