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OBTAINING CELLULOSE WITH CRYSTALLOGRAPHIC ORIENTATION OF MACROMOLECULES FROM THE HUSK OF A HYBRID SUNFLOWER

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Currently, the obtaining and implementation of self-degradable, harmless to the nature of composite materials based on cellulose, allows to solve a number of environmental problems. In this regard, the number of raw materials sources from which cellulose can be obtained increases, the economic and environmental efficiency of its usage and the properties of the obtained cellulose can be studied.

In this study, from sunflower husk (SFH)P63LE10 «Pioneer» (USA) using glacial acetic acid and based on acetic acid with 42% concentration peroxyacetic acid, process of obtaining microcrystalline cellulose comparatively was studied. As a result of the study, based on acetic acid with 42% concentration in combination peroxyacetic acid was determined, that yield of obtained MCC (MCC_2) was 3,7%, and also quantity of α-cellulose was higher to 3,6%. However, the quality of the residual lignin was 24.02%, and the trace quantity of amorphous structure was determined by IR spectroscopy and XRD diffractometry. On the contrary, the yield and quantity of α-cellulose obtained MMC_1 using glacial acetic acid in combination peroxyacetic acid is lower than MMC_2, obtained product was different by high degree of purity and content of the ordered part of cellulose with crystallographic orientation of macromolecules.

Keywords: *sunflower husk (SFH), microcrystalline cellulose (MCC), peroxyacetic acid (PAA), glacial acetic acid, crystallinity.*

INTRODUCTION

Nowadays, as the volume of agriculture increases, so does the amount of waste from that industry, and the problem of its recycling and utilization is becoming urgent [1]. In this regard, the use of rice husks, wheat straw, sunflower seed husks (SFH) and other agricultural wastes as raw materials for obtaining cellulosic materials is intensively studied [2–3]. Since the East Kazakhstan region is in the forefront of sunflower cultivation and sunflower oil production in the republic, the amount of accumulated SFH waste is also quite large [4].

Due to the development of green technologies, there is a growing need to use cellulose to obtain nanocrystals and cellulose nanofibrils from them, which can serve as a potential basis for polymer matrices [5]. Cellulose-based composite materials are used in pharmaceuticals, medicine and electronics, due to such qualities as biodegradability, biocompatibility and low cellular toxicity [6].

In general, since cellulose is a semi-crystalline biopolymer, which contains amorphous and crystalline structures that do not have clear boundaries [7]. The main objectives of the methods for obtaining cellulose are to receive high-quality pure cellulose with a high yield and content of α-cellulose, with less amorphous lignin [8]. The yield and crystallinity of nanocrystals and nanofibers obtained from cellulose depend on the amount of α-cellulose in the cellulose. This is because the form of cellulose with high crystallinity is α-cellulose [9]. Due to the simultaneous process of bleaching and delignification in organosolvent oxidation, it is a more environmentally and cost-effective method for removing lignin during the extraction of cellulose from annual plants [3].

According to the results of the previous research, it was determined that up to 50% cellulosic materials can be obtained from SFH [3, 10]. This is considered a relatively high yield for cellulose from agricultural residues. Whereas, when using the organosolvent method, the yield of cellulose from rice straw and wheat stems is 31.72% and 49–65%, respectively [11].

The waste from the oil extraction plant - sunflower seed husk, which is an indispensable raw material for obtaining cellulose, is often briquetted and used as fuel. In this regard, it is very important to consider the issue of extracting cellulose fibers from sunflower seed husks and develop its effective technology.

Although there are no morphological features of the sunflower seed husk depending on the variety and hybrid type, the size of the husks varies. For example, if the amount of husk in oil varieties and hybrid seeds is 22.5–30%, the amount of husk in confectionery seeds is up to 1.5–2 times more [12]. This, in turn, is a factor that affects the mode of technology for extracting cellulose from it. Currently, sunflower seeds in circulation of the agro-industrial complex of the country are mainly hybrid seeds, so the main source of raw material for cellulose production is sunflowers grown from hybrid seeds. Therefore, the study of extracting cellulose from hybrid seed husks, whose husk size is 1/3 of the seed, is also an urgent issue.

