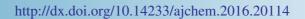


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# Effect of Sodium Borohydride on Microwave Assisted Reductive Cleaning of Dyed Polyester Fabrics

B. YESIM BÜYÜKAKINCI<sup>1,\*</sup>, NIHAL SÖKMEN<sup>2</sup> and BÜSRA BAYENDER<sup>1</sup>

<sup>1</sup>Department of Textile Engineering, Faculty of Engineering, Istanbul Aydin University, Istanbul, Turkey

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In this study, after dyeing polyester fabrics with disperse dyes, reductive cleaning process was applied using two different reduction agents, sodium dithionite  $(Na_2S_2O_4)$  and sodium borohydride  $(NaBH_4)$  and two different methods (conventional method and microwave irradiation). Results compared in terms of CIELab values and washing fastness properties of dyed samples. It was observed that the K/S values of the cleaned samples using  $NaBH_4$  were better than those using  $Na_2S_2O_4$ . As a result of combined use of  $NaBH_4$ , with microwave energy were also advantageous in terms of cost, energy consumption, time and chemical saving.

Keywords: Polyester, Reductive Cleaning, Microwave irradiation, Sodium borohydride, Sodium dithionite.

### INTRODUCTION

Textile industries use different chemicals in dyeing process, consuming large quantities of water and producing large volumes of wastewater. As a conventional method to remove surface loose colours from polyester fiber fabrics dyed with disperse dyes, reductive cleaning is applied with usually sodium dithionite (Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub>) and sodium carbonate. This conventional method is environmentally unfavourable because of the resultant contaminated wastewater. In addition sodium dithionite affects the aerobic processes in the water treatment and toxic hydrogen sulphide can form anaerobically from the sulfate deposits present in the wastewaters [1,2]. Therefore, it is important to replace sodium dithionite with other alternatives in order to achieve cleaner process, such as the use of sodium borohydride is a well-known reducing agent. It has been extensively used in chemical synthesis [3-5]. The use of sodium borohydride in aqueous solution presents many advantages such as facility of storage in alkaline solution and by products formed in the hydrolysis of sodium borohydride are mostly water and sodium metaborate (NaBO<sub>2</sub>) and its hydrates which are completely non-toxic and have minimum effects on environment [6,7]. Limited work can be found in technical literature on using as a reduction agent of indigo dye [8] and instead of sodium sulphide [9], as a bleaching agent for wool [10] and for cellulosic fibers [11].

On the other hand, textile processing consumes huge amount of energy as in dye fixation and heat setting. Saving time and energy is of immediate interest to textile industry. The use of new techniques such as microwave energy, which will allow less energy in less time. In conventional thermal processing, energy is transferred to the material through convection, conduction and radiation of heat from the surfaces of the material, but microwave heating is the transfer of electromagnetic energy to thermal energy and is energy conversion, rather than heat transfer. Microwave energy is delivered directly to materials through molecular interaction with the electromagnetic field. Volumetric heating can also reduce processing times and save energy [12]. Various but limited work can be found in the technical literature on pretreatment [13,14], dyeing [15-18], finishing [19-22], surface modification [23], surface grafting [24] and drying/fixation of textile materials [25-28], in which microwave is used as a heat source.

In this work, we report a novel technique to reductive cleaning process for polyester fabric by sodium borohydride without the addition of alkali. Besides, the microwave heating was applied as an alternative technique to conventional heating. Results were compared by CIELab values and fastness properties of dyed samples.

### **EXPERIMENTAL**

In this study, for the dyeing of 100 % PES fabric (62.55 g/m², 59 warp ends/cm, 33 weft ends/cm) Dianix Red C-4G, Dianix Navy XF, Dianix Yellow Se-G (Dispers Dyes-DyStar) were used. A dispersing agent (Levegal DLP, Bayer), a nonionic surfactant (Perlavin, Dr. Petry) were used as the dyeing auxiliaries.  $Na_2CO_3$ ,  $Na_2S_2O_4$  and  $NaBH_4$  were used for reductive cleaning processes and supplied by Merck.

