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PRODUCTION OF SUSTAINABLE FIBROUS MATERIAL FROM RENEWABLE RESOURCE

Abstract: The article comprehensively studies the possibility of obtaining microcrystalline cellulose (MCC) from the husk of sorghum (SbH) (Sorghum Bicolor), an agricultural crop growing in the country. The research comprehensively studied the effective parameters for the production of MCC from SbH, its yield and quality characteristics, as well as physicochemical properties. When using a hydromodule for the extraction of MCC from SbH at a SbH/peroxyacetic acid ratio of 1:16 g/ml, the yield of MCC was high and reached 57.24%. The amount of α-cellulose in the obtained MCC was 61.32%. It has been established that the yield of MCC obtained by this organosolvent oxidation method is 10-25% higher than with the traditional method of alkaline treatment. The obtained MCC can be used as an ingredient in paper and cardboard production, pharmaceuticals, and composite materials.

Keywords: Agricultural waste, sorghumbicolor husks, microcrystalline cellulose, organosolvent oxidation, FTIR, crystal structure.

Introduction

In line with sustainable development goals, interest in biomaterials derived from environmentally friendly and renewable raw materials is increasing every day. Cellulose fibers are one such natural polymer. It is the most abundant carbohydrate in plant cells. It is widely used in industry as an environmentally friendly, biocompatible material. Cellulose and its derivatives are of great importance in the food industry, paper and textile products, pharmaceuticals and biomedicine. (Roohallah et al., 2024). However, existing cellulose fibers are still produced from traditional raw materials (wood, cotton fiber). This process uses forest resources, causing enormous damage to the environment (Shehrawat & Sindhu, 2015). In this regard, obtaining cellulosic material from agricultural waste and renewable raw materials becomes a pressing issue for the transition to technology compatible with the circular economy within the framework of the UN Sustainable Development Goals No. 9 “Industry, Innovation and Infrastructure”, No. 12 “Responsible Consumption and Production” and No. 13 “Actions related to environmental change”.

In this regard, agricultural waste has great potential as an alternative source of cellulose fibers (Battalova et al., 2024b; Battalova et al., 2023; Imasheva et al., 2020; Khalil et al., 2012; Li et al., 2020; Rasaq et al., 2024). In the study of Jiang et al. (2019), high-purity cellulose fibers were obtained from rice husk and wheat straw by alkaline delignification. The studies (Battalova et al., 2024b; Battalova et al., 2023; Imasheva et al., 2020) investigated the possibility of obtaining microcrystalline cellulose (MCC) from agricultural waste such as sunflower husk (SH) and rice husk (RH) by organosolvent oxidation. The MCC yield was 43.49–52.58%, and the α-cellulose content was 40.5–65.57%. Similarly, in (Battalova et al., 2024a), the possibility of obtaining cellulose nanofibers (CNF) from agricultural waste – SFH by formic acid hydrolysis was investigated, resulting in CNF with a yield of 71% and a zeta potential of -15.8 mV. In (Akatan et al., 2022; Shahi et al., 2020), the authors of the study developed biofilms based on nanocellulose obtained from sugar beet pulp and residues accumulated during sunflower oil production and investigated their potential use as a “green” material. The results of

this study showed that agricultural waste is a potential source of raw material for the obtaining of cellulose-based biopolymers.

In this regard, sorghum husk (*Sorghum Husk*) has recently been studied as a promising raw material for pulp production (Sani et al., 2020; Waghmare et al., 2018). Sorghum is one of the five most important grain crops grown in sustainable agriculture worldwide. It is grown primarily in tropical and arid climates (Charyulu et al., 2024; Dalton & Hodjo, 2020). Sorghum is highly nutritious and contains a variety of bioactive compounds such as sterols, carotenoids, phenolic acids, and flavonoids, which are known for their potent antioxidant, anti-inflammatory, cholesterol-lowering, and anticancer activities (Bakari et al., 2023). A significant share of global sorghum seed production occurs in the United States, Nigeria, Sudan, Mexico, India, and Ethiopia (Pereverzin 2016). Annual global production is approximately 70 million tons. Sorghum production in our country, according to 2024 data, increased by 231% compared to previous years and is intended for the feed and food industries (West Kazakhstan Region, Taskala District Akimat [WKR TDA], n.d.). This indicates that, in addition to waste from manufacturing plants, the share of agricultural waste is significantly higher.

Sorghum hulls (SH), formed during sorghum processing, account for 15% of the total grain weight. Its composition has been shown to be rich in carbohydrates, such as polysaccharides, phenols, and proteins, as well as silicon (Dong et al., 2019; Kshirsagar et al., 2017).

Research has shown that sorghum pulp is a potential raw material for the production of cellulose fibers (da Costa et al., 2024; Kumar et al., 2015; Yulianto et al., 2020). The results indicate the possibility of obtaining cellulose in the range of 30-40%. However, these studies used a two-stage extraction method. Alkaline treatment was applied first, followed by hydrogen peroxide. This, in turn, increases the time and cost of extraction, as well as the amount of additional process fluids. Therefore, the efficiency and environmental safety of the method used for processing cellulose raw materials are of great importance.

Therefore, the aim of this study was to isolate high-quality cellulose microcrystals through the efficient utilization of husks – a byproduct of sorghum production grown in the southern region of the Republic of Kazakhstan. The resulting cellulose fibers have high potential for use as key components in the production of paper and cardboard, as well as biocomposite materials.

Materials and methods

Materials

Sorghum bicolor husks were obtained from southern region of Kazakhstan. Hydrogen peroxide (35%, H_2O_2), acetic acid (≥44%, CH_3COOH), potassium permanganate (99%, $KMnO_4$), ethanol (96%, C_2H_5OH), sulfuric acid (98%, H_2SO_4), orthophosphoric acid (98%, H_3PO_4), sodium hydroxide (≥99%, NaOH), potassium bichromate (≥99%, $K_2Cr_2O_7$), sodium thiosulfate (99%, $Na_2S_2O_3$), potassium iodide (≥99%, KI) and starchwere provided by Sigma-Aldrich (Bangalore, India). All other reagents were of analytical grade and were utilized without further purification.

