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STRUCTURAL AND MORPHOLOGICAL FEATURES OF MICROCRYSTALLINE CELLULOSE FROM INDUSTRIAL HEMP HURD

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Currently, there is increased interest in growing hemp as well as in large-scale hemp products. The main research focuses on the use of seeds and fibres. At the same time, the remaining hurd is proposed to be used for mulching, making insulation and bedding for animals. Due to the cellulose's high content in its composition with a relatively low content of inorganic components, it can be a promising raw material for obtaining microcrystalline cellulose (MCC). Therefore, our work aimed to obtain MCC from hemp husks, establish its physicochemical characteristics and compare them with the indicators of MCC previously obtained from another flax culture. Air-dry hemp hurd, waste after the fibre extraction from technical hemp, was used for the research. It has the following characteristics: humidity of 8 %, the proportion of organic components to dry weight of 97.3 % (cellulose – 48.4, hemicellulose – 25.8, lignin – 20.9 % mass) and inorganic components – 2.7 %. To obtain microcrystalline cellulose, the hemp hurd was subjected to organo-solvent cooking. The structure and morphology of the MCC were studied using methods such as XRD, XRF, FTIR-ATR, low-temperature nitrogen sorption-desorption, AFM, TGA, and DSC. It was found that by the organo-solvent cooking method, it is possible to obtain MCC with a yield of 83.2 %. The resulting product was a white, tasteless, and odourless substance with 96.9 % organic components (including 98.5 % cellulose and 1.5 % lignin) and 3.1 % inorganic components (including 91.4 % SiO_2). The XRD method confirmed the presence of a crystalline component in the obtained MCC due to the availability of the intensity of the peak reflex in the region 2θ = 22–23° which corresponds to the plane 002 of the crystal lattice of natural cellulose I. Based on these data, the crystallinity index was calculated – 0.88. The FTIR spectrum of the sample shows typical functional groups corresponding to MCC. There are two distinct mass loss steps in thermograms (TGA). It was found that the obtained samples had a specific surface area of 2.6 m^2/g and a pore diameter of 3.6 nm, which indicates an MCC's non-porous structure. The AFM method shows that the particles are distributed throughout the scan, while there are no clusters of particles and their agglomerates, the height of which elements varies from 5.0 to 11.1 nm. Surface roughness Ra = 1.3–1.4 nm.

***Keywords**: microcrystalline cellulose, hemp hurd, flax, organo-solvent cooking, relief of the surface*

INTRODUCTION

Hemp was one of the first cultivated fibre plants. It is known about its use in the ancient civilization in Northern China as early as 10.000 BC [1]. Over time, hemp became a valuable raw material for many industries: textiles (fabrics, threads, ropes), pharmaceuticals (medicines), cosmetology (cosmetics, shampoos, soaps, creams), fuel and lubricants (fuels, oils, lubricants, special liquids), paint and varnish (paints, solvents, varnishes), food (flour, food additives, beer, teas, *etc*.), veterinary (bedding and feed for animals), agriculture (soil remediation, crop rotation, mulch, compost), construction (insulation, building materials), paper and other materials [2–5]. In 1961, the United Nations adopted the Single Convention on Narcotic Drugs, which are not used for medical purposes [6]. It still works today. Its introduction led to a rapid decrease in the area of technical hemp, which, at that time, few people

distinguished from narcotic hemp. At the time of its adoption, Ukraine was considered the leader in its cultivation among the countries of the Soviet Union [3]. According to studies [7], over the last century, the cultivated area of industrial hemp in Ukraine has decreased more than 600 times. At the same time, technical hemp, unlike narcotic cannabis, contains less a specified concentration of delta-9 tetrahydrocannabinol [8], which in turn makes it possible to classify them as safe cultures. Therefore, currently, in Canada, the USA and the EU countries, legal technical hemp is equated with ordinary crops [8]. These large-scale hemp products prompted the rapid attention of scientists and entrepreneurs to technical hemp. The main research focuses on the use of seeds and fibres. At the same time, the remaining hurd is proposed to be used for mulching, making insulation and bedding for animals. The cost of one ton of hurd is from roughly €200–€450 ($230–$515) [9]. However, considering that more than a third of the hurd consists of cellulose and is a low-ash raw material, it can be a promising cheap raw material for obtaining cellulose products. Because of the rapid growth of demand and the expansion of the fields of application, one of its most popular varieties is microcrystalline cellulose (MCC) [10–16]. In work [17] it was shown that MCC with slightly various characteristics is obtained from different raw materials. Therefore, the aim of our work was to obtain MCC from hemp hurd, evaluate its physicochemical characteristics and compare them with the indicators of MCC previously obtained from another flax culture.

