Development auxiliaries for dyeing polyester with disperse dyes at low temperatures

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Development auxiliaries for dyeing polyester with disperse dyes at low temperatures

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Abstract. High-molecular weight organic compounds known as carriers are widely used to expedite polyester dyeing at atmospheric pressure at 100 ºC. However, carriers are usually poorly biodegradable and can partially plasticize fibres. Also, dyeing at temperatures above 100 ºC in the absence of a carrier entails using expensive equipment. In this work, we developed an alternative method for dyeing polyester at temperatures below 100 ºC that reduces energy expenses, dispenses with the need to invest in new equipment and avoids the undesirable effects of non-biodegradable carriers. The method uses disperse dyes in a micro-emulsion containing a low proportion of a non-toxic organic solvent and either of two alternative development auxiliaries (coumarin and *o*-vanillin) that is prepared with the aid of ultrasound.

1. Introduction

PET fibres are hydrophobic [1] and contain no chemically active groups, which hinders their dyeing in aqueous media [2] and requires using disperse dyes that are almost insoluble in water [3,4] . In fact, dyeing polyester typically entails dispersing a swelling agent (a carrier) and the dye among fibres at a high temperature and pressure [5] Efficiently using disperse dyes with polyester fibres entails operating at temperatures above their glass transition point (80 ºC) [6–8] Some organic solvents used as carriers to swell polyester fibres for dyeing contain phenyl-phenol, phenyl-chloride or phenyl-alkyl groups, among others [9–11]´ As noted earlier, carriers are often scarcely biodegradable and tend to plasticize fibres, thereby lowering their glass transition temperature (T_g) [12] .

Carrión-Fite [7] developed an alternative method for dyeing polyester with reduced energy costs, investments and unwanted effects [13]. The method uses temperatures as low as 40 ºC or even lower in combination with disperse dyes in a micro-emulsion containing a low proportion of an organic solvent (an alkyl halogen) and phosphoglyceride as emulsifier that is prepared under sonication [7].

Growing ecological concerns have led to strict regulation of textile dyeing and finishing processes [14], which has promoted a search for natural, safer substances for use by the wet textile processing industry [7, 14, 15]. For example, Pasquet et al [15] used *o*- and *p*-vanillin at high concentrations as alternatives for toxic carriers in the low-temperature dyeing of polyester fabrics with low-molecular weight disperse dyes in the presence of ethanol as co-solvent.

In this work, we developed an alternative method for dyeing polyester with disperse dyes at a low temperature. The method uses a micro-emulsion consisting of a non-toxic organic co-solvent *n*-butyl acetate, which was previously used by Carrion-Fite [15] and either of two agro-sourced development

auxiliaries[*o*-vanillin, which was previously employed by Pasquet et al [16], or coumarin. The microemulsion is prepared with the aid of ultrasound. The dyeing process was conducted by using various low-molecular weight disperse dyes at different temperatures below 100 ºC and its kinetics at each temperature examined.

2. Experimental

2.1. Materials

Fabric. Standard polyester, Type 30 A (Code 30,000), from wfk Testgewebe GmbH (Brüggen, Germany) (ISO 105-F10).

Chemicals. The low-molecular weight disperse dyes Rubin Foron E-5R (C.I. Disperse Red 73), and Blue Foron E-BL (C.I. Disperse Blue 56), both supplied by Clariant (Muttenz, Switzerland) (see Figs 1 and 2).

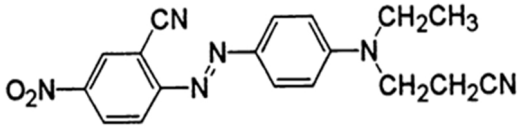

Figure 1. Structure of C.I. Disperse Red 73 (C.I. 11116, CAS 16889104, MW = 348.36 g·mol^{-1})

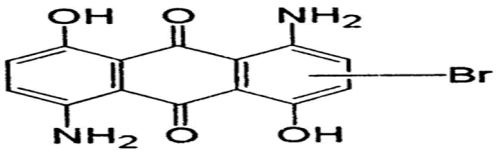

Figure 2. Structure of (C.I. Disperse Blue 56, C.I. 63285, CAS 12217797, MW = 365.18 g·mol^{-1})

Organic solvent. Pure *n*-butyl acetate (MW = 116.16 g·mol^{-1}) supplied by Panreac (Barcelona, Spain).

