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Effect of hemicelluloses and lignin on the sorption and electric properties of hemp fibers

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ABSTRACT

This study investigated the influence of hemicelluloses and lignin removal on the sorption and electric properties, as well as fineness of hemp fibers. Hemp fibers with different content of either hemicelluloses or lignin were obtained by chemical treatment with 17.5% sodium hydroxide or 0.7% sodium chlorite. The sorption properties of hemp fibers were evaluated as moisture and iodine sorption, while electric properties were evaluated as volume electric resistance. Hemicelluloses removal increases in higher extent fiber liberation than lignin removal; the modified hemp fibers were finer up to 12 times in relation to unmodified fibers. Furthermore, hemicelluloses removal increased the moisture and iodine sorption, as well as electric resistance of hemp fibers compared to unmodified fibers, while lignin removal decreased both moisture and iodine sorption, and only slightly increased the electric resistance of modified hemp fibers.

1. Introduction

Intensive growth of world population imposes the need for comfortable, biodegradable, biocompatible and ecological fibers. Textile fibers and textile materials are in continuous contact with consumer bodies during their use and comfort of clothing materials, among the other things, implies hygienic materials with good sorption properties and low values of electric resistance. Good sorption properties of clothing material mean extremely quick absorption of humidity accompanied with quick drying. High values of the electric resistance of these materials are causing static electricity, which create unfavorable effects. Some of these effects include increased dirt, cleaning problems and increased tendency of materials to form the rolled-up ends of fibers on their surface. Also, static electricity produces expressive unpleasant sensation to human bodies during the utilization and some physiological disturbances (which not yet been definitely solved), which appear like pathological reactions of the nervous system, heart and blood vessels (Asanovic, 2003; Asanovic et al., 2007).

Therefore, good sorption properties and low value of static electricity are an important task in textile industry, especially for production of clothing, working and protection textiles materials. After quite a long period of intensive application, synthetic fibers, created to replace natural fibers, are today considered inferior to natural ones, especially in respect to comfort and ecological properties. From this point, the total substitution of natural with synthetic fibers is not desirable. These facts, together with limits in the yield of cotton, the main comfort providing fibers, were main reasons for the beginning of an unexpected, worldwide return to almost forgotten bast fibers, among them hemp (Keller et al., 2001; Kostic et al., 2008; Kozlowski et al., 2000; Mankowski, 2003; Muessing et al., 1998; van der Werf and Turunen, 2008).

Technical (multi-cellular) hemp fibers, obtained from hemp plant (*Cannabis sativa*), are composed of elementary fiber bundles. The middle lamella joining the elementary fibers is mostly build of the woody component-lignin, while the interfibrillar regions are fulfilled with hemicelluloses. Each elementary fiber can be considered as a network of ultrafine cellulose fibrils embedded in a matrix of hemicelluloses and lignin. The elementary fiber bundles are in a further order integrated into the multi-cellular fiber by means of the plant glue, pectin, which also takes part in the construction of the hemp fiber cell wall. Chemical composition of hemp fibers is very heterogeneous. In raw hemp fibers the cellulose content is about 67–78%, the rest is approximately 5.5–16.1% hemicelluloses, 3.7–8% lignin, 0.9–4.3% pectin, and some fats and waxes (Buschle Diller et al., 1999; Mark, 1954; Pejic et al., 2008; Wang et al., 2003).

As a textile fiber, hemp fiber has the specific properties, namely aseptic properties, high absorbency and hygroscopicity, good thermal and electrostatic properties, as well as good UV protection properties and lack of any allergenic effect, that make them different from other fibers. However, high quantity of noncellulosic components in hemp fibers (hemicelluloses, lignin, pectin and

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Table 1
The chemical treatment scheme and list of samples.

