The International Year of the Periodic Table 2019 IYPT

СХД 1897

УНИВЕРЗИТЕТ У НИШУ ПРИРОДНО-МАТЕМАТИЧКИ ФАКУЛТЕТ

56. savetovanje Srpskog hemijskog društva

KNJIGA RADOVA

56th Meeting of the Serbian Chemical Society

PROCEEDINGS

Niš 7. i 8. juni 2019.
Niš, Serbia, June 7-8, 2019

ISBN 978-86-7132-074-0

Srpsko hemijsko društvo

Serbian Chemical Society

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CIP- Каталогизација у публикацији
Народна библиотека Србије

54(082)(0.034.2)
577.1(082)(0.034.2)
66(082)(0.034.2)
66.017/.018(082)(0.034.2)
502/504(082)(0.034.2)

СРПСКО хемијско друштво. Саветовање (56 ; 2019 ; Ниш)
Knjiga radova [Elektronski izvor] = Proceedings / 56. savetovanje Srpskog hemijskog društva, Niš, 7. i 8. juni 2019. = 56th Meeting of the Serbian Chemical Society, Niš, Serbia, June 7-8, 2019 ; [urednici, editors Dušan Sladić, Niko Radulović, Aleksandar Dekanski]. - Beograd : Srpsko hemijsko društvo = Serbian Chemical Society, 2019 (Beograd : Razvojno-istraživački centar grafičkog inženjerstva TMF). - 1 elektronski optički disk (CD-ROM) ; 12 cm

Sistemski zahtevi: Nisu navedeni. - Dostupno i na: www.shd.org.rs/56shd.pdf. - Nasl. sa naslovne strane dokumenta. - Tekst ćir. i lat. - Tiraž 6. - Bibliografija uz svaki rad. - Abstracts ; Apstrakti. - Registar.

ISBN 978-86-7132-074-0

а) Хемија -- Зборници б) Биохемија -- Зборници в) Технологија -- Зборници г) Наука о материјалима -- Зборници д) Животна средина -- Зборници

COBISS.SR-ID 276611852

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Izdaje */ Published by*
Srpsko hemijsko društvo */ Serbian Chemical Society*
Karnegijeva 4/III, 11000 Beograd, Srbija
tel./fax: +381 11 3370 467; www.shd.org.rs, E-mail: Office@shd.org.rs

Za izdavača */ For Publisher*
Vesna Mišković STANKOVIĆ, *predsednik Društva*

Urednici */ Editors*
Dušan SLADIĆ
Niko RADULOVIĆ
Aleksandar DEKANSKI

Dizajn korica, slog i kompjuterska obrada teksta
Cover Design, Page Making and Computer Layout
Aleksandar DEKANSKI

OnLine publikacija */ OnLine publication*
www.shd.org.rs/56shd.pdf

ISBN ***978-86-7132-074-0***

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Savetovanje podržalo / ***Supported by***

Ministarstvo prosvete, nauke i tehnološkog razvoja Republike Srbije
Ministry of Education, Science and Technological Development of Republic of Serbia

Ova knjiga sadrži ***20 radova***
(obima od najmanje četiri stranice)
pojedinih saopštenja prezentovanih na
56. savetovanju Srpskog hemijskog društva.

This book contains ***20 Proceedings***
of some of the contributions presented at
the 56th Meeting of the Serbian Chemical Society.

Effect of the alkali treatment on the structure, moisture sorption and volume electrical resistivity of woven jute fabrics

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Woven jute fabric was treated with NaOH solution of different concentrations for 5 min to obtain jute fabrics with gradually decreased content of hemicelluloses and increased moisture sorption. With increasing the concentration of NaOH the crystallinity index decreased; in the case of NaOH concentration ≥ 10 % the conversion from cellulose $I_β$ to cellulose II occurred. The obtained decrease of the volume electrical resistivity after the alkali treatments can be attributed to the changes in hemicelluloses content, crystallinity index and moisture sorption. Increased conversion from cellulose $I_β$ to cellulose II leads to an increase in the volume electrical resistivity of the mercerized jute fabrics.

Introduction

Jute is the second most common natural fiber (next to cotton), cultivated in the world and extensively grown in Bangladesh[1]. This multi-cellular fiber is a major bast fiber and has many desirable properties such as high breaking and tear strength, high absorbency and hygroscopicity, good electrical properties, low carbon footprint, *etc.* In addition, the jute fibers are available in large quantities, they are inexpensive, biodegradable, recyclable and renewable[2,3]. Thanks to their good physico-mechanical properties, jute fibers have been employed for centuries in a production of packaging materials such as sacks and hessian cloth, as well as for carpet and twines, ropes, cords, *etc*[4].

