



UNION OF ENGINEERS AND TEXTILE
TECHNICIANS OF SERBIA

VI INTERNATIONAL SCIENTIFIC CONFERENCE
**CONTEMPORARY TRENDS
AND INNOVATIONS IN THE
TEXTILE INDUSTRY**

VI MEĐUNARODNA NAUČNA KONFERENCIJA
**SAVREMENI TRENDovi I
INOvACIJE U TEKSTILNOJ
INDUSTRIJI**

PROCEEDINGS

EDITOR:
Prof. dr SNEŽANA UROŠEVIĆ

Belgrade, 14-15th September, 2023
Union of Engineers and Technicians of Serbia
Dom inženjera „Nikola Tesla“



**UNION OF ENGINEERS AND TEXTILE TECHNICIANS
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TABLE OF CONTENTS

PLENARY LECTURES	1
<hr/>	
Radomir Folić, Damir Zenunović TEXTILE REINFORCED CONCRETE	3
<hr/>	
Marija Vukčević, Biljana Pejić, Marina Maletić, Katarina Trivunac, Mirjana Kostić CONTRIBUTION TO THE CIRCULAR ECONOMY THROUGH THE UTILIZATION OF FIBROUS TEXTILE WASTE AS BIOSORBENTS FOR WATER PURIFICATION	17
<hr/>	
Andreja Rudolf, Zoran Stjepanović, Tadeja Penko DIGITAL VERSUS REAL DRAPING OF TEXTILES	27
<hr/>	
Iva Ćurić, Doris Curiš, Leonarda Bambić, Davor Dolar ENZYMATIC TREATMENT OF DYED COTTON KNITTED FABRIC WITH REUSED TEXTILE WASTEWATER	37
<hr/>	
Ineta Nemeša, Marija Pešić THE MAIN CHARACTERISTICS OF SMART SEWING MACHINES	45
<hr/>	
Kosana Vićentijević SUSTAINABILITY REPORTING IN THE TEXTILE INDUSTRY	62
<hr/>	
CONFERENCE PAPERS	
SESSION I	71
<hr/>	
Irem Çelik, Gizem Karakan Günaydın EVALUATION OF YARN PROPERTIES PRODUCED FROM MECHANICAL COMPACT AND CONVENTIONAL RING SPINNING SYSTEMS	73
<hr/>	
Matea Korica, Mirjana Kostić THE INFLUENCE OF LONG-TERM AGING ON THE PROPERTIES OF TEMPO OXIDIZED COTTON	82
<hr/>	
Hülya Kesici Güler, Funda Cengiz Çallıoğlu EFFECT OF PHASE RATIO ON CORE-SHEATH COMPOSITE NANOFIBERS MORPHOLOGY	92
<hr/>	
Ana Santiago, Cláudia Pinheiro, Nuno Belino DEVELOPMENT OF NEW TEXTILE APPLICATIONS USING NETTLE FIBRE	101
<hr/>	



Ivanka Ristić, Aleksandra Mičić, Nebojša Ristić CHEMICAL CATIONIZATION OF COTTON FABRIC FOR IMPROVED ABSORPTION OF DIRECT DYES	374
Mehmet Çağlayan, Banu Hatice Gürcüm DEMATEL METHOD FROM MULTI-CRITERIA DECISION MAKING METHODS AND EXAMPLES IN TEXTILE FIELD	383
Marina Maletić, Marija Vukčević, Biljana Pejić, Mirjana Kostić, Aleksandra Perić Grujić ORGANIC DYES ADSORPTION ON CARBON ADSORBENTS DERIVED FROM WASTE COTTON AND COTTON/POLYESTER YARN	394
Svitlana Arabuli, Anastasiia Truba, Arsenii Arabuli, Liudmyla Hanushchak- Yefimenko OPTICAL PROPERTIES OF MODIFIED FABRICS INTENDED FOR PROTECTION AGAINST UV RADIATION	403
Đorđić Dragan, Petrović Vasilije, Samir Pačavar, Enis Kolić, Anita Milosavljević ANALYSIS OF MECHANICAL CHARACTERISTICS AND WATER VAPOR PERMEABILITY OF FABRICS USED FOR MAKING THE LINING OF GARMENTS	409
Silvana Zhezhova, Sonja Jordeva, Sashka Golomeova Longurova, Sanja Risteska, Vanga Dimitrijeva Kuzmanoska FINAL CONTROL OF MEN'S SHIRTS	415
Julieta Ilieva, Mariya Koleva FROM NATURE TO FASHION – DIGITALIZED ELEMENTS AS INSPIRATION FOR PERSONALIZED CLOTHING AND ACCESSORIES	424
Subrata Das, Amitabh Wahi, Nivetha Sharathi S COLOUR MATCHING OF TEXTILES USING DEEP LEARNING CONVOLUTIONAL NEURAL NETWORKS	434
Marija Petrović, Samir Pačavar, Vasilije Petrović, Dragan Đorđić, Anita Milosavljević, Predrag Pecev THE INFLUENCE OF SOCIAL MEDIA ON PROMOTIONAL ACTIVITIES OF CLOTHING PRODUCERS	441
Dimitrios Chaidas, Tatjana Spahiu, John Kechagias AN INVESTIGATION OF 3D PRINTING ON TULLE TEXTILE CHANGING THE PLATFORM LEVELLING	451