Modification conditions			Sample code
Concentrations of modification means	Temperature	Time, min	
Unmodified sample–control	–	–	C
17.5% NaOH	Room temperature	5	H5
		10	H10
		30	H30
		45	H45
0.7% $NaClO_2$	Boiling temperature	5	L5
		15	L15
		30	L30
		60	L60

waxes) and impurities negatively influence further fiber processing and fiber properties (fineness, elasticity, evenness, and sorption properties). In order to make them finer, cleaner, softer, and more suitable for processing on machines of higher efficiency than traditional hemp machines, numerous chemical, mechanical, enzymatic and combined treatments of hemp fibers are applied which are mostly directed towards elimination of hemicelluloses, lignin and pectin. Modification of hemp fibers has to be performed in such way that attaining the desired goal is not followed by decreasing the most important positive properties of hemp fiber, especially very important comfort properties of fibers – their relation to water (vapor or liquid) and low static electricity charges, which defines their physiological properties (Buschle Diller et al., 1999; Kostic et al., 2008; Pejić et al., 2006; Pejic et al., 2008; Thomsen et al., 2006; Wang et al., 2003; Wong et al., 1997).

In this paper the influence of the hemicelluloses and lignin removal on the sorption and electric properties, as well as fineness of hemp fibers was studied. Hemp fibers with different content of either hemicelluloses or lignin were obtained by chemical treatment with 17.5% sodium hydroxide or 0.7% sodium chlorite. The first treatment progressively removed hemicelluloses keeping the lignin content unchanged and the last vice versa. The sorption properties of hemp fibers were evaluated as moisture and iodine sorption, while electric properties were evaluated as volume electric resistance. On the bases of the results obtained in this study, an attempt has been made to explain the individual roles of the hemicelluloses and lignin removal on the fineness, sorption properties and electric resistance of the chemically modified hemp fibers.

2. Experimental

2.1. Materials

Domestic water-retted long hemp fibers from Backi Brestovac (Serbia) were used in this investigation. The content of long fibers in the bundle was 74%, of short and tangled ones 24.2% and of shives and dust 1.8%. All used chemicals obtained from commercial sources are p.a. grade.

2.2. Chemical treatment

Hemp fibers were modified by chemical treatments in order to gradually remove either hemicelluloses or lignin according to the procedure described in literature (Garner, 1967; Pejic et al., 2008). In brief, the progressive removal of hemicelluloses and keeping the lignin content unchanged was brought by treating the fiber samples with 17.5% sodium hydroxide solution, 1:50 liquor ratio, at room temperature, for 5, 10, 30 and 45 min, followed by neutralization with 1% acetic acid, washing and drying. The progressive removal of lignin and keeping the hemicelluloses content unchanged was achieved by treating hemp fibers with 0.7% sodium chlorite at pH 4, 1:50 liquor ratio, at boil temperature, for different periods of time (5, 15, 30 and 60 min), followed by washing and drying. The chemical treatment scheme and list of samples are shown in Table 1.

2.3. Determination of chemical composition

Chemical composition of unmodified sample and each of modified samples was determined according to the scheme of Soutar and Bryden (Garner, 1967) by successively removal of water solubles, fats and waxes, pectin, lignin and hemicelluloses. Reported values are the mean values of three parallel determinations.

2.4. Fineness of modified hemp fibers

Fineness in tex was determined as per standard method (SRPS F.S2.212, 1963) by dividing the mass of fibers by their known length.

2.5. Scanning electron microscopy (SEM) analysis

Scanning electron microscopy (SEM) photographs were taken on a FE-SEM JEOL JSM-6330 F instrument operating at 2 μV after sputtering with gold.

2.6. Determination of moisture sorption

Moisture sorption of hemp fibers was determined according to standards (ASTM D 2654-76, 1976). Hemp fibers were exposed to standard atmosphere: 20 ± 2 °C, 65 ± 2% relative humidity, for 24 h (ASTM D 1776-74, 1974). Moisture sorption was calculated as a weight percentage of absolute dry material. Reported values are the mean values of three separate determinations. Variation between analyses was less than 8% in the case of alkali modified fibers and less than 5% in the case of sodium chlorite modified fibers.

