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3rd INTERNATIONAL CONGRESS OF INNOVATIVE *Textiles*

PROCEEDINGS

18-19 *May* 2022
ONLINE CONGRESS

TEKİRDAĞ NAMIK KEMAL UNIVERSITY
2006

3rd International Congress of Innovative Textiles
ICONTEX 2022 (18-19 May 2022)

ISBN: 978-605-4265-68-8

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With Our Thanks…

**listed in alphabetical order*

Keynote Speakers

Prof. Dr. Ray H. BAUGHMAN

The University of Texas at Dallas, Alan MacDiarmid Nanotech Institute, U.S.A.
"Powerful, Large Stroke Electrochemical Carbon Nanotube Yarn Artificial Muscles"

Prof. Dr. Long LIN

University of Leeds, Department of Colour Science, U.K.

"Recent Advances in Sustainable Dyeing Technologies"

Prof. Dr. Xungai WANG

Deakin University, Institute for Frontier Materials, Australia

"Fibre Materials Research that Makes a Difference"

Prof. Dr. Paul KIEKENS

Ghent University (retired from), Belgium

"Textiles and Clothing: Challenges for an Increasingly Environmentally Demanding World"

Scientific Programme

3rd International Congress of Innovative Textiles, ICONTEX2022 (18 - 19 May 2022)
PROGRAMME

18 May 2022, Wednesday

09:30-10:30	**HALL A** **OPENING CEREMONY** **Prof. Dr. Fatma GÖKTEPE, Chairperson of Organizing Committee** **Prof. Dr. Lokman Hakan TECER, Dean of Çorlu Engineering Faculty** **Ahmet ÖKSÜZ, Chairman of İstanbul Textile and Raw Materials Exporters Association (İTHİB)** **Mustafa GÜLTEPE, Chairman of İstanbul Apparel Exporters Association (İHKİB)** **İsmail GÜLLE, Chairman of Turkish Exporters Assembly (TİM)** **Prof. Dr. Mümin ŞAHİN, Rector of Tekirdağ Namık Kemal University**		
10:30-10:45	**BREAK**		
10:45-11:30	**HALL A** **PLENARY TALK:** **Prof. Dr. Ray H. BAUGHMAN , University of Texas at Dallas, U.S.A.** **"Powerful, Large Stroke Electrochemical Carbon Nanotube Yarn Artificial Muscles"**		
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14:15-15:15	**HALL A** **Technical Textiles** **Chairman: Prof. Dr. Mirela BLAGA (Romania)**	**HALL B** **Weaving, Knitting and Nonwoven Technologies** **Chairman: Prof. Dr. H. Ziya ÖZEK (Turkey)**	**HALL C** **Nanotechnology Applications in Textiles** **Chairman: Prof. Dr. Ali Akbar GHAREHAGHAJI (Iran)**
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19 May 2022, Thursday

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10:15-11:15	**HALL A** **Functional and Smart Textiles** **Chairman: Prof. Dr. Emel ÖNDER KARAOĞLU (Turkey)**	**HALL B** **Fibre Science and Technology/Nanotechnology Applications in Textiles** **Chairman: Prof. Dr. Yasemin KORKMAZ (Turkey)**	**HALL C** **Textile Pretreatment, Coloration and Finishing Technologies Chairman: Prof. Dr. Erhan ÖNER (Turkey)**
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11:30-12:30	**HALL A** **Technical Textiles** **Chairman: Prof. Dr. Saber BEN ABDESSALEM (Tunisia)**	**HALL B** **Weaving, Knitting and Nonwoven Technologies** **Chairman: Prof. Dr. Yordan KYOSEV (Germany)**	**HALL C** **Textile Pretreatment, Coloration and Finishing Technologies** **Chairman: Prof. Dr. Habip DAYIOĞLU (Turkey)**
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PREFACE

Textiles, being a craft in early times but then evolving into a huge technology, was the pioneering force for industrial revolution following application of steam power to textile machinery. Furthermore, it would be right to say that it presented foundation of digital age when one considers holed cards developed by Jacquard for weaving well-designed fabrics. As we all know, the first step in developing high value added products begins with innovation and only those who are able adopt this in a very short time would be able to have sustainable competition in this world. And textiles today has become an innovative field covering a wide area ranging from industrial applications to artificial organs, civil engineering to electronics, protective clothes to automotives as some examples of these applications are presented in this Proceedings.

