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STRUCTURE AND PROPERTIES OF TEMPO-OXIDIZED COTTON FIBERS*

In this paper, the influence of the catalytic oxidation using water soluble and stable nitroxyl radical 2,2',6,6'-tetramethylpiperidine-1-oxyl (TEMPO) on the structure and properties of cotton fibers was studied. In particular, the selective TEMPO-mediated oxidation has become a very interesting way for introduction of functional groups into cellulose fibers with the aim to obtain oxycellulose fibers with specific properties. Unmodified and modified fibers were characterized in terms of weight loss values, introduced functional groups and crystallinity index. Additionally, oxidized fibers were characterized in terms of the sorption, morphological, and physico-mechanical properties. The TEMPO-oxidized cotton fibers show a minimum increase of fineness (from 1.32 to 1.28 dtex) and increase of crystallinity index (up to 91.9%), while the tensile strength of fibers decreases (up to 10.82 cN/tex). By the TEMPO-mediated oxidation of cotton fibers, a significant amount of carboxyl groups (up to 0.795 mmol/g cell) can be introduced into cellulose fibers. Introducing hydrophilic carboxyl groups increases the sorption properties of oxidized fibers, which can be used directly or for further chemical modification.

Keywords: TEMPO-mediated oxidation; cotton fibers, structure, sorption and mechanical properties.

The main source of cellulose, which presents the most abundant, low cost and renewable biopolymer, is found in plant cell walls such as those of wood and cotton fibers. In almost pure form, cellulose is available in the seed hairs of cotton that is one of the most important types of fibers in the world [1,2]. Due to its excellent properties, such as softness, affinity to skin, strength and hygroscopic property, cotton is used for clothing with a broad range of application, as well as like raw material for preparing numerous products on the basis of cellulose (paper, medical supplies, home furnishings, food additives,...). For specific applications of cotton fibers in medical devices, like drug delivery matrix or absorbable adhesion barriers, etc., it is necessary to obtain fibers with improved sorption properties. Three hydroxyl groups of cellulose

are responsible for sorption properties and they are available for different chemical reactions. Among chemical reactions, such as esterification, nitration, etherification, oxidation is a quite frequent procedure in cellulose chemistry common to all primary and secondary hydroxyl groups [3,4].

Catalytic oxidation using water-soluble and stable nitroxyl radicals such as 2,2',6,6'-tetramethylpiperidine-1-oxyl (TEMPO) under aqueous conditions has become one of the interesting ways for introduction of functional groups in cellulose [5]. Oxidative conversion of cellulose by the system TEMPO/sodium hypochlorite/potassium bromide was first reported in 1994 [6] and has been the subject of several studies since then [7,8]. The TEMPO-mediated oxidation was described to cause selective oxidation at C-6 of the anhydroglucose units to carboxyl groups via the intermediate carbonyl stage, finally producing water-soluble polyglucuronic acids (Figure 1). The nitroxyl radical affects the oxidation from the alcohol to the carbonyl oxidation state, while the hypobromite generated *in situ* from hypochlorite and bromide performs the further oxidation of the carbonyl to the carboxylic acid. The reaction is usually carried out at around pH 10-11 since at lower pH, hypochlorite becomes an

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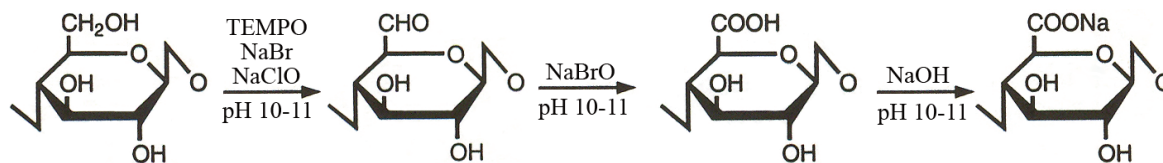


Figure 1. Scheme of TEMPO-mediated oxidation of cellulose [8].

overly aggressive and non-selective oxidant, and TEMPO reactivity is decreased [8,9].

According to the literature [5,10-13] TEMPO-mediated oxidation of cellulose pulp is widely investigated, while TEMPO-mediated oxidation of cellulose textile fibers has been investigated in a considerably smaller scope [14,15]. The aim of this paper is to study influence of the selective TEMPO-mediated oxidation on structure and properties of cotton fibers. Unmodified and modified fibers were characterized in terms of weight loss values, introduced functional groups and sorption properties (moisture sorption, water retention power and iodine sorption). Also, oxidized fibers were characterized in terms of the physico-mechanical (tensile strength and elongation) and geometric (fineness) properties. Based on the results obtained in this study, an attempt has been made to explain the individual roles of introduced functionalities and changed fiber structure in the sorption and mechanical properties of oxidized cotton fibers.

