

Eco-friendly conversion of corn husk agro-waste into microcrystalline cellulose and cellulose nanofibers

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ABSTRACT

Efficient use of bioresources is essential in order to achieve sustainable development goals, and plays an important role in the reduction of any environmental impact and the improvement of resource efficiency. To this end, the present study investigates the process of obtaining microcrystalline cellulose (MCC) and cellulose nanofibers (CNFs) from corn husks (CHs), which are agricultural waste, under “mild” conditions by employing the organosolvent oxidation method using peroxyacetic acid (PAA). The results obtained at an effective hydromodule of CH:PAA 1:18 g/mL, were: MCC yield – 51.60 %, α -cellulose content – 58.73 ± 3 %, residual lignin – 12.92 ± 0.5 %, hemicellulose – 13.8 ± 0.5 %, ash content (SiO_2) – 10.73 ± 0.5 %, and moisture content – 3.57 ± 0.5 %. Furthermore, the effective MCC:FA ratio that was used to obtain CNFs from MCC_{CH} using formic acid (FA) was found to be 1/30 g/mL. The CNF yield achieved with this ratio was 68.67 %, and the average particle size was reduced by 177 times in length and 33 times in width, as compared to the initial MCC_{CH} size. The ζ -potential of the resulting MCC was -19.3 mV and the crystallinity index was 80.9 %. The effectiveness of the PAA/FA approach arises from mild oxidation that selectively removes amorphous cellulose, while preserving crystalline regions, thereby maintaining high crystallinity and contributing to favorable material yields. The physicochemical properties of the resulting high-quality cellulose micro- and nanofibers were compared, and found to have been obtained at good efficiency. The MCC and CNFs obtained have excellent potential for use in the medical field as essential components, and in the development of bioplastics and biocomposites, among other applications.

1. Introduction

In the context of increasing global attention to the problems of pollution and climate change, in addition to the desire to achieve carbon neutrality, modern chemistry has gained a focus on the the development of environmentally-friendly production technologies, which are aligned with the goals of the Sustainable Development Goals (SDGs), in particular: SDG 12, “Responsible consumption and production”, and SDG 13, “Climate action” [1]. In this regard, there has been a shift in the focus of research activities from traditional hydrocarbons as raw materials towards renewable and low-value feedstocks, primarily waste from the agro-industrial complex, due to the need to reduce any negative

environmental impact and develop a circular economy [2].

One promising type of biomass in this context is corn husks (CHs), an agro-waste product that is typically disposed of through burning, thus contributing to the emission of greenhouse gases into the atmosphere. The shift in focus to renewable sources, such as agro-industrial waste, is a direct response to the necessity of reducing the carbon footprint of the chemical industry [3]. In the sustainable development paradigm, such waste is increasingly viewed as a valuable lignocellulosic resource [4]. Zea mays L (CLs), being one of the most common and renewable types of crop residues, represent a promising, economically viable, and environmentally sustainable source of raw materials for the production of chemicals. In 2024, Kazakhstan produced approximately 1 million tons

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of corn, which accounts for approximately one-third of the total volume of agricultural waste generated in the country [5]. Global corn production shows an upward trend, and is currently at approximately 1.228 billion tons per year [6]. Motivated by the global transition toward a carbon-neutral economy and the implementation of the SDGs, modern scientific research is increasingly focused on renewable and economically accessible carbon sources. CLs are an attractive feedstock for chemical transformation due to their high mass, homogeneity, availability, and ease of pre-processing. Among various types of biomass, CLs are one of the largest types of agricultural raw material, the chemical composition of which includes approximately 40–45 % cellulose, 25–35 % hemicellulose, and 7–10 % total lignin by dry weight, as well as non-structural components such as moisture, ash elements, and solvent-extractable compounds [7].

Compared to other types of lignocellulosic residues, such as grain and other types of crop husks, the use of CLs allows for optimized technological processes, reduction of energy used, lowered logistics costs, and assurance of a sustainable raw material supply chain. Furthermore, processing CHs helps reduce the cost of the final product by eliminating competition with food resources and efficiently utilizing locally available waste. Thus, this approach is highly efficient, both environmentally and economically, thus meeting modern sustainable development requirements [8].

Previously, various fractions of corn biomass have been utilized as raw materials for cellulose production, including corn stalks [9,10], and straw [9–11]. Compared to other corn residues such as stalks and cobs, corn cob outer leaves, the husks, exhibit a softer structure with a lower lignin content, which facilitates their processing under mild conditions. Unlike stalks, which largely remain as field residues after harvesting, CHs are generated in substantial quantities during industrial preparation of corn for boiling and food processing, and are typically discarded as waste. Therefore, CHs have stable availability, and their concentrated accumulation after corn processing makes them a particularly attractive and underutilized feedstock for sustainable cellulose production. An existing approach is alkaline and acid pretreatment, which is aimed at degrading lignin and extracting the cellulose fraction [12]. Despite the promise of such methods, CLs remain an understudied source, with high potential for bioconversion and the production of environmentally-friendly materials [13]. The properties of the isolated cellulose depend significantly on its source and the method of isolation, leading to the formation of two main classes of cellulose: microcrystalline cellulose (MCC) and cellulose nanofibers (CNFs) [14].

Cellulose, one of the most common polymers in nature, possesses a remarkable capacity for various modifications. These changes can be both structural and chemical, allowing for the production of materials with diverse properties for a wide range of applications [15–17].

There have been a number of studies devoted to the production of cellulose materials through chemical pretreatment of various bio-based raw materials. The dominant method for the isolation of nanocellulose particles remains acid hydrolysis, using 60 % sulfuric acid (H_2SO_4) [18–20]. Along with varying the concentrations of strong acids, numerous studies report the use of varying concentrations of alkalis and bleaching agents, so as to effectively remove non-cellulosic components such as lignin and hemicellulose [21].

However, the abovementioned concept for production of cellulosic materials is characterized by a number of significant environmental drawbacks associated with the use of aggressive acids and alkalis, the formation of toxic wastewater, the highly corrosive nature of the reaction medium, and the significant costs of neutralization and disposal. Taken together, such processing leads to an increased carbon footprint of the process, and, thus, limits the potential for industrial implementation of these technologies within the framework of green chemistry and sustainable development principles. Although several studies have successfully demonstrated the production of cellulosic materials with various morphologies, scaling up such processes inevitably requires careful optimization of reaction parameters from an environmental

perspective, including reactant concentrations, reaction time and temperature, and target product yield. Despite numerous studies on cellulose extraction from corn residues, the available scientific literature contains virtually no data on obtaining cellulose directly from corn cob outer husks. Furthermore, environmentally-friendly methods for processing of this type of raw material have yet to be thoroughly investigated.

From this perspective, the scientific novelty of the present study lies in the use of CHs as a previously unexploited cellulose-containing raw material for the production of MCC and CNFs, by using a gentle, environmentally-friendly method (Fig. 1a). The proposed approach involves subsequent extraction and characterization of cellulose materials through the application of mild acid hydrolysis, with minimal use of chemicals to remove non-cellulosic components. An additional advantage of the selected raw material is its high availability, because CHs are produced in significant quantities not only during the harvest but also year-round where boiled corn is industrially prepared and CHs are disposed of as waste.

The aim of this study is to determine effective processing parameters for production of MCC and CNFs from agricultural corn waste in Kazakhstan, as well as to investigate their structure and physicochemical properties. This research focuses on the environmentally efficient recycling of agricultural waste in order to reduce anthropogenic impacts on the environment and to promote the rational use of natural resources.

2. Experimental section

2.1. Materials

Acetic acid ($\geq 55\%$, CH_3COOH), hydrogen peroxide (15 %, H_2O_2), potassium permanganate (99 %, KMnO_4), ethanol (96 %, $\text{C}_2\text{H}_5\text{OH}$), sulfuric acid (98 %, H_2SO_4), potassium bichromate ($\geq 99\%$, $\text{K}_2\text{Cr}_2\text{O}_7$), sodium thiosulfate ($\geq 99\%$, $\text{Na}_2\text{S}_2\text{O}_3$), potassium iodide ($\geq 99\%$, KI), and starch were obtained from Sigma-Aldrich (Bangalore, India). Formic acid ($\geq 99\%$, HCOOH) was obtained from Sigma-Aldrich (Labtime, Kazakhstan). All reagents were of analytical grade and were used without additional purification.