In this study, the work of determining the mode of obtaining microcrystalline cellulose (MCC) by the organosolvent oxidation method using concentrated (glacial acetic acid) and acetic acids whose concentration is reduced by half, i.e. 42%, from the husks of hybrid sunflower seeds "Pioneer 10" grown in the region of East Kazakhstan was conducted. During the experiment, peroxyacetic acids were prepared from glacial acetic and 42% acetic acids in the presence of hydrogen peroxide, and samples of microcrystalline cellulose were obtained. The yield, amount of residual lignin and α-cellulose of the obtained MCC was determined, also its chemical structure by IR-spectroscopic method, as well as its morphology by optical microscopy, and its crystal structure by X-ray phase analysis was determined.

2. Experimental Part

2.1 Materials

P63LE10 "Pioner" (USA) hybrid sunflower seed husk from the circulation of LLP "Mileiko" peasant farm was used to obtain MCC by the of organosolvent oxidation method. This seed has been registered in the State Register of Kazakhstan since 2015, is a hybrid with high fat content and early ripening, mainly grown in East Kazakhstan. The average size of the seeds is 1.1 cm, oval-oblong, the average amount of oil in the seeds is 42.5%, the average amount of husk is 24.3% [13].

In order to prepare SFH, P63LE10 "Pioneer" (USA) hybrid sunflower seed husks were dried at a temperature of 50±20^{0}C to a constant mass in a drying cabinet (CS-80, Belarus).

Peroxyacetic acid was prepared from glacial acetic acid and 42% acetic acid to obtain microcrystalline cellulose by the organosolvent oxidation method and its concentration was determined [14–15]. In order to prepare peroxyacetic acid (PAA) (solvent) glacial acetic acid (CH_3COOH, 99.9%, STST 19814-74), acetic acid (CH_3COOH, 42%, STST 61-75) hydrogen peroxide (H_2O_2, 34,5–36,5%, STST 177-88), sulfuric acid (H_2SO_4, 95–98%, STST 4204-77), distilled water (STST 6709-72) was used. In order to determine qualitative indicators of MCC potassium iodide (KI, chemical pure, Sigma-Aldrich), starch ($(C_6H_{10}O_5)_n$ ACS, Sigma-Aldrich), sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$, 0,1 n, Sigma-Aldrich), drying cabinet (SC-80-01-SPU), orthophosphoric acid (≥85% H_3PO_4), sodium chloride (NaCl, Sigma-Aldrich), sodium hydroxide (NaOH ≥98%) was used. All reagents were used without further purification.

2.3 Methods

2.3.1 Obtaining MCC by the method of organosolvent oxidation

PAA_1 and PAA_2 peroxyacetic acids were developed from glacial acetic acid and 42% acetic acid to obtain microcrystalline cellulose from sunflower seed husks, respectively. The samples MCC_1 and MCC_2 were prepared using PAA_1 and PAA_2 at a ratio of 1/12 g/ml as raw material and solvent hydromodule, respectively. The delignification process was carried out at a temperature of 90±20 °C for 120±1 minutes in a reflux flask with continuous stirring. The obtained gray mass was cooled to a temperature of 25±20 °C, filtered using filter paper and washed with distilled water until the pH medium became neutral (pH=6–7). The neutralized cellulosic mass was dried at a temperature of 70±20 °C for 6 hours to a constant mass. The yield of obtained microcrystalline cellulose was calculated according to Formula 1:

$$X(\%) = (m_{SFH} - m_{MCC}) / m_{SFH} \cdot 100\% \quad (1)$$

where: m_{SFH} – mass of sunflower seed husk, m_{MCC} – mass of obtained MCC.

2.3.2 Determination of qualitative indicators of MCC_1 and MCC_2 samples

The α-cellulose in MCC samples was determined according to STST 6840-78 and the amount of residual lignin was determined according to STST 11960-76.

