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PREFACE

The 2nd International conference "Contemporary Trends and Innovations in the Textile Industry" CT&ITI 2019, is co-organized by the Union of Engineers and Textile Technicians of Serbia, the Union of Engineers and Technicians of Serbia, the Faculty of Technology and Metallurgy in Belgrade, the University of Belgrade and the Faculty of Technology in Leskovac, University of Niš, Faculty of Technology, Shtip, Nort of Macedonia and Society for Robotics of Bosnia i Herzegovina.

The Ministry of Education, Science and Technological Development of the Republic of Serbia recognized the importance of this Conference, and thus, supported it. The aim of this Conference is to consider current technical, technological, economic, ecological, R&D, legal and other issues related to the textile industry, then the application of contemporary achievements and the introduction of technical and technological innovations in the production process of fiber, textile, clothing and technical textile by applying scientific solutions in order to improve the business and increase the competitive advantages of the textile industry on the domestic and global market.

Leading scientists and experts from the Balkans and other countries, working at faculties, textile colleges and institutes, but also individuals who professionally deal with the issues at hand are taking part in this Conference.

The Conference program involves papers dedicated to the scientific and practical aspects of the following topics: Textile and Textile Technology, Textile Design, Management and Marketing in the Textile Industry and Ecology and Sustainable Development in the Textile Industry. The Conference program includes 54 papers, and a total of 112 participants from 12 countries: Bosnia and Herzegovina, Bulgaria, India, Moldova, North of Macedonia, Portugal, Romania, Russia, Serbia, Slovenia, Turkey and Ukraine.

Therefore, this Conference is an opportunity for establishing scientific, educational and economic cooperation of our country with other countries. Certain number of papers by domestic authors present the project results dealing with fundamental research and technological development, financed by the Ministry of Education, Science and Technological Development of the Republic of Serbia.

I would like to thank all those who have made it possible to organize the conference Contemporary Trends and Innovations in the Textile Industry and make it a success. First, I would like to thank the Scientific and Organizing Committee for working hard, spending countless hours and finding the best solutions for numerous organizational aspects of our Conference. Also, I would like to express my gratitude to all sponsors who believed in the importance of this Conference and co-financed it. I also thank all the other institutions that supported the Conference in various ways, because without their support, the Conference could not have been organized. Last but not least, I would like to thank plenary lecturers, all authors and co-authors and guests for their participation in the Conference.

On behalf of the Organizing Committee *Prof. dr Snežana Urošević, president*



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DIELECTRIC LOSS TANGENT OF ALKALI TREATED JUTE WOVEN FABRICS: EFFECT OF HEMICELLULOSES CONTENT

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ABSTRACT: The jute woven fabric was treated with sodium hydroxide at room temperature for 5, 30 and 45 min in order to obtain jute fabric with different hemicelluloses content and to study their influence on the dielectric loss tangent. After the alkali treatments, as the result of the hemicelluloses removal, the moisture sorption of the jute fabrics increased. The X-ray diffraction analysis shows that the jute fabrics with lower hemicelluloses content have a lower crystallinity index, which slightly increased after the intensive hemicelluloses removal. These results are in a good agreement with the results obtained for dielectric loss tangent measured at 30% and 80% relative air humidity.

Keywords: jute, alkali, hemicelluloses, crystallinity index, dielectric loss tangent

TANGENS DIELEKTRIČNIH GUBITAKA ALKALNO TRETIRANIH TKANINA OD JUTE: EFEKAT SADRŽAJA HEMICELULOZA

ABSTRACT: Tkanina od jute je tretirana sa natrijum-hidroksidom na sobnoj temperaturi u toku 5, 30 i 45 min u cilju dobijanja tkanina od jute sa različitim sadržajem hemiceluloza i proučavanja njihovog uticaja na tangens dielektričnih gubitaka. Nakon alkalnih tretmana, kao rezultat uklanjanja hemiceluloza, sorpcija vlage se povećava. Analiza rezultata dobijena rendgenskom difrakcijom pokazuje da tkanine sa manjim sadržajem hemiceluloza imaju manji indeks kristalnosti, koji se malo povećava nakon intenzivnog uklanjanja hemiceluloza. Dobijeni rezultati su u saglasnosti sa rezultatima dobijenim za tangens dielektričnih gubitaka merenim pri 30% i 80% relativne vlažnosti.