As a lignocellulosic fiber, jute has a very heterogeneous chemical composition which includes α-cellulose, hemicelluloses and lignin, and some other minor components. The α-cellulose is rich in hydroxyl groups, which partially affects the moisture sorption of the jute fibers. The highly crystalline cellulose fibrils are embedded in a matrix composed of hemicelluloses and lignin[5]. The hemicelluloses consist of polysaccharides built up from hexoses, pentoses and uronic acid residues. The lignin is a long-chain amorphous cross-linked substance of high molecular weight[6]. A large part of the general understanding of the jute fiber properties is based on the ratio of their main components which varies depending on their growing conditions and geographical location for cultivation[7].

The ratio of the above mentioned jute components can be changed by different chemical treatments. Among them, the alkali treatments or mercerization have been widely used as the most direct and efficient treatments[8]. Namely, when the jute fibers are treated with sodium hydroxide profound changes, such as lateral swelling together with considerable shrinkage in length, occur in their structure[2]. At the same time, the hemicelluloses content decreases. In this paper, the effect of the alkali treatment severity on the hemicelluloses removal, moisture sorption, crystallinity index as well as dc volume electrical resistivity of the woven jute fabric was investigated. These properties are very important since they influence jute fabric processing and end-use. Widespread use of the textile materials in the field of electromagnetic protection, electronics as well as for production of protective clothes cause a need for examination of their volume electrical resistivity. It is well known that a decrease in volume electrical resistivity helps reduce the problem of static electricity during the processing of textile materials[9].

Experimental

Materials

Commercially produced raw woven jute fabric with the following chemical composition: 1.88 % water-soluble components, 1.92 % fats and waxes, 0.84 % pectin, 13.48 % lignin, 21.76 % hemicelluloses and 60.09 % α-cellulose, was used as experimental material. All used chemical agents are *p.a.* grade.

Methods

Alkali treatments of woven jute fabrics

Raw woven jute fabric was treated with 5, 10 and 17.5 % NaOH solution for 5 min. The alkali treatments were done in slack conditions at room temperature. They were followed by neutralization with 1 % acetic acid, rinsing with 0.5 % $NaHCO_3$, washing and drying at room temperature. Three alkali treated woven jute samples (A5, A10, A17.5) as well as a raw sample (R), were investigated, Table 1.

Determination of the chemical composition of woven jute fabrics

The chemical composition of woven jute fabrics was determined according to the procedure reported in the literature[10] by successive removal of the non-cellulosic components; after that, α-cellulose remains as a solid residue.

Determination of moisture sorption of woven jute fabrics

The moisture sorption (MS) was determined by the thermo-gravimetric method using an infrared moisture analyzer (Sartorius MA 35). Prior to moisture sorption measurement, woven jute fabrics were exposed to 65 % relative air humidity for 24 h. The average of three measurements for each sample was considered.

X-ray diffraction analysis

The X-ray measurements were performed on a Rigaku Ultima IV diffractometer in a Bragg-Brentano configuration using CuKα radiation. The diffraction data were acquired over the 2θ scattering angle (from 10° to 40°) with a step of 0.05° and an acquisition time of 2°/min. The obtained X-ray diffraction patterns were resolved into proportions of cellulose $I_β$, cellulose II lattice[11] and amorphous region using Gaussian and Lorentzian distribution function. Fitting of the X-ray diffraction patterns to estimate the integrated peak area was done using commercial software (Peakfit v4.12). The crystallinity index (CrI) is calculated from the ratio of the area of all crystalline peaks to the total area.

Determination of dc volume electrical resistivity of woven jute fabrics

The dc volume electrical resistance of woven jute fabrics was determined with the device developed at the Department of Textile Engineering of the Faculty of Technology and Metallurgy at the University of Belgrade. The measurement of dc volume electrical resistance was performed using the voltage method that has been presented in the paper[12]. The dc volume electrical resistance of the investigated fabrics was determined in the warp as well as in weft direction. For each sample, two measurements were conducted, whereby during each measurement two samples were connected to electrodes in order to increase the sensitivity of the method. The resistance of woven jute fabrics was measured under different relative air humidity (in further text humidity). The measurement was performed by increasing the humidity in the chamber (from 35 % to 55 %), *i.e.* during moisture sorption, at room temperature (23±2°C). On the basis of the determined dc volume electrical resistance of the woven fabrics (R_x), the dc volume electrical resistivity ρ (GΩ·cm) is calculated according to the Eq. 1[13]:

$$\rho = \frac{R_x S_F}{l} \qquad (1)$$

where: R_x is measured volume electrical resistance (GΩ), S_F is surface of the sample cross-section (cm^2) and l is sample length, *i.e.* length between electrodes (1 cm). Samples' cross-section S_F is calculated by multiplying the thickness of the sample and their width.