Methods

Preparation of raw materials

To obtain microcrystalline cellulose, a variety of SbH raw material called “Sorghum bicolor” grown in the southern region of Kazakhstan was used. In sorghum, the fruit is a caryopsis. The sorghum fruit coat is the membrane in which the seed coat is fused with the pericarp. The pericarp is the outer part of the fruit, developing from the wall of the ovary. The pericarp is divided into three layers: 1) Exocarp (outer layer): consists of small, tightly adjacent cells with thickened walls covered with a waxy coating. The cells are prismatic in shape, elongated perpendicular to the surface. The cells are pigmented dark red, brown. 2) Mesocarp (middle layer): is represented by loose tissue consisting of parenchymatous cells. The cells are larger than in the exocarp, with thin cellulose walls. 3) Endocarp (inner layer): thin, tightly adjacent to the seed coat. The pericarp is rich in grain fibers, primarily cellulose, hemicellulose, starch, and lignin. The seed coat is the membrane surrounding the embryo. The seed coat is fused with the pericarp, forming a single covering (Badigannavar et al., 2018; Makhatov et al., 2017).

To remove residual substances from Sorghum bicolor husks, they were pre-washed with distilled water in a flask with a rotary condenser at 90±2°C for 60 minutes with stirring. They were then filtered through filter paper and dried in an oven at 60±2°C for 5 hours until constant weight was reached.

The raw material samples from Sorghum bicolor seed husks used in the study were conventionally referred to as sorghum bicolor husks (SbH), and the MCC obtained from them was referred to as MCC_{SbH}.

Preparation of peroxyacetic acid (PAA)

Peroxyacetic acid (PAA) was used to extract MCC from the raw material. The concentrations of the starting reagents for PAA production were re-

duced by half and prepared according to the method described in (Akatan et al., 2022; Battalova et al., 2024b; Battalova et al., 2023; Imasheva et al., 2020). The resulting PAA was stored in a freezer at 5±0.5°C.

Chemical composition of sorghum bicolor husks (SbHs)

The mineral composition of raw SbH was determined using X-ray fluorescence (XRF) spectroscopy on an EDX3600H (Skyray Instruments, China). In order to determine the amount of water-soluble hydrocarbons, 5 ± 0.001 g of raw material was heated for 2 h, with continuous stirring in 50 mL of distilled water. After this process, the SbHs were filtered, dried at 80°C ± 2°C for 5 h, and then the mass was measured on an LV 210-A analytical scale (Sartogosm, Russia). The quantity of hydrolyzable hydrocarbons in water was determined from the difference between the initial mass and the final mass.

To assess the ash content of SbH, 5 g of MCC was incinerated in an 8.2/1100 L muffle furnace (SNOL, Lithuania) at a temperature of 700°C ± 5°C for a duration of 90 minutes. The weight of the resulting ash (SiO_2) was recorded using an LV 210-A analytical scale (Sartogosm, Russian) until an average measurement was achieved. The ash content was calculated by subtracting the final mass from the initial mass.

Preparation of MCC from SbH by organosolvent oxidation and determination of its yield

The synthesis of microcrystalline cellulose (MCC) based on SbH was carried out according to the method described in (Akatan et al., 2022; Battalova et al., 2024b; Battalova et al., 2023; Imasheva et al., 2020). The SbH and PAA ratio was extracted under hydromodule conditions with proportions of 1:10, 1:12, 1:14, 1:16, 1:18, and 1:20 g/ml. To obtain MCC, the raw material and delignifying agent were boiled in a flask fitted with a rotary condenser at a temperature of 90±2°C for 90 minutes, with continuous and vigorous stirring. The resulting MCC solution was then cooled to 25±2°C, filtered using a paper filter, and neutralized with distilled water until reaching a pH of 7 (Fig. 1). The resulting product was dried at 80±2°C for 6 hours to constant weight, weighed on an analytical balance with an accuracy of 0.0001 g, and the yield was calculated using the formula:

$$\eta = (m_{SbH}-m_{MCC})/m_{SbH}\times 100\% \quad (1)$$

where, m_{SbH} is the mass of SbH, g; m_{MCC} – the mass of obtained MCC, g.

Figure 1
Scheme of obtaining MCC from SbH by the organosolvent oxidation

Determination of microcrystalline cellulose quality indicators

The moisture content of the synthesized MCC was determined using the GOST-16932 method (state standard), the α-cellulose content – using the GOST-6840 method, the residual lignin content – using the GOST-11960 method, the hemicellulose content – using the GOST-9002 method, and the ash content – using the GOST 18461-93 method.

Scanning electron microscopy (SEM)

The surface characteristics of the MCC were analyzed using a Quanta 200i 3D SEM from FEI in the Netherlands. The analysis was conducted in high vacuum mode with a secondary electron detector operating at an accelerating voltage of 15 kV. To enhance electron transfer, the MCC surface was coated with gold nanoparticles, and the samples were affixed to aluminum pins using carbon tape.

FTIR spectroscopy

The chemical composition of the samples was analyzed using an FTIR FT-801 IR spectrometer (Simex, Russia). This analysis employed an internal and variable-diffuse reflection technique, achieving an accuracy of 1 cm^{-1} across a wave number range of 450–4700 cm^{-1}. The procedure involved 50 scans at a temperature of 25±20°C, with the sample placed on the attachment's surface.

X-ray diffraction

The structural and phase properties of the samples were investigated by X-ray diffraction (XRD) analysis using monochromatic copper radiation (CuKα, λ = 0.1542 Å) on an X´PertPRO diffractometer (Malvern Panalytical Empyrean, Netherlands). The diffraction patterns were recorded within a 2θ range of 10–45° with a scanning increment of 0.02°. The X-ray tube was operated at 45 kV and 30 mA, while the counting time for each step was set to 0.5 s. A universal aluminum sample holder (PW1172/01) was employed during the measurements. The obtained diffraction data were interpreted using the ICDD PDF-4/AXIOM X-ray diffraction database.

Thermogravimetric analysis (TGA)

The thermal stability of the obtained samples was evaluated under an argon atmosphere using a LabSys Evo differential thermogravimetric analyzer (Setaram, France). The measurements were carried out over a temperature range of 30 ± 5 to 800 ± 5°C at a heating rate of 10 ± 1°C/min. The sample mass used for analysis was 20 ± 2 mg.