Auxiliaries. Coumarin 99% pure (MW = 146.15, CAS No. 91-64-5) supplied Acros (New Jersey,USA**)** and *o*-vanillin (2-hydroxy-3-methoxybenzaldehyde, MW = 152.15, CAS No. 148-53-8) in 99% purity supplied by Acros (New Jersey, USA**)**.

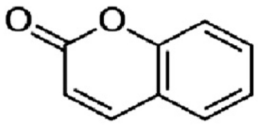

Figure 3. Chemical structure of coumarin.

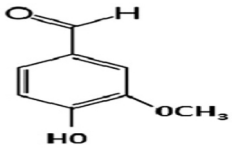

Figure 4. Chemical structure of *o*-vanillin.

Solvent for extraction of disperse dyes on polyester. *N*,*N*-Dimethylformamide for analysis (MW = 73.10 g·mol^{-1}) supplied in 99.8% purity by Panreac (Barcelona, Spain).

Non-ionic surfactant for pre-dyeing washing. Hostapal, obtained from Archroma GmbH (Sulzbach, Germany).

Post-dyeing reductant. QP-grade 85% pure sodium dithionite (MW = 174.11 g·mol^{-1}) and analytical-grade sodium hydroxide purissimum, both supplied by Panreac.

2.2. Equipment

Dyeing. A Linitest dyeing apparatus furnished with 300 ml sealed cans that was purchased from Atlas MTT GmbH (Germany)

Washing. A Launder-Ometer with 550 ml sealed cans also purchased from Atlas (Illinois, USA).

Spectrophotometry. A model M 330 UV–Visible spectrophotometer from Camspec, Ltd. (Cambridge, UK).

Sonication. A Labsonic 1510 shaker equipped with a standard probe of 19 mm Ø that was supplied by B. Braun (Hessen, Germany).

2.3. Procedures

2.3.1 Fabric pre-washing. Cleanliness in the standard polyester fabric used was ensured by washing with Hostapal detergent at a 0.5 $g \cdot L^{-1}$ concentration at 40 ºC for 30 min.

2.3.2 Dyeing conditions. The dyeing bath contained 3 g of polyester fabric, a 2% concentration of disperse dye, a 1 ml/1 g o.w.f. concentration of solvent (*n*-butyl acetate) and a 4% o.w.f. concentration of auxiliary. The bath ratio was 1:60 and the temperature 65, 75, 83 or 95 ºC. The dyeing apparatus was used with closed containers at a constant agitation rate and temperature. The operation time was 120 min —by exception, that used at the lowest temperature (65 ºC) was 150 min.

2.3.3 Dyeing procedure. Each dyeing bath was prepared from a 4% o.w.f. concentration of auxiliary (*o*-vanillin or coumarin) that was dispersed in 3 ml of *n*-butyl acetate and made to 180 ml (B.R: 1:60) with distilled water. Emulsification was facilitated by mechanical stirring with a propeller shaker, followed by ultrasound (2 w/ml, 1 min). The auxiliary emulsion contained a 2% o.w.f. concentration of disperse dye —previously dissolved in the solvent— in the required volume for the applicable bath ratio (180 ml). Each emulsion was warmed to the present temperature for each assay, which was kept constant throughout the dyeing process. The temperatures used were 65, 75, 83 and 95 °C. One-half of all tests used *o*-vanillin and the other half coumarin as auxiliary.

2.3.4 Post-dyeing washing. After dyeing, the fabric was washed in a reductive medium to remove all dye absorbed onto fibre surfaces. The reduction washing process consisted of 0.5 g/l soda and 2 g/l sodium hydrosulphite in distilled water and was followed by washing at 50 °C for 30 min. The bath ratio was 1:50 (150 ml per sample). The equipment used to wash dyed fabric was a Launder-Ometer with 550 ml cans for each specimen. An amount of 3 g of specimen was washed in a 150 ml wash bath under the above-described conditions. After, the wash fastness of a dyed fabrics during 120 min was obtained.

2.3.5 Determination dye absorption. The amount of dye absorbed by the polyester fabric was determined by extraction with *N*,*N*-dimethylformamide (DMF) and calculated from a linear line for the calibration of each disperse dye. As per the Beer–Lambert law, the absorbance at the maximum wavelength in the visible spectrum was a linear function of the concentration of dye dispersed in DMF.

3. Result and discussion

3.1 Dyeing kinetics and rate constant

Figures 5–8 illustrate the kinetics of dyeing with the two disperse dyes in the presence of the two auxiliaries used, namely: C.I. Disperse Red 73 with coumarin (Figure. 5), C.I. Disperse Red 73 with *o*-vanillin (Figure 6), C.I. Disperse Blue 56 with coumarin (Figure. 7) and C.I. Disperse Blue 56 with *o*-vanillin (Figure. 8).

