

Effect of Ultrasonic Energy on Reductive Cleaning of Dyed Polyester Fabrics

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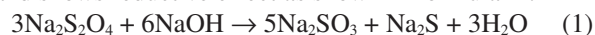
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After dyeing of polyester with disperse dyes, reductive cleaning processes were applied by using sodium ditionite, sodium hydroxide and thiourea dioxide. In this study effects of ultrasonic and conventional methods on reductive cleaning processes were investigated in terms of ΔE^* , ΔL^* , Δa^* , Δb^* , ΔC^* and ΔH^* colour values, K/S values and fastness properties. After conventional (70 °C, 20 min) and ultrasonic (70 °C, 10 min) reductive cleaning processes, values were compared and total colour differences for ultrasonic process for all reductive cleaning agents were in acceptable range. K/S values were higher for ultrasonic sodium ditionite and sodium hydroxide reductive cleaning processes depending on the sodium hydroxide concentration. K/S values were generally higher for conventional thiourea dioxide and sodium hydroxide reductive cleaning processes. It was obtained that ultrasonic energy process does not have a negative effect on fastness properties of the samples.

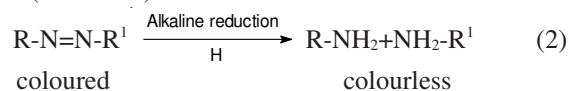
Key Words: Disperse dyed polyester fabric, Reductive cleaning, Ultrasonic method, Conventional method.

INTRODUCTION

Reductive cleaning process is applied for disperse dyeing including washing. Reductive cleaning substances are widely used for reductive cleaning. These substances are inorganic chemical agents, which have high conductivity and contain reductive groups. Sodium ditionite is the most commonly used reductive cleaning substance which is applied in alkaline condition and shows reductive effect as shown in formula 1¹.



It is supposed that chromoform of the azo structured disperse dyes break into pieces and form to colourless amino compounds by process of alkaline solution of reductive substance (formula 2).



At the reductive cleaning process of anthraquinoid structured disperse dyes, low substantive, leuco formed water soluble compounds are formed. Nowadays carrier is only used for polyester/wool blends. Reductive cleaning process is applied to prevent forming of aggregate and accumulation of carrier on fabrics. For high temperature dyeing (HT-130 °C) especially for medium and dark colours, which requires less carrier, reductive cleaning process is applied. It is not possible to obtain good fastness values without reductive cleaning. Reductive cleaning decreases the oligomer forming for light colour dyeing. Neutralization process after reductive cleaning

and setting up the pH of alkaline dyeing baths for alkaline reductive cleaning are increased the cost of dyeing. To prevent these problems some commercial supporter substance blends can be provided to increase the cleaning and conductive substance effect without changing the pH and can be added to the acidic baths directly².

Ecology is an important factor for the reductive washing. Reductive substances such as sodium dithionite, which contains sulfur are hazardous for biologic purifying plants because these substances consume too much oxygen and show toxic effect. Sodium dithionite has some disadvantages such as presence of aromatic amines in disperse dyes. Sodium dithionite is an alternative to boron contain products for continue reductive washing and has advantages to bobbin dyeing and stability control of stock tanks³.

Two most important factors of determining of application fields of reductive washing substances are cost and redox potential of the washing substances. The grade of colour change can be changed according to redox potential of reductive washing substance⁴⁻⁶.

Detergent based chemicals are suggested instead of sodium dithionite based reductive cleaning substances for final process of disperse dyed polyesters. It is indicated that no differences are observed at λ_{max} values and hue-chroma values, but low decrease is observed at colour strength when washed at 98 °C for 15 min, also washing fastnesses at 60 °C are higher and obtains high decrease in terms of BOD, COD and TOC².

Good washing fastness results without colour damage are obtained by 1 min ozone process of disperse dyed polyethylene terephthalate fibres. But more than 1 min ozone process is decreased the tenacity of fabrics without any colour damage. It is indicated that by ozone process, which is applied at room temperature in 1 min is decreased the energy cost and also chemicals which are used in conventional reductive cleaning are not used in this process⁷.

Ecology and environmentally friendly production concepts are forced companies for cleaner production. In this study colour and washing fastness properties of the conventional reductive cleaning substances and thiourea dioxide were investigated.

