

SELECTIVE PERIODATE OXIDATION OF C6-CARBOXYCELLULOSE TO C6-CARBOXY-2,3-DIALDEHYDE CELLULOSE: PROCESS OPTIMIZATION AND STRUCTURAL CHARACTERIZATION

Bakhriddin .Z. Kholturaev

*Institute of Polymer Chemistry and Physics, Academy of Sciences of the Republic of Uzbekistan,
Tashkent 100128, Uzbekistan*

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Corresponding author

bakhriddinjumayevich@gmail.com

ABSTRACT

In this study, the selective oxidation of C6-carboxycellulose with sodium periodate (NaIO_4) to obtain C6-carboxy-2,3-dialdehyde cellulose was investigated. The effects of NaIO_4 concentration and reaction duration on the formation of aldehyde groups and product yield were evaluated. Increasing the oxidant concentration and reaction time promoted aldehyde group formation; however, excessive oxidation conditions resulted in a decrease in product recovery. The optimal oxidation conditions were established at 2.5% NaIO_4 , pH 3.0-3.5, 25-30 °C, and a reaction time of 20 min. Under these conditions, the aldehyde group content reached 6.0 mmol/g, while the product yield was maintained at 80%.

FTIR analysis confirmed the formation of aldehyde-containing structures resulting from selective oxidation of the vicinal hydroxyl groups at the C2 and C3 positions, while XRD analysis revealed a decrease in crystalline ordering and an increase in amorphous regions after oxidation. SEM observations demonstrated pronounced morphological changes, including increased surface roughness, heterogeneous structural features, and partial disruption of the fibrillar organization of cellulose after periodate treatment. Nevertheless, preservation of the general fibrillar framework indicated controlled oxidation without complete destruction of the cellulose structure. The obtained results demonstrate that optimization of periodate oxidation parameters enables the preparation of C6-carboxy-2,3-dialdehyde cellulose with a balanced combination of aldehyde functionality, structural preservation, and potential applicability as a reactive cellulose-based intermediate for further functional material development.

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Introduction

The presence of hydroxyl groups with different reactivities in the cellulose macromolecule makes it a promising raw material for the preparation of multifunctional polymer derivatives through sequential selective chemical transformations. Targeted modification of the secondary hydroxyl groups located at the C2 and C3 positions and the primary hydroxyl group at the C6 position of the anhydroglucose unit enables the introduction of new reactive sites into the cellulose structure [1,2]. This approach makes it possible to regulate the hydrophilicity, sorption capacity, ion-exchange properties, and reactivity of the polymer toward subsequent chemical transformations while preserving its cellulosic backbone. [1-3].

Selective oxidation methods are of particular importance in the functionalization of cellulose. By selecting an appropriate oxidizing system and reaction conditions, hydroxyl groups can be converted into aldehyde or carboxyl groups at specific positions. For example, selective oxidation of the primary hydroxyl group at the C6 position results in the formation of cellulose derivatives bearing carboxyl functionalities [2,4]. The introduction of carboxyl groups enhances

the ionogenic character of the macromolecule and broadens its ability to interact with various cations and functional compounds [1,4].

For the functionalization of cellulose at the C2 and C3 positions, periodate oxidation is considered one of the most effective selective methods [3, 5-7]. The distinctive feature of sodium periodate is its ability to selectively oxidize adjacent hydroxyl groups in the anhydroglucose unit. During the reaction, the periodate ion forms an intermediate cyclic complex with the vicinal diol moiety at the C2 and C3 positions. Decomposition of this complex is accompanied by cleavage of the C2-C3 carbon-carbon bond and the formation of aldehyde groups at both positions [3, 5]. As a result, reactive 2,3-dialdehyde cellulose structures are formed.

The introduction of aldehyde groups into the cellulose macromolecule significantly expands its potential for further modification. These groups can participate in condensation reactions with amino-containing compounds, promote the formation of interpolymer linkages, and serve as reactive sites for attaching various functional moieties to the macromolecular chain [6,8,9]. Therefore, dialdehyde cellulose is regarded not only as an oxidized cellulose product but also as an important intermediate polymer system for the preparation of novel functional materials [3,6].

The subsequent periodate oxidation of cellulose in which the C6 position has already been converted into a carboxyl group is of particular interest. In this case, aldehyde groups can be formed at the C2 and C3 positions while preserving the C6-carboxyl functionality of the macromolecule. As a result, C6-carboxy-2,3-dialdehyde cellulose containing reactive sites of different chemical nature within the same macromolecular structure is formed. Such a structure enables the ionogenic properties of carboxyl groups and the high reactivity of aldehyde groups to be combined within a single polymer system.