ORGANIC DYES ADSORPTION ON CARBON ADSORBENTS DERIVED FROM WASTE COTTON AND COTTON/POLYESTER YARN

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ABSTRACT: In an attempt to reuse fibrous textile waste, and at the same time to reduce its quantity, waste cotton-based yarn was used as a cheap and sustainable precursor for carbon adsorbents production. Cotton and cotton/polyester yarn samples were carbonized using hydrothermal and classical carbonization and activated using KOH as activating agent. Nitrogen adsorption-desorption isotherms, scanning electron microscopy, Fourier transform infrared spectroscopy, and Boehm titration methods were used to analyze the structural and surface properties of obtained carbon samples. The adsorption properties of cotton-based carbon materials were tested through the adsorption of selected organic dyes from water. The influence of contact time, initial concentration, and pH value of adsorbate solution on adsorption was examined and experimental data were processed by theoretical models. It was found that the adsorption properties of carbonized samples were negatively affected by the presence of a polyester component in the precursor structure. Nevertheless, the cotton-based carbon adsorbents obtained by carbonization, and subsequent activation proved to be highly efficient adsorbents for organic dye removal from water.

Keywords: cotton, polyester, carbonization, chemical activation, adsorption, organic.

ADSORPCIJA ORGANSKIH BOJA NA UGLJENIČNIM ADSORBENTIMA DOBIJENIM OD OTPADNOG PREDIVA PAMUKA I PAMUK/POLIESTRA

APSTRAKT: U cilju iskorišćenja vlaknastog tekstilnog otpada, kao i smanjenja njegove količine, otpadna prediva na bazi pamuka su korišćena kao jeftina sirovina za proizvodnju ugljeničnih materijala. Pamučno predivo, kao i predivo mešavine pamuka i poliestra, karbonizovani su primenom klasične ili hidrotermalne karbonizacije, i naknadno aktivirani u prisustvu KOH kao aktivirajućeg agensa. Strukturne i površinske karakteristike dobijenih ugljeničnih materijala ispitane su analizom adsorpciono-desorpcionih izoterma azota i rezultata Bemovih titracija, kao i skenirajućom elektronskom mikroskopijom i infracrvenom spektroskopijom sa Furijeovom transformacijom. Adsorpcija odabranih organskih boja iz vodenih rastvora je korišćenja



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za ispitivanje adsorpcionih karakteristika, a ispitan je i uticaj vremena kontakta, početne koncentracije i pH vrednosti rastvora adsorbata na adsorpcione kapacitete ugljeničnih adsorbenata. Pokazano je da prisustvo poliestarske komponente u strukturi prekursora ima negativan uticaj na adsorpcione karakteristike materijala dobijenih samo karbonizacijom. S druge strane, svi uzorci ugljeničnih materijala dobijeni karbonizacijom pređa na bazi pamuka i naknadnom aktivacijom pokazuju visoku efikasnost u uklanjanju boja iz vode.

Ključne reči: pamuk, poliestar, karbonizacija, hemijska aktivacija, adsorpcija, organske boje.