When we look at the global picture of the industry, we see that global market size of textiles has reached 1.6 trillion USD, of which approximately 180 billion USD is made of technical textiles. And global picture of the sector shows us that developed countries, opposite to the common feeling, have not withdrawn from textiles but instead focused on high value added fashion products and technical textiles. In this direction, it is a clear necessity for our industry to follow the trends very closely as well as the technological developments in the world, to read the next 5, 10 and 20-year periods correctly and to determine its strategies in this direction. The Turkish textile industry is among the most important players in the world. Behind this, there is both a centuries-old textile tradition and a strong textile education that goes back many years. In addition, the demand for textile engineering by talented young people and distinguished minds for many years and the work of very successful engineers in this sector have also play a significant role in this success. However, we are all aware of the fact that we need to be successful in R&D and innovation efforts in order to realize a transformation that will carry this sector into the 22nd century. As Tekirdağ Namık Kemal University, Çorlu Faculty of Engineering, Textile Engineering Department, this was the main thought and emotion behind organising 1st of ICONTEX, International Congress of Innovative Textiles back in 2011, and we organize the 3rd of this congress with similar thoughts and enthusiasm. In this respect, we aimed to gather researchers and textile professionals to promote advanced technologies and innovations in textiles. We have tried to fulfil this aim by devoted work of our Organizing Committee, which has been going on for almost a year, as well as the local and national industry and institutions that have shared similar vision with us and supported this organization generously. However, since we could not predict the course of the Covid-19 pandemic, we've decided to organise ICONTEX2022 as online this time.

At ICONTEX2022, we aim to share the latest innovations in textiles from fiber to fabrics in textiles together with our distinguished Scientific Committee members from 22 different countries (28 members from abroad, 17 members from Turkey); 89 oral presentations and 24 poster presentations from 12 different countries; our distinguished plenary session speakers from U.S.A., U.K., Australia and Belgium sharing their outstanding research results and future vision with our participants and over 300 participants from various regions of the world during three parallel sessions in two days.

The content of this congress book includes topics of;

- Fibre science and technology
- Yarn spinning technologies
- Weaving, knitting and nonwoven technologies

- Textile pretreatment, coloration and finishing technologies
- Textile chemistry
- Textile testing and quality control
- Textile modeling and simulation
- Technical textiles
- Functional and smart textiles
- Nanotechnology applications ın textiles
- Ecology and sustainability
- Textile and fashion design
- Apparel technology
- Textile management

Before ending this preface, I would like to thank our Rector Prof. Dr. Mümin Şahin, our Vice-rectors and our Dean Prof. Dr. Lokman H. Tecer for their continuous support. I also would like to express our gratitude to each institution and company that supported our congress as a sponsor for their valuable contributions. Last but not least, I would like to thank our organizing committee members, Prof. Dr. Rıza Atav, Prof. Dr. Pelin Gürkan Ünal and Res. Assist. Volkan Yalı, for their great effort during last one year.

With the hope of ICONTEX2022 Proceedings being useful for textile researchers and technologists…

On Behalf of Organizing Committee

Prof. Dr. Fatma Göktepe

ORAL PRESENTATIONS

SECTION IV: TEXTILE PRETREATMENT, COLORATION AND FINISHING TECHNOLOGIES / TEXTILE CHEMISTRY

VAT DYEING PROCESS OF INHERENT FLAME RETARDANT FABRICS

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ABSTRACT

The paper will present an experiment of vat dyeing of specific fabrics with protective, fire retardant properties that have a high content of aramid fibres in their composition. The research was performed on fabric samples that differ in raw material composition and aramid content. Also, the samples were dyed in raw from (group 1) as well as after pre-treatment with alkaline scouring (group 2). Based on the values obtained by measuring the LOI index, it was shown that the selected fabrics meet the properties of inherently FR fabrics. Dyeing was performed with Indanthren® Olive Green HB Colloisol (manufactured by DyStar) vat dye, in exhaustion process, with a bath ratio of 1:30. The dye concentration was 3%, and Na-hydrosulphite (Na2S2O4) was used as a reducing agent. The colouristic analyses were performed based on spectrophotometric measurement and results interpretation according to CIELAB system. The evaluation of primary tactile properties was performed which shown the increase of smoothness and softness after scouring and dyeing. Also, wash fastness as well as light fastness tests shown satisfactory fastness properties.