Similar functional approaches have also been investigated for other cellulose derivatives containing carboxyl groups. In particular, it has been demonstrated that dialdehyde carboxymethyl cellulose can be obtained by oxidizing carboxymethyl cellulose with NaIO_4 and that the resulting aldehyde groups can be utilized in interpolymer crosslinking reactions [8,9]. These findings confirm that the simultaneous incorporation of carboxyl and aldehyde functional groups into cellulose is an effective approach for the development of novel functional materials.

The outcome of periodate oxidation is determined not only by the nature of the oxidizing agent but also by its concentration, reaction duration, temperature, medium pH, and the initial structure of cellulose [3,5-7]. Changes in the reaction conditions directly affect the degree of conversion of the C2 and C3 hydroxyl groups into aldehyde groups. Generally, increasing the NaIO_4 concentration and reaction time enhances the degree of functionalization. However, an excessively high oxidant concentration or prolonged reaction duration may also induce unfavorable changes in the macromolecular and supramolecular structure of cellulose [3,5,11].

Because cleavage of the C2-C3 bond is an integral part of the periodate oxidation mechanism, increasing the degree of oxidation may intensify structural changes in the cellulose chain. This may increase the likelihood of a decrease in the degree of polymerization, partial disruption of the structure, and, consequently, a reduction in product yield [3,6,11]. Therefore, evaluation of the periodate oxidation process should not be based solely on achieving the maximum aldehyde content. It is important to determine the optimal balance between the degree of functionalization and the preservation of the macromolecular structure and product yield.

The functional-group-rich structure of oxidized cellulose derivatives enables their use in the preparation of sorption materials, interpolymer crosslinking reagents, functional films, and biologically active materials [8-12]. In particular, the ability of aldehyde groups to participate in subsequent reactions is important for the formation of multicomponent and covalently crosslinked polymer systems based on dialdehyde cellulose. From this perspective, the preparation of cellulose derivatives combining carboxyl and aldehyde functional groups and the control of their synthesis conditions are of both theoretical and practical significance.

However, during the periodate oxidation of C6-carboxycellulose, the ratio between NaIO_4 concentration and reaction duration must be optimized so that a sufficient amount of aldehyde

groups is formed at the C2 and C3 positions while maintaining a high product yield. Therefore, experimental substantiation of the oxidation conditions for this system and determination of the optimal balance between the degree of functionalization and product yield represent an important research objective.

Accordingly, the aim of this study was to obtain C6-carboxy-2,3-dialdehyde cellulose by periodate oxidation of C6-carboxycellulose in the presence of NaIO₄ and to evaluate the effects of NaIO₄ concentration and reaction duration on the aldehyde group content and product yield. In addition, the main objective of the study was to determine the reaction conditions that provide an optimal balance between aldehyde functionality and product yield.

The present study focuses on the selective introduction of aldehyde groups at the C2 and C3 positions of C6-carboxycellulose while retaining the C6-carboxyl functionality, with particular emphasis on identifying oxidation conditions that provide a favorable balance between aldehyde content and product yield.

2. Materials and Methods

2.1. Materials

C6-carboxycellulose was used as the starting cellulose derivative for the preparation of C6-carboxy-2,3-dialdehyde cellulose. Sodium periodate (NaIO₄, analytical grade, CAS No. 7790-28-5) was used as an oxidizing reagent for the selective conversion of vicinal hydroxyl groups at the C2 and C3 positions of the anhydroglucose units into aldehyde functional groups. Distilled water (CAS No. 7732-18-5) was used for the preparation of NaIO₄ solutions and reaction mixtures, as well as for washing the oxidized cellulose samples. During the periodate oxidation process, the pH of the reaction medium was maintained within the range of 3.0-3.5. All reagents were of analytical grade and were used without further purification.

2.2. Preparation of C6-Carboxy-2,3-Dialdehyde Cellulose

C6-carboxy-2,3-dialdehyde cellulose was obtained by selective oxidation of C6-carboxycellulose using sodium periodate (NaIO₄). During the periodate oxidation process, the effects of NaIO₄ concentration and reaction duration on the conversion of vicinal hydroxyl groups at the C2 and C3 carbon atoms of the anhydroglucose units into aldehyde groups were investigated.

