J. of Supercritical Fluids 86 (2014) 108–114

Contents lists available at ScienceDirect

The Journal of Supercritical Fluids

journal homepage: www.elsevier.com/locate/supflu

Synthesis of reactive disperse dyes containing halogenated acetamide group for dyeing cotton fabric in supercritical carbon dioxide

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ARTICLE INFO

Article history:
Received 9 October 2013
Received in revised form 1 December 2013
Accepted 2 December 2013

Keywords:
Synthesis
Halogenated acetamide dyes
Reactive disperse dyes
Supercritical carbon dioxide
Dyeing
Cotton fabric

ABSTRACT

A series of reactive disperse dyes incorporating halogenated acetamide group were synthesized and applied to dye cotton fabric in supercritical carbon dioxide ($scCO_2$). Dyeing experiments were conducted in $scCO_2$ with dye concentration of 0.5% owf (% on weight of cotton fabric), varying from 80 to 120 °C, for 1–3 h at a constant pressure of 200 bar. The results showed that the color strength of dyed cotton fabric increased favorably when increasing temperature and time. The color characteristics were studied as well in terms of the reflectance spectra. And the color fastness to washing and rubbing were also reasonably good.

1. Introduction

The supercritical carbon dioxide ($scCO_2$) is a natural and innocuous fluid. Replacing organic solvent or water by $scCO_2$ is very advantageous to environmental protection [1,2]. Supercritical carbon dioxide dyeing, which was introduced at the first time in textile dyeing as an alternative to traditional water bath by E. Schollmeyer et al. in 1988 [3], has been worldwide investigated. Synthetic fibres, such as polyester, have been successfully dyed in $scCO_2$ [4–8] to meet an industrial requirement. However, the real challenge of supercritical carbon dioxide dyeing is connected to the coloration of natural textiles, especially the cotton dyeing. Since 37% of the world market are represented by cotton [9], developing methods for dyeing cotton is still now significant.

In order to achieve a good dyeing performance for cotton fibers, one of the concepts is reactive disperse dye that is to modify CO_2-soluble disperse dyes by reactive groups which are able to react with the cotton fibers with the formation of a chemical bond. The reactive disperse dyes are not only have to provide sufficient dye solubility in supercritical carbon dioxide, but also supply possibility of permanent dyestuff fixation on fiber and improved color fastness properties. So far many different reactive groups (sulphonylazide, triazine, bromoacrylic acid, and vinylsulphone) [10–13] on reactive disperse dyes have been examined. But in the last decade, although a lot of work has been focused on trying to achieve good coloration of cotton, no specific reactive functional groups was able to guarantee acceptable results, reviewed by Bach and co-workers [14].

Our research presented report a dyeing method for cotton fabric in supercritical carbon dioxide with reactive disperse dyes containing halogenated acetamide as reactive group, and cotton fabric was colored in one step process without any pre-treatment. The color fastness (washing and rubbing), color strength ($(K/S)_{dyed}$ and $(K/S)_{extracted}$), and fixation efficiency of dyed cotton fabric were investigated.

2. Experimental

2.1. Materials

All reagents was commercially purchased, and not further purified. The carbon dioxide gas (99.6 vol.%) obtained from Fuzhou Huaxinda Industrial Gases Co., Ltd was used for supercritical carbon dioxide dyeing of the cotton fabric. Cotton fabric (20.0 g of the fabric sample with a dimension of about 10.0 cm × 150.0 cm) used in this study was obtained from Shandong Weiqiao Pioneering Group Co., Ltd. The reactive disperse dyes used in this study were synthesized at our laboratory. Their structures are shown in Table 1.

2.2. Dyeing apparatus

The our own assembly apparatus of supercritical carbon dioxide to dye cotton fabric contains systems of pressurization, temperature control, dyeing, and carbon dioxide gas circulation, and recycle

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http://dx.doi.org/10.1016/j.supflu.2013.12.006

