



## A step towards tuning the jute fiber structure and properties by employing sodium periodate oxidation and coating with alginate

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### ABSTRACT

This paper outlines a novel simple protocol for tuning the structure and properties of jute using sodium periodate ( $\text{NaIO}_4$ ) oxidation and coating with alginate. When compared to the raw jute, fabrics oxidized with a 0.2 or 0.4 %  $\text{NaIO}_4$  solution for 30–120 min exhibited an increased aldehyde group content (0.185 vs. 0.239–0.398 mmol/g), a significantly increased negative zeta potential (from  $-8.57$  down to  $-20.12$  mV), a slight disruption of fiber crystallinity, 15.1–37.5 % and 27.9–49.8 % lower fabric maximum force and stiffness, respectively. Owing to the removal of hydrophobic surface barrier, decreased crystallinity index and the presence of micropores on the fabrics' surfaces, oxidized fabrics have a 22.3–29.6 % improved ability for moisture sorption compared to raw fabric. Oxidized fabrics characterized by very long wetting times and excellent antioxidant activities ( $> 98$  %), can find applications as hydrophobic packaging materials. To further extend the utilization of jute in biocarpet engineering such as water-binding geo-prebiotic supports, oxidized fabrics were coated with alginate resulting in 7.9–24.9 % higher moisture sorption and 352–660 times lower wetting times than their oxidized counterparts. This modification protocol has never been applied to lignocellulosic fibers and sheds new light on obtaining jute fabrics with tuned structure and properties intended for various applications.

### 1. Introduction

Nearly a century has passed since sodium periodate ( $\text{NaIO}_4$ ) oxidation was employed for the structural analysis of polysaccharides [1]. More recently, with the development of fast analytical methods and tools for polysaccharide structural characterization, it has evolved from a structural analysis method to a modification technique [2]. Sodium periodate oxidation in an aqueous solution at low temperatures has attracted significant attention in cellulose chemistry due to its ability to tune the content of introduced aldehyde groups by simply varying the reaction conditions. This selective oxidation agent cleaves the carbon–carbon bond at C2–C3, resulting in the formal generation of two aldehyde groups per anhydroglucose unit, leading to the formation of 2,3-dialdehyde cellulose (DAC) [3]. An important piece of information is that the aldehyde functionalities appear only to a small extent in the free

aldehyde form, while most of them are predominantly hydrated or crosslinked to form hemialdals and hemiacetals, both intra- and inter-molecularly [4].

Over the last decade, extensive scientific efforts have been devoted to obtain cellulose, cotton, and viscose fibers with different functionalities for a wide range of practical applications by the utilization of sodium periodate oxidation [5–9]. Sodium periodate oxidation of cellulose has been the subject of numerous studies and dialdehyde cellulose has been explored as a bio-based plastic for packaging applications [8], self-healing nanocomposite hydrogels [10], drug delivery [5,11], wound healing, tissue engineering [5], etc. Fliri et al. [12] have reported on well-known issues related to the cost and (eco)toxicity of the periodate oxidant, which can be overcome by the recycling protocols [13,14], aligning with the principles of green chemistry.

A detailed literature survey has revealed that a neglected area in

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sodium periodate oxidation is its employment in a modification protocol for lignocellulosic fibers like hemp, ramie, kenaf, jute, etc. Hence, in this paper, particular attention is paid to the periodate oxidation of the most important lignocellulosic fiber – jute. It is ranked as the world's second most important natural fiber after cotton (Food and Agriculture Organization, FAO). The primary objective of this work was to tune the jute structure and properties. Several attempts have been already made to achieve the above-mentioned through selective hemicellulose or lignin removal using alkali treatment with sodium hydroxide or oxidation with sodium chlorite [15–19], both having certain environmental drawbacks. To overcome some of the shortcomings of existing chemical modifications, in this fundamental research, raw jute fabric was oxidized with sodium periodate aqueous solution of two different concentrations during different reaction times. Well-known spectroscopy and microscopy methods and different standards were used for fabric characterization (contents of aldehyde and carboxyl groups, fiber surface chemistry and morphology, and crystallinity index) and determination of their mechanical (maximum force and stiffness), electrokinetic (zeta potential), antioxidant, and sorption (moisture sorption and wetting time) properties.

Adjusting the sorption properties of raw and oxidized jute fabrics (ranging from highly hydrophobic to highly hydrophilic) by a simple coating with alginate was also the objective of this research. Alginate, a naturally occurring polysaccharide known for its versatility, sustainability, nontoxicity, and relatively low cost, is widely exploited for obtaining functional materials [20,21]. Also, it can undergo mild gelation by interacting with most polyvalent metal ions [22]; thus, alginate is easily incorporated into multiple products such as fibers, films, and hydrogels. The adjustment of the jute's sorption properties and obtaining hydrophobic oxidized jute fabrics and hydrophilic fabrics coated with alginate allows us to make it a perfect starting material for both conventional (as matting, awning, and packaging materials for different commodities) and novel bio-based materials with several targeted applications. More specifically, highly hydrophilic alginate-coated oxidized fabrics can be applied in the field of biocarpet engineering, i. e., water-binding geo-prebiotic polysaccharide supports which in combination with cyanobacteria strains could be used for the rehabilitation of various degraded lands, sediments, and substrates, as well as for air and water pollution control.

The research described in the manuscript aimed to confirm two hypotheses: (1) sodium periodate oxidation tunes the jute molecular and fine structures as well as its electrokinetic, mechanical, and sorption

properties; and (2) the alginate coating of periodate-oxidized jute fabrics enhances their moisture sorption and wettability. The overall novelty of this paper is emphasized in Fig. 1.

## 2. Material and methods

### 2.1. Materials

Raw jute fabric in the plain weave procured from Dekoteks Trade (Belgrade, Serbia) with the following chemical composition: 21.76 % hemicelluloses, 13.48 % lignin, 60.09 %  $\alpha$ -cellulose, and 4.67 % extractives, was used as a starting material. All chemicals, purchased from commercial suppliers, were of p.a. grade and used as received. Employed alginic acid sodium salt (Thermo Scientific Chemicals, CAS No: 9005-38-3) was of low viscosity.

### 2.2. Sodium periodate oxidation of raw jute fabric

To tune the structure and properties of jute, previously washed (with distilled water at boiling temperature for 30 min) and dried, raw jute fabric (denoted as J) was oxidized with  $\text{NaIO}_4$  aqueous solution according to the procedure given by Ivanovska et al. [23]. Briefly, J was immersed in 0.2 or 0.4 %  $\text{NaIO}_4$  aqueous solution (material-to-liquid ratio of 1:50) prepared by dissolving  $\text{NaIO}_4$  in an acetic buffer having a pH value of 4.00. Different oxidation reaction times (30, 60, 90, or 120 min) were carried out in the dark at room temperature with constant stirring. The oxidation process was halted by immersing the fabrics in ice-cold distilled water and then drying them at room temperature. The fabrics' codes are presented in Fig. 2.

### 2.3. Jute fabric treatment with sodium alginate solution

After washing or immersing in ice-cold distilled water, raw jute and selected oxidized jute fabrics (J0.2/60, J0.2/120, J0.4/60, and J0.4/120) were squeezed and immediately immersed in a 0.5 % sodium alginate solution having a pH of 7 (material-to-liquid ratio of 1:20) for 24 h at room temperature. After that, the fabrics were squeezed and immersed in a 1.5 %  $\text{CaCl}_2$  aqueous solution (material-to-liquid ratio of 1:20) for 24 h at room temperature, rinsed with distilled water, and dried in a laboratory oven for 24 h at 60 °C.