Keywords: Inherently flame retardant fabric, vat dyeing, colour characterization, K/S, wash fastness, light fastness, primary tactile properties.

1. INTRODUCTION

There are two main directions in current research in the field of non-flammability of textiles: research of flame-retardants of innovative structure and pre-treatment methodologies, and research into inherently non-flammable compositions. While the first method of finishing treatment aimed in achieving the fire resistance properties of fabric, has been developed early as in 1735, the development of inherently fire-retardant fabrics started some 60 years ago. Inherently flame-retardant fabrics do not need any of external finish or treatment to achieve the optimal level of flame retardancy [1]. In general, there are three types of inherently fire retardant fabrics: one involves fibres pre-treatment and modification during production, the other refers to fibres that modify the structure to achieve fire retardancy without the need for further processing and treatments, and the third group includes inorganic and ceramic fibres [2-4]. Composition of inherently fire retardant fabrics is often characterized by high content of aramid fibres, which is why the processes of applying dyes by dyeing or printing to such fabrics are highly demanding. Difficulties in the processes of aramid fibres dyeing arise from their chemical structure, which implies a high degree of order of macromolecules in the polymer chain, high crystallinity and density of the structure, as well as from their pronounced base colour [5]. Given the problematic nature of the application of traditional dyeing techniques, the literature finds research on the application of some treatments or process modifications [5-9]. Holsten and Smith patented the cationic dying of aramid fibres using the N,N-diethyl m-toluamide as a carrier [10]. Tajul et al. researched the possibility of applying basic dyes by using N-methylformanilide as the swelling agent, achieving satisfactory washing and rubbing fastness properties [11]. Han and Jaung applied acid dyes on m-aramid fibres pre-treated with

PEO45-MeDMA and achieved some positive effect of the pre-treatment on obtained colour strength (K/S) in compare to untreated samples, although K/S value they reported was still rather low [12]. As for the vat dyes, E. R. Kim et al. have dyed m-aramid fibres, achieving satisfactory washing fastness properties, but rubbing and light fastness were rather low as well as the colour yield on m-aramid fibres [13]. Vat dyes are one of the oldest dye class used in textile and one of the major dye classes for cellulose fibres and cellulose fibre regenerates. They are characterized by high fastness properties and are therefore more applicable to functional textiles such as textiles with different protective properties. Vat dyes are insoluble, so during the process they must be converted from keto-substituted colorant by reduction to soluble form – enolate leuco form which has substantivity. The reduction is most often carried out with sodium dithionite (Na2S2O4), and through the dyeing process the dye is in soluble form, while at the end of the process it is again converted into an insoluble form by the oxidation process. Oxidation is most often carried out using oxidizing agents (e.g., hydrogen peroxide) [14, 15]. Inherently fire retardant textiles often contain cellulosic components, such as cotton and FR viscose, which makes vat dyes a suitable dye class for this type of fabric [16]. In this paper, the possibility of applying vat dyes on inherently fire retardant fabrics which, in addition to the content of aramid fibres, also contain cellulose components of FR viscose, was researched. The analyses were performed on non-commercial, inherent FR fabrics of the targeted composition developed at the University of Zagreb, Faculty of Textile Technology as part of the activities within the project Development of multifunctional non-flammable fabric for dual use, supported by the European Union from the European Regional Development Fund.

2. MATERIALS AND METHODS

The analysis was performed on two groups of three non-commercial fabrics characterized by the constant weave (Plain 1/1) but varied in weft composition. The content of warp and weft of the tested fabrics is shown in Table 1.