EXPERIMENTAL

Materials. Cotton fibers: Russian, I class, 32/33 mm. All the chemicals used were obtained from commercial sources are p.a. grade.

Preparation of TEMPO-oxidized cellulose fibers. The oxidation procedure was based on the literature methodology [5,16]. In brief, cellulose fibers (10 g) was suspended in water (750 ml) containing TEMPO (0.025 g) and sodium bromide (0.25 g). Subsequently, a designed amount of NaClO solution containing 13% available chlorine, corresponding to 0, 0.30, 2.42, 4.84 and 9.67 mmol/g cellulose, was added to the cellulose slurry under continuous stirring for 1-4 h, at room temperature. The obtained water-insoluble fractions were filtered on a Büchner funnel, and dried at room temperature for 48 h.

Determination of weight loss. Loss in weight, as result of chemical treatment, was determined by the direct gravimetric method [17].

Determination of carboxyl and carbonyl groups in the TEMPO-oxidized cellulose fibers. a) The carboxyl content of the fibers was determined using acid-base titration method according to the method described by Kumar and Yang [18] and Yackel and Kenyon [19]. The cellulose should be obtained in the aci-

dic form by replacement of its cations by hydrogen ions by the treatment of cellulose samples (0.5 g) with 0.01 M HCl for 1 h, followed by washing with distilled water. In the next step, 50 ml of distilled water and 30 ml 0.25 M of Ca-acetate solution were added to the fibers suspension. After standing during 2 h with frequent shaking, to facilitate completion of the interchange, 30 ml portions of the suspension were titrated with 0.01 M sodium hydroxide, using phenolphthalein indicator. The carboxyl contents are calculated as follows:

$$\text{COOH} = \frac{80}{30} \frac{0.01MV(\text{NaOH})}{m \left(1 - \frac{w}{100}\right)}, \text{ mmol/g} \quad (1)$$

where 0.01 M is concentration of NaOH, $V(\text{NaOH})$ is volume (ml) of NaOH solution used for titration, m is weight of treated fibers (g), and w is moisture content (%).

b) The carbonyl content was measured according to the method described in literature [16,20]. The water-insoluble fractions were further oxidized with sodium chlorite at pH 4-5 for selective conversion of the carbonyl groups to carboxyl ones, and carboxyl content was determined by the above mentioned calcium-acetate method. The carboxyl groups formed by the NaClO_2 oxidation were regarded as carbonyl groups present in the original water-insoluble fractions.

Determination of moisture sorption and water retention value. a) Moisture sorption of oxidized cellulose fibers was determined according to standards (ASTM D 2654-76, 1976). Fibers were exposed to standard atmosphere: 20 ± 2 °C, $65 \pm 2\%$ relative humidity, for 24 h (ASTM D 1776-74, 1974). Moisture sorption was calculated as weight percentage of absolute dry material. b) Water retention of cellulose fibers was determined by standard centrifuge method (ASTM D 2402-78, 1978).

Determination of iodine sorption value. The Schwestassek method was used for evaluation of the sorption properties of the cellulose fibers [21,22]. Cellulose fibers (0.3 g) were treated with 2 ml of iodine solution KI_3 (5 g I_2 + 40 g KI + 50 ml H_2O) for 3 min, then 100 ml saturated sodium sulphate (200 g/l) was added and the sample was shaken for 1 h. The iodine concentration of the sample and blank was deter-

mined by titration with sodium thiosulphate (0.02 mol/g). The iodine sorption values (ISV) in mg I₂ per g of sample was calculated as follows:

$$ISV = \frac{(b-t) \times 2.04 \times 2.54}{m_a}, \text{ mg/g} \quad (2)$$

where b is volume (ml) of Na₂S₂O₃ solution for blank titration, t is volume (ml) of Na₂S₂O₃ solution for the titration of sample solution, M is the molarity of the sodium thiosulphate (mol/g), 102 is a total volume (ml) of the solution, and m_a is the weight of absolute dry cellulose fibers (g). According to Schwertassek, the absorption takes place in the amorphous phase. A ratio of ISV (per 1g cellulose) to 412 (mg iodine absorbed per 1g of methyl cellulose) determines the amorphous fraction. The crystallinity index (CrI) was calculated using equation (3) [21].

$$CrI = 100 - \left(\frac{ISV}{412} \times 100 \right), \% \quad (3)$$

Geometrical and mechanical properties - fineness, tenacity and flexibility. Fineness in tex was determined as per standard methods (SRPS F.S2.212., 1963) by dividing the mass of fibers by their known length. The tensile strength and elongation of individual fibers was carried out using the prescribed standard SRPS F.S2.213. For this test the hydraulic drive dynamometer Werkstoffprüfmaschinen brands was used, which enabled the determination of breaking load up to 98 cN. The distance between terminals was set to 20 mm, and for each sample 10 measurements was carried out.