Results and discussion

Chemical composition of SbH

A quantitative analysis of the chemical composition of the initial SbH raw material was carried out, and the obtained results are presented in Table 1.

Table 1
Chemical composition of the initial SbH sample

Compounds	Quantity, %
K	16.3
P	2.55
Ca	3.76
Si	65.8
Al	4.23
Fe	2.28
Mn	0.08
Ni	0.06
Cu	0.07
Zn	0.08
Cr	0.18
Ti	0.57
Cl	1.84
S	1.94
Water soluble hydrocarbons	0.26

The MCC_{SbH} from SbH by organosolvent oxidation method

Figure 2a shows the initial material—sorghum seed husk (SbH), and Figure 2b shows the obtained MCC_{SbH}. The initial material, SbH, had a reddish-brown color, while the obtained MCC_{SbH} had a flaky, fibrous structure and a light grayish tint.

Figure 2
Sorghumbicolor husks and MCC samples from SbH: a – SbH; b – MCC_{SbH} (hydromodule 1:16)

The yield and quality indicators of MCC_{SbH}

The yield and quality indicators of MCC_{SbH} obtained at different SbH:PAA g/ml ratios under "mild" conditions of the organosolvent oxidation method are presented in Table 2. The results of the study showed that the yield of SbH:PAA obtained at an SbH:PAA ratio of 1:16 g/ml was 57.24%, moisture content was 4.23%, α-cellulose content was 61.32%, residual lignin was 0.71%, hemicellulose was 0.55%, and SiO_2 was 29.78%. Since the yield and quality indicators determined in other hydromodules were relatively low, 1:16 g/ml was recognized as the effective hydromodule. A study (Reddy & Yang, 2007) found that up to 35–40% MCC can be obtained from sorghum stems and leaves using alkaline and acid hydrolysis. It was found that at an

SbH:PAA ratio of 1:16 g/ml, the yield of MCC_{SbH} obtained using the organosolvent method increases by 10.6–25%. In addition, an additional bleaching process occurs for the cellulose obtained using traditional alkaline and mineral acids. This increases economic and energy costs. It should be noted that the organosolvent oxidation method does not require additional technological processes and increases the possibility of extracting cellulose under "mild" conditions.

Table 2
Effect of SbH:PAA ratio on MCC_{SbH} quality

SbH:PAA, g/ml	Quality indicators of MCC_{SbH}, %					
	Yield of MCC	Humidity	α-cellulose	Residual lignin	Hemi-cellulose	Ash content (SiO_2)
1:10	50.81±2	4.82±0.5	44.61±3	0.68±0.5	0.48±2	30.27±0.5
1:12	52.25±2	4.41±0.5	54.20±3	0.65±0.5	0.48±2	29.82±0.5
1:14	54.60±2	4.44±0.5	61.02±3	0.59±0.5	0.66±2	30.65±0.5
1:16	57.24±2	4.23±0.5	61.32±3	0.71±0.5	0.55±2	29.78±0.5
1:18	53.22±2	4.26±0.5	55.31±3	0.70±0.5	0.63±2	29.97±0.5
1:20	52.61±2	4.16±0.5	52.91±3	0.76±0.5	0.56±2	30.19±0.5

FTIR spectroscopy

Figure 3 presents the FTIR spectrum of the initial SbH material. The broad absorption band observed in the range of 3500–3000 cm^{-1} is attributed to the stretching vibrations of O–H groups. The peaks at 2920 cm^{-1} and 2850 cm^{-1} correspond to C–H stretching vibrations typical of polysaccharides (Zghari et al., 2018). In addition, the absorption bands in this region are associated with silanol (Si–OH) groups present in SbH (Cichosz & Masek, 2020; Zghari et al., 2018). The bands at approximately 1700 cm^{-1} and 1600 cm^{-1} are related to acetyl groups of hemicellulose and carbonyl groups within the lignin structure (Battalova et al., 2024b; Battalova et al., 2023; Kacuráková et al., 2002). The absorption region between 1600 cm^{-1} and 1510 cm^{-1} corresponds to the vibrations of aromatic rings in lignin (Battalova et al., 2024b; Battalova et al., 2023; Imasheva et al., 2020). The peaks in the range of 1250–1020 cm^{-1} are assigned to β-1,4-glycosidic bonds as well as C–O–C and C–O–H vibrations of cellulose (Akatan et al., 2025; Battalova et al., 2024b; Battalova et al., 2023; Guzman et al., 2017; Imasheva et al., 2020). These results confirm that sorghum husk is a lignocellulosic raw material rich in cellulose, hemicellulose, lignin, and other hydrocarbon compounds.

Comparative IR spectra of the chemical structure of MCC_{SbH}, obtained in different ratios, are shown in Figure 4. All spectra of the MCC_{SbH} samples contain signals of the main functional groups characteristic of cellulose. In particular, a broad and intense stretching vibration of the O–H groups is recorded in the region of 3300–3400 cm^{-1}. This indicates a large number of hydroxyl groups and the presence of intermolecular hydrogen bonds (Cichosz & Masek, 2020). In addition, the absorption bands at 2895 cm^{-1} are attributed to the symmetric and asymmetric stretching vibrations of C–H bonds (Cichosz & Masek, 2020; Zghari et al., 2018).

