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Research Article

Removal of Residual Hydrogen Peroxide in Cotton Bleaching using Catalase Enzyme

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Abstract

Industrial use of biotechnology is bringing about new products and processes aimed at the use of renewable resources, as well as the application green technologies with low energy consumption and environmentally healthy practices. Textile processing is a growing industry that traditionally has used a lot of water, energy and harsh chemicals. Due to the ever-growing costs for water and energy worldwide investigations are carried out to substitute conventional chemical textile processes by environment-friendly and economically attractive bioprocesses using enzymes. In the present study, Catalase enzyme is used to investigate the efficacy of removal of residual hydrogen peroxide after bleaching. An alternative cleanup option for hydrogen peroxide based bleaching by this option instead of chemical reducing agents found to be useful in saving water in successive washing, energy, cost and processing time beside significant improvement in dyeing of cotton fabrics with a reactive dye subsequently.

Keywords: Catalase enzyme; Hydrogen peroxide; Cotton bleaching; Textile processes.

Introduction

In the last decade biotechnological processes are gradually invading traditional industries like the textile industry. In some cases they are substituting traditional processes, in other cases biotechnological products are used to produce new product properties. The principal advantages are associated to less severe reaction conditions, lower processing time and temperatures and non-toxic and biodegradable products. Due to their nature, enzymes increase selectively and significantly the reaction rate of a series of specific reactions [1-3].

In today's textile industry the removal of starch sizes from cotton fabrics with amylases is a standard procedure and cellulases are indispensable for the finishing of fabrics made from cellulosic fibers like stone washing of denim garments or biopolishing. The application of pectinases for biobleaching of cotton and catalase for hydrogen peroxide removal is still under investigation [4,5]. Enzymes can work at atmospheric pressure and in mild conditions with respect to temperature and acidity (pH). Most enzymes function optimally at a temperature of 30ºC-70ºC and at pH values, which are near the neutral point (pH 7). Enzyme processes are potentially energy saving and save investing in special equipment resistant to heat, pressure or corrosion. The various types of enzymes used in textile wet processing are presented in Table 1.

The bleaching with hydrogen peroxide is a fundamental stage that precedes dyeing of cotton fabrics. Hydrogen peroxide as an oxidizing agent leads to degradation of reactive dyes and may increase dye hydrolysis so that remaining peroxide residues on the fabric have a negative influence on the result of dyeing. Thus residues from bleaching baths must be removed from fabric and machinery before adding the dye [6].

Usually successive washes of the fabric with water or chemical reducers diminish the residual bleaching agent concentration and improve dyeing. However, this involves the use of a big volume of water, a longer process time or the use of chemical products noxious to the environment, resulting in additional costs and the generation of more effluents. An alternative to decrease the consumption of water, energy and

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time is the use of catalase, which catalyze the decomposition of hydrogen peroxide. And thus the elimination of its residues on cotton fabrics originated from the bleaching process [7].

Table 1. Function of enzyme in Textile Processing

Type	Action
Amylase	To decompose starch applied in sizing
Catalase	Act on H_2O_2 to decompose it into water and oxygen
Protease, lipase and pectinase	When combined ,act on proteins, pectins and natural waxes to effect scouring
Laccase	Decomposes indigo molecules for washing down effect on denim
Cellulose	Break down cellulosic chains to remove protruding fibes by degrading &create wash-down effect by surface etching on denims
Lipase	Elimination of natural triglycerides(in scouring) or present in desizing (tallow compounds)
Pectinase	Breaks down pectin in scouring of cotton

In textile industry, peroxide bleaching is used as it is less toxic than the chlorine bleaching. if any trace of bleach chemicals is left on yarn or fabric, it leads to uneven dyeing. In the normal process, after bleaching the traditional reducing agents like sodium bisulphate are used to neutralize the residual hydrogen peroxide. In the latest and advanced method of bleach clean-up specifically developed for removal of residual hydrogen peroxide. Enzymes are more convenient alternative because they are easier and quicker to use. With the increasingly important requirement for textile manufacturers to reduce pollution during production, the use of enzymes is rapidly gaining wider recognition because of their non-toxic and eco-friendly characteristics [8].