2.2. Methods

2.2.1. Preparation of raw materials

An annual agricultural plant waste, corn husks (CHs), were used as the raw material in order to obtain microcrystalline cellulose (MCC) (Fig. 1). CHs from locally-produced *Zea mays* L. were collected from agricultural fields located in the village of Zevakino, Shemonaikha district, in the East Kazakhstan Oblast of the Republic of Kazakhstan ($50^\circ 16' 11.1''\text{N}$, $81^\circ 55' 41.7''\text{E}$), during the growing season from May to August 2022.

For the study, an early-ripening three-line corn hybrid, Darina MV, of universal use, was selected, as it has been included in the “State Register of Breeding Achievements Approved for Use in the Republic of Kazakhstan” since 2021 and is recommended for use as silage (Fig. 1a)

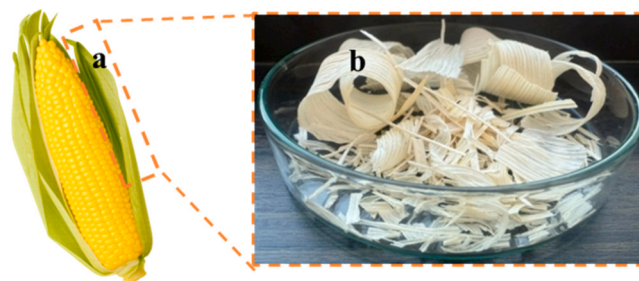


Fig. 1. *Zea mays* L.: a) An ear of the Darina hybrid; and, b) shredded CHs of the Darina hybrid.

[22].

Darina is an early-maturing, three-line corn hybrid that is suitable for general use. The plants average 256 cm in height, with the height of the lower ear attachment at approximately 46 cm. The hybrid has slightly curved leaves with a small angle between the leaf blade and stem, and reaches maturity in 90–100 days. The lateral branches are curved. The cob has a medium anthocyanin color [23].

For preliminary preparation, the CHs were not treated with sodium hydroxide (NaOH), as is often practiced when processing plant materials containing phytomelanin [24,25], because that pigment is absent from the composition of Darina (Fig. 1b), thus the natural straw-yellow color does not require a bleaching stage. Raw material preparation consisted of washing with water, only to remove dust and mechanical impurities, after which the material was placed in a drying oven (DC-80, Grod-torgmash, Belarus) at $60 \pm 2^\circ\text{C}$ for 6 h, until a completely dry and stable mass was achieved. During the study, samples obtained from the husks of Darina hybrid corn were conventionally designated as CHs.

2.2.2. Preparation of peroxyacetic acid (PAA)

PAA was synthesized through the following the method, as described by Kydyrmolla et al. [26]. The synthesized PAA was subsequently stored at $5 \pm 0.5^\circ\text{C}$ in a refrigerator. The final concentration of the PAA obtained was 12 %.

2.2.3. Preparation of microcrystalline cellulose (MCC) via the “soft” organosolvent oxidation method

In order to determine the optimal hydromodule during the interaction of crushed CHs with PAA, the method described by Akatan, K. et al. was used [26]. In the present study, due to the high hygroscopicity and good wettability of crushed raw CHs, the range of hydromodule was expanded, and amounted to 1:16, 1:18, 1:20, 1:22 and 1:24 g/mL. For this purpose, 10 g of raw material were individually weighed from CH samples, after which it was mixed with PAA in the above CH:PAA ratios.

MCC was prepared by boiling the raw material with PAA under reflux at a temperature of $90 \pm 2^\circ\text{C}$, with constant, vigorous stirring. The MCC samples obtained was cooled to a temperature of $25 \pm 2^\circ\text{C}$, then filtered using filter paper and washed with distilled water until a neutral pH of 7 was achieved. The neutralized MCC sample was dried at a temperature of $70 \pm 2^\circ\text{C}$ for 4 h, until constant weight was achieved (Fig. 2).

The dried product was weighed on an analytical balance (Sartogsm LV 210-A, Russia), with an accuracy of 0.0001 g, after which the MCC yield was calculated using the following formula:

$$\eta = m_{\text{MCC}}/m_{\text{CH}} \times 100\%$$

Where, m_{CH} is the mass of the CH sample, in grams; and, m_{MCC} is the mass of the MCC sample obtained, in grams.

During the study, the MCC obtained from CHs was conventionally designated as MCC_{CH} .

2.2.4. Determination of MCC_{CH} quality indicators

The moisture content of the final microcrystalline cellulose (MCC) product was determined by following the ASTM D 1348–94 (2008) protocol. The α -cellulose content was measured in accordance with ASTM D 588 (2000), while residual lignin and hemicellulose contents

were determined using ISO/DIS 21436 and ASTM D 5896–96 (2019) e1, respectively. In order to measure the ash content (SiO_2), a 3 g sample of MCC was incinerated in a muffle furnace (8.2/1100 L, SNOL, Lithuania) at 700°C for 90 min. The remaining ash mass was then measured with an analytical balance (LV 210-A, Sartogsm, Russia), so as to obtain a stable average value.

2.2.5. Preparation of cellulose nanofibers (CNFs) from MCC_{CH}

As an environmentally benign alternative to conventional mineral acid-based and hydrothermal methods, which typically require high temperatures and elevated pressures in autoclave systems [27–29], the present approach employs formic acid (FA), an organic acid, as the hydrolyzing agent. FA is characterized by relatively low toxicity, easy biodegradability, and high selectivity toward cellulose domains, making it a promising reagent for sustainable CNF production. The CNF preparation process was based on the method used by Battalova A. et al. [28]. MCC_{CH} to FA ratios were 1:30, 1:40 and 1:50 g/mL. The hydrolysis process was carried out in a reflux flask, along with with continuous magnetic stirring, at a reaction temperature carefully maintained at $90 \pm 2^\circ\text{C}$. Throughout the process, vigorous stirring ensured uniform reaction conditions, while the hydromodule was kept constant.

After 6 h of hydrolysis, the reaction mixture was neutralized using deionized water at a controlled temperature of $25 \pm 2^\circ\text{C}$. The resulting suspension was centrifuged in a tabletop high-speed centrifuge (ZT-TG16, Hunan Zetop Medical Co., Ltd., China), initially at 16 000 rpm, and then at 5000 rpm, so as to separate the nanofibers. The cellulose nanofiber suspension was subjected to ultrasonication using an ultrasonic homogenizer (3000 F, Scientz, China), at 40 kHz for 30 min, in order to further individualize the nanofibers. The dispersion obtained was subsequently freeze-dried in a lyophilizer (LGJ-12S, Beijing Songyuan Huaxing Biotechnology, China) (Fig. 3 and 4). The final weight of CNFs was measured using an analytical balance (LV 210-A, Sartogsm, Russia) with a precision of ± 0.0001 g.

The dry yield of CNFs was calculated using the following formula:

$$\eta = m_{\text{CNF}}/m_{\text{MCC}} \times 100\%$$

Where, m_{CNF} is the mass, in grams, of the CNFs obtained; and, m_{MCC} is the mass, in grams, of MCC.

2.2.6. Analysis of particle size and ζ -potential

Dynamic light scattering (DLS) was performed on a particle size analyzer (Zetasizer Nano ZS 90, Malvern, UK), in order to determine the average particle size and ζ -potential of the MCC and CNF suspensions.

Aqueous MCC_{CL} and CNF suspensions, of 2 wt%, were first dispersed using an ultrasonic bath (3000 F, Scientz, China) for 30 min at 30 kHz. Three samples from each of the selected ratios were prepared and analyzed.

2.2.7. Fourier-transform infrared spectroscopy (FTIR)

FTIR was performed for the purpose of characterizing the chemical structure of the initial CH and the processed MCC and CNFs. The measurements were carried out using an FTIR spectrometer (FT-801, Simex, Russia) equipped with attenuated total reflection (ATR) and specular-diffuse reflection (SDR) accessories. Spectra were recorded in the range of $500\text{--}4700\text{ cm}^{-1}$, with a spectral resolution of 1 cm^{-1} , at a constant temperature of 25°C . For each sample, 100 scans were averaged in accordance with standard procedures, so as to ensure reproducibility and accuracy of the spectral data.