1. INTRODUCTION

Various carbon materials have been widely used as adsorbents for the removal of organic and inorganic pollutants from wastewater. The high adsorption capacity of these materials is a consequence of the developed specific surface area and porosity, as well as the presence of a wide range of surface functional groups. These surface characteristics depend on the nature of the starting raw material, applied method for production, and production parameters such as temperature program, presence and amount of activation agents [1]. It has been shown in the literature that by carbonization and subsequent chemical activation in the presence of KOH, as an activating agent, microporous carbon materials with a highly developed specific surface can be obtained [2]. In recent years, great attention has been paid to the use of different types of lignocellulosic waste materials as starting materials for obtaining efficient carbon adsorbents, due to their specific structure and chemical composition rich in carbon [3-7]. This kind of lignocellulosic waste reuse reduces its amount in the environment and reduces a cost of waste disposal.

The aim of this work was to examine the possibility of using textile waste in the form of yarn, as precursors for obtaining carbon materials. Waste cotton and cotton/polyester yarn samples were transformed into carbon materials using hydrothermal and classical carbonization, as well as activation of the carbonized material in the presence of KOH. The adsorption properties of cotton-based carbon materials were tested through the efficiency of materials to adsorb selected organic dyes from water. The influence of contact time, initial concentration, and pH value of adsorbate solution on adsorption was studied, and experimental data were processed by theoretical models. Also, the influence of the starting raw material on the surface and adsorption characteristics of obtained carbon materials was examined.

2. MATERIALS AND METHODS

Textile waste in the form of cotton and a mixture of cotton and polyester (1:1) yarn, obtained from the Simpo Dekor factory (Vranje), was used as a raw material for obtaining carbon adsorbents by hydrothermal and classical carbonization, as well as



chemical activation. Hydrothermal carbonization of the yarn was carried out in an autoclave at a temperature of 180 °C, under increased generated pressure, for 24 hours. The carbonization reaction mixture consisted of 6 g of yarn, 400 cm³ of distilled water and 0.015 g of citric acid, which was used as a catalyst. After hydrothermal carbonization, the solid product was filtered and washed with methanol and distilled water. Classical carbonization was performed in an inert nitrogen atmosphere at 900 °C, with a heating rate of 5 °C/min. After hydrothermal and classical carbonization, the obtained materials were activated in the presence of KOH, as an activating agent, where the mass ratio of KOH and carbonized yarn was 1:2. Activation was performed in an electric furnace, in an inert nitrogen atmosphere at a temperature of 900 °C, with a heating rate of 5 °C/min. Eight carbon materials were obtained, marked with Cott_h, Cott/PES_h, Cott_c, Cott/PES_c, AcCott_h, AcCott/PES_h, AcCott_c and AcCott/PES_c, where Cott stands for cotton yarn as the starting raw material, Cott/PES for a mixture of cotton and polyester, h for hydrothermal, and c for classic carbonization, while Ac represents the designation for activation.

The morphological characteristics of the obtained materials were determined by scanning electron microscopy (Mira Tescan 3X, Tescan Orsay Holding, Czech Republic). The specific surface area (S_{BET}), and microporous surface area (S_{micro}) of the material, as well as the total pore volume (V_{total}), were obtained by the BET method, using a Micromeritics ASAP 2020 instrument. Fourier transform infrared spectroscopy (FTIR) was used to characterize functional groups on the surface of carbon materials. FTIR spectra of the samples were recorded in the range of wave numbers 400-4000 cm⁻¹, on Bomem MB-Series, Hartmann Braun. The Boehm method was used for the determination of acidic and basic oxygen groups, present on the surface of the carbon adsorbents. For the determination of the acidic sites, 0.1 g of tested samples were mixed with 10 cm³ of 0.1 M NaOH solutions in 25 cm³ beakers. The beakers were sealed and shaken for 24 h. The solutions were then filtered and titrated with 0.05 M H₂SO₄. Similarly, the basic sites were determined by mixing 0.1 g of the examined materials with 10 cm³ of 0.1 M HCl. The obtained solutions were titrated with 0.1 M NaOH. Adsorption experiments were performed in a batch system, with constant mixing (200 rpm). For adsorption efficiency examination, a sample of carbon adsorbent (0.02 g) was immersed in 25 cm³ of an aqueous dye solution with an initial concentration of 10 mg/dm³. For this purpose, solutions of methylene blue (MB), brilliant green (BG), Congo red (CR), methyl orange (MO) and crystal violet (CV) were used. The influence of contact time was examined by adsorption of methylene blue onto 0.05 g AcCott_c and AcCott/PES_c from 100 cm³ of MB solution (50 mg/dm³). At certain time intervals (3, 5, 10, 15, 30, 60, 120, and 180 minutes) samples were taken, and the MB concentration was determined by a visible spectrophotometer. The experimental data were analyzed using the pseudo-first (Eq 1) and the pseudo-second order model (Eq 2):