NaIO₄ solutions with concentrations of 1.5, 2.5, and 3.5% were used in the experiments. The oxidation process was carried out at pH 3.0-3.5 and a temperature of 25-30 °C. For each oxidant concentration, the reaction duration was set at 10, 15, 20, and 25 min. The aldehyde group content and product yield of the obtained samples were determined, and the efficiency of the oxidation conditions was evaluated based on these parameters.

Based on the experimental results, 2.5% NaIO₄, pH 3.0-3.5, 25-30 °C, and a reaction duration of 20 min were selected as the optimal conditions for the periodate oxidation of C6-carboxycellulose. Under these conditions, the aldehyde group content was 6.0 mmol/g, while the product yield was 80%.

2.3. Determination of Product Yield

The recovery of C6-carboxy-C2,C3-dialdehyde cellulose after periodate oxidation was evaluated gravimetrically using an approach adapted from Han et al. [13]. At the end of each oxidation experiment, the solid cellulose derivative was separated from the reaction medium and thoroughly washed to eliminate residual oxidizing agent and soluble reaction products. The recovered material was subsequently dried until a constant mass was obtained.

$$Y (\%) = m_1/m_0 * 100 \quad (1)$$

where (Y) represents the product yield (%), (m₁) is the dry mass of C6-carboxy-C2,C3-dialdehyde cellulose recovered after oxidation (g), and (m₀) is the initial dry mass of C6-carboxycellulose introduced into the reaction (g).

2.4. Determination of Aldehyde Group Content

The concentration of aldehyde functionalities introduced into C6-carboxycellulose was quantified by the hydroxylamine hydrochloride titration procedure, with adaptation of previously

reported protocols [7,14]. A 0.10 g portion of the dried oxidized cellulose sample was dispersed in distilled water, followed by the addition of 25 mL of 0.25 M hydroxylamine hydrochloride ($\text{NH}_2\text{OH}\cdot\text{HCl}$). The suspension was adjusted to pH 4.0 and maintained under continuous stirring at room temperature to allow the aldehyde functionalities to react with hydroxylamine.

Reaction of the carbonyl groups with hydroxylamine generates the corresponding oxime functionalities and releases an equivalent amount of hydrochloric acid. To correct for the contribution of the starting material, an unoxidized C6-carboxycellulose sample was treated in parallel under identical experimental conditions.

After completion of the reaction, the acidity generated in the oxidized and reference systems was determined by titration with a standardized NaOH solution until the initial pH of 4.0 was re-established. The aldehyde content was calculated from the difference between the NaOH volumes required for the oxidized and reference samples:

$$A = C_{\text{NaOH}} (V_s - V_b) / m \quad (2)$$

where (A) is the aldehyde group content (mmol/g), (C_{NaOH}) is the molar concentration of the standardized NaOH solution (mol/L), (V_s) is the NaOH volume consumed by the oxidized sample (mL), (V_b) is the corresponding volume for the unoxidized reference sample (mL), and (m) is the dry mass of the sample used for analysis (g).

2.5. FTIR Analysis

Fourier-transform infrared spectroscopy was used to monitor the chemical changes induced by periodate oxidation. Spectra of the initial C6-carboxycellulose and the resulting C6-carboxy-2,3-dialdehyde cellulose were collected with an INVENIO-S FTIR spectrometer (Bruker, Germany) using the KBr pellet technique. Measurements were performed within the $500\text{--}4000\text{ cm}^{-1}$ spectral range following a previously reported procedure with minor experimental adaptation [14]. Particular attention was given to variations in the hydroxyl, carbonyl, carboxylate, and cellulose fingerprint regions in order to assess the structural consequences of NaIO_4 treatment.

2.6. X-ray Diffraction Analysis

Changes in the ordered structure of cellulose caused by periodate oxidation were examined by X-ray diffraction. Diffraction patterns of C6-carboxy-2,3-dialdehyde cellulose were obtained using a Rigaku MiniFlex 600 diffractometer equipped with a $\text{Cu K}\alpha$ radiation source. The measurements were carried out over a 2θ interval of $5\text{--}40^\circ$ at a scanning rate of 2° min^{-1} , with the X-ray generator operated at 22 kV and 12 mA, based on the analytical procedure reported previously [15]. Structural changes were assessed by comparing the location, relative intensity, and broadening of the principal diffraction reflections of the two samples.

