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## SORPTION PROPERTIES OF PERIODATE OXIDIZED COTTON<sup>#</sup>

*The effect of periodate oxidation on the chemical and sorption properties of cotton yarn was investigated by determining aldehyde group content, moisture sorption, water retention and iodine sorption. Oxidation of cotton yarn was performed by varying concentration of sodium periodate solution and reaction time. To measure the aldehyde content present in the oxidized cotton, the aldehyde groups were selectively oxidized to carboxyl groups with sodium chlorite at pH 4-5, at room temperature for 48 h, and carboxyl group content was determined by modified calcium-acetate method. Differences in the sorption properties of untreated and oxidized cotton samples were obtained using conventional methods. The aldehyde groups were introduced into the oxidized cotton up to 99.2 μmol/g. Compared to the untreated fibers, oxidized cotton samples exhibited higher moisture sorption (up to 9%) and lower water retention values (up to 19%) and iodine sorption values (up to 31%). Based on the obtained results, it can be concluded that the crystalline structure of cellulose is not significantly changed by periodate oxidation, which is of great importance for textile material production.*

*Key words: cotton; sodium periodate oxidation; moisture sorption; water retention power; iodine sorption.*

Cellulose is the most abundant renewable natural polymer on Earth and hence can be regarded as a very important raw material for several purposes. Recently, cellulose has been in the public eye due to its possible use in the production of biofuels. However, cellulose has shown its versatility in numerous applications. Moreover, it can be chemically modified to yield cellulose derivatives [1,2]. Social concerns for sustainable green products are encouraging the efficient exploitation of cellulose.

Chemical modification of cellulose using oxidizing agents is quite a frequent procedure in cellulose chemistry. Due to the three hydroxyl groups available for oxidation within one anhydroglucose units and due to the polymeric character of the cellulose, a great variety of structural modifications and combinations is possible. Periodate oxidation is one of the rare ex-

amples of highly selective modification of cellulose [3-8]. Periodates specifically react with molecules that have adjacent hydroxyl groups. When applied to cellulose, the C2-C3 bond in the glucopyranoside ring is cleaved and the adjacent hydroxyl groups at these positions are converted to aldehydes producing dialdehyde cellulose. This reaction proceeds without significant side reactions [9]. Conversion of dihydroxyl groups to dialdehyde by periodate oxidation is a useful method widely used in derivatization of cellulose to activate the polymer to further reactions [10-12]. Partial periodate oxidation of cellulose leads to polymers having aldehyde groups in addition to the primary and secondary hydroxyl groups, which broadens the range of applications of cellulose, as well as cotton fibers [13,14].

The most pure natural form of cellulose is cotton. Cotton fibers are one of the most important types of fibers in the world, despite the high volume of wood cellulose and increasing number of synthetic fiber types available. Cotton is widely used in clothing fields due to its excellent characteristics including regeneration, biodegradation, softness, affinity to skin, hygroscopic property, strength and dyeability. Also, cotton is the base raw material for a wide assortment of

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products, such as paper, chemicals, food additives, medical supplies, some cosmetic ingredients [15-17], etc.

As is known, the chemical composition, macro- and microstructure of cotton fibers have significant influence on the properties of the final cellulose products. Cotton fibers consist of almost chemically clean cellulose, and have a crystalline/amorphous microfibrillar structure. Elementary fibrils are built from ordered crystallites and less-ordered amorphous regions statistically alternated along the fibril. Lateral tie molecules connect laterally adjacent amorphous regions [18]. The ratio between crystalline and amorphous areas (degree of crystallinity) strongly influences the characteristics of any fiber.

Oxidation of cellulose causes changes in the structure and crystallinity of the resulting molecules and affects its chemical and sorption properties. For a better understanding of the behavior of textile fibers during the wet finishing processes a knowledge of the correlation between their structure and their sorption properties is very important. Sorption properties of fibers can be obtained on the basis of various methods for determining water vapor sorption, water retention value, iodine and dye sorption, acid hydrolysis, and other methods [19-21]. In order to characterize cellulose accessibility, interaction with water is often employed, which is able to destroy weaker hydrogen bonds but cannot penetrate into the regions of high order. The amount of adsorbed water is a very important indicator for the determination of sorption properties. The iodine sorption test is a convenient empirical method of measuring cellulose accessibility.

