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PRODUCTION AND PROPERTIES OF OXIDATIVE-ORGANOSOLVENT

Iryna Trembus, Anna Haponiuk

Department of Ecology and Technology of Plant Polymers
Igor Sikorsky Kyiv Polytechnic Institute
37/4 Beresteyskyi Avenue, Kyiv, 03056, Ukraine
E-mail: anna_kpi.ihf@ukr.net (A.H.); tivkpi@gmail.com (I.T.).

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ABSTRACT

The article presents the results of research on the production of cellulose from non-wood plant raw material - Miscanthus giganteus stems - using the oxidative-organosolv pulping method. It was established that, in terms of the content of major components (cellulose and lignin), the raw material is comparable to wood, although it exhibits higher ash content and increased pentosan levels. Optimal delignification parameters in the acetic acid - water - hydrogen peroxide system at a temperature of 100°C and a duration of 180 min provide a cellulose yield of 57.92 % with a residual lignin content of 1.84 %. Morphological and fractional analysis revealed a decrease in fiber length after refining and an increase in the proportion of fine fractions. The experimental results substantiate the feasibility of using citric acid as a catalyst, with an optimal concentration of 0.5 % relative to the oven-dry weight of the raw material. It was determined that the residual concentration of peracetic acid after cooking is only 27 % of the initial level, which prevents the effective reuse of spent liquor in subsequent delignification of plant biomass. The obtained results confirm the potential of miscanthus as a promising raw material for pulp and paper production.

Keywords: Miscanthus giganteus, oxidative-organosolv pulping, chemical composition, fiber length, mechanical properties, spent liquor.

INTRODUCTION

The growing global demand for paper and paperboard products necessitates the continuous development of new environmentally friendly and resource-efficient technologies in the pulp and paper industry [1, 2]. An alternative to conventional methods for producing fibrous semi-finished products (FSP) is the application of oxidative-organosolv pulping processes for plant-based raw materials. These methods are characterized using more readily available and cost-effective chemical reagents. Hydrogen peroxide or nitric acid of varying concentrations is employed as the oxidizing agent in such delignification processes, while titanium oxide, sodium molybdate, sodium tungstate, and citric acid serve as catalysts. Organosolv technologies enable the pulping of plant biomass at temperatures below 100°C and under atmospheric pressure. Among the advantages of these delignification methods is the selective action of organic solvents towards lignin removal, which results in fibrous semi-products with higher polysaccharide yields [3 - 10].

At present, a wide variety of organosolv pulping modifications have been developed to produce cellulose from annual plants, particularly from Miscanthus stems. In terms of its properties, the resulting material is comparable to cellulose produced by conventional methods [8 - 14].

Miscanthus giganteus (Latin: *Miscanthus sinensis* forma *Giganteus*) is a perennial plant belonging to the Poaceae (grass) family. It possesses a set of unique properties and characteristics, including a high cellulose content, rapid growth rate, and high biomass yield.

Miscanthus giganteus, due to its distinctive properties and characteristics, has become an important resource in various industrial and environmental sectors. Its widespread application reflects the global trend toward the utilization of renewable resources and highlights its significance as an alternative to traditional materials such as wood [8, 15 - 17]. The plant has found application in various fields, including biofuel production, where it serves as an environmentally friendly alternative to fossil fuels. In the pulp and paper industry, Miscanthus is utilized due to its favorable chemical composition. The plant is also employed in the chemical industry [15]. *Miscanthus giganteus* is not merely a technical crop but a symbol of ecological and economic efficiency. Its cultivation and utilization across various sectors demonstrate how innovation and sustainable resource use can contribute to the development of a greener and more sustainable future economy [17].

Promising directions in the development of organosolv pulping technologies for plant biomass are primarily associated with the use of various process catalysts. This approach enables an increase in the cellulose content in the resulting solid residue. The literature reports the use of transition metal peroxo-complexes, such as sodium tungstate and sodium molybdate, as catalytic additives [18, 19]. To reduce the cost of cellulose production, Sokolovska et al. proposed the use of citric acid as a catalyst in the pulping process [20]. Citric acid is significantly less expensive compared to molybdenum- and tungsten-based compounds and is produced on a large industrial scale.

In the oxidative-organosolv pulping process, citric acid can facilitate the activation of the cooking reagent and promote effective interaction with cellulose. The catalytic properties of citric acid may enhance the reaction rate and ensure more efficient cellulose transformation. To achieve optimal results, it is essential to consider process parameters such as acid concentration, temperature, and reaction duration. Citric acid acts as a catalyst in these reactions, accelerating both the oxidation of cellulose by the oxidizing agent and the polymerization of citric acid itself. This contributes to improved cellulose solubility, reduced lignin content, and a higher cellulose yield from plant biomass.