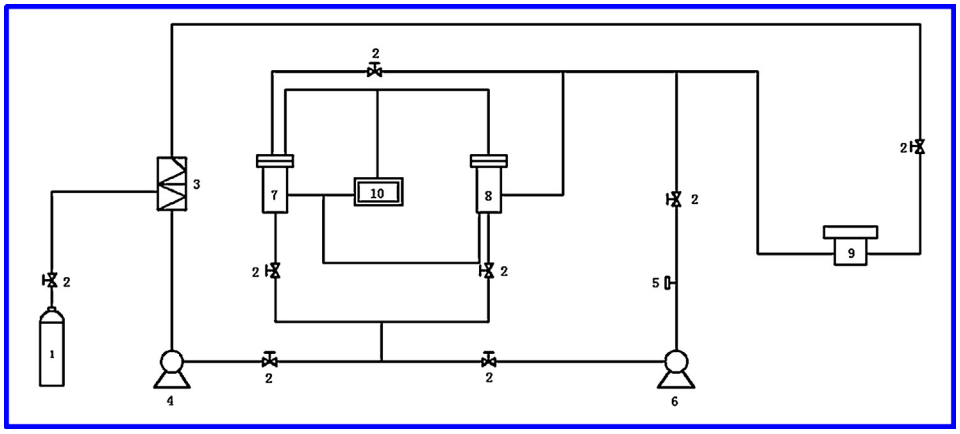


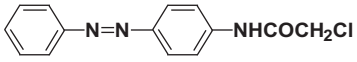
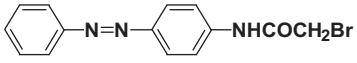
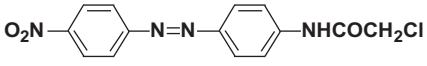
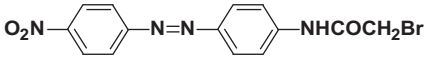
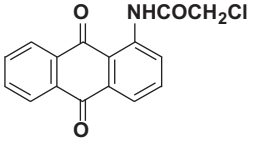
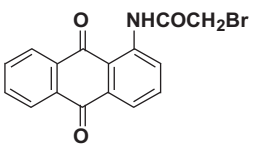
Fig. 1. Dyeing apparatus: (1) CO_2 cylinder, (2) shut-off valve, (3) cooling unit, (4) high-pressure syringe pump, (5) flow gauge, (6) CO_2 circulating pump, (7), (8) dyeing autoclave, (9) segregator, and (10) main controller.

sections. Fig. 1 is a diagram of the whole apparatus. The CO_2 leaving from a cylinder is passed through a cooling unit and introduces into a high-pressure syringe pump, and the high-pressure CO_2 eventually flows into the dyeing autoclave. The dyeing section is a stainless steel autoclave of 500 cm^3 volume equipped with a stainless steel shaft which is full of holes, as shown in Fig. 2. The dyes are put into a groove of the shaft top, together with cotton fabric wrapped around this shaft. The pressure and temperature of the dyeing process are monitored by main controller in a cabinet. Moreover, CO_2 gas is circulated by circulating pump carrying and its flow velocity and volume were recorded by a flow gauge. At the end of dyeing, the residual dyes and CO_2 are separated by segregator, then, the CO_2 is recycled.

2.3. Dye synthesis

The synthesis procedure is shown in Scheme 1.

Table 1
Molecular structure of the reactive disperse dyes.

	Molecular Structure	Name in the text
1		Dye1
2		Dye2
3		Dye3
4		Dye4
5		Dye5
6		Dye6

To a vigorously stirred solution of 4-Phenylazoaniline (1a, 0.02 mol), 4-Nitrophenylazoaniline (1b, 0.02 mol) respective and triethylamine (0.02 mol) in an appropriate dried solvent (CH_2Cl_2, 150 ml) was added a solution of chloroacetyl chloride or bromoacetyl chloride (2, 0.02 mol) in the same solvent (50 ml) at 0 °C. The reaction mixture was stirred at room temperature for 4–5 h. Then the reaction mixture was concentrated with decompress distillation and then was added 5% aqueous $NaHCO_3$ solution. The precipitate obtained was filtered, washed with water, and dried to produce the final products (3a, 3b) that were dye1 of saffron yellow powder, dye2 of red powder, dye3 of orange powder, dye4 of dark red powder respectively.

A similar procedure, the solution of 1-amino-9,10-anthraquinone (1c, 0.02 mol) and triethylamine (0.02 mol) in an appropriate dried solvent *N*,*N*-dimethylformamide (DMF, 80 ml) was added a solution of chloroacetyl chloride or bromoacetyl chloride (2, 0.02 mol) in the same solvent (40 ml) at 0 °C. The reaction mixture was vigorously stirred at room temperature for 4–5 h. The precipitate was obtained by adding 400 ml water,

Fig. 2. Photograph of the dyeing autoclave and stainless steel shaft.