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Features of microcrystalline cellulose produced from sunflower seeds of different oil content

Abstract. The work is dedicated to study the production of the microcrystalline cellulose (MCC) from sunflower seed husks (SFH) by oxidation in an organic solvent, the dependence its hydromodule, yield and quality index of the MCC on the sunflower type. According to the morphological characteristics of SFH with different fat content, the effective hydromodulus of the MCC obtained from low-fat SFH is 1:12 g/mL with a yield of 50.69%, while the effective hydromodulus for high-fat SFH is SFH:PAA 1:14 g/mL, it was determined that the yield will be equal to 43.49%. During the organic solvent oxidation, it was found that low fat sunflower husk undergoes delignification 3 times faster than high-fat, accordingly, the yield of cellulose and the content of β-cellulose are higher by 7.2% and 2.23%. That is, it became known that low-fat sunflower seed husk is an economically and ecologically effective raw materials in the production of the cellulosic materials.
Key words: sunflower husks, sunflower variety, organosolvent oxidation, microcrystalline cellulose, FTIR, crystal structure, surface morphology, fibers.

Introduction

Currently, research is being actively conducted on the extraction of cellulose fibers from annual plants, including agricultural waste. This is due to the fact that these wastes are rich in hydrocarbon compounds. The waste accumulated in the fields and during the processing of biomass requires additional utilization [1].One of these agricultural waste is sunflower seed husk (SFH), which is formed during the extraction of oil from sunflower seeds. Currently, sunflower husks are used in fields such as, fertilizer [2], animal feed [3], sintered panel production, xylose extraction [4] and furfural production [5], but using of SFH as a fuel is predominant [6, 7].

According to the research results, it was found that the fiber content in the seeds of sunflower husk (SFH) ranges from 41% [8] to 50% [9, 10]. In this works [11, 12], the effective parameters for obtaining MCC from SFH under «soft» conditions of the organic solvent oxidation method were studied, the yield of cellulose was 47.8%. This indicates that SFH is a potential feedstock source for cellulose production.However, in studies [13, 14], the composition and amount of chemical compounds in SFH change depending on their varietal characteristics, soil and climatic conditions and agricultural practices of cultivation, that is, sunflower seeds with a high fat content have a certain difference from low-fat types, it was found that in the husk of high grades there is more ash, phosphorus, nitrogenous substances, oil, sugar and lignin, less crude fiber and cellulose. Therefore, it is very important to determine the effective parameters, quality indicators and cellulose yield during the extraction of sunflower seed husks of different varieties. This is due to the fact that the determination of the quality of the raw materials utilized in the extraction of cellulose, the yield of cellulose and quality indicators allows minimizing the amount of reagents, time, energy and economic costs used in production.

The investigation is based on a comparative study of yield and quality indicators, chemical composition, surface morphology and crystal structure of sunflower seed husks of Limited Liability Partnership «East Kazakhstan Agricultural Experimental Station» «Belosnezhka» sort and «Altai» sort with various fat content.

Materials and methods

Materials. Hydrogen peroxide (15%, H_2O_2), acetic acid (≥55%, CH_3COOH), potassium permanganate (99%, $KMnO_4$), ethanol (96%, C_2H_5OH), sulfuric acid (98%, H_2SO_4), orthophosphoricacid (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 obtained from Sigma-Aldrich (Bangalore, India). All other reagents were of analytical grade and were used without additional purification.

Preparation of raw materials. The SFH raw material used to obtain microcrystalline cellulose was obtained from Limited Liability Partnership «East Kazakhstan Agricultural Experimental Station», located in Ust-Kamenogorsk. Two different varieties of sunflower «Belosnezhka» and «Altai» were used for research and were de-seeded (Figure1a-1b). Sunflower variety «Belosnezhka» – annual herbaceous plants, tall plant, 270-340 cm high. Fruits are oblong-ovate achenes, slightly faceted, slightly compressed 23-25 mm long and 10-12 mm wide, with a leathery pericarp (husk) of white color. The pericarp is a hard woody multilayer formation. Flowering time is very late. The ripening period (ripeness group) is late-ripening. Sunflower variety «Belosnezhka» is drought-resistant, resistant to lodging, withstands frosts down to – 80^0C. Oil content of seedsis 33.7% [15].

