

Dyeing of cotton fabrics using reactive dyes by microwave irradiation technique

K Haggag, M M El-Molla^a & Z M Mahmoud

Textile Research Division, National Research Centre, Dokki, Cairo, Egypt

Received 11 November 2013; revised received and accepted 13 March 2014

Dyeing of cotton fabrics with reactive dyes using exhaustion method has been done using microwave irradiation technique. Different parameters such as dye concentration, alkali concentration, salt concentration, power of microwave used and liquor ratio have been studied and the results are compared with conventional technique. It is observed that the microwave technique saves 90 min in the dyeing time 75% in the salt used and 20% in alkali used, indicating the saving in time, energy and money. The overall fastness properties to rubbing, washing, light and perspiration for the dyed samples are ranging from very good to excellent.

Keywords: Cotton fabrics dyeing, Exhaustion dyeing, Microwave irradiation, Reactive dyes

1 Introduction

Reactive dyes are widely used for the dyeing of cotton. These dyes are anionic in character and, in general, are water soluble due to the presence of sulphonic groups (SO_3^-) in the chemical structure. However, since cotton itself adopts an anionic surface charge in water, these dyes have low intrinsic affinity for the fibre. The repulsive charge between dye and cotton can be overcome by adding an electrolyte such as sodium chloride or sodium sulphate, which has the effect of screening the surface charge on the fibre. The large quantity of salt in the effluent, however, causes pollution of rivers and streams and upsets the biological equilibrium¹. In order to both reduce the usage of salt and increase in dye bath exhaustion, number of attempts have been made to modify the cotton fibre using compounds containing cationic groups²⁻⁵. These are mainly quaternary ammonium salts or polymeric amines or amides^{6,7}. Unfortunately many of the chemicals used for this purpose are also undesirable environmentally². The chemical modification of cotton in order to improve its dye ability with anionic dyes such as reactive, direct, acid, sulphur and vat dyes have received considerable attention in recent years⁸. The advantages of this process include reduced environmental impact, and energy consumption⁹. The microwave irradiation has been used in the dyeing processing of cellulose fabric. In the conventional processing of fabric, a large amount of energy is

consumed. Some new techniques and methods for saving energy were investigated¹⁰⁻¹². Microwave heating, as an alternative to conventional heating technique, has been proved to be more rapid, uniform and efficient. Microwave irradiation is one of powerful techniques of non-contact heating, because the dielectric substances with large dielectric loss constant are vigorously affected by vibration and rotation of permanent dipoles in microwave field. Microwave has been used for reacting, heating and drying cellulose materials. The microwave energy can easily penetrate to particle inside and all particles can be heated simultaneously, thus reducing heat transfer problems. However, the microwave irradiation could affect the chemical and morphological structure of cellulose, including some physical properties. It is found that the report on the effect of microwave irradiation on the physical properties and morphological structure of cellulose is scanty.

The present study is therefore aimed at dyeing the cotton fabrics with reactive dyes using microwave irradiation to save time, energy, and money.

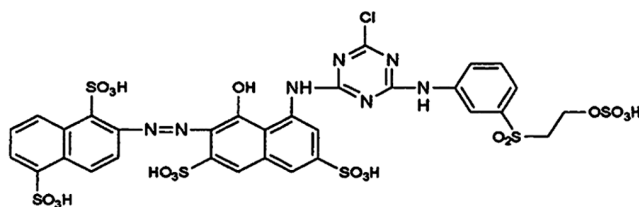
2 Materials and Methods

2.1 Materials

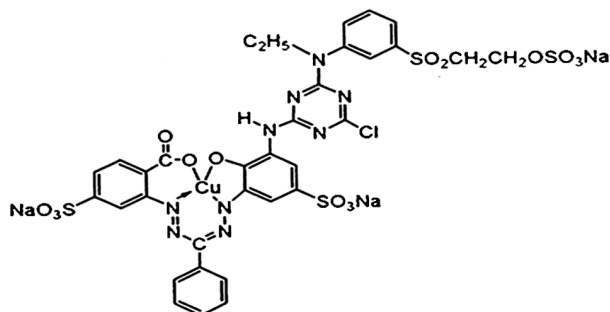
The grey cotton fabric (supplied by the Miser Helwan Company), having the specification was 140g/m^2 , warp 36 threads/cm, yarn count 30/1, weft 34 threads/cm, and yarn count 30/1, was used.

