UNION OF ENGINEERS AND TEXTILE TECHNICIANS OF SERBIA

IV INTERNATIONAL SCIENTIFIC CONFERENCE

CONTEMPORARY TRENDS AND INNOVATIONS IN THE TEXTILE INDUSTRY

IV MEĐUNARODNA NAUČNA KONFERENCIJA

SAVREMENI TRENDOVI I INOVACIJE U TEKSTILNOJ INDUSTRIJI

PROCEEDINGS

EDITOR:
Prof. dr SNEŽANA UROŠEVIĆ

Belgrade, 16-17th September, 2021.
Union of Engineers and Technicians of Serbia
Dom inženjera „Nikola Tesla"

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PREFACE

The 4 th International conference "Contemporary Trends and Innovations in the Textile Industry" CT&ITI 2021, 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 Faculty of Technology, Shtip, North of Macedonia, Society for Robotics of Bosnia i Herzegovina and Balkan Society Of Textile Engineering-BASTE of Greece.

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 56 papers, and a total of 129 participants from 16 countries: Albania, Bosnia and Herzegovina, Bulgaria, Croatia, Greece, India, Latvia, North of Macedonia, Montenegro, 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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SIMPLE PROTOCOLS FOR OBTAINING MULTIFUNCTIONAL JUTE FABRICS

Aleksandra Ivanovska[1], Koviljka Asanović[2], Dragana Cerović[3], Leposava Pavun[4], Biljana Dojčinović[5], Katarina Mihajlovski[2], Mirjana Kostić[2]

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ABSTRACT: *This study aims to obtain multifunctional jute fabrics by using simple alkali and oxidative protocols. Performed chemical treatments contributed to obtaining jute fabrics with enhanced electro-physical and sorption properties. Moreover, the jute fabrics were functionalized by incorporation of* Ag^{+} *(resulted in decreased volume electrical resistivity and excellent antibacterial activity) and in-situ synthesis of Cu-based nanoparticles (resulted in 38-88 times higher energy storage from an external electric field). Following the increased focus on the concept of circular economy, the possibility of jute fabric waste revalorization for wastewater purification (from* Cu^{2+}, Zn^{2+}, Ni^{2+}*) was examined.*

Keywords: *jute, multifunctional fabrics, electro-physical properties, sorption properties, antibacterial activity, wastewater purification*

JEDNOSTAVNI POSTUPCI ZA DOBIJANJE MULTIFUNKCIONALNIH TKANINA OD JUTE

APSTRAKT: *Cilj ovog istraživanja je dobijanje multifunkcionalnih tkanina od jute upotrebom jednostavnih alkalnih i oksidativnih postupaka. Ovi hemijski tretmani omogućili su dobijanje tkanina od jute sa poboljšanim elektro-fizičkim i sorpcionim svojstvima. Tkanine od jute su, takođe, bile funkcionalizovane jonima srebra (što je rezultovalo smanjenom specifičnom zapreminskom električnom otpornošću i odličnom antibakterijskom aktivnošću) i in-situ sintezom nanočestica na bazi bakra (što je rezultovalo 38-88 puta većim skladištenjem energije iz spoljašnjeg električnog polja). U skladu sa načelima cirkularne ekonomije, ispitana je mogućnost revalorizacije otpada tkanina od jute za prečišćavanje otpadnih voda (od* Cu^{2+}, Zn^{2+}, Ni^{2+}*- jona).*

Ključne reči: *juta, multifunkcionalne tkanine, elektro-fizička svojstva, sorpciona svojstva, antimikrobna aktivnost, prečišćavanje otpadnih voda*

1. INTRODUCTION

Jute fiber (also recognized as „golden fiber“) represents one of the most important lignocellulosic fibers, and the second most important natural fiber (after cotton) from the point of its global production, consumption, and availability. Namely, due to their excellent mechanical properties, jute fibers have been mostly used for the manufacturing of packaging materials (*i.e.*, sacking cloth, hessian, *etc*.), household textiles, carpet backings, and cords. Although their main component is cellulose, the non-cellulosic components such as hemicelluloses (polysaccharides of comparatively low molecular weight that occupy spaces between the fibrils in both primary and secondary wall) and lignin (high molecular weight, three-dimensional phenyl propane-based polymer located in the middle lamella and the secondary wall) play a vital role in the jute fiber properties.