2.7. Determination of iodine sorption

The Schwertassek method was used for evaluation of the sorption properties of the hemp fibers (Nelson et al., 1970; Siroka et al., 2008; Stankovič Elesini and Pavko, 2002). The hemp fibers (0.3 g) were immersed in 2 ml of iodine solution KI_3 prepared from 5 g I_2, 40 g KI and 50 ml H_2O for 3 min, 100 ml saturated sodium sulphate ($w(Na_2SO_4)$ = 200 g/l) was then added and shaken for 1 h. The iodine concentration of the sample and blank was determined by titration with sodium thiosulphate (0.02 mol/l). The iodine sorption value (ISV) in mg I_2 per 1 g of sample was calculated as follows:

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

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 (mol/l), 102 is a

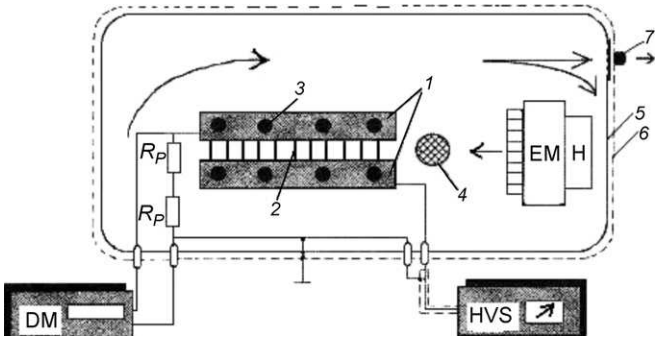

Fig. 1. The scheme of the device for measuring the volume electric resistance of fibers.

total volume (ml) of the solution, and m_a is the weight of absolute dry hemp fibers (g). Reported values are the mean values of three separate determinations. Variation between analyses was less than 8.6% in all cases.

2.8. Determination of volume electric resistance of hemp fibers using voltage method

The volume electric resistances of fibers were determined by the device, which was developed at the Textile Engineering Department of the Faculty of Technology and Metallurgy at the University of Belgrade (Asanovic, 2003; Asanovic et al., 2007). The scheme of this device, with electrodes adjusted for the measurement of volume electric resistance of fibers using voltage method, is given in Fig. 1.

The sample of parallel sheaf of fibers (2), whose fineness is 1000 tex, is clamped between the electrodes (1) by means of screws (3). The adjustable plate electrodes have been electrolytically silver-plated to achieve a stable electrode resistance. It is well known that silver oxide has a resistance similar to that of metallic silver. The electrodes are serially connected with protective known resistors (R_p) to the high voltage source (HVS) ($E = 1200$ V). Electrodes, placed into a chamber (5) that provides controlled measurement conditions, are fixed at distance of 1 cm. The shield from the external electric fields, which could produce unfavourable effects on the precision of the measurement, is realized by Faraday's cage (6). Air humidity in the chamber is established by humidifier (H), and registered by the sensors (4) of the digital-measuring device. Electromotor (EM), with a compression circuit, is providing air circulation inside the chamber. Aperture (7) is providing a connection between the chamber and the external atmosphere.

Volume electric resistance of fibers was determined by voltage method measuring a fall voltage (U_m) on the protective known resistor ($2R_p$). The voltage fall was registrated using a digital "Philips" multimeter (DM) Model PM 2528, whose resistance R_i is 10 MΩ. Volume electric resistance of fibers was determined by equation:

$$R = \frac{2R_pR_i}{2R_p + R_i}\left(\frac{E}{U_m} - 1\right) \approx \frac{2R_pR_i}{2R_p + R_i} \cdot \frac{E}{U_m}$$

where $R \gg R_p$, $R \gg R_i$, $E \gg U_m$, $R_p = 820$ kΩ, $R_i = 10$ MΩ, and $E = 1200$ V.

3. Results and discussion

3.1. Influence of chemical treatment on chemical composition of hemp fibers

The chemical compositions of modified hemp fibers and those of the control sample are given in Table 2.

The level of hemicelluloses removal during the alkaline treatment was high, while lignin content was almost unchanged. Content of hemicelluloses decreased up to 70% in relation to unmodified fibers. In contrast to hemicelluloses, lignin showed low reactivity during the alkaline treatment, mainly because of strong carbon–carbon linkages and aromatic groups and rings, which were very resistant to chemical attack (Wang et al., 2003). The sodium chlorite treatment of hemp fibers progressively removed lignin up to 50% in relation to unmodified fibers, with slightly changed hemicelluloses content.

Changes in hemp fibers composition, i.e. the separate removal of hemicelluloses and lignin, affect the structure and properties of modified fibers, among them fiber fineness, sorption and electric properties, which are very important for both fiber processing and performance.

