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Simultaneous biopolishing and reactive dyeing of cotton: A sustainable processing approach

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Abstract
The textile sector is increasingly adopting sustainable and resource-efficient methods, with enzymatic treatments offering eco-friendly alternatives to chemicals. This study investigates the integration of biopolishing with reactive dyeing of cotton in a single bath to reduce water, energy, and process time. Cotton twill fabric was processed using three approaches: Method A (dyeing followed by biopolishing), Method B (biopolishing followed by dyeing), and Method C (simultaneous dyeing and biopolishing) using neutral and acid cellulase enzymes. Treated samples were evaluated for colour yield (K/S values), colour difference (dE), bending length, and crock fastness. Method B showed the highest colour yield, likely due to improved dye binding sites created by enzymatic pretreatment. Simultaneous processing (Method C) achieved good softness and fastness, especially with neutral cellulase, though colour yield was slightly lower. Acid cellulase in Method C resulted in weaker dye uptake. Overall, simultaneous processing offers an efficient and sustainable alternative without compromising fabric quality.

Keywords: Acid cellulase, neutral cellulase, reactive dyes, colour yield, softness, crock fastness

1. Introduction

The textile industry continually seeks innovative methods to enhance sustainability and efficiency in its processing chains [1]. One promising avenue involves the integration of enzymatic treatments, particularly biopolishing, with conventional dyeing processes [2]. This approach aims to streamline operations, reduce resource consumption, and mitigate the environmental impact commonly associated with traditional textile wet processing [3]. Specifically, the simultaneous biopolishing and reactive dyeing of cotton fabric represents a significant advancement, offering a single-bath process that combines the enzymatic modification of cellulose fibers with the application of reactive dyes [4]. This integrated method leverages the benefits of cellulase enzymes for surface modification, leading to improved fabric aesthetics and performance characteristics, while simultaneously achieving vibrant and durable coloration through reactive dye chemistry [5]. The primary objective of biopolishing is to remove protruding surface fibers, often referred to as fuzz, thereby enhancing fabric smoothness, reducing pilling tendency, and improving luster [6]. Enzymes are biological catalysts that accelerate the rate of chemical reaction without themselves undergoing any permanent chemical change [7, 8]. Enzymes have been found as an efficient and cheap alternative to many chemicals & auxiliaries for textile applications. They work at lower temperatures and are less hazardous to the environment & therefore easy to dispose of after use. This treatment can be applied to knit and woven cellulosic fabrics such as cotton, viscose, linen and their blends. The elimination of superficial micro fibrils of the cotton fibre through the action of cellulase enzymes is obtained by the controlled hydrolysis of cellulose leaving the surface of the fibres free and conferring a more even look [9].

Concurrently, reactive dyeing forms covalent bonds with cellulosic fibers, ensuring excellent colorfastness and brilliance [10, 11]. The improvement in fabric softness and smoothness are permanent in contrast to the softeners applied to the fibre surface. Many researchers well established the effect of the cellulase enzymes on varies cellulosic material [12-15]. Cellulase comprise a multi-component enzyme system, includingendoglucalases (EG's) that hydrolyse cellulose chains randomly, cellobiohydrolases (CBHS) that splits cellobiose from cellulose ends, and cellobiases that hydrolysecellobiose to glucose [16, 17].

The cotton fabric may be dyed by four classes of dyes - direct, reactive, vat and sulphur. The dyeing mechanism is different and so is the mechanism of bonding with cellulose for the different class of dyes [18]. Cellulase treatment on the direct dyed fabric was studied and found that cellulase-catalysed hydrolysis would occur mainly in the amorphous region at smaller weight loss and tend to decrease with increasing dye uptake in every case of dye [19]. A role of reactive dyes on the action of cellulase on cellulosic fibres was studied and found that bifunctional dyes leads to less loss in weight as compared to monofunctional dyes as the formation of the cross-linkage between two cellulose molecules via bridging with the dye. The reactive dyes with higher substantivity and with more bifunctional property were assumed to retard the hydrolysis of the dyed fibre to a greater extent compared to the dyes of lower substantivity and less bifunctional property [20-22].

A study on the simultaneous processing of dyeing and enzymatic process on wool fabric was carried out with success [23]. In conventional method of cotton biopolishing, a dyed fabric is biopolished with cellulase enzymes to get a desired effect on the fabric [24]. This requires two different process operations i.e. dyeing followed by biopolishing which leads to high consumption of water and energy. To overcome these problems, we tried to explore the simultaneously dyeing and bio-polishing of a fabric in a single bath and also to compare the results with a conventional method of bio-polishing. Thereafter, we also studied the effects of bio-polishing on RFD fabric followed by dyeing.