In order to meet the worldwide demand for jute fibers, their production, processing, and quality should be improved. Therefore, many researches all over the world are focused on jute fiber functionalization. Among many different methods, the alkali modifications using sodium hydroxide and oxidative modifications sodium chlorite are the simplest, the most direct, economical, and efficient methods for jute fibers' functionalization. Such chemical modifications reduced the content of non-cellulosic components and therefore contributed to obtain jute fibers with unique properties.

In this study, an attempt has been made to obtain multifunctional jute fabrics and to explain the individual roles of the alkali and oxidative modification on jute fabric chemical composition, fabric structural characteristics, mechanical properties, sorption, and electro-physical properties as well as antimicrobial activity. Moreover, the last part of this investigation is focused on resolving the issue for lignocellulosic textile waste (*i.e.*, jute fabric) by its utilization as a biosorbent for heavy metal ions (such as Ni^{2+}, Cu^{2+}, and Zn^{2+}). In such a way, the creation of a closed technological loop focused on the reduction of lignocellulosic textile waste and strengthening of recycling processes has been proposed.

2. EXPERIMENTAL

2.1. Material

A commercially produced raw jute fabric (sample C, Table 1) was used as experimental material.

2.2. Chemical modifications of raw jute fabric

Alkali and oxidative modifications under different conditions (Table 1) were performed to obtain jute fabrics with lower hemicellulose and lignin content and to study their effect on fabric properties.

Table 1: Sample codes, chemical modification conditions, and chemical composition of investigated jute fabrics

Sample code	Experimental conditions	Temperature	Hemicelluloses, %	Lignin, %	Cellulose, %
C	raw	/	21.76	13.48	60.09
A30/1	1% NaOH for 30 min	Room temperature	18.62	13.45	67.93
A5/5	5% NaOH for 5 min		16.28	12.54	71.19
A5/10	10% NaOH for 5 min		15.93	13.63	70.44
A5/17.5	17.5% NaOH for 5 min		13.79	12.91	73.31
A30/17.5	17.5% NaOH for 30 min		12.34	13.27	74.35
A45/17.5	17.5% NaOH for 45 min		11.60	12.52	75.88
O15	0.7% $NaClO_2$ for 15 min	Boiling temperature, pH (4-4.50)	21.79	8.39	69.81
O30	0.7% $NaClO_2$ for 30 min		21.29	8.25	70.45
O60	0.7% $NaClO_2$ for 60 min		24.93	4.96	70.11
O90	0.7% $NaClO_2$ for 90 min		25.31	2.83	71.87

2.3. Jute fabrics' characterization

The jute fabric chemical composition was determined according to the procedure described in the literature [1]. FE-SEM and X-ray diffraction analysis were used to characterize the fiber surface morphology, and crystallinity index, respectively. The jute fabric structural characteristics (fabric thickness, fabric weight, fabric density, and crimp) and their mechanical properties (fabric maximum force and elongation at maximum force) were also determined according to the appropriate standards. Fiber moisture sorption, water retention power, and degree of swelling were determined according to the procedure described by Ivanovska et al. [1]. Measurement of the dielectric properties (AC specific electrical conductivity, dielectric loss tangent, and effective relative dielectric permeability) was performed on Precise LCR Hameg 8118 instrument, (details given in the paper [1]), while the volume electrical resistivity was measured based on the procedure described in the literature [2]. The *in* situ synthesis of Cu-based NPs on selected jute fabrics was performed according to the method given by Marković et al [3]. The jute fabric antibacterial activity was tested against *E. coli* and *S. Aureus* using a standard test method (ASTM E 2149-01 (2001)). The jute fabric biosorption potential for heavy metal ions (Ni^{2+}, Cu^{2+}, and Zn^{2+}) was investigated, whereby the metal ion concentration in an aqueous solution was determined by ICP-OES [4]. The content of carboxyl groups was determined by using the Ca-acetate method [5].

3. RESULTS AND DISCUSSION

A simple alkali and oxidative protocols (Table 1) were used for obtaining multifunctional jute fabrics having improved sorption, and electro-physical properties as well as antibacterial activity. Namely, with increasing the NaOH concentration and/or modification time, hemicellulose removal from interfibrillar regions becomes more intensive and is followed by a decreased crystallinity index and pronounced elementary fibers' liberalization (Figure 1), allowing higher availability of cellulose hydroxyl groups and storing of water molecules inside fibers' free volume. Altogether contributed to improved sorption properties, *i.e.*, higher moisture sorption, water retention power, and degree of fiber swelling. During the most severe alkali modification (sample A45/17.5), the highest conversion (57.7%) of cellulose I to cellulose II was observed; the entire fibers are converted into a swollen state and newly exposed cellulose hydroxyl groups can interact with each other to form a different type of hydrogen bonds [6] contributing to cellulose chains' rearrangement in an ordered structure, *i.e.*, increased crystallinity index and decreased sorption properties' values, Figure 1.