Surface changes of hemp fibers as a result of the separate removal of hemicelluloses and lignin can be seen by comparison of scanning electron micrographs of unmodified and modified hemp fibers presented in Fig. 2. As a natural bast fiber, hemp showed great variation in multi-cellular fiber diameter. The fiber surface appeared relatively rough and uneven, the fibril bundles within the fiber seemed to be embedded in a somewhat soft (i.e. a resinous matrix of lignin and hemicelluloses) (Fig. 2a). After only 5 min of alkali treatment, there was surface peeling in various areas along the fiber followed by elementar fiber liberation (Fig. 2b), which is more pronaunce with longer treatment. In the case of lignin removal (sodium chlorite treatment) surface peeling of fibers is very intensive, disclosing smoother looking layers. Even after 5 min chlorite treatment the resinous material around the fibril bundles seemed to have been mostly removed.

As result of removing noncellulosic substances, hemp fibers acquired a high level of divisibility, which determines important properties of hemp fibers – their fineness, Table 2. Hemicelluloses removal increases in higher extent fiber liberation than lignin removal; fiber fineness was reduced from 21.5 tex for unmodified to 1.8 tex for H45 sample.

Table 2
The chemical compositions of unmodified and modified hemp fibers.

Sample code	Hemicelluloses		Lignin		Fineness, tex
	Content, %	Removed, %	Content, %	Removed, %	
C	10.72	–	6.06	–	21.5
H5	4.69	56.25	5.66	6.60	4.2
H10	4.17	60.91	5.12	15.51	3.8
H30	3.29	69.31	5.18	14.52	2.1
H45	3.59	66.51	5.41	10.73	1.8
L5	8.89	17.07	4.09	32.51	9.2
L15	9.78	8.77	3.74	38.28	8.6
L30	8.72	18.66	3.57	41.09	8.2
L60	8.99	16.14	3.09	49.01	8.0

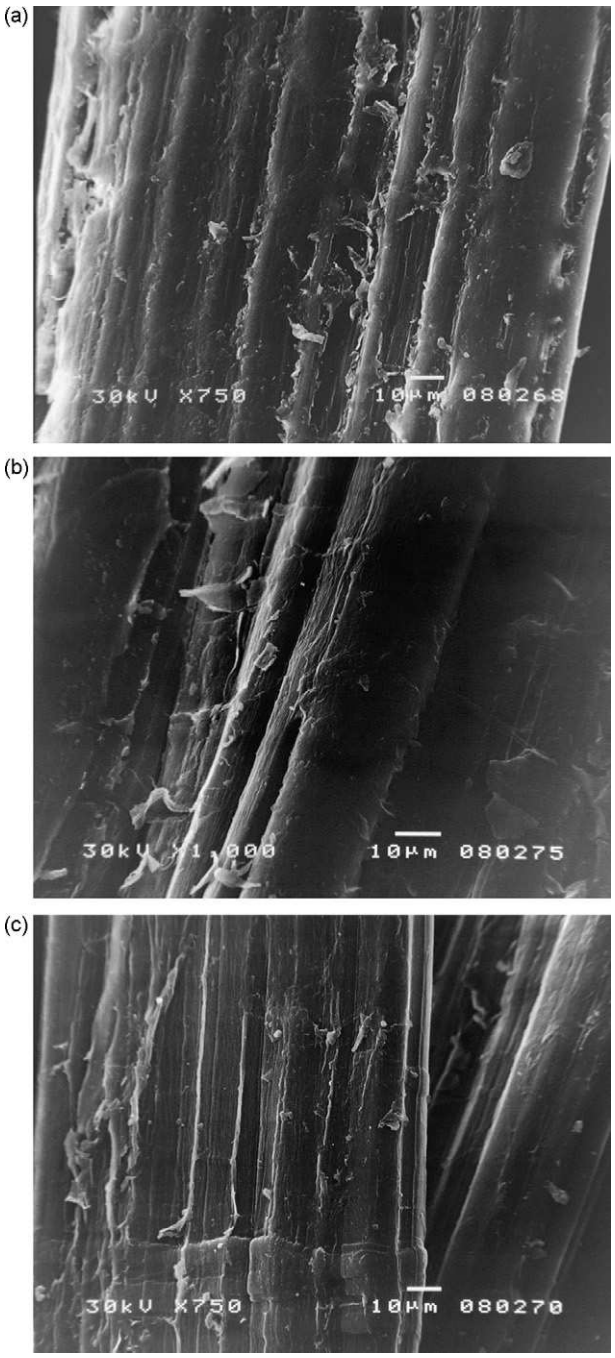

Fig. 2. SEM images of the surface of: (a) unmodified hemp fiber; (b) hemp fibers modified with 17.5% NaOH, 5 min (sample H5); (c) hemp fibers modified with 0.7% $NaClO_2$, 5 min (sample L5).