Ključne reći: juta, alkalija, hemiceluloze, indeks kristalnosti, tangens dielektričnih gubitaka

1. INTRODUCTION

Commercially grown in India, Bangladesh, China and Nepal, jute is the most important bast fiber. Thanks to their excellent properties (high tensile strength and tenacity, high absorbency, good thermal and electrical properties, biodegradability, recyclability, etc.),



jute has been traditionally used as packaging material and raw material for the production of different composites, etc. [1, 2]. Jute has a heterogeneous chemical composition, which includes α -cellulose, hemicelluloses and lignin, as well as minor components. The high content of non-cellulosic components in the jute fibers has a negative influence on their properties and processing and therefore, their further treatments may be necessary. The alkali treatments are the most economical and efficient treatments to selective hemicelluloses removal from the bast fibers. In this study, the jute woven fabrics with different hemicelluloses content were used to investigate the effect of the hemicelluloses content on the dielectric loss tangent. This parameter, as an indirect indicator of the tendency of fibrous materials to create a static charge [3], makes it possible to estimate the impact of the alkali treatment of jute fabrics on their properties.

2. EXPERIMENTAL

2.1. Materials

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

2.2. Methods

2.2.1. Preparation of the jute woven fabric with different hemicelluloses content

The raw jute fabric was alkali treated with 17.5% NaOH solution for 5, 30 and 45 min at room temperature. The alkali treatments were followed by neutralization with 1% acetic acid, rinsing with 0.5% NaHCO₃, washing and drying at room temperature, Table 1.

2.2.2. Determination of weight loss and chemical composition of the jute fabric

The weight loss (*WL*) which occurs as a result of hemicelluloses removal after the alkali treatments of the jute woven fabric was determined according to equation (1):

$$WL(\%) = \frac{m - m_c}{m} \cdot 100 \tag{1}$$

where: m (g) is mass of absolutely dry untreated jute fabric and m_c (g) is mass of absolutely dry chemically treated jute fabric.

The chemical composition of the jute woven fabrics was determined according to the procedure reported by Soutar and Bryden showed in the literature [4].

2.2.3. X-ray diffraction analysis

The X-ray measurements were performed on a Rigaku Ultima IV diffractometer in a Bragg-Brentano configuration using CuK α radiation. The diffraction data were acquired over the 2 θ 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 and an amorphous region. 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.

2.2.4. Detrmination of moisture sorption

The moisture sorption was determined according to a thermo-gravimetric method using *Infrared Moisture Analyzer* Sartorius MA35. Prior to moisture sorption measurements, the samples were exposed to 30% and 80% relative air humidities (RH) for 24h.

2.2.5. Determination of the dielectric loss tangent of the jute woven fabric

The Precise LCR Hameg 8118 instrument connected to the LD-3 solid dielectric cell was used to determine the dielectric loss tangent.[3]. Twelve measurements were performed over a range of frequencies from 30 Hz to 140 kHz, at 22 °C. Two sets of measurements were done, for samples exposed 24h to 30% and 80% relative air humidity. Acquisition of the conductance, G (S), and susceptance, B (S), as well as the conductance, G_0 (S) and susceptance, B_0 (S) of an empty cell, is carried out with specially programmed software. Based on the experimental data, the dielectric loss tangent ($tan\delta$) was determined using the equation (2):

$$\tan \delta = \frac{G - G_0}{B - B_0 + 2 \cdot \pi \cdot f \cdot \varepsilon_0 \cdot A \cdot l^{-1}}$$
(2)

where: f (Hz) is frequency, ε_0 is permittivity of a vacuum (8.854·10⁻¹² F/m), A (m²) is an area of the sample under the electrode, l (m) is the space of the electrodes which is equal to the thickness of the sample.