Enzymes provide a more convenient alternative process than conventional because they are easier and quicker to use. A small dose of Catalase is capable of decomposing hydrogen peroxide to water and oxygen. The enzyme can be applied directly after the bleaching stage. But conventional removal of residual hydrogen per oxide results high levels of energy and water consumption, longer processing time, release of toxic substance such as sodium hydrosulphite and it's by products, toxic and hazardous chemical, lower bleaching clean up efficiency [9]. Most of the reducing agents require high temperature in order to react efficiently with hydrogen peroxide and thus increase the energy consumption. An attractive alternative to neutralize the residual bleach chemical is catalase enzyme. It removes hydrogen peroxide without rinsing. It decomposes the hydrogen peroxide as follows eq. (1).

$$2H_2O_2 \xrightarrow{\text{Catalase}} 2H_2O+O_2 \quad (1)$$

The catalase actes in cold water and thus saves valubel energy.noremally, 30-40 liter of water per kg of fabric is required for rinsing to remove the excess of reducing agent.after catalase treatement rinsing is not at all required,thereby ahuge quantity of water is saved hyderogen peroxside is broken down to water and oxygen neither of which adds to pollution load.there are no sulphates or nitrogen saltes in the waste water which otherwise are present while using reducing agent.thuse,the use of catalase not only saves water,time and energy but also reduces the pollution load considerably [10].

Catalase has one of the highest turnover numbers of all enzymes; one catalase molecule can convert millions of molecules of hydrogen peroxide to water and oxygen each second. The residual peroxide present in the fiber will oxidize the reactive dyes' chromophore that contain the metal ions and lead to tonal variation and sometimes uneven dyeing. In some cases the chrmophore itself is getting destroyed into a colorless product and lead to pale or white patchy dyeing. Hence the removal residual alkali and peroxide are very much essential before starting a good dyeing operation [11-19].

Materials and Methods

Materials

Plain woven 100% cotton grey fabric (28 ends/cm, 18 picks/cm) was used in the study. The various chemicals used are Hydrogen peroxide (30%), sodium hydroxide, wetting agent, sequestering agent, sodium silicate, Sodium persulphate, For removal of residual hydrogen peroxide: Catalase (Invazyme cat), sodium dithionite, successive washing; For dyeing process: Bezactive Blue V-RN, Bezaktive Red V-RB, Bezaktive Yellow V- GR, sodium chloride (NaCl), sodium carbonate (Na_2CO_3), acetic acid. The equipments used for the work are pH meter, peroxide test strip (Quantofix Peroxide 100), Thermometer, Eco-infrared dyeing machine, dryer, Gretag Macbeth color-eye 3100 Spectrophotometer, crock meter, Lauder-O-meter, Grey scale as per ISO-05-A02 and ISO-105-AO3, Microsoft excel 2007 software for analyzing the data obtained from the tesing of dyed samples with in different treatements.

Methods

Combined pretreatment

The pretreatment was carried out using the recipe given in Table 2. After combined pretreatment, for removal of residual peroxide, three different treatments such as catalase enzyme, sodium dithionite, washing four times (2 cold and 2 hot washings) were used.

Table 2. Recipe for combined pretreatment

Parameter	value
Material to Liquor Ratio	1:20
Time (min)	45
Temperature (°C)	95
Caustic soda (gpl)	2.5
Hydrogen per oxide (gpl)	2.5
Sodium persulphate (gpl)	0.5
Wetting agent (gpl)	0.5
Sequestering agent (gpl)	0.5
Sodium silicate (gpl)	0.5

Methods of Removal of Residual H_2O_2

Using catalase: Hot wash 80° for 10 min with material to liquor ratio (MLR) 1:20 → Fill with 0.5 g/l acetic acid neutralize alkali → Check that pH in the range 6.7-7 and the temperature 45°C → Add 0.5 g/l catalase BF 35, 40 and 45°C for 10 min with MLR 1:20 → After 10 minutes, check removal of residual hydrogen peroxide by using a Peroxide test strip.