Results and discussion

The influence of the alkali treatment on the chemical composition of woven jute fabrics

The effect of the alkali treatment severity (*i.e.* treatments with NaOH solution of different concentrations) on the chemical composition of jute woven fabric was given in Table 1. From the obtained results it is evident that with increasing the alkali treatment severity, the hemicelluloses content significantly decreased. Namely, after the alkali treatment with 5 and 10 % NaOH, the hemicelluloses content decreased for 25.2 and 26.8 % respectively, compared to the raw jute fabric. The most severe alkali treatment results in a removal of only 36.6 % hemicelluloses which is attributed to the existence of strong hydrogen bonds between hemicelluloses and cellulose fibrils[7]. Moreover, the lignin content decreased for maximum of 7 % (sample A5) since it contains aromatic chemical groups and strong C-C bonds which are resistant to degradation or fragmentation[14]. It is good to mention that the alkali treated woven jute fabrics have higher α-cellulose content (70.44-73.31 %) compared to the raw fabric (60.09 %) due to the removal of hemicelluloses and other minor non-cellulosic components.

Table 1 Chemical composition of jute woven fabrics

Fabric codes	Treatment conditions		Content, %		
			Hemicelluloses	Lignin	α - cellulose
R	Raw – untreated		21.76	13.48	60.09
A5	5 min	5 % NaOH	16.28	12.54	71.19
A10		10 % NaOH	15.93	13.63	70.44
A17.5		17.5 % NaOH	13.79	12.91	73.31

X-ray diffraction analysis of the woven jute fabrics

After the alkali treatments, some rearrangement of the cellulosic chains occurs; which further contributed to the changes of the crystalline and amorphous regions ratio. Hemicelluloses and lignin are amorphous components, while the cellulosic chains are arranged in crystalline and amorphous regions[15]. According to that, the calculated crystallinity index of the raw woven jute fabric is 0.728, Fig. 1. After the alkali treatments as the result of hemicelluloses removal, the crystallinity index decreased. For example, in the case of the mild alkali treatment (sample A5), in parallel with the hemicelluloses removal (about 25.2 % of the hemicelluloses were removed), the crystallinity index decreased for about 5.6 % (CrI = 0.687). Compared to the sample A5, sample A10 has very similar hemicelluloses content (Table 1), while small conversion from cellulose I_β to cellulose II (about 8.9 %) occurred as the consequence of the mercerization (alkali treatment with 10 % NaOH). With further increase in the alkali treatment severity (17.5 % NaOH, sample A17.5), the mercerization is more pronounced and was manifested through decreasing of the crystallinity index (CrI = 0.591) and increasing conversion from cellulose I_β to cellulose II (about 37.1 %), Fig. 1.