2.7. Scanning Electron Microscopy (SEM) Analysis

The surface morphology of C6-carboxycellulose and C6-carboxy-2,3-dialdehyde cellulose samples was analyzed using a JEOL JSM-IT200 scanning electron microscope (JEOL Ltd., Japan) at an accelerating voltage of 5-10 kV. Prior to analysis, the samples were dried, mounted on conductive carbon tape, and coated with a thin conductive layer to reduce charging effects. SEM images were obtained at different magnifications to evaluate changes in surface morphology, fibrillar structure, and structural integrity after NaIO_4 oxidation.

3. Results and Discussion

The extent of periodate oxidation of C6-carboxycellulose was evaluated as a function of NaIO_4 concentration and reaction time. Aldehyde content was used as an indicator of functionalization, whereas product recovery was considered as a measure of material preservation during oxidation. The experimental results obtained under the investigated conditions are summarized in Table 1.

Table 1

Effect of NaIO₄ concentration and reaction time on aldehyde group content and product yield during periodate oxidation of C6-carboxycellulose

Sample	NaIO ₄ , %	pH of the medium	Reaction duration, min	Temperature, °C	Aldehyde group content, mmol/g	Product yield, %
C6-carboxycellulose sample	1.5	3.0-3.5	10	25-30	2.5	95
			15		3.8	91
			20		4.9	87
			25		5.3	82
	2.5		10		3.7	90
			15		4.6	86
			20		6.0	80
			25		6.2	71
	3.5		10		3.9	78
			15		4.8	72
			20		6.2	65
			25		6.4	60

At a NaIO₄ concentration of 1.5%, increasing the reaction duration from 10 to 25 min increased the aldehyde group content from 2.5 to 5.3 mmol/g, corresponding to an approximately 2.1-fold increase. At the same time, the product yield decreased from 95% to 82%. Under these conditions, the oxidation proceeded relatively mildly and a high product yield was maintained; however, the amount of aldehyde groups formed was lower than the values obtained at higher NaIO₄ concentrations.

When the NaIO₄ concentration was increased to 2.5%, the oxidation process proceeded more efficiently. In particular, the aldehyde group content was 3.7 mmol/g after 10 min and increased to 6.0 mmol/g after 20 min. Over the same time interval, the product yield decreased from 90% to 80%. Extending the reaction duration from 20 to 25 min increased the aldehyde group content only slightly, from 6.0 to 6.2 mmol/g, whereas the product yield decreased from 80% to 71%. Thus, oxidation beyond 20 min intensified product loss without a substantial increase in the degree of functionalization.

At a NaIO₄ concentration of 3.5%, the highest aldehyde group contents were recorded. When the reaction duration was increased from 10 to 25 min, the aldehyde group content increased from 3.9 to 6.4 mmol/g, while the product yield decreased from 78% to 60%. At 20 min, increasing the NaIO₄ concentration from 2.5% to 3.5% increased the aldehyde content only from 6.0 to 6.2 mmol/g, whereas the product yield decreased markedly from 80% to 65%. These results indicate that increasing the NaIO₄ concentration from 2.5% to 3.5% provides no substantial advantage in the degree of functionalization, while significantly increasing product loss.

Comparison of the obtained results showed that, with increasing NaIO₄ concentration and reaction duration, aldehyde group formation proceeded rapidly up to a certain point, after which the rate of increase slowed. At the same time, a pronounced decrease in product yield was observed at higher oxidant concentrations and prolonged reaction times. This behavior is consistent with increased material loss under more severe oxidation conditions and may also reflect progressive structural degradation of the cellulose matrix.

Within the investigated experimental range, 2.5% NaIO₄, 20 min, pH 3.0-3.5, and 25-30 °C provided the most favorable balance between aldehyde functionalization and product yield. Under these conditions, the aldehyde group content reached 6.0 mmol/g, while the product yield remained at 80%.

FTIR analysis was employed to evaluate the structural changes induced by periodate oxidation of C6-carboxycellulose. The corresponding spectra are presented in Figure 1.

