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The role of auxiliaries in the immersion dyeing of textile fibres: Part 11 residual inorganic electrolyte levels present during the wash-off of commercial grade reactive dyes

Na₂SO₄ in dyeing.

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ARTICLE INFO	A B S T R A C T
Keywords:	When both 2% omf and 5% omf dyeings on cotton were produced using three commercial grade reactive dyes in
Dyeing cotton	the complete absence of added inorganic electrolyte, a substantially modified wash-off process could be used in
Inorganic electrolyte	which both the number of wash-off baths and the liquor ratio utilised for each wash-off bath could be reduced. Measurements of the chloride ion concentration of residual dyebath, wash-off bath and final rinse bath liquors revealed that the reason why such a modified wash-off process could be utilised is due to the inherently very low dye-fibre substantivity that exists within the dyed substrate that results from the absence of added NaCl or
Chloride content	
TDS	
Reactive dyes	
Salt free dueing	

1. Introduction

Salt-free dyeing

This paper concerns the manner by which some of the auxiliary chemicals that are utilised in dyeing impart their particular, often unique, assistance to exhaust dyeing processes [1]. The work described thus far has sought to elucidate the precise mode of action of the most important dyeing auxiliary used in the dyeing of cellulosic fibres using direct dyes [2-5] and reactive dyes [6-9], namely inorganic electrolyte, in the guise of either NaCl or Na₂SO_{4.} Briefly, adding inorganic electrolyte to a reactive dye dyebath or direct dye dyebath promotes dye uptake because of the combined effects of electrolyte-induced dye aggregation and reduced dye solubility which shifts the inherent preference of the highly water-soluble dyes to favour the aqueous phase towards the fibre phase. Theoretical models were derived [3,4,7], based on the concept of interstitial water in dyeing, to explain the marked promotional effect of added NaCl or Na₂SO₄ on direct dye and reactive dye uptake. These models were able to interpret practical results obtained when cotton had been dyed using commercial grade direct dyes [5], commercial grade reactive dyes [8] as well as pure reactive dyes [9]. In so doing, the theoretical models were able to explain why cotton can be dyed to realistic depths of shade using direct dyes [5] and reactive dyes [8,9] in the complete absence of added inorganic electrolyte using low liquor ratios.

In the previous part of the paper [10] it was found that when commercial grade reactive dyes were applied to cotton in the absence of added inorganic electrolyte, modified wash-off processes could be utilised to remove unfixed reactive dye which required \geq 50% fewer wash-off baths than conventional wash-off processes used for similar depth of shade dyeings obtained in the presence of added inorganic electrolyte; it was also shown that the use of such modified wash-off processes enabled savings in energy, water and proprietary wash-off agent to be achieved. It was assumed [10] that such modified wash-off processes could be employed in the case of dyeings undertaken in the absence of added NaCl or Na₂SO₄, because large amounts of residual inorganic electrolyte were not present in the dyed material and, therefore, the characteristically very high level of electrolyte-induced dye-fibre substantivity within the dyed samples would be reduced or even nullified.

Clearly, such a supposition requires confirmation. As very few publications are available in the public domain that concern the level of residual electrolyte present in reactive dyed materials at the end of dyeing and their effects on wash-off, especially in the case of dyeings undertaken in the absence of added electrolyte, it was decided to secure relevant data. This is the purpose of the work reported in this part of the paper, insofar as measurements were made of the chloride ion concentration, unfixed dye concentration and the total dissolved solids content of residual dyebaths, wash-off baths and final rinse baths, in the cases of both 2% and 5% omf dyeings on cotton obtained in the presence and absence of 50 gl⁻¹ added NaCl.

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1.1. Reactive dyes and wash-off

An introduction to reactive dyes utilised for dyeing cellulosic fibres was presented previously [6,9]. Briefly, aqueous, alkaline conditions (eg using NaOH and/or Na₂CO₃) are employed to apply reactive dyes to cellulosic fibres in order to expedite dye-fibre fixation (ie the covalent reaction between electrophilic groups in the dye molecules and nucleophilic, ionised hydroxyl groups in the cellulosic substrate). Unfortunately, the aqueous alkaline conditions that must be used during dyeing to achieve dye fixation also result in alkali-induced hydrolysis of the reactive dye. Consequently, all immersion dyeing processes currently utilised for reactive dyes are intrinsically inefficient (\sim 50–70% fixation efficiency) because hydrolysed dye cannot enter into dye-fibre covalent linkage.