Sunflower variety «Altai»are annual herbaceous plants, medium-sized plant, 170-180 cm high. The fruits are oblong-ovate achene, the marginal stripes of the achene are strongly pronounced, slightly compressed, 14-16 mm long and 6-8 mm wide, with a leathery pericarp (husk) of black color. The pericarp is a hard woody multilayer formation. Flowering time is average. The ripening period (ripeness group) is early. Sunflower varieties of «Altai» drought resistance is high. Oil content of seeds is 52-54% [15].

To remove the phytomelanin pigment in the SFH of the Altai variety, pre-treatment is carried outwith a 5% NaOH solution in a flaskwith a rotary condenser. After it was washed for 60 min., with continuous stirring in an oven with a magnetic stirrer at 90±2°C temperature. Then it was filtered through filter paper, washed with distilled water until the pH of the medium was 7, and dried in an oven at 60±2°C for 5 hours.

During the research, the samples obtained from the raw materials of sunflower seed husks of different varieties were conventionally designated as for the variety «Belosnezhka» – B-SFH, and MCC obtained from it – $MCC_{B\text{-}SFH}$, for the variety «Altai» – A-SFH, and MCC from it – $MCC_{A–SFH}$.

Figure 1–Sunflower seed husk samples: a – B-SFH and b **–** A-SFH

Preparation of peroxyacetic acid (PAA). Preparation of the PAA was prepared according to the procedure [16].Obtained PAA was stored in a freezer at a 5±0.5 °C temperature.

Obtaining of MCC from SFH by organic solvent oxidation and determination of its yield. Obtaining MCC from SFH was conducted according to the methodology of these [11,12] studies. That is, for samples of B-SFH and A-SFH, measured separately at 10 g, the ratio of SFH:PAA was: 1:12, 1:14, 1:16, 1:18, 1:20, 1:22, 1:24 g/mL, respectively. Obtaining MCC was carried out, by boiling raw materials and delignificator, in a flaskwith a rotary condenser,at a temperature 90±20°C with continuous intensive stirring. The obtained $MCC_{B\text{-}SFH}$ and $MCC_{A\text{-}SFH}$ were cooled at a temperature of 25±20^{0}C, filtered through filter paper and neutralized with distilled water to pH=7. The neutralized MCC was dried at a temperature of 80 ± 2°C for 6 hours until a constant weight was reached, then MCC was weighed on an analytical balance with an accuracy of 0.0001 g and the yield was calculated by the formula:

$$\eta = (m_{SFH}\text{-}m_{MCC})/m_{SFH}\times 100\%$$

Herein: m_{SFH}–mass of SFH, g; m_{MCC}–mass of obtained MCC, g.

Figure 2 – The scheme of obtaining MCC by the method of organosolvent oxidation

Determination of microcrystalline cellulose quality indicators. Moisture content of $MCC_{B\text{-}SFH}$ and $MCC_{A\text{-}SFH}$ was determined according to state standard -16932 (STST), β-cellulose content according to STST-6840, residual lignin content according to STST-11960, hemicellulose content according to STST-9002.

Optical microscopy. The surface morphology of the obtained MCC was studied by placing the substance on the glass surface, passing light from below, at a temperature of 25±20 ^{0}C using an XSZ-146 optical microscope (LabSol, China).

FTIR spectroscopy. FTIR analysis was performed on a spectrometer FTIR FT-801 (Simex, Russia), with a resolution of 1 cm^{-1} and a wavelength 4500-4700 cm^{-1}, by placing the sample on the surface of the attachment with the method of internal and variable-diffuse reflection, at a temperature of 25°C and a number of scans of 100.

X-ray diffraction. The crystal structures of substances were studied by X-ray diffraction on X´PertPRO diffractometer (Malvern Panalytical Empyrean, Netherlands) using monochromatized copper (CuKα) radiation with a scan step of 0.02°, K-Alpha1 [Å] 0.1542. The measurement angle was 10-40°, the X-ray tube voltage was 40 kV, the current intensity was 30 mA, the measurement time at each step was 0.5 s and an aluminum rectangular multi-purpose sample holder (PW1172/01) was used for the measurement in reflection mode. The ICDD PDF-4/AXIOM database of XRD patterns was used for the analysis of the XRD.