Thiourea dioxide [$\text{H}_2\text{NC}(=\text{NH})\text{SO}_2\text{H}$], is the derivative of the sulphite acid and it is an organic compound, low odor, easily soluble, environmentally friendly reductive substance to be effective above 65 °C in alkaline condition. It is resistant to high temperatures and keeps the redox potential for a long time. It can stay stable in non alkaline condition for 6 h at room temperature. It is used in leather, textile, paper, photograph industries as reduction substance and also used for decolourization processes. In a study thiourea dioxide and benzene sulfonate zinc based abrasion substance (Decrolin-BASF) were compared and it is indicated that the most close results to 50 g/kg Decolin pigment abrasion substance were obtained by using 20 g/kg thiourea dioxide containing pigment abrasion substance concentrations^{8,9}.

Ultrasonic sound waves have frequencies that human can not hear. The chemical effect of the power of ultrasonic energy is emerged by cavitation. Ultrasonic energy is conveyed by waves. These waves create compression and relaxation in molecular structures of the environment pass. Liquid is decomposed and cavitation bubbles are occurred when negative pressure is applied to the liquid. These bubbles are crashed to each other and caused to come out an energy after consecutive compression periods¹⁰.

The use of ultrasonic energy in textile wet processes has some advantages such as short processing time, low energy and chemical consumption and improved product quality. As a result of the cavitation at solid/liquid interface an increase in mass transfer is obtained from liquid to solid¹¹.

High amount of water, electricity and thermal energy are used in textile wet processes. The chemical substances are also used to accelerate or decelerate the process time in some wet processes. High temperatures are required to transfer the mass from liquid condition to textile material. This transferring process is dependant to temperature and time. Radiofrequency, microwave and IR heating techniques are used to decrease process time and energy consumption. Ultrasonic energy is an important alternative technique for textile processes however it is not used for industrial processes. In order to use ultrasonic energy in industrial processes some problems such as machine design, homogenite in baths, ultrasonic pressure distribution, position of the transducers, fabric position in the machine and temperature of the bath should be solved¹².

The use of ultrasonic energy in sizing baths, preparation of emulsion paths, alkaline and bleaching processes, dyeing, final washings and enzymatic processes are carried out. Process time is decreased and whiteness index is increased

even at low temperatures in hydrogen peroxide bleaching process by using ultrasonic energy¹³. After biocleaning of raw cotton with pectinase by using ultrasonic energy, tenacity, wettability and whiteness index of the raw cotton textile material is increased¹⁴. Combination of conventional and ultrasonic processes is decreased enzyme consumption, process time and fibre damage. Ultrasonic energy is improved the effect of enzyme without decreasing the fabric strength in cotton's enzymatic pre-processes^{15,16}. It is indicated that chemical consumption, fiber damage and waste flote ratio are decreased by using ECE detergent instead of reductive substance in reductive cleaning of disperse dyed PLA fibres¹⁷.

EXPERIMENTAL

Woven polyester fabric was supplied from Sahinler holding (170 g/m²). Fabric was cleaned for 15 min at 60 °C with 1 g/L Sandozin NIN (surfactant; clariant) and 2 g/L 1 Na₂CO₃ at 1:20 flote ratio. Cleaned samples were dried at open atmosphere after rinsed well. In this study Dianix Black CCR (Dy Star) was used as disperse dye without purification.

Dyeing: Polyester fabric was dyed with alkaline poliglicoleter structured non-ionic egalition substance Lyogen DFT (Clariant) at 5 % colour strength in Polimat HT sample dyeing machine (Type A11612N-Emsey), which has 300 mL stainless steel tubes. Dyeing recipe and temperature-time diagram were shown in Table-1 (Figs. 1 and 2).

TABLE-1
DYEING RECIPE

Lyocol WPN	1 %
Albegal FFA	0.5 g/L
Lyogen DFT	0.5 %
Sodium acetate	3 %
pH	5.5 (acetic acid)
Material amount	5 g
Bath ratio	1:20
Dyeing time	110 min
Disperse dyes	5 % o.w.f.

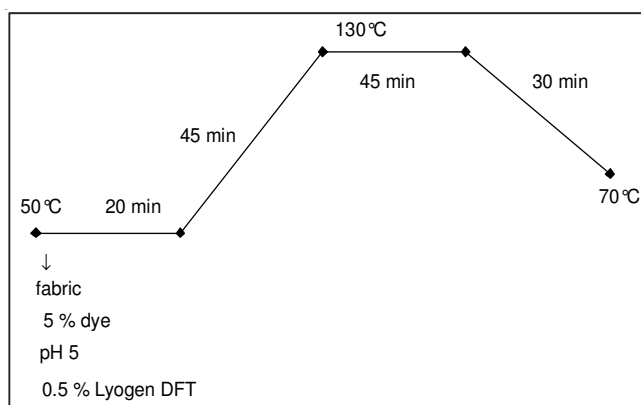


Fig. 1. Temperature-time diagram

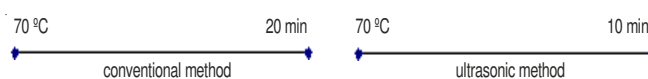


Fig. 2. Temperature-time diagram for reductive cleaning

Reductive cleaning: Reductive cleaning conditions were given in Table-2.