Table 1. Composition of analyzed fabrics

	Fabric 1 (B1)	Fabric 2 (B2)	Fabric 3 (B3)
Warp	22% PA 6.6 38% FR Viscose 40% M-Aramid	22% PA 6.6 38% FR Viscose 40% M-Aramid	22% PA 6.6 38% FR Viscose 40% M-Aramid
Weft	22% PA 6.6 38% FR Viscose 40% M-Aramid	30% PA 6.6 35% FR Viscose 35% M-Aramid	2% PA NoShock 20% PA 6.6 38% FR Viscose 40% M-Aramid

The samples were dyed in raw form without any pre-treatment (group 1) as well as pre-treated in the process of alkaline scouring (group 2). The recipe and conditions of alkaline scouring are given in Table 2.

Table 2. Recipe and conditions of alkaline scouring

Samples	Chemicals	Conditions
Fabric 1	Kemonecer WET: 2 g/l (wetting agent) Alkali Na2CO3: 2 g/l	T = 45 min T = 100 °C
Fabric 2		
Fabric 3		

2.1 LOI (Limiting Oxygen Index)

LOI-Limiting Oxygen Index was measured using Limiting Oxygen Index Apparatus (Concept Equipment Ltd.) according to EN ISO 4589-2: 2017 Plastics - Determination of burning

behavior by oxygen index - Part 2: Ambient - temperature test. Samples were conditioned (RH 65%, 24 h) according to standard ISO 139:2008/A1 Textiles - Standard Atmospheres for Conditioning and Testing. LOI values were measured prior to dyeing.

2.2 Dyeing

Untreated samples (group 1) and samples treated with alkaline washing (group 2) were vat dyed by the exhaustion dyeing process, in a laboratory apparatus for wet and dyeing processes Polycolor, Mathis, at 90 °C for 60 min (heating gradient 3°/min). The bath ratio (BR) was 1:40 and the dye concentration was 3% (over weight of the fabric). The dye used was Indanthren®Olive Green HB (DyeStar). According to the diagram prescribed for vat dyeing the content of sodium dithionite Na2S2O4 (2 g/L) and sodium hydroxide NaOH (11 ml/L) were determined, with the addition of the wetting agent Kemonecer WET (2 g/L). After dyeing, the samples were rinsed, oxidized in hydrogen peroxide H2O2 (4 ml/L) wit addition of Oxidol KR (Sodium-m-nitrobenzene sulphonate, 5 g/L), again rinsed and air-dried.

2.3 Color fastness and tactile properties testing

Dyed samples were tested for wash and light fastness properties. The wash fastness was tested by washing the samples in a laboratory apparatus for wet and dyeing processes Polycolor, Mathis, according to standard ISO 105-C06:2010 (A2S) Textiles — Tests for color fastness — Part C06: Colour fastness to domestic and commercial laundering, using 5 g/l standard detergent (ECE Non phosphate detergent without optical brightener agent), with bath ratio 1:8, temperature 40 ± 2 °C, time 30 minutes, through 5 cycles. Lightfastness testing were performed on Xenotest 440 (SDL Atlas), according to the modified ISO 105-B02 and 13 B04 test methods using Xenotest 440. The primary tactile properties were also measured on the Fabric Touch Tester M293 (SDL Atlas) in the form of ratings before and after the dyeing process.

2.4 Spectrophotometric measurement

The spectrophotometric measurement was performed by remission spectrophotometer DataColor 850, with constant instrument aperture, standard light D65 and d/8°geometry. The results of colouristic analysis of dyed samples are shown in terms of colour coordinates (a*/b*), basic colour parameters L*, C*, h°, and colour strength (K/S), expressed according to CIELAB system. The results of wash fastness are given as numerical values of total colour difference (dE) calculated according to CMC (l:c) equation accepted by ISO 105-J03:2009, for colour fastness and colour changes evaluation in the field of textiles [17]. The lightfastness properties were also expressed as total colour differences (dE), as well as in blue scale grades, obtained by comparing samples before and after exposure.