Cybulska et. al. investigated the production of cellulose from agricultural residues using an oxidative organosolv system consisting of glacial acetic acid and water in a 75 : 25 volume ratio, with hydrogen peroxide serving as the oxidizing agent [5, 7, 12, 13]. Citric acid was introduced into the reaction system to enhance the efficiency of delignification. The efficiency of delignification was mainly determined by the process temperature and the duration of treatment. The resulting cellulose was characterized by low lignin content, high brightness, and correspondingly high mechanical strength properties.

EXPERIMENTAL

Miscanthus giganteus stems were used as the plant raw material in this study. The plants were cultivated in Zhytomyr Oblast at the Botanical Garden of Polissia National University and harvested in March 2023. After harvesting, the miscanthus stems were air-dried and mechanically chopped into particles with a size of 20 ± 5 mm. The prepared raw material was stored in a desiccator to prevent moisture variation and to ensure the stability of its chemical composition before the experimental studies.

Prior to delignification, the main chemical constituents of the miscanthus stems were determined, including cellulose, lignin, pentosans, ash content, and other parameters characterizing the suitability of the plant raw material to produce fibrous semi-finished products. The quantitative determination of the chemical composition was carried out according to standard analytical procedures [19]. The obtained results are presented in Fig. 1. Oxidative-organosolv delignification of miscanthus stems was carried out in a cooking liquor consisting of glacial acetic acid and water in a volumetric ratio of 75 : 25. Hydrogen peroxide was used as the oxidizing agent in an amount of 50 % based on the oven-dry weight of the raw material. The pulping process was performed in glass flasks under atmospheric pressure at a temperature of 100°C for 180 min. These conditions were selected to ensure effective lignin removal while preserving the carbohydrate fraction of the plant raw material.

To investigate the effect of the catalyst on the delignification process, citric acid was added to the cooking liquor in an amount of 0.25 - 2.0 % based on the oven-dry weight of the raw material. The efficiency of the process was evaluated by the yield of the solid residue and the residual lignin content in the obtained cellulosic semi-finished product. The cellulose yield was calculated as a percentage of the oven-dry weight of the

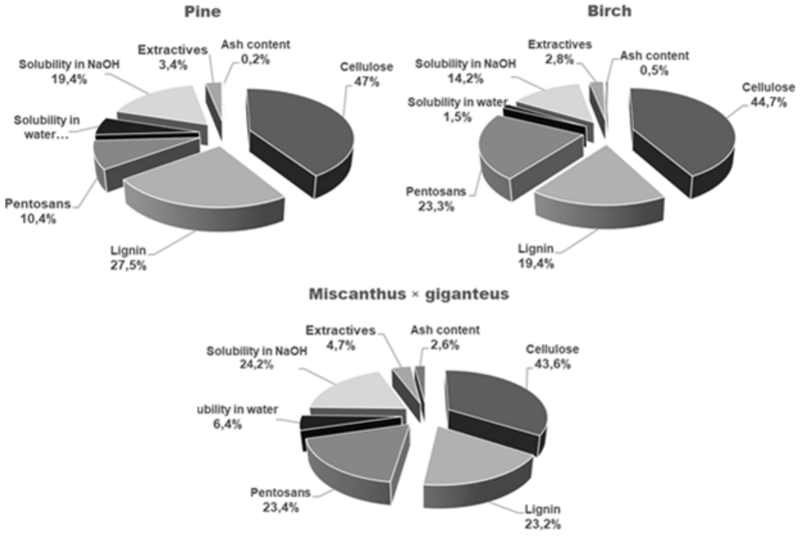

Fig. 1. Chemical composition of plant raw materials.

initial raw material, while the residual lignin content was determined as a percentage of the oven-dry weight of the cellulose.

After completion of pulping, the obtained solid residue was separated from the cooking liquor, thoroughly washed with water to a neutral reaction of the wash water, and dried to an air-dry state. The resulting cellulosic semi-finished product was further used for the determination of physico-mechanical properties, morphological analysis, and evaluation of the fractional composition of fibers.

All experiments were carried out under identical raw material preparation conditions and temperature-time regimes, which ensured the comparability of the obtained results and made it possible to assess the influence of the cooking liquor composition and catalyst concentration on the efficiency of delignification.