2.2.8. XRD analysis

The crystalline structure of the CH, MCC and CNF samples was analyzed utilizing a diffractometer (X'Pert PRO, Malvern Panalytical Epyrean, Netherlands) equipped with monochromatized $\text{CuK}\alpha$ radiation ($\lambda = 0.1542\text{ \AA}$). XRD measurements were carried out in the 2θ range of $10^\circ\text{--}40^\circ$, with a step size of 0.02° . The diffractometer was operated at

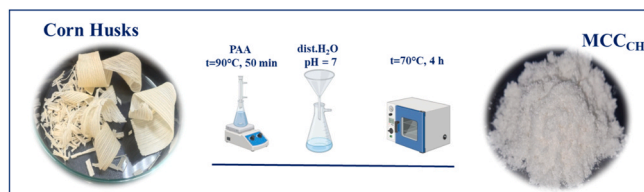


Fig. 2. Diagram showing MCC being processed using the organosolvent oxidation method.

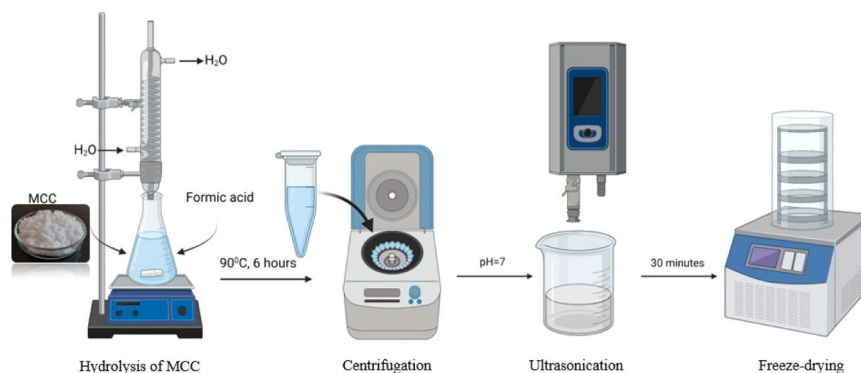


Fig. 3. Diagram of CNF Extraction from MCC_{CH}.



Fig. 4. MCC_{CH} obtained at the most efficient hydromodule of 1:18 g/mL.

an accelerating voltage of 40 kV and a tube current of 30–45 mA. The measurement time per step was set to 0.5 s, so as to ensure sufficient resolution and intensity of the diffraction peaks.

The crystallinity index was calculated using the following equation:

$$CI (\%) = (A_{\text{cryst}} / A_{\text{cryst}} + A_{\text{amorph}}) \cdot 100\%$$

Where, A_{cryst} is the area under the calculated pattern for crystalline cellulose; and, A_{amorph} is the area under the pattern calculated for the amorphous content.

CSL was determined via the Scherrer formula:

$$CSL = k\lambda / \beta \cos\theta$$

Where, k is a shape factor that is often 0.89 [26]; λ is the wavelength of the diffractometer (0.1542 nm); β is the FWHM (maximum half of the full width) of the (200) diffraction peak in radians; and, θ is the diffraction angle.

2.2.9. Thermogravimetric analysis

The thermal stability and degradation behavior of the CNF samples were evaluated using a thermogravimetric analyzer (STA200 Synchro- nous Thermal Analyzer, Wuhan Bonnin Technology Ltd. China), under an argon atmosphere, so as to prevent oxidative degradation. Approximately 25 ± 2 mg of each sample was placed in the crucible for analysis. The temperature range was set from 30 ± 5 °C to 700 ± 5 °C, with a heating rate of 10 ± 1 °C/min. The mass loss profiles were recorded in order to assess the decomposition stages and thermal resistance of the CNFs.

2.2.10. Scanning electron microscopy (SEM) analysis

The surface morphology of MCC and CNF was analyzed using a scanning electron microscope (Quanta 200i 3D, FEI, Netherlands).

Surface morphology was visualized under high vacuum conditions using a secondary electron detector at an accelerating voltage of 15 kV. For sample preparation, 1 g of each, MCC and CNF powder, was dispersed separately in 50 mL of deionized water containing silver nanoparticles at a concentration of 10^{-3} mol/L. The suspensions were subjected to mechanical stirring for 2 h, then filtered using standard filter paper, and then dried in an oven at 50 ± 2 °C for 1 h. The dried samples were mounted on aluminum stubs using carbon adhesive tape prior to imaging.

3. Results and discussion

3.1. MCC_{CH} via the organosolvent oxidation method

Fig. 4 shows the microcrystalline cellulose (MCC_{CH}) obtained using the organosolvent oxidation method. The resulting product is clearly a light and cotton-like material. Despite the natural greenish-yellow color of the initial corn husks (CHs), the resulting MCC_{CH} is completely white. This indicates that the method utilized does not require additional bleaching processes, including delignification.

3.2. Quantitative and qualitative parameters of obtained MCC_{CH}

The effect of different CH:PAA hydromodules (g/mL) on the yield and quality indicators of MCC obtained under the “mild” conditions of the organosolvent oxidation method is presented in Table 1. According to the experimental results, the highest yield of MCC was determined to be at the ratio of 1:18 g/mL, and was calculated to be 51.60 ± 2 %. The content of α -cellulose in MCC obtained at this ratio also had the highest value, and amounted to 58.73 ± 3 %, with residual lignin at 12.92 ± 0.5 %, hemicellulose at 13.8 ± 0.5 %, ash (SiO₂) at 10.73 ± 0.5 %, and moisture at 3.57 ± 0.5 %. Although aggressive chemical treatments may yield higher α -cellulose contents (typically above 70 %), the value

Table 1

Effect of CH:PAA ratio on the yield and quality indicators of MCC_{CH}.

CH: PAA, g/mL	Quality indicators of MCC _{CH} , %					
	Yield, %	α -cellulose	Humidity	Ash content (SiO ₂)	Residual lignin	Hemi- cellulose
1:16	49.57 ± 2	54.96 ± 3	3.77 ± 0.5	11.35 ± 0.5	13.08 ± 0.5	15.6 ± 0.5
1:18	51.60 ± 2	58.73 ± 3	3.57 ± 0.5	10.73 ± 0.5	12.92 ± 0.5	13.8 ± 0.5
1:20	50.58 ± 2	56.63 ± 3	3.86 ± 0.5	11.09 ± 0.5	14.71 ± 0.5	12.9 ± 0.5
1:22	49.38 ± 2	55.30 ± 3	3.47 ± 0.5	11.15 ± 0.5	15.26 ± 0.5	13.0 ± 0.5
1:24	49.30 ± 2	55.80 ± 3	3.82 ± 0.5	12.02 ± 0.5	14.41 ± 0.5	13.1 ± 0.5

obtained in this study (~60 %) is consistent with the mild processing conditions, and remains adequate for sustainable material applications [15]. The yield and quality of MCC_{CH} obtained with a CH:PAA ratio of 1:20 g/mL are close to those achieved with a ratio of 1:18 g/mL. For resource efficiency and environmental sustainability, a CH:PAA ratio of 1:18 g/mL was determined to be the most effective, as it represents a balanced combination of compositional, kinetic and economic factors. At this ratio, a higher α -cellulose content and a relatively lower amount of amorphous residues indicate efficient delignification and controlled oxidation kinetics, while sufficient liquid volume ensures improved mass transfer and reagent penetration into the biomass matrix. In addition, reduced PAA consumption at this hydromodule enhances the economic feasibility of the process without compromising cellulose quality. Clearly, the yield of MCC_{CH} obtained from the remaining ratios is 2 % lower than the effective ratio, and the α -cellulose content is 3.2 % lower, while the residual lignin content is 2 % higher. Previous studies examined the process of obtaining MCC from agricultural waste using the oxidation method with organosolvents, and similar results were obtained, which indicates the efficiency and resulting product quality of the method used in the present study to obtain MCC [28,29]. According to the data reported by Saberi-Riseh et al., residual lignin contents in cellulose obtained from various agricultural wastes using conventional alkaline or combined chemical treatments are typically higher than 10–15 % [17]. Notably, in the present study, corn husks processed without preliminary sodium hydroxide (NaOH) treatment exhibited a lower residual lignin content, highlighting the effectiveness of the proposed mild oxidation approach.