2.3.3 Fourier-transform infrared spectroscopy (FTIR)

FTIR analysis of obtainedMCC_1andMCC_2samples were performed on an FT-801 FTIR spectrometer (Simex, Russia), with a resolution of 1 cm^{-1} and a wavelength 4500–4700 cm^{-1}, at a temperature of 25 °C and a scan rate of 100.

2.3.4 Optical microscope

The surface morphology of the MCC_1 and MCC_2 samples was captured on an optical microscope of the XSZ-146 (China) model at x10 and x40 magnification. The samples were examined under a microscope at a temperature of 25±20 °C using a glass.

2.3.5 XRD analysis

The crystal structure of MCC_1 and MCC_2 samples was investigated by X-ray diffraction on an X´PertPRO spectrometer (PANanalytical, Netherlands) using monochromatized copper radiation with a scan step of 0.02°. The measuring angle 10–40°, X-ray tube voltage 40 kV, current 45 mA and measurement time at one point 0.5 s.

3. Results and Discussion

As a result of the study, light gray MCC_1 and MCC_2 cellulose samples were obtained from P63LE10 "Pioneer" (USA) hybrid sunflower seed husks using PAA_1 and PAA_2 at a ratio of 1/12 of raw material and solvent.

The obtained microcrystalline cellulose yields, α-cellulose and residual lignin content are presented in Table 1. The yield of MCC_1 was 32.8%, while that of MCC_2 was 36.5%. It was found that almost doubling the concentration of acetic acid did not affect the yield of MCC from sunflower husks obtained from hybrid seeds, but on the contrary, the yield of MCC_2 obtained by using 42% acetic acid increased by 3.7%. Accordingly, the content of the pure form of cellulose - α-cellulose, obtained from the inner layer of the plant in the MCC_2 sample was 57%, and compared to MCC_1, its amount was 3.6% higher. It is observed that the amount of residual lignin is 24.02% in the MCC_2 sample, while it is 20.92% less in the MCC_1 sample. Therefore, the use of glacial acetic acid during the organosolvent oxidation process, due to the greater destruction of amorphous structures in sunflower seed husks, a significant part of amphoric substances passes

into the solution and ensures the smooth progress of the delignification process. This is because hydroxonium cations formed by peroxyacetic acids in an acidic environment attack the reaction centers of lignin, the reactivity of lignin increases and it gradually undergoes fragmentation. That is, the more hydroxonium cations are formed, the better the delignification process is [16]. Compared to 42% acetic acid, glacial acetic acid with a higher concentration has a higher ability to form hydroxonium cations during the delignification process, so the residual amorphous structure is destroyed to a greater extent, and the amount of residual lignin in MCC_2 obtained by using acetic acid with twice the concentration is 7.75 times less (Table 1).

Table 1. Qualitative indicators of MCC_1 and MCC_2 samples, %

Qualitative indicators	MCC_1	MCC_2
Yield of MCC	32.80□2	36.50□2
α-cellulose	□.□2	57.00□3
Residual lignin	3.1□0.5	24.02□0.5

From Figure 1 can be seen that the IR spectra of MCC_1 and MCC_2 microcrystalline cellulose samples do not differ from each other. Here, the in-plane skeletal vibrations of the phenolic aromatic ring at wavelengths of 1505–1600 cm^{-1} indicate the presence of lignin [10]. However, the intensity of bands at wavelengths of 1125 cm^{-1} and 1510, 1600, 1659, 1726 cm^{-1}, which are typical models of lignin, in the spectrum of MCC_2 sample may be related to the high content of lignin in MCC_2 sample, as compared to MCC_1.And, between 3337.8 cm^{-1} and 2848.5–2915.4 cm^{-1} absorption interval, the signal of valence vibrations of the −OH group and C–H bond is observed.

If the vibrational signal of the C=C valence double bond in carbon is observed at a wavelength of 1622.4 cm^{-1}, the deformation movement of the CH_2 group can be observed in the range of 1434.3–1361.5 cm^{-1}. The absorption bands and the intensity of functional groups of the MCC molecule obtained from sunflower seed husks in the IR spectrum are similar to the structure of MCC obtained from annual plants in the works of other authors [8].