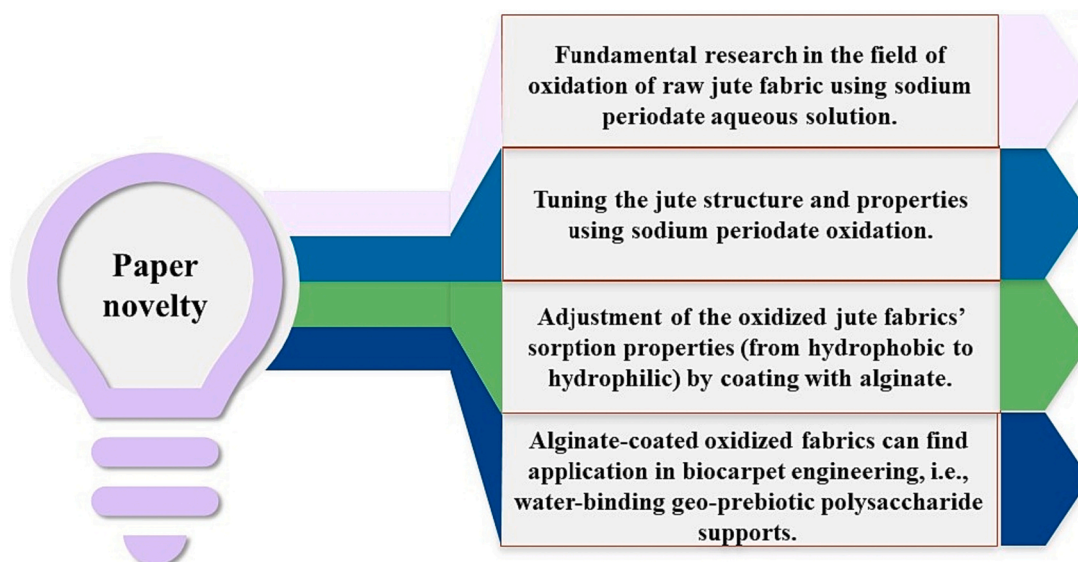


Fig. 1. Paper novelty.

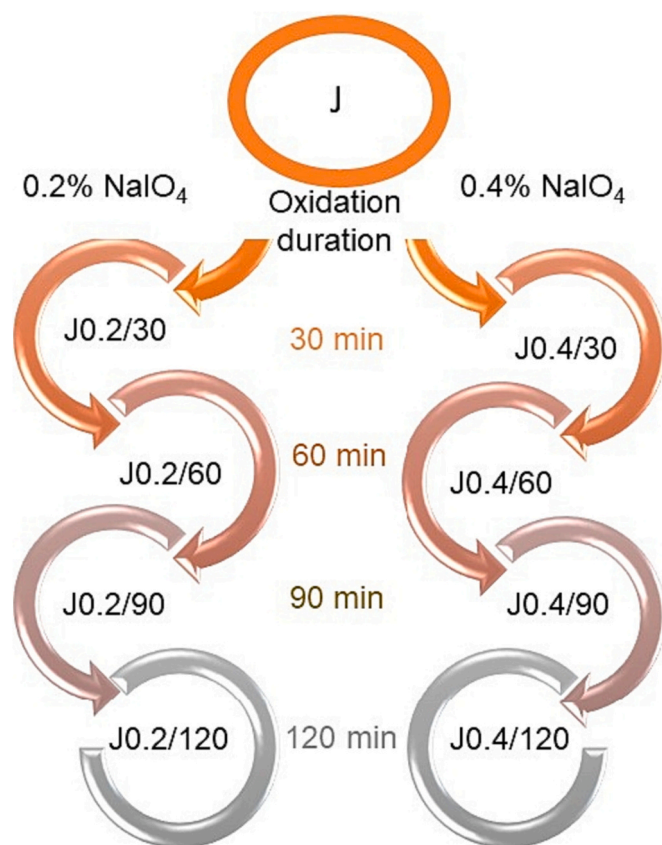


Fig. 2. Jute fabrics' codes.

#### 2.4. Characterization of raw and oxidized jute fabrics

The carboxyl (COOH) and aldehyde (CHO) group contents of studied jute fabrics were determined according to the methods outlined by Ivanovska et al. [24] with some modifications. The presented results for COOH and CHO group contents represent the mean values of five and three measurements per sample, respectively.

Fabric surface chemistry was assessed using ATR-FTIR spectroscopy (Nicolet™ iSTM 10 FT-IR, Thermo Fisher 2 SCIENTIFIC); FTIR spectra were recorded in the range of 4000–400 cm<sup>-1</sup> with 32 scans per spectrum. Field Emission Scanning Electron Microscopy (FESEM, Tescan MIRA 3 XMU) was used to evaluate changes on fabrics' surfaces. Before the FESEM analysis, all samples were sputter-coated with a thin layer of gold.

The fibers' crystallinity index (*CrI*) was calculated following the method described by Ivanovska et al. [16]. X-ray diffraction patterns were recorded using a Rigaku Ultima IV diffractometer with CuK $\alpha$  radiation. The diffraction data were acquired with a step of 0.05° and an acquisition time of 0.5°/min.

#### 2.5. Determination of fabric properties

The fabric zeta potential as a function of pH value was determined using a streaming potential method with a SurPASS electrokinetic analyzer (Anton Paar GmbH, Austria) according to the procedure described by Ivanovska and Kostić [25].

The fabric maximum force in the warp direction was determined based on EN ISO 13934-2:2014 standard using Tinius Olsen H5KT dynamometer, while the fabric stiffness was measured on the Digital Pneumatic Stiffness Tester M003F following ASTM D 4032–94 standard. The reported results are the average value of three measurements per sample.

The sorption properties of jute fabrics were assessed based on changes in moisture sorption and wetting time. Before moisture sorption determination on an Infrared Moisture Analyzer MA 35 (Sartorius, Göttingen, Germany), fabrics were conditioned in accordance with the standard SRPS EN ISO 139:2007/A1:2014. The fabric wetting time was measured following the standard AATCC 79–2018. For each fabric, moisture sorption, and wetting time were determined in triplicate.

Fabric antioxidant activity was tested according to the protocol published by Kraševac Glaser et al. [26]. The presented results represent the mean value of three parallel measurements, wherein the coefficients of variations were below 3.51 %.

#### 2.6. Statistical analysis

The results obtained in this study were statistically analyzed using the *t*-test. The parameter *t* was determined according to Eq. (1):

$$t = \frac{\bar{x}_1 - \bar{x}_2}{\sqrt{\frac{s_1^2(n_1-1) + s_2^2(n_2-1)}{n_1+n_2-2} \cdot \frac{n_1+n_2}{n_1 \cdot n_2}}} \quad (1)$$

where:  $\bar{x}_1$  and  $\bar{x}_2$  are the samples' means of the determining characteristic,  $s_1$  and  $s_2$  are the standard deviations of the determining characteristic,  $n_1$  and  $n_2$  are the sample sizes.

### 3. Results and discussion

#### 3.1. The influence of sodium periodate oxidation on the functional group contents

The raw jute fabric was oxidized with a NaIO<sub>4</sub> aqueous solution, a simple and effective method for introducing aldehyde functionalities to polysaccharides and altering the fibers' molecular and fine structure. This selective oxidation stands as one of the most effective routes for tuning the content of aldehyde groups by varying the reaction conditions. In theory, sodium periodate oxidation of cellulose (the main component of jute fibers) leads to the oxidation of glucopyranose ring's secondary hydroxyl (OH) groups (positioned at the C2 and C3 atoms) to aldehydes (CHO), which in parallel results in ring cleavage, and the formation of 2,3-dialdehyde cellulose [6], Fig. 3.

In this investigation, the oxidations of raw jute were carried out while varying the NaIO<sub>4</sub> concentration (0.2 and 0.4 %) and/or reaction duration (30–120 min), Fig. 2. The effect of both variables on the jute molecular structure was firstly assessed by determining the aldehyde and carboxyl group contents (Fig. 4), whereby the potentiometric titration was chosen as an appropriate method for their quantification. The results shown in Fig. 4a undoubtedly indicated that the jute fabrics exhibited an increase in CHO group content with an increase in the NaIO<sub>4</sub> concentration and/or reaction duration time. This observation is in coherence with the conclusions drawn by Nikolić et al. [27] and Kramar et al. [6] for the periodate oxidized viscose yarns and viscose fabrics, respectively. The CHO group content in the raw jute fabric is 0.185 mmol/g, while the fabrics oxidized with 0.2 % NaIO<sub>4</sub> for 30, 60, 90, or 120 min displayed about 28.8, 44.2, 57.8, and 76.7 % higher CHO group contents than the raw one. As expected, higher contents of CHO groups were introduced upon the oxidation with a higher concentration

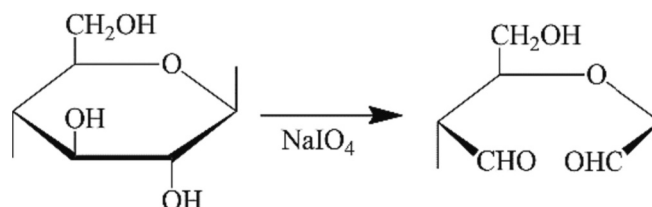


Fig. 3. Sodium periodate oxidation of cellulose.