3.2. Influence of hemicelluloses and lignin removal on moisture and iodine sorption of hemp fibers

As it has been reported earlier (Kostic et al., 2008; Pejic et al., 2008) together with the hemicelluloses and lignin removal during the chemical modification of hemp fibers either by alkaline or sodium chlorite treatment, the fibrous morphology of hemp fibers has been changed, depending on the modification conditions. Changes in hemp fibers chemical composition, crystallinity and pore structure during the modification affect the sorption properties which have been evaluated by determination of moisture sorption (MS) and iodine sorption value (ISV). Moisture and iodine sorption values for unmodified and modified hemp fibers are presented in Fig. 3.

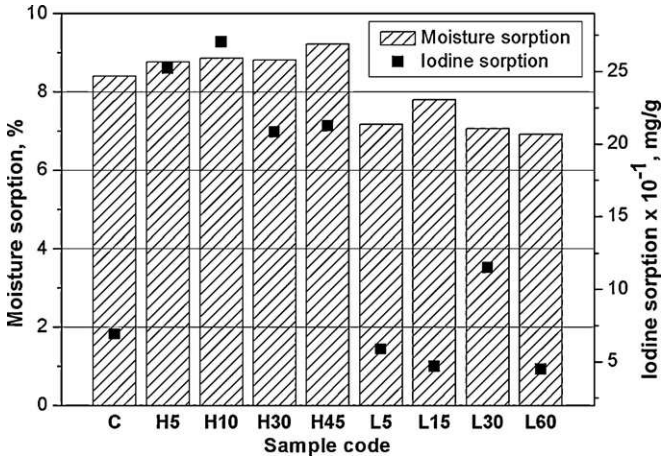

Fig. 3. Moisture and iodine sorption of unmodified and modified hemp fibers.

Moisture sorption values yield information on the extent of areas accessible to water vapor within a fiber. Free hydroxyl groups at the hemp fiber amorphous regions and at the crystallites' surfaces are responsible for the moisture sorption. The sorption of water vapor starts with the formation of a monolayer, where one molecule of water is bonded to each accessible hydroxyl group and continues with the formation of a multilayer of progressively increasing thickness. The mechanism of iodine sorption differs from water sorption at the partially-positive hydrogen atoms of the cellulose polar hydroxyl group because three-iodide ions (built up when an iodide ion is added to an iodine molecule) are preferentially adsorbed in a monomolecular layer, whereas additional water molecules are bound to the water monomolecular layer by hydrogen bounds. Therefore, water sorption is a measure of swelling ability and the accessibility of fibers whereas iodine sorption is a measure of the accessibility of fibers and the crystallinity index (Fakin et al., 2006).

The obtained data showed that hemicelluloses removal increased the moisture and iodine sorption of hemp fibers compared to unmodified fibers, while lignin removal decreased both moisture and iodine sorption of chlorite modified hemp fibers. The increase of the moisture sorption values for hemp fibers with lower hemicelluloses content is the most likely consequence of removing the hemicelluloses from interfibrillar regions, followed by swelling and shrinkage of ultimate cells, which result in some disorientation of the fibrils and changed of amorphous and crystalline regions ratio, in favor of amorphous ones. Last one can be easily seen from the data for the iodine sorption value, which is inversely proportional to the fiber crystalline phase. Additionally, when hemicellulosic components have been progressively removed, interfibrillar regions become less dense and less rigid, which with the greater content of amorphous regions enable easier penetration of larger quantity of water molecules into hemp fiber structure.