Using sodium dithionite: Fill with cold water → Hot wash 80°C for 10 min with MLR 1:20 → Add 0.5 g/l sodium dithionite fresh cold water → Fill with 0.5 g/l acetic acid, neutralize with alkali → Hot wash 80°C for 10 min → check that pH in the range 6.7-7 → After 10 minutes, check removal of residual hydrogen peroxide by using a Peroxide test strip.

Rinsing with water: Fill with cold water : Hot wash 80°C for 10 min → Add 0.5 g/l acetic acid neutralize alkali → Hot wash 80°C for 10 min → Fill fresh cold water → Check that PH in the range 6.7-7→ After 10 min, check removal of residual hydrogen peroxide by using a peroxide test strip

Peroxide test strip Evaluation: Break down of residual hydrogen peroxide into non-active oxygen and water is most efficient at pH 6.7-7 beyond this range large amount of the enzyme are needed to achieve complete decomposition of hydrogen peroxide.

According to the Figure 1, the Peroxide Test detects inorganic and organic compounds containing peroxide or a hydroperoxide group. It is well suited to the routine checking of simples that the Peroxide Test strip gives adequate results.

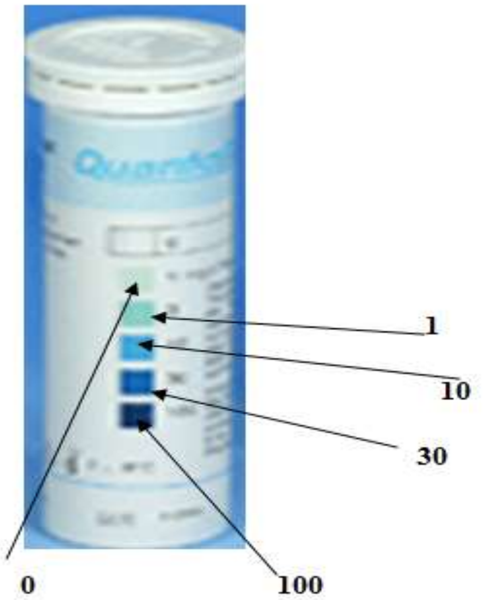


Figure 1 Measurements in Peroxide test stripe

The conventional and enzyme based bleach cleanup methods followed by dyeing was carried out as given below.

1. Rinsing with water : Bleach → Rinse → Rinse → Rinse → Rinse → Dye
2. Reduction with chemical reducing agents: Bleach → Reduce→ Rinse → Dye.
3. Using catalase enzyme: Bleach → Rinse → Catalase → Dye

In the method of catalase enzyme clean up from the experiment, complete breakdown of residual hydrogen peroxide takes place in 10-20 minute and has no adverse effect on either the fabrics or the subsequent dyeing process. Once the treatment is completed, dyeing can be carried out in the same liquor, without any need for further heating.

Dyeing of Treated Samples

Dyeing samples treated using catalase enzyme, sodium dithionate and water washes in eco-infrared dyeing machine was carried out using a recipe given in Table 3.

Table 3. Dyeing Recipe for reactive dyeing

Dye / Chemicals & conditions	Bezactive Blue V-RN	Bezactive Red V-RB	Bezactive Yellow V-GR
Shade	1%, 1.5%	1%, 1.5%	1%, 1.5%
NaCl (gpl)	50	50	50
Na_2CO_3 (gpl)	15	15	15
MLR	1:20	1:20	1:20
Temp. (°C)	80	80	80
Time (Min.)	60	60	60

Dyeing procedure for reactive dye

Take samples after three different treatment method of removal of residual H_2O_2 → Prepare a dye solution → Then immerse the sample in the dye bath and run at 60°C for 15 min. and add half of pre-dissolved salt → Continue further for 10 min. with stirring at temperature of 80°C and added the reminder of the salt → Continued dyeing for 10 minutes at 80°C and take 1.5ml solution for checking up of concentration of solution and add the soda ash solution → Dyeing for 10 min. at 80 - 85°C and take 1.5ml solution from the bath for checking concentration of the soda ash solution → Then wash the sample by cold water and neutralize by 2g/l acetic acid and soaping with 5g/l detergent at 95°C for 10 min. then dry.