3.3. FTIR analysis of MCC_{CH}

Fig. 5 shows the comparative IR spectra of MCC_{CH} obtained in all hydromodules of the CH:PAA ratio, in g/mL. According to the results of IR spectroscopic analysis, in all spectra the absorption band at 661.3 cm⁻¹ corresponds to out-of-plane vibrations of the C–OH bond, and in the range from 760 cm⁻¹ to 820 cm⁻¹ to aromatic out-of-plane vibrations of the residual lignin molecule [30,31]. The absorption regions from 895 cm⁻¹ to 1160.2 cm⁻¹ characterize β -(1,4)-glycosidic C–O–C bonds, characteristic of the cellulose molecule [32]. The absorption bands at 1320 cm⁻¹, 1335 cm⁻¹ and 1370 cm⁻¹ indicate O–H and C–H bonds in the glucoside units, respectively [33]. The absorption band at 1428 cm⁻¹ is characteristic of the signal of the hydrogen bond in the C–H₂ group, which determines the crystal structure of the cellulose molecule [34]. Weak absorption observed in the region of 1512.3 cm⁻¹ corresponds to the C=C bond in the aromatic ring of the

residual lignin molecule, and the absorption signal at 1728 cm⁻¹ corresponds to vibrations of the acetyl groups C=O in the hemicellulose molecule [31,32]. The signal in the region of 1640 cm⁻¹ indicates the O–H bond in the water molecule sorbed by cellulose from the air [33]. As well, the pike at 2892 cm⁻¹ clearly corresponds to vibrations of the CH groups, and 3332 cm⁻¹ to the OH groups [34,35].

The ratio of the intensity of the signal characteristic of residual lignin at 1512.3 cm⁻¹ to the intensity of the signal characteristic of β -(1,4)-glycosidic bond at 895 cm⁻¹ in the FTIR spectrum of the an effective ratio of CH:PAA 1:18 g/mL, the A1512/A895 ratio, was approximately 0.3. That is, the index of lignin is less than 1. The delignification process was shown not to be aggressive when using the organosolvent oxidation method. Similar results were obtained in other studies [36,37].

3.4. Scanning electron microscopy (SEM) analysis of MCC_{CH}

The surface morphology of MCC_{CH} obtained at an effective hydromodule of CH:PAA 1:18 g/mL was examined using SEM (Fig. 6). The micrograph shows that the surface morphology of the sample studied is ribbon-like. The average fiber size was approximately 500 μ m in length and 20 μ m in diameter. The surface of the fibers was clean and slightly rough. This indicates that the waxy compounds, lignin, and hemicellulose, which surrounded the fibers during the extraction process, were completely removed. The surface morphology of MCC obtained from rice and sunflower waste under the same, “mild”, organosolvent oxidation conditions, as determined in other studies, is consistent with the results of MCC_{CH} obtained in the present study [38–40].

3.5. Preparation of cellulose nanofibers (CNFs) from MCC_{CH}

CNFs were obtained from MCC_{CH} at an effective CH:PAA hydromodule of 1:18 g/mL using formic acid (FA) (Fig. 7). In the present study, the MCC_{CH}/FA hydromodule was set at ratios of 1/30, 1/40, and 1/50 g/mL. The resulting CNF yield and quality parameters at the three different hydromodules are shown in Table 2, in comparison with the original MCC_{CH}. The highest CNF yield was found at the MCC_{CH}/FA ratio of 1/30 g/mL, which amounted to 68.67 %. The average size of the CNF particles obtained at this ratio was 2.824 μ m in length and 60 nm in width, which indicates a decrease in length by 177 times and in width by 33 times, as compared to the original MCC_{CH}. The crystallinity index reached 80.9 %. The CSL was 2.9 nm, which can be attributed to the selective removal of amorphous cellulose regions by the FA, a mild and environmentally friendly reagent, potentially enhancing mechanical, thermal and barrier properties. The zeta potential value of –19.3 mV indicates moderate dispersion stability of the CNF suspension over time, which is sufficient for short-term processing and handling. The extremely low ζ -potential of the CNFs provides a good opportunity for the surface modification of water to purify metal ions and to prepare membranes for the separation of proteins from biomaterials [41,42]. This stability can be further improved by simple approaches such as pH adjustment, mild surface modification, or ultrasonication, so as to enhance electrostatic repulsion and dispersion homogeneity of the nanofibers.

The yield of CNF was 54.21 % and 53.83 %, respectively, at the MCC_{CH}/FA ratios of 1/40 and 1/50 g/mL. The crystallinity index was approximately 2.7 % and 3.1 % lower, and the zeta potential was –4.2 and –4.6 mV lower than those of CNF obtained at the ratio of 1/30 g/mL. The particle size was found to decrease by approximately 147–152 times in length and by approximately 25–27 times in width as compared to the initial MCC_{CH} particle size. In addition, the CLS and CI values were observed to decrease with increasing FA content, which indicates that the use of excessive amounts of acid has a negative effect on the quality parameters of CNFs. In this regard, the MCC_{CH}/FA ratio of 1/30 g/mL was found to be the most effective in obtaining CNFs.

Liu et al. produced nanocellulose from bleached corncob residues

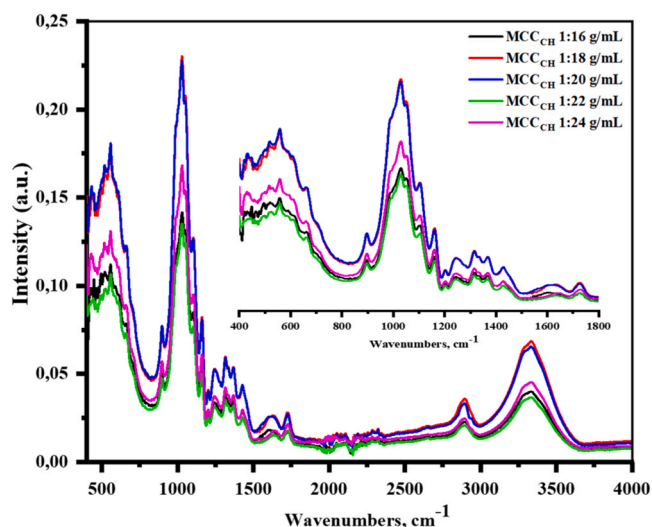


Fig. 5. FTIR spectra of MCC_{CH}.

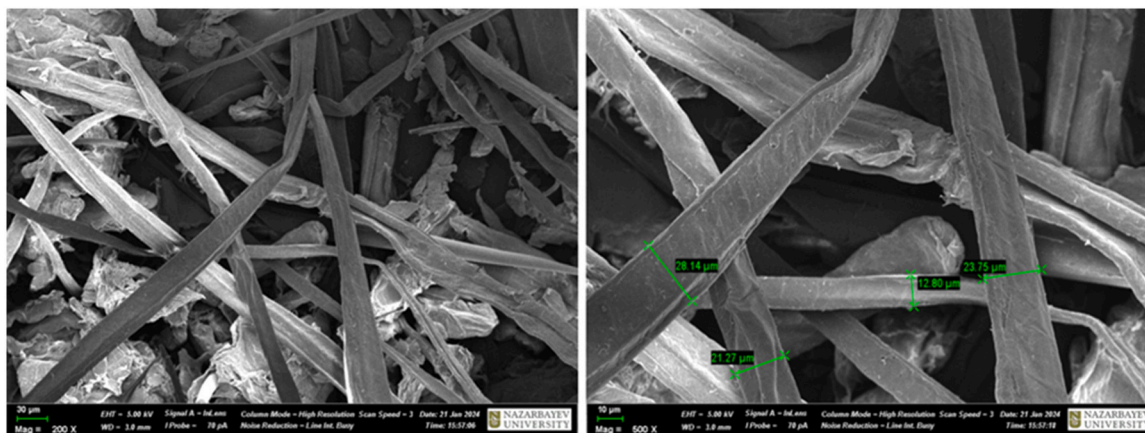


Fig. 6. SEM micrograph of MCC_{CH} obtained at an effective hydromodule of CH:PAA 1:18 g/mL.