Several characteristic peaks associated with the structural features of cellulose are observed within the 1800–800 cm^{-1} region. The band at approximately 1430 cm^{-1} corresponds to the deformation vibrations of C–H bonds in the crystalline regions of cellulose and is commonly used to evaluate the crystallinity degree of cellulose (Kacuráková et al., 2002; Zghari et al., 2018). The peak at 1370 cm^{-1} is assigned to C–H deformation vibrations, while the intense band at 1160 cm^{-1} is related to the stretching vibrations of C–O–C bonds within the pyranose ring, characteristic of β-1,4-glycosidic linkages. Furthermore, the peaks observed in the range of 1050–1030 cm^{-1} correspond to the stretching vibrations of C–O bonds in the aromatic ring, confirming the polysaccharide nature of cellulose (Kacuráková et al., 2002; Zghari et al., 2018). The absorption peak at 895 cm^{-1} indicates the presence of β-glycosidic bonds and reflects the crystalline structure of cellulose (Battalova et al., 2024b; Battalova et al., 2023; Cichosz & Masek, 2020). Meanwhile, weak absorption bands at 1508.9 cm^{-1} and 1734.4 cm^{-1} are associated with the stretching vibrations of aromatic C=C bonds in

residual lignin, as well as C=O vibrations of acetyl and ester groups in hemicellulose present in MCC (Akatan et al., 2025; Guzman et al., 2017; Imasheva et al., 2024). This clarifies the quantitative values of residual lignin and hemicellulose given in Table 2. The obtained results (Bakari et al., 2023; Charyulu et al., 2024) are in good agreement with the FTIR absorption spectra of microcrystalline cellulose obtained from sorghum residues are presented in Figure X. A noticeable decrease in the intensity of the absorption peaks was observed with increasing PAA content in the SbH:PAA ratio (g/mL). This behavior can be attributed to the progressive hydrolysis process, which weakens the glycosidic bonds in the cellulose macromolecule and alters the symmetry of its molecular structure (Battalova et al., 2024b; Battalova et al., 2023; Imasheva et al., 2024; Zghari et al., 2018). In particular, the reduced intensity of the peaks corresponding to O–H and C–H bond vibrations in the samples with SbH:PAA ratios of 1:18 and 1:20 indicates structural deformation of the cellulose matrix.

Figure 3
FTIR spectra of SbH

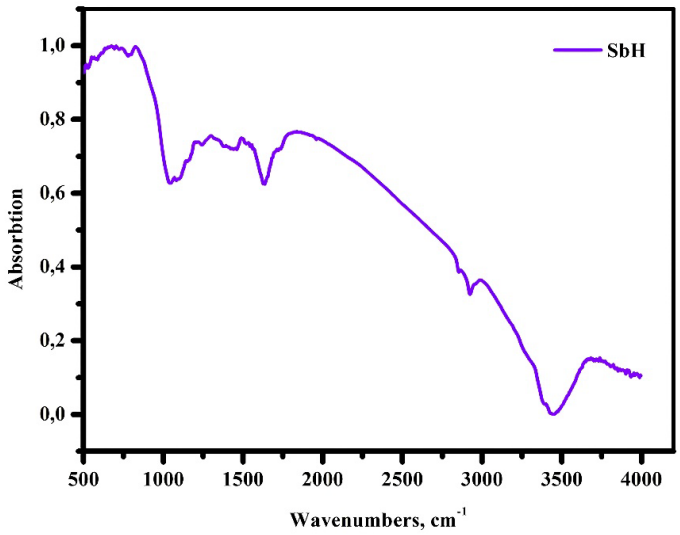
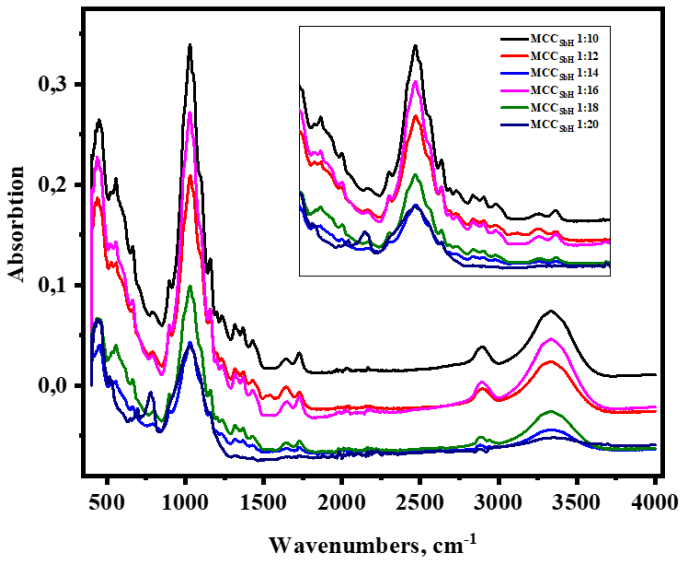


Figure 4
FTIR spectra of MCC_{SbH} in different ratios



X-ray diffraction

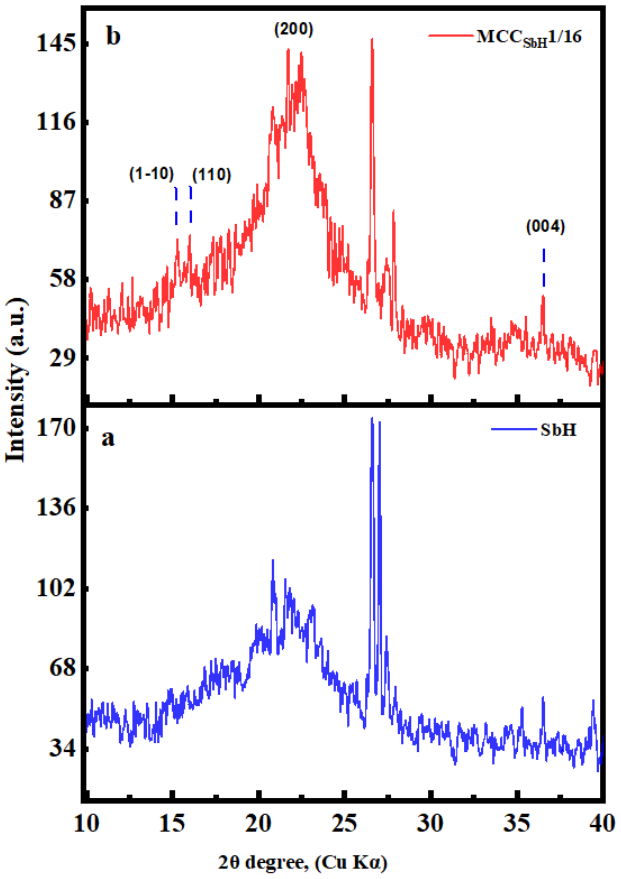
The results of comparative X-ray diffraction analysis carried out to determine the structural and phase composition of the initial SbH raw material and MCCSbH samples (1:16 g/ml) obtained in the effective hydromodulus are shown in Figures 5a and 5b. The crystalline structure of the samples was determined in accordance with the studies (French, 2014; Imasheva et al., 2024; Podgorbunskikh et al., 2018). Peaks 2θ=16.1°(110), 22.4°(200), 34.1°(004) recorded in the diffraction pattern of SbH (Figure 5a) indicate an amorphous and semi-crystalline structure of sorghum fiber. Moreover, an intense signal at the diffraction reflection angle 2θ=27.2°(101) is characteristic of the signal of silica in the raw material (Bolatkan et al., 2025). It is evident that this corresponds to the elemental composition and quality indicators of the initial SbH raw material given in Tables 1 and 2.