2. Experimental

2.1 Material: The substrate used in the study was 100% cotton twill weave fabric (Weave - 3/1 twill; ends per inch 72 & picks per inch 52). Both acid and neutral cellulase used for the study was provided by Dystar India Pvt. Ltd. The chemicals used were buffer (sodium acetate/acetic acid), Sodium Sulphate, sodium carbonate (provided by Merck Specialties Pvt. Ltd.) and soaping agent (provided by Dystar India Pvt. Ltd.). The dye used for dyeing was Remazol Ultra Red RGB (provided by Dystar India Pvt. Ltd.). The experiment was carried out in Infra colour IR dyeing machine. Agitation required for effective bio-polishing was given by using steel balls. The ratio steel balls/fabric was taken as 20:1.

2.2 Process Method

Biopolishing and dyeing of fabric was done by three different methods which are discussed in the upcoming sections.

2.2.1 Method A (Conventional Method)

RFD fabric was first dyed with reactive dyes followed by bio-polishing with both classes of enzymes. The method and recipe used for bio-polishing and dyeing was as follows:

2.2.1.1 Dyeing method

Cotton fabric was dyed by exhaust method using Remazol Ultra Red RGB dye having 1% shade with concentration of salt as 40 g/l for exhaustion and soda ash as 10 g/l for fixation of reactive dyes at temperature of 60°c with dyeing cycle as shown in Fig 1 [16].

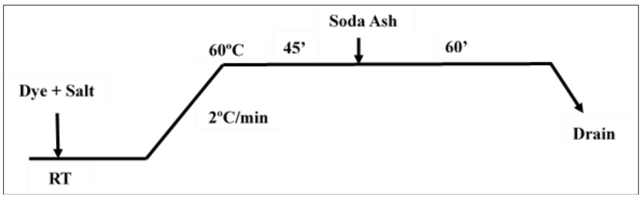

Fig 1: Dyeing cycle of cotton fabric dyed with Remazol Ultra Red RGB

2.2.1.2 Biopolishing method

Dyed fabrics were treated with acid and neutral cellulase separately with the process conditions as mentioned in table 1. A mechanical agitation was provided by the steel balls and rotation of the beaker. The pH of the bath was maintained with the help of buffer according to the cellulase used i.e. pH of 4.5-5.5 and 6-7 for acid and neutral cellulase respectively. After 45 mins of dwell time, the temperature of the bath was increased to 90°c for 10 mins to inactivate the cellulase enzyme. After drying of the fabric, K/S, bending length and rubbing fastness were evaluated.

Table 1: Process conditions for conventional method of biopolishing

	Neutral Cellulase	Acid Cellulase
Enzyme	3% o.w.f	3% owf
pH	6-7	4.5-5.5
Time	45 min	45 min
Temperature	55ºC	55ºC

2.2.2 Method B

RFD fabric was first bio-polished with both classes of enzymes followed by dyeing with reactive dyes. The recipe and conditions used for this method were same as mentioned in method A.

2.2.3 Method C (Simultaneous method)

Dyeing and Bio-polishing of the RFD fabric was done simultaneously. The recipes and process cycle for the same were shown table 2 and fig 2 & 3 respectively:

Table 2: Recipes for simultaneously biopolishing and dyeing of cotton with reactive dyes

	Neutral Cellulase	Acid Cellulase
For exhaustion of dyes and Biopolishing process		
Enzyme	3% owf	3% owf
Dye shade	1%	1%
pH	6-7	4.5-5.5
Salt	40 gpl	40 gpl
For inactivation of cellulase and fixation of dye		
Soda Ash	10 gpl	10 gpl

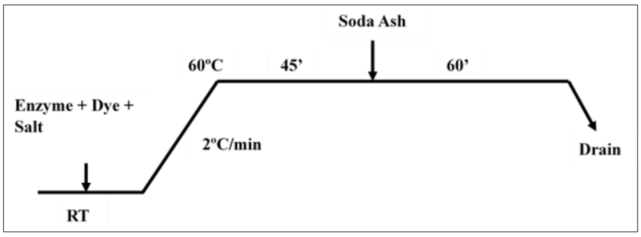

Fig 2: Process cycle for simultaneously biopolishing and dyeing of cotton with neutral cellulase

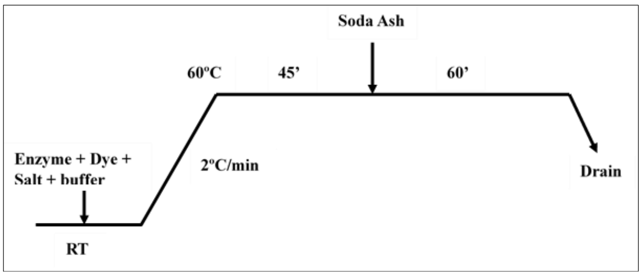

Fig 3: Process cycle for simultaneously biopolishing and dyeing of cotton with Acid cellulase

Evaluation of samples

- Colour yield and colour difference was determined by measurement of K/S values and dE (on Datacolor SF 600) at the wavelength of maximum absorption under D65 illuminant.
- Bending length of the treated samples was done as per ASTM (E290-09).
- Crock Fastness of the treated samples was done as per AATCC 116

Results and Discussion

Colour yield (K/S values)

From the results of K/S as shown in table 3, for both neutral and acid cellulase enzymes, it was observed that the colour yield for the sample treated by method B was higher as compared to method A and method C. It was also found that neutral cellulase gives comparable and better colour yield as compared to acid cellulase in case of method A and C.