MATERIALS AND METHODS

Air-dry hemp hurd, a waste of the technical hemp crop Hlesia varieties of the 2023 harvest, (fraction 20–50 mm) from Chernigiv's region of Ukraine with the following characteristics: humidity 8 %, the proportion of organic components to dry weight of 97.3 % (cellulose – 48.4, hemicellulose – 25.8, lignin – 20.9 % mass) and the proportion of inorganic components – 2.7 % was used. The chemical composition of hemp hurd ash (inorganic components) determined by using XRF are shown in Table 1.

Table 1. Composition of initial industrial hemp hurd ash

Elements	Content %mass.
O	36.012±0.157
Si	13.695±0.163
P	0.838±0.067
S	1.887±0.030
Cl	1.771±0.040
K	5.596±0.090
Ca	36.904±0.144
Fe	2.557±0.016
Mn	0.314±0.007
Ti	0.427±0.016

MCC was obtained from hemp hurd by the method of organ-solvent cooking described in [18, 19]. The cellulose yield (Y) was calculated using Equation (1) in [18]. The contents of cellulose, hemicellulose, lignin and other chemical components in cellulosic products were determined by standard chemical analysis, described earlier [20]. All chemical analysis was carried out twice allowing calculating the mean values and standard deviations, which do not exceed 5 %. The inorganic components were determined using Expert 3L XRF (INAM, Ukraine). The degree of polymerization (DP) of cellulose samples was determined by viscosity measurements in a cadmium ethylenediamine solution using an Ostwald viscometer [21]. The phase identification of the products was examined under X-ray diffraction (XRD) using the MiniFlex 300/600 diffractometer (Rigaku, Japan). The diffraction patterns were recorded using Cu$K\alpha$ radiation ($\lambda = 0.15418$ nm), the operating voltage of 40 kV and a current of

15 mA. XRD pattern of samples was obtained in the 2θ range between 10° and 50° with a step of 0.02°. The crystallinity index (CI) [22] was calculated according to Eq. 2 in [18]. FTIR analysis of the obtained cellulose was performed using an IRAffinity-1S FTIR spectrometer (Shimadzu, Japan) equipped with a Quest ATR Diamond GS-10800X (Specac, UK) within the wavenumber range of 4000 to 400 cm^{-1}. The surface morphology was investigated with an atomic force microscope (AFM) NT-206 (double liability company "Microtestmachines", Belarus) equipped with standard sonde CSC37 and rigidity of console 0.3–0.6 N/m. The scan was run in a contact static mode at 10 mcm/s with a step of 0.3 nm. Samples of cellulose (4 mg) were stirred in ethyl alcohol (5 ml) for 15 min. The resulting suspension (0.25 ml) was applied to quartz glass and dried at 50 °C to constant weight. Then the scan was performed on AFM. Thermogravimetric analysis (TGA) and differential scanning calorimetric analysis (DSC) were performed with a PT1600 TG-DTA/DSC (STA Simultaneous Thermal Analysis, LINSEIS Messgeräte GmbH, Germany). The samples (13.0 ± 0.1 mg) were collected in a standard corundum pan. The scan was run at 5 °C/min under a flow of air. The mass change was measured from 15.8 to 800 °C. The sample was analysed three times. The porous properties of the MCC were studied using N_2 adsorption at − 195.8 °C on the specific surface area and a porosity analyser Nova 1200e (Quantachrome, USA). Prior to analysis, 0.05–0.08 g of powder sample was degassed at 120 °C for 3 h to remove previously adsorbed gases and dead space by vacuum pump to cool down the sample at room temperature. The specific surface area and porosity of the catalyst were calculated using methods Brunauer-Emmett-Teller (BET) and Barrett-Joyner-Halenda (BJH) respectively.

RESULTS AND DISCUSSION

A white, tasteless and odorless MCC was obtained under the earlier described method from hemp hurd. The MCC's percentage yield was calculated by Eq. 1 [18] (Table 2). The results of the chemical composition of obtained MCCs were determined using the above-described methods and the data were summarised in Table 2. The chemical composition of MCC ash (inorganic components) determined by using XRF are shown in Table 3.

Table 2. The main characteristics of obtaining MCC

	Cellulose yield (Y), %mass.	Ash, %mass.	Cellulose, %mass.	Klason lignin, %mass.	CI, %	DP
Hemp hurd	83.2	3.1	98.5	1.5	0.88	180
Flax [23]	99.6	1.6	97.7	2.3	0.86	277

Table 3. Composition of hemp hurd MCC ash

Elements	Content %mass.
O	50.009±0.183
Si	43.432±0.178
S	0.062±0.007
K	3.992±0.077
Ca	0.952±0.028
Fe	0.890±0.011
Ti	0.663±0.006