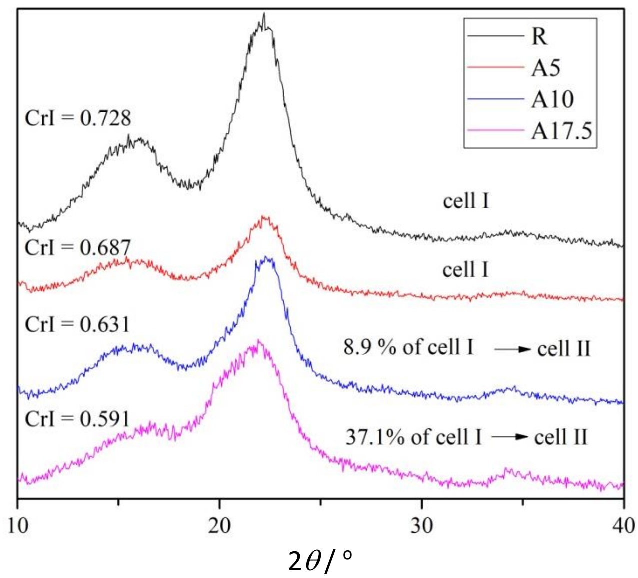

Fig. 1. X-ray diffraction patterns of woven jute fabrics

Moisture sorption of the woven jute fabrics

As it was expected, the hemicelluloses removal, as well as the rearrangement of the cellulosic chains, contributed to the increased accessibility of the cell wall components to water vapor. Fig. 2 presents the relation between the hemicelluloses content, moisture sorption and crystallinity index of woven jute fabrics. According to the results, the raw jute fabric which has the highest hemicelluloses content and crystallinity index has the lowest moisture sorption value (7.41 %). In addition, the jute fabric with the lowest hemicelluloses content and crystallinity index can absorb the highest amount of moisture (9.01 %). This can be explained by the higher exposer of the cellulose and its hydroxyl groups at the jute amorphous regions and crystallite's surface as well as increased effective surface area after the alkali treatment[16]. Furthermore, the changes in the moisture sorption are also the consequence of the rearrangement of the cellulose chains and conversion from cellulose I_β (parallel arrangement) to cellulose II (anti-parallel arrangement), which should enable better moisture sorption.

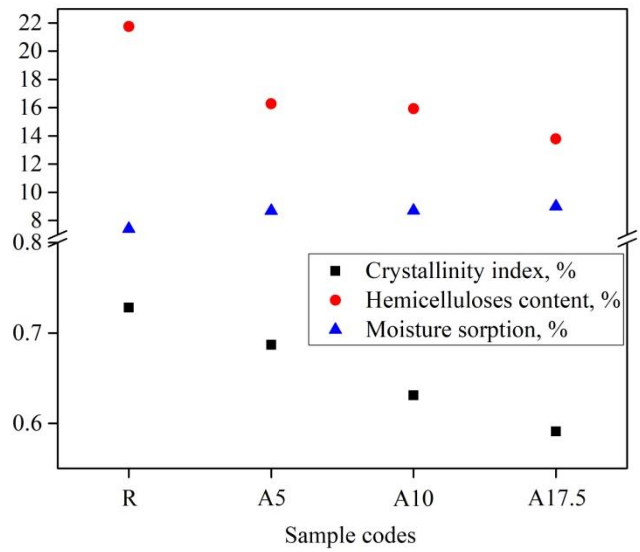

Fig. 2. Relation between the content of hemicelluloses, moisture sorption and crystallinity index of woven jute fabrics

The dc volume electrical resistivity of woven jute fabrics

Fig. 3 shows the values of dc volume electrical resistivity (in further text resistivity) at different humidities (35, 40, 45, 50 and 55 %) for raw and alkali treated woven jute fabrics, determined at the same sorption dynamics of moisture. The humidity can influence partly ionization of water molecules, which were around the fabrics, and neutralization of electric charges on fibers surface by these molecules[12]. A trend of decreasing values of the dc volume resistivity along the warp and weft direction with increasing the humidity in the chamber was noted. The highest difference between the values of the volume electrical resistivity measured at 35 and 55 % humidity was noticed for raw jute fabric, even 9.5 and 23.4 times (warp and weft direction, respectively). The mentioned differences are lower for the alkali treated jute fabrics; they have about 2.5-10.6 times lower volume electrical resistivity at 55 % than at 35 % humidity. With increasing the humidity in the chamber, these differences significantly decreased, which can be clearly seen in Fig. 3b.

As it can be seen, the resistivity is very sensitive to humidity, but the influence of the chemical composition, moisture sorption and crystallinity index as well as conversion from cellulose I_β to cellulose II on the resistivity should not be neglected. At milder alkali treatment conditions (sample A5), when the hemicelluloses content decreased for 25.2 % (Table 1) compared to raw fabric, the resistivity (at 35 % humidity) decreased for 8.4 and 15.9 times (warp and weft direction, respectively), Fig 3. In addition, according to Morton and Hearle[17] the most important factors that influence the resistivity are moisture sorption and fiber amorphous regions. The presence of moisture facilitates the flow of current through the jute fiber amorphous regions resulting in decreased resistivity. From our results, it is obvious that jute fabric with lower crystallinity index (greater amorphous fraction), *i.e.* samples after alkali treatments, have lower resistivity. After the mercerization (samples A10 and A17.5), the resistivity increased, compared to the sample A5. The reason for such behavior can be related to the existence of antiparallel cellulose II in the jute crystalline regions. Namely, about 4.2 times higher conversion from cellulose I_{β} to cellulose II (in sample A17.5 compared to the sample A10) contributed to increased resistivity (at 35 % humidity) for 30.2 and 24.1 % (warp and weft direction, respectively).

