

## Analyses of some undesirable effects in industrial exhaust dyeing of knitted fabrics of different grades of cotton with reactive dyes

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*Dyeing of knitted fabrics from different grades of cotton in industrial conditions by a single recipe with bi-functional reactive dyes has been investigated in order to estimate the efficiency and reproducibility of the process. Quality control in different stages of the process was followed through the content of the non-cellulose components, alkaline and alkaline earth elements in raw and scoured cotton, the hydrophilic properties, and CIELab coordinates, color difference between different batch dyeing, purity of the electrolyte as well as the quality of the water through permanent hardness, temporary bicarbonates hardness, alkaline, earth-alkaline and heavy metal ions. Black dyed fabrics had high variance coefficients of  $\Delta E^*$ , indicating shade variations and unacceptable color levelness and reproducibility. Enzymatic scouring, quality of the raw cotton, water and electrolyte have significant influence on the dyed fabrics.*

**Key words:** scouring, cotton, reactive dyes, electrolyte, water

### 1. Introduction

Reactive dyeing of cellulosic materials is of great commercial importance, but it is also the dyeing process difficult to control. The growing application of reactive dyes requires out-standing grade to grade cotton level dyeing with good reproducibility and good fastness properties [1]. Dyers who are working with these dyes, have been faced with many problems during the dyeing as are: physical and chemical damage to the cotton during preparation, poorly penetrated streaky dyeing, reduced depth of shade, dulling of shade, un-level dyeing, poor shade reproducibility, reduced fastness, some dye-stuff swimming on the surface of the

fiber, cloudy dyeing, spots and stains which are difficult to remove, change of shade, and even contaminated machines which can run subsequent dyeing [2]. Reactive dyes are sensitive to alkali, chlorine, residual peroxide and metals.

The fixing of water-soluble reactive dyes on cellulose fibers is a heterogeneous reaction which takes place at the interphase between the dyeing solution and the fibers. Its proceeding includes diffusion of the dye in the bulk liquid phase, adsorption of the dye on the surface of the fibers, diffusion in the bulk of the fibers, adsorption on the inner surface of the fibers [3]. Each one of these steps can become limiting and hence determining the overall rate and the kinetic rela-

tions of the process. For successful dyeing, factors arising from the heterogeneity of the reaction (diffusion, reactivity, substantivity), process parameters (dye concentration, pH, temperature, process sequence, recipe, dyeing time) as controllable factors influencing the reaction, must be taken into consideration and properly set [4]. Even if dyers had essentially perfect control over the process, shade variations could sometime result from uncontrollable factors such as quality of the raw cotton, dyes and chemical variances, particular electrolyte and water quality. It is not possible properly to optimize processing conditions and equipment while important raw material and/or water variation is significant. Good water

treatment and well-prepared fabrics are essential [5]. Depending on shades desired, careful pre-treatment stages, enzyme or alkali scouring and peroxide bleaching before dyeing have essential influence on dyeing process. Therefore, it is important to devise dyeing protocols which are highly resistant to variations. Situations which lead to inherently unstable shade repeats can be identified by examination of dyebath data.

In this work the efficiency of the process, levelness and reproducibility was investigated by statistical analysis of *CIELab* coordinates of greige, scoured and dyed knitted fabrics and  $\Delta E^*$  between black dyed fabrics of both grades of cotton and standard for black color. Critical quality control factors as quality of raw cotton, water and electrolyte purity were also investigated.