Solazol Red SP-3B (C.I. Reactive Red 195) and Solazol Blue SP-BRF (C.I. Reactive Blue 221) were supplied by New Trend Co. Egypt. The structure of

^a Corresponding author.
E-mail: melmolla@yahoo.com



C. I. Reactive Red 195



C. I. Reactive Blue 221

Scheme 1

both the dyes are shown in Scheme 1. Sodium sulfate, sodium carbonate and acetic acid, all of laboratory grade, were used.

2.2 Methods

2.2.1 Scouring and Bleaching

The grey cotton fabric was bleached using a combined scouring and bleaching method with the bath liquor containing 0.75% of the wetting agent sandozin NIE, 4.0% hydrogen peroxide and 2.5% sodium hydroxide at pH 10.5, liquor ratio 1:10, temperature 90°C, and 60 min. The test samples were then neutralized with 0.5g/L acetic acid and air dried.

2.2.2 Dyeing Procedure

Cotton fabrics were dyed with Reactive Dye 0.5- 4% (owf), Na₂SO₄ 10-50 g/L, sodium carbonate 5-20 g/L with liquor ratio 1: 20. The temperature was slowly raised to 60°C and dyeing was continued further for 1 h. Another sample was dyed with Reactive Dye 0.5- 4% (owf), Na₂SO₄ 10-50 g/L, sodium carbonate 5-20 g/L, having liquor ratio 1:5-1:20. Using microwave (Milestone Start synthesis Microwave Synthesis Lab station, USA), irradiation was done with power 150 W for 5-20 min. Then all the dyed samples were rinsed, and neutralized by acetic acid (1 g/L) and soaped (Ciba Pone R) followed by hot rinsing and air drying. Finally, the samples were dried and assessed for color strength and overall fastness properties.

2.3 Measurements and Analysis

2.3.1 Color Measurements

Color strength expressed as *K/S* was measured according to a previously reported method¹³ using light reflectance technique, and the relative color strength was calculated by applying the following Kubelka-Munk equation:

$$\text{Color strength } (K/S) = \frac{(1-R)^2}{2R} - \frac{(1-R^\circ)^2}{2R^\circ}$$

where *R* and *R*[°] are the decimal fraction of the reflectance of the colored and uncolored fabrics respectively; *K*, the absorption coefficient; and *S*, the scattering coefficient.

2.3.2 Fastness Properties

Fastness to washing, rubbing and perspiration was assessed according to the standard methods of AATCC technical manual, viz Method 8 (1989) 68, 23 (1993); Method 36 (1972) 68 (1993) 23; and Method 15 (1989) 68, (1993) 30 respectively.

3 Results and Discussion

3.1 Dyeing of Cotton Fabric Using Conventional Technique

In the dyeing with reactive dyes, the primary exhaustion occurs before addition of alkali, and then the secondary exhaustion occurs after addition of alkali. The rate of exhaustion can be increased by selecting dyes of high substantivity, and increasing the temperature, electrolyte concentration and dyeing time. The effect of dye concentration has been studied on the color strength of dyed cotton fabrics dyed with Solazol Red SP-3B using conventional heating technique at liquor ratio 1:20, dyeing temperature 60°C, dyeing time 100 min, Na₂SO₄ 8-50 g/L, and Na₂CO₃ 5-20 g/L. It is noticed that the increase in the dye concentration leads to increase in color strength. With the increase in dye concentration from 0.5% to 1, 2, 3 and 4% the *K/S* increases from 3.13 to 7.32, 12.59, 15.92 and 16.34 respectively.

Generally, the fastness properties to rubbing (wet and dry), washing, light and perspiration to both acidic and alkaline for the dyed samples range from very good to excellent.

3.2 Dyeing of Cotton Fabric using Microwave Irradiation Technique

3.2.1 Effect of Dyeing Time on *K/S*

Microwave-assisted organic synthesis^{14,15} has gained popularity in recent years, because microwave