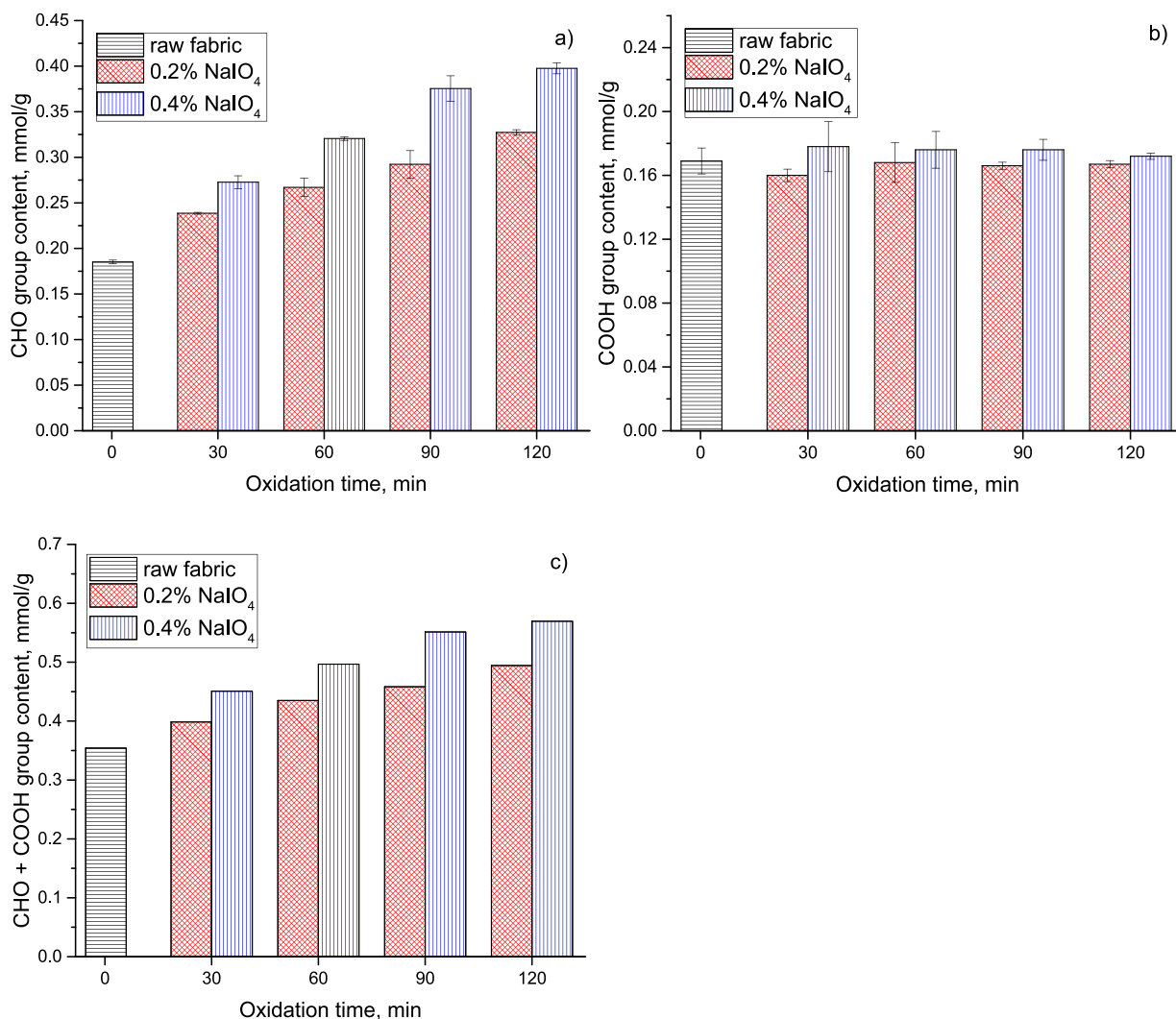


Fig. 4. The effect of oxidation conditions on fabric: a) CHO, b) COOH, and c) CHO + COOH group contents.

of oxidizing agent. The differences between the CHO group contents between the raw fabric and those oxidized with 0.4 % NaIO<sub>4</sub> for 30, 60, 90, or 120 min accounted for 47.2, 73.1, 102.7, and 114.7 %, respectively, Fig. 4a. It is worth mentioning that properly selected reaction conditions contributed to obtaining jute fabrics with tuned and significantly different contents of CHO groups (with exception of those between J0.2/60 and J0.2/90, and between J0.4/90 and J0.4/120, Supplementary material, Table S1). The capability of sodium periodate oxidation for tuning the CHO group content is further supported by the linear correlations of 0.995 and 0.990 that exist between the oxidation time and CHO group content in jute fabrics oxidized with 0.2 % and 0.4 % NaIO<sub>4</sub>, respectively (Supplementary material, Fig. S1). On the other hand, the changes in COOH group content after the performed oxidations are generally insignificant (Fig. 4b and Supplementary material Table S1), proving that the increased total content of CHO and COOH groups (Fig. 4c) is solely owed to the introduction of CHO groups.

In our quest to determine the optimal oxidation parameters, we devoted special attention to the possibility of sodium periodate regenerating after oxidation, a matter of utmost importance. Fliri et al. [12] reported on known issues regarding the cost and (eco)toxicity of the periodate oxidant, which can be overcome by the recycling protocols [13,14,28], which is in line with the green chemistry guidelines. Liimatainen et al. [28] found out that under mild oxidation conditions (aldehyde content around 0.4 mmol/g), it is possible to regenerate 100 % of the periodate used in the reaction with a minor excess of sodium

hypochlorite. Therefore, after all the performed oxidations in the current study (CHO group content ranging from 0.239 to 0.398 mmol/g, Fig. 4a), the periodate solutions used in the reaction can be successfully regenerated.

An in-depth investigation of fabric surface chemistry and morphology, fiber fine structure, and different properties was performed on raw jute (labeled as J) and fabrics oxidized with 0.2 % or 0.4 % sodium periodate for 60 or 120 min (marked as J0.2/60, J0.2/120, J0.4/60, J0.4/120), Fig. 2.

### 3.2. Fabric surface chemistry

As fabric surface chemistry significantly influences its interaction with water, the changes in the jute fabric surface chemistry induced by sodium periodate oxidation were monitored using ATR-FTIR spectroscopy. From FTIR spectra shown in Fig. 5, it was spotted the presence of an absorption band at 1735 cm<sup>-1</sup>, which is characteristic for carbonyl (C=O) valence vibration of acetyl- or COOH-groups in hemicelluloses as well as non-cross-linked dialdehyde cellulose [29,30]. The efficiency of periodate oxidation was analyzed through the deconvolution of the band belonging to C=O group and a comparative analysis of the changes in the band area assigned to ester, aldehyde, and carboxyl groups (detailed results in Supplementary material, Fig. S2 and Table S2). Notably, the band area corresponding to ester groups is diminished in oxidized jute fabrics compared to the raw jute fabric,

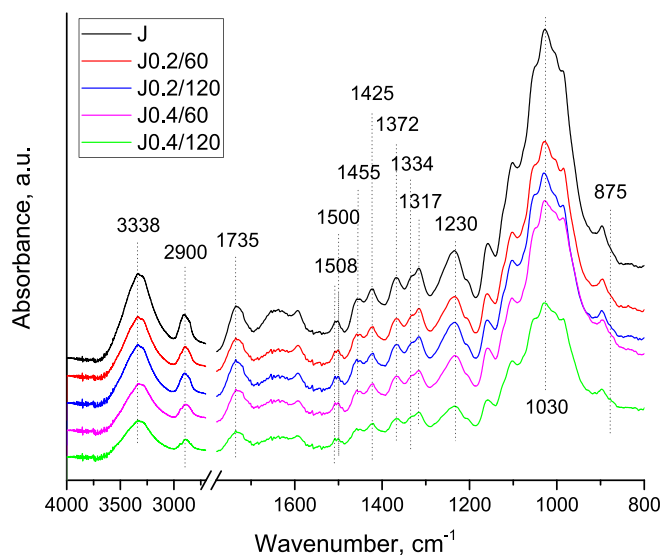


Fig. 5. ATR-FTIR spectra of raw and periodate oxidized jute fabrics.