## 2. Experimental

### 2.1. Materials

Single jersey weft-knitted fabrics made of combed ring-spun 100 % yarns of two different grades cotton, cotton 1 (India) and cotton 2 (Pakistan) were carried out from circular-knitting machines (Orizio JOHN/C, E 28, 30 coll). Tubular-knitted fabrics were kept on a flat surface for two weeks under standard atmospheric condition and after dry relaxing, structural characteristics (weight per unit area in  $\text{g/m}^2$ , thickness in mm, vertical and horizontal density in  $\text{cm}^{-1}$ ) were measured. In this manner analyses were made on 15 samples, 8 made of cotton 1, and 7 made of cotton 2. Greige knitted fabrics had following structural characteristics: weight per unit area from 118.21 to 155.01  $\text{g/m}^2$ , thickness from 0.50 to 0.62 mm, vertical density from 18.83 to 23.5  $\text{cm}^{-1}$ , horizontal density from 11.17 to 13.17  $\text{cm}^{-1}$ . The water used for this process was technical.

The chemicals used, were 96 %  $\text{C}_2\text{H}_5\text{OH}$  and  $\text{CH}_3\text{Cl}$  Alkaloid (Macedonia) of laboratory grade. Sodium

hydroxide, sodium chloride, sodium carbonate and acetic acid were chemicals of technical grade. A wetting agent Felosan NFG (BEZEMA), an alkaline pectinase for enzymatic scouring Beisol PRO (BEZEMA), a leveling agent Sarabit LDR (BEZEMA), a crease preventing agent based on polyamide Biavin BPA (BEZEMA), a sequestering and dispersing agent based on carbonic acids and polyphosphonate Heptol NWS (BEZEMA), a soaping agent Heragan CSB (BEZEMA), a cationic after treatment product for reactive dyed fabrics based on polyammonium compound, for improving wash, wet fastness without impairing the light fastness, shade, handle and re-wettability Revin ACP from BEZEMA (Switzerland). Black bi-functional monochlorotriazine-vinylsulphone (MCT/VS) reactive dye (Bezactiv S Cosmos S-MAX) from BEZEMA (Switzerland) was used. The standard for back color according to prospect of BEZEMA (Switzerland) has *CIELab* coordinates values  $L^* 0.74$ ,  $a^* 0.36$  and  $b^* 0.65$ .

### 2.2. Pretreatments and treatments

#### 2.2.1. Enzymatic scouring

Alkaline pectinase scouring was done using 2 % Beisol PRO 10:1 liquor ratio (LR) containing 1 % Felosan NFG as wetting agent and 1 % NaOH at 55 °C for 20 min. After that the temperature was raised to 80 °C for 20 min to stop enzymatic activity. Fabrics were rinsed twice at 80 °C for 10 min, pH dropped to 10.5-11 after first and to 9.7-10 after second rinsing.

After scouring, knitted fabrics were neutralized with 1 %  $\text{CH}_3\text{COOH}$  at 45 °C for 10 min (pH dropped to 4.9-5.7) and rinsed with cold water (pH between 7.5-7.8) and submitted to dyeing.

#### 2.2.2. Dyeing and finishing

The scoured samples under investigation were dyed with 6 % Bezactiv S Cosmos S-MAX. Dyeing took place

in winch dyeing machine at a LR10:1 in the presence of: salt, dye,  $\text{Na}_2\text{CO}_3$  and NaOH, recommended by prospect recipes of the producer. The dyeing started in the bath with 2 % Sarabit LDR, 1.5 % Biavin BPA, 1 % Heptol NWS, 0.5 %  $\text{Na}_2\text{CO}_3$  (pH 10) at 50 °C for 10 min. Then in 10 min time 100 g/L NaCl was added and the material continued to circulate in the next 30 min (pH between 8.3-9). In the next 10 min, 6 % Bezactiv Cosmos S-MAX was added and the material continued to circulate for 30 min (pH 7.6 to 8.2). Then 20 g/L  $\text{Na}_2\text{CO}_3$  at 50 °C was added in portions (pH between 10.3-10.6) and within 10 min the temperature was raised to 60 °C and after 20 min at 60 °C 1.7 g/L NaOH was added (pH between 11.3-12) and fixing the dye continued in the next 45 min. The fabric was then rinsed with cold water, neutralized with 1 %  $\text{CH}_3\text{COOH}$  at 45 °C for 10 min, soaped with 1 % Heragan CSB at 90 °C for 10 min and rinsed twice with hot water at 90 °C and 80 °C (pH dropped from 11.9, to 8.3 and 8.2, respectively). At the end knitted fabrics were softened with 3 % Dimicol Soft 6073, 0.5 % Rewin ACP, 0.3 %  $\text{CH}_3\text{COOH}$  at 45 °C for 10 min, dried and thermo stabilization by compacting.