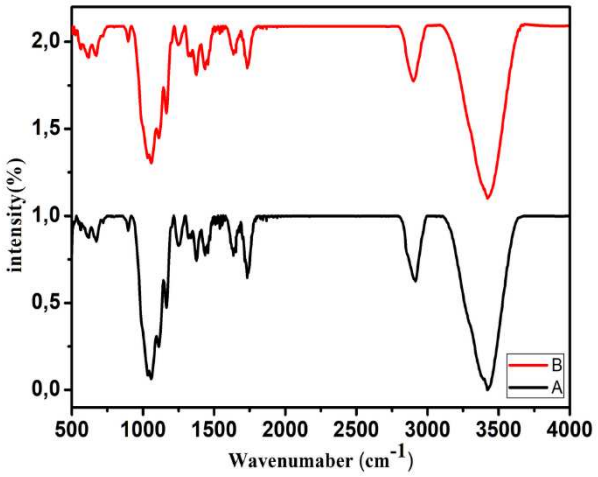

Figure 1. IR spectra of MCC from hybrid sunflower seed husk: A – MCC_2, B – MCC_1

Micrographs of MCC_1 and MCC_2 samples are presented in Figure 2. The structure of the obtained MCC samples is similar to each other and consists of broken and flat fibers of different lengths and widths, with an uneven surface. The average size of the fibers is 224–280 μm in length and 27–32 μm in width for MCC_1 (pictures 2a, b), and 219–285 μm and 29–30 μm for MCC_2, respectively (pictures 2c, d). It can be observed that the concentration of peroxyacetic acid does not affect the morphology and dimensions of MCC fibers obtained from hybrid sunflower seed husk. The morphological dimensions of the obtained MCC fibers are consistent with the results of studies on the extraction of microcrystalline cellulose from sunflower seed husks [8].

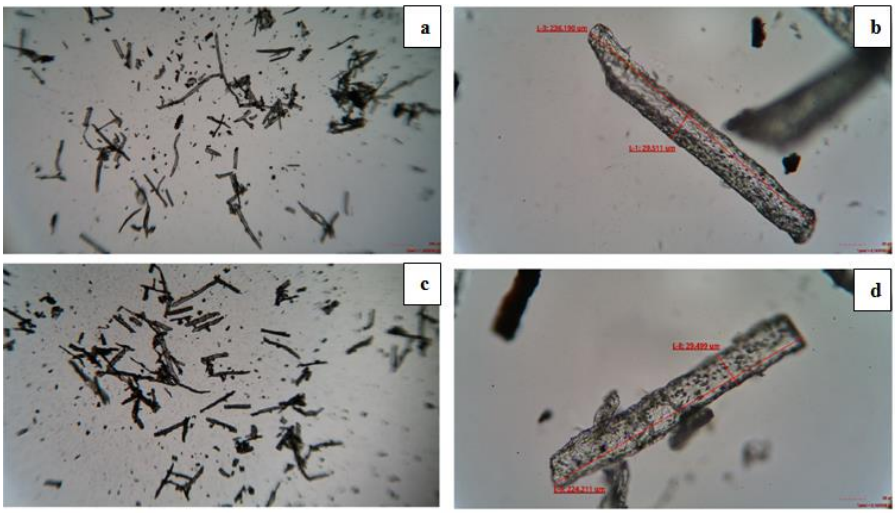

Figure 2. Micrographs of MCC from hybrid sunflower seed husk (with magnification): a) MCC_1x10; b) MCC_1x40; c) MCC_2x10; d) MCC_2x40