The diffusion of dyes into fibres depends on the local rate of dye transport in the substrate in relation to the concentration gradient, the geometry of the medium, and the radius of the fibre cross-section. The difficulty of measuring these variables led us to use the following Cegarra-Puente kinetic equation [8]:

$$L\left(1-\frac{C_t^2}{C_\infty^2}\right) = -\mathrm{K}t \qquad (1)$$

Where
Ct = dye concentration on fibres at time t
C_∞ = initial concentration in the dye bath

K = dyeing rate constant
t = time

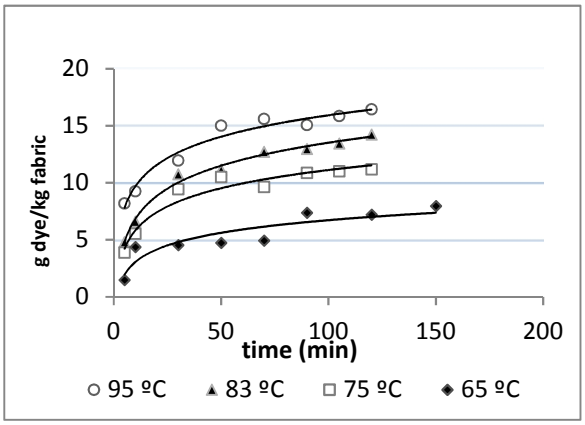

Figure 5 Kinetics of dyeing with C.I. Disperse Red 73 absorbed by polyester fabric at different temperatures in the presence of butyl acetate (16.666 g·l^{-1}) and coumarin (0.666 g·l^{-1}).

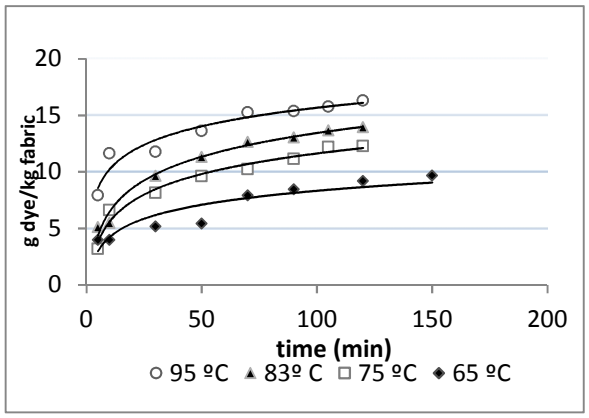

Figure 6 Kinetics of dyeing with C.I. Disperse Red 73 absorbed by polyester fabric at different temperatures in the presence of butyl acetate (16.666 g·l^{-1}) and *o*-vanillin (0.666 g·l^{-1}).

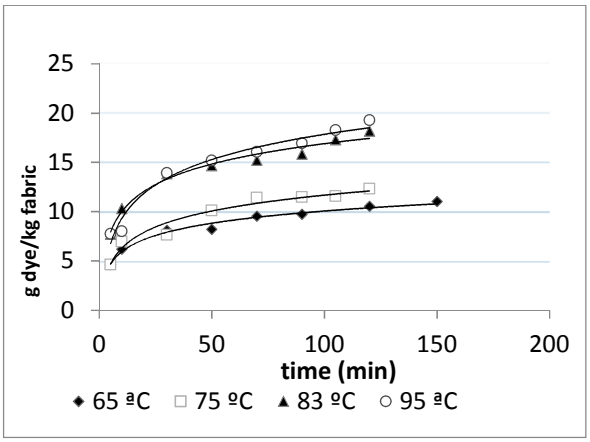

Figure 7 Kinetics of dyeing with C.I. Disperse Blue 56 absorbed by polyester fabric at different temperatures in the presence of butyl acetate (16.666 g·l^{-1}) and coumarin (0.666 g·l^{-1}).