RESULTS AND DISCUSSION

Studies by Kim, Barbash, Cybulska and their co-authors have shown that the delignification of non-wood plant stems in peracid-based cooking media is characterized by high selectivity [8, 9, 13]. Taking this into account, an oxidative organosolv system containing acetic acid, water and hydrogen peroxide was selected for the processing of miscanthus stems intended for further production of filtration materials. The experimental results showed that cellulose could be isolated from miscanthus stems with a yield of 57.92 %, while the residual lignin content was 1.84 % based on oven-dry raw material.

For cellulose used in the preparation of laboratory filtration material samples, which operate mainly under wet conditions and increased pressure, the most important performance characteristics are wet strength and burst resistance. These parameters are largely governed by the intensity of interfiber bonding and the elastic properties of the fibers. The physico-mechanical properties of cellulose isolated from miscanthus stems are shown in Fig. 2 and compared with those of softwood kraft pulp, which is commonly used as a basic raw material for cellulosic membrane production.

The experimental results indicate that miscanthus-derived cellulose is lower than bleached softwood kraft pulp only in terms of wet strength, with a decrease of 33.3 %. However, it shows higher burst resistance and breaking length, exceeding the reference pulp by 32.5 % and 23 %, respectively. These results confirm the potential of miscanthus cellulose as a promising raw material for filtration media production.

The morphological structure of bleached softwood kraft pulp and miscanthus cellulose obtained via oxidative-organosolv pulping is presented in Fig. 3. The cellulose samples were refined to a freeness level of 92 ± 2°SR (where °SR denotes the Schopper-Riegler

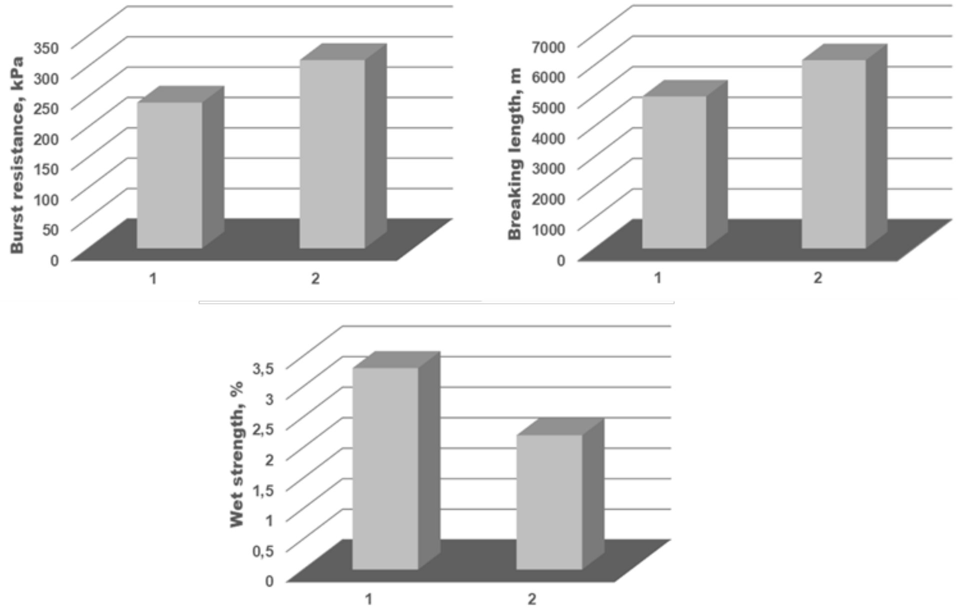

Fig. 2. Strength properties of cellulose: (1) bleached softwood kraft pulp; (2) oxidative-organosolv miscanthus cellulose.