### 2.3. Methods

#### 2.3.1. Wax content

Wax content, reported as the fraction (w/w) present on the dried fiber, was determined by six-hour Soxhlet extraction with chloroform with solvent siphoning every 3-4 min. Three replicates of 8 grams each were performed for each grade of cotton. The resultant solutions were evaporated at 105 °C, which left a wax residue that was subsequently weighted. Alcohol extractable substances, which are traditionally accepted as an indicator of the non-cellulosic content of cotton, were determined in the same way, except the solvent was absolute ethanol.

2.3.2. *Metals determination*

Preparation and analysis of samples by ashing followed the procedure previously reported [6]. Analysis were conducted using an atomic absorption (Perkin Elmer 700) spectrophotometer with an air/acetylene flame in the absorbance mode. Individual metal concentrations were determined from the calibration curves constructed with minimum of three standard concentrations. Dilutions made to obtain effective absorbencies at each metal respective wave lengths in their linear operating ranges were 1250X for potassium and 625X for both calcium and magnesium and 6000X for sodium analysis.

2.3.3. *Electrolyte purity*

Purity of the electrolyte was followed through content of chloride and alkaline earth elements. The chloride content was determined by Mohr method [7] while the Ca and Mg content was determined using an atomic absorption (Perkin Elmer 700) spectrophotometer.

2.3.4. *Water quality*

Analysis of water is made in laboratory of PE Water Supply and Sewerage, Skopje, Macedonia.

2.3.4. CIELab coordinates, whiteness index and color difference

The degree of whiteness (WI) and CIELab coordinates lightness  $L^*$ ,  $a^*$ ,  $b^*$ , chroma  $C^*$ , and hue  $H^*$  were mea-

sured on X-RITE CA 22 spectrophotometer (D65 light source, observer 10°) according to AATCC Evaluation Procedure 6 [8]. The degree of whiteness was expressed as Berger Whiteness. The differences between the standard and dyed cotton knitted fabrics were calculated as  $\Delta E^*$  values. Each reported value represents the average of five sample readings with a CV less than 5 %.

2.3.5. *The water absorbency test*

The water absorbency test was measured by AATCC Test Method of Evaluation of Wettability which determines fabric wettability by counting the elapsed seconds between the contact of water drop with the fabric and the disappearance of the drop into the fabric [9].

3. Results and discussion

Exhaust dyeing of knitted fabrics in dark shades is usually done by a two-stage process, scouring and dyeing with reactive dyes. Scouring can be done by enzymatic (acid or alkaline pectinase) or alkaline process. Location of the anomalies and their characterization during the technological process of industrial knitted fabrics dyeing in dark shade was done on 15 samples made from two grades of cotton with the characteristics previously done. The knitted fabrics were scoured with alkaline pectinase and dyed with black reactive dye. The

reproducibility of pre-treatment and dyeing processes between all the substrates under investigation (two different grades of cotton fibers, 15 repeating processes of scouring and dyeing) was determined by statistical analysis after each stage. CIELab coordinates  $a^*$ ,  $b^*$  and WI values of greige and scoured knitted fabrics are given in Fig. 1 and 2. The values of color coordinates  $a^*$  and  $b^*$  WI, before and after each treatment were statistically analyzed in order to validate each pre-treatment process in covering the differences in knitted fabrics from different grade cotton by statistical analysis of the mean deviation (M.D) and variance coefficient (V.C). The color coordinates  $a^*$  and  $b^*$  and WI values of greige knitted fabrics made of two different grades cotton fibers, as shown in Fig. 1 and 2, have  $a^*$  values lower or near to 1 negligible red hue,  $b^*$  values between 10.61 and 12.34, and WI values between 16.63 and 22.40 respectively, emphasizing yellow hue. After scouring, knitted fabrics are less red and less yellow with WI almost 15 units higher comparing with greige. The reproducibility of pre-treatment process among two grades of cotton fibers in several repeating processes of scouring under investigation was evaluated and the results obtained set in Tab.1. Both grades enzymatic scoured fabrics have similar CIELab coordinates and WI but different V.C. values indica-