<sup>&</sup>lt;sup>2</sup>Department of Textile Engineering, Faculty of Technology, Marmara University, Istanbul, Turkey

<sup>\*</sup>Corresponding author: Tel: +90 5322955584; E-mail: byesimb2@gmail.com

**Dyeing method:** 10 g material was used throughout this work. Each dyeing was repeated three times. HT dyeing was carried out in TERMAL HT-dyeing machine. Each dye pot which has 200 mL capacity contained 1 % o.w.f. dye and 1 g/L Levegal DLP (dispersing agent) at a liquor ratio of 20:1. In the HT dyeing, the dyeing process was started at 50 °C and kept at this temperature for 10 min. Then the temperature was raised to 130 °C (at a heating rate of 1.5 °C/min). The dyeing went on for 45 min at 130 °C and, eventually, the bath was cooled to 80 °C. Total dyeing time was 110 min.

Reductive cleaning processes: According to the conventional and microwave irradiation method, after the dyeing process, reductive cleaning was applied to the materials using reducing agents either Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub> or NaBH<sub>4</sub>. The details of reductive cleaning processes are given below.

Conventional method: Conventional reductive cleaning process was applied using Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub> at 60 °C for 15 min at liquor ratio of 1:20 containing 1 g/L Na<sub>2</sub>CO<sub>3</sub> as an alkaline and 1 g/L non-ionic surfactant. In contrast, in the NaBH<sub>4</sub> process, other chemicals were not used.

Microwave reductive cleaning procedure: Microwave-assisted reductive cleanings were carried out in a Samsung (Malaysia) microwave oven (model ME732K, with a maximum input power of 1150 W and output power of 800 W, operating at 2 450 MHz).

Each reductive cleaning-bath (same amount of substance and liquor ratio in the conventional method) in a 250 mL Erlenmayer flask was placed into the oven at 20 °C and its energy level was adjusted to 800 W and the temperature rose up to 60 °C in 1 min. Later, the energy level was immediately shifted to 100 W and the bath was kept at this temperature for 4 min. Then cleaned samples were rinsed with cold water and dried at a room conditions.

Reductive cleaning processes for both methods were given in Table-1.

TABLE-1 REDUCTIVE CLEANING PROCESS CONDITIONS							
Method	Reduction agent (2 g/L)	Na <sub>2</sub> CO <sub>3</sub> (g/L)	Non-ionic surfactant (g/L)	Time (min)			
Conventional	Na <sub>2</sub> S <sub>2</sub> O <sub>4</sub>	1	1	15			
	NaBH <sub>4</sub>	-	-	15			
Microwave	Na <sub>2</sub> S <sub>2</sub> O <sub>4</sub>	1	1	5			
	NaBH <sub>4</sub>	_	_	5			

Measurements and standards: Colour values of the dyed samples were assessed using Datacolor SF600+ spectrophotometer (a USAV 6.6 mm measuring plate was used) and then the CIELab values were calculated using illuminant D65 and 10° standard observer values. From the reflectance values (R) at the maximum absorption wavelength (k<sub>max</sub>) for each dye, the corresponding colour strength (K/S) values of the samples were calculated by using the Kubelka-Munk equation (eqn. 1).

$$K/S = (1 - R)^2 / 2R$$
 (1)

where K is the absorption coefficient of the substrate, S is the scattering coefficient of the substrate and R is the reflectance of the dyed fabric at  $\lambda_{\text{max}}$ .

The rubbing and washing fastness values of the dyed PES fabrics were determined according to ISO105-X12 and ISO105:C06 (A1S) standards, respectively.

#### RESULTS AND DISCUSSION

The colour strength values of all dyed and reductive cleaned samples by using  $Na_2S_2O_4$  and  $NaBH_4$  for both methods (conventional and microwave energy) are shown in Table-2. According to Table-2, there is no significant difference between the colour strength of all dyed and reductive cleaned samples. This result was supported with the values of colour differences ( $\Delta E$ ) as shown in Table-3. The colour differences of all samples were acceptable and less than 1 CIELab unit, except YBc.