Color strength

Color strength (K/S) were determined at maximum dye absorption wave length using Kubelka-Munk equation (2).

$$\frac{K}{S} = \frac{(1-R)^2}{2R} \qquad (2)$$

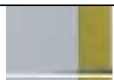
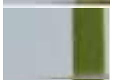
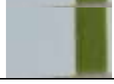
where, K is the light absorption coefficient, S is the light scattering coefficient while R is the D65/10 light reflection. K/S values were calculated based on reflection values of untreated and treated cotton fabrics. This value symbolizes the reduction ratio of light owing to absorption and scattering achieved based on reflectance. Using color eye 3100 reflectance spectrophotometer with light source D-65 and 10° observer the color strength (K/S) was measured.

Results and Discussion

Peroxide Test Stripe Measurement

This test stripe is ideal to test level of peroxide in solution. Peroxide test stripe change color with reaction type from white to blue in gradation of 0 - 1 - 3 - 10 - 30 - 100. This easy dip-and-read procedure provides reliable results within 15 sec.

Table 4. Peroxide test stripe detection

S. No.	Sample result	Catalase Conc. (gpl)	Temp. (°C)	Time (Min.)	Catalase test result
1		0.6	45	10	0
2		0.5	45	10	0
3		0.5	40	10	1
4		0.4	35	10	1

The bleached samples were treated using catalase enzyeme in order to eliminate resdual hyderogen peroxide. Four trials were taken by varying the concentration of catalase enzyeme, temprature and by keeping the other parameteres constant. The concentration used were 0.4g/l, 0.5g/l and 0.6g/l and temprature 35°C, 40°C & 45°C, and time of 10 min. In this experiment, the effect on the result of catalase treated samples was investigated. It can be seen from Table 4 that the treatment with catalase of 0.5g/l concentration and 45°C tempreature was effective compared to the other levels of concentrations.

From Table 5 where the test result average value are presented, one can understand that there was no significant difference in treated samples between catalase, sodium dithionite and successive washing

Colour Strength (K/S Value)

Color Strength of Bezactive Blue V-RN, Red V-RB and Yellow V-GR Dyed Fabric

The color strength or dye-uptake was determined by measurement of the K/S value of the dyed samples on Gretag Macbeth color eye 3100 spectrophotometer. Before measuring (K/S), samples were pressed with hot iron and left for about 2 hour to cool down. The results for the 3 Reactive dyes selected, dyed in 1% and 1.5% shade is presented in Table 6-11.

The treatment with the enzyme catalase presented color strengths similar to the treatments with the methods of chemical reducer and successive washes, with the advantage of less water consumption, avoiding the use of noxious chemicals, reduced processing time and cost effectiveness. The physical properties of the final product did not show any significant alterations and it can be visualized from Figure 2-7.

Table 5. Peroxide stripe test result after different treatments

Treated fabric	Gradation 0 · 1 · 3 · 10 · 30 · 100 / H_2O_2		
Samples	Catalase	Sodium Dithionite	Water wash
1	0	0	0
2	0	0	0
3	1	1	0
4	0	0	1
5	0	1	0
6	0	0	0
7	0	0	0
8	0	0	1
9	1	0	1
10	0	0	0
11	0	1	0
12	1	0	0
13	0	0	0
14	0	0	0
15	0	1	0
16	0	0	0
17	0	0	0
18	0	0	0
Sum	3	4	3
Average	0.1666	0.222	0.1666

Table 6. Color Strength (K/S) of 1% shade Blue dyed samples after different treatment

Blue dyed sample	Catalase	Sodium dithionite	Water washing
1	2.8176	3.2057	2.9094
2	3.2682	3.0103	3.1709
3	3.7606	2.7831	2.9671
4	2.9843	3.0569	3.1801
5	2.8695	2.9817	2.9432
6	3.6319	2.4530	3.0129
8	2.9890	3.2603	2.9632
9	3.3751	3.0581	3.0432

Longer processing times at temperature of 40°C increased the efficiency of the enzymatic treatment. The maximum efficiency of the enzyme was reached at a concentration of 0.5 g/l of the enzyme solutions, 45°C and a reaction time of the enzyme of 10 min.