while the band area attributed to aldehyde groups is significantly higher for oxidized jute fabrics. However, a slight difference between the area of the bands attributed to aldehyde and carboxyl groups (Table S2) contradicts the findings obtained through titration methodology (Fig. 4b). This inconsistency can be explained by the fact that the development of the aldehyde moiety in cellulose is masked by the presence of hydrates and hemiacetals, which are formed depending on the fabric's moisture content [29]. During the oxidation process, the acidic pH (4.0) promotes the formation of hemiacetals between  $-CHO$  and adjacent  $-OH$  groups or the hydration of aldehydes. Confirmation for this lies in the detected shoulder at  $875\text{ cm}^{-1}$  (corresponding to hemiacetal vibrations [3]) and the weakened signals at  $1030\text{ cm}^{-1}$  (C–O–C pyranose ring vibration [3]),  $1317\text{ cm}^{-1}$  (O–H bending vibrations) and at  $3338\text{ cm}^{-1}$  (O(2)H...O(6) intramolecular hydrogen bond [31]) in the spectra of oxidized fabrics, Fig. 5. A progressive weakening of the signals corresponding to O–H bending vibrations and intramolecular hydrogen bonds during the periodate oxidation of cellulose was also observed by Simon et al. [3].

The intensities of the bands at  $3338\text{ cm}^{-1}$  and  $1334\text{ cm}^{-1}$  were used to calculate the Hydrogen Bond Intensity (HBI), a parameter related to the regularity of intramolecular hydrogen bonding in cellulose I [31]. The decrease in HBI of J0.2/60, J0.2/120, J0.4/60, and J0.4/120 compared to J (Table 1) suggests the disruption of OH groups (due to the conversion to CHO groups) and their inter- and intramolecular bonding during the oxidation [6].

The decrease in the intensity of the bands at  $1230\text{ cm}^{-1}$  (C–O stretching vibration of the acyl group of lignin [32]),  $1425$ ,  $1455$ , and  $1508\text{ cm}^{-1}$  (aromatic ring vibrations of the lignin phenyl propane groups [33,34]), and  $1500\text{ cm}^{-1}$  (C=C stretching vibration of the benzene ring of lignin [32]) in the spectra of oxidized fabrics can be explained by the less condensed and less exposed lignin on the fabric's surface. The appearance of broadband positioned at  $2900\text{ cm}^{-1}$

**Table 1**  
Parameters calculated from the intensities of different absorbances (A) in fabrics' FTIR spectra.

Samples	HBI ( $A_{3338}/A_{1334}$ )	Lignin/Cellulose ( $A_{1508}/A_{1317}$ )	TCI ( $A_{1372}/A_{2900}$ )
J	1.025	0.708	1.401
J0.2/60	0.944	0.621	1.357
J0.2/120	0.987	0.553	1.374
J0.4/60	0.924	0.573	1.347
J0.4/120	0.958	0.677	1.336

(comprising of bands' shoulders centered at  $2849\text{ cm}^{-1}$  and  $2917\text{ cm}^{-1}$ ) is connected with the C–H stretching vibration of  $CH_2$  and CH groups in cellulose and hemicelluloses [30]. After oxidation, the reduced intensities of the bands at  $2917\text{ cm}^{-1}$ ,  $2849\text{ cm}^{-1}$  and the earlier mentioned band at  $1735\text{ cm}^{-1}$  (originating from hemicelluloses and the non-cross-linked dialdehyde cellulose) and those assigning to lignin (at  $1508$ ,  $1500$ ,  $1455$ ,  $1425$ , and  $1230\text{ cm}^{-1}$ ) indirectly indicate more exposed cellulose, as also evident from the decreased lignin/cellulose ratio (Table 1).

Due to oxidation under different conditions, changes in the bands at  $2900$  and  $1372\text{ cm}^{-1}$  provide information that the cellulose crystalline areas are affected by the oxidation agent [6], as also obvious from the lower Total Crystallinity Index (TCI) of oxidized jute fabrics, Table 1. Since the decline in TCI is interpreted as less ordered cellulose molecules, this parameter suggests a decrease in fiber crystallinity, which is discussed below.

### 3.3. Crystallinity index of raw and oxidized jute

Besides the fact that after studied oxidations, negligible weight loss (0.24–2.28 %) was observed due to the agent consumption by the fiber non-cellulosic components (primarily by the surface impurities), this process is very complex. It proceeds via three reactions related to the evolution of crystallinity in the periodate oxidation system: (1) an initial random oxidation that occurs in the cellulose amorphous regions, followed by (2) the oxidation of the crystallite surfaces, and (3) a slow reaction due to the oxidation of the crystalline core [35]. Therefore, the sodium periodate oxidation is accompanied by the rearrangement of the cellulosic chains, i.e., a slight disruption of the fiber crystallinity.

The crystallinity index (*CrI*), an essential parameter of the fiber's fine structure was determined using the peak area method. The recorded X-ray patterns were deconvoluted and fitted using Gaussian and Lorentzian functions [16]. All patterns consist diffraction from an amorphous region and sharp peaks assigned to a crystalline region. The cellulose I structure remained unchanged after the periodate oxidations and its characteristic peaks are located at  $2\theta$  of  $14.7^\circ$ ,  $16.8^\circ$ , and  $22.7^\circ$  corresponding to the reflections (1–10), (110), and (020), respectively [36], Fig. 6.

With increasing the  $NaIO_4$  concentration from 0.2 % to 0.4 %, a slight decrease in the fiber *CrI* was observed due to the opening of glucopyranose ring, which led to slight disruption of the cellulose ordered structure. This phenomenon mainly occurred at the ends of the accessible polymeric chains generating the fragmentation of particles by

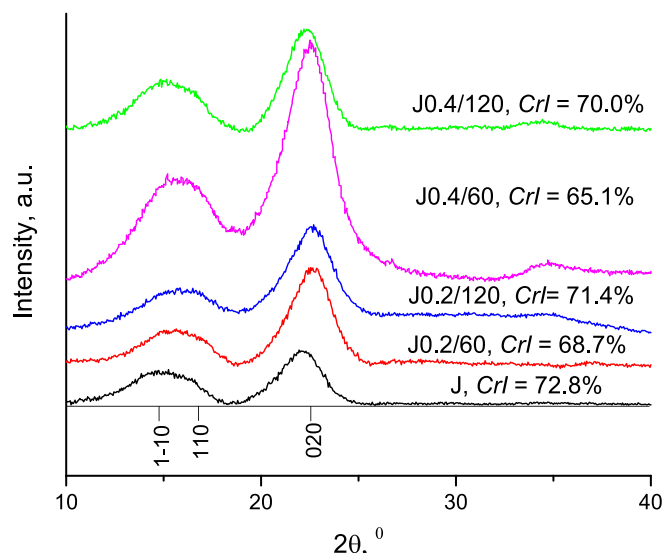


Fig. 6. X-ray diffraction patterns and crystallinity index (*CrI*) of jute.

disruption of intramolecular hydrogen bonds. Hence, there is a conformity between the  $CrI$  (Fig. 6) and HBI (Table 1); the linear correlation between these parameters is 0.930 pointing out that the  $CrI$  is proportional to the regularity of intramolecular hydrogen bond. Fig. 6 also provides quantitative evidence of a slightly improved alignment of cellulose crystalline domains by prolonging the oxidation duration from 60 to 120 min (independently of the  $NaIO_4$  concentration used) which is attributed to: (1) the introduction of a significant amount of aldehyde groups within J0.2/120 and J0.4/120 fabrics (Fig. 4a) enabling the rearrangement of amorphous regions and (2) the formation of intermolecular and intramolecular hemiacetals with concomitant ample crosslinking possibilities [9], as discussed in the previous section. These observations are in line with already published data [6,27].