In the literature there are no references about the effect of selective oxidation of periodate on the sorption properties of cotton fiber. In this paper, the influence of sodium periodate oxidation on the chemical and sorption properties of cotton yarn was investigated. The sorption properties of cotton fibers having different aldehyde contents achieved by varying the parameters used during oxidation (concentration of sodium periodate and period of time) were defined by moisture sorption, water retention power and iodine sorption value. The present study aims to gain a better understanding of the changes in the structure of cotton fibers during periodate oxidation, by studying the changes in their sorption properties.

## EXPERIMENTAL

### Materials

Raw cotton ring yarn (fineness: 20.85 tex,  $CV = 3.4\%$ ; yarn twist: 755 t.p.m.,  $CV = 5.5\%$ ), which is intended for gauze production, was obtained from

Strumičanka (Strumica, FYR Macedonia). All chemicals used were of analytical grade.

### Preparation of periodate oxidized cotton

A sample of cotton yarn was immersed in solutions of sodium periodate in 0.1 M acetic buffer (ratio 1:50, w/v), pH 4.0 at concentrations of 2.0 and 4.0 mg/ml, *i.e.*, 0.2 and 0.4% (w/v). The mixture was then stirred in the absence of light, at room temperature, for 15, 30, 45, 60, 120, 180, 240, 300 and 360 min. After completion of the oxidation, the cotton yarn was washed thoroughly with ice-cold distilled water on a filter paper set in a Büchner funnel to remove the oxidant. The obtained oxidized cotton samples were then dried at room temperature for 72 h.

### Determination of weight loss

Loss in weight of oxidized cotton yarn samples, as a result of chemical treatment, was determined by applying the direct gravimetric method [22].

### Determination of aldehyde group content

The aldehyde content present in the oxidized cotton was measured according to the method described in literature [23-25]. The aldehyde groups were selectively oxidized to carboxyl groups with sodium chlorite at pH 4-5, at room temperature for 48 h, and carboxyl group content was determined by modified calcium-acetate method [24]. Before titrations all cotton samples were ion-exchanged into acid form by suspending in 0.01 M HCl for 1 h, followed by washing with distilled water. The aldehyde group content was calculated by subtracting the carboxyl content value determined in the starting cotton sample from that of chlorite oxidized samples.

### Determination of moisture sorption

Moisture sorption of oxidized cotton was determined according to standards ASTM D, 2654-76. Cotton samples were exposed to standard atmosphere:  $20 \pm 2$  °C,  $65 \pm 2\%$  relative humidity, for 24 h (ASTM D, 1776-74). Moisture sorption was calculated as weight percentage of absolute dry material. Reported values for each cotton sample (raw and oxidized) are the mean values of three separate determinations.

### Determination of water retention value

Water retention power of cotton fibers was determined in triplicate by standard centrifuge method (ASTM D, 2402-78). This method is based on a determination of the quantity of water which the fibers can absorb and retain under strictly controlled conditions. This property is expressed as a ratio between the

mass of water retained in the fiber after soaking and centrifuging, and the mass of absolute dry sample.

### Determination of iodine sorption value

The iodine sorption of the cotton fibers was evaluated using the Schwertassek method [26–28]. Cotton sample (0.3 g) is treated with 2 ml of iodine solution  $KI_3$ , prepared from 5 g  $I_2$ , 40 g  $KI$  and 50 ml  $H_2O$ , for 3 min, and then 100 ml saturated sodium sulphate (200 g/l) was added and shaken for 1 h. The iodine concentration of the sample and blank was determined by titration with 0.02 M sodium thiosulphate and the iodine sorption values (*ISV*) in mg  $I_2$  per g of sample was calculated as follows:

$$ISV = \frac{(b-t)(102M)(126.91M)}{m_a} = \frac{2.04(b-t) \cdot 2.54}{m_a} \text{ (mg/g)} \quad (1)$$

where  $b$  is volume (ml) of  $Na_2S_2O_3$  solution for blank titration,  $t$  is volume (ml) of  $Na_2S_2O_3$  solution for the titration of sample solution,  $M$  is the molarity of the sodium thiosulphate, 102 is a total volume (ml) of the solution, and  $m_a$  is the weight of absolute dry cotton yarn (g).

