

The role of auxiliaries in the immersion dyeing of textile fibres: Part 10 the influence of inorganic electrolyte on the wash-off of reactive dyes



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ABSTRACT

The depth of shade of dyeings obtained using three commercial reactive dyes on cotton in the absence of added electrolyte at a 1:2 liquor ratio were of similar magnitude to those secured in the presence of 50 g l⁻¹ NaCl at a 1:10 liquor ratio. In the case of both the 2% omf (on mass of fibre) and 5% omf dyeings which had been produced in the complete absence of added inorganic electrolyte at low liquor ratio, substantially modified wash-off processes could be used that required $\geq 50\%$ fewer wash baths than conventional wash-off processes that must be used in the case of dyeings produced in the presence of added inorganic electrolyte. Use of the modified wash-off processes enables substantial savings in both energy and water compared to their conventional wash-off counterparts; a 50% saving in proprietary wash-off agent was also secured when a 1:5 liquor ratio was employed for the modified wash-off processes. The ensuing electrolyte-free dyeings displayed excellent levels of wash fastness that were characteristic of those exhibited by commercial reactive dyes on cotton and other cellulosic fibres.

1. Introduction

Of the many different types of dyeing auxiliary that are available for exhaust dyeing processes [1], inorganic electrolyte, in the form of either NaCl or Na₂SO₄, is the most fundamentally important auxiliary employed in the dyeing of cotton and other cellulosic fibres using several classes of dye, notably direct dyes and, of greatest commercial significance, reactive dyes. In essence, the addition of inorganic electrolyte to a direct dye or reactive dye dyebath promotes dye uptake. Despite the widespread use, over many decades, of NaCl or Na₂SO₄ in cellulosic fibre dyeing, the precise manner by which such marked promotional effects are imparted, was not adequately resolved.

Previous parts of this paper have sought to elucidate the mechanism by which inorganic electrolyte promotes the uptake of commercial grade direct dyes [2–5] as well as both commercial grade [6–8] and pure [9] reactive dyes on cellulosic fibres. In this context, theoretical models were derived, based on the concept of interstitial water in dyeing, which attributed the marked promotional effect of added NaCl or Na₂SO₄ in the immersion application of direct dyes [3] and reactive dyes [7] to induced dye aggregation in the dyebath which reduces the solubility of both the direct and reactive dyes in the dyebath, which, in turn, favours increased dye uptake. This particular theoretical approach also was able to explain the promotional effect which reducing the liquor ratio used for dyeing has upon both direct dye [4] and reactive dye

[7] adsorption on cellulosic fibres. It was therefore considered [5,8] that the two, seemingly dissimilar dyebath actions, namely adding inorganic electrolyte and reducing liquor ratio exert the same promotional effect on both direct dye and reactive dye adsorption. When used to interpret results obtained for the application of commercial grade direct dyes [5] and both commercial grade [8] and pure [9] reactive dyes, the theoretical models also explained why cotton can be dyed to realistic depths of shade in the complete absence of added NaCl or Na₂SO₄ using low liquor ratios.

Since cotton can be dyed using commercial grade reactive dyes in the absence of added inorganic electrolyte [8], the dyed material will not contain residual inorganic electrolyte at the end of dyeing, and, therefore, it should be possible to utilise a simplified wash-off process to remove unfixed dye. This paper describes the development of such a modified wash-off process.

1.1. Reactive dyes

Aspects of both the chemistry and application of reactive dyes used for dyeing cotton and other cellulosic fibres have been described [6,9]. Essentially, as reactive dyes are able to form a covalent linkage with ionised hydroxyl groups within the cellulosic substrate, the ensuing dyeings characteristically display excellent levels of fastness to wet treatments (eg washing, perspiration, etc.). To expedite dye-fibre

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fixation (ie covalent reaction with the fibre), the dyes are applied under aqueous alkaline (eg NaOH, Na₂CO₃) conditions to generate the strongly nucleophilic, ionised hydroxyl group (Cell-O⁻) within the substrate with which electrophilic groups in the dye can form a covalent bond. However, such aqueous alkaline application conditions result not only in covalent dye-fibre fixation but also alkali-induced hydrolysis of the reactive dye, with the result that all types of commercial reactive dyes for cellulosic fibres exhibit inherently low levels of dye-fibre fixation efficiency (~50-70%).

Conventional immersion application processes employed for reactive dyes customarily involve the use of significant amounts of added inorganic electrolyte to promote dye uptake; indeed, traditional dyeing theory dictates that either NaCl or Na₂SO₄ must be added to a reactive dye dyebath in order to achieve adequate dyeing. The amount of inorganic electrolyte utilised in conventional exhaust dyeing processes for reactive dyes varies, typically within the range 50 and 100 g l⁻¹ of NaCl or Na₂SO₄, which corresponds to between 400 and 800 kg of either NaCl or Na₂SO₄ per T of fibre assuming a 1:8 liquor ratio is used for dyeing. However, the continuing usage of such high levels of electrolyte must be regarded as untenable, globally, when it is considered that in 2015, in the case of the estimated ~15 × 10⁶ T of cellulosic fibres that were dyed using reactive dyes, if this fibre tonnage had been dyed using immersion processes that employed a 1:8 liquor ratio, then between ~6 × 10⁹ kg and 12 × 10⁹ kg of added inorganic electrolyte would have been consumed [6].