Results and discussion

The obtaining $MCC_{B\text{-}SFH}$ *and* $MCC_{A\text{-}SFH}$ *from SFH by organosolvent oxidation method.* Figures 3a and 3b show $MCC_{B\text{-}SFH}$ and $MCC_{A\text{-}SFH}$ obtained from two different varieties of SFH. Based on the white color of the B-SFH feedstock, the received $MCC_{B\text{-}SFH}$ is a perfectly thin cotton-like soft mass composed of very fine fibers (Figure 3a). However, $MCC_{A\text{-}SFH}$ obtained

from A-SFH was found to be light gray in color and cotton-like in softness due to residual pigment in the feedstock (Figure3b).

Theyield and quality indicators of MCC. Table 1 shows the yield and quality indicators of $MCC_{B\text{-}SFH}$ and $MCC_{A\text{-}SFH}$ obtained at various ratios of SFH:PAA g/ml by organic solvent oxidation under «mild» conditions.According to the results of the study, the yield of cellulose at a ratio of hydromodule SFH:PAA 1:12 g/mL for B-SFH is high, i.e. yield is 50.69%, moisture 3.7%, β-cellulose 67.53%, residual lignin 2.4.%, hemicellulose 8.1% and ash content (SiO_2) 1.2%. An increase in the amount of PAA did not lead to an increasing in the MCC yield. Whereas, for A-SFH, the yield of MCC was determined to the maximum value at a ratio of SFH:PAA 1:14 g/mL, and its value 43.49%, moisture 4.7%, β-cellulose 65.3%, residual lignin 2.6%, hemicellulose 8.1% and ash content (SiO_2) 1.3%. The yields, of $MCC_{A\text{-}SFH}$ obtained at other ratios of SFH:PAA g/mL ranged from 40 to 42%.

Figure 3 – MCC samples from two different varieties of SFH:
a – $MCC_{B\text{-}SFH}$ (hydromodule 1:12); b – $MCC_{A\text{-}SFH}$ (hydromodule1:14)

The yield of $MCC_{B\text{-}SFH}$ obtained from B-SFH is higher to 7.2%, than MCCfrom A-SFH, which is directly related to the large fiber size in the inner layer of the B-SFH husk. During the investigation [11,12], the effective hydromodule of SFH:PAA g/mL for the extraction of MCC from SFH was studied. It was found that its value is 1:20 g/mL. According to the results of this study, it was found that the amount of PAA consumed in effective hydromodule is 8 mL less for B-SFH and 6 mL less for A-SFH compared to the previous study.In addition, it was found that it takes 40 minutes to delignify B-SFH and 120 minutes to delignify A-SFH. It can be assumed that the delignification process in A-SFH takes longer than in B-SFH, since due to the higher fat content in A-SFH, the sorption capacity of the delignifying agent decreases and the process takes more time. In addition, types of raw material characteristics indicate, that MCC yield from SFH, effective water content of SFH:PAA g/mL and extraction time are among the main factors influencing the variation for each raw material.

Optical microscopy. Figures 4a and 4b show in comparison the surface morphology of B-SFH and A-SFH raw materials under an optical microscope. The fruit coat (pericarp) consists of thin and colorless cells forming a uniform layer of skin. The fruitsurface can be seen paired fibers. Skin cells are covered with a thin film – integumentary tissue. On the inner surface of the fruit shell there are cells of the fibrous layer with pronounced pores (sclerenchyma). Sclerenchyme and internal parenchyme are composed of lignified cells [15]. The morphological characteristics of sunflower achene varieties of A-SFH differ significantly from B-SFH, primarily in the structure of integumentary tissues. In achenes of A-SFH varieties, the thickness of the fruit coat is 100 μm, while in case B-SFH it was 400 μm. The color of the achenes is determined by the color of the skin of the pericarp.The black color of the achenes of varieties A-SFH is associated with a coloring matter – phytomelanin. Achenes of varieties B-SFH arewhite. As a result of the study, it was found that on the inner surface of the fruit

shell there are smooth undulating protrusions, which are called sclerenchyme, in the fruit coat of sunflower varieties B-SFH, the distance between the protrusions is greater both in the middle of the sclerenchyme layer and on the side of the parenchyme, the cells are oval in shape and larger than in varieties of A-SFH. Due to a very developed active surface, sclerenchyme has a high sorption capacity with respect to various gases, vapors, as well as water, oil and good mass conductivity.