According to all the above discussed, it can be concluded that the fiber's fine structure was not significantly influenced by the applied periodate oxidations. Guigo et al. [37] highlighted that at carbonyl content between 0.38 and 1.75 mmol/g (as in the current paper), cellulose microfibrils were oxidized exclusively at their surfaces and cellulose did not seem to be substantially affected by the oxidation. However, cellulose oxidation is a self-accelerating process and the slight decrease in crystallinity improves fiber accessibility and reactivity [35], which has also been demonstrated for sodium periodate oxidized cotton [38] and viscose [6].

### 3.4. The effect of sodium periodate oxidation on the fabrics' electrokinetic properties

The aforementioned accessibility of fabric functional groups along with their dissociation ability affect the distribution of surface charge and the thickness and distribution of the electrochemical double layer, which in turn, leads to changes in fabric electrokinetic properties [39]. Following these facts, raw and oxidized jute fabrics were subjected to zeta potential ( $\zeta$ ) measurements in a wide pH range (Fig. 7), while fabric isoelectric point (IEP) was determined by extrapolation. The pH of an aqueous solution is the driving mechanism for acid-base reactions, i.e. a higher pH promotes the dissociation of acidic groups on the fibers' surfaces while suppressing the protonation of basic groups and vice versa [40].

Raw jute (J), like other cellulosic fibers, exhibited a negative zeta potential in the entire measured pH range, which is a consequence of the presence of hydroxyl and carboxyl groups on its surface that are susceptible to dissociation upon contact with an electrolyte solution resulting in a negative charge [41]. Due to the removal of fabric surface

impurities during the oxidation, such groups become more accessible leading to an increase in the fabrics' active sites which is evident from more negative  $\zeta_{\text{plateau}}$  value. The changes induced by sodium periodate oxidations are manifested as a decrease in the fabrics'  $\zeta$  (from  $-8.57$  for J down to  $-20.12$  mV for J0.4/60) and shift the pH value where the minimum  $\zeta$  value is detected to lower pH values (from 4.10 down to 3.88), Fig. 7. For the oxidized fabrics, protonation contributes to the formation of a higher positive charge in the electrochemical double layer causing increased adsorption of  $Cl^-$  ions (originating from electrolyte), and hence, resulting in a significant decrease in  $\zeta$  at lower pH values [25].

In addition to the  $\zeta$ , IEP (a pH value at which the  $\zeta$  value is 0) was monitored as an indicator of the nature of the functional groups present on the fabrics' surfaces. As shown in Fig. 7, the IEP of J is 2.61, suggesting a positive fabric surface at pH values below 2.61, and a negatively charged surface above this pH value. The IEPs of oxidized fabrics shifted to higher pH of a 2.75–2.84 because the number of the groups prone to deprotonation is lowered due to the conversion of OH to CHO groups that are not charged. It should be kept in mind that fabrics J0.2/60, J0.2/120, and J0.4/120 exhibit very similar surface chemistry since their IEPs are nearly identical.

### 3.5. Jute fabric mechanical properties

When considering the potential applications of oxidized fabrics, special attention should be paid to their fundamental mechanical properties like maximum force and stiffness. These properties are not only associated with fabric applications, they are also responsible for the product's durability. Fig. 8a presents the maximum force and stiffness of raw and differently oxidized jute fabrics. The difference in maximum force between raw fabric and fabric oxidized with 0.2 %  $NaIO_4$  for 60 min is negligible and not statistically significant (Supplementary material, Table S3); fabric J0.2/60 retained 93.9 % of the maximum force of the raw jute fabric. However, as the severity of oxidation increases, the maximum forces of the oxidized fabrics J0.2/120, J0.4/60, and J0.4/120 decrease by 15.1, 27.5, and 37.5 %, respectively. The most likely explanation for this trend is the cellulose destruction to a certain extent, i.e. breaking of glycosidic bonds and tending of cellulose macromolecules to depolymerize, which is caused by subsequent reactions and not solely by oxidation itself [42]. Although the maximum force does not exhibit a good linear correlation with the fiber  $CrI$ , it should be mentioned that the slight disruption of the cellulose's ordered structure (i.e., increasing in the amount of amorphous regions, Fig. 6) upon sodium periodate oxidation weakened the fabric maximum force. An additional reason for the lowest maximum force of J0.4/120 is the observed fiber damage (Fig. 8b), which is not the case for raw fabric (covered by an outer layer of surface impurities that protect the fiber from damage) and other oxidized fabrics, which is detailed discussed in the next section.

Due to the oxidation with sodium periodate, the intrinsically rigid cellulose structure is converted to a flexible open-chain structure while keeping the backbone integrity [43]. As a consequence, the cellulose hydrogen bond network, which is largely responsible for the fabrics' mechanical properties, is destroyed inducing a major release of molecular motions within and between the chains. The decrease in the regularity of hydrogen bonds in cellulose is evident from the lower HBI values (Table 1) and the weakened FTIR signals at  $3338\text{ cm}^{-1}$  (O–H stretching vibrations) and  $1315\text{ cm}^{-1}$  (O–H bending vibration) and  $1030\text{ cm}^{-1}$  (C–O–C band in cellulose) of the oxidized fabrics compared to the raw fabric, Fig. 5. All of the discussed factors contribute to the reduced fabrics' ability to withstand deformation under stress, i.e., 27.9, 38.7, 40.9, and 49.8 % lower stiffness of J0.2/60, J0.2/120, J0.4/60, and J0.4/120 in relation to J, Fig. 8a. The differences in stiffness between the fabrics are statistically significant at levels of significance 0.05, 0.01, and 0.001, Supplementary material, Table S3.

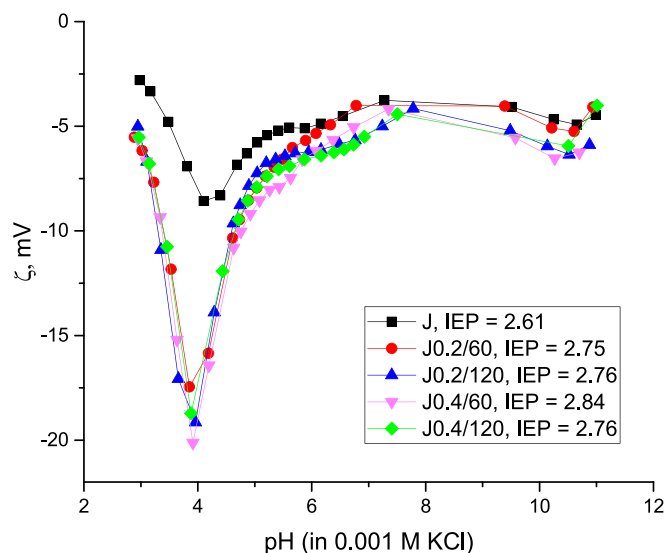


Fig. 7. Fabrics' zeta potential ( $\zeta$ ) as a function of pH.