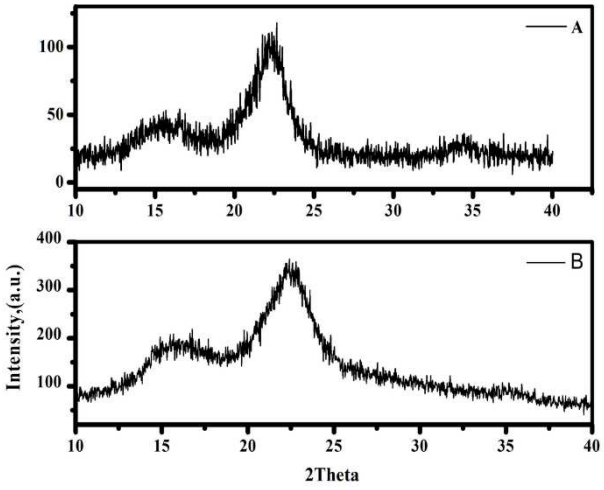

Figure 3. XRD analysis of MCC from hybrid sunflower seed husk: A – MCC_2, B – MCC_1

Intense diffraction peaks 16.81°, 22.25°, 35.78° at 2θ in the X-ray diffraction pattern of MCC_1 and MCC_2 samples indicate that most MCC macromolecules are crystallographically oriented [7, 17]. That is, MCC samples can be conditionally called a crystalline structure. But, unlike MCC_2, the diffractogram of the MCC_1 sample is distinguished by a higher intensity of diffraction peaks (Figure 3).

Therefore, the use of glacial acetic acid in the organosolvent oxidation process reduces the amount of amorphous structure in cellulose and causes an increase in the amount of crystallographically oriented structures.

The amount of residual lignin in MCC_1 and MCC_2 samples obtained by using glacial acetic acid is 7.7 times lower in MCC_1 sample compared to the amount of lignin in MCC_2 obtained using 42% acetic acid, which is an additional evidence of a significant reduction of the amorphous part. The results obtained are in good agreement with previous studies [2–3, 18].

Conclusion

Microcrystalline cellulose samples MCC_1 and MCC_2 were obtained from P63LE10 "Pioneer" (USA) hybrid sunflower seed husks by the organosolvent oxidation method using glacial acetic and 42% acetic acids in a 1/12 ratio of raw material and solvent. The yield of the MCC_1 sample was 32.80%, and the amount of α-cellulose was 53.4%, the yield of the MCC_2 sample was 3.7% higher than MCC_1, and the amount of α-cellulose was 3.6% higher, respectively. The amount of residual lignin in the obtained MCC samples was equal to 3.1% for MCC_1 and 24% for MCC_2. This was further confirmed by the X-ray diffractogram results, and it was found that MCC_1 and MCC_2 samples have a crystalline structure, and the MCC_1 sample has a significantly higher crystalline area. Although the chemical structure of the samples was similar according to the IR spectrum, it was observed that the lignin signals were more intense for the MCC_2 sample compared to the MCC_1 sample. It was known that the fibers of MCC samples are similar in size, that is, their length is 219–285 μm, and their width is 27–32 μm.

In conclusion, the use of glacial acetic acid in the production of MCC by organosolvent oxidation of P63LE10 "Pioneer" (USA) hybrid sunflower seed husks allows the development of MCC with a low amount of residual lignin and a crystalline structure.

That is, in the delignification process, glacial acetic acid fragments more amorphous particles than 42% acetic acid, and as a result of the effect on their transition to the melting solution, MCC with a large number of crystallographically oriented parts is obtained. Therefore, the concentration of acetic acid used in the production of MCC by the organosolvent method is an important factor directly affecting the yield and quality of the resulting cellulose.

However, the use of 42% acetic acid is effective in obtaining high-yield microcrystalline cellulose in an environmentally and economically optimal way.

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ГИБРИДТІК СҰРЫПТЫ КҮНБАҒЫС ТҰҚЫМЫ ҚАУЫЗЫНАН КРИСТАЛЛОГРАФИЯЛЫҚ БАҒДАРЛАНҒАН МАКРОМОЛЕКУЛАЛЫ ЦЕЛЛЮЛОЗА АЛУ

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Қазіргі таңда, целлюлоза негізіндегі өздігінен ыдырайтын, табиғатқа зиян келтірмейтін композиттік материалдарды алу және оны қолданысқа енгізу – бірқатар экологиялық мәселелерді шешуге мүмкіндік беруде. Осыған байланысты, целлюлоза алуға болатын шикізат көздерінің қатары көбейіп, оларды қолдануда экономикалық және экологиялық тиімділігі, алынатын целлюлозаның қасиеттері кеңінен зерттелуде.

