



Study of Exhaustion of Direct Dyes under Optimized Conditions Spectrophotometrically

SHAZIA NAHEED¹, H.B. AHMAD^{2*}, KHURRAM SHAHZAD¹, MUHAMMAD ABDULLAH¹, MUHAMMAD ZUBER¹ and MAZHAR HUSSAIN²

¹Department of Chemistry, Government College University Faisalabad, Pakistan

²Department of Chemistry, Bahauddin Zakariya University Multan, Pakistan

*Corresponding author: E-mail: hafiz_badar@hotmail.com

(Received: 22 September 2010;

Accepted: 7 September 2011)

AJC-10365

CI Direct blue 67 and CI direct red 81 were studied for optimization of conditions in exhaust dyeing process by varying different parameters like salt concentration, time, temperature and liquor ratio. Glauber's salt ($\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$) or common salt (NaCl) is used as exhausting agent. The exhaustion of dye in dye bath was determined spectrophotometrically and its retention on fabric was tested on spectra flash (data colour 600). The dyeing results were evaluated by measuring colour yield, washing-fastness, rubbing-fastness and light-fastness. The best results were obtained when 4 g cotton fabric was dyed using 50 g/L $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$, at 95 °C, for 1 h, in liquor ratio 1:10. After treatment, a fixer (Tino-fixer) was necessary for the best results.

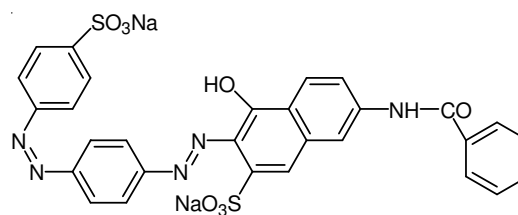
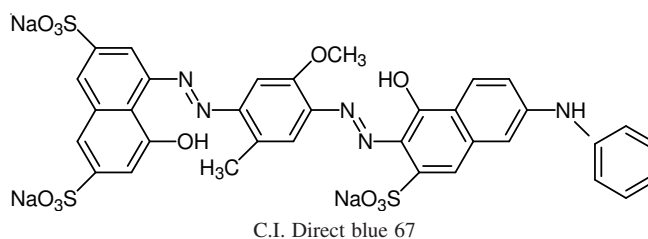
Key Words: Direct dyes, Exhaustion, Optimization.

INTRODUCTION

Dyes are normally water soluble or water-dispersible organic compounds that are capable of being absorbed into substrate. The dye molecules are usually chemically bonded to the surface and become a part of material on which they are applied¹. Direct dyes are colourants that are able to dye cellulose fibers without the need for a pre-treatment of the fibers with mordant². The first direct dye for cotton fibers was Congo Red which was discovered by Professor Paul Boettiger³. Despite their existence for over 100 years, direct dyes continue to be one of the most important groups of dyes for the textile industry. Since the development of new dyes, direct dyes are also used for dyeing cellulose fibres because of favourable characteristics such as intense colours, hue, bright shades, high substantivity, ease of synthesis and low cost⁴. The direct dyes derived from benzidine, owing to their excellent substantivity towards cellulose fibers, have gained considerable importance. However, due to carcinogenic behaviour, its use for the production of dyes was forbidden⁵. The majority of direct dyes are azo compounds, mostly bisazo and trisazo in nature having sulfonate group for making it water soluble which is important for dyeing. These dyes are normally applied in the form of their sodium salts. Other substituents that direct dyes contain are hydroxyl and amino groups. These electron-rich groups are capable of forming hydrogen-bond with the hydroxyl groups on cellulose fiber⁶.

EXPERIMENTAL

Direct dyes (C.I. direct blue 67 and C.I. direct red 81), cellulose fabric (cotton) and tinofix (formaldehyde based fixing agent) and oilified liquid soap were purchased from local market. Sodium sulphate, perchloric acid, sodium carbonate, sodium dihydrogen phosphate, potassium dihydrogen phosphate, 1-propanol were purchased from Merck laboratories Germany.



pH meter (WTW pH 525), perspirometer (James Heals and Co.), UV-Visible double beam spectrophotometer (Perkin-Elmer Lambda 20), spectra flash (Data Colour 600), digital crock meter (James Heals and Co.), glycerin bath high temperature beaker dyeing machine (H-T Dyeing Machine), electrical balance were used.

Exhaust dyeing: The dyeing of cotton fabric was carried out with the help of direct dyes by using exhaust dyeing process. To get 4 % shade, dye solution (16 mL, 1 % w/v), OLS solution (4 mL, 5 %) and distilled water (40 mL) were taken in a dye bath. Glauber's salt (30 g/L) was then added and a cotton fabric (4 g) was dipped in this dye bath and lid was closed correctly.

