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Removal of Hydrolyzed Reactive Dyes from Cotton Fabric Using Spent H_2O_2: A Green Approach

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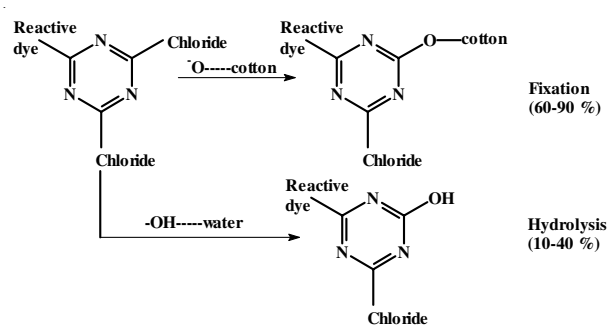
In this paper, the effectiveness of spent H_2O_2 bleach liquor was evaluated for the removal of hydrolyzed reactive dyes from cotton fabrics. This study was conducted with an aim to develop a new textile dyeing method with lesser amount of water and chemicals, without compromising quality of dyeing. In this context, spent bleach water from a textile factory was collected and used in wash-off step of reactive dyeing to ascertain colour fastness properties of selected reactive dyes. Four dyeings, using C.I. Reactive Yellow 145, C.I. Reactive Red 194, C.I. Reactive Blue 19 and C.I. Reactive Black 5,were carried out and dyed fabric samples were subjected to both conventional washing and new washing method containing spent H_2O_2 bleach liquor. Fabrics washed-off with both methods were compared in terms of washing fastness, rubbing fastness, change of colour and magnitude of total colour difference (ΔE*). The colour fastnesses properties and final shade of fabrics washed-off with spent bleach were found to be comparable to those washed-off conventionally. This study concludes that spent of H_2O_2 bleach is a potential candidate for removing the unfixed reactive dyes from cotton fabrics.

Keywords: Textile Dyeing, Bleaching, Washing-off, H_2O_2, Wastewater, Fastness.

INTRODUCTION

Principles of chemistry play very important role in textile industry. Major environmental problems of textile sector is the use of considerably high amount of fresh water and discharge of highly polluted waste water[1,2]. Clothing made of cellulosic fibersis usually dyed with reactive dyes due to their brightness, colour fastness properties and ease of application[3-5]. Although reactive dyes make covalent bonding with cotton fibers during the fixation phase[6], fiber fixation is always accompanied by some degree of dye hydrolysis, leading to the formation of unfixed or hydrolyzed dyes[3,5,7]. The reactions of reactive dye are as follow:



It is critical to remove these hydrolyzed dyes from the textile materials to achieve commercially acceptable colour fastness properties. The conventional washing method to remove hydrolyzed dyes is to rinse at high liquor ratio with frequent changes of liquor baths, including soaping and boiling steps at high temperature. Consequently, this makes textile washing operation as the most water intensive processes in textile production[8,9]. Several researchers have tried to reduce water consumption in washing process by developing special washing detergents, cationic fixing compounds and easy-to-wash dye stuffs[8,10,11].

In this study, efficiency of spent bleach bath as a washing agent to remove hydrolyzed reactive dyes from fabric was evaluated. Bleaching of cotton textiles using H_2O_2 at high temperature is widely practiced[12]. Since H_2O_2 is a weak acid ($K_{diss} = 1.78 \times 10^{-12}$ at 20 °C), it dissociates at alkaline pH (10-12) to generate perhydroxylanions (HO_2^-). These ions react with cellulose to yield bleaching action.

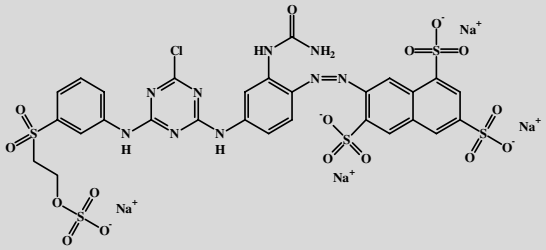
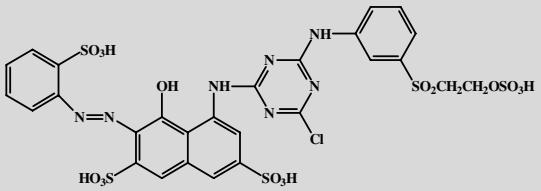
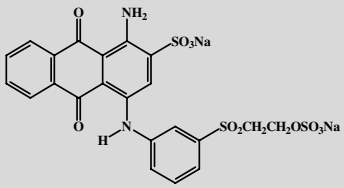
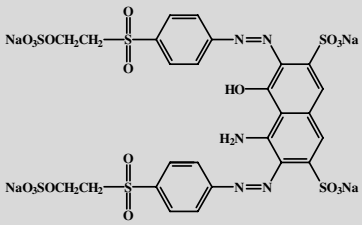
$$H_2O_2 \rightleftharpoons H^+ + HO_2^- \quad (1)$$

The results obtained in the study suggest that the use of the spent bleach bath can replace conventional washing method without compromising fastness properties or altering final shade. The new method also consumes reduced amount of water and energy and minimize pollution load.