$$q_t = q_e (1 - e^{-k_1 t}) \quad (1)$$

$$q_t = q_e - \left(\frac{1}{\frac{1}{q_e} + k_2 t} \right)^{-1} \quad (2)$$

where q_e (mg/g) is the equilibrium adsorbed amount of adsorbate per unit mass of adsorbent, q_t (mg/g) is the amount of adsorbate per unit mass of adsorbent in time t

(min), k_1 (1/min) and k_2 (g/(mg min)) are the pseudo-first, and the pseudo-second order rate constants.

The influence of initial adsorbate concentration on adsorption was examined through the adsorption of MB (25 cm³, initial concentration of 10, 30, 40, 50, and 70 mg/dm³) onto 0.02 g AcCott_c and AcCott/PES_c. Collected data were fitted with Langmuir (Eq 3) and Freundlich (Eq 4) adsorption isotherm:

$$q_e = \frac{Q_0 b C_e}{1 + b C_e} \quad (3)$$

$$q_e = K_f C_e^{1/n} \quad (4)$$

where C_e (mg/dm³) is the equilibrium concentration of adsorbate in the solution, Q_0 (mg/g) is the maximum adsorption capacity, b (dm³/mg) is the Langmuir constant, and K_f (mg^{1-1/n}dm³/ng⁻¹) and $1/n$ are empirical Freundlich constants.

3. RESULTS AND DISCUSSION

The adsorption efficiency of hydrothermally and classically carbonized and activated cotton and cotton/polyester yarn to remove selected organic dyes from water is given in Figure 1. Generally, among the selected organic dyes, all tested cotton-based carbon adsorbents show good adsorption efficiency for the removal of MB. It was found that samples obtained by hydrothermal and classical carbonization show lower adsorption efficiencies than activated samples. For all tested dyes, samples AcCott_c and AcCott/PES_h showed the highest adsorption efficiencies. It seems that the presence of a polyester component in the starting material affects the adsorption efficiency of activated samples differently, depending on the applied carbonization method: for the samples obtained by hydrothermal carbonization and activation, the presence of PES component increases adsorption efficiency, while for the samples obtained by classic carbonization and activation adsorption efficiency decreases. In order to examine the influence of the PES component on surface and adsorption properties, characterization of the samples AcCott_c and AcCott/PES_c was performed, along with the detailed examination of the MB adsorption.

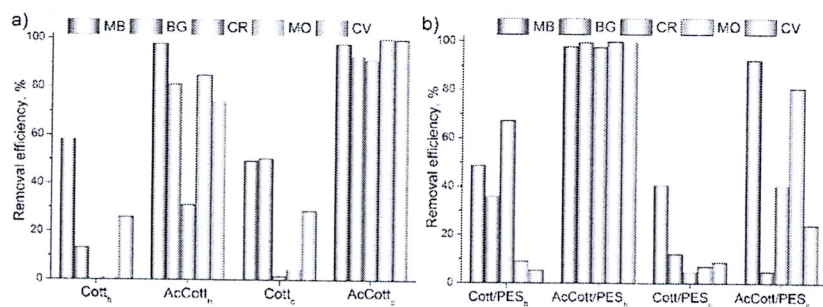


Figure 1: Adsorption efficiency of cotton-based carbon adsorbents for removal of selected dyes from water

Scanning electron micrographs of starting cotton and cotton/polyester yarn samples are shown in Figures 2a and 2b, while Figures 2c and 2d show the morphology of carbonized and activated samples.