The dye bath was placed in H-T dyeing machine. The temperature was set at 45 °C for 15 min and then increased to 95 °C for 1 h. After 1 h, the dye bath was taken out from machine and cooled it under tap water. The dyed fabric was taken out from dye bath and after a cold wash it was dipped into fixer solution (50 mL, 50 %) for 0.5 h. Tino-fix (a formaldehyde based fixer) was used as fixer. The fabric was then cut into two pieces. One piece remained unwashed and other was washed to remove unfixed colour. Change in shade was noted and washed piece was preserved for further investigation.

Exhaustion and fixation: The degree of dye exhaustion (E) was determined spectrophotometrically, measuring dye concentrations in the bath before and after dyeing using eqn. 1.

$$E = \frac{A_1 - A_2}{A_1} \times 100 \quad (1)$$

where A_1 , A_2 are the absorbances of the dye solutions before and after dyeing, respectively⁷.

The extent of direct dye fixation (F %) was determined with the help of spectra flash (Data Colour 200) by using eqn. 2.

$$F (\%) = \frac{K/S_{\text{after DMF}}}{K/S_{\text{after soaping}}} \times 100 \quad (2)$$

This method assumes that K/S values are proportional to concentration of dye on fibre⁵.

Determination of fastness to rubbing and washing: The dyed fabrics were evaluated using a standard procedure *i.e.*, fastness to rubbing according to ISO 105-X12 and fastness to washing according to ISO 105-C01. The change in shade and staining of adjacent undyed fabrics were assessed using grey scales 1 (poor)-5 (excellent)⁸.

RESULTS AND DISCUSSION

To optimize direct dyeing on cotton fabrics different parameters like salt concentration, time, liquor ratio and temperature were varied. Optimization conditions were evaluated by exhaustion and fixation of dyes, rubbing and washing fastness.

Effect of salt concentration: In this study the salt concentrations 30, 50 and 70 g/L were used keeping the other parameters *i.e.*, temperature, time and liquor ratio constant. It has been observed that without adding salt, exhaustion was very low. It was improved by addition of salt. Low quantity of salt

(below 30 g/L) did not have considerable effect. At 50 g/L exhaustion was maximum and on further increase in concentration effect is not significant. The results are summarized in Figs. 1 and 2.

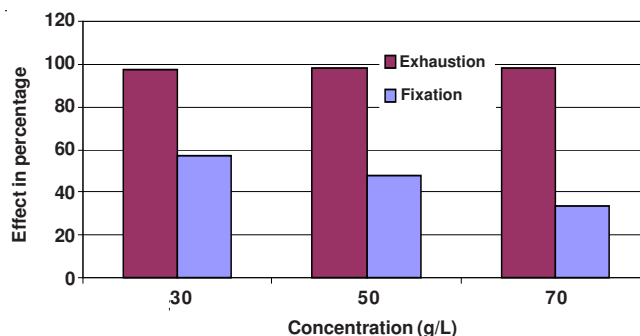


Fig. 1. Exhaustion and fixation of direct blue 67 in contrast with salt concentration

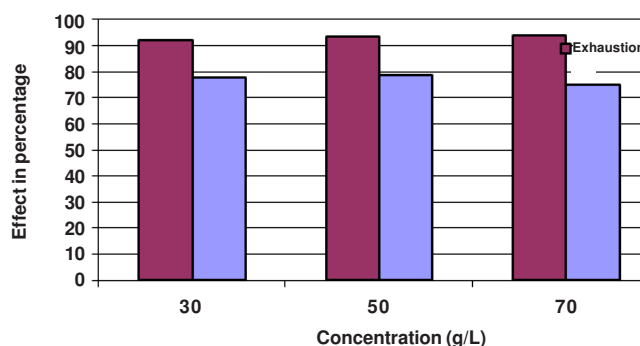


Fig. 2. Exhaustion and fixation of direct red 81 in contrast with salt concentration

Keeping in view of the Figs. 1 and 2, which indicated that 50 g/L is the best concentration of salt for direct dyeing.

Temperature effect: To determine the effect of temperature in exhaust dyeing process on direct dyes observations were made at 80, 95 and 110 °C. This study shows that the amount of dye taken by the material depends upon the temperature of the dye bath. The rate of dyeing increased with increase in temperature (Figs. 3 and 4). However uneven dyeing was observed at high temperatures. The best way for obtaining even dyeing, the dyeing process should be started at low temperature and it should be raised gradually.