3. RESULTS AND DISCUSSION 3.1. Chemical composition of the jute woven fabrics

In order to study the influence of the hemicelluloses content on the dielectric loss tangent, hemicelluloses were selectively removed from the jute woven fabric by alkali treatment with 17.5% NaOH for 5, 30 and 45 min, Table 1. From the results obtained for the weight loss and hemicelluloses content, it is clear that the weight loss after the alkali treatments occurs as a result of removal of the hemicelluloses and other non-cellulosic components. The weight loss increases, while the hemicelluloses content decreases with increasing the treatment duration. After the alkali treatment for 5 min (sample A5) and 30 min (sample A30), the hemicelluloses content decreased for 36.6% and 43.3%, respectively, compared to the untreated fabric (sample C). The highest weight loss (13.92%) and hemicelluloses removal (about 46.7%) was noticed after the alkali treatment for 45 min (sample A45). The lignin content after the alkali treatments slightly decreased, pointing out that after the alkali treatments it remains almost unchanged, due to the presence of aromatic groups, as well as a strong chain of carbon-carbon bonds [5].

Fabric codes	Treatment conditions		Weight loss, %	Hemicelluloses, %	Lignin, %
С	Control – untreated		/	21.76	13.48
A5	17.5% NaOH	5 min	10.35	13.79	12.91
A30	at room	30 min	12.07	12.34	13.27
A45	teperature	45 min	13.92	11.60	12.52

 Table 1: Jute woven fabric codes, weight loss, hemicelluloses and lignin content

3.2. X-ray diffraction analysis

The hemicelluloses removal was followed by some rearrangement of the cellulosic chains, which further affects the changes in the ratio of the crystalline and amorphous regions. The X-ray diffraction patterns are the combination of the diffraction from an amorphous region (a broad featureless halo) and sharp peaks from the crystalline region. The peaks which are characteristic for cellulose I_{β} are located at around $2\Theta = 14.7^{\circ}$, 16.8° and 22.7° , while the peak at around $2\Theta = 18^{\circ}$ corresponds to the amorphous region [6], Figure 1. 0.728 is the crystallinity index of the untreated fabric.



Figure 1: X-ray diffraction patterns of the jute fabrics: a) C, b) A5, c) A30 and d) A45

The X-ray diffraction patterns of the alkali treated jute fabrics contain peaks characteristic for cellulose I_{β} and cellulose II, which confirmed the incomplete conversion from cellulose I_{β} (chains aligned in parallel) to cellulose II (anti-parallel). The peaks of cellulose II polymorph are shifted at $2\Theta = 12.1^{\circ}$, 20.1° and 21.9° , while the peak of the amorphous region is shifted to $2\Theta = 16^{\circ}$ [6]. In the case of short alkali treatment duration (sample A5), the hemicelluloses removal was followed by the decrease in the crystallinity index for 18.8%, as well as polymorphic conversion (about 46%) of cellulose I_β to cellulose II. Since the alkali penetration induced fibers swelling and disrupting of the crystalline regions [7], after 30 min alkali treatment (sample A30) the cellulosic chains rearrangement is accompanied by a higher decrease (about 28.2%)



in the crystallinity index, Figure 1. With higher hemicelluloses removal (sample A45), a slight increase of the crystallinity index (CrI = 0.673) was observed due to the higher conversion from cellulose I_β (about 69%) to cellulose II and sufficient time for rearrangement of cellulose chains in an ordered structure.

3.3. Moisture sorption of the jute woven fabrics

The raw jute fabric can absorb a high amount of moisture from the atmosphere (6.5% at 30% RH and 14.8% at 80% RH), (Figure 2), which comes from the presence of hydroxyl and other polar groups in the jute amorphous regions and at crystallite's surface. The hemicelluloses removal from the interfibrillar regions, as well as higher availability of the hydroxyl groups [8] lead to higher moisture sorption values (independent of the relative air humidity) compared to the control jute woven fabric.