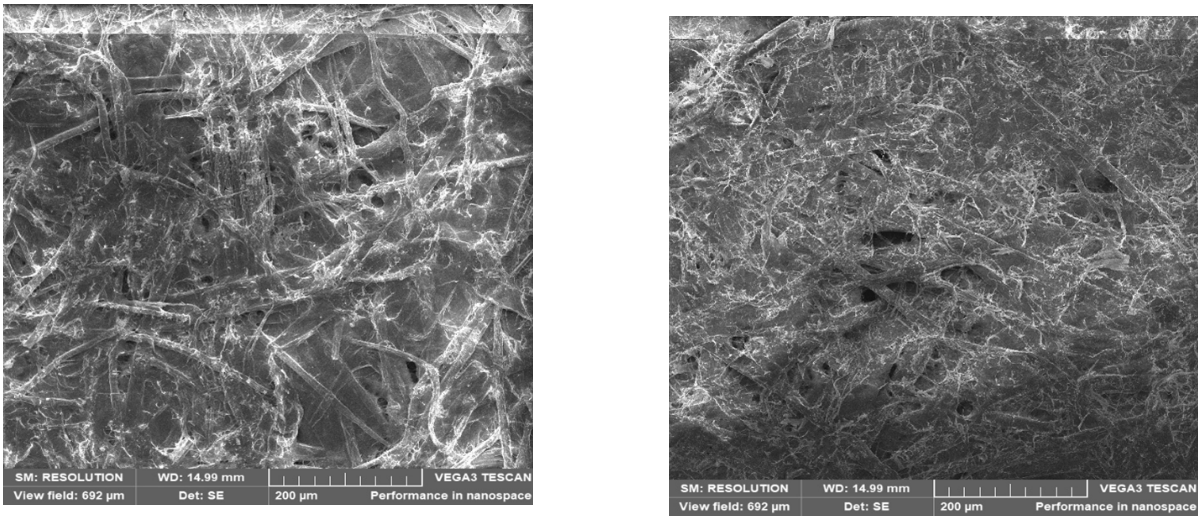

Fig. 3. SEM images of cellulose samples: (a) oxidative-organosolv miscanthus cellulose; (b) bleached softwood kraft pulp.

degree, a conventional index used to assess the degree of cellulose fiber refining). The presented images indicate that the use of different pulping conditions (chemical reagents, temperature, and pressure) leads to significant changes in fiber structure: fiber dimensions are reduced, and the uniformity of distribution is improved.

The primary components of the samples are xylem and phloem cells, which perform several functions: conduction, mechanical support, and storage. Miscanthus stems are composed predominantly of very short, oval-shaped cells. As a result of the delignification process, the cellulose derived from miscanthus consists of thin fibers of varying widths and lengths, which can form fiber bundles.

According to literature sources, oxidative-organosolv pulping reduces the fiber dimensions of plant raw materials by an average of 10 % [21 - 24]. Consequently, miscanthus fibers after delignification are reported to have an average length of 0.4 ± 0.28 mm. The cellulose samples also contain a significant amount of fines [25].

In contrast, softwood kraft pulp fibers after cooking have a fiber length of 0.24 ± 0.2 mm, but contain only 0.4 % fines [26]. Therefore, the structure of the cellulose matrix used for the production of filtration materials is expected to be denser when oxidative-organosolv miscanthus cellulose is used.

Fiber length analysis and fractional distribution by length were carried out for the investigated fibrous semi-finished product (FSP) samples. The measurements were performed using a Kajaani FS-200 analyser. The results (Fig. 4) confirmed previously reported literature data [25, 26]. Specifically, the average fiber length of oxidative-