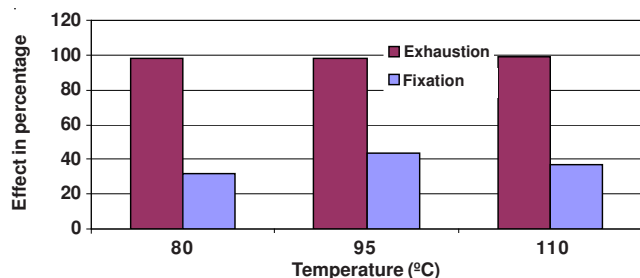


Fig. 3. Exhaustion and fixation of direct blue 67 in contrast with temperature

Time variation effect: To optimize time duration, dyeing period was continued for 45, 60 and 75 min. The time effect is vital for direct dyeing (Figs. 5 and 6). The dye molecules remain mainly on the surface of the fiber but when time is

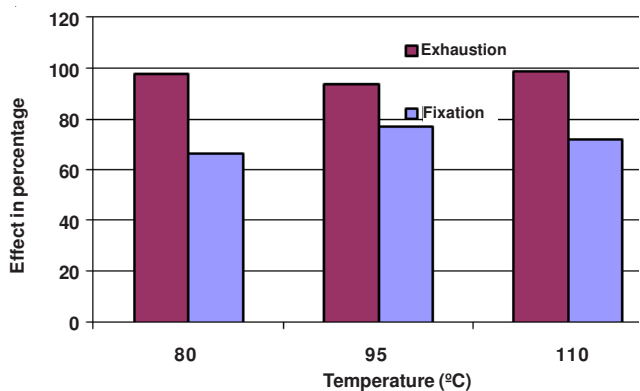


Fig. 4. Exhaustion and fixation of direct red 81 in contrast with temperature. The above graphs show that exhaustion and fixation of dye was maximum at 95 °C which may be the best temperature for direct dyeing

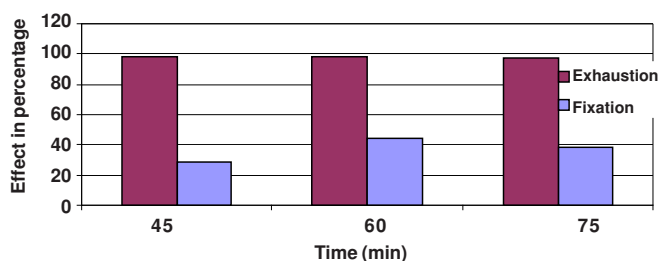


Fig. 5. Exhaustion and fixation of direct blue 67 in contrast with time

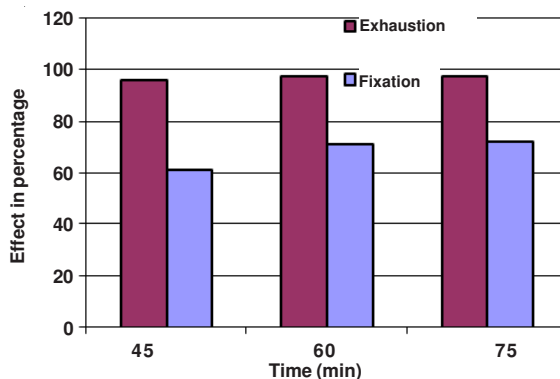


Fig. 6. Exhaustion and fixation of direct red 81 in contrast with time

sufficiently prolonged, they enter into the interior structure of the fiber. Figs. 5 and 6 indicated that 1 h is the best time period for direct dyeing because maximum fixation was obtained during this time.

Liquor ratio effect: To study liquor ratio effect the ratios used were 1:10, 1:15 and 1:20. It is observed that when liquor ratio was increased, the concentration of dye per liter is decreased and *vice versa* (Figs. 7 and 8). It was observed that best results obtained at liquor ratio 1:10.

Effect of fixer application: The common reasons of using direct dyes for cellulose fibers are their low cost and simplicity of applications. However these dyes are soluble in water, hence washing fastness of these dyes is not so good. Most of them faint in light. Therefore, direct dyes are undesirable for the materials which are frequently washed and dried in sunlight. Many after treatment methods have been developed to overcome these defects. Some of them increase the molecular weight of the dye while others make the dye less soluble in