The persistent use of such inexcusably high levels of inorganic electrolyte requires large expenditure, not simply because of the direct cost of the large amounts of inorganic electrolyte utilised in dyeing (NaCl = 10 US\$ T⁻¹; Na₂SO₄ = 20 US\$ T⁻¹ [10]) but also the expense of treating the very high volumes of reactive dyeing wastewater that is contaminated with high levels of residual NaCl or Na₂SO₄ as well as unfixed reactive dye. Indeed, the effluent from reactive dyeing processes presents many difficulties as it is remarkably resilient to biodegradation and, whilst many effluent treatment strategies have been considered, the pronounced salinity of reactive dyeing wastewater is a major concern.

1.2. Wash-off

Because alkali-induced dye hydrolysis always accompanies dye-fibre fixation, hydrolysed dye, as well as any unreacted dye, will be present in both the residual dyebath and the final dyed material. All hydrolysed dye and non-covalently bound reactive dye (ie unreacted dye) must be removed from the dyed material at the end of dyeing so that the dyeing displays maximum fastness to wet treatments such as domestic laundering. All reactive dyeings are therefore subjected to a rigorous wash-off process at the end of dyeing so as to remove all unfixed reactive dye.

Conventional reactive dyeing wash-off processes commonly involve treating the dyed material in several baths that contain water, usually at different temperatures, and, in at least one of the baths, a proprietary wash-off agent that expedites the removal of residual hydrolysed and unreacted reactive dye from the dyeing; the liquor ratio used for wash-off is normally 1:10 (Fig. 1).

Owing to the intrinsic inefficiency of dye-fibre fixation imparted by alkali-induced dye hydrolysis, immersion dyeing processes for reactive dyes therefore involve two distinct stages, namely the dyeing stage in which the dye is applied under aqueous alkaline conditions and the wash-off stage in which residual unfixed dye is removed.

1.2.1. The nature of wash-off

Although the particular conditions recommended by dye makers for the wash-off of their particular range(s) of reactive dyes differ depending on factors such as type of dye used, depth of shade, fibre type, etc., all conventional wash-off methods routinely consume very large amounts of water and entail the use of a proprietary wash-off auxiliary.

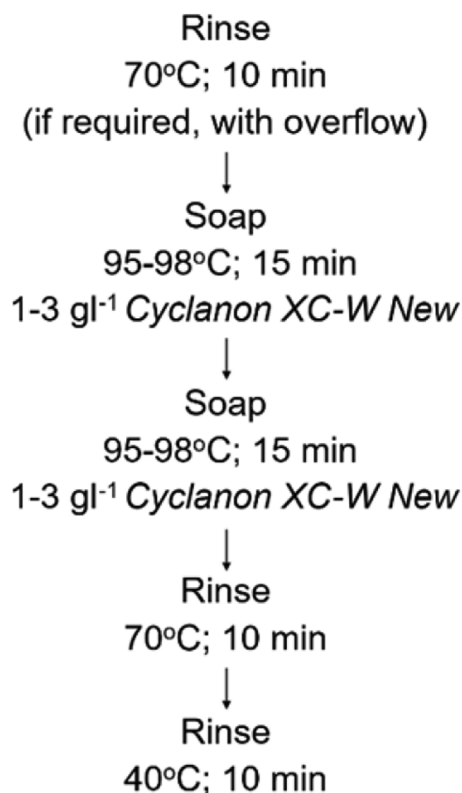


Fig. 1. Wash-off process recommended for monochlorotriazine reactive dyes in deep shades [11].

As such, conventional wash-off processes that are utilised in the application of reactive dyes to cellulosic fibres are a major contributor to the effluent load generated by reactive dyeing processes, owing to the presence of residual hydrolysed and unreacted dye as well as proprietary wash-off agent.

Despite the global popularity of reactive dyes for cellulosic fibres and the astonishingly high number of research papers, reports and patents that have concerned this apparently most intriguing of all dye classes over the past 60 or so years, the wash-off stage has received surprisingly modest attention. In this context, several authors have sought to modify/improve reactive dye wash-off processes (eg see Refs. [12–14]) and replace the proprietary wash-off agent [15]. As conventional reactive dye wash-off processes routinely consume large amounts of water, attempts have been made to reduce water usage; studies [16,17] revealed that it was possible to replace the vast majority of the water employed in a conventional reactive dye wash-off process with a novel, re-usable and re-cyclable polyamide bead medium which had been developed for a novel dyeing process [18] and laundry process [19–22]. In the latter context, the wash-off process employed for reactive dyes on cellulosic fibres was considered [17] to be analogous to a domestic laundering process insofar as wash-off processes rely upon the combination of time, temperature, detergency and water to expedite dye removal and, thereby, conform to the ‘Sinner’s Circle’ which is commonly used to represent the four main factors that determine domestic laundering performance [17]. It has been demonstrated that both the duration of wash-off as well as the amounts of water and chemicals employed could be reduced by replacing a five-bath wash-off process recommended for bis(aminochlorotriazine) dyes [23] and bis(vinyl sulfone), aminochlorotriazine/vinyl sulfone and bis(aminochlorotriazine/vinyl sulfone) dyes [24] on cotton, with a novel, wash-off agent-free, three-bath process. Environmental indicators (BOD₅, COD and COD/BOD₅) were employed to describe potential improvements in the biodegradability of the wastewater generated using the chemical-free, three-bath wash-off process [23,24]. It was found that

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