TABLE-2
REDUCTIVE CLEANING CONDITIONS

Code	Conditions
Na ₂ S ₂ O ₄ - NaOH (38° Bé)	
1	2 g/L Na ₂ S ₂ O ₄ - 2 mL/L NaOH
2	2 g/L Na ₂ S ₂ O ₄ - 3 mL/L NaOH
3	2 g/L Na ₂ S ₂ O ₄ - 4 mL/L NaOH
4	2 g/L Na ₂ S ₂ O ₄ - 5 mL/L NaOH
Thiourea dioxide - NaOH (38° Bé)	
5	0.3 g/L TUDO - 0.6 mL/L NaOH
6	0.3 g/L TUDO - 0.7 mL/L NaOH
7	0.3 g/L TUDO - 0.8 mL/L NaOH
8	0.4 g/L TUDO - 0.8 mL/L NaOH
9	0.4 g/L TUDO - 0.9 mL/L NaOH
10	0.4 g/L TUDO - 1.0 mL/L NaOH

TUDO – Thiourea dioxide

Sodium ditionite and sodium hydroxide reductive cleaning: After dyeing process fabrics were rinsed with hot water and reductive cleaned by conventional method, flotte ratio of 1:200, at 70 °C for 20 min the reason of high flotte ratio is to compare with ultrasonic process which needs high flotte ratio for effective sonication. All reductive cleaned dyed fabrics were rinsed first in hot water then in running water, finally they were dried at room temperature. Branson ultrasonic bath was used as the sonication source for ultrasonic process and sample was reductive cleaned, at flotte ratio of 1:200 at 70 °C for 10 min. Properties of the ultrasonic bath, 220 V and 205 W, wave range 50-60 Hz, wave sensitivity 47 Hz ± 6 %. All methods were repeated 3 times.

Thiourea dioxide and sodium hydroxide reductive cleaning: Samples were reductive cleaned by conventional process at flotte ratio of 1:200 at 70 °C for 20 min. Samples were reductive cleaned by ultrasonic process at flotte ratio of 1:200 at 70 °C for 10 min.

Colour measurement: Colour values of dyed samples were measured by data colour spectra flash 600 plus reflectans spectrophotometer by using datamaster software according to CMC 2:1 CIELab and CIELch system. Colour measurements were performed by using 10° observer and D65 light source and conventionally reductive cleaned samples were accepted as standard. In order to identify the colour L*, a* and b* coordinates, which are calculated from tristimulus values were used (Fig. 3).

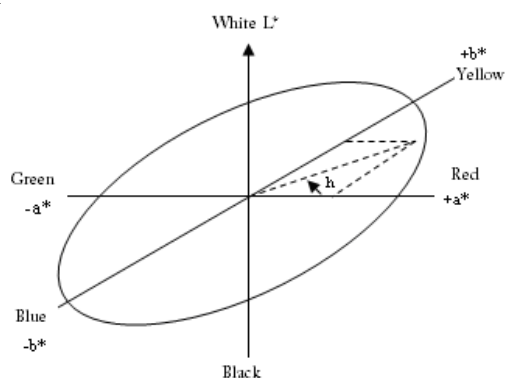


Fig. 3. 3-Dimension colour space

Different tones of same colour are taken place on a line extending outward, which is made up by a* and b* coordinates. The angle of rotation "h" which increases from red to yellow is a

colour measure. For example h = 0° corresponds to red colour tone, h = 90° corresponds to yellow colour tone and h = 270° corresponds to blue colour tone. A remote point from neutral point is expressed the chroma (C*) and this the measure of the colour saturation at a defined L* value. Colour differences were calculated in CIELab units according to formula 3.¹⁸

$$\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2} \quad (3)$$

L*a*b* Cartesian coordinates, L*C*h* can be converted to cylindrical coordinates by formula 4 and 5.

$$C^* = [(a^*)^2 + (b^*)^2]^{1/2} \quad (4)$$

$$h = \arctan b^*/a^* \quad (5)$$

L*_{numune} - L*_{standard} if it is positive, sample is lighter than the standard, if it is negative sample is darker than the standard.