Scanning electron microscopy (SEM) analysis. Fiber morphology was observed by a JEOL JSM 5300 scanning electron microscope (SEM), at 20 kV after sputtering with gold.

RESULTS AND DISCUSSION

Weight loss of TEMPO-oxidized cotton fibers. Cellulose is very sensitive to oxidizing media due to its polyalcoholic character. The severity of treatment is generally characterized by weight loss, which is a result of water solubility of oxidation products and/or some of low-molecular weight products obtained due to proceeding β -elimination at the C-6 carbonyl intermediate. In addition, in the case of cotton fibers the weight loss is a result of decomposition of impurities and natural pigments in cotton fibers [23,24]. The highest weight loss for oxidized cotton fiber (11.34%) was determined for the sample treated in the most severe conditions of TEMPO-mediated system, with 9.67 mmol NaClO/g cellulose for 4 hours (Figure 2).

Carbonyl and carboxyl contents in the TEMPO-oxidized cotton fibers. The effect of oxidation time and amount of added NaClO on carbonyl and carboxyl group contents in the water-insoluble fractions of TEMPO-oxidized cotton fibers is shown in Figure 3.

The carbonyl groups formed in the water-insoluble fractions are mostly due to C-6 carbonyls present as the intermediate structure during the TEMPO-mediated oxidation and reducing ends formed from glycoside bonds by β -elimination at pH 10.5 and other depolymerization reactions during the oxidation [9,16]. The carbonyl group content in cotton fibers oxidized

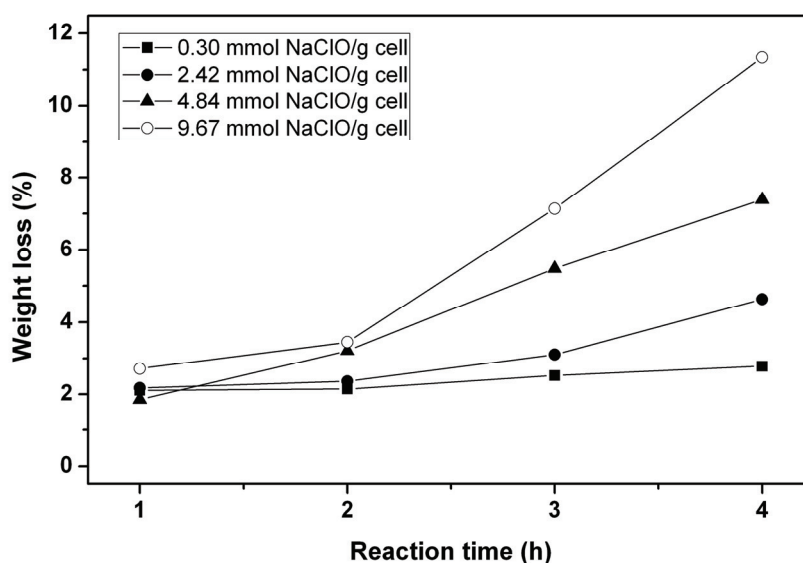


Figure 2. Relationships between oxidation time and weight loss value in the TEMPO-oxidized cotton fibers, where 0.30, 2.42, 4.84 and 9.67 mmol NaClO (per gram of cellulose) was applied to the cellulose slurry, at room temperature and pH 10.5.