The lowest value of moisture sorption (6.91%) and iodine sorption (44.9 mg/g) were obtained for sample L 60 (chlorite modified hemp fibers, 60 min). The decrease of the moisture and iodine sorption with gradual lignin removal can be explained by removing the less ordered and easily accessible noncellulosic moisture-absorbing materials in hemp fibers (i.e. lignin) during the chlorite treatment. Also, lignin removal occurred mostly in the middle lamella, making it more homogeneous (see Fig. 2c), which caused more difficult penetration of water molecules in these regions (Kostic et al., 2008).

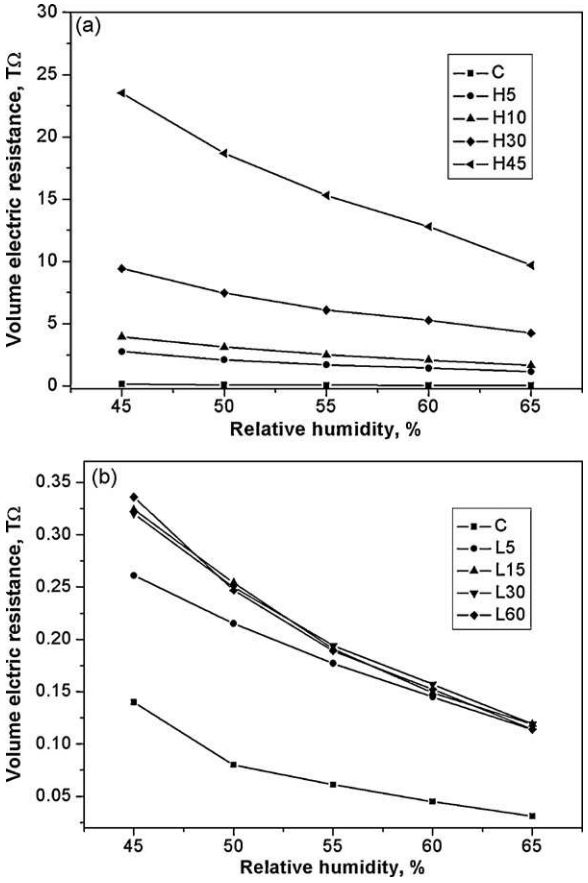

Fig. 4. Dependence of volume electric resistance on relative humidity of (a) hemp fibers modified with 17.5% NaOH and (b) hemp fibers modified with 0.7% $NaClO_2$.

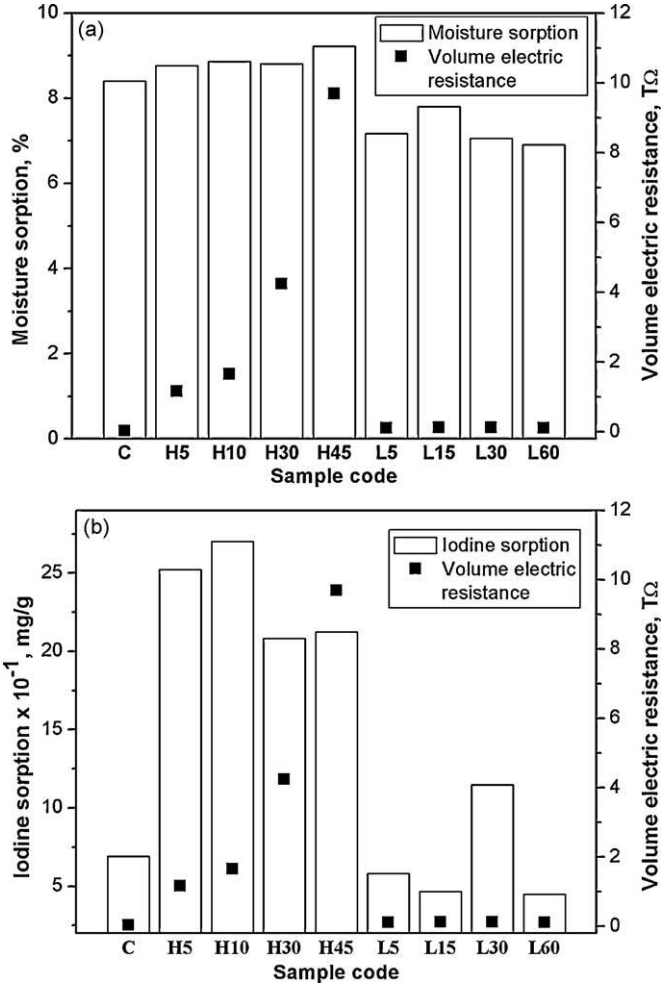

Fig. 5. (a) Moisture sorption and volume electric resistance and (b) iodine sorption and volume electric resistance of unmodified and modified hemp fibers.