Table 7. Color Strength (K/S) 1% Red dyed samples after different treatment

Red dyed sample	Catalase	Sodium dithionite	Water washing
1	4.5944	3.9876	4.4678
2	4.8302	4.6785	4.4678
3	4.4832	3.8652	3.9578
4	3.9809	4.0324	4.9670
5	4.9212	4.5642	4.0098
6	4.6578	4.9102	4.7652
7	4.3559	4.0567	4.3027
8	4.5467	3.9865	3.9783
9	4.6785	4.5410	4.0675

Table 8. Color Strength (K/S) 1% Yellow dyed samples in different treatment

Yellow dyed sample	Catalase	Sodium dithionite	Water washing
1	2.3091	2.3213	2.3212
2	2.3164	2.4051	2.3340
3	2.3901	2.2794	2.3960
4	2.1419	2.3035	2.1386
5	2.1280	2.3451	2.3411
6	2.2210	2.3010	2.3247
7	2.2125	2.3113	2.1223
8	2.4025	2.0133	2.4079
9	2.3041	2.1332	2.2131

Table 9. Color Strength (K/S) 1.5% Blue dyed samples after different treatment

Blue dyed sample	Catalase	Sodium dithionite	Water washing
1	4.1718	3.8957	3.9094
2	3.7264	4.0103	4.1709
3	3.7606	3.7831	3.9671
4	4.2043	4.0569	4.1801
5	3.9781	4.0120	3.9432
6	3.8319	3.7530	3.8929
7	3.9098	3.9306	4.0807
8	3.9890	3.8639	3.9632
9	4.2751	3.9581	3.8143

Table 10. Color strength (K/S) 1.5% Red dyed samples after different treatments

Red dyed sample	Catalase	Sodium dithionite	Water washing
1	6.4962	6.3560	6.4631
2	6.2540	6.3203	5.9872
3	6.1293	5.8976	6.0210
4	6.0345	5.9983	6.1670
5	6.2154	6.0670	6.0098
6	5.9870	5.9983	5.9652
7	6.2932	6.0170	6.3027
8	5.8932	5.9740	5.9783
9	6.0129	6.0219	6.0675

Table 11. Color Strength (K/S) 1.5% Yellow dyed samples after different treatments

Yellow dyed sample	Catalase	Sodium dithionite	Water washing
1	2.3528	2.2192	2.4325
2	2.2319	2.4101	2.5246
3	2.3154	2.4507	2.3359
4	2.4031	2.3031	2.2204
5	2.6180	2.3451	2.4506
6	2.3410	2.2091	2.3247
7	2.2496	2.3013	2.2123
8	2.4005	2.4033	2.2079
9	2.3021	2.0332	2.2079

In this experiment, the effect of different treatement of catalase enzyeme, sodium dithionite and seecssive washing dyed in 1.5% shade concentration were studied.

Figure 2. Catalase treated samples dyed in 1% shade

Figure 3. Sodium dithionite treated samples dyed in 1% shade

Figure 4. Successive washing treated sample dyed in 1% shade

Figure 5. Catalase treated samples dyed in 1.5% shade

Figure 6. Sodium dithionite treated samples dyed in 1.5% shade

Figure 7. Successive washing treated sample dyed in 1.5% shade

Comparison between Colors strength (K/S) during dyeing was investigated. It can be seen from tables that there is no significant difference in color strength between conventional and enzyme treatments.

Color Fastness

Color fastness to washing of the dyed fabric samples was determined as per method using launder-O-meter (Mesdan Lab) following wash fastness test method. The wash fastness rating was assessed using grey scale as per ISO-05-A02:1993 (change in colour; loss of shade depth) and ISO-105-AO3: 1993 (extent of staining on white).