The X-ray diffraction (XRD) pattern of MCCSbH exhibits characteristic diffraction peaks at 2θ = 15.3° (110), 22.3° (200), and 34.8° (004), confirming the crystalline structure of MCCSbH (Fig. 5b). The observed diffraction peaks indicate that the crystal structure of MCC is a double-chain monoclinic form corresponding to cellulose Iβ (Imasheva et al., 2024; Kacuráková et al., 2002). These results confirm the effective removal of lignin, hemicellulose, and other non-cellulosic hydrocarbon components during the extraction of cellulose from sorghum husk. Furthermore, the intensity of the diffraction peak associated with silica at 2θ = 27.2° (101) decreased by approximately 1.2 times in MCC compared to the initial SbH material. This reduction may be attributed to the partial hydrolysis of silica occurring during the organosolvent oxidation treatment of the original SbH raw material.

The results of the X-ray diffraction analysis demonstrated that the initial SbH material possesses a predominantly semicrystalline or amorphous structure, whereas the microcrystalline cellulose obtained after treatment exhibits well-defined crystalline characteristics. These findings are in good agreement with previously reported studies (Akatan et al., 2022; Battalova et al., 2024b; Battalova et al., 2023; Imasheva et al., 2024; Imasheva et al., 2020).

Figure 5

XRD diffractions of: a – SbH; b – MCCSbH

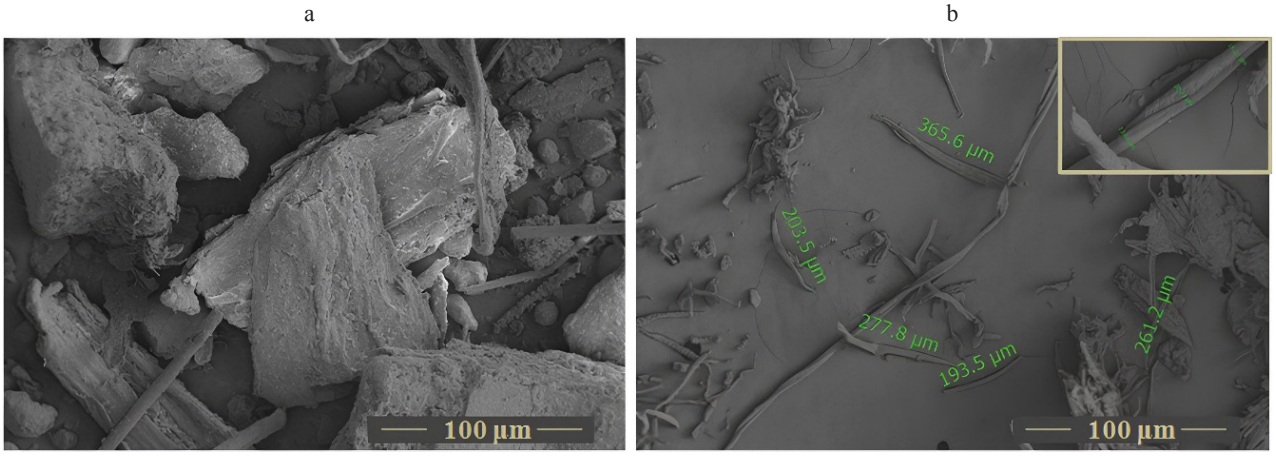


SEM analysis

Figure 6 shows the surface morphology of the initial material SbH (Fig. 6a) and MCCSbH obtained at an effective hydromodulus of 1:16 g/ml (Fig. 6b). It is evident that the initial material – sorghum husk – has a multilayer structure. It can be observed that the cellulose fibers on the surface of the husk are densely covered with silica, waxes, pectin, lignin, hemicellulose, and other hydrocarbon compounds, which contribute to the high mechanical strength and hydrophobic properties of the material (Li et al., 2018; Zhang et al., 2018). This indicates that the composition of the starting material SbH, shown in Figure 3, is consistent with the results obtained by IR analysis.

In Fig. 6b, the surface morphology of MCCSbH extracted at an effective hydromodulus of 1:16 g/ml has a ribbon-like shape consisting of fibrils of varying sizes. The length of the cellulose fibers is approximately 193.5–993.0 μm, and the width is 13.66–21.55 μm (Fig. 6b). This indicates a fibrous structure of MCCSbH fragments. In addition, roughness of the surface of the cellulose fibers is observed. This is explained by partial hydrolysis of the amorphous regions and the release of crystalline fibers during hydrolysis (Cichosz & Masek, 2020). In the study (Zhang et al., 2018), it was found that the rough surface structure of the cellulose fibers indicates a large surface area and high sorption properties.

Figure 6
SEM images of: a – SbH; b – MCCSbH



Thermogravimetric analysis of MCC_{SbH}

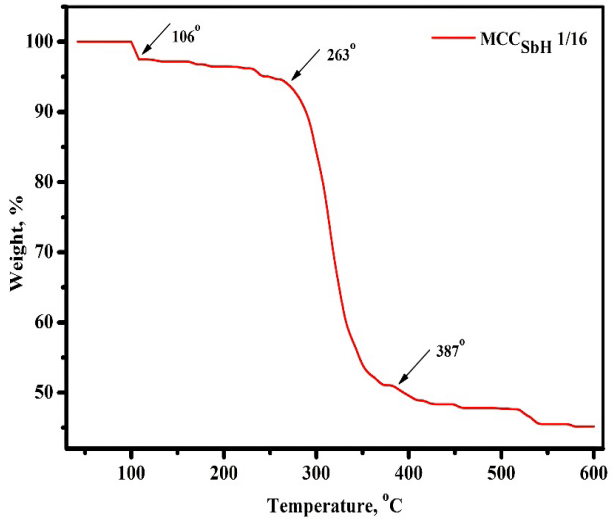
The resulting thermogram of MCC_{SbH} is shown in Figure 7. According to the obtained results, initial mass loss was observed in the range from 25 to 106°C. It is evident that over this period, the mass of the material decreased by approximately 3.3% due to the evaporation of moisture from the air and water sorbed by the sample.

