



Electrochemical reduction in vat dyeing: greener chemistry replaces traditional processes

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ABSTRACT

In application of vat dyes the substitution of non-regenerable reducing agents such as $\text{Na}_2\text{S}_2\text{O}_4$ by cathodically regenerable reducing agents offers great ecological advantages. In a demonstration project a 45 A multi-cathode electrolyser successfully was coupled to a dyeing apparatus for package dyeing with a capacity of 1–10 kg material. Iron-complexes with triethanolamine and Na-D-gluconate were used as cathodically regenerable reducing agents. Dyebath regeneration was performed by ultrafiltration. Substantial reduction in chemical consumption and reduction in wastewater volume, released from the dyeing process could be demonstrated. Quality of dyeings was assessed with regard to levelness and iron content analysed in the dyed samples.

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1. Introduction

Textile industry belongs to the big consumers of high quality water. Dependent on the type of product the water consumption for dyeing and finishing of cotton textiles can be estimated between 100 and 200 l of water per kg cotton goods [1]. As a result a comparative assessment of textile processes with regard to the EC Council Directive Concerning Integrated Pollution Prevention and Control (IPPC) has been initiated rather early [2–4]. Among potential techniques which exhibit substantial potential to become best available technologies (BAT) electrochemical processes also have been mentioned [5].

Considerable chemical load in the wastewater results from the dyeing step, where dyestuff and specific chemicals are used to fix the dyestuff on the goods. At the end of the dyeing process non-fixed dye and surplus chemicals are released into the wastewater. Demand to dye different colours, presence of non-regenerable chemicals e.g. hydrolysed reactive dye molecules or dyeing processes with dosage of chemicals e.g. alkali dosage in reactive dyeing prohibit recycling of used dyebaths, which thus is not performed in practice [1]. An emerging field covers the recycling of water by membrane processes, however reuse of chemicals from dyeing processes is not practicable today [6,7].

In case of vat dyes, a class of dyes for premium quality level textiles, the released dyebath contains spent reducing agents, alkali, dispersing agents and other auxiliaries e.g. levelling agents and sequestration agents [8–12].

Strong reducing agents are required to transfer the dispersed oxidised vat dye into the alkali soluble leuco-form. The reduced form of the vat dye exhibits affinity to cellulose fibres and thus becomes exhausted from the dyebath [9,10]. In a following oxidation step the exhausted dyestuff is re-oxidised on the fibre into the oxidised insoluble form [11–14].

The most widely used reducing agent for vat dyeing is sodium dithionite $\text{Na}_2\text{S}_2\text{O}_4$, which reduces the dyestuff under formation of sulfite and sulfate [8]. As regeneration of dithionite is not possible up to now, the used dyebath has to be discharged into the wastewater [15–17].

A promising concept for regeneration of used vat-dyebaths applies redox couples which can be regenerated by cathodic reduction. A number of alkali stable redox couples suited for this purpose has been described in the literature [18–22]. Most important redox couples base on iron(II/III)-complexes which are able to establish a redox potential sufficiently negative to reduce dispersed vat dyes. According to literature data for the formal redox potential required to reduce vat dyes, a suited redox system has to be able to establish a formal redox potential of approximately -1000 mV vs. Ag/AgCl/ 3 M KCl at pH 13 [13,14,23].

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A schematic presentation of the indirect electrolysis of vat dyes is given in Fig. 1.

The possibility to regenerate reducing agents in spent dyebaths permits recovery of these heavily polluting baths. The insoluble oxidized vat dyes can be removed from exhausted dyebaths during the regeneration step by ultrafiltration. At present in the literature a number of reports has been presented describing properties of redox couples, electrolyzers and laboratory dyeings, however studies describing the technology at pilot scale level are not available [15,19,21–27].

In this article results of an experimental study, performed at pilot scale level with the first complete installation of the technical concept, are shown. Coupling of a standard dyeing apparatus for package dyeing with a capacity of 1–10 kg goods and a multi-cathode cell, permits a demonstration of the technology under realistic conditions [28,29]. The study includes removal of dyestuff from spent dyebaths by membrane filtration and reuse of recycled dyebaths. Besides description of the performance of the installation data with regard to cell efficiency, process time, and product quality level, aspects of chemical savings and recycling are discussed.

2. Experimental

2.1. Chemicals

FeCl_3 (40% w/w), Na-D-gluconate, triethanolamine (85% w/w), NaOH (aqueous solution 50% w/w), acetic acid (80% w/w), Na_2CO_3 , were technical grade chemicals.

Primasol[®]NF (wetting agent; alkylphosphate), Setamol WS[®] (dispersing agent; lignosulfonate) Trilon TA or B (complexing agent; nitrilo-triacetic acid, ethylenediamine-tetraacetic acid), Dekol SN (sequestering agent; polyacrylate), Laventin CW (detergent; alkanol-polyglycol-ether), Peregol P (levelling agent; polyvinylpyrrolidone) were commercial products supplied by BASF AG, Ludwigshafen a.R., Germany.

