



## The wash-off of dyeings using interstitial water: Part 3. Disperse dyes on polyester

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### ABSTRACT

Poly(ethylene terephthalate), which had been dyed at 0.5, 1.0 and 2.0% depths of shade using three disperse dyes, was reduction cleared using a traditional, four-stage process that comprised two water rinses at 40 °C, treatment with aq. Na<sub>2</sub>CO<sub>3</sub>/Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub> at 60 °C and one cold water rinse. A novel, two-stage wash-off method was also employed that consisted of treatment with damp nylon beads and surfactant at 70 °C and one cold water rinse. In terms of fastness to repeated washing at 60 °C and colorimetric characteristics, the traditional, four-stage reduction clearing treatment using aq., alkaline Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub> could be replaced by the two-stage, bead wash-off with detergent at 70 °C. As the detergent-based, bead wash-off process used lower amounts of water than reduction clearing and did not employ sodium dithionite, it avoided the environmentally unacceptable generation of aromatic amines in the case of the reduction clearing of azo dyes. Calculations indicated that considerably less heat energy was consumed in bead wash-off than reduction clearing not only because two, rather than four stages were involved but also since the bead process used only a 2:1 water:fibre ratio rather than the 20:1 liquor ratio employed in the reduction clear process; also, the much lower specific heat capacity of nylon than water meant that much less heat was required to heat the beads. The beads adsorbed vagrant disperse dye during wash-off, thereby offering the potential of a lower effluent load compared to a traditional reduction clearing treatment for disperse dyes on polyester.

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### 1. Introduction

Surplus dye and dyeing/printing auxiliaries are removed from dyeings and prints by means of an aqueous treatment, to which the generic term 'wash-off' has been ascribed [1]. In the case of poly(ethylene terephthalate) (PET) and other hydrophobic fibres, such as poly(lactic acid) (PLA) which has been dyed with disperse dyes, particulate dye molecules accumulate at the fibre surface at the end of dyeing because of the dye's sparing solubility in water. As this surface deposited dye impairs brightness of shade and reduces fastness, a specific wash-off process, commonly referred to as *reduction clearing* is employed, which entails treating the dyed material at 50–80 °C in an aqueous, alkaline solution of sodium dithionite (sodium hydrosulfite; Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub>). In the case of azo disperse dyes, the typically, 4–5 stage reduction clearing process, such as that shown in Fig. 1, cleaves the azo bond, generating colourless amino compounds whilst anthraquinoid disperse dyes are converted to the almost colourless, water-soluble, low substantivity, *leuco* variant (Scheme 1). The ability of the reduction clearing process to reduce dye that is present only at the fibre surface accrues

from the marked hydrophobicity of the fibres (e.g. PET, PLA) which are traditionally dyed using disperse dyes together with the fact that the process is carried out below the glass transition temperature ( $T_g$ ) of the fibre (for example, ~55–65 °C for PLA and ~80–90 °C for PET, respectively), with the result that the aqueous alkaline, Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub> solution does not penetrate the fibre. Reduction clearing is widely used, especially in the case of medium/heavy depths of shade and, for some forms of dyed polyester, regardless of depth of shade, to remove surface deposited polyester oligomers [2].

However, reduction clearing suffers from several disadvantages insofar as the multi-stage process necessitates three changes in pH [acidic dyebath (~pH 5) to alkaline reduction clear (~pH 12) to neutralisation with aq. CH<sub>3</sub>COOH] and also consumes large amounts of water, energy and chemicals; in addition, sodium dithionite generates an environmentally unacceptable effluent that is further compromised by the presence of aromatic amines in the case of azo disperse dyes. Attempts have been made to reduce the environmental impact and cost of reduction clearing, as exemplified by research efforts directed towards temporarily solubilised disperse dyes [3–16], microencapsulated disperse dyes [17], and, in the late 1970's, the introduction of commercial ranges of alkali-clearable disperse dyes [18–20], which, under hot, aq. alkaline conditions, either generate water-soluble derivatives via hydrolysis or undergo decolourisation via cleavage of the chromophoric system, thereby enabling surface

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deposited disperse dye to be removed without recourse to reducing agents; alkali-clearable disperse dyes continue to attract research attention [7–9,11–13,15,21–31]. Various replacements to sodium dithionite have been investigated, including acetone [32], ozone [25,33] and detergents [34–37], the latter approach having been shown [34] to not only reduce the BOD, COD, TOC and suspended solids generated during a typical traditional reduction clearing treatment of disperse dyes on polyester but also to offer a potential way of avoiding the environmentally unacceptable generation of aromatic amines in the case of azo dyes. However, despite such interest, the traditional reduction clearing process using aq. alkaline,  $\text{Na}_2\text{S}_2\text{O}_4$  treatment continues to enjoy widespread use, mostly in unchanged guise since its inception in the mid 1950's [38,39].