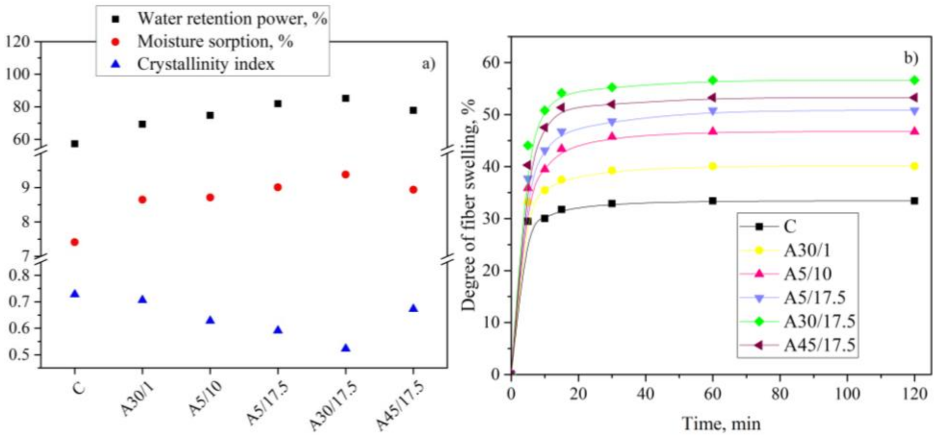

Figure 1: Sorption properties of alkali modified jute fabrics (Part of the results published in [1])

The oxidative modifications for different times enable to obtain jute fabric with gradually decreased lignin content (Table 1) and improved moisture sorption, water retention power, and degree of fiber swelling, Figure 2. When lignin was almost removed (sample O90), the homogenization of middle lamellae occurred resulting in difficult penetration of water molecules within fibers, and thus lower moisture sorption and swelling ability compared to other oxidized fibers (samples O30 and O60). It has to be emphasized that due to the lignin removal, the cellulose content increased (Table 1), its hydroxyl groups become more exposed and ready to form new hydrogen bonds within surrounding amorphous regions. According to Agarwal et al. [7], these hydrogen bonds caused a higher number of cellulosic molecules in the amorphous regions to move closer to the crystalline regions and become aligned causing increased crystallinity index.

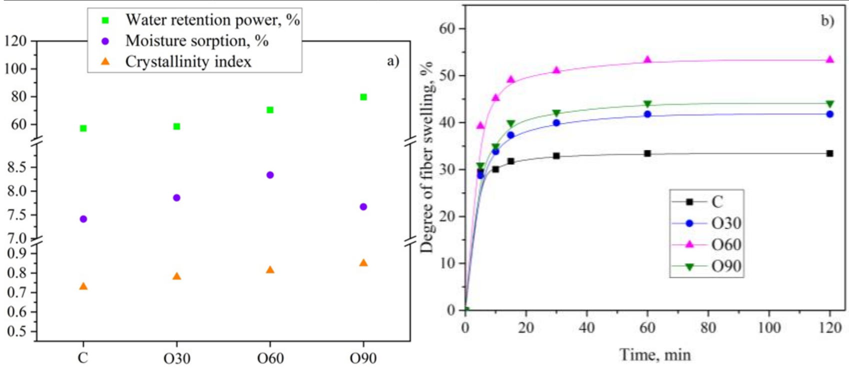

Figure 2: Sorption properties of oxidatively modified jute fabrics (Part of the results published in [1])

The influence of alkali and oxidative modifications on the jute fabric structural parameters is given in Table 2. Namely, with increasing the alkali modification intensity the values of all investigated structural parameters increases. In contrast, the changes in the oxidized jute fabric structural parameters can not be directly connected with the degree of delignification.