3.3. *Influence of hemicelluloses and lignin removal on volume electric resistance of hemp fibers*

Unmodified hemp fibers have exceptionally low electric resistance in comparison with other natural and synthetic fibers (Asanovic et al., 2007). Data obtained during the experiments (Fig. 4) showed that with increase of modification times (i.e. degree of hemicelluloses and lignin removal), the volume electric resistance of all samples of modified fibers increased in comparison with the volume electric resistance of unmodified fibers. Increase of volume electric resistance of hemp fibers modified with sodium hydroxide (i.e. hemicelluloses removal) was higher than for fibers modified with sodium chlorite (i.e. lignin removal). Values of volume electric resistance, at standard relative humidity (65%), were 34–285 times and 3–3.5 times higher for hemp fibers modified with alkali and chlorite, respectively, than for unmodified fibers. In the case of lower relative humidity, increase of volume electric resistance for modified hemp fibers in comparison to unmodified fibers is more pronounce. Volume electric resistance of alkali modified fibers for all modification time, at 45% relative humidity, was about 57–58% higher in comparison with values obtained for the same samples at 65% relative humidity (Fig. 4a). Volume electric resistance for samples, from which lignin was gradually removed, was higher about 56–66% at 45% relative humidity than at 65% relative humidity (Fig. 4b). This can be explained by influence of relative humidity on partly ionization of water molecules, which were around the fibers, and neutralization of electric charges on fibers surface by these molecules. Furthermore, according to literature data (Dutta et al., 1980; Morton and Hearle, 1975) moisture and amorphous regions of the fibers are the most important factor in determining their resistance. Taking in consideration that, in Fig. 5a and b data of moisture sorption and volume electric resistance and iodine sorption values and volume electric resistance of unmodified and modified hemp fibers are presented, respectively. Because of the above mentioned fact that the fiber volume electric resistance is sensitive to the relative humidity, data determined at standard relative humidity (65%) were presented. From these data it is clear that hemp fibers with higher moisture content and greater amorphous fraction, i.e. hemp fibers from which hemicelluloses were removed, have higher volume electric resistance in comparison with unmodified or fibers from which lignin was removed gradually. This looks like unpredictable result just at the first moment, but from these data we can conclude that electric resistance of hemp fibers is mainly determined by their chemical composition, i.e. content of noncellulosic substances and at the first place hemicelluloses. Electric resistance of hemp fibers from which lignin was removed increased slightly mainly because of two reasons: lignin was removed only up to 50% and, as we mentioned earlier, lignin removal occurred mostly in the middle lamella, not in secondary wall. Furthermore, detailed comparison of the results from the literature (Asanović et al., 2004; Asanovic et al., 2007) for cotton, viscose, flax and hemp shows that they are in reasonable agreement with the assumption that the low electric resistance values of hemp fibers are consequence of deposited noncellulosic substances (i.e. hemicelluloses, lignin). Accepting of this assumption means that any refinement of hemp fibers, which includes removing of noncellulosic substances, should be very care-

fully done, with taking in consideration data presented in this paper.

4. Conclusion

This work presents an attempt to explain the individual influences of lignin and hemicelluloses on sorption and electric properties of hemp fibers. The progressive removal of hemicelluloses or lignin influenced the accessibility and sorption properties of hemp fibers differently, i.e. hemicelluloses removal increases while lignin removal decreases the moisture and iodine sorption of hemp fibers. The most interesting features of obtained results are a sharp increase of volume electric resistance with hemicelluloses removal and its smaller changes with lignin removal, which is certainly not due to changes in moisture sorption and amorphous fraction of modified fibers. From obtained data it is clear that electric resistance of hemp fibers is mainly determined by their chemical composition, i.e. content of noncellulosic substances and at the first place hemicelluloses. Furthermore, obtained results show that the modified hemp fibers acquired a high level of divisibility, with good levels of the comfort properties as a result of the removing noncellulosic substances (i.e. hemicelluloses and lignin).

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