2.5 Microscopic imaging

The microscopic examination of dyed samples was performed using DinoLite AM7013. The imaging was performed before and after the 5th cycle of washing.

3. RESULTS AND DISCUSSION

The aim of the research, part of which is presented in this paper, is the development of inherently non-flammable fabric, which, in addition to fire retardant properties, will have added aesthetic and comfort properties. Therefore, the part is dedicated to researching the possibility of applying dyes in dyeing or printing processes, in general, for the purpose of obtaining patterning or colouration. The problem lies in the fact that due to the composition and high content of aramid fibres that cannot be dyed in conventional dyeing processes, it is difficult to achieve optimal dye binding and satisfactory colour strength, as well as in the fact that aramid fibres have their basic colour of pronounced yellowish hue, which makes it difficult to achieve

the target shade in the dyeing processes. In the first step of experimental work, since the primary goal is to achieve fire retardant properties of the fabrics, the measurement of LOI index was performed, and the results are shown in Table 3.

Table 3. LOI of fabrics

	Fabric 1	Fabric 2	Fabric 3
LOI [%]	27,21	27,27	27,77

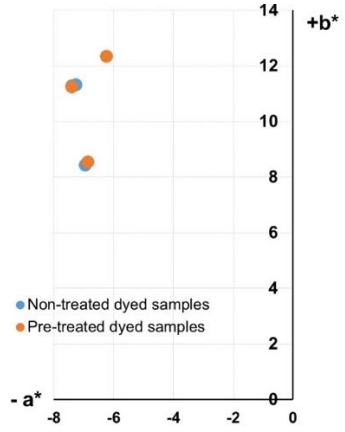
The results show that all of the samples meet the criteria specified for the inherently flame resistant fabrics that LOI> 26%. This is followed by dyeing of the samples, the first (Fabric 1 to 3 - untreated) and the second group (Fabric 1 to 3 - treated by alkaline scouring). After dyeing, and before testing the washing and light fastness properties, the analysis of primary tactile properties was performed as well as the analysis of coloristic parameters based on spectrophotometric measurement and their numerical, objective evaluation, according to CIELAB system (L *, a *, b *, C *, h). Table 4 shows the results of testing the primary tactile properties using the Fabric Touch Tester laboratory instrument. Smoothness, softness and warmth were evaluated and the values of the overall grade were given.

Table 4. Primary tactile properties of dyed fabrics

Group 1 (untreated fabrics)	Smoothness		Softness		Warmth		Total grade	
	BD	AD	BD	AD	BD	AD	BD	AD
Fabric 1	2	3	1	2	4	4	2	3
Fabric 2	2	3	1	2	4	4	1	3
Fabric 3	2	3	1	2	4	4	2	3
Group 1 (fabrics treated with alkaline scouring)	Smoothness		Softness		Warmth		Total grade	
	BD	AD	BD	AD	BD	AD	BD	AD
Fabric 1	2	3	1	3	4	4	2	3
Fabric 2	2	2	1	1	4	4	1	2
Fabric 3	2	3	1	2	4	4	2	3

*BD – before dyeing; AD – after dyeing

The results show that the pre-treatment of fabrics by alkaline scouring did not contribute to the change of primary tactile properties, while after the dyeing process there is an increase in values for smoothness and softness, which indicates an increase in these tactile properties - greater softness and smoothness after dyeing, to which the addition of wetting agent in the dyeing bath certainly contributes.



	L*	C*	h°
Fabric 1 (untreated)	39,52	13,43	122,68
Fabric 2 (untreated)	39,28	13,47	123,2
Fabric 3 (untreated)	35,8	10,91	129,51
	L*	C*	h°
Fabric 1 (treated)	43,66	13,82	116,77
Fabric 2 (treated)	39,83	13,43	123,33
Fabric 3 (treated)	37,32	10,94	128,75

Figure 1. Fabrics colours placing in a*/b* colour space, with table of L*, C* and h° values.