Figure 2: Moisture sorption of the jute fabrics measured at a) 30% and b) 80% RH

The highest moisture sorption value has sample A30 (7.87% at 30% RH, 16.92% at 80% RH). With prolonged alkali treatment and a higher decrease of the hemicelluloses content (sample A45), the moisture sorption of the jute fabric decreased, pointing out that not only hemicelluloses content affect moisture sorption. From the previously mentioned results, it is evident that the crystallinity index also influenced moisture sorption: as the crystallinity index increased (Figure 1), the moisture sorption of the sample A45 decreased, Figure 2.

3.4. Dielectric loss tangent of the jute woven fabrics

The results obtained for the dielectric loss tangent $(tan\delta)$ of the investigated jute woven fabrics measured at 30% and 80% RH in the frequency region between 30 Hz and 140 kHz were present in the Figures 3 and 4. The lowest value of $tan\delta$ at 30% and 80% RH



for the untreated jute woven fabric (Figures 3 and 4) can be explained by its lowest moisture sorption ability and highest crystallinity index.



Figure 3: Dielectric loss tangent $(tan\delta)$ of the jute woven fabrics measured at 30% RH

A significant increase in the *tand* values after the hemicelluloses removal was observed. Namely, with decreasing the content of hemicelluloses from 21.76% (sample C) to 13.79% and 12.34% for samples A5 and A30, the tan δ (at 30 Hz) increased from 0.174 (sample C) to 0.545 and 0.637, respectively, Table 1 and Figure 3. Such behavior occurs due to the increase in the number of polar groups, i.e. hydroxyl groups of cellulose became more accessible as the result of the hemicelluloses and other non-celliulosic components removal. This further increase the orientation polarization at lower frequencies leading to higher $tan\delta$ values. In addition, with decreasing the hemicelluloses content, the crystallinity index also decreased, while the moisture sorption increased, that additionally contributed to the higher $tan\delta$ values. According to the literature [10], the hemicelluloses not only change the structure in such a way that the mobility of the ions in the electric field is restricted, but they also restrict the freedom of the water molecules to take part in the polarization process. An exception is the sample A45 which has lower hemicelluloses content compared to A5, but still has about 13.8% lower tan δ value (at 30 Hz), Figure 3, which can be associated with its higher crystallinity index and lower moisture sorption value. With increasing the frequency above 100 Hz, tan δ decreased because the orientation polarization of the polar groups does not take place completely.





Figure 4: Dielectric loss tangent $(tan\delta)$ of the jute woven fabrics measured at 80% RH

The relative air humidity affects significantly on the $tan\delta$ values of the jute woven fabrics. The results obtained for the $tan\delta$ at 80% RH followed the same trend as the results obtained for the $tan\delta$ at 30% RH, Figure 4. With increasing the relative humidity from 30% to 80%, the $tan\delta$ increased for about 2-4.7 times (at 30 Hz). The presence of water in the jute woven fabrics increases the number of polar groups resulting in an increase of dielectric properties [3], in this case in an increase of tan\delta. As in the case of the 30% RH, also in the case of 80% RH, the $tan\delta$ is in a good agreement with the moisture sorption and crystallinity index of the jute fabrics.

4. CONCLUSION

Jute woven fabric was treated with sodium hydroxide for different periods of time in order to obtain jute fabrics with different hemicelluloses content, which further influences their dielectric loss tangent. 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 and conversion from cellulose I_{β} to cellulose II when the hemicelluloses content decreased down to 43.3%. But, with a further decrease, the crystallinity index increased due to the higher conversion from cellulose I_{β} to cellulose II and sufficient time for rearrangement of cellulose chains in an ordered structure. Jute fabrics with lower hemicelluloses content have increased dielectric loss tangent compared to the untreated fabric, which depends on different factors. At both relative humidities, 30% and 80%, the moisture sorption and crystallinity index dominantly influenced the dielectric loss tangent, while the hemicelluloses content slightly affect the dielectric loss tangent. In the condition of increased relative air humidity, the dielectric loss tangent increased, since the presence of water in the jute fabrics increases the number of polar groups. The measurements of dielectric loss tangent enable to predict the behavior of the jute woven fabrics in real application conditions. The obtained dielectric loss tangent values suggest that the jute fabrics with lower hemicelluloses content and crystallinity index, as well as higher moisture sorption can be used as a substrate for various applications.

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