

Novel Dyeing Methods for Dyeing Cotton and Polyester

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Introduction

The fibre-reactive dyes constitute one of the largest, fastest growing and the most important range of dyes for cellulosic materials because of their flexible application at a moderate cost, their brightness of shade and wide colour gamut and all-round good colour fastness properties [1]. The traditional methods for dyeing cotton with reactive dyes rely on two-stage process: firstly, exhaustion of dye in the presence of neutral salt and secondly, dye-fibre bond formation in the presence of alkali, which may cause some disadvantages such as the hydrolysis of dye, unlevelness and irreproducibility especially with very high reactivity dyes [2]. If these two-step dyeing method can be replaced by the dyeing processes that achieve the covalent-bonding without adding alkali, the following advantages may obtain: cheaper and simpler dyeing processes, reduced dye hydrolysis, simultaneous one-bath dyeing for blend fabrics containing cellulosic component [3]. The idea is to dye with pre-activated vinyl sulphone dyes so that the covalent bond formation occurs in a neutral medium.

Supercritical fluid dyeing (SFD) is one of the typical applications of supercritical fluids to polymer processes which have been studied since 1990s [4,5]. The conventional wet-dyeing method for polyester textiles should be replaced because it requires many dispersing agents and surfactants producing a large amount of hard-to-destroy little-biodegradable wastewater. The environmentally friendly supercritical fluid dyeing does not require any water, dispersing agents and surfactants in the dyeing process. It can also offer a method to save energy as it does not require any drying stage after dyeing. In this study the sorption amount of disperse dyes in the PET, PTT textile fibers were measured in a high-pressure sorption cell equipped with a magnetic pump.

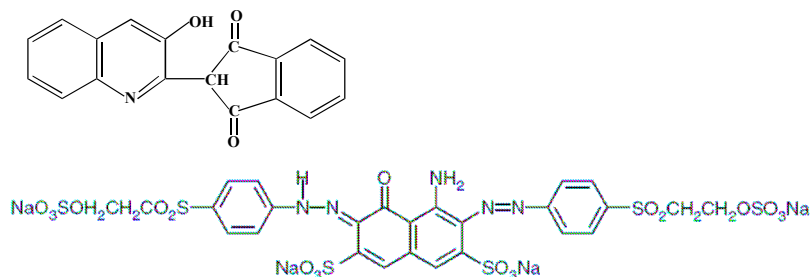
Experimental

Materials

Scoured, bleached and unmercerised cotton fabrics (Testfabric Inc. – USA (127 g.m⁻²)), PET (Hyosung Dyeing Co.) and PTT (Namseung Textile Co.) textiles were used. The commercial C. I Reactive Black 5 (RBB) which contains sulphatoethylsulphone (SES) groups from Dystar for cotton, C. I. Disperse Yellow 54 (Y54), disperse dye from LG Chemical Co. (Figure 1) for PET and PTT were employed. Carbon dioxide (99%) was obtained from Daedong Oxygen Co. All other chemicals used in the experiments were obtained as laboratory grade materials without any further purification.

Procedures

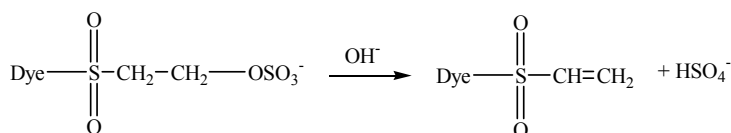
Remazol dyes, which usually contain a sulphatoethylsulphone group, were pre-activated to the vinylsulphone (VS) form prior to dyeing cotton by adding 0.5M Na₂CO₃ solution to an aqueous solution of dye to raise pH value to 11. The temperature was maintained at 20°C in the water bath for 90 minutes; in this way the SES group was activated and the VS reactive group generated, as shown in Scheme 1. The course of the activation of C. I. Reactive Black B was followed by micellar electrokinetic capillary chromatography.



C. I. Disperse Yellow 54

C.I. Reactive Black 5

Figure 1. The structure of dyes used in this study.



Scheme 1. Activation of sulphatoethylsulphone group.