Tab.1 Effect of scouring and dyeing with reactive dye on average color coordinates of knitted fabrics made of different grades of cotton; MD = Mean deviation, VC = Variance coefficient

Treatments	Substrate Number														
	1								2						
	No		$L^*$	$a^*$	$b^*$	$C^*$	$H^*$	WI	No	$L^*$	$a^*$	$b^*$	$C^*$	$H^*$	WI
Greige	4	x	85.32	0.97	12.6	11.95	85.44	16.95	7	84.91	0.85	11.4	11.39	85.58	19.69
		MD	0.97	0.13	0.35	0.51	0.5	0.54		0.74	0.16	0.49	0.50	0.42	1.89
		VC	1.13	12.37	2.88	0.58	0.58	3.18		0.87	18.82	4.22	4.38	0.47	9.54
Scouring	7	x	87.51	0.55	8.29	8.29	86.2	35.04	7	87.87	0.53	7.84	7.86	86.15	37.44
		MD	0.38	0.05	0.11	0.09	0.29	0.74		0.32	0.17	0.15	0.16	1.20	1.04
		VC	0.43	9.09	1.32	1.08	0.33	2.11		0.36	32.07	1.91	2.03	1.39	2.77
Dyeing-Thermostabilization	8	x	2.74	-0.87	0.76	1.18	138.93	-	7	2.79	-0.69	0.64	0.96	137.7	-
		MD	0.38	0.13	0.16	0.11	8.78			0.76	0.13	0.18	0.05	12.77	-
		VC	<b>13.86</b>	<b>14.94</b>	<b>21.05</b>	<b>9.32</b>	<b>6.32</b>			<b>27.24</b>	<b>18.84</b>	<b>28.12</b>	<b>5.20</b>	<b>9.27</b>	-

ting uneven pre-treatment scouring process (Tab.1).

The *CIELab* results of black dyed fabrics (Tab.1) showed high V.C., indicating unlevel dyeing. Fabrics made of cotton 2 have higher  $a^*$  and  $b^*$  V.C. than fabrics made of cotton 1, 18.84 %, 28.12 % and 14.94 % and 21.05 %, respectively. Calculated  $\Delta E^*$  values between black dyed fabrics and standard of black color showed high color differences,  $\Delta E^*$  higher than 1 (Fig.4). The dyed samples of both grades of cotton fibers have higher values of  $L^*$  compared to standard of black color indicating the significant reducing depth of shade (Fig.3).  $\Delta E^*$  values of fabrics made of cotton 1 and 2 which are presented on Fig.4 were between 1.44 and 2.45 for cotton 1 and between 1.84 and 3.10, for cotton 2. It means that there were visually noticeable differences in depth of the shade indicating poor reproducibility between fabrics made of different grades of cotton as well as between the fabrics from the same grade of cotton but from one batch to the others Fig.5.

From the results presented so far it can be concluded that the processes of enzymatic scouring and dyeing with black reactive dye were followed by certain anomalies.

There are many process parameters which can influence the final shade of any given reactive dye recipe. The shade can be influenced by the presence of impurities, especially metallic impurities. These metallic impurities include calcium, magnesium, copper, iron and manganese.