Washing Fastness

Washing fastness test has been carried out to investigate effect of different treatments on washing fastness of vinyl sulphone reactive dyd samples It can be seen from Table 12 that washing fastness test result of Bezactive Blue V-RN, Red V-RB and Yellow V-GR Dyed Fabric, the shade fading or change in colour and staining on white lies between good and excellent. The results obtained are comparable among the treatments.

Table 12. Wash fastness test results

Depth of shade	1% dye		1.5% dye	
Treatment / Dyed samples	Staining on white	Change in colour	Staining on white	Change in colour
Catalase treated				
Blue dyed S1	5	5	4/5	5
Blue dyed S2	4/5	4/5	4/5	5
Red dyed S1	4/5	5	4/5	4/5
Red dyed S2	4/5	5	4/5	5
Yellow dyed S1	5	4/5	4/5	4/5
Yellow dyed S2	4/5	4/5	4/5	4/5
Sodium dithionite treated				
Blue dyed S1	4/5	4/5	5	5
Blue dyed S2	4/5	5	4/5	5
Red dyed S1	4/5	4/5	4/5	4/5
Red dyed S2	4/5	5	4/5	4/5
Yellow dyed S1	4/5	5	4/5	5
Yellow dyed S2	4/5	4/5	4/5	5
Successive washing				
Blue dyed S1	4/5	5	5	5
Blue dyed S2	4/5	5	4/5	5
Red dyed S1	4/5	5	5	5
Red dyed S2	5	5	4/5	4/5
Yellow dyed S1	4/5	5	4/5	5
Yellow dyed S2	4/5	5	4/5	5

Rubbing Fastness

Color fastness to rubbing (dry and wet) was assessed as per IS: 766-1984 method using a manually operated crock meter and grey scale as per ISO-105-AO3:1993 (extent of staining). In rubbing fastness experiment, the effect of different treatement on rubbing fastness of Bezactive Blue V-RN, Red V-RB and Yellow V-GR dyed fabric was investigated. It can be seen from the Table 13 that staining on white in case of wet and dry rubbing lies between good and excellent and comparable among treatments. Dry rubbing fastness is better than wet rubbing in all cases.

Table 13. Rubbing fastness test results

Treatment / Dyed samples Depth of shade	Staining on white in Wet & Dry rubbing			
	1% dye		1.5% dye	
	Wet	Dry	Wet	Dry
Catalase treated				
Blue dyed S1	4/5	5	4	5
Blue dyed S2	4	5	4/5	5
Red dyed S1	4/5	5	5	4/5
Red dyed S2	4	5	4	4/5
Yellow dyed S1	4/5	5	4/5	5
Yellow dyed S2	4/5	5	4	5
Sodium dithionite treated				
Blue dyed S1	4/5	5	4	4/5
Blue dyed S2	4/5	5	4	4/5
Red dyed S1	4/5	5	4/5	4/5
Red dyed S2	4	5	4/5	4/5
Yellow dyed S1	4/5	5	4	4/5
Yellow dyed S2	4/5	5	4/5	5
Successive washing				
Blue dyed S1	4	5	4	4/5
Blue dyed S2	4/5	5	4/5	5
Red dyed S1	4	4/5	4/5	5
Red dyed S2	4/5	5	4	4/5
Yellow dyed S1	4/5	5	4/5	5
Yellow dyed S2	4	4/5	4/5	5

Conclusion

The use of a catalase enzyme for the elimination of residual hydrogen peroxide from bleached cotton fabrics is good by considering the dyed quality parameters (K/S value, color difference and colour fastness) of the fabric dyed with reactive dye. Similar results were observed while it removes the residual H_2O_2 with sodium dithionite and successive washing processes. The catalase can be applied after two washing, then addition can be done directly in the dyeing bath. As a consequence, it decreases the washing steps, water consumption and effluent volume. Therefore, catalase enzyme is an alternative clean up option for removal of residual hydrogen peroxide. Due to complete and quick elimination of residual hydrogen peroxide, it can be improved right first time and reproducibility in subsequent dyeing and by-products of the residual H_2O_2 are completely inert to dyestuffs and fabrics, so it can be use the bath as it is for dyeing section.

Conflicts of interest

Authors declare no conflict of interest.

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