C*_{numune} - C*_{standard} if ΔC* is positive sample has higher chroma (saturation), if it is negative sample has lower chroma.

K/S Values: Colours of the dyed fabrics were evaluated by colour strength (K/S), which is calculated by using Kubelka-Munk equality (formula 6) % reflectance values of the samples were measured by datacolour spectra flash 600 plus reflectans spectrophotometer by using datamaster software and according to CMC 2:1 CIELab and CIELch system. Colour measurements were performed by using 10° observer and D65 light source¹⁸.

$$K/S = (1-R)^2/2R \quad (6)$$

where, R; reflectance value of the fibre at maximum absorption wavelength, K; absorption coefficient, S; scattering coefficient.

Fastness determination: Washing fastness tests of the dyed fabrics were performed according to ISO 105-C06 standard and washing fastness test machine (Gyrowash/James H. Heal Co. Ltd.) was used. Light fastness of the dyed fabrics were performed according to TS 1008 EN ISO 105-B02 standard and light fastness test machine (James H. Heal) was used for the test.

RESULTS AND DISCUSSION

Reductive cleaned samples were accepted as standard for the colour measurement of dyed samples by datacolour spectra flash 600 plus reflectance spectrophotometer. Results were given in Table-3.

TABLE-3
COLOUR MEASUREMENT VALUES

Code	ΔL*	Δa*	Δb*	ΔC*	ΔH*	ΔE*
Na ₂ S ₂ O ₄ -NaOH						
0 original	0.52	0.03	0.08	-0.07	0.05	0.47
1	-0.88	-0.01	0.41	-0.40	0.09	0.94
2	1.55	0.03	0.08	-0.08	0.04	1.45
3	-0.64	0.20	-0.15	0.18	0.18	0.65
4	-0.11	-0.12	-0.28	0.26	-0.17	0.44
Thio-urea dioxide-NaOH						
5	1.00	-0.20	-0.05	0.02	-0.20	0.93
6	0.22	-0.05	-0.08	0.06	-0.07	0.23
7	0.40	-0.04	-0.10	0.09	-0.06	0.38
8	-0.43	-0.04	-0.48	0.47	-0.11	0.78
9	0.35	0.03	0.17	-0.16	0.07	0.39
10	-1.04	0.16	0.08	-0.04	0.17	0.92

Colour differences of the conventionally reductive cleaned samples' colour and reductive cleaned sample's colour which was cleaned by ultrasonic method were acceptable when Na₂S₂O₄ and NaOH used in reductive cleaning. (ΔE* < 1) (Fig. 4).

Saturation of the colour increased and the colour became darker and turned to red and blue by increasing the alkaline concentration (Table-3).

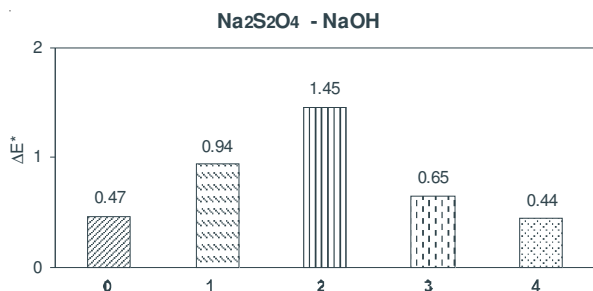


Fig. 4. ΔE* Values of Na₂S₂O₄-NaOH reductive cleaned dyed samples

Total colour differences (ΔE*) of the samples were acceptable at the spectroscopic measurements when thiourea dioxide and NaOH concentration were increased. (Fig. 5). But colours obtained from reductive cleaned samples by ultrasonic method were lighter than standard sample. Colours were turned from green gradation to red and were turned from blue gradation to yellow according to blue-yellow coordinate when sodium hydroxide concentration were increased. Chroma (saturation) decreased and colour became dull when thiourea dioxide and alkaline concentration were increased. (Table-3).