Calcium and magnesium are reported as the hardness of the bath. Most of the companies use soft water or use sequestering agent to soften the water but had failed to recognize the danger of hardness from other sources. Problems during dyeing with reactive dyes usually come from the temporary hardness. Not all reactive dyes are affected to the same extent, or in precisely the same way, but all are susceptible. Broadly speaking, the potential dangers of dyeing in the

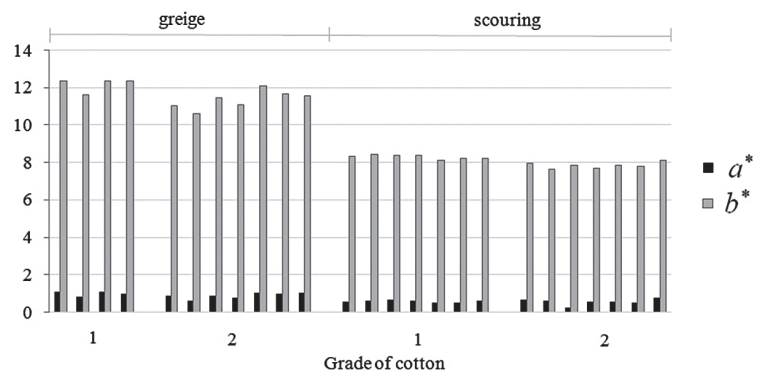


Fig.1 Color coordinates results of greige and enzymatic scoured cotton knitted fabrics from different grade cotton fibers

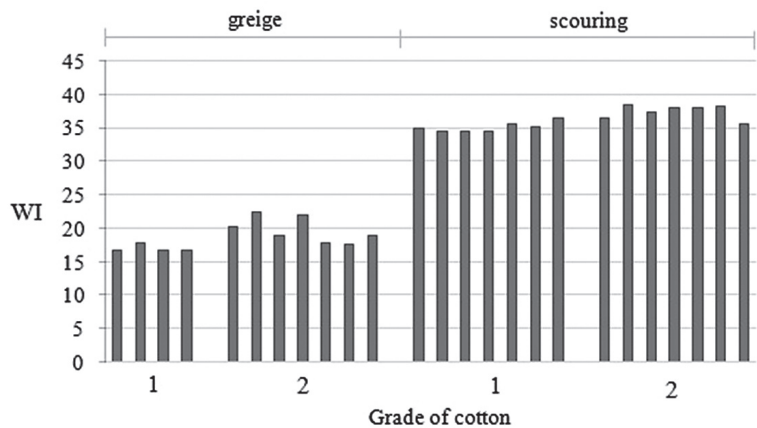


Fig.2 Whiteness index results of greige and enzymatic scoured cotton knitted fabrics from different grade cotton fibers

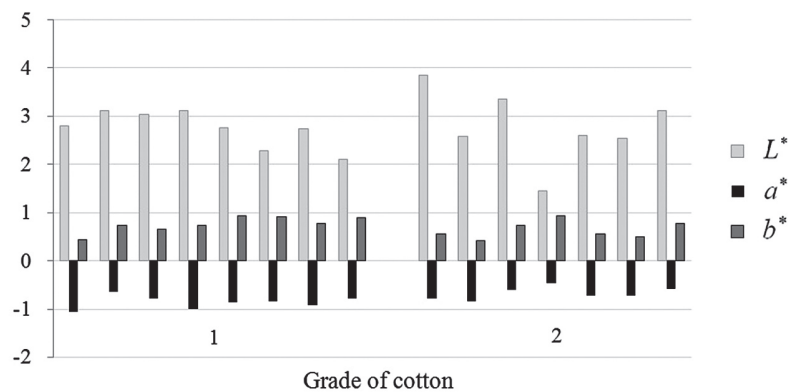


Fig.3 Color coordinates results of black dyed cotton knitted fabrics from different grade cotton fibers

presence of mainly metallic impurities are: reduced depth, dulling of shade, unlevel dyeing, poor shade reproducibility, reduced fastness, and quasi unlevelness (complain form

garment confection). The potential sources of hardness of the dyeing bath are: cotton, electrolyte, chemical auxiliaries, and water.