Consequently, at the end of dyeing, residual hydrolysed dye as well as unreacted reactive dye (ie unfixed dye) will be present in both the dyebath and, more importantly, in the dyed material. In order for the dyed sample to exhibit the very high level of wet fastness that is required of reactive dyes on cellulosic fibres, all traces of hydrolysed dye and unfixed dye must be removed from the dyed material at the end of dyeing. This is achieved using an aqueous wash-off process [10] in which the dyeing is treated in a succession of aqueous wash-off baths at different temperatures; usually, a wash-off agent is employed to expedite removal of residual dye. Such is the importance of such wash-off processes that they are an integral part of the immersion process of dyeing cellulosic fibres with reactive dyes. Conventional wash-off processes utilise four, five or more separate wash-off baths, as well as a final water rinse bath; such processes therefore characteristically consume large amounts of both water and energy.

The exhaust dyeing of cellulosic fibres using reactive dyes suffers from another major drawback which has significant cost and environmental consequences. Conventionally, large amounts of inorganic electrolyte (eg 50–100 gl⁻¹ of either NaCl or Na₂SO₄) are added to reactive dye dyebaths to promote dye uptake. Thus, at the end of dyeing, the dyed substrate will contain not only hydrolysed dye and unfixed dye, but also large amounts of inorganic electrolyte. In order to remove all residual dye from the dyed cellulosic fibre at the end of the dyeing process, the concentration of NaCl or Na₂SO₄ within the dyed fibre within the substrate must be reduced because the inorganic electrolyte promotes the substantivity of both hydrolysed reactive dye and unfixed reactive dye towards the substrate. Thus, the presence of residual inorganic electrolyte in the dyed fibre at the end of dyeing makes it very difficult to remove all traces of residual dye during wash-off. Indeed, the primary function of the first one or two wash-off baths in a conventional wash-off process for reactive dyes is to reduce the high concentration of inorganic electrolyte within the substrate, so that electrolyte-induced, dye-fibre substantivity is lowered, which favours removal of both hydrolysed dye and unfixed dye.

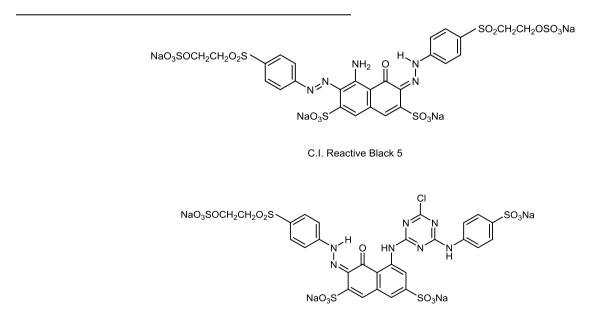
The presence of NaCl or Na_2SO_4 within the dyed cellulosic fibre is a major reason why conventional wash-off processes for reactive dyes on cellulosic fibres require the use of multiple rinse baths, high temperatures, high liquor ratios and proprietary wash-off agents. It follows therefore, that if reactive dyes can be applied to cellulosic fibres in the absence of added inorganic electrolyte, it should be possible to utilise a modified, shortened, wash-off process because it is not necessary to remove large amounts of residual electrolyte. This was indeed found to be the case [10] insofar as when cotton had been dyed using three commercial grade reactive dyes in the complete absence of added NaCl, both the number of wash-off baths and the liquor ratio that was utilised for each wash-off bath, could be reduced.

In this part of the paper, the reasons why such a modified wash-off process could be used for electrolyte-free dyeings of commercial grade reactive dyes on cotton are discussed.

2. Experimental

2.1. Materials

The scoured and bleached knitted cotton fabric (235 gm⁻²) and the three commercial grade reactive dyes, *Duractive Black B* (C.I. Reactive Black 5), *Remazol Red RB grain* 133% (C.I. Reactive Red 198) and *Remazol Yellow R grain* (no C.I. Generic Name ascribed) described previously [10], were employed. The amount of diluent NaCl present in the commercial dye samples was: C.I. Reactive Black 5 11.6%, C.I. Reactive Red 198 11.5% and Remazol Yellow R 12.3% [8]. All other chemicals were of general purpose grade except for a commercial sample of the wash-off auxiliary *Cyclanon XCW* which was kindly supplied by BASF.



C.I. Reactive Red 198

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