This paper describes the use of a wash-off process for dyeings that employs a novel, re-usable and re-cyclable polyamide bead medium to replace the vast majority of the water and chemicals that are traditionally used in wash-off. The patented, novel process [40], which is currently being commercialised by Xeros Ltd. [41] does not employ a large reservoir of water to immerse the dyeing/print but, rather, uses only the relatively small amount of water present within the interstices of both the damp fabric and the bead material. The first part of the paper [42] comprised an introduction to the wash-off process, describing the theoretical and empirical considerations that underpinned the selection of the nylon bead material whilst the second part of the paper [43] compared the wash-off of 2% and 5% omf dyeings of bis(aminochlorotriazine) dyes reactive dyes from cotton, using the dye maker's recommended, five- and six-stage, wash-off processes as well as a novel method that utilised two water rinses and treatment with damp nylon beads. Similar depths of shade of comparable fastness to repeated wash fastness at 60 °C were achieved using the various wash-off methods [43]. As the beads adsorbed a sizeable amount of vagrant reactive dye that was removed during wash-off, the bead wash-off process generated less rinse liquor compared than the recommended wash-off processes, thereby constituting a lower effluent load [43]. This part of the paper describes the use of the novel, bead wash-off process for PET which had been dyed with disperse dyes and compares the colorimetric and fastness data secured for the washed-off dyeings to those which had been obtained in a previous study [34] using a traditional reduction clearing process.

## 2. Experimental

### 2.1. Materials

Scoured, woven polyester fabric (122 g m<sup>-2</sup>; Whaleys) together with commercial samples of the three disperse dyes, namely *Dianix*

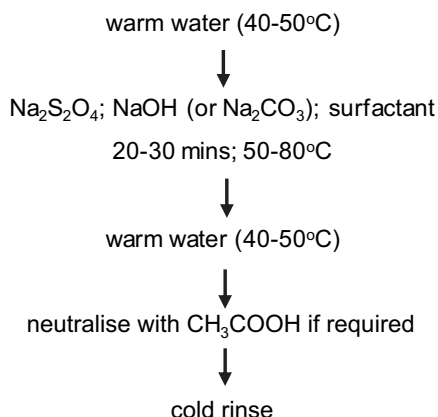
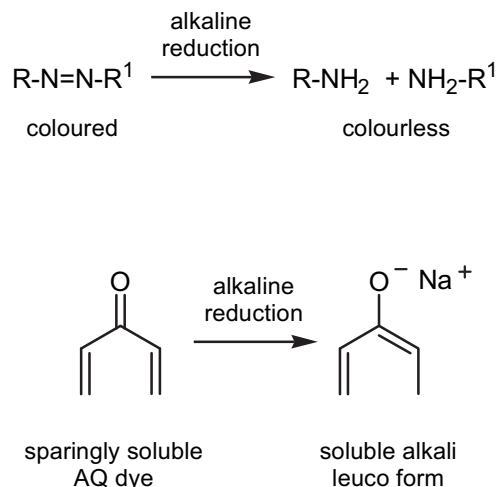


Fig. 1. Typical reduction clearing process for disperse dyes on hydrophobic fibres.



Scheme 1. Effect of reduction clearing on azo and AQ disperse dyes.

*Blue UN-SE, Dianix Yellow Brown XF and Dianix Cherry CC* (no C.I. Generic Names ascribed), were used. Commercial samples of the non-ionic surfactant, *Sandozin NIN* was kindly supplied by Clariant, the proprietary anionic levelling agent *Levegal DLP* from DyStar and *ECE Reference Detergent B* from SDC Enterprises Ltd. Owing to commercial confidentiality, details of the polyamide bead material used cannot be disclosed.

### 2.2. Dyeing

0.5, 1.0 and 2.0% omf depths of shade were carried out in sealed, 300 cm<sup>3</sup> capacity, stainless steel dyepots housed in a Roaches *Pyrotec S* dyeing machine, employing a 20:1 liquor ratio (Fig. 2); the pH was adjusted using McIlvaine buffer [44].

### 2.3. Reduction clearing

After dyeing, the fabrics were reduction cleared using the method shown in Fig. 3, employing a 20:1 liquor ratio. The rinsed, reduction cleared dyeings were allowed to dry in the open air.

### 2.4. Bead wash-off

Dyeings were squeezed and treated, using the method shown in Fig. 4, in a sealed polypropylene container using sufficient beads to provide a 1:15 dyed fabric:bead ratio and sufficient aq 4 gl<sup>-1</sup>

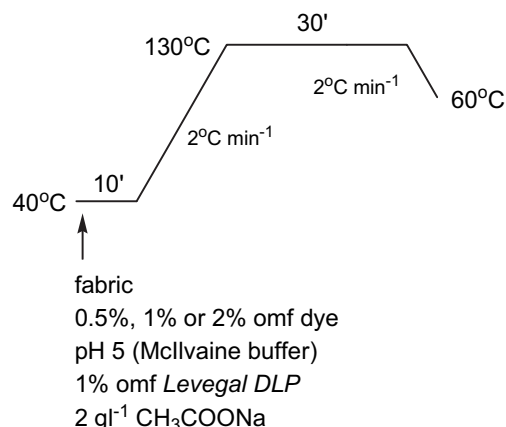


Fig. 2. Dyeing method.

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