Tab.2 Metal contents in different grades of greige and scoured cotton; MD = Mean deviation, VC = Variance coefficient

Metallic ion (ppm)	Greige				Scoured			
	Substrate number		Statistical analysis		Substrate number		Statistical analysis	
	1	2	MD	VC	1	2	MD	VC
Ca	771.75	850.00	55.33	6.82	1015.25	747.25	189.50	21.50
Mg	479.75	462.50	12.20	2.59	200.00	169.50	21.57	11.67
K	3417.50	3630.00	150.26	4.26	116.25	101.50	10.43	9.58
Na	7442.50	8061.25	437.52	5.64	3020.00	2710.00	219.2	7.65
Ca+Mg+K+Na	12111.5	13003.75	630.92	5.02	4351.5	3728.25	440.7	10.91

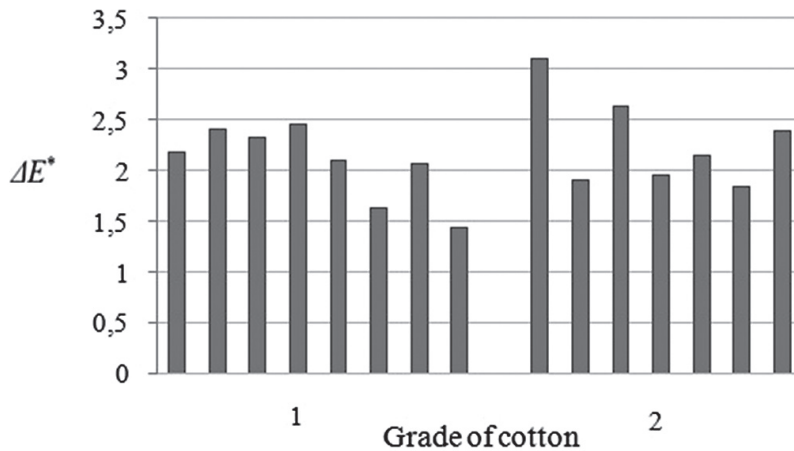


Fig.4 Color difference between standard for black color and black dyed knitted fabrics from two grade of cotton

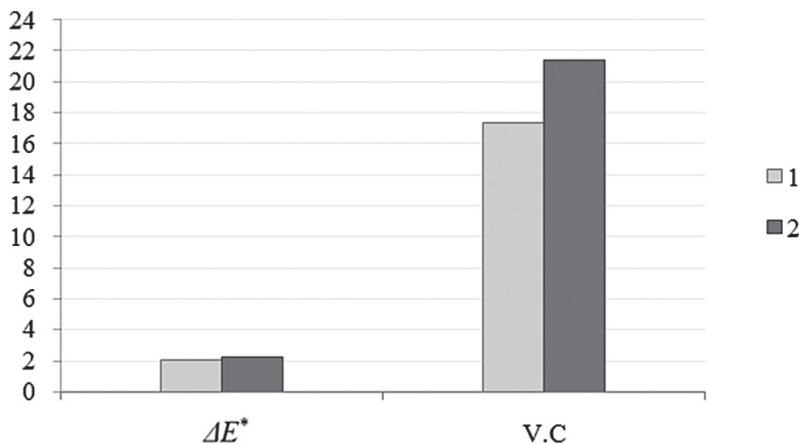


Fig.5 Statistical analysis (V.C) of the color difference in shade (ΔE\*) for knitted fabrics made of different grades of cotton from one bath to the others

The total level of impurities in cotton can range from 4 to 12 % of the total weight of fiber. The impurities mainly concentrated in the cuticle include pectin (0.7-1.2 %), protein (1.1-1.9 %), wax (0.4-1.0 %), ash (0.7-1.6 %), and others (resins, pigments, hemicellulose, sugars, organic acids) (0.5-1.0 %) [10].