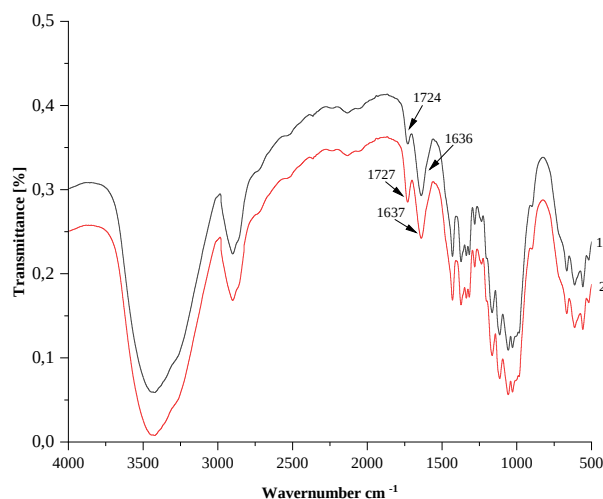


Figure 1. FTIR spectra of C6-carboxycellulose (1) and C6-carboxy-2,3-dialdehyde cellulose (2) samples

The FTIR profiles of C6-carboxycellulose and C6-carboxy-2,3-dialdehyde cellulose are compared in Figure 1. Both spectra retain the principal absorption features associated with the cellulose backbone, whereas distinct changes appear after treatment with sodium periodate.

A broad absorption envelope within 3200-3500 cm^{-1} is associated predominantly with O-H stretching vibrations and reflects the extensive hydrogen-bonding network characteristic of cellulose. The absorptions observed at approximately 2850-2950 cm^{-1} are attributed mainly to C-H stretching vibrations of the polysaccharide framework.

The most relevant spectral changes following NaIO_4 treatment occur in the carbonyl region, particularly between 1700 and 1750 cm^{-1} . These changes are consistent with conversion of the vicinal hydroxyl functionalities at the C2 and C3 positions into aldehyde-containing structures as a consequence of periodate-mediated cleavage of the C2-C3 unit. Consequently, modification of the carbonyl absorption profile provides spectroscopic evidence for the formation of C6-carboxy-2,3-dialdehyde cellulose.

The aldehyde-related C=O absorption does not appear as an exceptionally intense isolated band. This behavior can be associated with the well-known tendency of aldehyde functionalities in oxidized cellulose to undergo hydration and to participate in intra- and intermolecular hemiacetal formation. Such equilibria reduce the fraction of aldehyde groups present exclusively in the free carbonyl form.

Additional modifications observed in the approximately 1580-1630 cm^{-1} interval and throughout the 1200-900 cm^{-1} fingerprint region suggest alterations in the local environment of carboxylate functionalities and in the C-O-C/C-O vibration pattern. Taken together, the FTIR results are consistent with modification of the C2-C3 positions while indicating retention of the principal cellulose framework after oxidation.

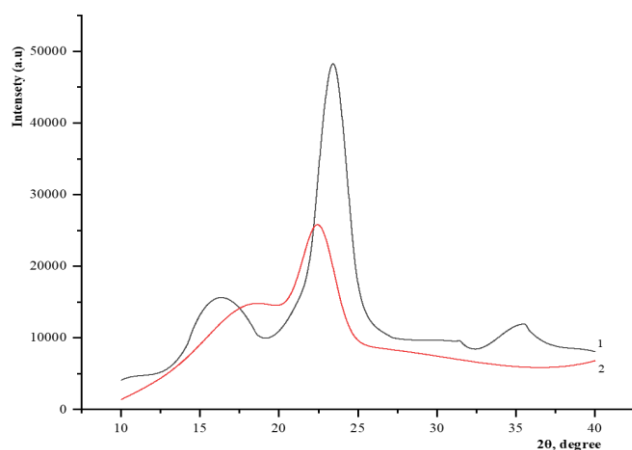


Figure 2. X-ray diffraction (XRD) patterns of C6-carboxycellulose (1) and C6-carboxy-2,3-dialdehyde cellulose (2) samples

Figure 2 compares the XRD patterns of the starting C6-carboxycellulose and the corresponding C6-carboxy-2,3-dialdehyde cellulose obtained after NaIO_4 treatment. The diffraction profile of C6-carboxycellulose contains prominent reflections in the vicinity of $2\theta \approx 16^\circ$ and $22-23^\circ$, which are associated with ordered domains characteristic of cellulose I. In particular, the strong reflection centered around $22-23^\circ$ indicates the persistence of a substantial crystalline fraction in the starting material.

Following periodate oxidation, clear modifications in the diffraction pattern are observed. The principal reflection becomes substantially less intense and broader, while the feature around 16° is markedly weakened. Minor reflections at higher 2θ values are also diminished or no longer clearly distinguishable. These observations indicate that the level of structural ordering decreases during conversion of C6-carboxycellulose into the dialdehyde derivative.

