



## Standardization of Dyeing Variables of Reactive Dye for Tie and Dye on Cotton

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### ABSTRACT

A study was conducted to standardize the dyeing variables of hot reactive dye for tie and dye technique. The optimum wave length was 520 nm, out of a set of wave length ranging from 400-700 nm on the basis of highest optical density. Eight dye concentrations (1, 2, 3, 4, 5, 6, 7, and 8 %) were tried and two dye concentrations were selected on the basis of dye absorption and colour fastness properties. 4 percent dye concentration gave maximum percent dye absorption and 2 percent dye concentration showed excellent fastness to wash and light. Five different dyeing temperatures (60, 70, 80, 90 and 100°C) were tried and 80°C was found best for 2 and 4 percent dye concentration respectively. Five different dyeing times (80, 90, 100, 110, 120 minutes) were tried and 90 minutes was found to be the best for both 2 and 4 percent dye concentration. Dyeing was carried out at seven different pH values (9, 9.5, 10, 10.5, 11.0, 11.5 and 12.0), the maximum dye absorption was observed at 10.5 percent for both the dye concentration. 60g/l sodium sulphate was found best for 2% dye concentration and 70g/l was found best for 4% dye concentration. Similarly 15g/l of sodium carbonate was found best for 2% dye concentration and 20g/l was found best for 4% dye concentration in two instalments of both the auxiliaries.

**KEYWORDS:** Reactive dye, Standardize, Dyeing variables, Dye absorption

### Introduction

Reactives are the most important class of colours for the dyeing of cotton. Dyeing is a complex process, where number of variables are involved. Dyeing process is broadly governed by fabric, dye and machine type, MLR, time, temperature, pH of the fabric and liquor, type of auxiliary used etc. Any minor variation in any of these variables causes problems in dye reproducibility, though it is possible to achieve reproducibility in dyeing results. By standardizing each and every variable consistent reproducible results can be achieved. (<http://www.shivananddyes.com/faq.html>)

In this class of dyes, the molecules permanently bind with cellulose based fibers (cotton, rayon, hemp, linen) as well as silk, when the pH is raised. Soda ash (sodium carbonate) is generally used to raise the pH and is either added directly to the dye or in a solution of water in which garments are soaked before dyeing. They do not fade with washing. (Anonymous, 2007)

The optimum temperature for hot reactive dye fixation was 80°C. It was observed that the percent dye absorption increased with increase in temperature upto 80°C and after that, the dye absorption decreased. Taylor, 2001; Bae et al., 1998 used hot dyeing for monochlorotriazine reactive dye and good colour yield was obtained when dyed at 80°C. The temperature of the dye bath affects the affinity of the dye molecules towards fibre, rate of hydrolysis, migration and covalent bond formation, therefore the dyeing temperature selected must be as per the dye sub class.

Auxiliaries play an important role in dyeing of reactive dyes. They help in better exhaustion of the dyes. Alam et al., 2008; Farha et al., 2010 reported that the addition of electrolyte to the dye liquor of anionic dye increased the uptake of dye by the fabric. The electrolyte used in dyeing dissociates completely in aqueous dye liquor. For entering into the fabric, the charge on surface (negative in fabric) will have to be neutralized since both anionic dyes and fabric have the same charge. Sodium ion (Na<sup>+</sup>) from sodium sulphate is cationic and in the dye liquor is attracted by the negatively charged fabric. By bonding the sodium cations neutralize the anionic surface charge of the fabric. Now the neutralized fabric can attract the organic dye molecules which have a greater affinity for the fabric than the aqueous solution.

Cotton based tied articles have been dyed mostly with direct dyes till today, which always pose problems both to dyers as well as consum-

ers as these articles fades very easily so an attempt has been made to standardize tie and dyeing process for cotton using reactive dye which have good colour fastness.

### Materials and Methods

Grey cotton fabric was desized, scoured and bleached and preliminary data of fabric was also taken. The samples were tied and dyed with reactive red H brand dye to optimize dyeing conditions.

Standardization of dyeing process for tie dyeing of cotton fabric with reactive dye: Experiments were carried out to optimize dye concentration, dyeing time, dyeing temperature, dyeing pH and concentration of dyeing auxiliaries for tie dyeing of cotton fabric with reactive dye.