According to Schwertassek, the absorption takes place in the amorphous phase. A ratio of *ISV* per g cellulose to 412 (mg iodine absorbed per 1 g of methyl cellulose) determines the amorphous fraction. The crystallinity index (*CrI*) was calculated using following equation [26]:

$$CrI = 100 - \left( 100 \frac{ISV}{412} \right) (\%) \quad (2)$$

## RESULTS AND DISCUSSION

Cotton yarn was oxidized with sodium periodate to cleave the 2,3-vicinal diol of the anhydroglucose units and produce aldehyde groups at those two positions (Figure 1). This reaction is thought to proceed via a cyclic diester of periodate ion with vicinal hydroxyls, which subsequently undergoes an intramolecular redox process with C-C bond cleavage of the cellulose glucose units, according to a concerted mechanism [29].

In this study, several oxidations with different reaction time and different concentration of sodium

periodate solution were carried out. The effect of periodate oxidation on cotton fibers was initially assessed by determining the aldehyde group content. Figure 2 shows the effect of oxidation time and concentration of sodium periodate on CHO group content in cotton fibers. The aldehyde group content in unmodified cotton was 26  $\mu\text{mol/g}$  cellulose. During the periodate oxidation of cotton fibers, there was no increase in aldehyde group content during the first 30 min of oxidation; after that, with increase of oxidation time, the aldehyde group content in oxidized fibers increased for both periodate concentrations used. The cotton fibers oxidized with 0.4%  $NaIO_4$  had higher increase in aldehyde group content (up to 282%) compared to the fibers oxidized with 0.2%  $NaIO_4$  (increase up to 210%). The maximum amount of aldehyde groups, 99.2  $\mu\text{mol/g}$  cellulose, was introduced into the cotton fibers oxidized under the most severe conditions (0.4%  $NaIO_4$ , 360 min).

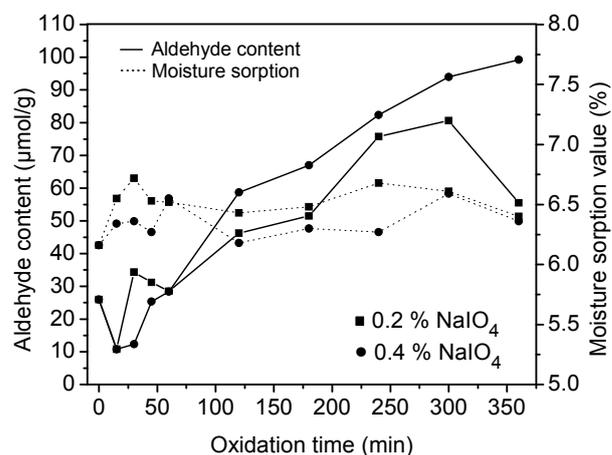


Figure 2. Aldehyde group content and moisture sorption value of the cotton fibers oxidized with different concentrations of  $NaIO_4$  and for different oxidation times.

During the first 30 min of oxidation treatment, periodate was consumed probably by non-cellulosic material, since raw unbleached cotton yarn was used in this study. The non-cellulosic material at the cotton fiber surface was determined to be a complex mixture of fatty acids, alcohols, alkanes, esters and glycerides [30]. This is in agreement with the weight loss data of oxidized cotton, namely samples of cotton yarn oxidized during the first 30 min showed the weight loss

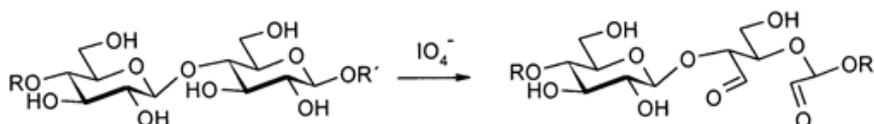


Figure 1. Periodate oxidation of cellulose.

values of ~2.5%, which remained almost constant with increasing reaction time and periodate concentration (data not shown).

