



## PHYSICO-CHEMICAL PARAMETERS OF RESIDUAL WATER FROM DIFFERENT SCOURING TREATMENTS OF HEMP/COTTON FABRIC

DOCHIA Mihaela<sup>1</sup>, GAVRILAȘ Simona<sup>2</sup>

<sup>1</sup> "Aurel Vlaicu" University of Arad, Research Development Innovation in Technical and Natural Science Institute,  
Postal address, 310330, 2-4 Elena Dragoi Street, Arad, Romania, E-Mail: [dochiamihaela@yahoo.com](mailto:dochiamihaela@yahoo.com)

<sup>2</sup> "Aurel Vlaicu" University of Arad, Faculty of Food Engineering, Tourism and Environmental Protection, Department of  
Technical and Natural Sciences, Postal address, 310330, 2-4 Elena Dragoi Street, Arad, Romania, E-Mail:  
[simona2213@yahoo.com](mailto:simona2213@yahoo.com)

Corresponding author: Gavrilaș, Simona, E-mail: [simona2213@yahoo.com](mailto:simona2213@yahoo.com)

**Abstract:** *Impact of economic development - ecological balance rises numerous and complex problems for the textile industry. It is known that all textile processes need important quantities of water and chemicals. This situation generates large amounts of wastewater which cannot be further use without additional treatments because of the environmental problems. The issue of environmental protection has become an essential part of the textile industry development strategy. Many studies in the field present alternatives for the conventional scouring to reduce water pollution. The applied ecological strategy is focused on process optimization and pollution prevention.*

*The paper presents a comparison of the physico-chemical parameters of the wastewater resulted from the various scouring treatments applied on a cotton/hemp fabric. Three types of scouring treatments have been carried out: two with enzymes for which a commercial enzyme product, a washing agent and EDTA or sodium citrate as complexing agents were used, and one classical scouring treatment with sodium hydroxide. The main physico-chemical parameters of the residual water analyzed were: pH, salinity, turbidity, TDS (total dissolved solids), conductivity, chemical oxygen demand (CCOMn), total dissolved oxygen and dry residue.*

*From the two enzymatic treatments, the values of physico-chemical parameters of the residual water were in accordance with the legislation in case were the sodium citrate was used. For the alkaline treatment, the data were even with 100 % higher compared to enzymatic treatments. For some parameters, higher values were registered also for the enzymatic scouring where EDTA was used as a complexing agent.*

**Key words:** *wastewater, physico-chemical analysis, pretreatment, ultrasound, commercial enzyme product.*

### 1. INTRODUCTION

Cotton fibres are characterised by a trilamellar structure. It is considered that the second layer has in its composition de-esterified pectins. Those may be a result or contribute to the fibre cells elongation [1]. Both cotton and hemp fibres are characterised by a certain amount of native pectin. For further utilisation in the textile industry it is mandatory to remove all noncellulosic components, along with other impurities: lignin, waxes, natural pigments, or organic acids. Independent of the classical method chosen (acid, alkaline or peroxide scouring) the main objectives of the treatment are to be non harmful for cellulose fibre and remain with less impurities residues. In



case of the hemp fibres it was shown that a scouring treatment which includes sodium hydroxide and sodium sulphite is recommended for the removing of both lignin and pectin and also to reduce the treatment time [2].

Using a conventional pretreatment on cotton/hemp fabric with high temperatures (80-90 °C) and aggressive alkaline ( $\text{pH} \geq 10$ ) will have a major negative impact on the ecosystems. In this situation the wastewaters resulted have to be subject to additional purge steps to accomplish the environmental requirements [3].

Impact of economic development - ecological balance rises numerous and complex problems for the textile industry. Textile ecological technologies occupy an important place in the research projects developed at national and international levels. The issue of environmental protection has become an essential part of the textile industry development strategy. Many studies in the field present alternatives for the conventional scouring. The applied ecological strategy is focused on process optimization and pollution prevention. The suggested treatments imply the utilization of enzymatic products which are not harmful, are suitable for the process, economically advantageous by decreasing the quantity of energy, water and chemicals used, and eco friendly [4]. Other advantages of using an enzymatic treatment are the lower fibres damage degree and the utilisation of biodegradable chemicals. Replacement of the classical scouring treatment with the enzymatic one reduces the pollution factors and also streamlines the technological process from an economic point of view. Another aspect which should be taken into account is the lower quantity of effluents resulted after bioscouring treatment [5,6].