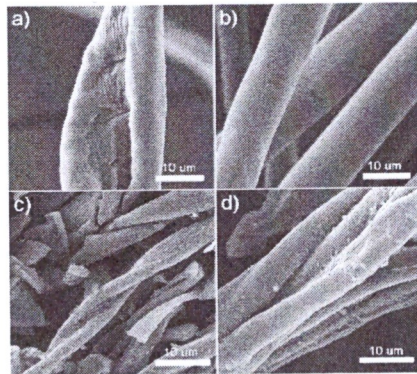


Figure 2: Scanning electron micrographs of a) cotton yarn, b) cotton/polyester yarn, c) AcCott_c and d) AcCott/PES_c.

The cotton fibers within the structure of starting cotton and cotton/polyester yarn samples are spirally twisted, with the structure looking like a twisted ribbon (Figures 2a and 2b). Also, the polyester component from the structure of starting cotton/polyester yarn is characterized by a straight filament, with a noticeably smooth surface. According to Figures 2c and 2d, carbonized and activated cotton components retain their fibrous structure, while in the case of the AcCott/PES_c polyester component most likely decomposes and disappears during activation, and appears to form a thin coating on the activated cotton component.

Figure 3 shows the FTIR spectra of the tested samples. Both samples show a broad band in the region of 3450-3650 cm⁻¹, which originates from stretching vibrations of the O-H bond in carboxyl or hydroxyl groups. Peaks at 2855 cm⁻¹ and 2925 cm⁻¹ originate from the symmetrical and asymmetrical vibrations of the C-H bond in methyl and methylene groups [8]. Also, a band at 1640 cm⁻¹ can be observed, which originates from the bending vibrations of the O-H bond, or the stretching vibrations of the aliphatic C=C bond, while the peak at 1384 cm⁻¹ originates from the deformational vibration of the C-O bond in the carboxyl group [9].

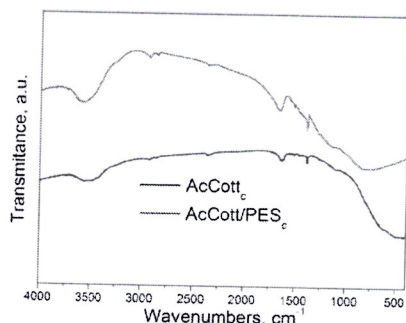


Figure 3: FTIR spectra of AcCott_c and AcCott/PES_c.

The specific surface area (S_{BET}), microporous surface area (S_{micro}), and the total pore volume (V_{total}) of AcCott_c and AcCott/PES_c, are given in Table 1. It is evident that starting from cotton and cotton/polyester yarn as carbon precursors, microporous materials with a highly developed specific surface area can be obtained by carbonization and activation in the presence of KOH. This double pyrolysis procedure of cotton/polyester yarn leads to some higher S_{BET} and S_{micro} values in the case of AcCott/PES_c, due to the decomposition of the synthetic component (also confirmed by SEM photographs) with the opening of the pores upon activation in the presence of KOH.

The Boehm method was used to determine the amounts of acidic surface oxygen groups (carboxyl, lactone, phenol), and basic functionalities (chromene, ketone, and pyrone groups, along with the delocalized π -electrons of graphene layers). It was found that sample AcCott_c contains a higher amount of surface oxygen groups, both acidic and basic, and that the surface of both examined samples has a distinctly acidic character, due to the much higher amount of acidic surface groups.

Table 1: Surface properties of AcCott_c and AcCott/PES_c.

Sample	S_{BET} , m ² /g	S_{micro} , m ² /g	V_{total} , cm ³ /g	Amount of acidic groups, mmol g ⁻¹	Amount of basic groups, mmol g ⁻¹
AcCott _c	885.8	663.3	0.518	3.939	0.713
AcCott/PES _c	913.1	756.1	0.511	3.763	0.361

The influence of the initial pH value of the MB solution on the adsorption capacities of AcCott_c and AcCott/PES_c is shown in Figure 4. Adsorption of MB onto AcCott_c does not depend on the initial pH, since this sample completely removed MB from the water solution. On the other hand, adsorption on AcCott/PES_c is influenced by the initial pH value of MB solution and the highest adsorption capacity was obtained on pH=8.