Periodate oxidation of cellulose is a complex process, since it proceeds gradually from the amorphous to the crystalline phase. The random initial oxidation occurs in the amorphous region of cellulose, followed by the oxidation of the surface of crystallites. Prolonged oxidation times and higher periodate concentrations could be necessary to access into the inner region of the polymer [6]. The oxidation of fibers with periodate has been shown to cause an uneven distribution of dialdehyde groups [4].

The molecular structure (chemical structure, degree of polymerisation, molecular mass) and supramolecular structure (degree of crystallinity, molecular orientation, amorphous regions and void fractions) have a strong influence on sorption properties of the fibers. Differences in molecular and supramolecular structure of original and oxidized cotton fibers cause different sorption properties of fibers which have been evaluated by determination of moisture sorption, water retention power and iodine sorption value.

The most significant influence on the adsorption properties of fibers have the accessible regions - the part of less ordered amorphous regions and void system. The ordered regions (crystalline) do not contribute significantly to the process of water adsorption. Actually, X-ray studies have shown that water molecules do not penetrate the crystalline region [31]. Thus, the moisture must be absorbed in the amorphous regions and on the surfaces of crystallites [32]. Free hydroxyl groups at the cotton fibers amorphous regions and at the crystallites' surfaces are responsible for the moisture sorption. The sorption of water starts with the formation of a strongly bound monolayer, where one molecule of water is bonded to each accessible hydroxyl group. Additional water molecules are bound to the present monomolecular layer by hydrogen bonds, forming additional water layers [33]. Therefore, moisture sorption values yield information on the extent of areas accessible to water vapor within a fiber.

Moisture sorption values obtained for unmodified and modified cotton fibers are presented in Figure 2. Moisture sorption value of oxidized cotton fibers increased during the first 30 min of oxidation, from starting 6.16 to 6.72% and 6.36% for samples oxidized with 0.2 and 0.4% NaIO<sub>4</sub>, respectively. Cotton samples oxidized for 60 min exhibited almost the same moisture sorption value of ~6.5%. When the reaction time of oxidant was over 60 min, the moisture sorption values remained more or less constant

for fibers oxidized by 0.2% NaIO<sub>4</sub>, while for cotton fibers oxidized by 0.4% NaIO<sub>4</sub> firstly decreased, and then became nearly constant at 6.34%. Generally, oxidized cotton fibers exhibited small changes in moisture content. In comparison with unmodified cotton fiber, the highest increase of moisture sorption was 9%, obtained for the sample oxidized for 30 min with 0.2% NaIO<sub>4</sub>.

The sorption properties of the fibers depend on less ordered amorphous regions, where the sorption processes take place, and therefore accessible hydroxyl groups are present in these regions. In periodate oxidized fibers, a number of accessible OH groups are converted to the aldehyde groups, which are probably only moderately less prone to absorb water molecules than the hydroxyl groups. Thus, oxidized fibers would be expected to exhibit lower moisture sorption values [34]. In addition, periodate creates covalent crosslinks in the fiber and this both improves the integrity of the fiber and removes possible sites for water sorption. A possible explanation of the higher moisture content after the periodate treatment is that, the impurities at the surface of raw cotton fibers, which represent a hydrophobic blockage, were partially removed in the early stages of oxidation, thus enabling water molecules to access cellulosic moisture-absorbing materials at the surface.

The interactions of cotton fibers with liquid water represent the total swelling of fibers which includes wide expansion of amorphous regions. When cellulose fibers are immersed in water, they absorb a large quantity of water and swell significantly. Water retention power represents the quantity of water which is retained in fibers after prescribed soaking in water and centrifugation. This quantity changes with supramolecular fiber structure as it represents a measure for the fibers' absorption ability. The total water holding capacity of a fiber can be estimated by determining water retention values. All water absorbing and holding surfaces, cracks, and cavities are included with the water retention measurement.