The first phase of thermal degradation begins at temperatures from 106 to 263°C, and in this temperature range, mainly residual hemicellulose and low molecular weight fractions of lignin undergo thermal degradation (Battalova et al., 2024b). The studies (Chen et al., 2011; Henrique et al., 2015) have shown that thermal decomposition of lignocellulosic biomass occurs in the range from 150 to 500°C. The research (Wang et al., 2021) has shown that most of the hemicellulose decomposes gradually and over a wide temperature range from 150 to 350°C, cellulose at 275–350°C, and lignin at 250–500°C.

The major thermal degradation of MCC_{SbH} occurred within the temperature range of 263–387°C. During this stage, the MCC_{SbH} sample lost approximately 49.5% of its initial mass, indicating the thermal decomposition of high-molecular-weight cellulose chains (Hou et al., 2017). This degradation process led to the formation of volatile compounds and the breakdown of the organic structure of MCC_{SbH}.

During the thermal degradation of cellulose at temperatures of 387–600°C, combustion of intermediate products occurs (Hou et al., 2017). At this stage, 54% of the total mass of the sample undergoes thermal degradation, and the residual substance – silica (SiO_2) – accounts for approximately 35%. This is consistent with the results of determining the mineral composition and ash content of the initial material and MCC_{SbH}, presented in Tables 1 and 2.

Figure 7
TGA curve of MCC_{SbH}



Conclusion

Summarizing the obtained results, it should be noted that in the course of the research work, the possibility of obtaining microcrystalline cellulose (MCC_{SbH}) from sorghum seed husk (SbH) grown in the southern region of Kazakhstan, using the organosolvent oxidation method was studied. The elemental analysis of the feedstock (SbH) showed that its composition is dominated by Si (65.8%), K (16.3%), Ca (3.76%) and P (2.55%). IR analysis showed that it is rich in hydrocarbons. In this regard, it was established that the effective hydromodulus for obtaining MCC_{SbH} from SbH is the SbH:PAA ratio of 1:16 g/ml. The yield of the obtained MCC_{SbH} sample was 57.24%, and the α-cellulose content was 61.32%.

The surface morphology of the obtained MCC_{SbH} sample was found to be fibrous, with a length of approximately 193.5–993.0 μm and a width of 13.66–21.55 μm. Fourier transform infrared (FTIR) spectroscopy analysis demonstrated that the obtained product contained the main functional groups characteristic of MCC and was effectively purified from residual lignin and hemicellulose components. X-ray diffraction analysis revealed that the original SbH sample had a semi-amorphous structure, while the obtained MCC_{SbH} was characterized by a distinct crystalline structure. The cellulose crystal structure was double-chain monoclinic, typical of cellulose Iβ. Furthermore, thermogravimetric analysis (TGA) showed that the thermal stability of the MCC_{SbH} sample was maintained at temperatures up to 263°C.

The obtained results confirm that this material is a promising raw material for producing high-quality microcrystalline cellulose using mild organosolv oxidation. The resulting MCC_{SbH} has significant potential as a promising bio-based raw material for the development of pharmaceuticals, food packaging, and the production of environmentally friendly biodegradable materials.

Author contributions

The manuscript was written through contributions of all authors. Conceptualization – Saule Nauryzova and Sana Kabdrakhmanova; Data curation – Kydyrmolla Akatan and Esbol Shaimardan; Formal analysis – Aidana Imasheva and Ainur Kabdrakhmanova; Investigation – Ainur Battalova and Ansagan Demeukhan; Methodology – Ainur Battalova and Kydyrmolla Akatan; Project administration – Ainur Battalova and Kydyrmolla Akatan; Resources – Ainur Kabdrakhmanova and Ansagan Demeukhan; Supervision – Saule Nauryzova and Sana Kabdrakhmanova; Validation – Saule Nauryzova, Sana Kabdrakhmanova and Kydyrmolla Akatan; Visualization – Saule Nauryzova, Kydyrmolla Akatan and Aibar Yeskaliyev; Writing – original draft – Ainur Battalova; Writing reviewing and editing – Ainur Battalova, Kydyrmolla Akatan and Saule Nauryzova. All authors have read and agreed to the published version of the manuscript.

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Conflict of interest

The authors declare that they have no conflicts of interest.

References

1. Akatan, K., Kabdrakhmanova, S., Kuanyshbekov, T., Battalova, A., Ibraeva, Z., & Beisebekov, M. (2022). Highly-efficient isolation of microcrystalline cellulose and nanocellulose from sunflower seed waste via environmentally benign method. *Cellulose*, *29*(7), 3787–3802. https://doi.org/10.1007/s10570-022-04527-4

2. Akatan, K., Kaiyrbekov, N., Demeukhan, A., Battalova, A., Shaimardan, E., Beisebekov, M., Kholiya, F., Ibraeva, Zh., & Kabdrakhmanova, S. (2025). Synthesis of cellulose based hydrogel with high absorption capacity. *International Journal of Biology and Chemistry*, *18*(1), 150–158. https://doi.org/10.26577/IJBCh202518115

3. Badigannavar, A., Teme, N., de Oliveira, A. C., Li, G., Vaksmann, M., & Viana, V. E. (2018). Physiological, genetic and molecular basis of drought resilience in sorghum [*Sorghum bicolor* (L.) Moench]. *Indian Journal of Plant Physiology*, *23*(4), 670–688. https://doi.org/10.1007/s40502-018-0416-2