Figure 1 shows the placement of colour of the samples in the a*/b* colour space. According to coordinate values of a* and b* there is no significant influence of pre-treatment of the fabrics on colour placement in colour space. Also, uniform values are obtained for colour parameters of lightness (L*), chroma (C*) and hue (h°). However, a certain influence of the composition and content of different yarns in the examined fabrics on the value of the colour hue (h°) can be seen, and a shift of hue from yellow-green with a more pronounced yellow shade to yellow-green hue with a more pronounced green shade is observed.

Due to the lower value of chrome and brightness, the overall appearance of the colour of the samples is dark grey-green, which can be seen in Figure 2, where a scanned and microscopic images of the fabrics surfaces are given.

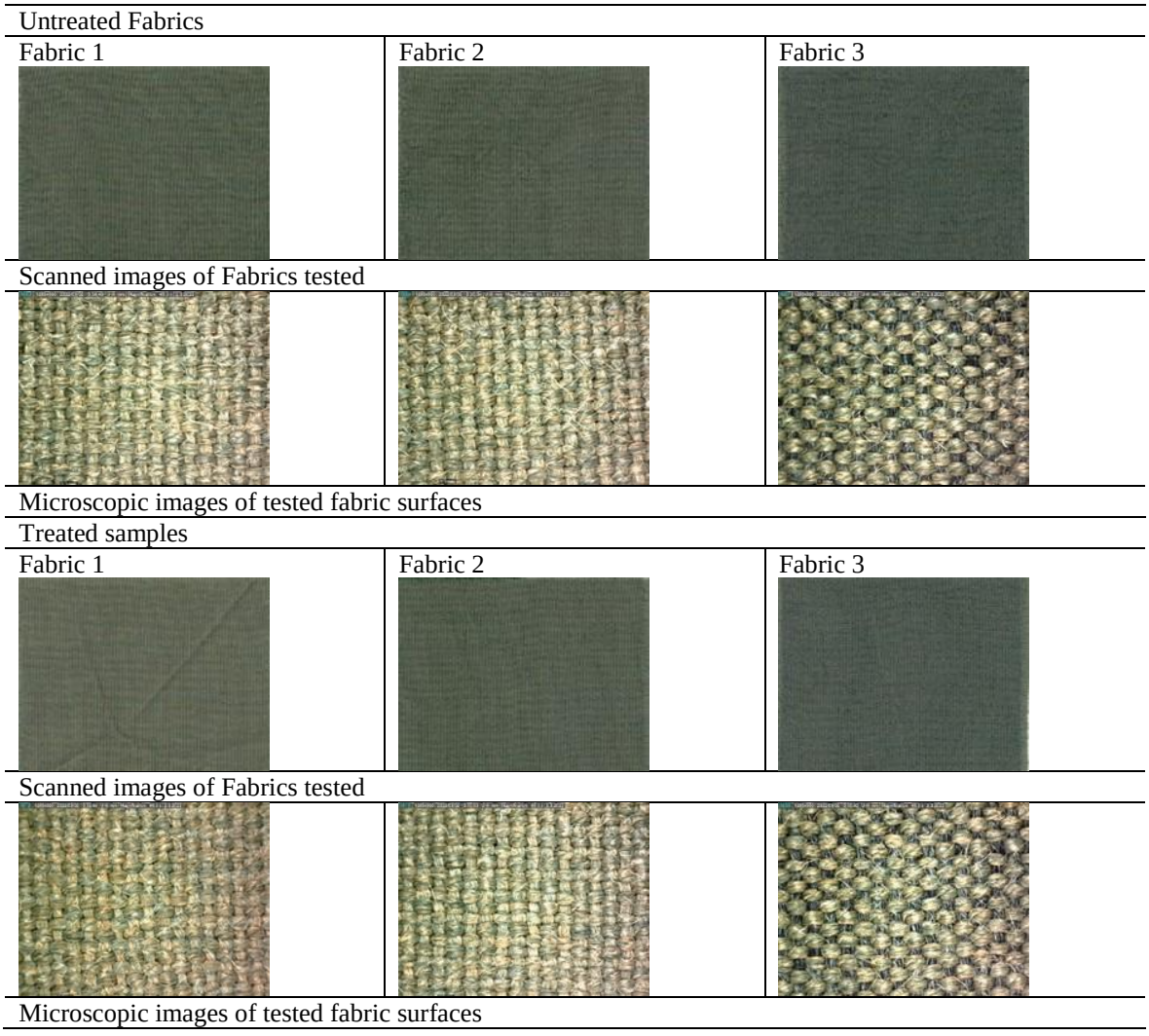


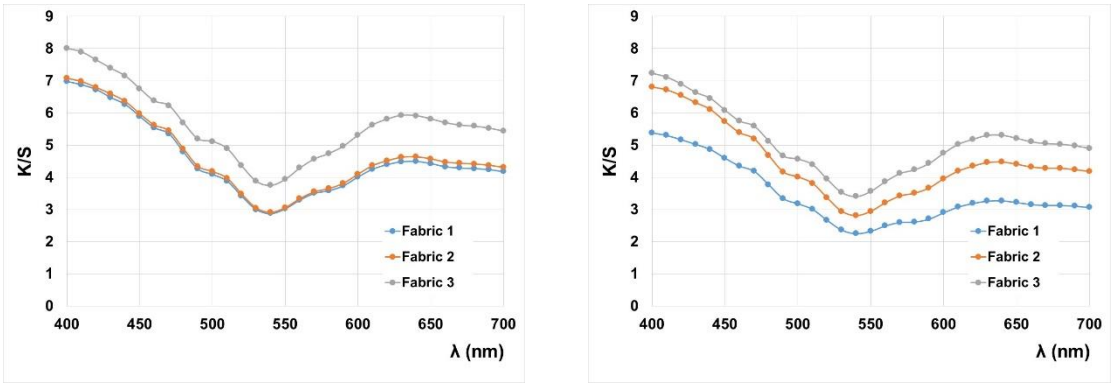
Figure 2. Scanned and microscopic images of tested fabrics surfaces

Given the content of fibre components that can bind the vat dye (cellulose and cellulose regenerates), the images show a satisfactory characteristic of the colour appearance. Microscopic images show that binding of dye occurred only with FR viscose and possibly PA 6.6 fibres, and the proportion of aramid fibres that remained undyed is clearly visible. Such a relationship will certainly affect the final appearance of the colour and the visual, but also the objective characteristics of the strength and intensity of colouration. In Table 5 the objective values of colour strength expressed in K/S values are shown, and also graphically presented on Figure 3.