Water retention values (*WRVs*) of unmodified and cotton fibers oxidized under different condition are presented in Figure 3. The obtained data showed that the *WRVs* of oxidized cotton fibers strongly decreased (starting from 30.6 to 22.7% and 22.4% for fibers oxidized with 0.2 and 0.4% NaIO<sub>4</sub>, respectively) during the first 30 min of modification. With further oxidation, a continuous slow decrease of *WRVs* was observed for fibers oxidized with 0.2% NaIO<sub>4</sub> to the lowest value of 19.9% (*i.e.*, 65% of the original value). In the case of fibers oxidized with 0.4% NaIO<sub>4</sub>, *WRVs* became nearly constant at 21.3%, with the lowest va-

lue of 19.0% reached after 180 min of treatment time. The obtained results can be interpreted as the presence of cross-links due to the aldehyde functionalities that were introduced into the fibers by periodate oxidation. Once formed in the cellulose fibers, the aldehyde groups are able to subsequently react with adjacent hydroxyl groups in the fiber to form hemiacetal linkages, as suggested in Figure 4. These hemiacetal links are responsible for the crosslinking. The aldehyde groups form stable, covalent hemiacetal linkages in dialdehyde cellulose, both intra- and intermolecularly [7]. This crosslinking effect makes the molecules more compact and removes possible sites for water sorption. Therefore, decrease in water retention value of crosslinked cotton fibers was caused probably both by the swellability reduction by the network crosslinking structures [35] and by eliminating adsorption sites available for water sorption.

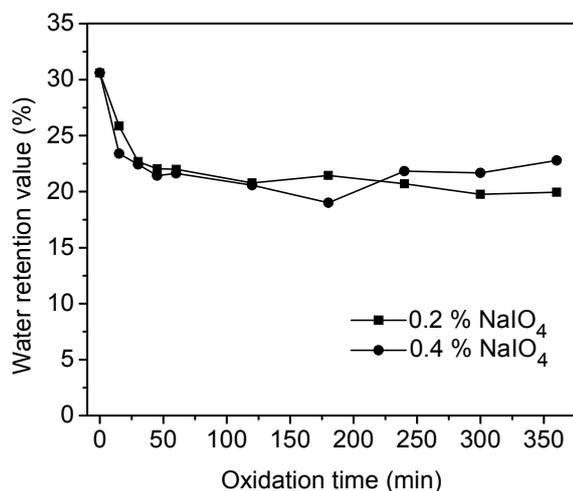


Figure 3. Water retention values of the cotton fibers oxidized with different concentrations of NaIO<sub>4</sub> and for different oxidation times.

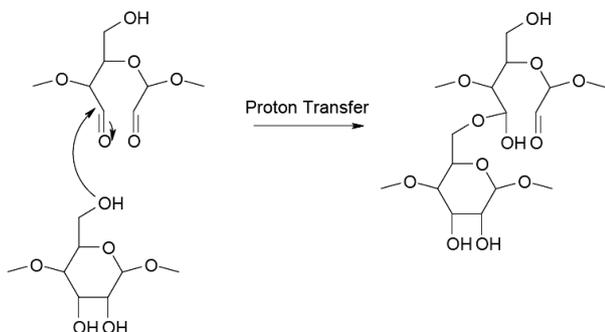


Figure 4. Schematic representation of a possible mechanism for the cross-linking reaction.

Although a difference in the aldehyde contents was observed between the samples oxidized with 0.2

and 0.4% NaIO<sub>4</sub>, especially for 360 min, nearly equal values of moisture sorption and water retention were observed between them. Most probably, with increasing amounts of aldehyde groups hemiacetal linkages were formed until saturation was achieved, thus the aldehyde groups further introduced by oxidation did not participate in crosslinking.