Table 2: Jute fabric structural parameters (Part of the results published in [2])

Sample code	Fabric thickness, mm	Fabric weight, g/m^2	Fabric density, 1/dm		Crimp, %	
			Warp	Weft	Warp	Weft
C	0.762	266	46	46	3.46	2.32
A30/1	1.274	313	50	52	17.26	12.28
A5/5	1.389	339	52	54	18.31	14.95
A5/17.5	1.773	495	63	67	21.15	20.15
A30/17.5	1.868	529	64	67	27.33	20.20
O15	1.118	317	49	51	13.04	5.77
O60	1.221	270	50	51	16.75	7.58
O90	1.133	262	51	51	11.04	10.41

In addition to chemical composition and structural parameters, the fabric mechanical properties are also affected by the applied chemical modifications, Figure 3. Decreased maximum force in the case of fabrics A30/1 and A5/5 compared to the raw fabric (sample C) could be ascribed to the fact that during the hemicellulose removal, the interfibrillar regions become less dense and, as it was previously shown (Figure 1a), the crystallinity index decreased. On the other hand, the mercerization (samples A5/17.5 and A30/17.5) in parallel with the higher fabric thickness, weight, and density (Table 2) contributed to a higher maximum force. Having in mind that the lignin is responsible for the cell wall rigidity, it was expected that the selective lignin removal will result in decreased fabric maximum force. By comparing the data obtained for the elongation at maximum force, it can be noticed that all chemically modified jute fabric have a higher elongation at maximum force (especially mercerized ones) than raw jute fabric.

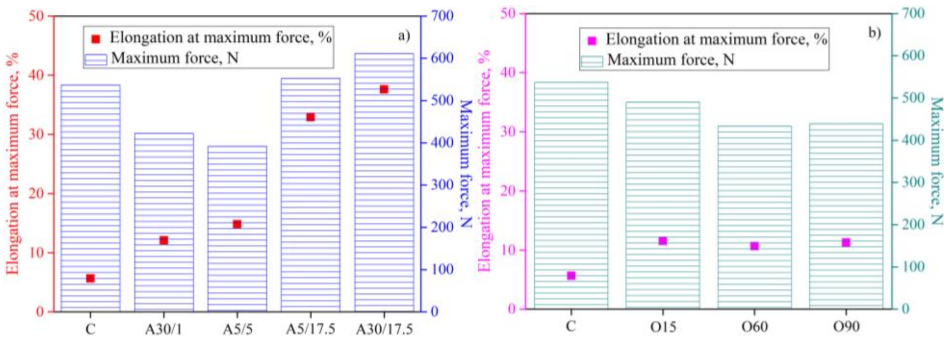

Figure 3: Jute fabric mechanical properties determined in the warp direction (Part of the results published in [2])

Among different fabric properties, the measurement of the electrical resistivity enables us to estimate and predict their tendency to produce static electricity. Alkali modification (sample A5/5) significantly decreased the fabric volume electrical resistivity (Table 3) which can be explained by the increased content of amorphous regions and the number of available hydroxyl groups that are able to sorbing and retaining moisture from the air. Moreover, the higher fabric density provides better contact between yarns, and thus an easier flow of charge. Compared to this sample (A5/5), the oxidized sample (O90) has about 4 times lower volume electrical resistivity. The incorporation of silver ions (Ag^+) decreases the fabrics' volume electrical resistivity and provides a maximum bacterial reduction for both tested bacteria, *E. Coli* and *S. aureus*, Table 3.

Table 3: Volume electrical resistivity and antibacterial activity of jute fabrics before and after incorporation of silver ions (Ag^+) (Part of the results published in [2])

Sample code	Humidity, %				Bacterial reduction, %	
	55	50	45	40		
	Resistivity measured in the weft direction, GΩcm				*E. coli*, %	*S. aureus*, %
C	6.02	7.97	9.53	12.21	99.11	99.28
CAg^+	0.20	0.22	0.25	0.32	99.99	99.99
A5/5	1.00	1.21	1.40	2.41	61.95	92.57
A5/5 Ag^+	0.16	0.18	0.20	0.24	99.99	99.99
O90	0.26	0.33	0.37	0.46		
$O90Ag^+$	0.10	0.11	0.13	0.15		

Taking into account that the fabrics' dielectric properties (AC specific electrical conductivity, dielectric loss tangent, and effective relative dielectric permeability) are very sensitive to their structural characteristics, moisture sorption, fiber molecular and fine structure, it is reasonable to assume that the dielectric properties could be changed after both chemical modifications. The increased AC specific electrical conductivity (σ_{AC}) of alkali modified fabrics (Figure 4a) can be explained by the fact that the hemicelluloses not only restrict the freedom of the water molecules to take part in the polarization process, they also change the structure in such a way that the mobility of the ions in the electric field is restricted. On the other hand, the jute fabrics with 37.8% and 63.2% lower lignin content (samples O15 and O60) and similar moisture sorption values (Figure 2a) have the same σ_{AC} (Figure 4b) revealing a moisture sorption dominant effect. The σ_{AC} of all investigated jute fabrics increases with increasing frequency, whereby the highest difference between the values is observed at the highest frequency (140 kHz).