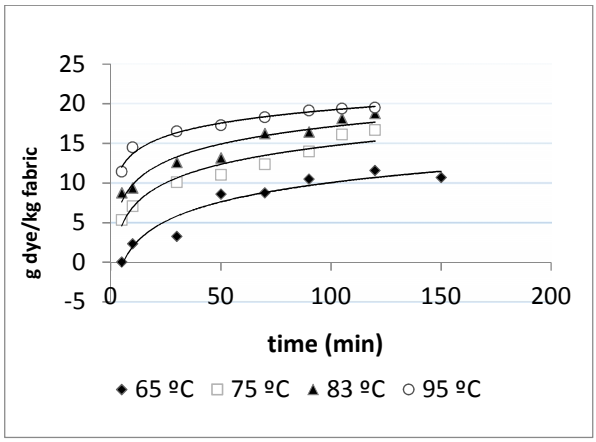

Figure 8 Kinetics of dyeing with C.I. Disperse Blue 56 absorbed by polyester fabric at different temperatures in the presence of butyl acetate (16.666 g·l^{-1}) and *o*-vanillin (0.666 g·l^{-1}).

Table 1 shows the dyeing absorption rate constants, and their respective correlation coefficients, as calculated from equation (1).

Table 1. Absorption rate constant (K) and correlation coefficient (r) for the kinetics of dyeing of polyester with disperse dyes in the presence of *o*-vanillin or coumarin as development auxiliary.

Disperse dyes	Auxiliary agent: o Vanillin			Auxiliary agent: Coumarin		
	Rate constant	Correlation coefficient	Exhaustion % after 120 min	Rate constant	Correlation coefficient	Exhaustion % after120 min
C.I. Disperse Red 73 at 95ºC	0.0073	0.9476	81.68	0.0078	0.8953	82.26
I. Disperse Red 73 at 83 ºC	0.0054	0.9672	69.88	0.0052	0.9491	71.06
C.I. Disperse Red 73 at75 ºC	0.0037	0.9723	61.47	0.0027	0.7989	55.84
C.I. Disperse Red 73 at 65 ºC	0.0017	0.9568	48.53	0.0011	0.88	39.76
C.I. Disperse Blue 56 at 95ºC	0.0221	0.9874	97.51	0.0183	0.912	96.31
C.I. Disperse Blue 56 at 83 ºC	0.0155	0.9316	93.85	0.0116	0.9355	90.81
C.I. Disperse Blue 56 at75 ºC	0.085	0.9455	83.32	0.0035	0.9358	61.62
C.I. Disperse Blue 56 at65 ºC	0.0027	0.8154	58.43	0.002	0.9437	55.06

As can be seen, the absorption rate constant for both dyes increased with increasing temperature. Also, the correlation coefficients were good (in general, higher than 0.94 with *o*-vanillin and somewhat lower but still exceeding, in general, 0.91 with coumarin). Exhaustion after dyeing was acceptable in all cases except below 75 ºC — this temperature is lower than glass transition temperature of polyester.

3.2 Wash fastness of polyester fabric dyed

Table 2 shows colour fastness of to domestic and commercial laundering according to the standard methodology and using the A2S test indicated in this standard [17].

Table 2 Colour fastness of the polyester dyed

PET fabric dyed 120 minutes	Auxiliary : O_Vanillin			Auxiliary : Coumarin		
	Gray Scale	PET	Cotton	Gray Scale	PET	Cotton
C.I. Disperse Red 73 at 65ºC	2	3	3	3-4	3-4	3
C.I. Disperse Red 73 at 75 ºC	3	3	3	4-5	3-4	3-4
C.I. Disperse Red 73 at 83 ºC	4	3	4	3	4	4-5
C.I. Disperse Red 73 at 95 ºC	4-5	4	4-5	5	4-5	4
C.I. Disperse Blue 56 at 65 ºC	2-3	3	2-3	3	3	2-3
C.I. Disperse Blue 56 at 75 ºC	4-5	3	3	4	3-4	2-3
C.I. Disperse Blue 56 at 83 ºC	4-5	4	3	3	4	3-4
C.I. Disperse Blue 56 at 95 ºC	4	4-5	3-4	4	4-5	4

Generally, in respect to Table 2 dyed fabric the washing fastness is acceptable for all samples and their values are quite similar for both dyes and slightly better in coumarin than o-vanillin agent. Staining of

the white polyester fabric is regular for all dyes and auxiliaries tested. In respect to the white cotton fabric values are almost similar and shows the regular level.

4. Conclusions

This paper reports a new method for dyeing polyester fabric with disperse dyes at temperatures below 100ºC. The dyes are dispersed in a small volume of *n*-butyl acetate to obtain a micro-emulsion to which a development auxiliary (coumarin or *o*-vanillin) is added. The low molecular weight disperse dyes used exhibited acceptable exhaustion after 120 minutes and gray scale values of washing fastness was slightly better than o-vanillin. The absorption rate constants values increases with increasing temperature.

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