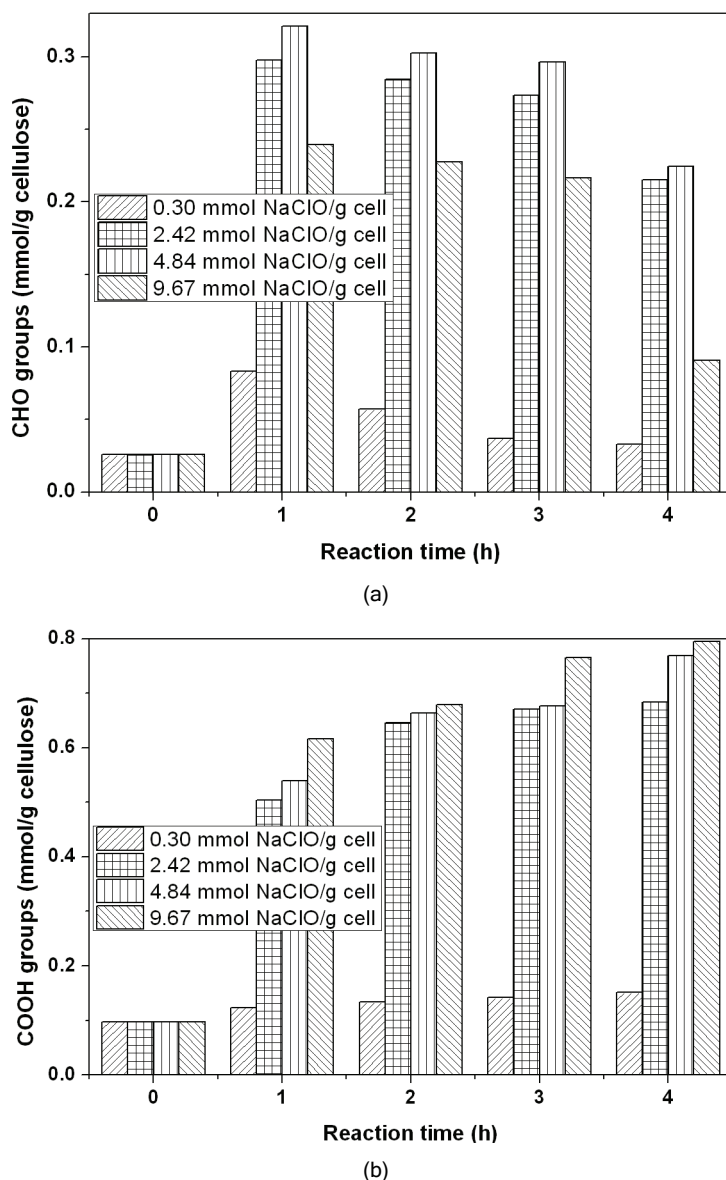


Figure 3. Relationships between oxidation time and a) carbonyl and b) carboxyl group content in the TEMPO-oxidized cotton fibers, where 0.30, 2.42, 4.84 and 9.67 mmol NaClO (per gram of cellulose) was applied to the cellulose slurry, at room temperature and pH 10.5.

by different amount of the primary oxidant (NaClO) reached the maximum value after the 1 h of oxidation (12.5 times higher than corresponding values for unmodified cotton fibers), and decreased slowly with increasing oxidation time (Figure 3a). The observed decrease of carbonyl group content can be explained by conversion of CHO groups into COOH groups due to the further oxidation process with hypobromite generated in situ from hypochlorite and bromide (Figure 1). The highest decrease was obtained in the case of modification under the most severe conditions (9.67 mmol NaClO/g cell and 4 h), because of the generation of the highest amount of hypobromite. In the same time, a certain amount of carbonyl groups pre-

sent in all oxidized fibers, even after the 4 h oxidation, can be explained according to the literature [5,9] by formation of intra- and inter-molecular hemiacetals with cellulose hydroxyls in the solid water-insoluble fractions, which are likely to have high resistance to the next TEMPO-mediated oxidation step (*i.e.*, converting to carboxyl groups) by steric hindrance.

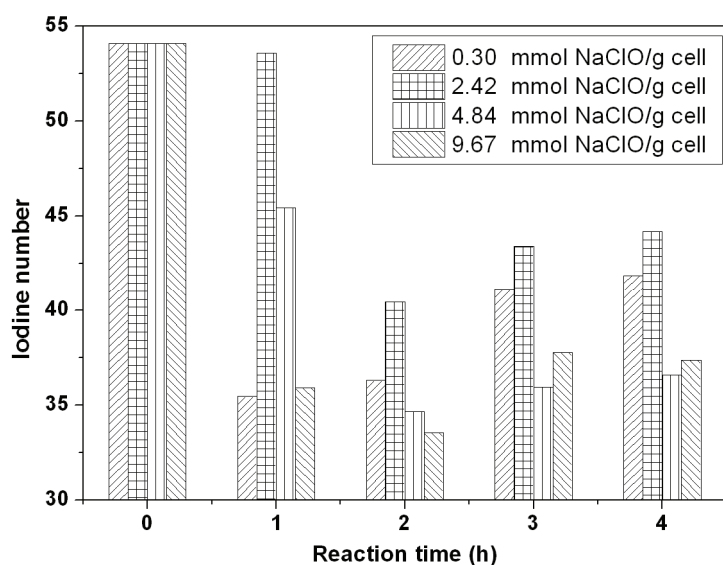
By changing the parameters of the oxidation (NaClO concentration and treatment time), it is possible to obtain cotton fibers with a different amount of carboxyl groups within the range of 0.123–0.795 mmol/g cellulose (Figure 3b). Comparison of data for carbonyl and carboxyl groups presented in Figures 3a and 3b revealed that decrease in carbonyl group content,