Table 1 – Effect of SFH:PAA ratio on MCC quality

B-SFH SFH:PAA, g/mL	Quality indicators of MCC_{B-SFH}, %					
	Yield	Moisture	β-cellulose	Residual lignin	Hemi-cellulose	Ash content (SiO_2)
1:12	50.69±2	3.7±0.5	67.53±3	2.4±0.5	8.1±2	1.2±0.5
1:14	48.79±2	4.8±0.5	64.93±3	2.6±0.5	10.8±2	1.2±0.5
1:16	49.32±2	4.7±0.5	63.80±3	3.1±0.5	8.1±2	1.3±0.5
1:18	48.30±2	4.8±0.5	62.87±3	3.0±0.5	8.1±2	1.2±0.5
1:20	48.71±2	5.1±0.5	60.61±3	3.0±0.5	13.5±2	1.4±0.5
1:22	48.58±2	5.1±0.5	59.20±3	3.1±0.5	8.1±2	1.4±0.5
1:24	48.51±2	5.1±0.5	56.90±3	2.9±0.5	13.5±2	1.3±0.5
A-SFH SFH:PAA, g/mL	Quality indicators of MCC_{A-SFH}, %					
	Yield	Moisture	β-cellulose	Residual lignin	Hemi-cellulose	Ash content (SiO_2)
1:12	42.53±2	4.8±0.5	63.93±3	2.8±0.5	8.1±2	1.4±0.5
1:14	43.49±2	4.7±0.5	65.30±3	2.6±0.5	8.1±2	1.3±0.5
1:16	42.85±2	5.2±0.5	65.23±3	2.6±0.5	13.5±2	1.3±0.5
1:18	41.99±2	4.8±0.5	64.50±3	3.0±0.5	13.5±2	1.3±0.5
1:20	40.58±2	5.7±0.5	62.53±3	3.1±0.5	13.5±2	1.4±0.5
1:22	41.17±2	4.5±0.5	61.40±3	2.8±0.5	10.8±2	1.5±0.5
1:24	41.10±2	4.2±0.5	61.37±3	3.0±0.5	10.8±2	1.5±0.5

Figure 5 shows the surface morphology of MCC_{B-SFH} and MCC_{A-SFH} obtained in an effective hydromodule.It can be seen, that MCC obtained from two different types of raw materials consists of fibers. It was determined that, with an MCC_{B-SFH} fiber length of about 697.5±150 μm, the width is 41.7±15 μm (Figure5a-5b), also that the length of the MCC_{A-SFH} fiber is 333.3±100 μm and the width is 20.4±15 μm (Figure 5c-5d). This is due to the biological features of SFH described above, that is, the thickness and length of the B-SFH fibers are 2 times greater than those of A-SFH, and the fibers in the inner layers of B-SFH have a greater thickness, respectively, the volume of the obtained MCC_{B-SFH} fibers appears to be much larger.In addition, in organic solvent oxidation, it was observed that the cellulose microfibers obtained from SFH, were extracted separately and purified from hemicellulose and lignin.Similar results were obtained by microscopic examination in research [11,12] work.

FTIR spectroscopy. Chemical structures of MCC_{B-SFH} and MCC_{A-SFH} obtained at PAA ratios (SFH:PAA, g/mL) 1:12, 1:14, 1:16, 1:18, 1:20, 1:22, 1:24 relative IR spectra are shown in Figure 6. According to IR spectroscopydescribes, the absorption region of 661.3 cm^{-1} in all spectra indicates an out-of-plane vibration of the C–OH bond [17,18], 896.1 cm^{-1} and 1160.2 cm^{-1}β-(1,4)-glycosidic bond C–O–C (amorphous part) [19,20], high intensity signals at 1029.4 cm^{-1} and 1160.2 cm^{-1} are C-O and C-C bonds in the aromatic ring, long-term vibrations of the C-O-C bond in the pyranose ring at 1050.3 cm^{-1},1315.5 cm^{-1}, 1333.4 cm^{-1} and 1368.3 cm^{-1} C–H, 1428.4 cm^{-1} C–H_2 group [19,21], 1639.6 cm^{-1}O–H bonds in a water molecule absorbed from air by cellulose [19], signals of the CH and OH groups at wavelengths of 2892.4 cm^{-1} and 3332.7 cm^{-1} [19, 22, 23]. In the entire spectrum, weak absorption at a wave number of 1512.3 cm^{-1} is characteristic of the C=C aromatic ring in the lignin

molecule [21], 1728.7 cm^{-1} indicates a long-range vibration of the acetyl and ester C=O groups in the hemicellulose molecule [24]. This explains the numerical values of the content of hemicellulose in the range of 8.1-13.5% given in table 1.In studies [11, 12], it can be seen that the absorption spectra of the MCC obtained as a result of the IR spectrometer are consistent with the above results.