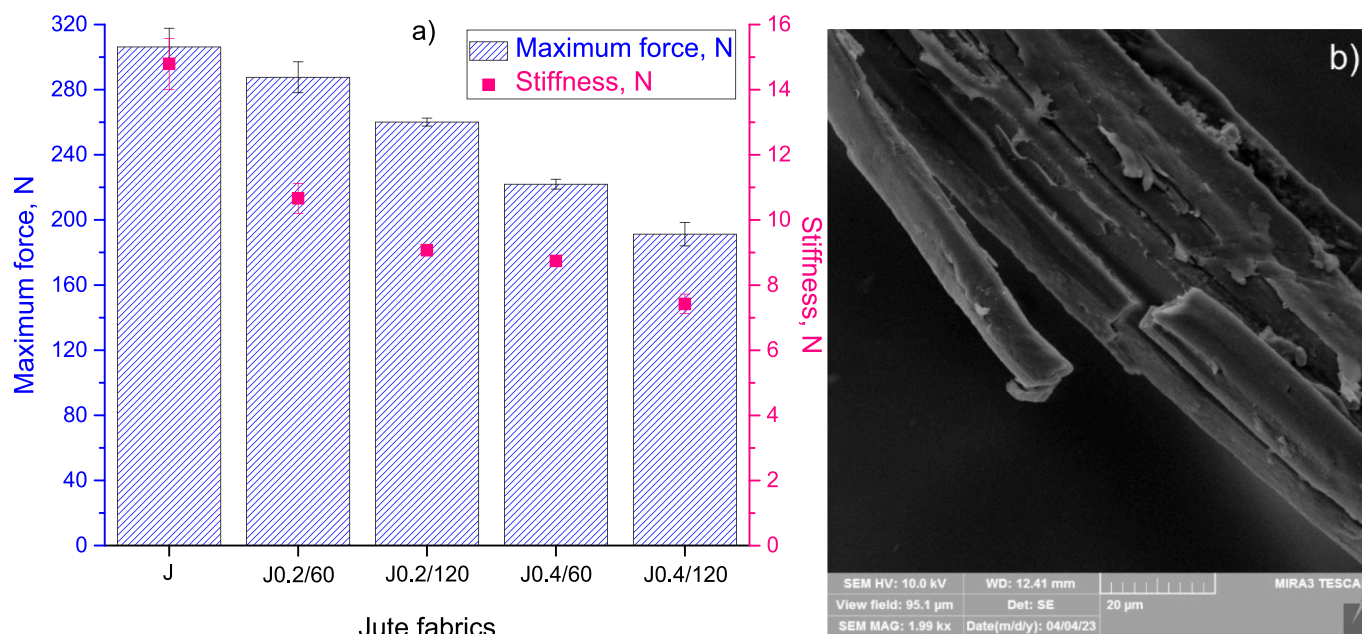


Fig. 8. a) Mechanical properties of investigated fabrics, b) SEM photograph of J0.4/120.

### 3.6. Investigation of the fabric sorption properties

Recent studies [16,17,44] suggest that the fiber molecular (content, nature, and accessibility of functional groups), and fine (degree of crystallinity) structures and surface morphology (pore system) play a significant role in the jute fabric sorption properties. Understanding the correlations between these variables and fabric sorption properties is of exceptional importance for comprehending fabric behavior during different wet processes and potential applications. Fabric sorption properties can be evaluated through various methods: (1) those that provide information about the accessibility of areas and cell wall components to water vapor (moisture sorption), or aqueous solution (fibers swelling, capillarity, and water retention) and (2) those related to fabric wettability (contact angle, and wetting time).

In this study, moisture sorption and wetting time were selected as the simplest and quickest experimental methods to assess the sorption properties of raw and oxidized jute fabrics. From Table 2 and Supplementary material Table S4, it is apparent that all oxidized fabrics exhibited statistically significant better ability for moisture sorption than the raw one which is the consequence of three different factors acting simultaneously. First of all, it is related to the removal of the hydrophobic surface barrier (evident from a weight loss after oxidation and SEM photographs, Fig. 9) allowing water vapor to reach the remaining cellulose, hemicelluloses, and lignin hydroxyl groups. The second factor influencing the enhanced moisture sorption of oxidized fabrics is the crystallinity index, i.e., oxidized fabrics' higher amount of

Table 2

Sorption properties of jute fabrics before and after coating with alginate (samples containing "+ AL" in their codes).

Sample code	Moisture sorption, %	Wetting time, s
J	7.41 ± 0.23	535.25 ± 18.4
J0.2/60	9.11 ± 0.50	1564.96 ± 5.55
J0.2/120	9.06 ± 0.90	2850.12 ± 43.65
J0.4/60	9.60 ± 0.46	> 3600
J0.4/120	9.26 ± 0.93	> 3600
J + AL	8.73 ± 0.51	8.68 ± 0.55
J0.2/60 + AL	9.83 ± 0.33	4.44 ± 0.36
J0.2/120 + AL	10.10 ± 0.25	4.32 ± 0.20
J0.4/60 + AL	11.86 ± 0.46	4.14 ± 0.30
J0.4/120 + AL	11.57 ± 0.61	3.96 ± 0.42

less ordered amorphous regions. Observing in parallel the results obtained for *CrI* (Fig. 6) and moisture sorption values (Table 2) of oxidized fabrics, it is obvious that as the *CrI* decreases by 3.8–10.6 %, the fabrics' moisture sorption values increase by 22.3–29.6 %. These results are logical since moisture sorption primarily occurs in amorphous regions or on the crystallites' surfaces, where free hydroxyl groups are responsible for moisture sorption [17]. Namely, the sorption of water vapor begins with the formation of a tightly bound monolayer, in which one water molecule is bound to each accessible hydroxyl group. Further binding of water molecules is achieved by forming hydrogen bonds with the existing monomolecular layer, leading to the formation of additional layers of water (multilayers) [45]. Moreover, the effect of the pore system on the fabrics' moisture sorption should not be overlooked. The SEM photographs shown in Fig. 9 revealed the existence of micropores on the fabrics' J0.2/120 and J0.4/120 surfaces upon a deeper oxidant penetration, making the fiber structure more open and accessible for water vapor. The existence of such micropores was not observed on the surface of raw fabric (J), which is covered with hydrophobic impurities.

The observed slightly and statistically insignificant (Supplementary material, Table S4) lower moisture sorption values of fabrics treated for 120 min (J0.2/120 and J0.4/120) compared to those treated for 60 min (J0.2/60 and J0.4/60) can be described by reducing the number of hydroxyl groups due to their conversion into aldehydes and the formation of stable covalent intra- and intermolecular hemiacetal bonds with adjacent hydroxyl groups altogether leading to the slightly reduced fiber accessibility to water in a gaseous state [46]. The resulting hemiacetal bonds are responsible for cross-linking [4,9,27,29,46 and discussion in Section 3.2.] causing fiber structure to become more compact (evident from increased *CrI* of J0.2/120 and J0.4/120 compared to J0.2/60 and J0.4/60, Fig. 6), and occupying some of the potential moisture binding centers [27].

Exactly the mentioned formation of CHO groups, which are less prone to interact with water molecules and the lack of binding centers due to the hemiacetal cross-linking are the main reasons for the extremely long wetting times of the oxidized fabrics, Table 2. Initially, these results may not appear satisfactory since the time taken for a water droplet to be fully absorbed in fabrics J0.4/60 and J0.4/120 exceeds 1 h. On the other hand, this characteristic could be advantageous since hydrophobic fabrics can find applications as matting, awning, and packaging materials for various commodities, preventing product spoilage

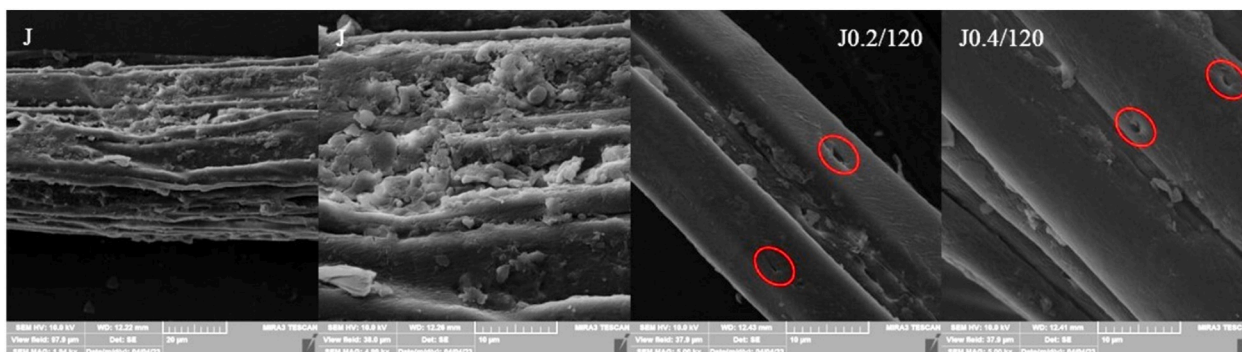


Fig. 9. SEM photographs of raw and oxidized jute.

regardless of storage conditions. It is good to mention that India is the world's leading jute producer, and in March 2023, its Ministry of Textile “issued a notification directing that certain commodities (100% of produced food grains such as rice, paddy, and wheat, and 20% of the sugar) shall be packed in jute packaging material for supply, or distribution” [47]. In light of that, jute antioxidant activity is not an essential but desired property for fabrics intended for packaging materials. Different periodate oxidation protocols did not change the excellent antioxidant activity of raw jute (97.61 %); the antioxidant activities of J0.2/60, J0.2/120, J0.4/60, and J0.4/120 are 98.37, 97.66, 98.93, and 99.09 %, respectively.