Жұмыста, P63LE10 «Пионер» (США) күнбағыс тұқымы қауызынан мұзды сірке қышқылы мен концентрациясы екі есеге төмендетілген 42%-дық сірке қышқылы негізінде алынған пероксисірке қышқылдарын салыстырмалы түрде қолдана отырып, микрокристалды целлюлоза алу процесі зерттелді. Зерттеу нәтижесінде концентрациясы екі есе төмен сірке қышқылы қолданылған пероксисірке қышқылы қатысында алынған МКЦ-ның ($МКЦ_2$) шығымы 3,7%-ға, ал құрамындағы α-целлюлоза мөлшері 3,6%-ға жоғары болатындығы анықталды. Дегенмен, ондағы қалдық лигниннің мөлшері 24,02% тең болып, аморфты құрылым іздері (следовые количество аморфных структур) ИҚ-спектроскопия және XRD дифрактограммасы арқылы анықталды. Керісінше, мұзды сірке қышқылы негізінде алынған пероксисірке қышқылын қолдану арқылы алынған $МКЦ_1$ шығымы мен құрамындағы α-целлюлоза мөлшері $МКЦ_2$ салыстырғанда аздау болғанымен, алынған өнім макромолекулалардың кристаллографиялық бағдары бар целлюлозаның реттелген бөлігінің жоғары мөлшерімен және жоғары тазалық дәрежесімен ерекшеленеді.

Кілт сөздер: *күнбағыс тұқымы қабығы (SFH), микрокристалды целлюлоза (МКЦ), еріткіш, пероксисірке қышқылы (PAA), мұзды сірке қышқылы, α-целлюлоза, қалдық лигнин, кристалдылық.*

ПОЛУЧЕНИЕ ЦЕЛЛЮЛОЗЫ С КРИСТАЛЛОГРАФИЧЕСКОЙ ОРИЕНТАЦИЕЙ МАКРОМОЛЕКУЛ ИЗ ЛУЗГИ ГИБРИДНОГО СОРТА ПОДСОЛНЕЧНИКА

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В настоящее время получение и внедрение к применению биоразлагающихся композиционных материалов на основе целлюлозы, не наносящих вреда природе, позволяют решить ряд экологических проблем. В связи с этим увеличивается количество сырья, из которого можно получить целлюлозу, широко изучаются экономическая и экологическая эффективность его использования и свойства получаемой целлюлозы.

В работе изучен процесс получения микрокристаллической целлюлозы из лузги семян подсолнечника P63LE10 «Pioneer» (США) с использованием пероксиуксусных кислот на основе ледяной уксусной кислоты и 42%-ной уксусной кислоты с уменьшенной в два раза концентрацией. В результате исследования установлено, что выход МКЦ ($МКЦ_2$), полученной в присутствии пероксиуксусной кислоты при вдвое меньшей концентрации уксусной кислоты, выше на 3,7%, а содержание α-целлюлозы – выше на 3,6%. Однако количество остаточного лигнина в нем равно 24,02%, следы аморфной структуры (следовые количества аморфных структур) определены методами ИК-спектроскопии и рентгенодифрактограммы. Напротив, хотя выход и содержание α-целлюлозы $МКЦ_1$, полученного с использованием пероксиуксусной кислоты на основе ледяной уксусной кислоты, ниже, чем у $МКЦ_2$, но полученный продукт характеризуется высокой степенью чистоты и высоким содержанием упорядоченных частей целлюлозы с кристаллографической ориентацией макромолекул.

***Ключевые слова**: лузга подсолнечника (SFH), микрокристаллическая целлюлоза (МКЦ), растворитель, пероксиуксусная кислота (PAA), ледяная уксусная кислота, α-целлюлоза, остаточный лигнин, кристалличность.*