Therefore, all following adsorption experiments were performed from MB solution with initial pH value adjusted on 8.

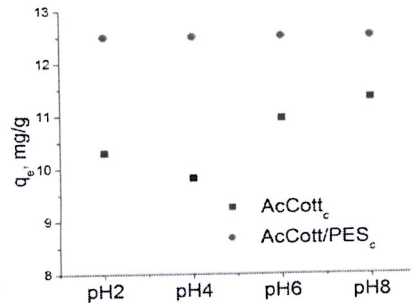


Figure 4: The influence of the initial pH value of MB solution on adsorption

The influence of contact time and initial MB concentration on adsorption is shown in Figure 5, along with the fitting of experimental data with theoretical models. Adsorption capacities of both samples increase with time and initial concentration, whereby AcCott_e shows a significantly higher adsorption capacity, despite the lower specific surface area. It is evident that specific surface area does not have a crucial influence on adsorption performance, and that a higher amount of surface oxygen groups increases the adsorption capacity of cationic MB. Sample AcCott_e exhibits fast adsorption, reaching the equilibrium within 10 minutes, while adsorption onto AcCott/PES_e slows down after 60 minutes, but does not reach equilibrium in examined time interval of 180 minutes (Figure 5a). According to the correlation coefficients (R²) MB adsorption on both samples follows the pseudo-second order kinetic. Although adsorption capacity increases with the initial concentration, there is no characteristic plot on the q_e-C_e dependence (Figure 5b), which indicate the lack of surface saturation in an examined concentration range, especially in the case of AcCott_e. Adsorption equilibrium data were fitted with Langmuir and Freundlich isotherm models and obtained parameters are summarized in Table 2.

Table 2: Langmuir and Freundlich parameters for MB adsorption on AcCott_e and AcCott/PES_e

Sample	Langmuir isotherm			Freundlich isotherm		
	Q ₀ , mg/g	b, dm ³ /mg	R ²	K _f , mg ^{1-1/n} /dm ³ /ng ⁻¹	1/n	R ²
AcCott _e	82.57	27.93	0.93757	73.30	0.178	0.75507
AcCott/PES _e	79.45	0.157	0.92797	14.40	0.401	0.91954

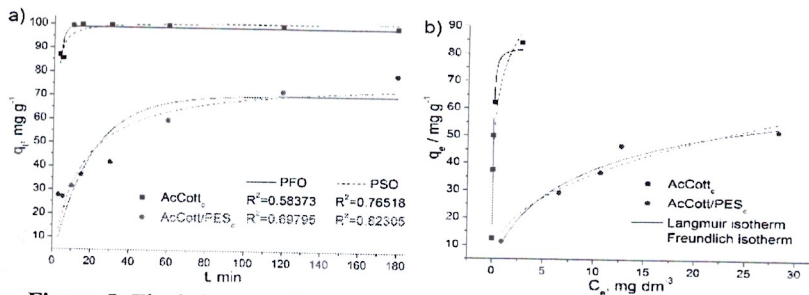


Figure 5: The influence of a) contact time and b) initial concentration on MB adsorption onto AcCott_c and AcCott/PES_c.

Adsorption equilibrium data were fitted with Langmuir and Freundlich isotherm models and obtained parameters are summarized in Table 2. Based on the correlation coefficients (R^2) adsorption of MB onto examined samples can be better described by Langmuir isotherm model.

The higher adsorption capacity of AcCott_c is confirmed by the values of Q_0 and K_f . Obtained values of heterogeneity factor ($1/n$) were less than unit, indicating that adsorption on examined cotton-based carbon adsorbents was a chemical process that occurs on the surface functional groups [4].