TABLE-2						
K/S VALUES OF ALL DYED AND						
	K/S		K/S			
RSc	4.49	RBc	4.34	$\lambda_{\rm max}$ 510 nm		
RSm	4.35	RBm	4.48			
NSc	4.49	NBc	5.34	$\lambda_{max}$ 610 nm		
NSm	4.91	NBm	5.25			
YSc	13.07	YBc	14.18	$\lambda_{\rm max}$ 450 nm		
YSm	13.41	YBm	13.35			

\*Key: R: Dianix Red C-4G; N: Dianix Navy XF; Y: Dianix Yellow Se-G; S: Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub>; B: NaBH<sub>4</sub>; c: conventional method; m: microwave energy.

Example: RSm: the samples dyed with Dianix Red C-4G using microwave irradiation and  $Na_2S_2O_4$  for reducing agent; NBc: the samples dyed with Dianix Navy XF using conventional method and  $NaBH_4$  for reducing agent.

TABLE-3								
	CIELab VALUES AND COLOUR DIFFERENCES OF THE DYED SAMPLES							
RSc (Standard)	$\Delta E^*$	ΔL*	Δa*	Δb*	$\Delta C^*$	ΔΗ*	Result	
RBc	0.839	0.636	0.527	0.148	0.547	-0.027	Light red	
RSm	0.775	0.591	0.488	0.117	0.500	-0.043	Lighter redder yellow	
RBm	0.736	0.216	0.589	0.385	0.681	0.177	Lighter redder yellow	
NSc (Standard)	$\Delta E^*$	$\Delta L^*$	Δa*	Δb*	$\Delta C^*$	$\Delta H^*$	Result	
NBc	0.976	-0.960	0.044	-0.175	0.166	0.069	Darker bluer	
NSm	0.138	0.117	-0.036	0.065	-0.059	-0.045	Lighter	
NBm	0.780	-0.764	0.046	-0.146	0.138	0.066	Darker bluer	
YSc (Standard)	ΔΕ*	$\Delta L^*$	Δa*	Δb*	$\Delta C^*$	$\Delta H^*$	Result	
YBc	1.078	-0.231	1.224	1.406	1.427	-1.200	Darker redder yellow	
YSm	0.516	-0.123	0.406	0.293	0.298	-0.402	Darker redder yellow	
YBm	0.722	0.075	0.534	0.480	0.488	-0.527	Redder yellow	

2754 Büyükakinci et al. Asian J. Chem.

TABLE-4 WASHING AND RUBBING FASTNESS VALUES OF POLYESTER MATERIALS									
Sample	Colour		Staining					Rubbing fastness	
code	change	Cellulose acetate	Cotton	Polyamide	Polyester	Acrylic	Wool	Dry	Wet
RSc	4-5	5	4-5	5	5	5	5	5	5
RBc	5	5	5	5	5	5	5	5	4-5
RSm	5	5	4-5	5	5	5	5	5	5
RBm	5	5	5	5	5	5	5	5	5
NSc	4-5	5	5	4-5	4-5	4-5	5	5	4-5
NBc	5	5	5	5	5	5	5	5	4-5
NSm	5	4-5	5	5	5	5	5	5	5
NBm	5	5	5	5	5	5	5	5	4-5
YSc	5	5	5	5	5	5	5	5	5
YBc	5	5	5	4-5	5	5	5	5	5
YSm	5	5	5	5	5	5	5	5	5
YBm	5	5	5	5	5	5	5	5	5

In the comparison of the K/S values (Table-2) for the two reducing agents, NaBH<sub>4</sub> reductive cleaning process gave slightly better results. The fastness values of dyed samples (washing and rubbing) were also investigated and the results were summarized in Table-4. The rubbing test results and the colour change values were found to be of grade "4-5" to "5" according to Grey scale ratings for all samples.

#### **Conclusions**

In this study reached the following conclusions:

- The K/S values of materials are almost the same in both methods for different processing time.
- The washing and rubbing fastness results of all samples were quite good and between 4-5 and 5.
- The microwave and the conventional reductive cleaning lasted 5 and 15 min, respectively. Therefore, the reductive cleaning time was considerably reduced the energy conservation, time saving and cost effectiveness by using microwave irradiation.
- Microwave irradiation and NaBH<sub>4</sub> process is also advantageous in terms of green chemistry and cost because Na<sub>2</sub>CO<sub>3</sub> and any other chemicals have not been used during the reductive cleaning.

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