Taking into account that jute is the second most important natural fiber (after cotton) and that its annual world production exceeded 3.46 million tons in 2021 (according to Food and Agriculture Organization, FAO), we have decided to further extend its application into non-conventional areas, i.e., we aim to obtain hydrophilic fabrics suitable for use in biocarpet engineering. For that purpose, coating with sodium alginate (a water-soluble salt of alginic acid) is an elegant method for adjusting the fabrics' wettability due to the alginate abundance of hydroxyl and carboxylic groups (providing additional sites for binding water) and its ability to preserve the desired moist microenvironment while reducing hornification that occurs during material drying [48]. Moreover, this natural anionic linear polysaccharide is environmentally friendly, biodegradable, non-toxic, cost-effective, and highly abundant in seaweed, especially in brown algae.

The successful coating of jute fabrics with alginate (fabrics containing “+ AL” in their codes) was confirmed by SEM and ATR-FTIR spectroscopy. Non-cellulosic components were absent on the J + AL surface because they were partially removed (by fabric boiling in water) before coating with alginate. J surface is covered by a homogeneously distributed thin net of alginate, Fig. 10. In the SEM photographs of oxidized jute fabrics, it is evident that alginate is present as a thicker layer like a curtain of biopolymer (J0.2/120 + AL), or it fills the fiber

structure (J0.4/120 + AL).

The changes in the fabric surface chemistry upon coating with alginate were well-documented by ATR-FTIR spectroscopy. In Fig. 11, the spectra of J and J0.4/120 are compared before and after coating with alginate. Additionally, to easily discuss the changes in bands' intensities and positions, the FTIR spectrum of sodium alginate was recorded. It displayed typical bands at  $3215\text{ cm}^{-1}$  (originating from O–H stretching vibrations), and at  $1595$  and  $1411\text{ cm}^{-1}$  (assigned to  $\text{COO}^-$  asymmetric and symmetric stretching vibrations, respectively [49]). Alginate

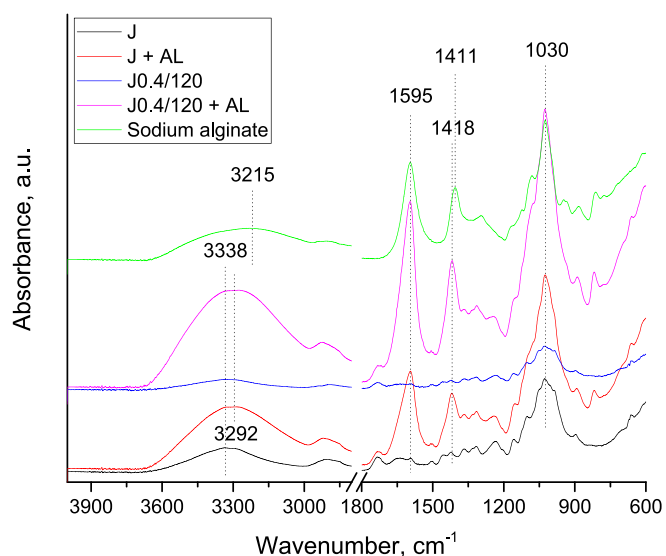


Fig. 11. ATR-FTIR spectra of sodium alginate and selected jute fabrics before and after coating with alginate.

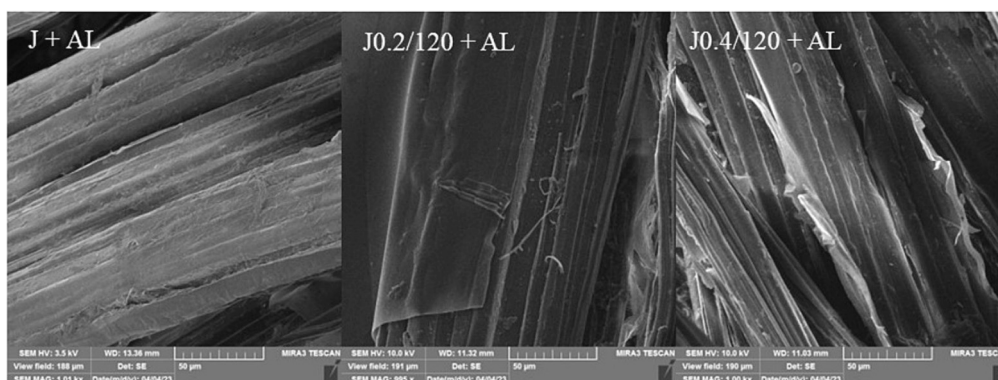


Fig. 10. SEM photographs of jute coated with alginate.



coating of J and J0.4/120 induces notable changes in their FTIR spectra. Specifically, a band at  $3338\text{ cm}^{-1}$  (ascribed to the absorbance of hydrogen-bonded O—H stretching in J and J0.4/120) becomes intense and shifts to a lower wavenumber ( $3292\text{ cm}^{-1}$ ) in the spectra of J + AL and J0.4/120 + AL. Both mentioned spectra show an appearance of significant arising of the dominant band at  $1595\text{ cm}^{-1}$  derived from the vibrations of the alginate  $\text{COO}^-$  groups, Fig. 11. Bands ascribed to symmetric  $\text{COO}^-$  vibrations are shifted from  $1411\text{ cm}^{-1}$  (AL) to  $1418\text{ cm}^{-1}$  in the spectra of the J + AL and J0.4/120 + AL due to the replacement of the  $\text{Na}^+$  ions of sodium alginate with  $\text{Ca}^{2+}$  ions of calcium chloride, which was added for crosslinking purposes [50]. Further, the band at  $1030\text{ cm}^{-1}$ , corresponding to C—O—C pyranose ring vibration, becomes more intense and sharper upon alginate coating. On the other hand, the characteristic bands coming from the lignin moiety of jute fabrics (positioned at  $1230, 1425, 1455, 1500,$  and  $1508\text{ cm}^{-1}$ ) show no significant changes after alginate coating. This suggests that lignin functional groups do not participate in the coating process and that coating of jute fabric with alginate originates solely from the interaction with cellulose, the main component of jute fabric. The absorption band of the carbonyl groups at  $1735\text{ cm}^{-1}$  and the hemiacetal vibration at  $875\text{ cm}^{-1}$  inherent to the formation of the dialdehyde group in oxidized jute fabric (J0.4/120) do not show significant changes upon alginate treatment indicating that these groups do not participate in the interaction with alginate.

The reason for introducing an additional step, i.e., oxidation, before fabric coating with alginate is to increase the negative fabric zeta potential which can “force” the alginate molecules to orient themselves in a way that is most suitable for establishing jute-alginate interactions, thereby promoting effective alginate coating. As is given in Fig. 7, the minimum zeta potential value of raw fabric is  $-8.57$ , while this electrokinetic parameter of oxidized fabrics ranges between  $-18.3$  and  $-20.12$ . The highly negatively charged surfaces of oxidized jute fabrics can strongly repel the negatively charged alginate carboxylate groups orienting them away from the jute surface and making available for further crosslinking, as depicted in Fig. 12. After treatment with  $\text{CaCl}_2$ , these carboxylate groups form an egg-box structure due to the  $\text{Ca}^{2+}$  mediated crosslinking process through the formation of chelates [51]. On the other hand, the OH groups of alginate align themselves towards the jute fabric's surface and establish hydrogen bonds with the OH groups of cellulose (Fig. 12) facilitating effective coating. When considering oxidized jute fabrics, their higher hydrophilicity (as

indicated by wetting time) can be attributed to the more negatively charged surface compared to the raw jute (Fig. 7), allowing the alginate chains to orient in a more favorable way for coating.