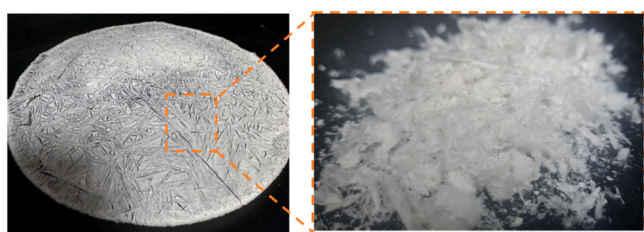


Fig. 7. CNFs obtained at MCC_{CH}:FA 1/30 g/mL.

under various hydrolysis conditions, applying sulfuric acid, formic acid, TEMPO-mediated oxidation, and pulp refining [43]. Formic-acid hydrolysis generated longer, rod-shaped CNFs with high crystallinity, while sulfuric-acid treatment produced shorter rod-like CNFs with lower crystallinity. Compared to the sulfuric acid and TEMPO-based methods, the PAA/FA route provides clear sustainability advantages, due to reduced chemical waste generation, lower corrosivity, and easier handling and recovery of reagents [44].

Battalova et al. studied the process of obtaining CNFs at different ratios of sunflower residue and FA [27]. According to the results of the study, the yield and quality of CNFs with the lowest ratio of raw material to FA were higher, which indicates that the results obtained in the present study are within established tolerances.

All qualitative and quantitative characterizations of MCC and CNFs were performed in triplicate, using three independent experimental batches, the results of which are presented as mean values with corresponding standard deviations.

3.6. FTIR analysis of CNFs

Fig. 8 shows the IR spectra of cellulose nanofibers obtained from MCC_{CH} with FA at an effective ratio of 1/30 g/mL (Fig. 8b) compared to MCC_{CL} (Fig. 8a). The chemical structure of the samples obtained is consistent with the results of the FTIR analysis presented in Fig. 5.

The intensity of signals characteristic of cellulose molecules were

observed to increase in the spectra of the CNFs. In particular, there was a rise in the intensity of the signal characteristic of the C–O bond at an absorption wavelength of 898 cm^{−1} and in the signal characteristic of the –C–H₂ group, indicating a hydrogen bond that characterizes the crystalline structure of cellulose, in the range of 1450–1420 cm^{−1} [45–47], which together demonstrate the accuracy of the crystallinity index values determined in Table 2. Nang et al. studied the process of deriving CNF from rice husks using FA, and showed that FA is effective in obtaining CNFs [48].

3.7. X-Ray analysis of MCC_{CH} and CNFs

The crystal structure of MCC_{CH} (Fig. 9a), at the effective CH:PAA hydromodule of 1/18 g/mL, and CNF (Fig. 9b), at the effective MCC_{CH}/FA ratio of 1/30 g/mL, were comparatively studied via X-ray diffraction. The diffraction pattern of MCC_{CH} in Fig. 9a shows peaks characteristic of cellulose I β , with a double-chain monoclinic crystal lattice at 2 θ reflection angles of 15.3° (1–10), 22.2° (200), and 34.6° (004) [40]. Furthermore, the diffractograms of the CNFs correspond to the crystal structure of MCC_{CH}. Battalova et al. found that the use of FA during the preparation of CNFs hydrolyzed the amorphous regions in MCC and produced cellulose nanofibers with high crystallinity [27].

3.8. SEM analysis of CNFs from MCC_{CH}

Fig. 10 shows a micrograph of the surface of CNFs obtained at an effective MCC_{CH}:FA ratio of 1/30 g/mL. The CNF surface is seen to consist of ribbon-like fibers with a diameter of approximately 57 ± 2–78 ± 2 nm, which is consistent with the average particle size values given in Table 2. Furthermore, the nanofibers are evidently of uniform distribution and are interconnected, which the present study demonstrates is crucial in obtaining materials with a large surface area and porosity for various applications [41]. The potential medical applications of the MCC and CNFs obtained are based on their material structure and measured physicochemical properties, as well as evidence from previous studies. Their high crystallinity, nanoscale dimensions, and absence of aggressive chemical residues indicate suitability for use in

Table 2

Effects of formic acid-based hydrolysis conditions on the yield and properties of CNFs prepared from MCC_{CH}.

Samples	Reactiontime, h	Yield, %	Crystallinity index (CI), (%)	ζ -potential, mV	Average particle size, nm	
					Length	Width
MCC _{1/18}	-	51.60 ± 2	46.5 ± 4	-	500 ± 50 µm	20 ± 3 µm
CNF _{1/30}	6	68.67 ± 2	80.9 ± 4	-19.3	2824 ± 50	60 ± 5
CNF _{1/40}	6	54.21 ± 2	77.8 ± 4	-15.1	3271 ± 50	72 ± 4
CNF _{1/50}	6	53.83 ± 2	78.2 ± 4	-14.7	3390 ± 50	78

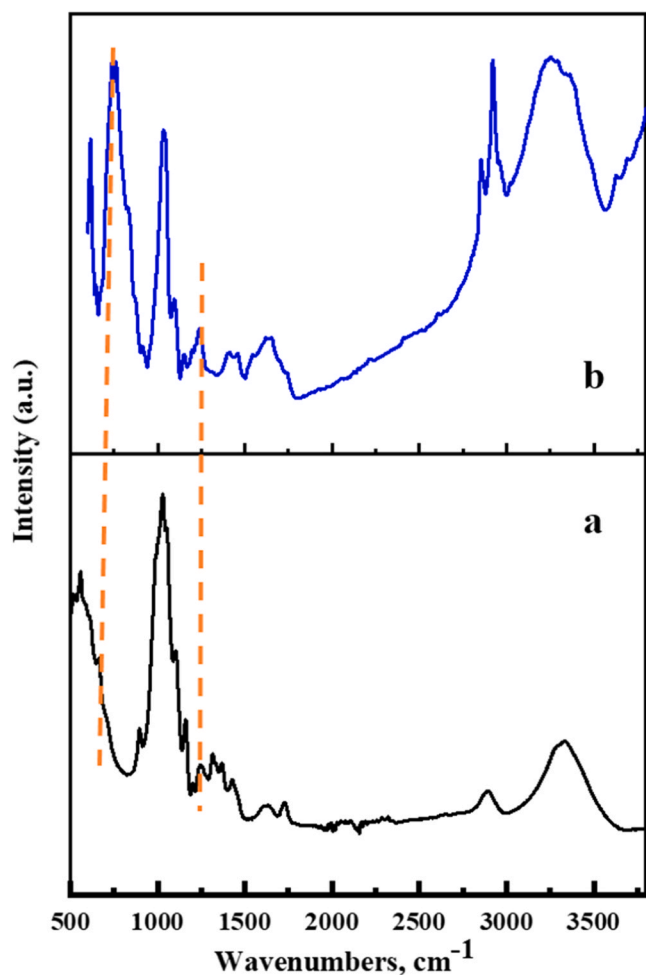


Fig. 8. IR spectra of CNFs obtained from MCC_{CH}. a) MCC_{CH}/18 g/mL; and, b) CNF1/30 g/mL.

biopolymer matrices and drug delivery systems [15]. The combination of mild processing conditions and organic acids confers high industrial relevance to the proposed method by minimizing corrosion, simplifying process control, and enabling cost-effective and scalable implementation.

3.9. TEM analysis of CNFs from MCC_{CH}

The transmission electron microscopy (TEM) results, shown in Fig. 11, reveal the characteristic long and flexible thread-like shape of the CNFs. The fibers are uniformly distributed, have a diameter of approximately $30 \pm 2 - 70 \pm 2$ nm, and range from 500 nm to several hundred microns in length. These sizes play a crucial role in the use of such CNFs as reinforcement in composite structures, which has been summarized in other studies [27].

3.10. TGA analysis of MCC_{CH} and CNFs

Fig. 12 shows the relative thermal stability of MCC_{CH} with an effective CH:PAA water modulus of 1/18 g/mL and CNFs with an effective MCC_{CL}:FA ratio of 1/30 g/mL. In the first temperature range of 30–280 °C, the CNFs lost approximately 5 % of their initial mass. This is due to the fact that CNFs have a larger surface area than does MCC, allowing absorption of more moisture from the air. No change in the mass of MCC was observed in this temperature range.