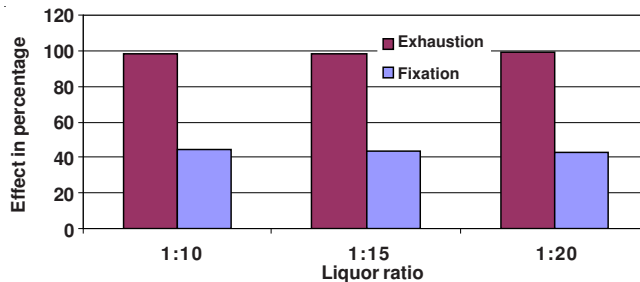


Fig. 7. Exhaustion and fixation of direct blue 67 in contrast with liquor ratio

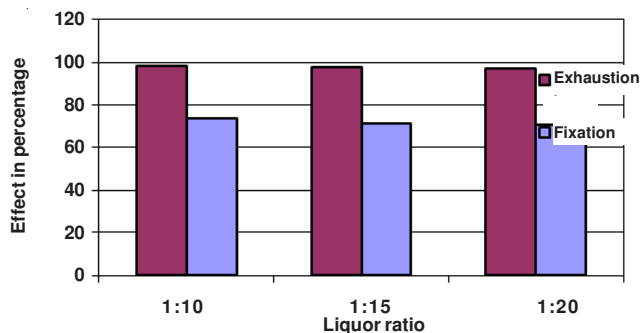


Fig. 8. Exhaustion and fixation of direct red 81 in contrast with liquor ratio

water. Increase in the size of dye molecule can be achieved by using formaldehyde based fixer (Tino-Fixer) after treatment with suitable dye. By this after treatment, washing fastness can be improved. Graphs show the comparison of exhaustion and fixation of dyes with and without fixer (Figs. 9 and 10). Formaldehyde is believed to link two molecules of dye through methylene group.

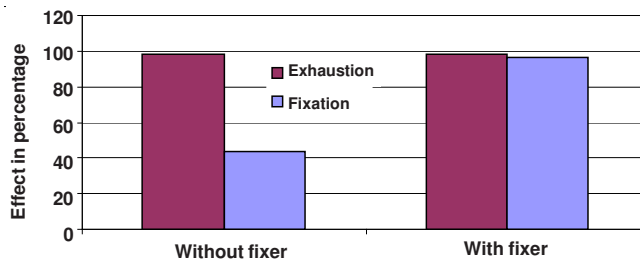


Fig. 9. Exhaustion and fixation of direct blue 67 in contrast with fixer application

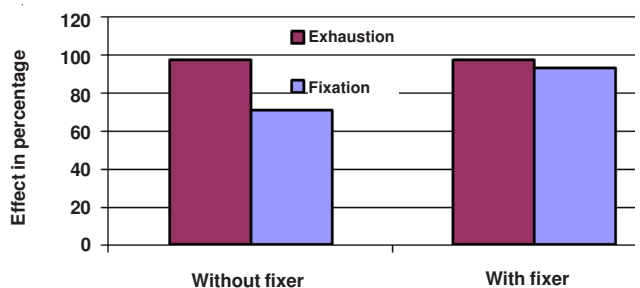


Fig. 10. Exhaustion and fixation of direct red 81 in contrast with fixer application

Conclusion

The present investigations were carried out to study the optimization conditions in exhaust dyeing process by using

direct dyes on cotton. Glauber's salt (Na_2SO_4) was used as exhausting agent, detergent (olified liquid soap) as wetting agent and Tino-Fixer as fixing agent. These investigations show that best results are obtained when 4 g cotton fabric is dyed using 50 g/L Na_2SO_4 , at 95 °C, for 1 h, in liquor ratio 1:10. After treatment with a fixer (Tino-fixer) is necessary for best results.

ACKNOWLEDGEMENTS

The authors wish to thank Hafiz Muhammad Tayyab, Manager, Sandal Dyestuff Industries, Faisalabad (Pakistan) for his guidance and support for this research work.

REFERENCES

1. A.M. Farhat, *Hand Book Ind. Chem.*, **8**, 260 (2000).
2. K. Sawada and M. Ueda, *Dyes Pigm.*, **58**, 37 (2003).
3. D.P. Steensma, *Arch. Pathol. Lab. Med.*, **125**, 250 (2001).
4. J. Bae and H.S. Freeman, *Dyes Pigm.*, **73**, 126 (2007).
5. R.M. El-Shishtawy, S.H. Nassar and N.S.E. Ahmed, *Dyes Pigm.*, **74**, 215 (2007).
6. C.V. Stead, *J. Chem. Technol. Biotechnol.*, **38**, 55 (1987).
7. K. Wojciechowski, A. Wyre bak and J. Gumula, *Dyes Pigm.*, **56**, 99 (2003).
8. Y. Li, W. Li, W. Zhang and D. Liu, *Dyes Pigm.*, **64**, 35 (2005).