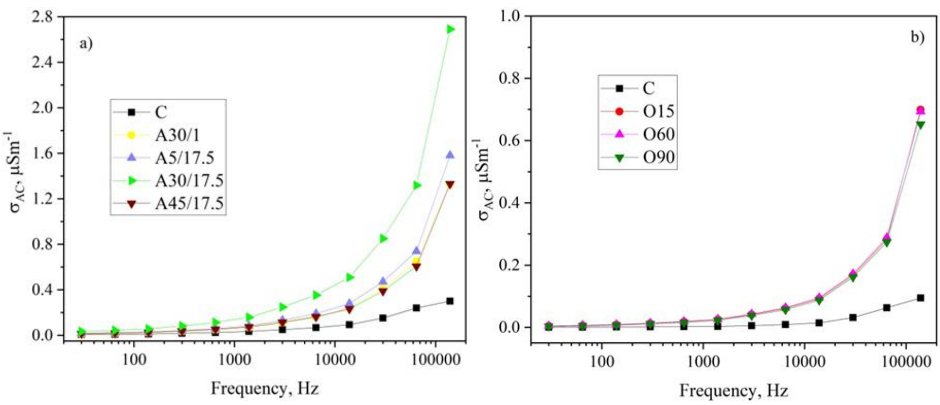

Figure 4: AC specific electrical conductivity (σ_{AC}) of: a) alkali and b) oxidatively modified jute fabrics (at 30% relative air humidity) (Part of the results published in [8])

The dielectric loss tangent (*tan δ*) values increased after the alkali modifications (Figure 5a) due to the progressive hemicellulose removal. Namely, the free spaces within/between the fibrils become larger enabling easy movement of structural components' molecules contributing to higher *tan δ* values. As is evident from Figure 5b, all oxidatively modified jute fabrics have almost the same *tan δ* values, pointing out that this property is not affected by the degree of delignification, moisture sorption, and crystallinity index. On the other hand, these fabrics have a similar thickness and density proving that the structural characteristics have the main role in the *tan δ* of oxidatively modified jute fabrics.

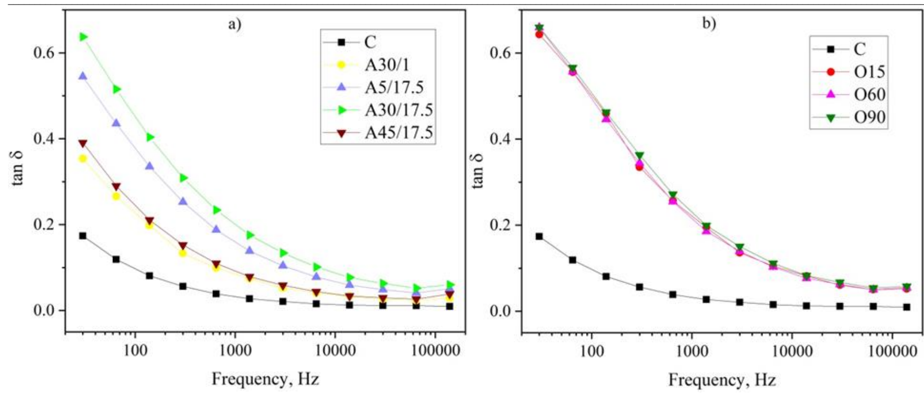

Figure 5: Dielectric loss tangent (*tan δ*) of: a) alkali and b) oxidatively modified jute fabrics (at 30% relative air humidity) (Part of the results published in [1])

The effective relative dielectric permeability (ε'_m) represents another dielectric property that is important for jute fabric utilization. The obtained increase in the ε'_m after the alkali modifications can be attributed to the changes in the fabric structural characteristics and decreased hemicellulose content, which further contributed to decreased crystallinity index and increased ability for moisture sorption. In the case of the oxidatively modified jute fabrics, ε'_m is in line with the degree of delignification.