- Determination of optimum wave length: For determining the optimum wavelength 1 ml of dye was diluted to 100 times, centrifuged at 2500 rpm for 20 minutes and optical density of the solution was taken on a spectrophotometer at different wave lengths from 400 to 700 nm. The wavelength reflecting the highest optical density was selected.
- Optimization of dye concentration: For determining the optimum dye concentration, eight different concentrations of reactive dye i.e. 1, 2, 3, 4, 5, 6, 7 and 8 percent were taken and samples were dyed at 80°C -100°C for 30 minutes with constant stirring using 50 g/l sodium sulphate. Sodium carbonate was also added to bring the pH at 11. Optical density of dye solutions before and after dyeing was recorded at optimum wavelength. The dye solution giving the maximum dye absorption was taken as optimum dye concentration. (Singh and Rose, 2001)
- Optimization of dyeing temperature: To optimize dyeing temperature, dyeing was carried out using optimum concentration of dye at five different temperatures i.e. 60, 70, 80, 90 and 100°C. The temperature giving maximum dye absorption was taken as the optimum dyeing temperature.
- Optimization of dyeing time: The cotton samples were dyed using optimum dye concentration for five different time durations i.e. 80, 90, 100, 110 and 120 minutes. The optimum dyeing time was selected on the basis of maximum dye absorption.
- Optimization of dyeing pH: Dye solutions were set to pH 9.0, 9.5, 10.0, 10.5, 11.0, 11.5, 12.0. The samples were added to the solution and dyed with optimized conditions and dyeing pH was optimized on the basis of maximum dye absorption.
- Optimization of sodium sulphate and sodium carbonate concen-

tration: Different concentrations of sodium sulphate i.e. 30, 40, 50, 60, 70, 80 and 90 g/l and sodium carbonate i.e. 5, 10, 15, 20 and 25 g/l were added in dye bath. Optimum concentration of both the auxiliaries was decided on the basis of maximum dye absorption. The number of instalments of both the auxiliaries was also optimized.

**Results and discussion**

i. Determination of wave length: Optimum wave length is the wave length at which maximum optical density was observed. The optical density was recorded from 400 to 700 nm. The maximum optical density was observed at 520 nm hence selected for further experiments. (Fig. 1).

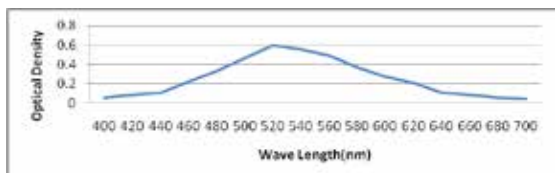


Fig.1: Wave length for hot reactive dye

ii. Optimization of dye concentration: Dye concentration was optimized on the basis of maximum dye absorption and colour fastness against washing and sunlight.

**a) On the basis of dye absorption:** To optimize dye concentration 1, 2, 3, 4, 5, 6, 7 and 8 % dye concentration were taken. The data in table 1 exhibits that percent dye absorption increased with increase in dye concentration upto 4 percent and decreased with the increase in dye concentration. Hence 4 % dye concentration was selected. Alam *et al.* (2008) observed that the dye absorption by cotton fabric decreased with the increase in dye concentration in the dye bath. This may be explained by the fact that the presence of more dye ions hinders the absorption of dye by the fabric whereas rare ions favor it. It is found that with the increase in dye concentration, the absolute quantity of the absorbed dye increases while the relative quantity diminishes.

**Table-1.Optimization of dye concentration on the basis of dye absorption**

Dye concentration (%)	1	2	3	4	5	6	7	8
Percent dye absorption	64.94	67.56	70.84	71.53	65.27	62.81	58.32	49.86

b) On the basis of colour fastness properties: The colour fastness properties of the samples dyed with 1 to 8 percent dye concentration are presented in table 2. It is clear from the table that 2 percent dye concentration showed maximum colour fastness with mean score 4.50. The samples were also evaluated visually and it was observed that 2 percent dye concentrations gave best colour and shade, hence used for further work. Vastsala (2003) emphasized that reactive dyes exhibit good fastness properties. This is due to the very stable electron arrangement and covalent bond that existed between the dye molecules and the fibre polymers which provide good resistance to washing and sunlight, respectively.

**Table-2.Optimization of dye concentration on the basis of colour fastness**

Percent dye concentration	Fastness grades			Mean score
	Washing		Sunlight	
	CC	CS	CC	
1	4/5	4/5	4	4.33
2	5	4/5	4	4.50
3	4/5	4	4	4.16
4	4/5	4	4	4.16
5	4	4	4	4
6	4	4	4	4
7	4	4	4	4
8	4	4	4	4

CC: colour change, CS: colour staining

Hence on the basis of dye absorption and colour fastness 4percent and 2 percent dye concentrations were selected.

iii. Optimization of dyeing temperature: Dyeing temperature is the

temperature that is suitable for dye absorption and fixation of dye on the textile material. For optimizing dyeing temperature, dyeing was carried out at five different temperatures i.e. 60, 70, 80, 90 and 100°C. The percent dye absorption at different temperatures using 2 and 4 percent dye concentration is given in table 3. It is clear from the table that the percent dye absorption increased with the increase in dyeing temperature up to 80°C and decreased with further increase in temperature, therefore 80°C was selected as the optimum dyeing temperature for both the dye concentrations. Bae *et al.* (1998) investigated that the adsorption of reactive mono chlorotriazine red dye on cellulose at 80°C was maximum.