Table 5. K/S values of dyed samples

Untreated Fabrics	λ (nm)	K/S	Treated Fabrics	λ (nm)	K/S
Fabrics 1	470	5,3551	Fabrics 1	470	4,1907
	630	4,4851		630	3,2647
Fabrics 2	470	5,4475	Fabrics 2	470	5,1994
	630	4,6219		630	4,4602
Fabrics 3	470	6,2288	Fabrics 3	470	5,3057
	630	5,9268		630	5,3057

Although visually, given the composition of the fabrics, a satisfactory colour depth has been achieved, the K/S values, objectively, are still relatively low. This can also be attributed to the nature of the olive-green colour itself, which is characterized by a grey component that places it in the marginal achromatic-chromatic area. Precisely because of this characteristic, objectively, a higher value of K/S cannot be achieved. The obtained characteristic of colour strength is confirmed by the shape of curves from which the chromatic-achromatic character of the colour can be seen. Also, a certain specificity of this colour can be seen in the K/S values. Namely, the K/S value is calculated, ie determined at the point of maximum absorption. For the olive-green colour, which spectrally belongs to the yellow-green area, the absorption takes place in the red-purple colour (magenta) which is not contained in the spectrum. Therefore, which corresponds to the nature of the magenta, the absorption takes place in two spectra that characterize the magenta, namely red (630 nm) and blue-violet (400 nm).



