Industrial Crops and Products 46 (2013) 350–358

Contents lists available at SciVerse ScienceDirect

Industrial Crops and Products

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

A promising route to dye cotton by indigo with an ecological exhaustion process: A dyeing process optimization based on a response surface methodology

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ARTICLE INFO

Article history:
Received 10 December 2012
Received in revised form 8 February 2013
Accepted 11 February 2013

Keywords:
Cotton modification
Indigo
Clean process
Optimization
Response surface methodology

ABSTRACT

The present paper investigates a non conventional but eco-friendly exhaust dyeing process of cotton with indigo. During this process, indigo was converted to its water-soluble leuco form by a green reducing agent: the glucose, in presence of alkali and at high temperature.

To improve the exhaust dyeing process, the dyeing step was carried out on modified cotton by several cationizing agents. Modified cotton fibres were characterized by Fourier transform infrared (FTIR) spectra and an X-ray diffraction analysis. The performances of the dyeing process were evaluated by measuring the bath exhaustion E (%), the colour yield (K/S), the brightness index BI (%) and the dyeing fastnesses of the coloured cotton. It was found that the colour yield and the brightness obtained from the exhaustion dyeing were improved when using cationized cotton giving fastness properties better than those obtained with untreated cotton dyed by the conventional process. The effect of the main operating conditions (cationizing agent nature and concentration, reducing temperature, dyeing duration, dyeing temperature) on the quality of this dyeing process were also studied. A surface design was employed for experimental design and optimization of results. Mathematical model equation and statistical analysis were derived by computer simulation programming applying the least squares method using Minitab 15.

1. Introduction

The application of indigo to cotton requires a very specific dyeing process. This process is continuous used for dyeing cotton yarns. It consists generally of 6 dip-6 nip in order to achieve deep shades (a completed '1-dip 1-nip' cycle consists of dipping fabric in dye liquor for 1 min than airing it for 2 min). This is due to the low substantively of the reduced form of indigo to cotton fibres. Currently, there is an important demand from consumers to garments dyed totally with indigo. Dyeing garments by indigo in machine is very advantageous but challenging because it needs the application of an exhaustion dyeing process. In fact, in the case of indigo, this process gives generally not satisfying results in terms of colour yield and level dyeing. So, it is necessary to develop new processes to accomplish a dyeing quality as good as the conventional processes using the environmentally unfavourable sodium dithionte for reducing indigo. In order to achieve this purpose, it is important to enhance the affinity of cotton fibres to the bienolate form (the leuco-indigo reduced form) (see Fig. 1). This would be possible when a chemical modification via cationisation process was operated on cotton using a cationic agent. This promising route has recently attracted interest from many researchers in order to enhance the dyeability of cellulosic fibres by several anionic dyes, such as acid dyes (Rong and Feng, 2006), direct dyes (Burkinshaw and Gotsopoulos, 1999), reactive dyes (Montazer et al., 2007), sulphur dyes (Burkinshaw and Gotsopoulos, 1996) as well as natural dyes (Kamel et al., 2007, 2009, 2011). The reason behind such pretreatment is that the increased cationicity imparted to the cellulosic substrate reduces the inherent ion ± ion repulsion that operates between the anionic dye and negatively charged groups in the fibre, thus resulting in enhanced dye uptake. Besides, it has been shown that adding cationic sites to the fibres brings significant advantages in reducing environmental impact following the dyeing process (Ma et al., 2005; Xie et al., 2008; Zhang et al., 2005, 2007, 2008). Chemically, cationized cotton is usually produced by the etherifying reaction of cotton with the tertiary amino or quaternary ammonium cationizing reagents, especially quaternary ammonium cationizing reagents (Hauser, 2000; Montazer et al., 2007).

The present study focuses on the dyeing of cationized cotton by indigo using an exhausting dyeing process. As the

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http://dx.doi.org/10.1016/j.indcrop.2013.02.009

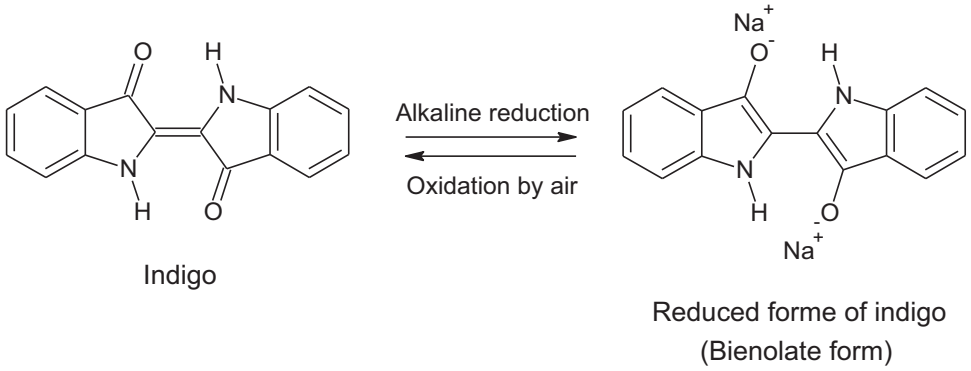


Fig. 1. Redox reaction of indigo.