irradiation is found to remarkably accelerate a wide variety of reactions. The effect of dyeing time has been studied on the color strength of dyed cotton fabrics with Solazol Red SP -3B using microwave irradiation. It is observed that the increase in dyeing time up to 10 min leads to increase in *K/S* and after that the increase in dyeing time leads to either constant *K/S* (0.5% shading) or decrease in the *K/S* (3% shading). Hence, the dyeing time of 10 min is recommended as optimum value. It is observed that the color strength ranges of dyeing cotton fabric using Solazol Red SP -3B and conventional technique (liquor ratio 1:20, temp. 60°C, dyeing time 100 min, Na₂SO₄ 8-50 g/L, Na₂CO₃ 5- 20 g/L and dye concentration 0.5- 3%) and that using Solazol Red SP -3B and microwave irradiation technique (liquor ratio 1:20, microwave power 150 W, dyeing time 10 min, Na₂SO₄ 8-50 g/L, Na₂CO₃ 5-20 g/L, dye concentration 0.5 & 3%) are 3.13 & 15.92 and 5.79 & 12.59 respectively. It is found that the microwave irradiation technique saves about 90 min which indicates saving in energy and power used for complete dyeing process.

The overall fastness properties to rubbing, washing, light and perspiration for the dyed samples range from very good to excellent.

3.2.2 Effect of Dye Concentration (Shading %) on *K/S*

The effect of dye concentration has been studied on the color strength of the dyed cotton fabrics upon using Solazol Red SP -3B and microwave irradiation. It is observed that the increase in dye conc. leads to increase in *K/S*. The increase in the dye conc. from 0.5 % to 1, 2, 3 and 4% shows the increase in *K/S* from 5.79 to 8.49, 11.18, 12.59 and 15.47 respectively.

The overall fastness properties to rubbing, washing, light and perspiration for the dyed samples range from very good to excellent

3.2.3 Effect of Dyeing Temperature on *K/S*

The higher temperature in dyeing with reactive dyes results in a higher rate of dyeing, better dye penetration, rapid diffusion, better leveling, and a higher risk of dye hydrolysis, lower colour yield, and lower substantivity. On studying the effect of dyeing temperature on the color strength of the dyed cotton fabrics upon using Solazol Red SP -3B and microwave irradiation, it is observed that the increase in dyeing temperature up to 60 °C leads to increase in *K/S* and after that the increase in temperature leads to decrease in *K/S*, irrespective of the dye conc. used. For dyeing temperature 40, 50, 60, 70 and 75 °C, the *K/S* values

are 8.48, 10.74, 12.59, 9.22 and 8.74 respectively upon using 3% dye conc. This may be due to the higher dye hydrolysis and hence the optimum dyeing temperature is recommended as 60 °C.

The overall fastness properties to rubbing, washing, light and perspiration for the dyed samples range from very good to excellent.

3.2.4 Effect of Alkali Concentration (Soda ash Na₂CO₃) on *K/S*

In the dyeing using reactive dyes, the increase in *pH* value by 1 unit corresponds to a temperature rise of 20 °C. The dyeing rate is best improved by raising the dyeing temperature when a *pH* of 11–12 is attained. Further increase in *pH* will reduce the reaction rate as well as the efficiency of fixation. Different types of alkalis, such as caustic soda, soda ash, sodium silicate or a combination of these alkalis, are used in order to attain the required dyeing *pH*. The choice of alkali usually depends upon the dye used, the dyeing method as well as other economic and technical factors. The effect of alkali concentration has been studied on the color strength of cotton fabrics dyed with Solazol Red SP-3B using microwave irradiation. It is observed that the increase in alkali concentration from 5 g/L to 20 g/L leads to increase in *K/S*, and after that the increase in alkali concentration to 25 g/L leads to slight increase in the *K/S*. For alkali concentrations 0, 5, 10, 15, 20 and 25 g/L, the *K/S* values are 5.79, 7.7, 10.9, 12.8, 13.1 and 13.6 respectively upon using 3% dye conc. This may be due to the increase in alkali concentration which increases the *pH* of the dyeing bath, i.e. dyeing rate is increased till it reaches to the best by raising the *pH* from 11 to 12 and after that the increase in *pH* beyond 12 leads to slight increase or decrease in *K/S* due to hydrolyses in the fixed reactive dye, and hence the optimum alkali concentration is recommended as 20 g/L.