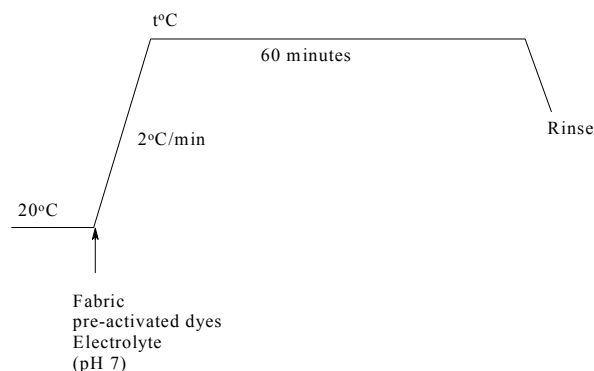


Figure 2. Temperature/time profile for applying pre-activated dyes under neutral conditions

After the activation, the pre-activated dyes were used for dyeing unmodified cotton in 100 cm³ sealed stainless steel dye pots, housed in the Mathis Labomat type BFA laboratory dyeing machine (Werner Mathis AG, Switzerland). The dyebaths contained the pre-activated dyes and various concentrations of auxiliaries at different liquor to goods ratios. For neutral fixation, the dyeing solution was adjusted to pH 7 by 1M HCl solution, and the bath raised to the selected temperature at 2°C.min⁻¹, hold at that temperature for 1 hour (Figure2). Na₂SO₄ was used as electrolyte.

A closed-loop high-pressure sorption apparatus circulated by a magnetic pump was placed in a constant temperature air bath controlled within ±1 K. A small amount of Y54 dye-powder was placed in one part (upstream side) of a stainless steel dye reservoir with both sides plugged with glass wool to prevent the dye powder from being entrained. Textile samples, separated by thin wire screen to prevent direct contact of samples, were then packed in the other part (downstream side) of the tube. After evacuation carbon dioxide was introduced into the system and was circulated in the loop with a magnetic pump. When the piston jumps up and down continuously, the fluid was circulated in the loop. One set of Sensotec TJE pressure transducer and Sensotec GM signal conditioner/ indicator reads the pressure to ±14 kPa. The

samples were taken out from the cell 500 minutes and the dye sorbed on the sample was extracted with chlorobenzene at its boiling point.

Results and Discussion

Figure 3a shows that when increasing the temperature, the dye exhaustion was decreased due to the hydrolysis of the dyes at the high temperatures employed. Although with increasing temperature there were reductions in dye exhaustion values, the absorbed dye fixation values were improved, leading to the increase in total fixation efficiency. Figure 3b indicates that with this pre-activated dye, maximum colour yield was obtained if the bath temperature raised at least 90°C.

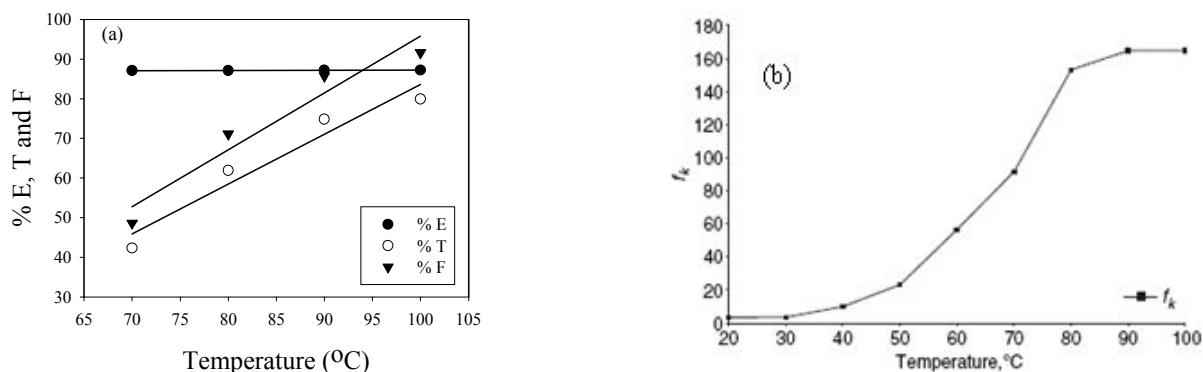


Figure 3. (a) Dye exhaustion, fixation and total fixation efficiency and (b) Colour intensity of the sample dyed with C. I. Reactive Black B (VS form) at different temperatures under neutral condition (80 g.dm^{-3} of Na_2SO_4 , liquor ratio 10:1 for 1 hour).

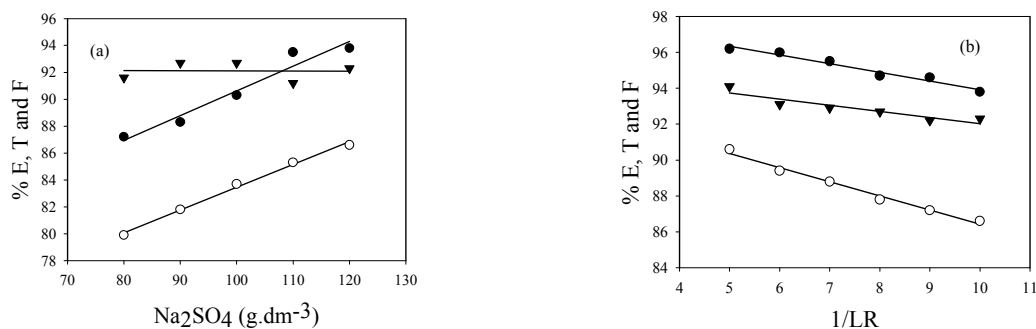


Figure 4. Dye exhaustion, fixation and total fixation efficiency values for C. I. Reactive Black B dyed at (a) different concentrations of sodium sulphate and (b) at different liquor ratios (at 100°C and liquor ratio 10:1).