EXPERIMENTAL

In this study, 100 % cotton knitted fabric having single jersey construction and 200 g/m^2 weight was used. Four reactive dyes used in the study are summarized in Table-1.

TABLE-1
REACTIVE DYES USED IN THE EXPERIMENTAL WORK

CI name	Commercial name	Chemical structure
C.I. Reactive Yellow 145	Synozol Yellow 2GR	
C.I. Reactive Red 194	Synozol Red 6BN	
C.I. Reactive Blue 19	Sinarcion Blue VR/specal	
C.I. Reactive Black 5	Sinarcion Black VBS	

Dyeing chemicalslike sodium sulphate and sodium carbonate, were of commercial grade and used without any further purification.

Dyeing and washing methods: The amount of dye used for all dyeing was 5 % o.w.f. because this depth of shade represents a dark shade. A 20 g sample consisted of four fabric swatches, each weighing 5 g, was used in all trials. The dyeing took place in an IR laboratory dyeing machine (Data colour, Ahiba Nuance) at a liquor ratio of 1:8, in the presence of 80 g/L Na_2SO_4 and 20 g/L Na_2CO_3. All dyeing was carried out at 60 ºC for 60 min using isothermal all-in-one laboratory method. At the end of fixation phase, dyed samples were removed from the dyeing machine, rinsed in tap water and subjected to wash-off treatment. One fabric swatch (5 g) from each dyeing was washed-off with conventional washing method as shown in Table-2. This fabric swatch was regarded as reference sample.

TABLE-2
CONVENTIONAL WASHING-OFF METHOD

Step	Washing steps	Temperature (ºC)	Time (min)
1	Cold rinse	30	10
2	Neutralization with CH_3COOH	30	10
3	Warm wash	50	10
4	Hot wash	80	10
5	Soaping	85	10
6	Cold rinse	30	10

The remaining three swatches were subjected to new wash-off method comprised of 1 to 3 washing treatments using 100 % spent H_2O_2 bleach water (Table-3). First, second and third fabrics watches under went washing step 3, steps 3-4 and steps 3-5, respectively. At the completion of washing process, fabric samples were removed from the machine, squeezed, dried and conditioned for 24 h before evaluating for the uniformity of dyeing, change of shade and colour fastness properties.

TABLE-3
NEW WASHING-OFF METHOD
USING SPENT H_2O_2 BLEACH WATER

Step	Washing steps	Temperature (ºC)	Time (min)
1	Cold rinse	30	10
2	Neutralization with CH_3COOH	30	10
3	Wash#1 with spent H_2O_2 bleach	50	10
4	Wash#2 with spent H_2O_2 bleach	80	10
5	Wash#3 with spent H_2O_2 bleach	85	10
6	Cold rinse	30	10

Colour measurement and fastness properties: The colour fastness of dyed fabrics was determined using AATCC test methods. AATCC test methods 61-2001-2A and 8-2001 were used to evaluate colour staining and rubbing fastness, respectively[13,14]. Colour difference values between samples washed-off with conventional and new methods were deter-

mined using a spectrophotometer (Data colour, Spectra flash SF-600 Plus CT) at the following settings: CIELAB colour equation, Illuminant D65, specular reflection included mode, 10º standard observer and aperture size of LAV 30 mm[4]. After folding each sample twice, four measurements were carried out at different positions on the fabric surface and averaged.

RESULTS AND DISCUSSION

In order to determine the effect of spent H_2O_2 bleach water on fastness properties of dyed samples were exposed to varied number (1 to 3) of treatments. Table-4 shows a comparison of fastness properties of reference and samples washed-off using spent H_2O_2 bleach water. The data indicates that both washing systems exhibited similar washing and rubbing fastness properties in terms of staining to the adjacent multi-fiber strip. For C.I. Reactive Yellow 145 dyeing, the fabric sample washed-off with spent H_2O_2 bleach water showed identical fastness properties, mainly in the range of 4.5 to 5.0. The change of shade was found to be 5, showing a similar shade compared to that of reference. In case of C.I. Reactive Red 194, the good fastness values confirm that new wash-off method under investigation removed all hydrolyzed dyes effectively. Rest of the dyes followed the similar trend.

Colour differences between reference fabric and those washed-off using spent H_2O_2 bleach water were reported in terms of ΔL*, Δc*, Δh* and ΔE* values (Table-5). Overall results clearly showed that total colour difference in all cases was negligible (ΔE* < 1). The results pertinent to C.I. Reactive Yellow 145 illustrate that 1st wash-off using spent H_2O_2 bleach water was good enough to achieve similar depth of shade. Negligible differences in lightness (ΔL* = -0.48), Hue (ΔH* = -0.35) and total difference (ΔE* = 0.51) confirm that colour