These changes are consistent with structural disruption expected from periodate oxidation of vicinal hydroxyl groups at the C2 and C3 positions, which involves cleavage of the C2-C3 bond and local rearrangement of the anhydroglucose structure.

Consequently, the oxidized material contains a higher proportion of disordered or amorphous regions than the initial C6-carboxycellulose. Nevertheless, the persistence of the main diffraction feature near $22-23^\circ$ shows that periodate oxidation under the selected conditions does not eliminate all ordered cellulose domains. Thus, the XRD data indicate partial disruption rather than complete loss of the initial supramolecular organization.

The surface morphology of C6-carboxycellulose before and after periodate oxidation was investigated using scanning electron microscopy (SEM), and the obtained results are presented in Figure 3. SEM images showed that significant morphological changes occurred in C6-carboxycellulose (1) and C6-carboxy-2,3-dialdehyde cellulose (2) samples after NaIO_4 oxidation.

Scanning Electron Microscopy (SEM) analysis

The surface morphology of C6-carboxycellulose before and after periodate oxidation was investigated using scanning electron microscopy (SEM). The SEM images of the initial C6-carboxycellulose and C6-carboxy-2,3-dialdehyde cellulose samples are presented in Figure 3.

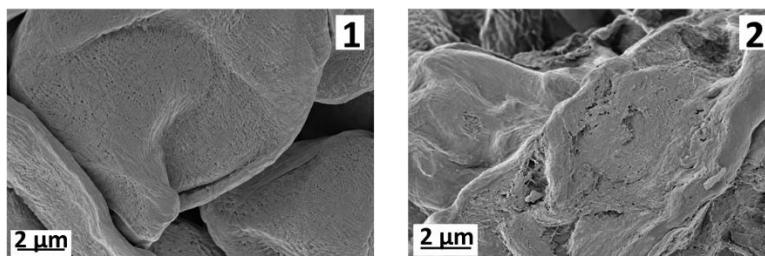


Figure 3. SEM images of C6-carboxycellulose (1) and C6-carboxy-2,3-dialdehyde cellulose (2) samples

The initial C6-carboxycellulose exhibited a relatively compact and continuous fibrillar structure, with a uniform surface morphology and limited structural deformation. After oxidation, the C6-carboxy-2,3-dialdehyde cellulose sample showed increased surface roughness, morphological heterogeneity, and partial disruption of the fibrillar structure.

The observed changes can be attributed to the cleavage of C2-C3 bonds in the anhydroglucose units under the action of NaIO_4 and the subsequent rearrangement of cellulose chains. The formation of aldehyde groups at these positions reduces the regularity of intermolecular interactions and promotes partial loosening of the fibrillar network.

These morphological transformations are consistent with the XRD results, which demonstrated a decrease in crystalline ordering and an increase in amorphous regions after oxidation. However, preservation of the general fibrillar framework indicates that the selected oxidation conditions enabled controlled functionalization without complete degradation of the cellulose structure.

Conclusion

Periodate oxidation of C6-carboxycellulose using NaIO_4 was investigated to obtain C6-carboxy-2,3-dialdehyde cellulose with controlled aldehyde functionalization. The results demonstrated that NaIO_4 concentration and reaction duration strongly influenced both aldehyde group formation and product yield. The optimal oxidation conditions were determined as 2.5% NaIO_4 , pH 3.0-3.5, 25-30 °C, and 20 min, under which the aldehyde group content reached 6.0 mmol/g with a product yield of 80%.

FTIR analysis confirmed the introduction of aldehyde functionalities through selective oxidation of the C2-C3 vicinal hydroxyl groups, while maintaining the characteristic cellulose backbone. XRD analysis showed partial disruption of the ordered cellulose structure, expressed by reduced diffraction peak intensity and increased contribution of amorphous regions. SEM analysis further confirmed these structural changes by revealing increased surface roughness, morphological heterogeneity, and partial disruption of the fibrillar organization after oxidation. At the same time, the preservation of the overall fibrillar morphology indicated that the selected reaction conditions provided controlled modification rather than extensive degradation of the cellulose matrix.

Overall, the obtained C6-carboxy-2,3-dialdehyde cellulose combines carboxyl and aldehyde functionalities within a single cellulose-based structure, making it a promising reactive intermediate for the development of functional polymer materials, sorption systems, and chemically modified cellulose derivatives.

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