Both samples underwent thermal degradation with significant mass loss in the range from 280 to 410 °C for CNF and from 361 to 420 °C for

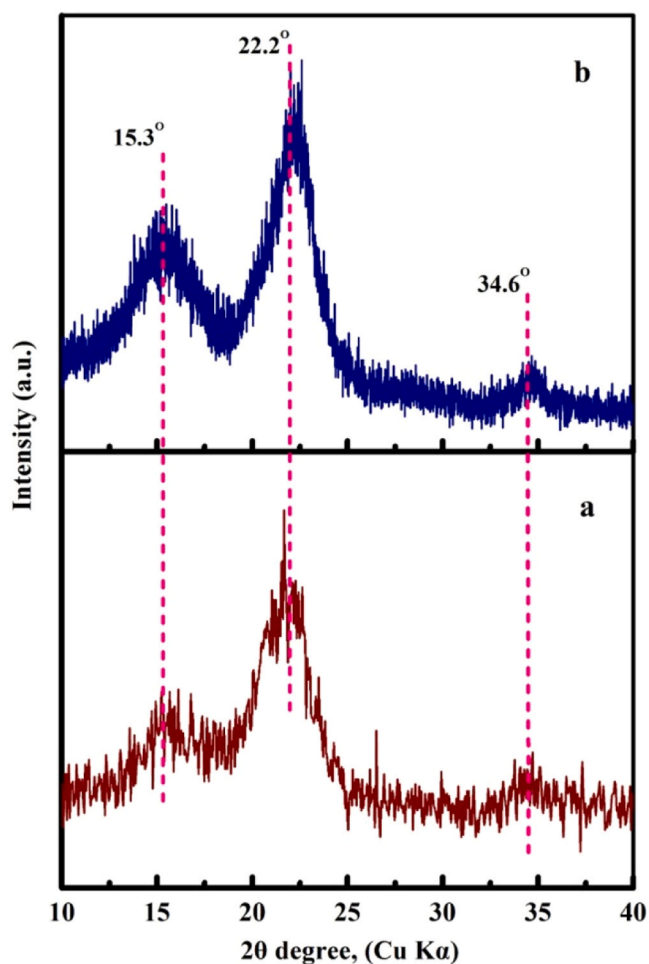


Fig. 9. X-Ray diffractograms of MCC_{CH} and CNFs: a) MCC_{CH}/18 g/mL; and, b) CNF1/30 g/mL.

MCC_{CH}. The early onset of thermal degradation of CNF may be due to the binding of carboxyl or formyl functional groups on the particle surface during formic acid hydrolysis, which appears to correspond to the ζ -potential value of -19 mV, as determined using DLS analysis [49–52].

4. Conclusions

This study demonstrates a highly efficient and environmentally benign strategy for conversion of low-value corn husks (CHs), considered to be agro-waste, into high-purity microcrystalline cellulose (MCC) and structurally-ordered cellulose nanofibers (CNFs). An effective water modulus for the extraction of MCC from cellulose using polyacrylic acid (PAA) was determined to be a ratio of 1:18 g/mL, which resulted in a yield of 51.60 ± 2 %, with contents of α -cellulose (58.73 ± 3 %), residual lignin (12.92 ± 0.5 %), hemicellulose (13.8 ± 0.5 %), ash content (SiO_2) (10.73 ± 0.5 %), and moisture content (3.57 ± 0.5 %). The chemical structure was characterized using FTIR, which found that the structural characteristics of the cellulose did not change. SEM analysis revealed the surface morphology of MCC to be ribbon-like, with average fiber dimensions of approximately 500 μm in length and 20 μm in diameter, which indicates that “mild” oxidation with an organosolvent is an effective method for extraction of MCC from CHs.

Furthermore, the effective MCC_{CH}:FA ratio for the preparation of CNFs from MCC_{CH} using formic acid (FA) was studied and ultimately found to be 1/30 g/mL. The yield of CNFs obtained at this ratio was 68.67 %, and the average particle size decreased by 177 times in length and 33 times in width, as compared to the original MCC_{CH} dimensions.

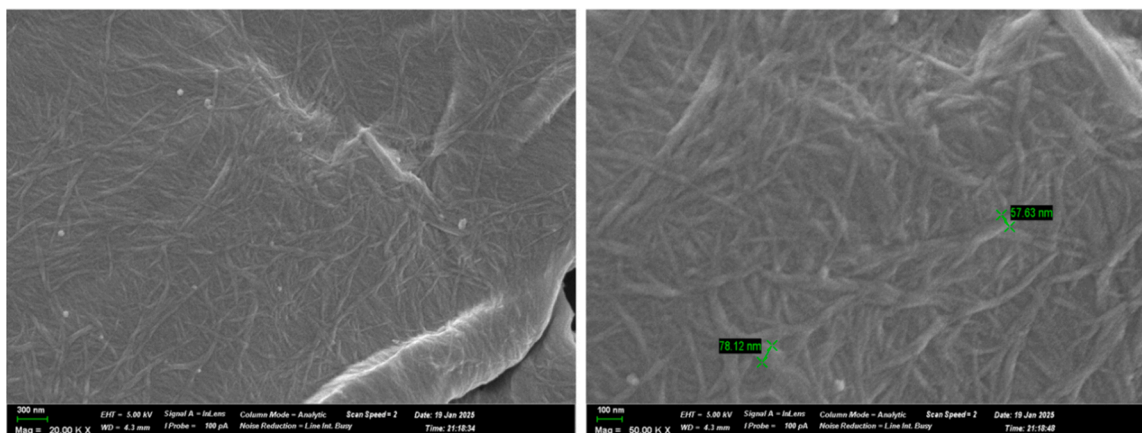


Fig. 10. SEM images of CNFs derived from MCC_{CH}.

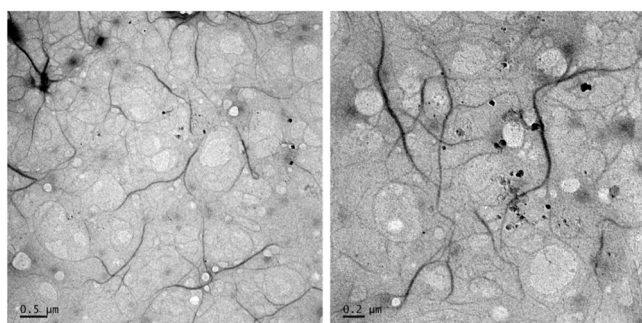


Fig. 11. TEM images of CNFs derived from MCC_{CH}.

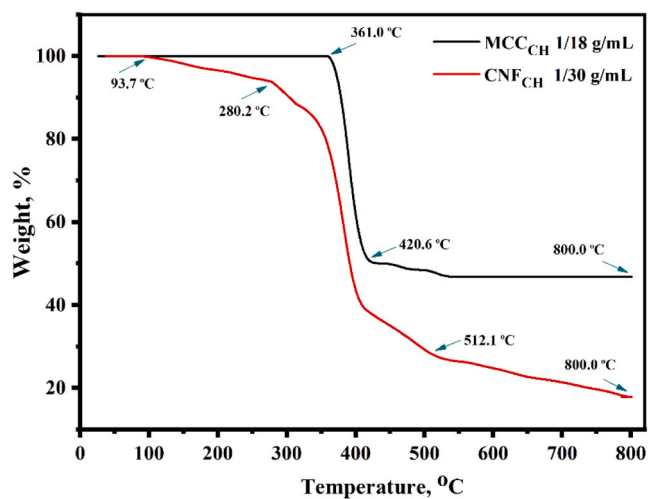


Fig. 12. TGA curing of MCC_{CH} and CNFs.

The ζ -potential was -19.3 mV and the crystallinity index was 80.9 %. According to X-ray diffraction analysis, the crystal lattice of the MCC_{CH} had a structure characteristic of cellulose I β , which is double-chain monoclinic. SEM and TEM analysis revealed that CNFs are ribbon-like fibers, with a diameter of approximately 57–78 nm. The thermal stability of both MCC_{CH} and CNFs was examined and degradation was found to occur in CNFs in the range of 280–410°C, with a loss of approximately 65 % of the initial mass, and in MCC_{CH} at 361–420°C, where it lost approximately 50 % of its initial mass.

The results of this research demonstrate that high-quality MCC can be obtained from cellulose by organosolvent oxidation under mild

conditions, and that CNFs with excellent physicochemical properties can be produced using FA. The application of these methods can facilitate the development of technologies for the production of highly-efficient, environmentally-friendly, and high-quality cellulose materials. Furthermore, the MCC and CNFs obtained demonstrate significant potential for applications in the paper industry, pharmaceuticals, and bionanocomposites, with future research directed toward their use in antibacterial nanocomposite systems. To this end, the present research team plan to extend their work in this extraction process to include several other promising sources of cellulose that result from agriculture and food processing in Kazakhstan, including wheat, potato, onion and sunflower stalks, rice chaff, and, in accordance with previous work, sunflower seed husks. Ultimately, the goal is to achieve a near zero-waste economy with regards to the major agricultural products that are grown in Kazakhstan, with the hope of developing technologies that can be licensed for use in similar regions.