Table 3: K/S values of sample treated with 3 different methods

Type of Enzyme	Method A	Method B	Method C
Acid Cellulase	6.5	8.0	6.3
Neutral Cellulase	7.3	8.0	7.2

The K/S value of the dyed unbiopolished sample was calculated and found to be 7.8 and while comparing K/S values with biopolished treated samples, it was observed that method A i.e. conventional and method C i.e. simultaneous, gives lower colour yield than unbiopolished sample. Lower colour yield in case of method A attributed to loss of the reactive dye during biopolishing process. The colour yield in case of method B is higher than unbiopolished sample may be attributed to alternation in dye binding sites on fibre surface and amorphous region by the action of cellulase pre-treatment.

The colour yield for neutral cellulase was found higher as compared to acid cellulase by using method C. This may be attributed to the fact that pH used in neutral enzyme didn't interfere with exhaustion of the reactive dyes whereas the acidic pH of acid cellulase bath i.e. pH 4-5 leads to decrease in the overall exhaustion which may be attributed to the slightly decrease in the dye solubility resulted in lower colour yield on the fabric i.e. 7.2 and 6.3 respectively.

Colour Difference (dE)

dE of the dyed samples was assessed against unbiopolished sample and minor changes in colour tone were observed in all samples as shown in table 4. In conventional method, colour difference with both enzymes was found higher as compared to standard and was under acceptable limit i.e.≤0.5. Maximum colour difference was found in method C with acid cellulase. This may be attributed due to low colour yield on the sample.

Table 4: dE of the sample treated with 3 different methods

Type of Enzyme	Method A	Method B	Method C
Acid Cellulase	0.44	0.18	0.57
Neutral Cellulase	0.34	0.13	0.17

Bending Length

Bending length determines the softness of the fabric and from the results mentioned in table 5, it was found that bending length of all treated samples decreased as compared to the standard unbiopolished sample, thus indicating successful bio-polishing for all samples. Although the results for all samples were comparable but method C with neutral cellulase provides best softness with least bending length of 2.8 cm against unboipolished sample i.e. 3.8 cm.

Table 5: Bending length of the sample treated with 3 different methods

Type of Enzyme	Method 1	Method 2	Method 3
Acid Cellulase	2.8	3.2	2.9
Neutral Cellulase	3.0	3.1	2.8

Crock Fastness

During biopolishing process, the protruding fibres got removed from the surface of the treated fabric and this may leads to increase in rubbing fastness of the fabric. To understand the effect of biopolishing treatment on the rubbing fastness of the dyed fabric, rubbing fastness was evaluated and results were shown in table 6. The crock fastness of the unbiopolished sample was evaluated in terms of dry and wet crock fastness and rated as 4/5 & 3 respectively. Overall fastness in terms of dry and wet was found comparable with unbiopolished and all 3 treatment methods.

A marginal gain in wet fastness rating was observed in case of method A treated with acid cellulase having value 4 against achieved 3/4 for methods B & C respectively. The wet fastness of neutral cellulase gives higher values than unbiopolished standard sample and was comparable to conventional sample. This may be attributed to the fact that neutral enzymes removed the protruded fibres from the surface more efficiently in all three treatment methods.

Table 6: Crock fastness of the sample treated with 3 different methods

Type of Enzyme	Method 1		Method 2		Method 3	
	Dry	Wet	Dry	Wet	Dry	Wet
Acid Cellulase	4/5	4	4/5	3/4	4/5	3/4
Neutral Cellulase	4/5	4	4/5	3/4	4/5	4

Conclusion

To reiterate, this study demonstrates that integrating biopolishing with reactive dyeing of cotton fabric can effectively streamline textile wet processing. Among the three approaches, biopolishing prior to dyeing (Method B) yielded the highest colour strength, while the simultaneous process (Method C) offered significant savings in time, water, and energy without compromising softness or fastness properties. Neutral cellulase performed better than acid cellulase, particularly under simultaneous conditions, due to favourable pH compatibility with reactive dyes. Overall, the combined approach highlights a sustainable, resource-efficient alternative to conventional methods, supporting cleaner production practices in the textile industry.

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