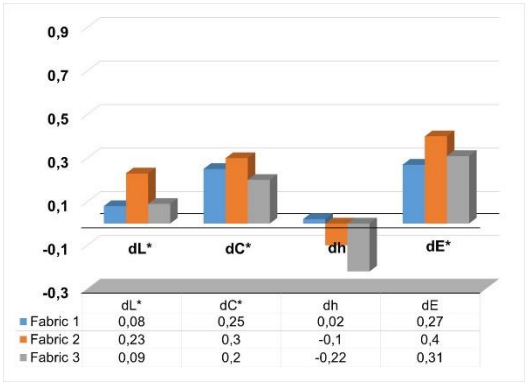
Untreated fabrics Treated fabrics

Figure 3. K/S curves of untreated and treated dyed fabrics

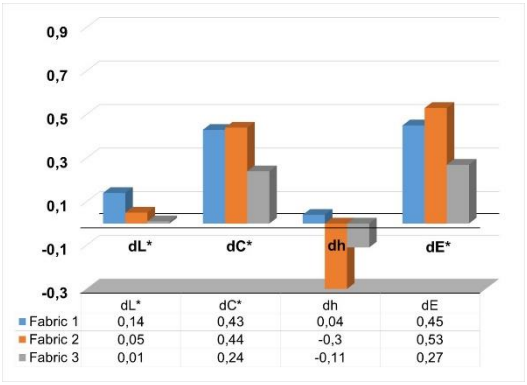
After the colouristic analysis of the dyed samples, the fastness tests were performed and the wash fastness and light fastness were tested. The results are expressed by the values of colour differences calculated according to the CMC (1: c) system (Figure 4), as the difference in individual colour parameters (dL*, dC*, dh), as well as the total colour differences (dE). The differences were calculated comparing the samples before washing with samples after the 5th cycles of washing.

The results of light fastness are shown on Figure 5, as the values of blue scale grades, as well as through the objective evaluation of colour differences between samples before and after light exposure (Figure 5). The results showed highly satisfactory colour fastness, which is confirmed by the values of differences in individual colour parameters, as well as in the values of the total colour difference, which do not exceed the numerical value of 0.5 for wash fastness and 0.9 for light fastness, which is definitely within the allowed tolerance limits. These are the expected results for vat dyes which, precisely because of the mechanism of transition from insoluble to

soluble form during dyeing, and then return to insoluble form, are characterized by highly satisfactory fastness.

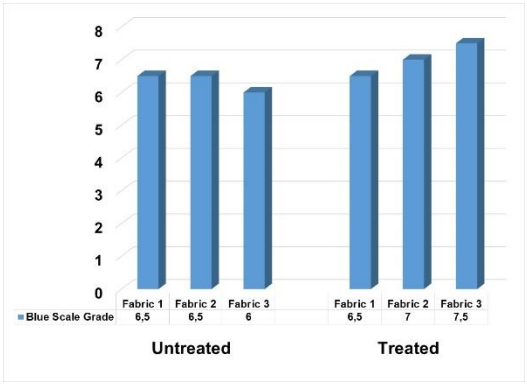


Untreated fabrics

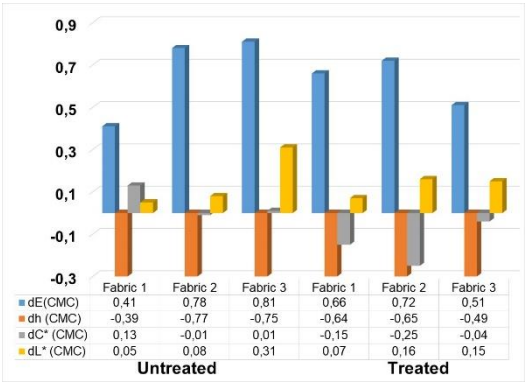


Treated fabrics

Figure 4. Wash fastness results expressed in colour difference values



Blue scale grades



Colour differences

Figure 5. Light fastness results expressed in Blue scale grade and in colour difference values

4. CONCLUSIONS

Based on the obtained results, it can be concluded that further research into the possibility of applying dyes on fabrics with a high content of aramid fibres is justified if they contain a component that can bind the dye in conventional dyeing procedures. It has been shown that satisfactory colouring of the target colour properties can be obtained. It is characteristic of vat dyes, and what was obtained in the research, that precisely because of their specific chemical constitution and the mechanism of returning to insoluble form after the dyeing, highly satisfactory fastness can be achieved.

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