TABLE-4
COLOUR FASTNESS PROPERTIES OF SAMPLES WASHED-OFF WITH SPENT H_2O_2 WATER

	No. of washes	Crocking		Multi-fiber staining			Change of shade
		Dry	Wet	Cotton	Nylon	Polyester	
C.I. Reactive Yellow 145							
Reference	-	5	5	5	5	5	-
Sample-1	1	5	5	5	5	5	4.5
Sample-2	2	5	5	5	5	5	4.5
Sample-3	3	5	5	5	5	5	4.5
C.I. Reactive Red 194							
Reference	-	5	5	5	5	5	-
Sample-1	1	5	5	5	5	5	4.5
Sample-2	2	5	5	5	5	5	4.5
Sample-3	3	5	5	5	5	5	4.5
C.I. Reactive Blue 19							
Reference	-	5	5	5	5	5	-
Sample-1	1	5	5	5	5	5	4.5
Sample-2	2	5	5	5	5	5	4.5
Sample-3	3	5	5	5	5	5	4.5
C.I. Reactive Black 5							
Reference	-	5	5	5	5	5	-
Sample-1	1	5	5	5	5	5	4.5
Sample-2	2	5	5	5	5	5	4.5
Sample-3	3	5	5	5	5	5	4.5

TABLE-5
CIELAB COLOUR DIFFERENCES OF SAMPLES WASHED-OFF WITH SPENT H_2O_2 WATER AND COMPARED WITH REFERNCE

Dyes	No. of washes	CIELAB Colour difference values					
		ΔL*	Δa*	Δb*	Δc*	Δh*	ΔE*cmc
C.I. Reactive Yellow 145							
Sample-1	1	-0.48	0.88	0.87	1.19	-0.35	0.51
Sample-2	2	-0.05	0.44	-0.31	-0.10	-0.53	0.38
Sample-3	3	0.23	0.44	-0.87	-0.61	-0.76	0.58
C.I. Reactive Red 194							
Sample-1	1	-0.67	-1.02	-1.21	-1.06	-1.17	0.61
Sample-2	2	-0.29	-1.27	-1.70	-1.41	-1.58	1.02
Sample-3	3	-0.11	-1.79	-2.62	-2.00	-2.46	1.53
C.I. Reactive Blue 19							
Sample-1	1	-1.47	0.45	0.34	-0.32	0.47	0.87
Sample-2	2	-0.67	0.10	0.48	-0.48	0.11	0.42
Sample-3	3	0.79	-0.63	1.14	-1.16	-0.60	0.78
C.I. Reactive Black 5							
Sample-1	1	-0.05	-0.04	0.04	-0.04	-0.05	0.08
Sample-2	2	0.46	-0.07	-0.40	0.40	-0.02	0.54
Sample-3	3	0.44	-0.11	-0.06	0.07	-0.10	0.46

properties of treated fabric were comparable to those of the reference sample. The total colour difference (ΔE*) was further reduced to 0.38 when sample under went 2nd wash-off.

In the case of C.I. Reactive Red 194, the shade of sample treated with spent H_2O_2 bleach was found to be slightly darker (ΔL* = -0.67), slightly duller (Δc* = -1.06) and within acceptable total colour difference (ΔE* = 0.61). The next two washes did not further improve results and thus only one wash using spent H_2O_2 seems to be sufficient to remove hydrolyzed C.I. Reactive Red 194 from the fabric. For C.I. Reactive Blue 19 and C.I. Reactive Black 5, total colour difference (ΔE*) values of 0.42 and 0.08 were achieved at 2nd and 1st washes, respectively.

A direct relation between the total colour difference (ΔE*) and number of wash-off treatments is shown in Fig. 1.

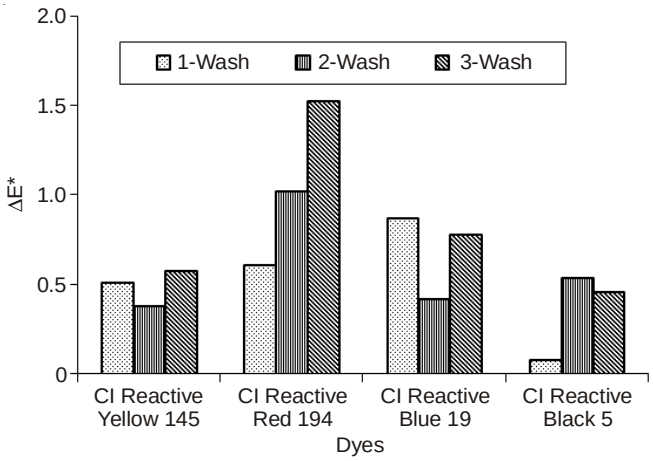


Fig. 1. Effect of washes on total color change (ΔE*)

Conclusion

This study investigated a new wash-off method for the removal of surface deposited hydrolyzed dyes from cotton fabrics with reduced quantity of fresh water. The efficiency of the new method using spent H_2O_2 bleach water was investigated on cotton fabrics dyed with C.I. Reactive Yellow 145, C.I. Reactive Red 194, C.I. Reactive Blue 19 and C.I. Reactive Black 5 in dark shades. Based on the results obtained in this study, it is found that spent H_2O_2 bleach based wash-off method was capable to produce similar colour fastness properties with minimal colour difference and with reduced water consumption. This study concludes that spent H_2O_2 bleachis a potential candidate for removing the unfixed reactive dyes from cotton fabrics.

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