α-hydroxycarbonyls recently represent an attractive ecofriendly alternative to sodium dithionite (Meksi et al., 2012), the reduction of indigo was carried out using glucose as green reducing agent.

The performances of this exhausting dyeing process were compared with those of the classical process. The effects of the main experimental conditions (cationizing agent nature and concentration, reducing temperature, dyeing duration, dyeing temperature) on the quality of this dyeing process were studied. Besides, modelling and optimization of some experimental conditions were investigated in order to improve the performances of this exhausting indigo dyeing process.

2. Experimental

2.1. Chemicals and materials used

Indigo (Bezema AG, Switzerland), Glucose (Sigma, France), and Sodium Hydroxide (CDM, Germany) were used for the reduction without further purification.

Croscolor DRT (Eurodye-CTC, Belgium), Evo Soft HS P (CPM, Tunisia), Sera Fast GMX (CPM, Tunisia) and Rewin Os (Bezema AG, Switzerland) were used as cationizing agents.

The cationic exchange capacity of these cationizing agents was determined firsty by a mineralization in concentrated nitric acid at 90 °C during 30 min under reflux, then titration of the chloride ions formed in solution using the method of Charpentier–Volhard (Rodier et al., 1996). The cationic exchange capacities of the used cationizing agents are given in Table 1.

Commercially bleached but unfinished cotton fabric with the following specifications was supplied from SITEX, Tunisia: plain weave; ends per inch, 33.02; picks per inch, 38.1; warp count, 10.5 open end; weft count, 15 open end; weight, 204 g m^{-2}.

2.2. Cotton cationization

Cotton samples were cationized using a specific preparative bath. The proposed method consists of treating cotton before the step of dyeing by indigo in a bath containing an appropriate amount of a cationizing agent during 60 min at 50 °C. After that, cotton was dried at room temperature.

Table 1
The cationic exchange capacity for different cationizing agents used.

Cationizing agent	Cationic exchange capacity (MmolEquiv. g^{-1})*
Sera Fast GMX (CPM. Tunisia)	125
Croscolor RDT (Eurodye-CTC. Belgium)	84.96
Evo Soft HS P (CPM. Tunisia)	74.21
Rewin Os (Bezema AG. Switzerland)	67.16

* 1 MmolEquiv. g^{-1} = 1 mmol g^{-1}.

2.3. Reduction process of indigo

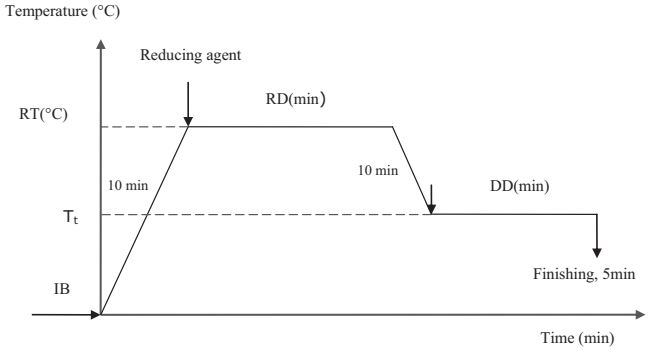
A solution containing 12 g L^{-1} of sodium hydroxide, 2 g L^{-1} of indigo and 10 g L^{-1} of glucose were prepared by adding them to 200 mL of distilled water. This solution was brought up to 75 °C for 120 min as describe in Fig. 2. Reaction was carried out in laboratory autoclave machine (Ahiba Datacolour International, USA) where duration and temperature of reduction were programmed.

2.4. Exhaustion dyeing process

The reaction medium obtained, and which resulted from the reduction process at an appropriate temperature, was used as a dyeing bath after decreasing the temperature to 25 °C (this step lasted about 10 min before the beginning of the dyeing process). Fabrics were dyed at a liquor ratio 50:1 (dyeing bath volume (mL): fabric weight (g)) at 25 °C during 45 min (see Fig. 2). When finished, fabrics were oxidized for 5 min. After that, the dyed samples were subjected to a hand washing with hot water for 5 min at 70 °C and were followed by a cold rinsing and finally dried at room temperature.

2.5. Conventional indigo dyeing process

The conventional indigo dyeing process of cotton used is continuous consisting of 6 dip-6 nip. It was carried out accordingly to



IB : the initial bath containing distilled water, sodium hydroxide and indigo
RT : the reducing temperature (°C)
RD : the reduction duration (min)
DT : the dyeing temperature (°C)
DD : the dyeing duration (min)

Fig. 2. The exhaust dyeing process investigated.