mainly due to their conversion into carboxyl groups, is not followed by expected increase in carboxyl group content, especially in the case of the most severe oxidation conditions. These can be explained by the water solubility of highly oxidized cellulose molecules (data for weight loss, Figure 2). In spite of this, the maximum increase of carboxyl content in oxidized cotton fibers of 8.16 times ($\text{COOH}_{\text{mod.}}/\text{COOH}_0$) is obtained for the sample oxidized in the presence of 9.67 mmol NaClO/g cellulose during 4 h.

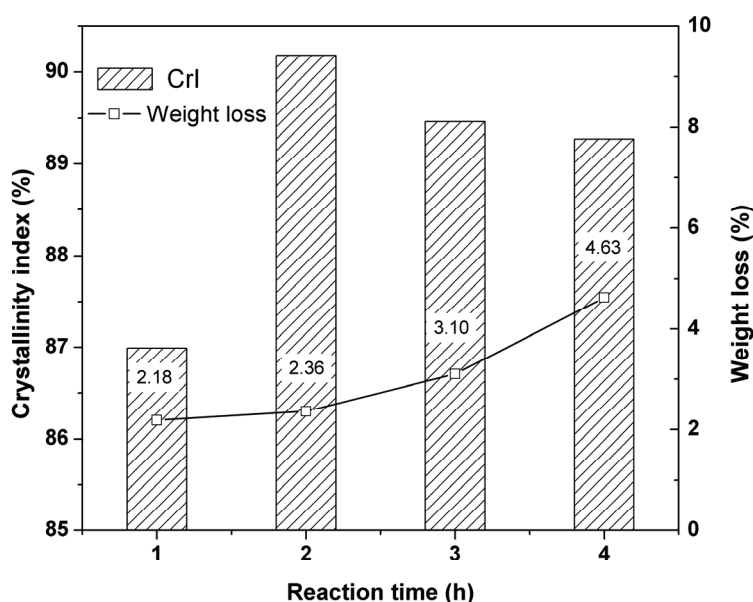
Iodine sorption value. As a consequence of the dissolution of highly oxidized cellulose molecules and/or

some of low-molecular weight products obtained due to proceeding β -elimination and introduction of carbonyl and carboxyl groups into cellulose by the TEMPO-mediated oxidation, the iodine sorption values has been changed (in the range from 35.5 to 53.6), depending on the oxidation conditions (Figure 4a).

As iodine takes place only in less ordered amorphous regions of cellulose [23,25], inverse of iodine sorption value is proportional to the fiber crystalline phase, and can be expressed by the crystallinity index (CrI). During the TEMPO-mediated oxidation, the crystallinity index was increased and changed in the



(a)



(b)

Figure 4. Relationships between: a) oxidation time and iodine sorption value in the TEMPO-oxidized cotton fibers where 0.30, 2.42, 4.84 and 9.67 mmol NaClO (per gram of cell) was applied to the cellulose slurry and b) crystallinity index (CrI) and weight loss of TEMPO-oxidized cotton fibers, modified with 2.42 mmol NaClO (per gram of cell) during 1-4 h, at room temperature and pH 10.5.

range from 86.9 for unmodified cotton fibers to 87.0–91.9 for TEMPO-oxidized cotton fibers. The resulting slight changes in crystallinity for the TEMPO-oxidized cellulose sample are due to partial loss of the disordered regions during the washing process because of their increased water-solubility [16]. This can be confirmed by the relationship between the *CrI* and weight loss, for the set of TEMPO-oxidized cotton fibers modified under the same conditions, in the presence of 2.42 mmol NaClO/g cellulose (Figure 4b). The obtained results are in agreement with literature data [14,16,26] and confirm that the oxidation did not reach to the interior of the crystalline region, *i.e.*,

carbonyl and carboxyl groups were introduced in disordered regions and crystal surface of cellulose.