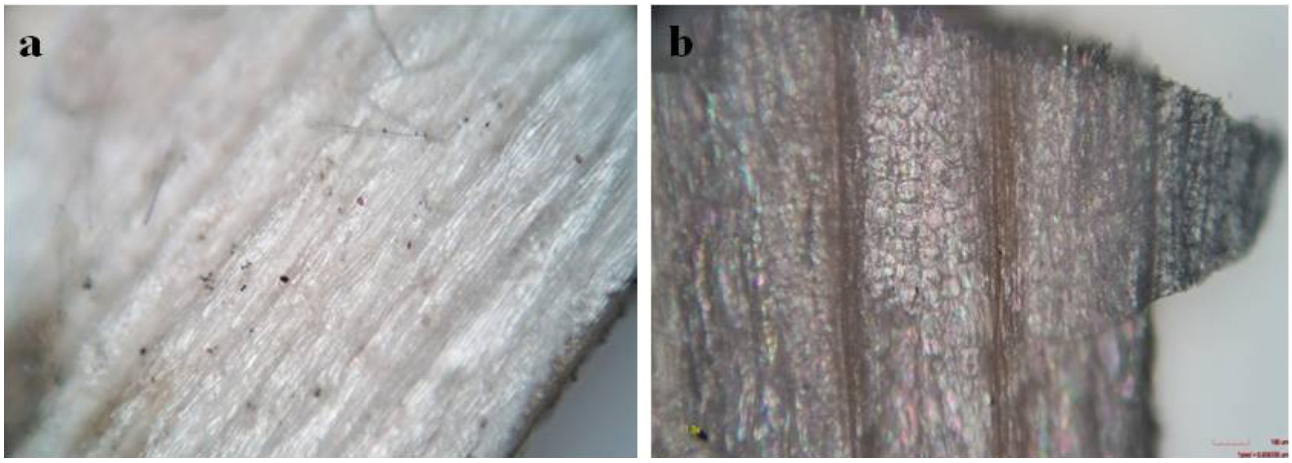

Figure 4 – Optical microscope images of: a -B-SFH and b -A-SFH

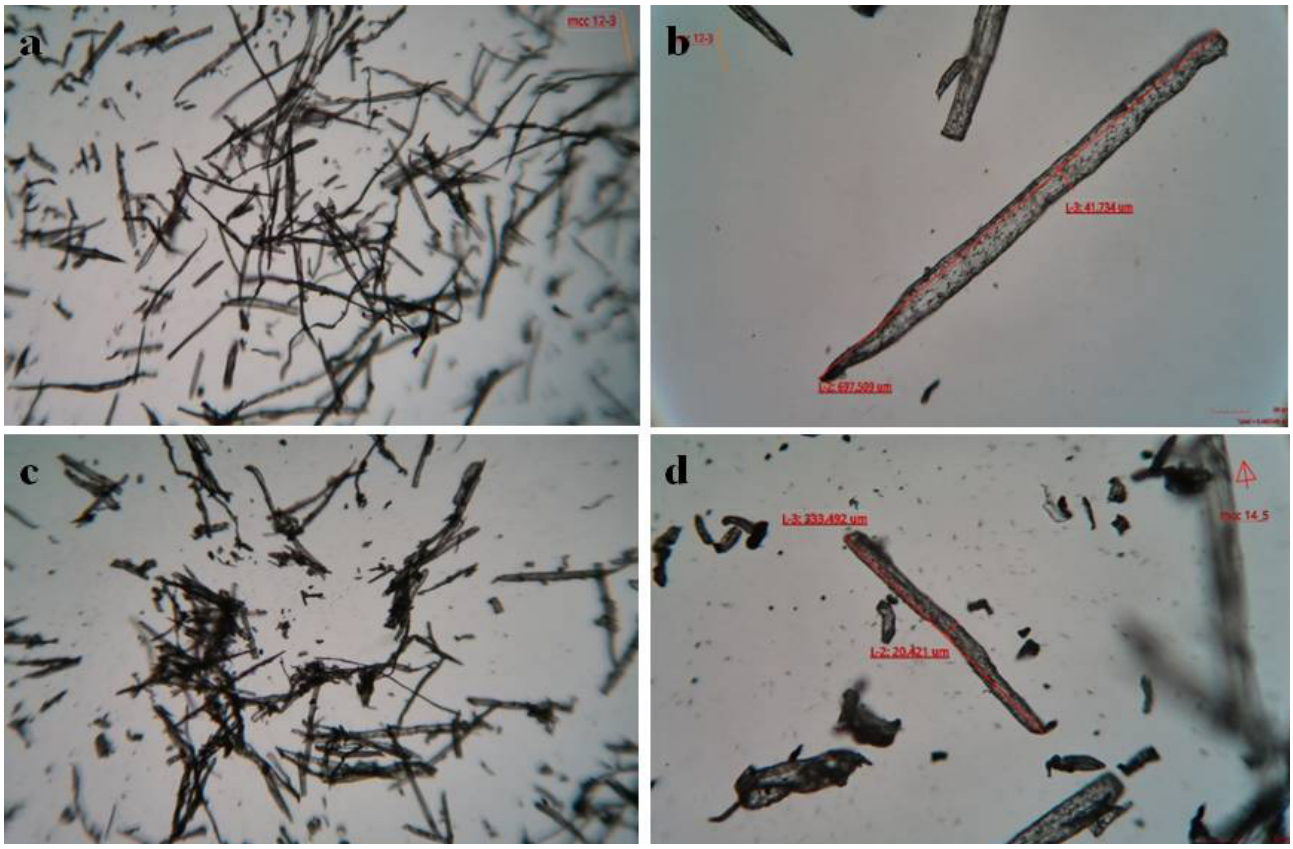

Figure 5 – Optical microscope images of: a.b – $MCC_{B\text{-}SFH}$; c.d – $MCC_{A\text{-}SFH}$

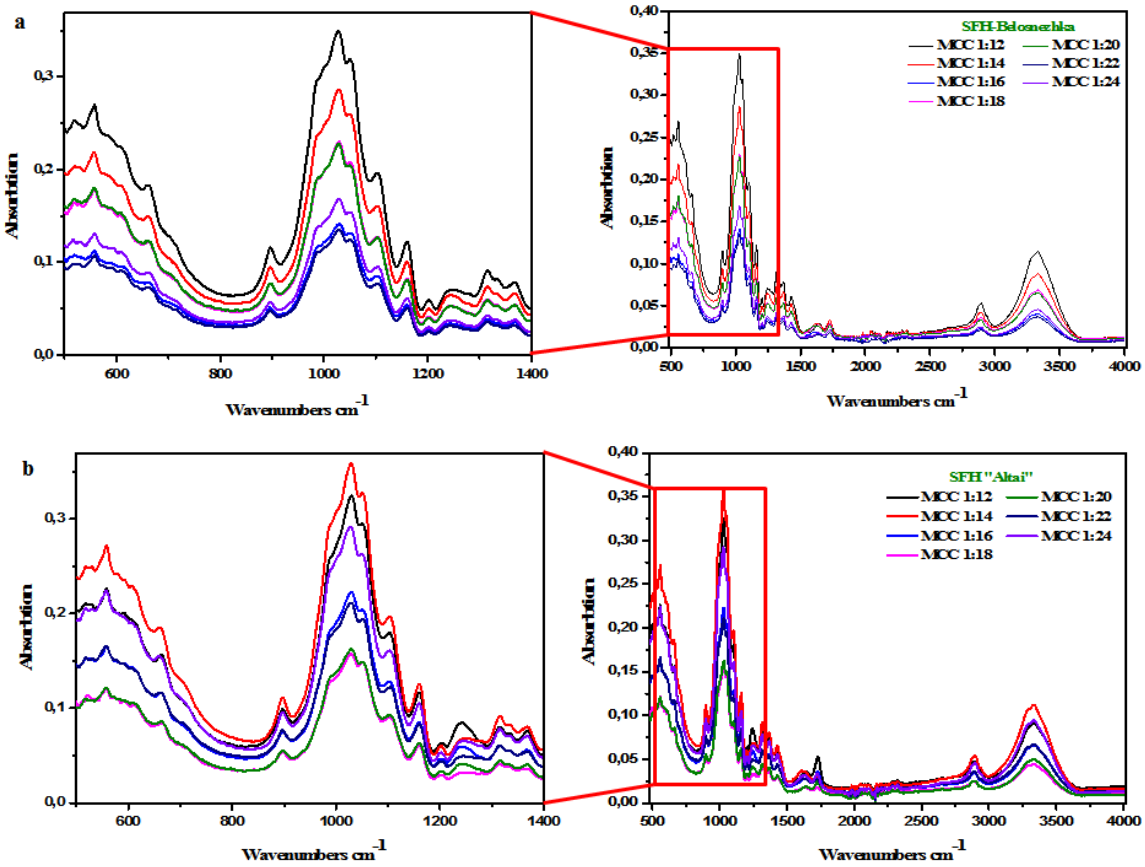

Figure 6 – FTIR spectra of MCC in different ratios: a – $MCC_{B\text{-}SFH}$; b – $MCC_{A\text{-}SFH}$