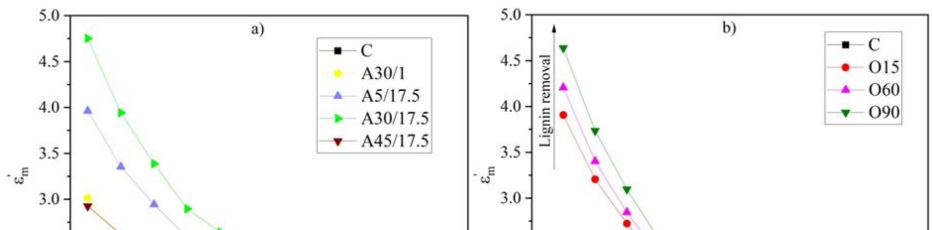

Figure 6: Effective relative dielectric permeability (ε'_m) of: a) alkali and b) oxidatively modified jute fabrics (at 30% relative air humidity) (Part of the results published in [1])

The ε'_m of selected jute fabrics (A30/17.5 and O90) was also studied after the treatment with $CuSO_4$ and coating with Cu-based nanoparticles, at 80% relative air humidity, Figure 7. A few single Cu-based nanoparticles were observed across the alkali modified fabric's surface, while single and agglomerated nanoparticles were distributed over the oxidatively modified fabric's surface. It can be concluded that fabrics with *in situ* synthesis of Cu-based nanoparticles can store 38-88 times more energy from an external electric field than before the copper reduction.

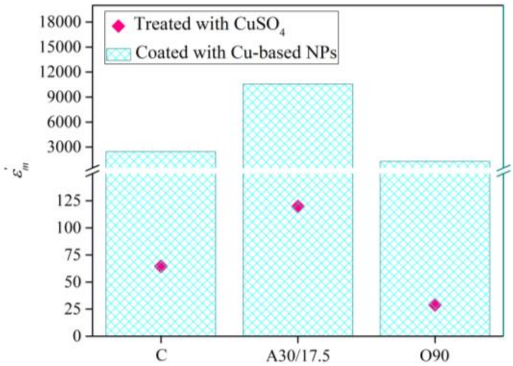

Figure 7: Effective relative dielectric permeability (ε_m') of jute fabrics: a) treated with $CuSO_4$ and b) coated with Cu-based nanoparticles (at 80% relative air humidity)

Following the increased focus on the concept of circular economy, the possibility of jute fabric waste revalorization for wastewater purification from heavy metal ions (such as Ni^{2+}, Cu^{2+}, and Zn^{2+},) was examined. From the results listed in Table 4, it is evident that the chemically modified fabrics' biosorption capacity is affected by the hemicellulose and lignin removal that is accompanied by the increasing amount of carboxyl groups. Among all studied samples, the best biosorption performance possesses jute fabric with 63.2 % lower lignin content as well as 81.1 % higher amount of carboxyl groups (sample O60).

Table 4: Jute fabrics' biosorption capacity for different heavy metal ions (at 20 mg/l initial ion concentration) and content of carboxyl (COOH) groups (Part of the results published in [4])

Sample code	Biosorption capacity, mg/g			Content of COOH groups, mmol/g
	Ni^{2+}	Cu^{2+}	Zn^{2+}	
C	3.78	2.45	2.77	0.207
A30/1	5.61	4.91	4.35	0.342
A5/17.5	5.00	4.44	3.82	0.284
O30	6.05	5.89	4.58	0.345
O60	6.11	6.02	4.50	0.375

3. CONCLUSION

The currents study shows that alkali and oxidative protocols can be successfully applied in order to obtain multifunctional jute fabrics for different applications such as advanced protective textiles, *i.e.* protective clothing in environments sensitive to electrical discharges and carpet backing. Alkali modifications lead to selective hemicellulose removal, increased fabric structural parameters, elongation at maximum force, sorption, and dielectric properties, as well as decreased volume electrical resistivity. On the other hand, the oxidations contributed to selective lignin removal, which consequently causes a decrease in the volume electrical resistivity and fabric maximum force as well as increased fabric sorption and dielectric properties. Silver ions incorporated in the jute fabric samples decreased their volume electrical resistivity even further and provided maximum bacterial reduction for *E. coli* and *S. aureus*. *In-situ* synthesis of Cu-based nanoparticles resulted in 38-88 times higher energy storage from an external electric field. By utilizing the jute fabric waste as a biosorbent for heavy metal ions (such as Ni^{2+}, Cu^{2+}, and Zn^{2+}), we also suggest how to create the closed technological loop focused on the reduction of lignocellulosic textile waste and strengthening of recycling processes.

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