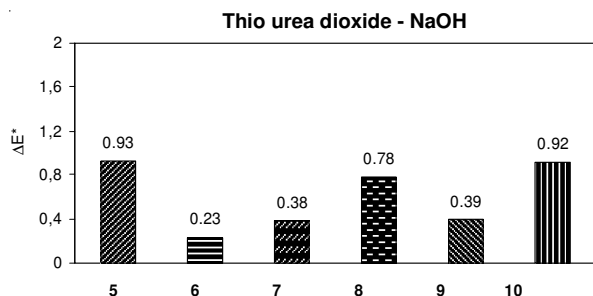


Fig. 5. ΔE* values of thiourea dioxide-NaOH reductive cleaned dyed samples

K/S values of reductive cleaned samples with sodium dithionite and sodium hydroxide at maximum absorption (520 nm) were given in Fig. 6. Higher K/S values were obtained with the increasing of sodium hydroxide concentration on reductive cleaned samples by ultrasonic energy.

K/S values of conventionally reductive cleaned samples with thiourea dioxide and sodium hydroxide were higher and results were given in Fig. 7.

Disperse dyed polyester materials were reductive cleaned with environment friendly thiourea dioxide and conventional sodium dithionite and sodium hydroxide. No decrease was observed at fade washing fastness values of reductive cleaned samples with thiourea dioxide. Slightly decrease was observed only on nylon part of multifibre material after stain washing fastness test of reductive cleaned samples with sodium dithionite and sodium hydroxide (Table-4).

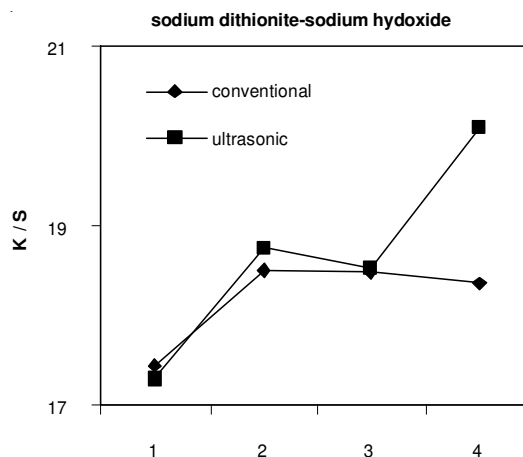


Fig. 6. K/S values of Na₂S₂O₄ - NaOH reductive cleaned dyed samples

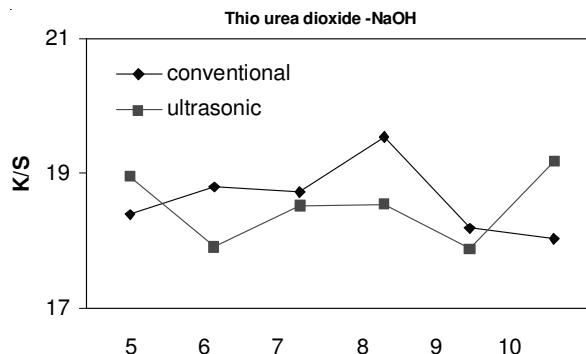


Fig. 7. K/S Values of thiourea dioxide-NaOH reductive cleaned dyed samples

TABLE-4 WASHING AND LIGHT FASTNESS RESULTS OF THE SAMPLES																
Test	Washing fastnesses (ISO 105 C06)														Light	
	Fade				Stain											
	CA		Co		PA		PES		PAN		Wo		k	u		
	k	u	k	u	k	u	k	u	k	u	k	u	k	u		
Na ₂ S ₂ O ₄ and NaOH																
Original	5	5	4/5	5	5	5	4/5	5	4/5	5	5	5	5	5	5/6	5/6
1	5	5	5	5	5	5	4/5	5	5	5	5	5	5	5	5/6	5/6
2	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5/6	5/6
3	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5/6	5/6
4	5	5	5	5	5	5	4/5	5	4/5	5	5	5	5	5	5/6	5/6
Thiourea dioxide and NaOH																
5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5/6	5/6
6	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5/6	5/6
7	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5/6	5/6
8	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5/6	5/6
9	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5/6	5/6
10	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5/6	5/6

Conclusion

By using environmentally friendly reductive cleaning substances instead of sodium dithionite which is widely used by the textile plants, it will be possible to decrease the environmental harmful substances of the waste water. High K/S values were obtained with the increasing of sodium hydroxide concentration at the reductive cleaned samples by using ultrasonic energy. K/S values of reductive cleaned samples with thio-urea dioxide and sodium hydroxide by using conventional method were higher. Total colour differences of reductive cleaned samples by using ultrasonic energy in different concentrations were acceptable also washing and light fastness properties were not affected negatively so ultrasonic process can be applied by the industrial plants because of its short process time and low energy requirement.

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