It is known that all textile process needs important quantities of water and chemicals. This situation generates large quantities of wastewater which cannot be further use without additional treatments due to the fact that they will probably cause environmental problems. The specific regulations demand a serious of precise determinations for: COD, pH, turbidity, salinity, TDS, and others. Good results have been reported in case of using different physical methods for wastewater treatment. In case of the ultrafiltration-electrodialysis system the values of the determined parameters were lower than the detection limit. This suggests that the treated water could be reintegrated in the technological process [7].

Independently of the method used for the wastewater treatment (coagulation-flocculation, oxidation, photocatalytic, chlorination, adsorption, electrocoagulation-ozone) all present different inconvenient and requires additional costs. This suggests a different approach of the problem. A development of modern techniques and technologies for reducing the amount of pollutants in the resulting effluents are needed. A possibility is the development of an ecological process for the pretreatment of the fabrics by using enzymes and biodegradable reagents. The main advantages of using enzymes are associated with less severe reaction conditions, lower processing temperatures, reduced treatment time, non-toxic and biodegradable products. Also, the water consumption is lower compared with the classical treatment and the wastewater parameters are in the limits specified by the legislation. For example, in case of a bioscouring cotton knits the pH and CCOMn values registered for the waters resulted from the process could be with approximately 50 % lower compared with the alkaline treatment, and the TDS is 10 % of the one registered for the classical treatment [8]. The lack of supplementary treatment applied to the waters resulted from the technology imply the process costs reduction. It is believed that up to one fifth of the water pollution could be determined by different specific textile processes [9].

The bioscouring treatment applied in our case tends to reduce the aggressive conditions compared to the conventional one. The working temperature had lower value (55 °C) and the pH was close to 7. As presented in the literature, also in our study the pectinolytic treatment used show similar efficiency as the classical one regarding the specific parameters of the fabrics (wettability, tensile strength, elongation at break, etc.).



## 2. EXPERIMENTAL PART

### 2.1 Materials and experimental procedure

The wastewater analysed was resulted after the bioscouring and alkaline treatment on cotton/hemp materials with the following characteristics: width ( $120 \pm 3$  cm), weight ( $220 \pm 10$  g/m<sup>2</sup>), warp density (10 yarns/cm), weft density B (10 yarns/cm), 100 % of cotton yarn, Nm 14 for warp direction and 100 % of hemp yarn, Nm 14 for weft direction.

The specific reagents have analytical purity and were purchase from: CHT Bezema Company (Beisol PRO-pectinolytic product, Denimcol Wash RGN- surfactant), Sigma-Aldrich (sodium hydroxide, sodium citrate, sodium carbonate, sodium silicate, sodium bisulfite), and Rotta Company (Sulfolen 148: S-148 alkyl polyglycol ether).

During the bioscouring treatment of the cotton/hemp material were used two different complexing agents EDTA or sodium citrate and the same enzymatic product (Beisol PRO-mixture of pectinase) in similar concentration and treatment time after an experimental program (see Table 1 and Table 2). The considered liquid to fabric ratio was 20:1, and the temperature 55 °C. The basic pH in case of the classical method was ensured with sodium hydroxide.

The pectinolytic product concentration used varied between 1-3 % o.w.f (over weight fiber) and the action time was between 15 and 55 minutes. The quantity of complexing agents (EDTA or sodium citrate) was 2 g/L and the surfactant (Denimcol Wash RGN) 0.5 %.