CRediT authorship contribution statement

Sana Kabdrakhmanova: Writing – review & editing, Validation, Supervision, Resources, Project administration, Funding acquisition. **Nazym Sagiyeva:** Writing – review & editing, Writing – original draft, Visualization, Validation, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Ainur Kabdrakhmanova:** Investigation, Data curation. **Madiar Beisebekov:** Data curation. **Gulfaridat Kampitova:** Project administration. **Esbol Shaimardan:** Project administration, Data curation. **Kydyrmolla Akatan:** Writing – review & editing, Validation, Supervision, Project administration, Data curation. **Ainur Battalova:** Visualization, Data curation.

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.

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Data availability

Data will be made available on request.

References

- [1] United Nations. (2015). Transforming our world: the 2030 Agenda for Sustainable Development. (<https://sdgs.un.org/goals>).
- [2] L. Chopra. Extraction of cellulose from agro waste – A short review. *Journal Name*, Volume(Issue), Page numbers. <https://doi.org/10.1016/j.matpr.2023.04.378>.
- [3] NV Lobus, Carbon footprint reduction and climate change mitigation: a review of current approaches and technologies, *Carbon* 120 (2023). (<https://www.mdpi.com/2311-5629/9/4/120>) (Article).
- [4] A.O. Ojo, An overview of lignocellulose and its biotechnological importance in high-value product production, *Fermentation* 9 (11) (2023) 990, <https://doi.org/10.3390/fermentation9110990>.
- [5] (https://www.apk-inform.com/en/news/1546374?utm_source).
- [6] USDA. (2024). World Agricultural Supply and Demand Estimates (WASDE), July 2024. Global maize production forecast: 1,222.77 million tonnes. (<https://www.usda.gov/oce/commodity/wasde>).
- [7] M. Balat, H. Balat, Recent trends in global production and utilization of bio-ethanol fuel, *Appl. Energy* 86 (11) (2009) 2273–2282, <https://doi.org/10.1016/j.apenergy.2009.03.015>.
- [8] Elsevier. (2023). Sustainable Development Goals Resource Centre. (<https://www.elsevier.com/connect/sustainable-development-goals>).
- [9] Y. Bin, S. Li, F. Jiao, S. Zhong, Y. Yuan, Comparative effects of pretreatment on composition and structure of corn stalk for biocomposites, *BioResources* 17 (3) (2022) 4395–4409, <https://doi.org/10.15376/biores.17.3.4395-4409>.
- [10] J. Suesat, P. Suwanruji, Preparation and properties of microcrystalline cellulose from corn residues, *Adv. Mater. Res.* (2011) 1781–1784.
- [11] H.K. Singh, T. Patil, S.K. Vineeth, et al., Isolation of microcrystalline cellulose from corn stover with emphasis on its constituents: corn cover and corn cob, *Mater. Proc.* (2020), <https://doi.org/10.1016/j.matpr.2019.12.065>.
- [12] Mateo S., Peinado S., Morillas-Gutiérrez F., La Rubia M.D., Moya A.J. (2021) Nanocellulose from agricultural wastes: products and applications – a review. <http://doi.org/10.3390/pr9091594>.
- [13] R. Singh, V. Chaturvedi, Maize leaf as a lignocellulosic feedstock for sustainable biofuel production, *Renew. Energy* 152 (2020) 1082–1090, <https://doi.org/10.1016/j.renene.2020.01.112>.
- [14] Ministry of Agriculture of the Republic of Kazakhstan (2023) State register of selection achievements permitted for use in the Republic of Kazakhstan. Section: Zea mays L. (Maize). Committee on State Inspection in Agro-Industrial Complex, Nur-Sultan. Available at: (<https://sortcom.kz>).
- [15] S. Thomas, K. Joseph, S.K. Malhotra, K. Goda, M.S. Sreekala (Eds.), *Polymer Composites*, Biocomposites, John Wiley & Sons, 2013. (<http://download.polympart.ir/polympart/ebook/Polymer%20Composites%20-%20Biocomposites.pdf>).
- [16] S. Thomas, A. Penkova, Development and study of sustainable edible coating from carrageenan/starch/nanocellulose for enhancing fruit/vegetable shelf life and preservation, *ACS Omega* (2025), <https://doi.org/10.1021/acsomega.5c00480>.
- [17] R. Saberi-Riseh, M.G. Vazvani, M. Hassaniasadi, V.K. Thakur, Agricultural wastes: a practical and potential source for the isolation and preparation of cellulose and application in agriculture and different industries (Article), *Ind. Crops Prod.* 208 (2024) 117904, <https://doi.org/10.1016/j.indcrop.2023.117904>.
- [18] O.O. Sadare, K.O. Yoro, K. Moothi, M.O. Daramola, Lignocellulosic biomass-derived nanocellulose crystals as fillers in membranes for water and wastewater treatment: a review, *Membranes* 12 (2022) 320, <https://doi.org/10.3390/membranes12030320>.
- [19] S.S. Qureshi, S. Nizamuddin, J. Xu, et al., Cellulose nanocrystals from agriculture and forestry biomass: synthesis methods, characterization and industrial applications, *EnvironSciPollutRes* 31 (2024) 58745–58778, <https://doi.org/10.1007/s11356-024-35127-3>.
- [20] H.M. Shaikh, A. Anis, A.M. Poulouse, S.M. Al-Zahrani, N.A. Madhar, A. Alhamidi, M. A. Alam, Isolation and characterization of alpha and nanocrystalline cellulose from date palm (Phoenix dactylifera L.) trunk mesh, *Polymers* 13 (2021) 1893, <https://doi.org/10.3390/polym13111893>.
- [21] P. Sukyai, P. Anongjanya, N. Bunyahwuthakul, K. Kongsin, N. Harnkarnsujarit, U. Sukatta, R. Sothornvit, R. Chollakup, Effect of cellulose nanocrystals from sugarcane bagasse on whey protein isolate-based films, *Food Res. Int.* 107 (2018) 528–535, <https://doi.org/10.1016/j.foodres.2018.02.052>.
- [22] Ministry of Agriculture of the Republic of Kazakhstan (2023) State register of selection achievements permitted for use in the Republic of Kazakhstan. Section: Zea mays L. (Maize). Committee on State Inspection in Agro-Industrial Complex, Nur-Sultan. Available at: (<https://sortcom.kz>) (accessed 5 December 2025).
- [23] (https://ssc-otbor.ru/product/darina-mv/?utm_source).
- [24] C. Liu, B. Li, H. Du, D. Lv, Y. Zhang, G. Yu, X. Mu, H. Peng, Properties of nanocellulose isolated from corn cob residue using sulfuric acid, formic acid, oxidative and mechanical methods, *CarbohydrPolym* 151 (2016) 716–724, <https://doi.org/10.1016/j.carbpol.2016.06.025>.
- [25] Yara International (2025) Key facts about world maize production. Yara Crop Nutrition – Maize. (<https://www.yara.kz/crop-nutrition/maize/key-facts/world-production/>) (accessed 5 December 2025).
- [26] K. Akatan, N. Kaiyrbekov, et al., Microporous aerogel based on microcrystalline cellulose as a sorbent for use as a gas capacitor, *Cellulose* 32 (2025) 8939–8952, <https://doi.org/10.1007/s10570-025-06766-7>.
- [27] Ainur Battalova, Kydyrmolla Akatan, Sana Kabdrakhmanova, Zhanar Ibraeva, Beisebekov Madiar, Shaimardan Esbol, Kantay Nurgamit, Asylan Dzhumanazarova, K.S. Joshy, Sabu Thomas. Isolation of cellulose nanofibers from sunflower seed husks via formic acid hydrolysis. 2024. <https://doi.org/10.1002/pol.20240485>.