Moisture sorption and water retention value of TEMPO-oxidized cotton fibers. Introduction of carbonyl and carboxyl groups into cellulose and changes in crystallinity and pore structure during the TEMPO-mediated oxidation, affect the sorption properties, which have been evaluated by determination of moisture sorption (MS) and water retention value (WRV). For unmodified cotton fibers, moisture sorption and water retention values are 5.77 and 18.16%, respectively. Figure 5 depicts moisture sorption and water retention values of the water-insoluble fractions of the TEMPO-

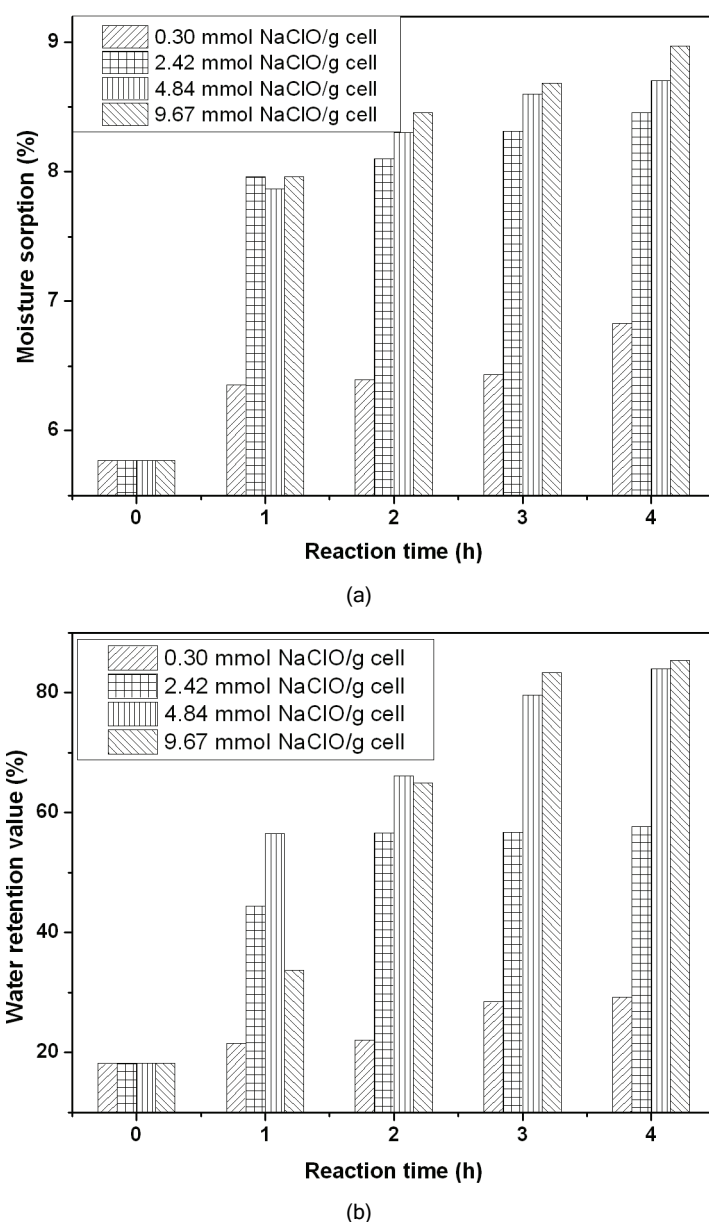


Figure 5. a) Moisture sorption (MS) and b) water retention values (WRV) of the TEMPO-oxidized cotton fibers where 0.30, 2.42, 4.84 and 9.67 mmol NaClO (per gram of cellulose) was applied to the cellulose slurry, at room temperature and pH 10.5, for the different oxidation times (1–4 h).

oxidized cotton fibers oxidized under different conditions (*i.e.*, oxidation times and NaClO charge). The obtained data showed that the moisture sorption value and WRVs of oxidized cotton increased with increasing NaClO charge and oxidation times approaching a maximum value of 8.97% (ratio $w_{mod}/w_0 = 1.55$) for MS, and 85.4% for WRV. Both maximum values are obtained for sample oxidized under the most severe conditions (4 h, 9.67 mmol NaClO/g cell). The increases are due to the introduction of hydrophilic carboxyl groups and morphological changes from fibrous forms to short fragments, which are for the samples modified with different amount of NaClO (2.42-9.67 mmol/g cell), during 4 h, shown in Figure 6. The SEM images also confirm removing of the surface layer of cotton fiber, which contains accompanying components (waxes and pectin). Effectively eliminated hydrophobic impurities from the surface of the fibers by the TEMPO-oxidation enabled water molecules to penetrate into the cotton fiber, which is in agreement with the results obtained for oxidative treatment under alkaline conditions [23,24].