Na-m-nitro-benzene-sulfonic acid (Ludigol, BASF, Ludwigshafen a.R., Germany) was used as oxidant.

All vat dyes were Indanthrene[®] dyes supplied by DyStar Frankfurt a.M., Germany.

Scoured and bleached yarn cones with a mass of approximately 800–900 g cotton yarn (Co, yarn number 15–30 tex (1 tex = 1 g kg⁻¹), Getzner Textil AG, Bludenz, Austria) or cotton/viscose fibre blended yarn (Co/CV, yarn number 36–37 tex, HTL-Dornbirn, Dornbirn, Austria) were used for the dyeing experiments.

2.2. Electrochemical dyeing apparatus

For the pilot scale installation a commercial package dyeing apparatus (Krantz Textiltechnik GmbH, Würselen, Germany) with a maximum capacity of 10 kg of goods was coupled to a modified

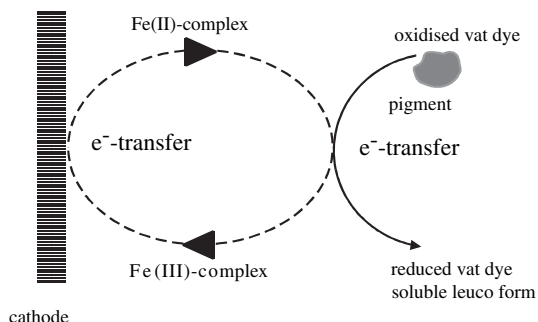


Fig. 1. Reaction scheme of the indirect electrolysis of vat dyes in a divided cell.

multi-cathode electrolysis cell (DE NORA DEUTSCHLAND, Rodenbach, Germany). A scheme of the construction is shown in Fig. 2.

Fresh dyebath/mediator solution was supplied from a storage tank with a capacity of 400 l. The used dyebath was collected in second polyethylene tank of the same size.

For standard procedures such as alkaline scouring, bleaching, as well as dyestuff oxidation after the vat dyeing, soaping and rinsing, the dyeing apparatus worked as a stand alone unit for normal package processing.

For the electrochemical dyeing process, apparatus and cell were filled with mediator solution from the storage tank.

The total volume to fill the dyeing apparatus was 100 l, the filling of the cell required another 100 l. Thus a total volume of 200 l process solution was required for a complete filling. Circulation between the dyeing apparatus and cell was performed with a flow of 150–200 l min⁻¹. Thus exchange of dyebath between apparatus and cell could be obtained within less than one minute.

Due to a throttle placed in the main pipe from the dyeing apparatus back to the cell, a static pressure of 2 bar was established in the dyeing apparatus.

The direction of the internal circulation in the dyeing apparatus, which is required to pump the dyebath through the yarn cones, was reversed regularly, to achieve uniform dyeing conditions through the yarn package.

The cathode stack was built up from 10 isolated cathode units attached to separately adjustable power supplies and connected to a common anode. Characteristic data of the electrochemical cell are shown in Table 1. Relevant data of the full installation are given in Table 2.

2.3. Mediator composition

To prepare the catholyte, a concentrated solution was prepared, from which a volume of 80 ml was diluted to 1 l to obtain the dyebath composition. To prepare the concentrated solution, the calculated amount of FeCl_3 solution was diluted with water to a reach approximately 10% of the final volume of concentrate. Sodium-D-gluconate was dissolved in water, triethanolamine (85% w/w) and part of the required amount of NaOH 50% (w/w) was added. This solution then was added to the FeCl_3 solution in portions to avoid overheating of the mixture formed. Then the rest

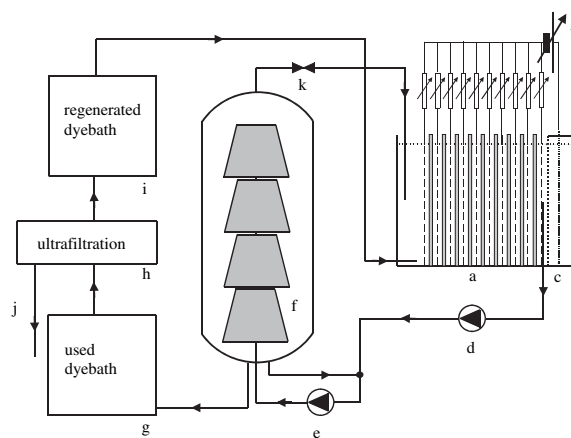


Fig. 2. Scheme of the electrochemical package dyeing apparatus: a. cathode compartment with three dimensional flow through cathodes, b. power supply, c. anode compartment, d. pump for circulation cell-dyeing apparatus, e. circulation pump of dyeing apparatus, f. dyebath with yarn cones, g. tank for used dyebath, h. ultrafiltration unit, i. storage tank for regenerated dyebath, j. drain of dyestuff containing residue, k. throttle to adjust static pressure in apparatus.

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