a

b

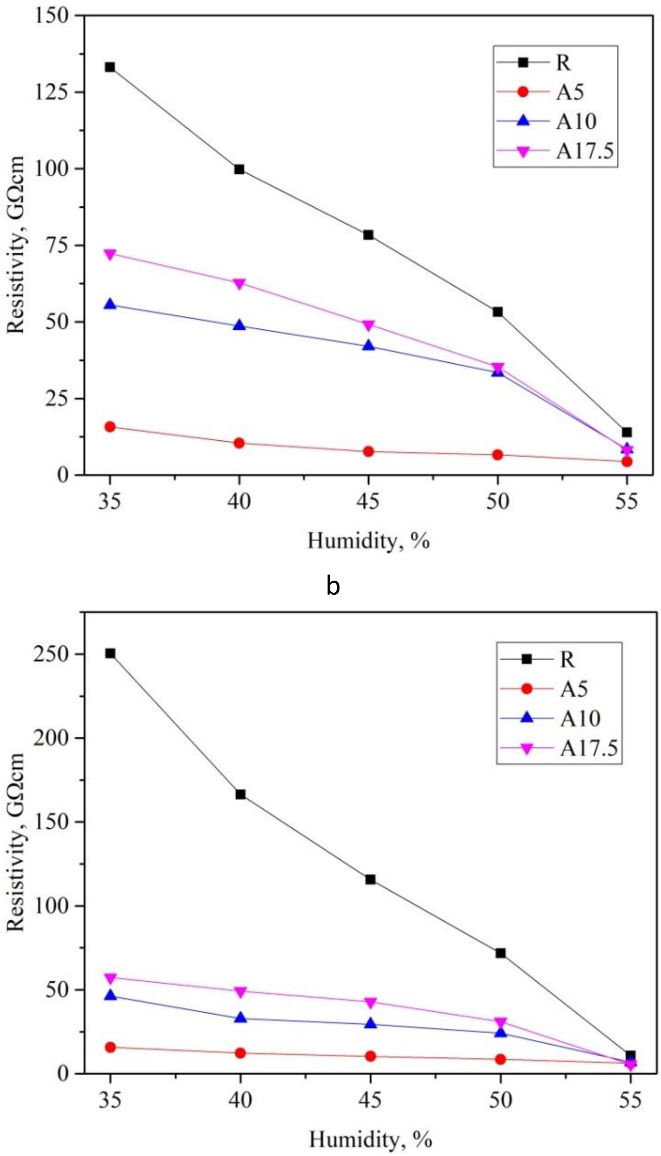

Fig. 3. The resistivity of woven jute fabrics: a) warp direction and b) weft direction

Conclusion

The alkali treatment of woven jute fabric for 5 min with NaOH solution of different concentrations resulted in jute fabrics with different hemicelluloses content as well as changed some properties such as moisture sorption, crystallinity index and volume electrical resistivity. The jute fabrics with lower hemicelluloses content were characterized by higher moisture sorption. The X-ray diffraction analysis showed that there was a gradual decreased in the crystallinity index when the hemicelluloses content decreased. In the case of NaOH concentration ≥ 10 %, the conversion from cellulose $I_β$ to cellulose II occurred. The values of volume electrical resistivity are very sensitive to above mentioned jute fabric properties. In general, when the relative air humidity was increased from 35 % to 55 %, the volume electrical resistivity of raw as well as alkali treated jute fabrics decreased since the humidity can influence partly ionization of water molecules, which were around the fabrics, and neutralization of electric charges on fibers surface by these molecules. All alkali treated jute fabrics have reduced volume electrical resistivity compared to the raw jute fabric. The obtained jute fabrics with lower volume electrical resistivity can be successfully used for protective clothes or textile of a specific behavior in environments sensitive to electrical discharges as well as for some ordinary product such as home textiles (carpet), filters, *etc.*

Funding: *This work was supported by the Ministry of Education, Science and Technological Development of the Government of the Republic of Serbia (Projects OI 172029 and 171029).*

Uticaj alkalnog tretmana na strukturu, sorpciju vlage i zapreminsku električnu otpornost tkanina od jute

Tkanina od jute je tretirana rastvorom NaOH različitih koncentracija u toku 5 min, što je dovelo do smanjenja sadržaja hemiceluloza i povećanje sorpcije vlage. Analiza rezultata dobijenih difrakcijom rendgenskih zraka pokazala je da tkanine sa manjim sadržajem hemiceluloza imaju manji indeks kristaliničnosti i da nakon mercerizovanja, dolazi do konverzije celuloze $I_β$ u cellulozu II. Manje vrednosti zapreminske električne otpornosti nakon alkalnih tretmana mogu se povezati sa smanjenjem sadžaja hemiceluloza i indeksa kristaliničnosti i povećanjem sorpcije vlage. Povećan stepen konverzije celuloze $I_β$ u celulozu II dovodi do povećanja zapreminske električne otpornosti mercerizovanih tkanina.

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