4. CONCLUSION

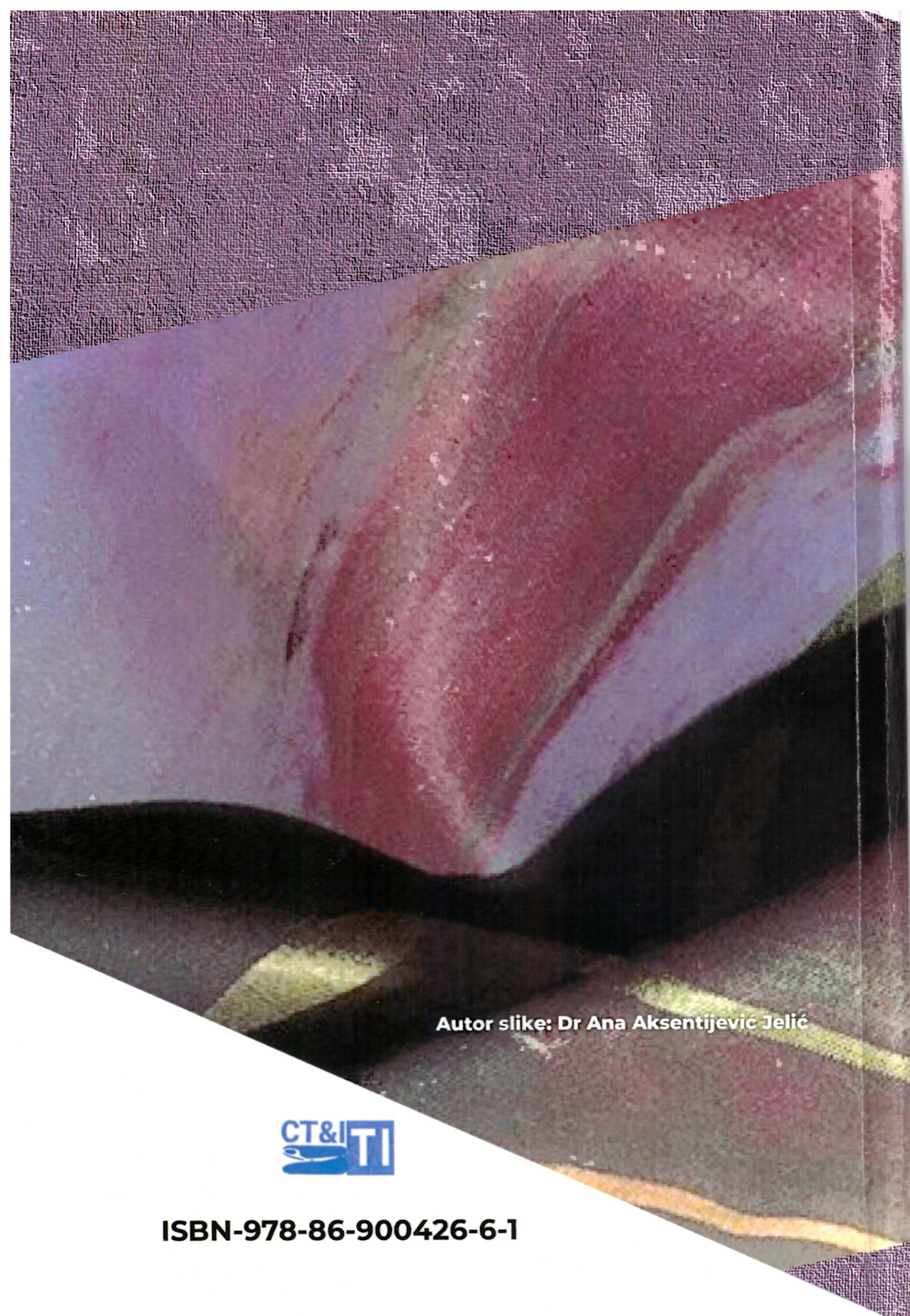
Different carbon adsorbents were obtained by classical and hydrothermal carbonization, and chemical activation, using textile waste in the form of cotton and cotton/polyester yarn as carbon precursors. Adsorbents obtained by carbonization and subsequent activation proved to be the most effective for the removal of selected organic dyes from water. It was found that the chemical composition of starting material, i.e., the presence of a polyester component in the structure of the yarn, affects the surface properties, morphology, and consequently, adsorption properties of the resulting cotton-based carbon adsorbents. In the case of cotton-based carbon adsorbents obtained by classic carbonization and consequent activation, the presence of the polyester component induced a higher specific surface area, but also a lower amount of surface oxygen groups, which led to the lower capacity for adsorption of methylene blue. Nevertheless, obtained results have shown that waste cotton yarn can be utilized for the preparation of highly efficient carbon adsorbent for the fast removal of methylene blue from water.

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