The overall fastness properties to rubbing, washing, light and perspiration of the dyed samples range from very good to excellent. This is true at higher alkali concentration used, but at lower alkali concentration (0 - 5 g/L) the overall fastness properties to rubbing, washing and perspiration for the dyed samples range from moderate to good

3.2.5 Effect of Salt Concentration (Glauber salt Na₂SO₄) on *K/S*

The addition of electrolyte results in increase in the rate and extent of exhaustion, and increase in dye aggregation. There may be impurities present in the salt to be used, such as calcium sulphate, magnesium sulphate, iron, copper and alkalinity, that can be a

source of many dyeing problems. The effect of salt concentration has been studied on the color strength of the dyed cotton fabrics upon using Solazol Red SP-3B and microwave irradiation. It is observed that the increase in salt concentration from 20g/L to 40 g/L leads to increase in *K/S*, and after that the increase in salt concentration to 70 g/L leads to decrease in *K/S*. For the salt concentrations 0, 20, 40, 50 and 70 g/L the *K/S* values are 5.19, 9.7, 13.6, 13.1 and 12.9 respectively upon using 3% dye conc. This may be due to the increase in salt concentration which leads to increase in exhaustion of the dye from the dyeing bath into the fabrics, i.e. dyeing rate is

increased till it reaches to the best and after that the increase in the salt concentration more than 40 g/L leads to decrease in *K/S* due to hydrolyses in the fixed reactive dye by increasing the salt concentration. Hence, the optimum salt concentration is recommended as 40 g/L.

The overall fastness properties to rubbing, washing, light and perspiration for the dyed samples range from very good to excellent. This is true at higher salt concentration used, but at lower concentration (0 and 20 g/L), the overall fastness properties to rubbing, washing and perspiration for the dyed samples range from good to very good.

Table 1—Comparison between cotton fabrics dyed with Solazol Red SP-3B using microwave irradiation and/or the conventional heating on color strength and overall fastness properties

[Dye concentration 3%, temperature used 60°C, alkali used 20g/L]

Dyeing method	Time min	MLR	Power Watt	Salt g/L	<i>K/S</i>	Light fastness	Rub fastness		Wash fastness			Perspiration				
							Dry	Wet	St on cotton	St on wool	Alt	St.	Acidic		Alkaline	
													Alt	St.	Alt	St.
Dyeing using microwave irradiation	10	1:5	150	40	15.8	6	4-5	4	4-5	4	4-5	4-5	4-5	4-5	4-5	
Dyeing using conventional heating	100	1:20	-	50	15.9	6	4-5	4	4-5	3-4	4-5	4-5	4-5	4-5	4-5	
Dyeing using conventional heating	10	1:5	-	40	9.3	5	3-4	3	3	3	3-4	4	4	3-4	3-4	
Dyeing using microwave irradiation	10	1:20	150	50	13.1	5	4-5	4	4-5	4	4-5	4-5	4-5	4-5	4-5	

St.- Staining. Alt. - Alteration.

Table 2—Comparison between cotton fabrics dyed with Solazol Blue SP-BRF using microwave irradiation and/ or the conventional heating on color strength and the overall fastness properties

[Dye concentration 3%, temperature used 60°C, alkali used 20g/L]

Dyeing method	Time min	MLR	Power Watt	Salt g/L	<i>K/S</i>	Light fastness	Rub fastness		Wash fastness			Perspiration					
							Dry	Wet	St on cotton	St on wool	Alt	Alt.	St.	Acidic		Alkaline	
														Alt	St.	Alt	St.
Dyeing using microwave irradiation	10	1:5	150	50	12.4	6	4-5	4	4-5	4	4-5	4-5	4-5	4-5	4-5		
Dyeing using conventional heating	100	1:20	-	50	12.6	6	4-5	4	4-5	4	4-5	4-5	4-5	4-5	4-5		
Dyeing using conventional heating	10	1:5	-	50	7.1	5	3-4	3	3	3	3-4	4	3-4	3	3-4		
Dyeing using microwave irradiation	10	1:20	150	50	11.2	6	4-5	4	4-5	4	4-5	4-5	4-5	4-5	4-5		

St. - Staining. Alt. - Alteration.

3.2.6 Effect of Power of Microwave on K/S

The study on the effect of power of microwave on the color strength of cotton fabrics dyed with Solazol Red SP-3B shows that the increase in power from 100 W to 150 W leads to increase in K/S, and after that the increase in power to 200 W leads to decrease in K/S. This may be due to the increase in power of microwave to more than 150 W. This means that the increase in temperature or the increase in hydrolysis of the reactive dye gives lower K/S. Hence, the optimum value of power used is recommended as 150 W.

The overall fastness properties to rubbing, washing, light and perspiration for the dyed samples range from very good to excellent.