For the bioscouring treatments were used also ultrasound of 45 kHz in an Elmasonic X-tra basic 2500 ultrasonic bath from Elma Company, Germany. The classical scouring treatment used for comparison was done with 10 g/L sodium hydroxide, 1 g/L sodium bisulfite, 5 g/L sodium carbonate, 2 g/L sodium silicate and 2 g/L wetting agent (Sulfolen 148) for 1 hour at 100 °C in an AATCC Launder Ömeter.

### 2.2 Wastewater analysis

The wastewater quality indicators determined such as: pH, salinity, turbidity, TDS (total dissolved solids), conductivity, chemical oxygen demand (CCOMn), total dissolved oxygen and dry residue were chosen according to the EU specific regulations. A WTW multi-parameter inoLab Multi 740 was used for conductivity, pH, salinity, TDS, and total dissolved oxygen measurements. For turbidity determination was used a HI 88713 HANNA Instruments Turbidimeter. The dry residue was determined gravimetrically as difference in mass before and after the drying process. For evaporation was used a Pura 14 water bath from Julabo, Germany and dried at ( $105 \pm 5$ ) °C to constant mass in an oven, according to [10]. The CCOMn (chemical oxygen demand) was performed by titration with KMnO<sub>4</sub> as described by R. Ballance [11].

## 3. RESULTS AND DISCUSSIONS

The purpose of this research was to study the impact of the wastewater resulted from two eco-friendly scouring treatments compared to the classical alkaline scouring. Further on will be presented the comparative results obtain for the investigated parameters in case of using EDTA or sodium citrate as complexing agents in the bioscouring treatment, and for the traditional scouring with sodium hydroxide. Table 1 shows a comparative analysis of some quality indicators (pH, TDS, salinity, conductivity, total dissolved oxygen) of the residual water resulted from classical and enzymatic pretreatments of the cotton/hemp fabric.



**ANNALS OF THE UNIVERSITY OF ORADEA  
FASCICLE OF TEXTILES, LEATHERWORK**

*Table 1: Comparative analysis of some residual water quality indicators resulted from classical and enzymatic treatments of the cotton/hemp fabric*

<b>Scouring with enzymes + sodium citrate</b>							
Sample	Enzyme [%]	Treatment time [min.]	pH	TDS [mg/L]	Salinity	Conductivity [( $\mu$ s/cm)]	Total dissolved oxygen [mg/L]
1	1.30	21.00	8.02	1778.00	0.90	1780.00	5.27
2	2.70	21.00	7.81	1932.00	1.00	1936.00	5.43
3	1.30	49.00	8.18	1768.00	0.90	1776.00	5.04
4	2.70	49.00	8.05	1912.00	0.90	1917.00	4.92
5	1.00	35.00	8.53	1778.00	0.90	1785.00	4.82
6	3.00	35.00	7.82	2027.00	1.00	2030.00	4.23
7	2.00	15.00	7.65	1914.00	0.90	1918.00	6.86
8	2.00	55.00	8.30	1856.00	0.90	1863.00	5.27
9	2.00	35.00	8.05	1883.00	0.90	1888.00	5.26
10	2.00	35.00	8.15	1885.00	0.90	1893.00	5.15
11	2.00	35.00	7.93	1916.00	0.90	1923.00	4.91
12	2.00	35.00	7.99	1887.00	0.90	1891.00	4.93
13	2.00	35.00	8.10	1881.00	0.90	1886.00	4.76
<b>Scouring with enzymes + EDTA</b>							
1	1.30	21.00	4.849	1072.00	0.50	1071.00	5.34
2	2.70	21.00	4.864	1183.00	0.50	1182.00	5.53
3	1.30	49.00	4.933	1039.00	0.50	1038.00	5.15
4	2.70	49.00	5.082	1199.00	0.50	1197.00	5.65
5	1.00	35.00	4.849	998.00	0.40	997.00	5.71
6	3.00	35.00	4.931	1209.00	0.60	1209.00	5.77
7	2.00	15.00	4.887	1105.00	0.50	1105.00	5.79
8	2.00	55.00	4.920	1107.00	0.50	1104.00	5.64
9	2.00	35.00	4.836	1112.00	0.50	1111.00	6.17
10	2.00	35.00	4.868	1107.00	0.50	1104.00	5.93
11	2.00	35.00	4.927	1101.00	0.50	1101.00	6.26
12	2.00	35.00	5.010	1109.00	0.50	1108.00	6.13
13	2.00	35.00	5.018	1109.00	0.50	1109.00	6.29
<b>Classical alkaline scouring</b>							
1	-	60.00	13.50	53000.00	35.00	53000.00	4.16