It can be seen from Figure 4a that the dye exhaustion, fixation and total fixation efficiency values increased when the concentration of sodium sulphate were increased. Sodium sulphate salt plays a very important role in this neutral dyeing process; the addition of large amounts of neutral salts to the dyebath is necessary to attain high fixation.

Small increases in dye exhaustion, absorbed dye fixation and total fixation efficiency values were observed as the liquor ratio decreased (Figure 4b). Changing the liquor ratio influences the equilibrium between dye absorption and desorption, lower liquor ratio favouring absorption hence more opportunity for dyes to react with the fabrics. Even dyeing at liquor ratio as low as 5:1 this new dyeing method still

produces dyeings of good levelness and penetration due to the fact that all the chemicals used as dyes, electrolyte, and fabric can be added into the dyeing solution at the beginning of the dyeing process, and also dyeing was carried out at the boil which maximises dye solubility and migration.

PET and PTT fibers were easily dyed with Y54. The amount of sorption of Y54 dye in the crystalline polyester textile fibers increased with increasing pressure up to 33 MPa at various temperatures (Figures 5). The amount of sorption for PTT was (about 10%) larger than that for PET because PET has a high degree of crystallinity of 30% which is larger than that of PTT.

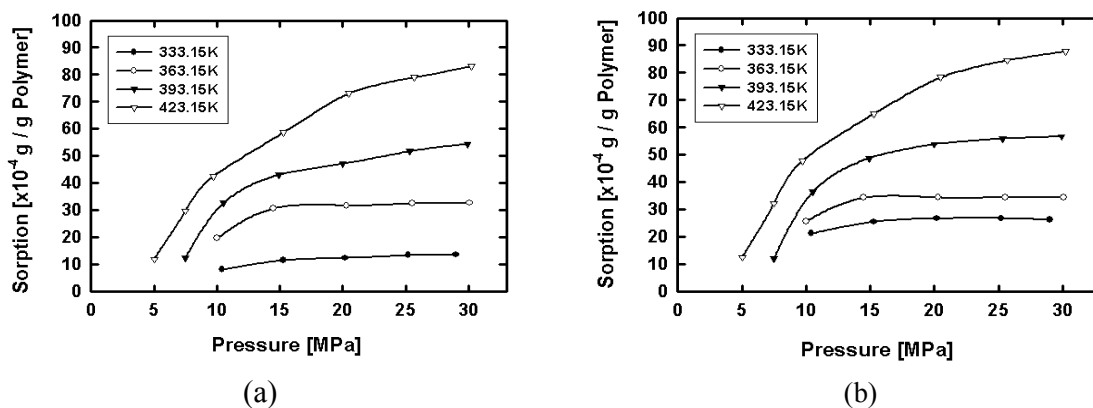


Figure 5. Experimental sorptions of Y54 dye in (a) PET textile and (b) PTT textile in the presence of supercritical carbon dioxide at different pressures and holding temperatures.

Conclusion

The concept of neutral fixation of the pre-activated reactive dyes has been introduced, giving fixation efficiency values comparable with the conventional two-step salt/alkali method. Although in this new process temperature and electrolyte concentration are higher than in the conventional method, using a short liquor ratio (5:1) reduces the use of electrolyte and the amount of energy required to heat the dyebath as well as the water consumption. This method also offers the opportunities for dyer dyeing blends, such as cotton/polyester blends, with simpler dye selection.

Sorption of Y54 dye in PET and PTT textile in the presence of supercritical carbon dioxide increased monotonically with pressure at the same temperature and increased with temperature at the same pressure. The increasing rate was reduced with increasing pressure.

Acknowledgment

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References

1. Rattee, I.D. and Breuer, M.M., *Fibre - reactive Dyes. The Physical Chemistry of Dye Adsorption*. London, New York: Academic Press, **1974**.
2. Beech, W.F., *Fibre-reactive Dyes*. London: Logos Press Limited, 1970.
3. English, R.J. and Lewis, D.M., *Reactive Dyes and Dyeing: A Critical Review*. In *Colour Science' 98*. University of Leeds: Department of Colour Chemistry. **1998**.
4. Saus, W., Knittel, D., Schollmeyer, E., *Textile Res. J.*, Vol. 63, No. 3, **1993**, p.135.
5. Chang, K.-H., Bae, H.-K., Shim, J.-J., *Korean J. Chem. Eng.*, Vol. 13, No. 3, **1996**, p.310.