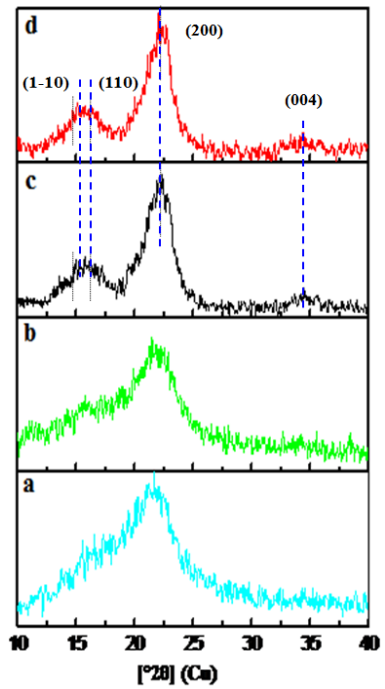

Figure 7 – XRD diffractions of SFH and MCC:
a) B-SFH; b) A-SFH; c) $MCC_{B\text{-}SFH}$ 1:12; d) $MCC_{A\text{-}SFH}$ 1:14

X-ray diffraction. The crystallinity of cellulose is a very important indicator that determines its thermal and mechanical properties. On figure 7 are shown comparative the X-ray diffraction patterns of MCC_{B-SFH} and MCC_{A-SFH} obtained from B-SFH and A-SFH raw materials in an effective hydromodule. The crystal structure of cellulose was determined on the basis of studies [11, 25, 26]. On the diffraction patterns of B-SFH and A-SFH in figures 7a and 7b marked peaks at $2\theta=15.7^0$ (1-10), (110), 22.3^0 (200). This is mainly characteristic of amorphous carbon [12]. This is due to the fact that the raw material contains unextracted lignin, hemicellulose, etc., which has an amorphous structure due to the action of hydrocarbons that cover the crystalline cellulose fibers [11]. On the X-ray diffraction pattern of the obtained MCC in Figures 7c and 7d three diffraction peaks were registered. They are equal to $2\theta=15.6^0$ (1-10), (110), 22.3^0 (200), 34.5^0 (004) and the crystal structure of the molecule is double-stranded monoclinic, cellulose I β showed diffraction peaks [11, 24]. This proves that the crystal structure of MCC obtained by the method of organic solvent oxidation does not change. The crystal structure of the MCC obtained during the research [11,12] is consistent with the results of the present study. This indicates that the crystal structure of the material obtained in this investigation is typical for MCC.

Conclusion

In conclusion, it was found that the yield, optimal hydromodule and quality indicators of MCC obtained from SFH are directly affected by the varietal characteristics of the feedstock. Due to the thicker fibrous layer (sclerenchyma) of B-SFH than A-SFH and the lower fat content, the effective hydromodule for obtaining MCC_{B-SFH} by organic solvent oxidation in B-SFH is SFH:PAA 1:12 g/mL, the yield was equal to 50.69%. Whereas, the effective hydromodule of SFH:PAA was 1:14 g/mL and the yield of MCC_{A-SFH} was 43.49%. That is, the yield of MCC_{B-SFH} obtained from B-SFH and the amount of β-cellulose are higher by 7.2% and 2.23%, respectively, compared with the A-SFH grade, and there was no significant difference in hemicellulose and ash content (SiO_2). It was investigated whether the delignification time of low fat SFH is shorter than of high fat SFH. It has been established that the chemical structure of MCC obtained from B-SFH and A-SFH varieties corresponds to cellulose, and it has also been studied that the surface morphology of cellulose is a thin ribbon-like fiber, and the crystal structure does not change during processing synthesis. As a result, it became known that the B-SFH brand is an effective raw material for resource saving in the production of cellulose raw materials.

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