In the textile processing units, pH is a very important factor and must be adjusted for each processing step for better results. The pH of the wastewater resulted from the three types of scouring treatments was found in a big range from ~ 4.50 to 13.50. In the case of pretreatment with sodium citrate, the pH of the wastewater is almost neutral, being in accordance with the legislation. Not the same situation is in the case of EDTA where the pH values are below 5, requiring a slight correction. As for salinity, this shows higher values for sodium citrate treatments. The highest value of pH shows the residual water resulting from the alkaline treatment (13.50), this being far beyond the limits allowed.

Electrical conductivity and TDS of wastewater from all enzymatic treatments were found to be in the range of ~ 1000 to 2000. Lower conductivity and TDS values were obtained in this case for the enzymatic treatments in which EDTA was used as a complexing agent. The same situation is for salinity values. Close values have been obtained for total dissolved oxygen in both cases of enzymatic treatments. In the case of classical alkaline treatment, with the exception of total



**ANNALS OF THE UNIVERSITY OF ORADEA  
FASCICLE OF TEXTILES, LEATHERWORK**

dissolved oxygen, the other values obtained for conductivity, TDS and salinity exceed the admissible values.

In Table 2 is presented a comparative analysis of turbidity, dry residue and chemical oxygen demand values of the residual water resulted from the classical and enzymatic treatments of the cotton/hemp fabric.

*Table 2: Comparative analysis of turbidity, dry residue and chemical oxygen demand of the residual water resulted from classical and enzymatic treatments*

<b>Scouring with enzymes + sodium citrate</b>					
Sample	Enzyme [%]	Treatment time [min.]	Turbidity [NTU]	Dry residue [mg/L]	CCOMn [mg O <sub>2</sub> /L]
1	1.30	21.00	2.85	19.26	19.64
2	2.70	21.00	2.98	26.36	21.71
3	1.30	49.00	2.49	29.28	29.07
4	2.70	49.00	2.61	28.00	32.34
5	1.00	35.00	2.63	25.64	22.86
6	3.00	35.00	2.71	36.70	27.81
7	2.00	15.00	3.31	15.16	18.96
8	2.00	55.00	1.92	57.60	25.28
9	2.00	35.00	2.48	34.96	27.81
10	2.00	35.00	2.60	36.88	22.64
11	2.00	35.00	2.90	35.96	25.28
12	2.00	35.00	3.09	38.40	25.28
13	2.00	35.00	2.74	40.86	28.96
<b>Scouring with enzymes + EDTA</b>					
1	1.30	21.00	1.64	34.34	644.64
2	2.70	21.00	1.59	46.56	620.80
3	1.30	49.00	1.49	32.00	379.20
4	2.70	49.00	1.56	60.06	606.72
5	1.00	35.00	1.61	31.06	379.20
6	3.00	35.00	1.41	37.20	442.40
7	2.00	15.00	1.83	20.44	265.44
8	2.00	55.00	1.20	64.82	689.60
9	2.00	35.00	1.36	34.44	151.68
10	2.00	35.00	1.51	35.26	145.36
11	2.00	35.00	1.46	37.98	170.64
12	2.00	35.00	1.84	33.32	139.04
13	2.00	35.00	1.60	36.00	169.60
<b>Classical alkaline scouring</b>					
1	-	60.00	84.00	1150	1180.00

Turbidity values are higher for sodium citrate treatments. Instead, similar values were obtained in the case of dry residue and much lower values for CCOMn compared to the treatments in which EDTA was used. In the case of classical alkaline treatment, the same high values are observed for all parameters (turbidity, dry residue, CCOMn) resulting high polluted wastewaters.