As can be seen on Fig.6 the amount of waxes and total extractable cotton impurities obtained by chloroform and ethanol is small, with similar content, ranged from about 0.76 % to 0.90 % and 0.97 % to 1.06 % for cotton 1 and 2, respectively. It can be concluded that both grades of cotton used in our examinations have simi-

Tab.3 Quality of used water

Total hardness	dH°	14.88
pH	6,5	
P-alkalinity	-	
Ca	mg/L	85.50
Mg	mg/L	19,00
Na	mg/L	3.43
K	mg/L	0.29
Fe	mg/L	0.65
Mn	mg/L	2.54
Cu	mg/l	0.172
Cr	mg/l	0.528
Cl <sup>-</sup>	mg/L	23.00
SO <sub>4</sub> <sup>2-</sup>	mg/L	54.15
HCO <sub>3</sub> <sup>-</sup>	mg/L	274.15
NO <sub>3</sub> <sup>-</sup>	mg/L	4.48
NO <sub>2</sub> <sup>-</sup>	mg/L	-
NH <sub>4</sub> <sup>+</sup>	mg/L	-

lar concentrations of non-cellulosic components.

The levels of metallic impurity have significant influence on the shade of the dyed knitted fabrics. The main contributory factor include geology and the cultivation area, soil contamination, weather conditions during the maturing period, cultivation techniques, the use of chemical pesticides, de-foliant and fertilizers, and harvesting techniques. If calcium and magnesium ions are not sequestered, there is the strong possibility of their combining with natural waxes during the enzymatic scouring. These deposits of waxes on the cotton carried forward into dyeing can cause many problems. Metal content of two different grades of cotton are given in Tab.2. As can be seen from the results from the Tab.2 the content of Ca, Mg, K and Na in both grades of cot-

ton have similar values ranging in the class of cotton with lower content of metal ions. The results in Tab.2 showed that content of Mg, K and Na ions decreased during the scouring process among both grades of scoured cotton while content of calcium ions increased by 31.55 % on fabrics of cotton 1 and decrease by 12.08 % on fabrics of cotton 2.

Water, as another essential source of permanent or temporary hardness and heavy metals ions is of grate importance during dyeing with reactive dyes. The results of the analyses of water as show in Tab.3, indicated total hardness of 14.88 dH°, m-alkalinity, bicarbonate 274.15 mg/L, Fe 0.65 mg/L, Mn 2.54 mg/L and Cr 0.528 mg/L, without traces of acids, NH<sub>4</sub><sup>+</sup> and NO<sub>2</sub><sup>-</sup> and low content of NO<sub>3</sub><sup>-</sup> of 4.48 mg/L. Permitted amount of Fe, Mn and Cr can vary 0.02-0.1 mg/L [11]. The water hardness, 14.88 dH°, is caused by the presence of total content of Ca 85.5 mg/L and Mg 19 mg/L and their salts, bicarbonate 274.15 mg/L, chloride 23 mg/L and sulfate 54.15 mg/L. The consensus view with regard to hardness is that, for complete safety, reactive dyes should not be used in an aqueous environment exceeding 3 degrees German hardness [12]. Bicarbonates hardness of water, also called "temporary hardness", is the water hardness determined by bicarbonates, i.e. calcium bicarbonate Ca(HCO<sub>3</sub>)<sub>2</sub> and magnesium bicarbonate Mg(HCO<sub>3</sub>)<sub>2</sub>, which decomposed during heating, releasing CO<sub>2</sub> and precipitating as carbonates. During the dyeing process 1% Heptol EMG a sequestering agent was added to form a complex with metal ions and keep them in solution. According to the prospects of the producer of Heptol EMG Beze-ma, 1 % of it has complexing power of chelating alkali earth and heavy metal ions from 10 dH° bath hardness. But as can be seen from the results it was not enough for chelating all the metallic impurities in the used water.