After characterizing the alginate-coated fabrics, their wetting times and moisture sorption values were determined and compared with those before the treatment, Table 2. Surprisingly, fabrics' J0.2/60 + AL and J0.2/120 + AL have about 350 and 660 times shorter wetting times than J0.2/60 and J0.2/120, respectively. Just to note that before coating, J0.2/60 and J0.2/120 have wetting times that are 2.9 and 5.3 times longer than raw jute (J). This trend reverses after the coating with alginate; about 2.0 times shorter wetting times were measured for J0.2/60 + AL and J0.2/120 + AL compared to J + AL. Among the group of alginate-coated fabrics, the shortest wetting time (3.96 s) is observed for J0.4/120 + AL, which is 2.2 times shorter than that of J + AL, Table 2. Such changes are undoubtedly related to the fabric's surface polarity and morphology (Fig. 10), which promote the quick and easy penetration and adsorption of water drops within the alginate-coated fabrics.

Moisture sorption values of all studied fabrics were also increased after coating with alginate. Fabrics J + AL, J0.2/60 + AL, J0.2/120 + AL, J0.4/60 + AL, and J0.4/120 + AL have 17.8, 7.9, 11.5, 23.5, and 24.9 % higher moisture sorption values compared to their counterparts before coating. The obtained differences before and after coating are statistically significant, Supplementary material, Table S5. The findings of this section support the second hypothesis that coating with alginate enhances the oxidized jute fabrics' moisture sorption and wettability. Also, the presented results are very precious since the oxidized jute fabrics coated with alginate fulfill the most important condition to be used as water-binding geo-prebiotic polysaccharide supports providing a new value and moving the jute fabrics' application from conventional to non-conventional areas.

The major innovation in our overall work with biocarpet is related to proposing an innovative (bio)technological solution: biocarpet engineering, which ensures the continuity and sustainability of life under various conditions. The synergistic association and interaction [52] between the substrate, polysaccharide-based support (i.e., jute fabric), and microorganisms (e.g. cyanobacteria) could provide an eco-friendly and cost-efficient method for the long-term rehabilitation and development of damaged terrains affected by anthropogenic activities (mining, traffic, etc.) and aridization/desertification. Cyanobacterial crusts, as primary colonizers, were proposed by Wei [53] as a potential solution for such environmental issues but it is well known that the proper

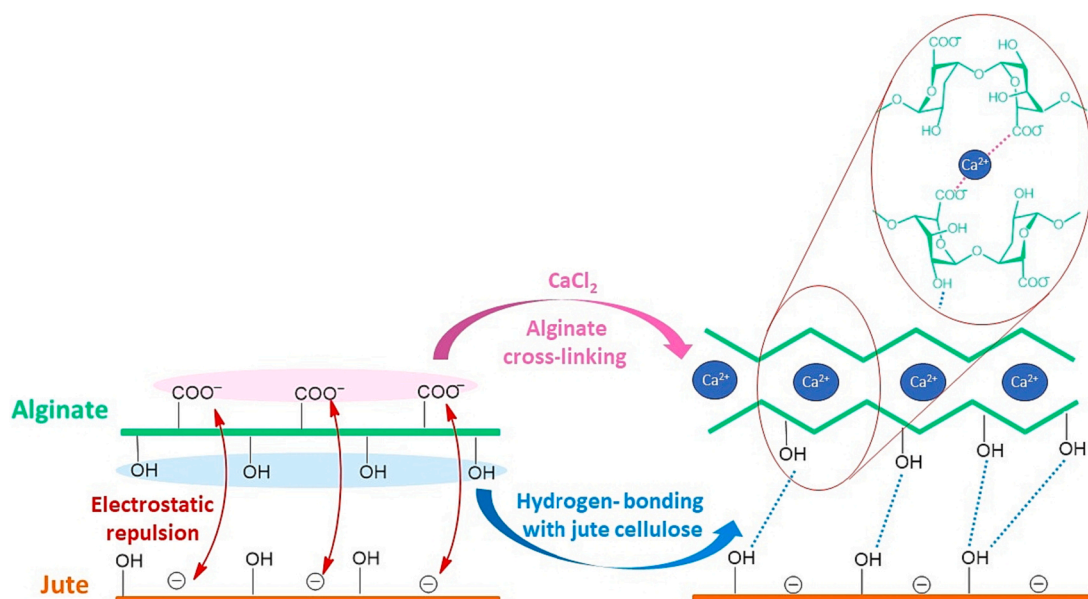


Fig. 12. Putative mechanism of coating jute fabrics with alginate.

growth of the initial inoculum requires more moisture than is typically available in arid and semi-arid environments [54].

The support developed in this paper has the potential to promote the growth and survival of cyanobacteria and other microorganisms that can be used for the rehabilitation of various degraded terrains by providing a water-rich geo-prebiotic microenvironment. Once the carpet is spread on lifeless terrain, the cyanobacteria will have enough minerals and moisture to begin reproduction and they will grow as the primary colonizers and pioneer organisms. As they create a synergistic relationship with the abiotic environment [52], they will create the conditions for the germination of fungi, lichens, mosses, and grass, eventually creating a sustainable ecosystem and conditions for higher organisms. The applicative value of cyanobacterial biocarpet engineering could be demonstrated by the restoration of lifeless surfaces, prevention of soil erosion leading to a reduction of air and water pollution, rehabilitation of deserts and damaged loess surfaces, and bioremediation of polluted areas. These activities would restore and enhance biodiversity, and promote agriculture and ecological balance. Our idea will not only support the above-mentioned services but it will introduce an inoculum (a starter community of organisms) that promotes life in all types of extreme conditions, including those prevailing after big catastrophes of any origin.

#### 4. Conclusion

This is the first study to provide evidence of tuning the jute structure and properties using selective oxidation with sodium periodate and coating with alginate. Raw jute fabric was treated with  $\text{NaIO}_4$  to oxidize cellulose secondary OH to CHO groups. As the  $\text{NaIO}_4$  concentration and/or reaction time increased, the CHO group content within oxidized fabrics increased by 28.8–114.7 % compared to the raw jute. Sodium periodate oxidation was accompanied by changes in fabric surface chemistry and a slight disruption of the fiber crystallinity resulting in improved accessibility of functional groups. The last one in combination with group dissociation contributed to an increase in the negative zeta potential of oxidized fabrics. With increasing oxidation severity, the maximum force and stiffness of the oxidized fabrics decreased by 6.1–37.5 %, and 27.9–49.8 % respectively. Oxidized fabrics exhibited 22.3–29.6 % higher ability for moisture sorption and very long wetting times, which can be advantageous for applications as packaging materials.

To further extend jute utilization in non-conventional biocarpet engineering, raw and oxidized hydrophobic fabrics were transformed into hydrophilic ones by coating with alginate. Fabrics treated with 0.2 %  $\text{NaIO}_4$  for 60 and 120 min and coated with alginate had about 350 and 660 times shorter wetting times than their oxidized counterparts. Additionally, after coating with alginate, all jute fabrics exhibited up to 24.9 % higher moisture sorption values. The study successfully verifies its first hypothesis concerning the effects of oxidation on jute structure and properties and the second hypothesis regarding the transformation of hydrophobic to hydrophilic jute fabrics through alginate coating.

The experiments in this paper were performed on real, commercially produced fabric, which is advantageous from the point of further application, i.e., obtaining hydrophobic and hydrophilic jute fabrics and moving their application from conventional to non-conventional areas. Another advantage is that the same oxidation protocol is particularly relevant and can be applied for tuning the structure and properties of other lignocellulosic fibers with similar chemical compositions as jute fabric used in this study.

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#### CRediT authorship contribution statement

**Aleksandra Ivanovska:** Conceptualization, Formal analysis, Investigation, Methodology, Visualization, Writing – original draft. **Marija Milošević:** Conceptualization, Investigation, Methodology, Visualization, Formal analysis. **Jelena Lađarević:** Formal analysis, Investigation, Visualization, Writing – original draft, Writing – review & editing. **Maja Jankoska:** Formal analysis, Investigation. **Tamara Matić:** Formal analysis, Investigation, Writing – review & editing. **Zorica Svirčev:** Funding acquisition, Writing – original draft, Writing – review & editing. **Mirjana Kostić:** Conceptualization, Supervision, Writing – review & editing.

#### Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

#### Data availability

Data will be made available on request.

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#### Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.ijbiomac.2023.128668>.

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