3.2.7 Effect of Liquor Ratio on K/S

The effect of liquor ratio has been studied on the color strength of cotton fabrics dyed with Solazol Red SP-3B using microwave irradiation. It is observed that the increase in liquor ratio from 1:5 to 1:40 leads to decrease in K/S. This may be due to increase in hydrolysis of the reactive dye. At lower liquor ratios, there is higher exhaustion and higher colour strength. For the liquor ratios 1:5, 1:10, 1:20, and 1:40, the K/S values are 15.8, 14.9, 13.6 and 12.9 respectively. Hence, the optimum value of liquor ratio used must be 1:5, which helps in saving water, salt and alkali.

The overall fastness properties to rubbing, washing, light and perspiration for the dyed samples range from very good to excellent

3.2.8 Comparison between Dyeing Techniques

Tables 1 and 2 show the comparison between dyeing of cotton fabric using microwave irradiation and conventional heating methods with regard to color strength and overall fastness properties using Solazol Red SP-3B and Blue SP-BRF respectively. It is found that the color strength and overall fastness properties of cotton fabric dyed with either Solazol Red SP-3B or Solazol Blue SP-BRF upon using microwave irradiation is better than the results obtained on using conventional heating. The K/S values are found to be 15.8 & 12.4 and 9.3 & 7.1 in case of microwave irradiation and conventional heating using Solazol Red SP-3B and Solazol Blue SP-BRF respectively. The overall fastness properties to rubbing, washing, light and perspiration for the dyed samples range from very good to excellent in case of microwave irradiation while their values range from good to very good in case of conventional heating. But with the increase in time of dyeing of the

conventional heating to 100 min the overall fastness is improved and ranges from very good to excellent.

3.2.9 Commercial Viability

From the results obtained, it is observed that the microwave technique saves 90 min in the dyeing time, 75% in the salt used and 20% in alkali used, indicating the saving in time, energy and money, irrespective of the dye used.

4 Conclusion

4.1 For better results, the power used in case of dyeing cotton fabric using exhaustion method should not exceed 150 Watt, the salt concentration should not increase beyond 40 g/L, and the alkali concentration should not increase more than 20 g/L.

4.2 The liquor ratio used in case of dyeing cotton fabric using exhaustion and microwave technique method must be 1:5 which saves water, salt and alkali.

4.3 The microwave technique saves ~ 90 min in the dyeing time, ~ 75% salt used and 20% alkali used, indicating saving in time, energy and money, irrespective of the dye used.

Acknowledgement

The authors gratefully acknowledge the funding support by Academy of Scientific Research and Technology, Cairo, Egypt under the project ID 3561.

References

- Schlaeppli F, *Text Chem Color*, 30(4) (1998)19.
- Chattopadhyay D P, *Indian J Fibre Text Res*, 26(3) (2001)108.
- El-Molla M M, Badawy N A, Abdel-Aal A Y, El-Bayaa A A & El-Shaimaa H M G, *Indian J Fibre Text Res* 36(2011)88
- Lewis D M & Lei X P, *Text Chem Color*, 21(10) (1989)23.
- Sekar N, *Colourage*, 46 (6) (1999)29.
- Burkinshaw S M, Lei X P & Lewis D M, *J Soc Dyers Colour*, 105 (1989)391.
- Burkinshaw S M, *J Soc Dyers Colour*, 106 (1990)307.
- Prasil M, *Proceedings International Textile Conference Texci- 2000* (Mönch Publishing Group Liberec, Gzech Republic), 2000, 448.
- Blackburn R S & Burkinshaw S M, *J Appl Polym Sci*, 89 (2003)1026.
- Liu L, Li Y & Fang Y, *Carbohydrate Polym*, 57 (2004) 97.
- Xie K, Liu Y & Li X, *Materials Chem Phys*, 105 (2007b)199.
- Ahmed K A, El-Molla M M, Abdel-Mottaleb M S A, Mohamed S Attia & El-Saadany S, *Res J Chem Sci*, 3(4) (2013) 3.
- Judd D B & Wyszenki G, *Color in Business, Science and Industry*, 3rd edn (John Wiley and Sons, New York), 1975.
- Biswal J, Kumar V, Bhardwaj Y K, Goel N K, Dubey K A & Chaudhari C V, *Radiation Phys Chem*, 76 (2007)1624.
- Takano T, Ishikawa J, Kamitakahara H & Nakatsubo F, *Carbohydrate Res*, 342 (2007) 2456.