- [28] A. Battalova, Z. Ibraeva, S. Kabdrakhmanova, K. Akatan, E. Shaimardan, A. Demeukhan, A. Tursyngazykyzy, M. Beisebekov, A. Maussumbayeva, Comparative characteristics of microcrystalline cellulose obtained from the rice waste production of Kazakhstan, *Chem. Bull. Kazakh Natl. Univ.* 113 (4) (2024) 14–23, <https://doi.org/10.15328/cb1386>.
- [29] K. Akatan, N. Kaiyrbekov, A. Demeukhan, A. Battalova, E. Shaimardan, M. Beisebekov, F. Kholiya, Zh Ibraeva, S. Kabdrakhmanova, Synthesis of Cellulose based Hydrogel with High Absorption Capacity, *Int. J. Biol. Chem.* 18 (1) (2025) 150, <https://doi.org/10.26577/IJBCh202518115>.
- [30] W. Rewini Kunusa, I. Isa, L.A.R. Lalliy, H. Iyabu, FTIR, XRD and SEM analysis of microcrystalline cellulose (MCC) fibers from corn cob in alkaline treatment, *J. Phys. Conf. Ser.* 1028 (2018) 012199, <https://doi.org/10.1088/1742-6596/1028/1/012199>.
- [31] Kian, L.K., et al. (2017). Isolation and characterization of microcrystalline cellulose from rose fiber. *Carbohydrate Polymers*. <https://doi.org/10.1016/j.jbiomac.2017.05.135>.
- [32] M.T. Baker, O.S. Oguntayo, Physical and spectroscopic characterization of the microcrystalline cellulose derivatives from corn cob and Daniella oliveri wastes, *J. Theor. Comput. Sci. Appl.* 10 (1) (2023) 31–38, <https://doi.org/10.18596/jotcsa.1107627>.
- [33] Z. Ahmad, et al., Isolation and characterization of microcrystalline cellulose (MCC) from rice husk (RH) //, *MATEC Web Conf.* 47 (2016) 05013.
- [34] H. Kargarzadeh, M. Ioelovich, I. Ahmad, S. Thomas, and A. Dufresne, “Methods for Extraction of Nanocellulose From Various Sources,” in, <https://doi.org/10.1002/9783527689972.ch1>.
- [35] S. Wu, S. Shi, R. Liu, The transformations of cellulose after concentrated sulfuric acid treatment and its impact on the enzymatic saccharification, *Biotechnol. Biofuels* 16 (2023) 36, <https://doi.org/10.1186/s13068-023-02293-4>.
- [36] A.A. Imasheva, S.K. Kabardhamova, Zh.Ye Ibraeva, S.Ye Kudabergenov, K. Akatan, M.B. Abilev, conditions for producing cellulose from wastes of oily cultures, research of morphology and properties (In Kazakh), *Nncrk Bull.* (1) (2020) 35–39, <https://doi.org/10.52676/1729-7885-2020-1-35-39>.
- [37] A.A. Imasheva, S.K. Kabdrakhmanova, K. Akatan, Zh.E. Ibraeva, G. Temirkhanova, M.M. Beisebekov, N. Kantay, A.M. Mausumbaeva, Obtaining cellulose with crystallographic orientation of macromolecules from the husk of a hybrid sunflower, *Nncrk Bull.* (3) (2024) 29–34, <https://doi.org/10.52676/1729-7885-2024-3-29-34>.
- [38] Lau Kia Kian, Mohammad Jawaid, Hidayah Ariffin, Othman Y. Allothman, Isolation and characterization of microcrystalline cellulose from roselle fibers, *Int. J. Biol. Macromol.* 103 (2017) 931–940, <https://doi.org/10.1016/j.jbiomac.2017.05.135>.
- [39] Garima Joshi, Shakti Singh Chauhan, Characteristic differences in cellulose nanofibers isolated from bamboo, eucalyptus wood, bagasse and waste paper, *Mater. Chem. Phys.* 346 (2025), <https://doi.org/10.1016/j.matchemphys.2025.131333>.
- [40] K. Akatan, S. Kabdrakhmanova, T. Kuanyshbekov, et al., Highly-efficient isolation of microcrystalline cellulose and nanocellulose from sunflower seed waste via environmentally benign method, *Cellulose* 29 (2022) 3787–3802, <https://doi.org/10.1007/s10570-022-04527-4>.
- [41] Xiaomeng Liu, Lang Wang, Ying Lei, Xiao Li, Chunzu Cheng, Leixin Yang, Long Jiao, Shuo Yang, Dengkun Shu, Bowen Cheng, Highly sensitivity and wide-range flexible humidity sensor based on LiCl/cellulose nanofiber membrane by one-step electrospinning, *Chem. Eng. J.* 503 (2025) 158018, <https://doi.org/10.1016/j.cej.2024.158018>.
- [42] Fujun Qi, Benkun Qi, Zhaoliang Cui, Xiangrong Chen, Yinhua Wan, Jianquan Luo, Cellulose-based separation membranes: A sustainable evolution or fleeting trend? *Adv. Membr.* 5 (2025) <https://doi.org/10.1016/j.advmem.2025.100153>.
- [43] H. Kargarzadeh, J. Huang, N. Lin, I. Ahmad, M. Mariano, A. Dufresne, S. Thomas, A. Gałęski, Recent developments in nanocellulose-based biodegradable polymers, thermoplastic polymers, and porous nanocomposites, *ProgPolymSci* 87 (2018) 197–227, <https://doi.org/10.1016/j.progpolymsci.2018.07.008>.
- [44] T. Abitbol, A. Rivkin, Y. Cao, et al., Nanocellulose, a Tiny Fiber With Huge Applications, *Curr. Opin. Biotechnol.* 39 (2016) 76–88, <https://doi.org/10.1016/j.copbio.2016.01.002>.
- [45] C. Miao, W.Y. Hamad, Cellulose reinforced polymer composites and nanocomposites: a critical review, *Cellulose* 20 (2013) 2221–2262, <https://doi.org/10.1007/s10570-013-0007-3>.
- [46] Rasaq S. Abolore, Swarna Jaiswal, Amit K. Jaiswal, Green and sustainable pretreatment methods for cellulose extraction from lignocellulosic biomass and its applications: A review, *Carbohydr. Polym. Technol. Appl.* 7 (2024), <https://doi.org/10.1016/j.carpta.2023.100396>.
- [47] F. Baraka, X. Erdocia, I. Velazco-Cabral, et al., Impact of deep eutectic solvent pretreatment on the extraction of cellulose nanofibers, *Cellulose* 31 (2024) 9645–9660, <https://doi.org/10.1007/s10570-024-06185-0>.
- [48] An. Nang Nguyen, Long Hoang Tran, Ha-Chi V. Yoshimura, Kimio Tran, Tap Duy Van Le, Hieu Nguyen, T. Ngoc-Uyen, Cellulose nanocrystals extracted from rice husk using the formic/peroxyformic acid process: isolation and structural characterization, *RSC Adv.* 14 (2024) 2048–2060, <https://doi.org/10.1039/D3RA06724F>.
- [49] P.M. Sabura Begum, et al., Valorization of avocado peel waste: extraction and characterization of cellulose nanofibers for multifunctional applications, *Biomass.. Conv. Bioref.* 15 (2025) 12789–12800, <https://doi.org/10.1007/s13399-024-06065-4>.
- [50] Juan Pablo Castaneda-Nino, Jose Hermisul Mina-Hernández, Jose Fernando Solanilla-Duque, Effect of cellulose nanofibers and plantain peel fibers on mechanical, thermal, physicochemical properties in bio-based composites storage

- time, Results Eng. 25 (2025) 104185, <https://doi.org/10.1016/j.rineng.2025.104185>.
- [51] Hiquang Gong, Jinsong Zeng, Pengfei Li, Bingyang Liu, Daxian Cao, Bin Wang, Jun Xu, Wenhua Gao, Formic-oxalic synergy unlocks bamboo nanocellulose with high-thermal-stability and superior dispersion, Int. J. Biol. Macromol. 308 (2) (2025) 142511, <https://doi.org/10.1016/j.ijbiomac.2025.142511>.
- [52] A.K Battalova, S.K. Kabdrakhmanova, K. Akatan, M.M. Beisebekov, N. Kantay, Zh. E. Ibraeva, A.M. Mausumbayeva, L.B. Merck, Features of microcrystalline cellulose produced from sunflower seeds of different oil content, Int. J. Biol. Chem. 16 (2023), <https://doi.org/10.26577/ijbch.2023.v16.i1.08>.