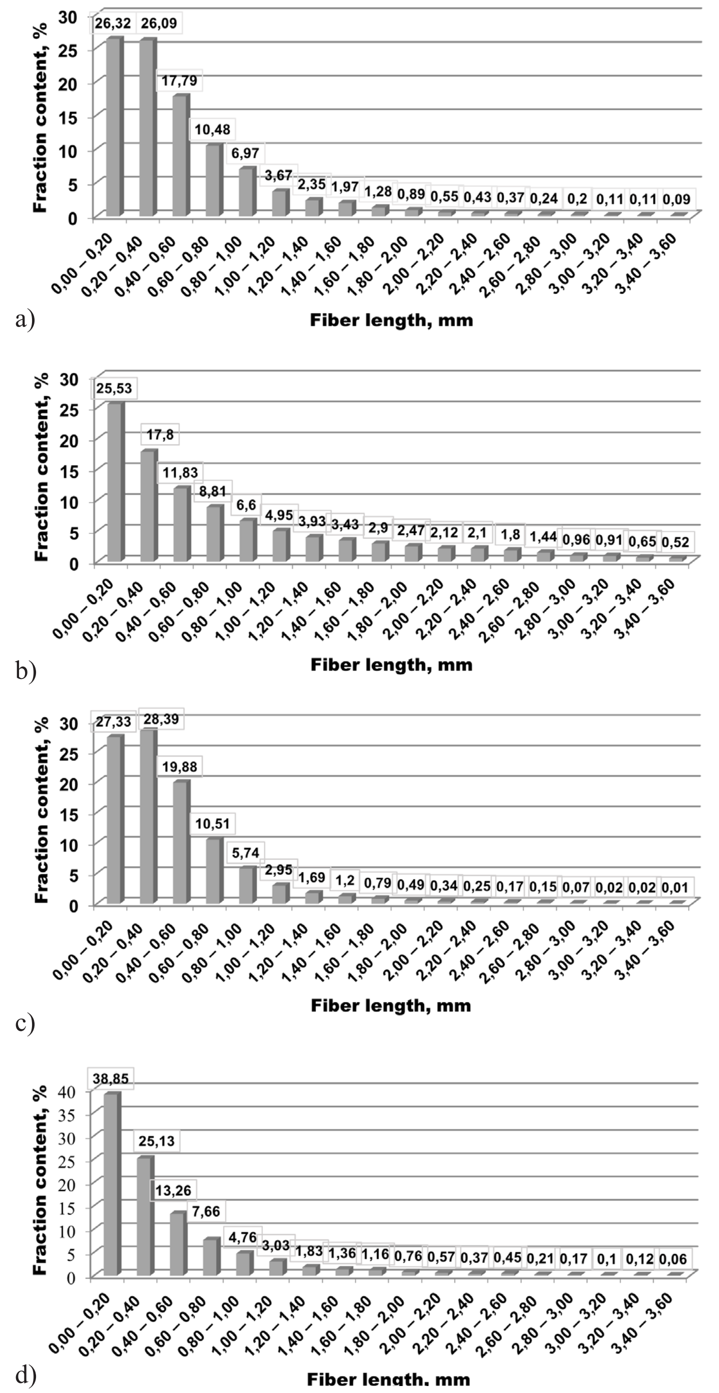

Fig. 4. Fractional composition of different types of cellulose: (a) unrefined oxidative-organosolv miscanthus cellulose; (b) unrefined softwood kraft pulp; (c) oxidative-organosolv miscanthus cellulose refined to 91°SR; (d) softwood kraft pulp refined to 91°SR.

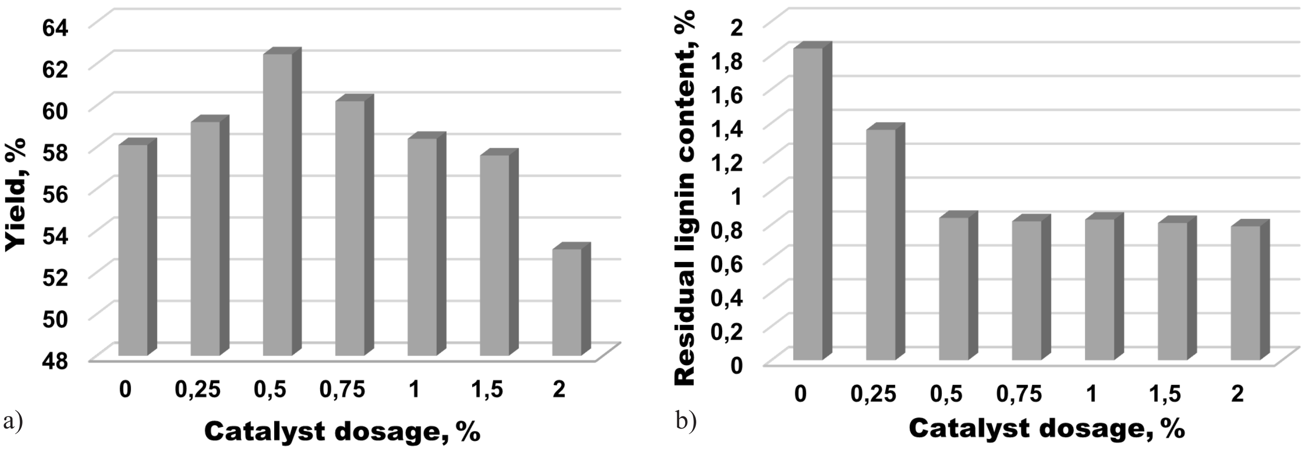

Fig. 5. Dependence of (a) yield and (b) residual lignin content of oxidative-organosolv miscanthus cellulose on catalyst dosage in the cooking liquor.

organosolv cellulose was 0.51 mm, with 51.41 % of the fibers belonging to the fine fraction (0.00 - 0.40 mm). After refining, the average fiber length decreased to 0.44 mm, and the fine fraction content increased to 55.72 %. Thus, refining miscanthus cellulose to a freeness of 90 ± 2°SR led to a 13.7 % reduction in fiber length and a 3.3 % increase in the proportion of fines.

