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INNOVATIVE AND ECOFRIENDLY METHODS OF COTTON DYEING

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Abstract: Colouration of textiles is an important aspect of the production and adds value to it. The specific requirement of coloured shade / hue and its fastness properties is a major quality parameter demanded by the end users. Organic dyes and pigments, whether natural or synthetic, are very intense in colour. Out of various classes of dyestuffs used for cellulosic colouration, reactive, vat, indigo, direct, sulphur, naphthol occupy the major share. Sulphur dyes are mainly used for dyeing cotton to medium or heavy shades, primarily because they provide excellent to good light and wash fastness at low cost. In present research it was found that that few enzymes viz. lipase, pectinase, protease and catalase can effectively reduce all sulphur dyes while other enzymes studied could not. Dye bath potential, dye yield (K/S), reduction bath stability and colour fastness of dyeing showed almost comparable results in both the reducing systems, thus opening scope for enzymatic dyeing of cotton with sulphur dyes.

Keywords: Sulphur dye, sodium sulphide, reduction potential, enzymatic dyeing, dye strength, colour fastness



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INTRODUCTION

Sulphur dyes are water insoluble after oxidation and therefore they are largely eliminated by adsorption on the activated sludge in waste water treatment plants. Colour removal due to sorption by bio mass is referred as bio-elimination. With the increasing demand of textile manufactures to reduce pollution in textile coloration and finishing, the use of enzymes in the chemical processing of fibres and textiles is rapidly gaining wide recognition because of their non-toxic and eco-friendly characteristics. The exact share of textiles is estimated to be 10% of industrial enzymes. Enzymes catalyse chemical reactions under optimum reaction conditions like acid base, catalysis, covalent bonding or electron transfer mechanism. Textile processing with enzymes is a new emerging and multidisciplinary area. Various enzymes viz. protease, lipase, pectinase, catalase, amylase, laccase, cellulase have been used in the present work to catalyse reduction of sulphur dyes to substitute sodium sulphide, a toxic and polluting chemical. Keeping in mind the different requirement of sulphur dyes in dyeing of cotton viz. high alkalinity (11.5-12 pH), high (90°C) temperature reduction potential (-400 mV approximately) etc., alkaline, thermo stable and nucleophilic catalysis capable or reduction-oxidation catalysis capable enzymes were chosen for enzymatic catalytic reduction and dyeing of sulphur dyes. Protease, pectinase and lipase carry out nucleophilic attack on the substrate with structurally analogous Asp-His-Ser triads. Whereas a catalase enzyme completes its catalytic cycle in two two-electron reduction and oxidation. Various enzymes had been used in the present work to catalyse reduction of sulphur dyes to substitute sodium sulphide.

In present work, new methods of reduction and dyeing of sulphur dyes on cotton by using enzymes. Obtained results suggested ability of enzymes under alkaline conditions to cause effective reduction and solubilisation of sulphur dyes.

2. Comparative performances of various enzymes on sulphur dyes

2.1 Reduction potential and pH

Reduction potential and pH of all reduction baths prepared with optimized sodium sulphide concentration (2T, two times wrt dye) as well as alkaline enzyme formulation, i.e. protease (0.25T), pectinase (0.5T), lipase (0.5T) and catalase (0.5T) at different stages of dyeing with Green 1 are detailed in table 1.

Table 1 Dye bath status in sulphide and enzymatic systems for Green 1 after optimizing all dyeing parameters

Reducing systems	Reduction potential (mV) and pH at various stages					
	Before reduction		After reduction		At the end	
	of dye		of dye		of dyeing	
	pH	mV	pH	mV	pH	mV
Sulphide	11.4	-556	11.0	-535	11.0	-518
Protease	12.0	-549	11.7	-527	11.5	-517
Pectinase	12.0	-525	11.7	-521	11.6	-512
Lipase	12.0	-519	11.7	-517	11.6	-507
Catalase	12.0	-539	11.7	-527	11.6	-517

It was found that the optimized sulphide as well as alkaline enzyme formulation developed adequate reduction potential in bath, sufficient for complete reduction of sulphur dyes. The reduction potential remained quite high even at the end of dyeing confirming retaining the reduced status throughout dyeing process. Though there was some difference in reduction potential developed by sulphide and different alkaline enzymes that was not substantially different. For other sulphur dyes, sodium sulphide and alkaline enzyme systems followed the same pattern. However, alkaline amylase, cellulase and laccase could not develop required reduction potential in bath.

2.2 Comparative dye strength

Comparative dye strength of cotton dyed with ten different sulphur dyes was analyzed. The hue of all the dyed cotton samples in enzymatic system showed shifting of the hues towards some other direction, different for different dyes under study, as could be seen in table 2 for various dyes.

Table 2 Dye strength with sulphide and alkaline enzymes on Brown 8

Reducing system	K/S	% CS	L	a	b	C	h	δd	Remark
Sodium Sulphide	10.7	100	32.4	7.3	10.8	13	56	-	-
Protease	9.8	92	37.9	9.5	11.6	13	51	5.9	More red & yellow
Pectinase	7.4	69	37.4	10.5	11.2	15.3	47.0	5.6	-do-
Lipase	6.2	60	38.5	11.6	10.6	15.7	42.4	7.1	-do-
Catalase	8.4	79	36	11.6	10.6	15.7	42.3	5.6	-do-

3. Stability of reduction baths

3.1 In absence of dye

Reduction baths were stored for different times in absence of dye. After stipulated time, dye was added to these baths. After the dye was reduced, cotton was dyed.