Fineness, tenacity and flexibility. As it is usual that the fibers tensile strength is expressed as a tenacity-specific value related to fineness (force per unit fineness), a brief review of the TEMPO-mediated oxidation influence on the fineness of cotton fibers should be assessed. In the modified fibers, which retained

the fibrous form after the oxidation, minimal changes in fineness (1.32 to 1.28 dtex) were observed. Finer fibers are obtained due to decomposition of oxidation products and depolymerization caused by the active species, and due to the removal of impurities in cotton fibers [23,27]. Increasing the amounts of modification agents and prolongation of the oxidation reduces the tensile strength of oxidized fibers up to 58%, compared to unmodified cotton fibers (25.79 cN/tex). Reduction of tensile strength of the modified fibers can be explained by the oxidation - destructive processes that lead to a reduction degree of polymerization and molecular weight [27], and hence to a deterioration of mechanical properties (Table 1). In the mildest conditions of modification (0.30 mmol NaClO per gram of cellulose, 1 h), the decrease in cotton fiber tensile strength was 21.3% compared to the unmodified fibers. Elongation of modified fiber increased only for samples modified with 9.67 mmol NaClO/g cell, 1 h (6.3%), while in all other cases decreases, depending on the modification of conditions, from 2.9 to 5.3%, compared to the unmodified sample (5.4%). Applying severe conditions (9.67 mmol NaClO/g cell) in the case of TEMPO-mediated oxidation of cellulose fibers results in loss of the fibrous structure, and for textile applications only oxidation under mild conditions should be used (Figure 7).

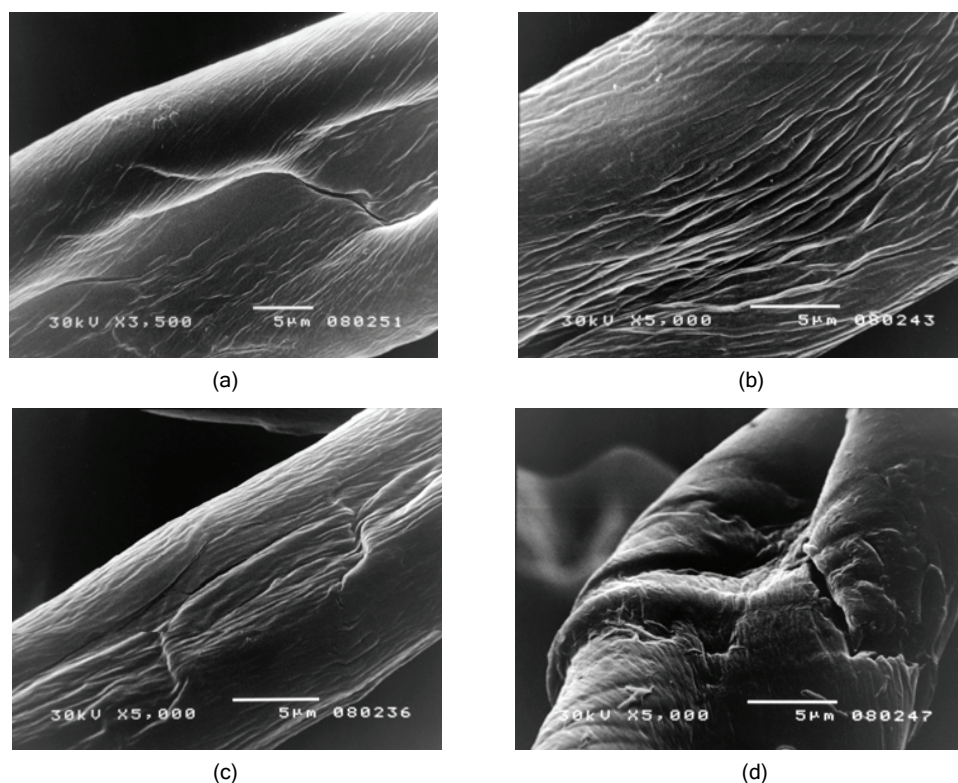


Figure 6. SEM Images of a) unmodified and cotton fibers modified during 4 h, with different amounts of oxidative agent: b) 0.30, c) 2.42 and d) 9.67 mmol NaClO/g cellulose.