4. Bakari, H., Djomdi, Ruben, Z. F., & Ali, M. (2023). Sorghum (*Sorghum bicolor* L. Moench) and its main parts (by-products) as promising sustainable sources of value-added ingredients. *Waste and Biomass Valorization*, *14*(4), 1023–1044. https://doi.org/10.1007/s12649-022-01992-7

5. Battalova, A., Akatan, K., Kabdrakhmanova, S., Ibraeva, Z., Beisebekov, M., Shaimardan, E., Kantay, N., Dzhumanazarova, A., Joshy, K. S., & Thomas, S. (2024a). Isolation of cellulose nanofibers from sunflower seed husks via formic acid hydrolysis. *Journal of Polymer Science*. Advance online publication. https://doi.org/10.1002/pol.20240485

6. Battalova, A., Ibraeva, Z., Kabdrakhmanova, S., Akatan, K., Shaimardan, E., Demeukhan, A., Tursyngazykyzy, A., Beisebekov, M., & Maussumbayeva, A. (2024b). Comparative characteristics of microcrystalline cellulose obtained from the rice waste production of Kazakhstan. *Chemical Bulletin of Kazakh National University*, *113*(4), 14–23. https://doi.org/10.15328/cb1386

7. Battalova, A. K., Kabdrakhmanova, S. K., Akatan, K., Beisebekov, M. M., Kantay, N., Ibraeva, Zh. E., Mausumbayeva, A. M., & Merck, L. (2023). Features of microcrystalline cellulose produced from sunflower seeds of different oil content. *International Journal of Biology and Chemistry*, *16*(1), 78–86. https://doi.org/10.26577/ijbch.2023.v16.i1.08

8. Bolatkan, D., Kerimkulova, A., Beisebekov, M., Akatan, K., Kantay, N., Shaimardan, E., Kukhareva, A., Bukunova, A., & Kabdrakhmanova, S. (2025). Quantitative assessment of the use of Kazakhstan montmorillonite clays as sorbent carriers for pharmaceutical substances. *Physical Sciences and Technology*, *12*(1-2), 121–133. https://doi.org/10.26577/phst202512112

9. Charyulu, D. K., Afari-Sefa, V., & Gumma, M. K. (2024). Trends in global sorghum production: Perspectives and limitations. In E. Habyarimana, M. A. Nadeem, F. S. Baloch, & N. Zencirci (Eds.), *Omics and biotechnological approaches for product profile-driven sorghum improvement* (pp. 1–21). Springer. https://doi.org/10.1007/978-981-97-4347-6_1

10. Chen, X., Yu, J., Zhang, Z., & Lu, C. (2011). Study on structure and thermal stability properties of cellulose fibers from rice straw. *Carbohydrate Polymers*, *85*(1), 245–250.

11. Cichosz, S., & Masek, A. (2020). IR study on cellulose with the varied moisture contents: Insight into the supramolecular structure. *Materials*, *13*(20), 4573.

12. da Costa, D. P. S. T., Dewi, I. A., Fahma, F., Hamid, Z. A. A., & Suryanegara, L. (2024). Isolation and characterization of cellulose nanofibers from sweet sorghum with different ultrafine grinding modes. *AIP Conference Proceedings*, *2973*(1), 040009. https://doi.org/10.1063/5.0184730

13. Dalton, T. J., & Hodjo, M. (2020). Trends in global production, consumption, and utilization of sorghum. In V. A. Tonapi, H. S. Talwar, A. K. Are, B. V. Bhat, C. R. Reddy, & T. J. Dalton (Eds.), *Sorghum in the 21st century: Food – fodder – feed – fuel for a rapidly changing world* (pp. 3–15). Springer. https://doi.org/10.1007/978-981-15-8249-3_1

14. Dong, M., Wang, S., Xu, F., & Li, Q. (2019). Pretreatment of sweet sorghum straw and its enzymatic digestion: Insight into the structural changes and visualization of hydrolysis process. *Biotechnology for Biofuels*, *12*, 276. https://doi.org/10.1186/s13068-019-1613-6

15. French, A. D. (2014). Idealized powder diffraction patterns for cellulose polymorphs. *Cellulose*, *21*(2), 885–896. https://doi.org/10.1007/s10570-013-0030-4

16. Guzman, J. T., Sillard, C., Plackett, D., Szabo, P., Bras, J., & Daugaard, A. E. (2017). Chemically extracted nanocellulose from sisal fibres by a simple and industrially relevant process. *Cellulose*, *24*(1), 107–118.

17. Henrique, M. A., Neto, W. P. F., Silvério, H. A., Martins, D. F., Gurgel, L. V. A., & Barud, H. S. (2015). Kinetic study of the thermal decomposition of cellulose nanocrystals with different polymorphs, cellulose I and II, extracted from different sources and using different types of acids. *Industrial Crops and Products*, *76*, 128–140.

18. Hou, X., Fang, F., Guo, X., Wizi, J., Ma, B., Tao, Y., & Yang, Y. (2017). Potential of sorghum husk extracts as a natural functional dye for wool fabrics. *ACS Sustainable Chemistry & Engineering*, *5*(6), 4589–4597. https://doi.org/10.1021/acssuschemeng.6b02969

19. Imasheva, A. A., Kabdrakhmanova, S. K., Akatan, K., Ibraeva, Zh. E., Temirkhanova, G., Beisebekov, M. M., Kantay, N., & Mausumbaeva, A. M. (2024). Obtaining cellulose with crystallographic orientation of macromolecules from the husk of a hybrid sunflower. *Bulletin of the National Nuclear Corporation of the Republic of Kazakhstan*, *(3)*, 29–34. https://doi.org/10.52676/1729-7885-2024-3-29-34

20. Imasheva, A. A., Kabdrakhmanova, S. K., Ibraeva, Zh. E., Kudaibergenov, S. E., Akatan, K., & Abilev, M. B. (2020). Study of the possibility of obtaining cellulose from oilseed residues by the organosolvent method. *Bulletin of the National Nuclear Corporation of Kazakhstan*, *1*(81), 35–38.

21. Jiang, F., Wang, T., Zhang, Y., & Dong, S. (2019). Extraction of cellulose from rice husk and wheat straw for bio-based materials. *Industrial Crops and Products*, *137*, 66–74.