For bleached softwood kraft pulp, the average fiber length was 0.82 mm for the unrefined sample and 0.43 mm after refining, indicating significant fiber shortening during the refining process. At the same time, the number of fines in bleached softwood kraft pulp was lower than that in oxidative-organosolv miscanthus cellulose by 9.08 % and 9.76 % for the unrefined and refined samples, respectively. Therefore, the cellulose material derived from miscanthus pulp has a denser structure and is more suitable for the production of membranes intended for water purification.

To reduce the cost of cellulose production during the pulping process, citric acid has been proposed as a catalyst. It is significantly less expensive than molybdenum- and tungstate-based compounds and is produced on a large industrial scale.

A series of experimental studies was conducted on the delignification of miscanthus stems using a cooking liquor composed of glacial acetic acid and water in a volumetric ratio of 75 : 25 %, with the addition of 50 % H_2O_2 (based on oven-dry raw material) and citric acid in amounts ranging from 0.25 % to 2.0 % (based on oven-dry raw material). The delignification process was carried out at constant temperature and duration (100°C, 180 min) in glass flasks under atmospheric pressure. The results of the pulping experiments were evaluated based on the yield of the solid residue (expressed as a percentage of oven-dry raw material) and the lignin content in the solid residue (percentage by weight) (Fig. 5).

According to the quality assessment results (Fig. 5), the use of citric acid leads to an increase in cellulose yield by 0.3 - 4.3 % (at catalyst dosages from 0.25 % to 1 %), while the residual lignin content decreases by 0.48 - 1.05 % based on oven-dry cellulose mass. These changes in solid residue quality indicators are attributed to the accelerated dissolution of lignin macromolecules and their transfer into the cooking liquor, along with stabilization of the carbohydrate component of the plant biomass. It is also worth noting that peracetic acid is chemically unstable due to the presence of trace amounts of transition metal ions. The addition of citric acid significantly improves the stability of peracetic acid.

It should be noted that at citric acid dosages exceeding 0.5 % of the oven-dry raw material, the cellulose yield decreases despite an almost identical degree of delignification compared to similar pulping without chemical additives. The optimal values of the examined delignification parameters for miscanthus stems are achieved at a citric acid dosage of 0.5 % based on oven-dry raw material.

In the pulping process of miscanthus stems using the acetic acid - water - hydrogen peroxide system, one of the key indicators is the extent of oxidant utilization. It was determined that the initial concentration of peracetic acid in the cooking liquor was 4.8 %, whereas after completion of the delignification process, its residual concentration decreased to 1.3 %. This indicates that only about 27 % of the initial peracetic acid content remains in the liquor after pulping, confirming its intensive consumption during delignification. According to literature data, the reuse of spent liquor with such a low level of active oxidant is ineffective due to the significant loss of oxidative capacity, making it impossible to achieve the required degree of delignification upon reuse in the preparation of fresh cooking liquor [27].

CONCLUSIONS

The conducted research confirms the feasibility of using *Miscanthus giganteus* stems as an alternative non-wood raw material for cellulose production via the oxidative-organosolv pulping process. Optimal delignification parameters were established: a temperature of 100°C and a duration of 180 min, which provided a cellulose yield of 57.92 % with a residual lignin content of 1.84 %, based on the oven-dry mass of the raw material.

The physico-mechanical testing of the obtained cellulose confirmed its compliance with the requirements for primary semi-finished products derived from wood-based raw materials. Morphological analysis revealed a reduction in average fiber length after refining, along with an increase in the proportion of fines structural characteristics typically associated with non-wood cellulose.

The effectiveness of citric acid as a catalyst in the oxidative-organosolv pulping process was experimentally confirmed. It was established that the optimal dosage is 0.5 % based on the oven-dry weight of raw material, which ensures an increased cellulose yield and reduced residual lignin content.

It was established that only 27 % of the initial oxidant concentration remains in the spent liquor after completion of the pulping process, resulting in low reactivity of the solution upon reuse.

Authors' contributions

I.T. made the primary contribution to the manuscript preparation, including the synthesis of scientific data, analysis of the obtained results, and their graphical and textual presentation. She was responsible for structuring the material and ensuring the manuscript's compliance with the journal's requirements.

A.H. was responsible for conducting experimental studies, including sample preparation, performing a series of measurements, and processing primary data. Additionally, she translated the manuscript into English and handled the formatting of the reference list.

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