3.2 In presence of dye

Dye was added to the reduction baths at 90 °C and was stored for varying times. After stipulated time, the baths were used for dyeing of cotton. The dye strength values are being shown in table 13. It was observed that, like stability of baths in absence of dye, dye strength of cotton was maximum after dyeing in baths stored for two hours in sulphide and catalase baths while that was maximum while dyed in baths stored for one hour in protease, pectinase and lipase systems.

4. Colour fastness of dyed cotton

4.1 Light fastness

Light fastness of dyed cotton in all the five dyeing systems resulted in very good to excellent light fastness grades though there was nominal difference in a very few cases as shown in table 3.

Table 3 Light fastness grades in sulphide and alkaline enzymatic systems

Dye	Light fastness grades				
	Sulphide	Protease	Pectinase	Lipase	Catalase
Black 1	6	6	6	6	6
Blue 4	5	5	5	5	5
Green 1	6	6	6	6	6
Red 10	6	6	6	5	6
Yellow 9	5	5	5	6	5

5. Mechanism of cotton dyeing using enzymes

In the proposed mechanism of action of enzymes, chymotrypsin, serine and histidine carry out nucleophilic attack on substrate.

In the first step, the electronegative oxygen atom of serine carries out a nucleophilic attack on the the substrate, cleaving the disulphide bonds so as to bring the imidazole ring of the histidine residue nearer to serine. This is accompanied by the removal of a proton from serine, which is donated to the nitrogen atom of the cleaved sulphur dye and the enzyme than covalently binds with the substrate.

In the second step, the electro-negative oxygen atom of a water molecule displaces the acyl group from the enzyme, making the enzyme free. Histidines also play a role in proton transfer and donate to the serine residues.

The lipase catalytic triad is composed of serine, aspartate or glutamate and histidine. Hydrolysis of the substrate follows a two step mechanism. The nucleophilicity of the active serine is enhanced by transferring a proton to the catalytic histidine with formation of an oxyanion that attacks the carbonyl carbon of the susceptible ester bond. A tetrahedral intermediate is formed carrying a negative charge on the carbonyl oxygen atom of scissile bond and it is stabilized through hydrogen bonding to main-chain N-H groups. Such residues build up the so-called oxyanion hole that in some lipases is performed in the correct orientation, whereas in others it is positioned upon the opening of the lid structure. The proton on the histidine is then transferred to the ester oxygen of the bond that is cleaved and a covalent intermediate forms with fatty acid from the substrate esterified to serine. The second step of the reaction is deacylation of the enzyme through a water molecule that hydrolyses the covalent intermediate. In addition the negatively charged tetrahedral intermediate is stabilized by hydrogen bonds to the oxyanion hole. Finally, histidine donates an oxygen atom of an active serine and the acyl component is released. Many esterases are multifunctional enzymes and they can work as carboxyesterases, lipases and others. In the hydrolysis reaction, the catalytic triad Ser-His-Asp can be involved in the same way as protease. The mechanism for ester hydrolysis or formation is essentially the same for lipases and is composed of four steps: first, the substrate is bound to the active serine, yielding a tetrahedral intermediate stabilized by the catalytic His and Asp residues. Next, the alcohol is released and an acyl-enzyme complex is formed. Attack of a nucleophile (water in hydrolysis, alcohol or ester in transesterification) reforms a tetrahedral intermediate, which after resolution yields the product (an acid or an ester) and free enzyme. Lipases observe the phenomenon of interfacial activation, lipase needs a minimum substrate concentration before high activity is observed. Structure elucidation revealed that this interfacial activation is due to a hydrophobic domain (lid) covering the lipase active site and only in the presence of a minimum substrate concentration, (a triglyceride phase or a hydrophobic organic solvent) will the lid open, making the active site accessible. Furthermore, lipases prefer water-insoluble substrates, typically triglycerides composed of long-chain fatty acids, lipase and esterases were among the first enzymes tested and found to be stable and active in organic solvents, but this characteristic is more apparent with lipases. 3-D structure of enzyme showed that the active site of lipases displays a negative potential in the pH-range (typically 8 pH).

Pectin esterases commonly employ a Ser-His-Asp catalytic triad such as acetylxylin esterase to catalyze deacetylation. Aspartate is buried in the interior part of the active site so as to

facilitate action of Histidine and Serine. This setup is believed to act as a charge relay system in which Asp removes a proton from His that makes easier for it to remove proton from Ser during later part of the reaction, known as catalytic triade. In the case of nucleophilic catalysis, catalysts are more nucleophilic, compared to normal attacking group and so rapidly form an intermediate, that itself rapidly gets broken down to give the product. Other mechanisms such as a Zn^{++} catalyzed deacetylation may also be considered for some families. A catalase enzyme completes its catalytic cycle in two two-electron reduction and oxidation. It develops sufficient reduction potential during its catalytic cycle of reduction.

6. CONCLUSIONS

Alkaline enzymes viz. protease, pectinase, lipase and catalase were found to be effective in dyeing of cotton alkaline condition with no such substantial difference in dye uptake; though tonal changes may be due to their differences in structure, mechanism favorable catalytic conditions and dye itself. Colour fastness grades remained on higher side for all the three fastness criteria studied, they are also found to be satisfactory. Hence enzymatic methods of sulphur dyeing could be considered to substitute the sulphide method as an environmental friendly process. It was concluded from the study that cotton could be successfully dyed with sulphur dye using these enzymes.

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