**Table-3.Optimization of dyeing temperature**

Temperature (0C)	Dye concentration (%)	
	Percent dye absorption	
	2	4
60	67.48	71.23
70	69.44	73.67
80	71.47	75.48
90	68.77	72.34
100	64.21	68.73

Dye conc.- 2 and 4%

iv. Optimization of dyeing time: Dyeing time is the time required to get the dye fixed on fabric. For the present study 80, 90, 100, 110 and 120 minutes were taken as dyeing time for standardization. The percent dye absorption at different dyeing time employing optimum dye concentration and dyeing temperature are given in table 4. It is evident that maximum percent dye absorption i.e. 74.30 and 80.65 for 2 and 4 percent dye concentration, respectively was observed at 90 minutes dyeing time and the dye absorption decreased further due to the saturation of dye bath as stripping of dye might have taken place, hence was selected for further work.

**Table-4.Optimization of dyeing time**

Time(minutes)	Dye concentration (%)	
	Percent dye absorption	
	2	4
80	70.18	74.34
90	74.30	80.65
100	73.04	77.52
110	72.54	76.07
120	71.16	75.47

Dye conc.- 2 and 4%, dyeing temp.- 800C

v. Optimization of dyeing pH: The role of pH is very important in dyeing as it affects the shade and percent dye absorption. The percent dye absorption at different pH values at optimum dye concentration, temperature and time is presented in table 5. The table shows that percent dye absorption at pH 9.0, 9.5, 10.0, 10.5, 11.0, 11.5 and 12.0 for 2 percent dye concentration was 66.42, 68.76, 73.33, 75.85, 74.54, 72.65 and 67.90 whereas for 4 percent it was 69.33, 76.29, 80.64, 82.78, 80.47, 74.51 and 72.91 respectively. The maximum absorption was at pH 10.5 therefore it was selected as optimum pH for both the concentrations of dye. The pH value of the dye bath affects both the degree and rate of fixation of reactive dyes on cotton.

**Table-5.Optimization of dyeing pH**

Dyeing pH	Dye concentration (%)	
	Percent dye absorption	
	2	4
9.0	66.42	69.33
9.5	68.76	76.29
10.0	73.33	80.64
10.5	75.85	82.78
11.0	74.54	80.47
11.5	72.65	74.51
12.0	67.90	72.91

Dye conc.- 2 and 4%, dyeing temp.- 800C, dyeing time-90 min.

vi. Optimization of concentration of auxiliaries: For optimization of auxiliaries different concentrations of sodium sulphate and sodium carbonate were taken. Sodium sulphate was used as electro-

lyte and exhausting agent and sodium carbonate was added for the constant control of pH value. The optimized auxiliaries were added in two equal instalments for better absorption of colour. The percent dye absorption at different concentrations of sodium sulphate and sodium carbonate employing the optimum dyeing conditions are given in table 6. Sodium sulphate (60 g/l) and sodium carbonate (15 g/l) were selected for 2% dye concentration and 70 g/l of sodium sulphate and 20 g/l of sodium carbonate was selected for 4% dye concentration. Farha et. al. (2010) envisaged that exhaustive dyeing of cotton fabric with reactive dyes requires the presence of sodium sulphate, which suppresses negative charge build-up at the fibre surface and promotes dye uptake.

#### Conclusion:

It can be concluded from the study that for 2 percent and 4 percent dye concentration all the dyeing variables were same i.e. 800C dyeing temperature, 90 minutes dyeing time, 10.5 dyeing pH except dyeing auxiliaries. The 2% dye concentrations gave maximum dye absorption at 60 g/l of sodium sulphate and 15 g/l of sodium carbonate whereas 70 g/l of sodium sulphate and 20 g/l of sodium carbonate showed maximum dye absorption for 4% dye concentration in two equal instalments. The amount of the auxiliaries increased if the dye concentration is increased.

**Table 6: Optimization of auxiliaries concentration**

Sodium sulphate			Sodium carbonate		
(g/l)	Dye concentration (%)		(g/l)	Dye concentration (%)	
	Percent dye absorption			Percent dye absorption	
	2	4		2	4
30	71.34	74.48	5	74.92	76.44
40	73.67	77.62	10	76.37	79.98
50	75.38	81.50	15	79.86	80.32
60	77.83	82.71	20	73.24	84.36
70	76.45	83.54	25	72.49	75.69
80	76.24	80.74			
90	74.48	78.89			

Dye conc.-2% & 4%, dyeing temp. - 800C, time- 90 min. and pH- 10.5

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