Table 1. Tenacity of TEMPO-oxidized cotton fibers

Oxidative conditions	Tenacity, cN/tex	Oxidative conditions	Tenacity, cN/tex
0.30 mmol NaClO/g cell, 1 h	20.29	2.42 mmol NaClO/g cell, 1 h	18.55
0.30 mmol NaClO/g cell, 2 h	19.60	2.42 mmol NaClO/g cell, 2 h	13.87
0.30 mmol NaClO/g cell, 3 h	18.34	4.84 mmol NaClO/g cell, 1 h	12.75
0.30 mmol NaClO/g cell, 4 h	17.97	9.67 mmol NaClO/g cell, 1 h	10.82

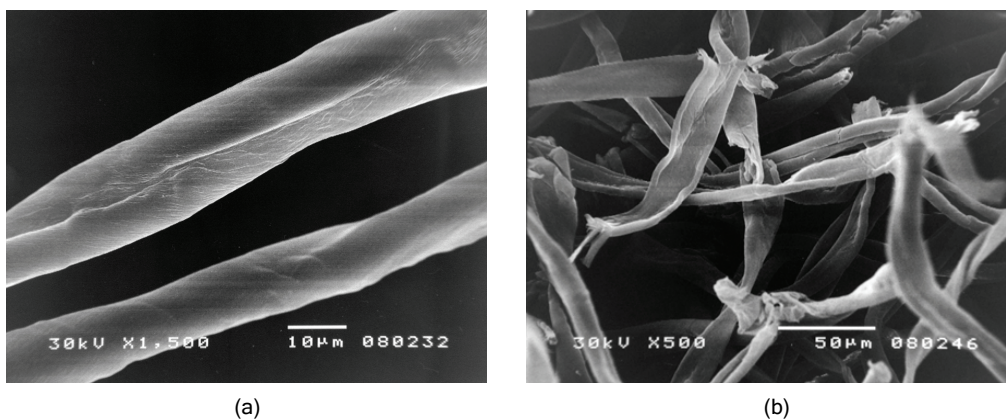


Figure 7. SEM Images of cotton fibers modified during 4 hour, with a) 0.30 and b) 9.67 mmol/g cellulose.

CONCLUSIONS

This work demonstrates the potential of the selective TEMPO-mediated oxidation on cotton fibers. The TEMPO-oxidized cotton fibers show minimal increase of fineness (from 1.32 to 1.28 dtex) and increase of crystallinity index (up to 91.9%), while the tensile strength of fiber decreases (up to 10.82 cN/tex). Applying severe conditions results in loss of fibrous structure and for textile applications only oxidations under mild conditions should be used. By changing the parameters of the oxidation (*i.e.*, HOCl conc., reaction time), it is possible to obtain fibers with a different amount of carboxyl groups (up to 0.795 mmol/g cell). Due to the introduced hydrophilic COOH groups, the hemisorption properties of oxidized fibers increase. The functional groups introduced by oxidation of cellulose can be used directly because of their special properties, *e.g.*, hydrophilization or even water solubility by introduction of carboxyl groups, or they can be used for further chemical modification.

Acknowledgment

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Nomenclature

CrI - Crystallinity index
 ISV - Iodine sorption value
 MS - Moisture sorption
 SEM - Scanning electron microscope

TEMPO - 2,2',6,6'-Tetramethylpiperidine-1-oxyl radical
 WRV - Water retention value.

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NAUČNI RAD

STRUKTURA I SVOJSTVA TEMPO-OKSIDISANIH VLAKANA PAMUKA

U ovom radu je proučavan uticaj selektivne oksidacije katalizovane 2,2',6,6'-tetrametil-piperidin-1-oksil radikalom, poznatim kao TEMPO radikal, na strukturu i svojstva vlakana pamuka. TEMPO-oksidacija je postala posebno interesantna za uvođenje funkcionalnih grupa u celulozu, u cilju dobijanja oksiceluloznih vlakana sa specifičnim svojstvima. Nemodifikovana i modifikovana vlakna pamuka okarakterisana su sa aspekta gubitka mase, sadržaja uvedenih funkcionalnih grupa i stepena kristalnosti. Takođe, određena su sorpciona, geometrijska i fizičko-mehanička svojstva vlakana pamuka. Za TEMPO-oksidisana vlakna pamuka uočava se minimalno povećanje finoće (1,32-1,28 dtex), a tačke i indeksa kristalnosti (do 91,9 %), dok se prekidna jačina vlakana smanjuje (do 10,82 cN/tex). Oksidacijom pomoću TEMPO radikala postiže se uvođenje značajnih količina karboksilnih grupa (do 0,795 mmol/g celuloze). Usled uvođenja hidrofilnih funkcionalnih grupa dolazi do povećanja hemisorpcionih svojstava oksidisanih vlakana, što se može upotrebiti direktno ili u daljem hemijskom modifikovanju.

Ključne reči: TEMPO-oksidacija; vlakna pamuka; struktura; sorpcione i mehaničke karakteristike.