The electrolyte NaCl is a significant source of temporary hardness and can be sourced from underground deposits or by evaporation of sea water. Unpurified rock salt from underground deposits, unpurified and purified sea salt by evaporation of sea water can contain high quantities of metallic impurities. So it was of interest to analyze the quality of the salt used during the dyeing with the black reactive dye. The purity of the used technical NaCl was 97.5 % with the calcium ions content of 0.628 % and magnesium ions content of 0.1175 %. After 100 g/L NaCl, electrolyte added, water hardness of the bath increased to 15.88 dH°, so 1 % chelating agent should reduce the hardness to 5.88 dH°.

Enzymatic scouring for both grades of cotton was performed in the bath with 14.88 dH° water hardness at pH 12, with 1 % Felosan NFG and 2 % Beisol PRO. Scoured fabrics were dried and hydrophilicity tested. Higher pH value of the scoured bath, decreased enzymatic activity and reduced the elimination of non-cellulosic components especially waxes. So residues of the waxes reduced the wettability of enzymatic scoured cotton fabrics, the time of disappearance of the water drop was higher than 5 s. After scouring and deactivation of the enzyme, the fabrics were several times rinsed with hot and cold water in order to remove residual alkali, neutralized with CH<sub>3</sub>COOH and then rinsed with cold water (pH dropped to 7.5 to 7.8). Effective rinsing and neutralization is a basic requirement for a successful start of the dyeing process. If the scoured fabrics are not completely neutralized and contained residual alkali, it could lead to premature dye fixation, before migration, resulting in uneven dyeing, premature hydrolysis with loss of dye and loss of wet fastness.

Scoured fabrics were submitted to dyeing at 60 °C with 6 % Bezactiv S Cosmos S-MAX black bi-funcional MCT/VS reactive dye by the previously discussed process: salt, dye,

Na<sub>2</sub>CO<sub>3</sub> at 50 °C and NaOH at 60 °C. Dyebath contained wetting agent, crease prevention agent, sequestering and dispersing agent based on carbonic acids and polyphosphonate, has pH 10 at 50 °C. The process continues by subsequent addition of 100 g/L NaCl, in portions. After adding 100 g/L NaCl, water hardness increased to 5.88 dH°. Calcium and magnesium ions present in water and salt increased the hardness of dyebath probably reducing the solubility of anionic dyes leading to greater risk of unevenness. Gradual addition of the dye into the bath with the hardness of 5.88 dH°, decreased the pH to 7.6-8.2. Weak alkaline conditions contributed to the activation of passive VS reactive group. But, this pH was not high enough for fixation, and formation of covalent bonds. With the addition of Na<sub>2</sub>CO<sub>3</sub> and NaOH, at 60 °C the pH risen to 10.3-10.6 and to 11.3-12, respectively and conditions for establishing covalent bonds between both functional groups were established.

After dyeing knitted fabrics were submitted to cold rinsing at 20 °C (pH 11), removing of the hydrolyzed dye, neutralized (pH 9), soaped at 95 °C (pH from 8 to 8.5), hot rinsed at 80 °C (pH 8.2) and a cold rinsed at 20 °C for complete removal of the hydrolyzed dye and softened at pH 7.4. However, increased hardness of the dyebath caused reduced depth of the shade (higher *L\** values), poor shade reproducibility ( $\Delta E^*$  higher than 1).

#### 4. Conclusion

Summarizing the results presented it can be concluded that the process of enzymatic scouring of the knitted fabrics and dyeing with black reactive dye used as single recipe to yield single results is not effective and stable to produce black dyed knitted fabrics with outstanding grade to grade cotton and batch to batch dyeing. Inappropriate scouring formulation, increased hardness and pH of the bath, gave scou-

red fabrics accompanied by wax residues and metallic impurities without satisfactory hydrophilicity and uniformity. Inappropriate quality of water and quantity of chelating agent reduced the depth of the shade and gave poor shade reproducibility.

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