Studying the sorption of iodine which takes place only in less ordered amorphous regions of cellulose is a classical method for the determination of sorption properties. The iodine sorption value is a measure of cellulose accessibility to aqueous solutions in the fiber amorphous areas and it is inversely proportional to the fiber crystalline phase, expressed by the crystallinity index [33,36]. Iodine sorption mechanism differs from water sorption in cellulose fibers. During the penetration of iodine into amorphous regions of fibers, electrostatic interactions appear between partly positively charged hydrogen atom of the cellulose hydroxyl group and negative charge of three-iodide ion (built up when an iodide ion is added to an iodine molecule). Contrary to water, iodine does not adsorb in multimolecular layers, but only in a monomolecular layer to available hydroxyl groups of the cellulose. Also, iodine does not entirely fill up the available volumes of voids in fibers such as water. Therefore, the results of determining iodine sorption value may be interpreted in terms of monolayer adsorption and hence may be used to calculate internal surface areas [36,37].

The iodine sorption values (*ISVs*) for unmodified and oxidized cotton fibers are shown in Figure 5. As can be seen from the values obtained, the different oxidation conditions differently influenced the accessibility of the oxidized cotton fibers. The *ISVs* of cotton fibers oxidized with 0.4% NaIO<sub>4</sub> firstly decreased with increasing oxidation time to a minimum value of 46.0 mg/g, then increased to a maximum value of 61.4 mg/g, and with further oxidation became nearly constant at 54.2 mg/g, which is 97.5% of the *ISV* of unmodified cotton fibers. Cotton samples oxidized with 0.2% NaIO<sub>4</sub> showed decrease in *ISVs* reaching the lowest value of 38.3 mg/g, obtained for sample oxidized during 120 min; after that, almost linear increase of *ISVs* was observed approaching a maximum value of 57.9 mg/g for sample oxidized for 360 min. Reduced *ISVs* for oxidized fibers are a consequence of the introduction of aldehyde groups during the oxidative treatment of cellulose, and the subsequent formation of crosslinks.

The three-iodide anion was incorporated into the fiber by specific interaction with partly positive hydrogen of the hydroxyl group of the cellulose. In the

case of the higher degree of crystallinity, these groups were included in the crystallites and therefore blocked (not accessible). This means that  $ISV$  is lowered with the reduction of the number of OH groups, or with the increase of the degree of crystallinity [37].

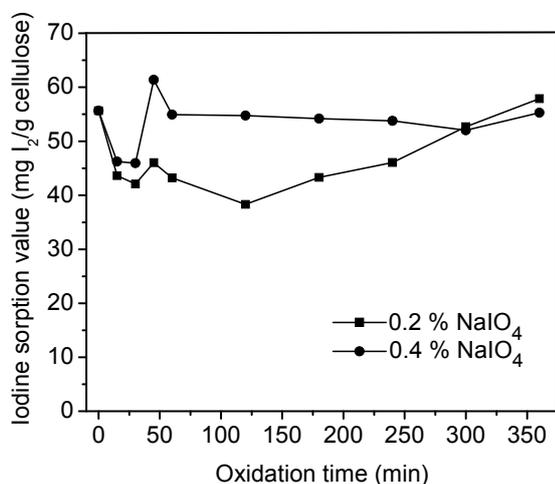


Figure 5. Iodine sorption values of the cotton fibers oxidized in different conditions.

According to the results presented in Figure 5, cotton fibers oxidized with 0.4% NaIO<sub>4</sub> had greater amounts of specific adsorption sites accessible for iodine anions in comparison with fibers modified with 0.2% NaIO<sub>4</sub>. Since cotton fibers oxidized with 0.4% NaIO<sub>4</sub> had higher aldehyde group content (i.e. lower amount of hydroxyl group) compared to the fibers oxidized with 0.2% NaIO<sub>4</sub> (Figure 2), the obtained differences in  $ISV$ s for fibers oxidized with 0.2 and 0.4% periodate are due to the changes in the degree of crystallinity and cross-linking. The crystallinity index of oxidized cotton fibers, calculated on the basis of  $ISV$ , increased from 86.5% (unmodified fibers) to 88.8%, then decreased to 85.1% and after 60 min of oxidation became nearly constant at 86.9% for fibers oxidized with 0.4% NaIO<sub>4</sub>, while for fibers oxidized with 0.2% NaIO<sub>4</sub> firstly increased to 90.7% and then slowly decreased to a value of 85.9% (data not shown). This can be explained by the fact that the oxidation by sodium periodate, especially at higher concentration, and long oxidation time, breaks to some extent the crystalline structure of cellulose in the native cotton yarn, as well as the reorganization of less ordered amorphous fraction of oxidized samples due to the crosslinking effect.