22. Kacuráková, M., Smith, A. C., Gidley, M. J., & Wilson, R. H. (2002). Molecular interactions in bacterial cellulose composites studied by 1D FT-IR and dynamic 2D FT-IR spectroscopy. *Carbohydrate Research*, *337*(12), 1145–1153.

23. Khalil, H. P. S. A., Bhat, A. H., & Ireana Yusra, A. F. (2012). Agricultural biomass and residues as potential sources for nanocellulose fibers: Preparation and applications. *Carbohydrate Polymers*, *88*(4), 1079–1085.

24. Kshirsagar, S. D., Bhalkar, B. N., & Waghmare, P. R. (2017). Sorghum husk biomass as a potential substrate for production of cellulolytic and xylanolytic enzymes by *Nocardiopsis* sp. KNU. *3 Biotech*, *7*(3), 163. https://doi.org/10.1007/s13205-017-0800-z

25. Kumar, C. G., Kumar, M. P., & Gupta, S. (2015). Isolation and characterization of cellulose from sweet sorghum bagasse. *Sugar Tech*, *17*(4), 395–403. https://doi.org/10.1007/s12355-014-0339-9

26. Li, T., Chen, C., Brozena, A. H., Zhu, J. Y., Xu, L., Driemeier, C., Dai, J., Rojas, O. J., Isogai, A., Lawoko, M., Wagberg, L., Yu, H., & Hu, L. (2020). Nanocellulose from agricultural waste for sustainable materials development. *ACS Sustainable Chemistry & Engineering*, *8*(12), 4937–4956.

27. Li, M., Yan, G., Bhalla, A., Maldonado-Pereira, L., Russell, P. R., & Ding, S. Y. (2018). Physical fractionation of sweet sorghum and forage/energy sorghum for optimal processing in a biorefinery. *Industrial Crops and Products*, *124*, 607–616.

28. Makhatov, Zh. B., Latif, A. S., Saparbekova, A. A., Isaev, E. B., Makhatov, B. K., & Bukharbaeva, A. E. (2017). Botanical and geographical characteristics of sorghum species. *Vestnik of the South-Kazakhstan State Pharmaceutical Academy*, *2*(79), 74.

29. Pereverzin, V. (2016). Sorghum: Cultivation technology for forage purposes. *Agrarian Review*, *3*(55), 51–53. https://doi.org/10.1016/j.apsusc.2024.159394

30. Podgorbunskikh, E.M., Bychkov, A.L., Bulina, N.V., & Lomovskii, O.I. (2018). Disordering of the crystal structure of cellulose under mechanical activation. *Journal of Structural Chemistry*, *59*(1), 201–208. https://doi.org/10.1134/s0022476618010328

31. Rasaq, S. A., Swarna, J., & Amit, K. J. (2024). Green and sustainable pretreatment methods for cellulose extraction from lignocellulosic biomass and its applications: A review. *Carbohydrate Polymer Technologies and Applications*, *7*, 100396. https://doi.org/10.1016/j.carpta.2023.100396

32. Reddy, N., & Yang, Y. (2007). Structure and properties of natural cellulose fibers obtained from sorghum leaves and stems. *Journal of Agricultural and Food Chemistry*, *55*(14), 5569–5574. https://doi.org/10.1021/jf0707379

33. Roohallah, S. R., Mozhgan, G. V., Mohadeseh, H., & Vijay, K. T. (2024). Agricultural wastes: A practical and potential source for the isolation and preparation of cellulose and application in agriculture and different industries. *Industrial Crops and Products*, *208*, 117904. https://doi.org/10.1016/j.indcrop.2023.117904

34. Sani, H. A., Yauri, A. U. B., Muhammad, A., Yahayya, Y., & Salau, M. (2020). Preparation and characterization of bioplastic from sorghum husk. *American Journal of Polymer Science and Technology*, *6*(2), 14–20. https://doi.org/10.11648/j.ajpst.20200602.12

35. Shahi, N., Min, B., Sapkota, B., & Rangari, V. K. (2020). Eco-friendly cellulose nanofiber extraction from sugarcane bagasse and film fabrication. *Sustainability*, *12*(15), 6015. https://doi.org/10.3390/su12156015

36. Shehrawat, P., & Sindhu, N. (2015). Agricultural waste utilization for healthy environment and sustainable lifestyle. *Annals of Agri-Bio Research*, *20*(2), 110–114.

37. Waghmare, P. R., Khandare, R. V., Jeon, B., & Govindwar, S. P. (2018). Enzymatic hydrolysis of biologically pretreated sorghum husk for bioethanol production. *Biofuel Research Journal*, *5*(3), 846–853. https://doi.org/10.18331/BRJ2018.5.3.4

38. Wang, Q., Nnanna, P. C., Shen, F., Huang, M., Tian, D., Hu, J., Zeng, Y., Yang, G., & Deng, S. (2021). Full utilization of sweet sorghum for bacterial cellulose production: A concept of material crop. *Industrial Crops and Products*, *162*, 113256. https://doi.org/10.1016/j.indcrop.2021.113256

39. West Kazakhstan Region, Taskala District Akimat. (n.d.). *Activities*. https://www.gov.kz/memleket/entities/bko-taskala/activities/2380

40. Yulianto, Putri, D. N. M., Perdani, M. S., Arbianti, R., Suryanegara, L., & Hermansyah, H. (2020). Effect of cellulose fiber from sorghum bagasse on the mechanical properties and biodegradability of polylactic acid. *Energy Reports*, *6*(1), 221–226. https://doi.org/10.1016/j.egyr.2019.08.048

41. Zghari, B., Hajji, L., & Boukir, A. (2018). Effect of moist and dry heat weathering conditions on cellulose degradation of historical manuscripts exposed to accelerated ageing: 13C NMR and FTIR spectroscopy as a non-invasive monitoring approach. *Journal of Materials and Environmental Science*, *9*(2), 641–652.

42. Zhang, L. M., Leng, C. Y., Luo, H., Wu, X. Y., Liu, Z. Q., & Zhang, Y. M. (2018). Sweet sorghum originated through selection of dry, a plant-specific NAC transcription factor gene. *The Plant Cell*, *30*(10), 2286–2307.

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