#### **4. CONCLUSIONS**

The main physico-chemical parameters of the residual water resulted from two types of enzymatic scouring treatments were analyzed in comparison with wastewater parameters of an classic alkaline treatment. Based on the experimental data obtained it was concluded that for



enzymatic pretreatments (EDTA or sodium citrate) all the parameters are in the limits with the exception of the pH for treatments where EDTA was used. We can not say the same thing in the case of classic alkaline treatment where higher values were obtained for all parameters. A constant monitoring of water quality is necessary to avoid further dreadful conditions. The effluents which are toxic in nature are needed imperative treatment before disposal on water bodies to create less pollution and an eco-friendly environment.

### ACKNOWLEDGEMENTS

This work was supported by a grant of the Romanian National Authority for Scientific Research and Innovation, CNCS-UEFISCDI, project number PN-II-RU-TE-2014-4-1370, and

„Centru de Cercetare în Științe Tehnice și Naturale-CESTN” co-funded by European Union through European Regional Development Fund Structural Operational Program “Increasing of Economic Competitiveness” Priority axis 2. Operation 2.2.1. POSCCE Nr. 621/2014 POS-CCE.

### REFERENCES

- [1] K. C. Vaughn, R. B. Turley, “*The primary walls of cotton fibers contains an ensheathing pectin layer*”, *Protoplasma*, vol. 209, pp. 226-237, 1999.
- [2] H. M. Wang, R. Postle, R. W. Kessler, W. Kessler, “*Removing Pectin and Lignin During Chemical Processing of Hemp for Textile Applications*”, *Textile Res. J.*, vol. 73, pp. 664-669, 2003.
- [3] C. R. Holkar, A. J. Jadhav, D. V. Pinjari, N. M. Mahamuni, A. B. Pandi, “*A critical review on textile wastewater treatments: Possible approaches*”, *J. Environ. Manage.*, vol. 182, pp. 351-366, 2016.
- [4] G. Garg, A. Singh, A. Kaur, R. Singh, J. Kaur, R. Mahajan, “*Microbial pectinases: an ecofriendly tool of nature for industries*”, *Biotech.*, vol. 6:47, 2016, DOI 10.1007/s13205-016-0371-4.
- [5] K. Mojsov, “*Bioscouring and bleaching process of cotton fabrics-an opportunity of saving water and energy*”, *J. Text. I.*, vol. 107, pp. 905-911, 2016.
- [6] F. El-Gohary, N. A. Ibrahim, F. Nasr, M. H. Abo-Shosha, H. Ali, “*A new approach to accomplish wastewater regulation in textile sector: an egyptian case study*”, *Cellulose Chem. Technol.*, vol. 47 (3-4), pp. 309-315, 2013.
- [7] R. Lafi, L. Gzara, R. H. Lajimi, A. Hafiane, “*Treatment of textile wastewater by a hybrid ultrafiltration/electrodialysis process*”, *Chem. Eng. Process.*, vol. 132, pp. 105-113, 2018.
- [8] E. K. Choe, C. W. Nam, S. R. Kook, C. Chung, A. Cavaco-Paulo, “*Implementation of Batchwise Bioscouring of Cotton Knits*”, *Biocatal. Biotransfor.*, vol. 22 (5/6), pp. 375-382, 2004.
- [9] S. M. A. Hoque, A. Y. M. A. Azim, “*Using Enzymes as an Aid of Better and Eco-Friendly Scouring Processing*”, *AJER.*, vol. 6 (5) pp. 167-182, 2016.
- [10] DS 204:1980 (in compliance with SFS 3008, NS 4764, and SS 028113); “*Determination of total residue and total fixed residue in water, sludge and sediment*”.
- [11] R. Ballance, “*Physical and chemical analyses, Water Quality Monitoring*”, in J. Bartram, R. Ballance (Eds), *A Practical Guide to the Design and Implementation of Freshwater Quality Studies and Monitoring Programmes*, UNEP/WHO ISBN 0 419 22320 7 (Hbk) 0 419 21730 4 (Pbk), 199.