## CONCLUSION

The cotton fiber was oxidized to dialdehyde cellulose by sodium periodate oxidation reaction. The

oxidized cotton samples having different oxidation level were obtained with adjusting the periodate concentration and oxidation time. The maximum amount of aldehyde groups, 99.2  $\mu\text{mol/g}$ , was introduced into the cotton fibers oxidized under the most severe conditions (0.4% NaIO<sub>4</sub>, 360 min). It has been shown that the introduced aldehyde groups have considerable influence on the sorption properties and the accessibility of oxidized cotton fibers. Compared to the unmodified fibers, oxidized cotton samples exhibited higher moisture sorption (increase up to 9%) and lower water retention values (WRV up to 19%, i.e. decrease up to 38%) and iodine sorption values (decrease up to 31%). From the obtained results it is evident that the water retention value exhibits the highest sensitivity to the changes in the structure of oxidized cotton fibers, followed by less sensitive iodine sorption value. In comparison, the moisture sorption exhibits the lowest sensitivity to changes in fiber structure. Thus, the periodate oxidation allows unique chemical modifications selectively in disordered regions and on crystal surfaces together with some morphological changes of cotton fibers under aqueous and moderate conditions.

Modern hygienic materials are composite products, designed with various functional components, and it is sometimes desirable to have layers exhibiting specific hydrophilic/hydrophobic gradients across the thickness of the absorbent structure. Gaining control over fiber wettability may allow cellulose to be applied in those cases where traditionally only synthetic materials have so far been used. Whole-cellulose composite hygienic materials would be of unquestionable advantage due to the "green" aspects of such products as well as the economy and user friendliness of natural fibers.

## Acknowledgment

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## Nomenclature

$CV$  - Coefficient of variation  
 t.p.m. - twist per meter  
 $WRV$  - Water retention value  
 $ISV$  - Iodine sorption value

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NAUČNI RAD

## SORPCIONA SVOJSTVA PAMUKA OKSIDOVANOG PERJODATOM

*U ovom radu ispitan je uticaj uslova perjodatne oksidacije na hemijska i sorpciona svojstva pamučne pređe preko određivanja sadržaja aldehidnih grupa, sorpcije vlage, sposobnosti zadržavanja vode i sorpcije joda. Oksidacija pamučne pređe izvedena je pri različitim koncentracijama rastvora natrijum-perjodata i vremenima reakcije. Sadržaj aldehidnih grupa u oksidovanom pamuku određen je na sledeći način: aldehidne grupe su selektivno oksidovane do karboksilnih grupa pomoću natrijum-hlorita pri pH 4-5, na sobnoj temperaturi u toku 48 h, a zatim je sadržaj karboksilnih grupa određen modifikovanom kalcijum-acetatnom metodom. Razlike u sorpcionim svojstvima nemodifikovanog i uzoraka oksidovanog pamuka dobijene su upotrebom konvencionalnih metoda. Rezultati su pokazali da su aldehidne grupe uvedene u oksidovana pamučna vlakna i do 99,2  $\mu\text{mol/g}$ . U poređenju sa nemodifikovanim vlaknima, oksidovani pamučni uzorci su pokazali neznatno povećanje sorpcije vlage (do 9%) i niže vrednosti sposobnosti zadržavanja vode (do 19%) i sorpcije joda (do 31%). Na osnovu rezultata sprovedenih ispitivanja može se zaključiti da se pod primenjenim uslovima perjodatne oksidacije kristalnost celuloze znatno ne menja, što je veoma značajno u procesima proizvodnje tekstilnih materijala.*

*Ključne reči: pamuk; oksidacija natrijum-perjodatom; sorpcija vlage; sposobnost zadržavanja vode; sorpcija joda.*