Fig. 1 presents the X-ray diffraction (XRD) patterns of the MCC from hemp hurd compared to the previously described MCC from flax [23]. On the diffraction patterns of cellulose samples (Fig. 2) the peaks were observed at: 14–16°; 22–23°; 34–35°, relating to the reflection of the planes 10–1, 101; 002; 040 cellulose crystal lattice, respectively. The intensity of the peak reflex in the region 2θ = 22–23° corresponds to the plane 002 of the crystal lattice of natural

cellulose I [24]. There is no doublet peak at the 002 plane (Fig. 1) which according to the work [25], indicates that all the samples comprised cellulose-I polymorph. Peaks in the region 2θ = 15–16° are associated with the diffraction of X-rays from the planes 10-1 and 101 of the crystal lattice of cellulose I. Profile of amorphous cellulose scattering has a characteristic diffusion character with a maximum of 2θ = 18.5–19°. Differences in peak height and width of samples in the angle (2θ) range of 18.5°–19° and 22°–23° are indicative of differences in their degree of crystallinity (Table 2). This is also confirmed by the values of the degree of polymerization (Table 2). As can be seen from those shown in Fig. 1, curves of MCC have closely related XRD patterns, which is consistent with the data given in Table 2. High crystallinity indicates an ordered compact molecuar structure, when the crystalline cellulose content is high.

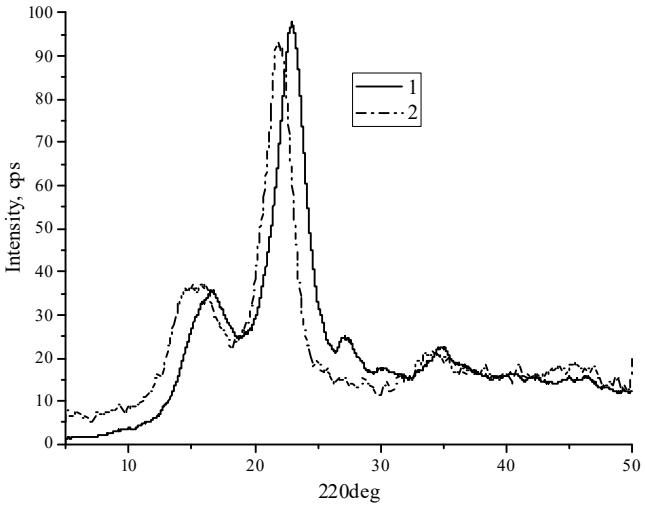


Fig. 1. XRD patterns of MCC obtaining from industrial hemp hurd (*1*) compared with flax (*2*) [23]

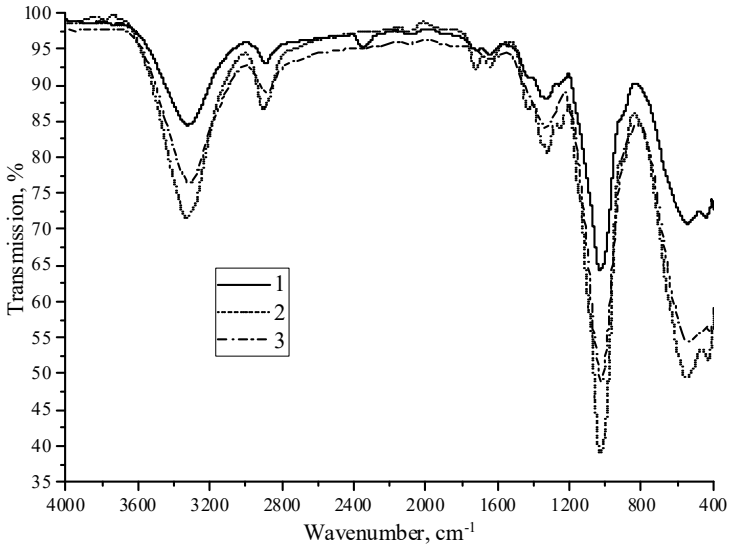


Fig. 2. FT-IR spectra of obtained MCC from industrial hemp hurd (*1*) compared to flax (*2*) [23] and commercial M-102 (*3*) MCC's

Fig. 2 demonstrates the FTIR-ATR spectra of the obtained MCCs compared to the flax and industrial design. Spectra at 3600–2995 (OH stretching, hydrogen bonds), 2890 (C–H asymmetric and symmetric tensile vibration), 1430 (symmetric CH_2 bendings), 1330 (CH_2

rocking vibration at C-6 position), 1030 (ring vibration and C–OH bending) and 900 cm^{-1} (corresponding to β-glycosidic linkages) indicate crystal and amorphous regions. A more detailed description of the FTIR spectrum of cellulose can be found in the literature [23]. According to the work [25], the presence of a MCC peak at 1330 cm^{-1} for all samples indicates that all the samples comprised cellulose-I polymorph. This is also supported by the XRD patterns of MCC.

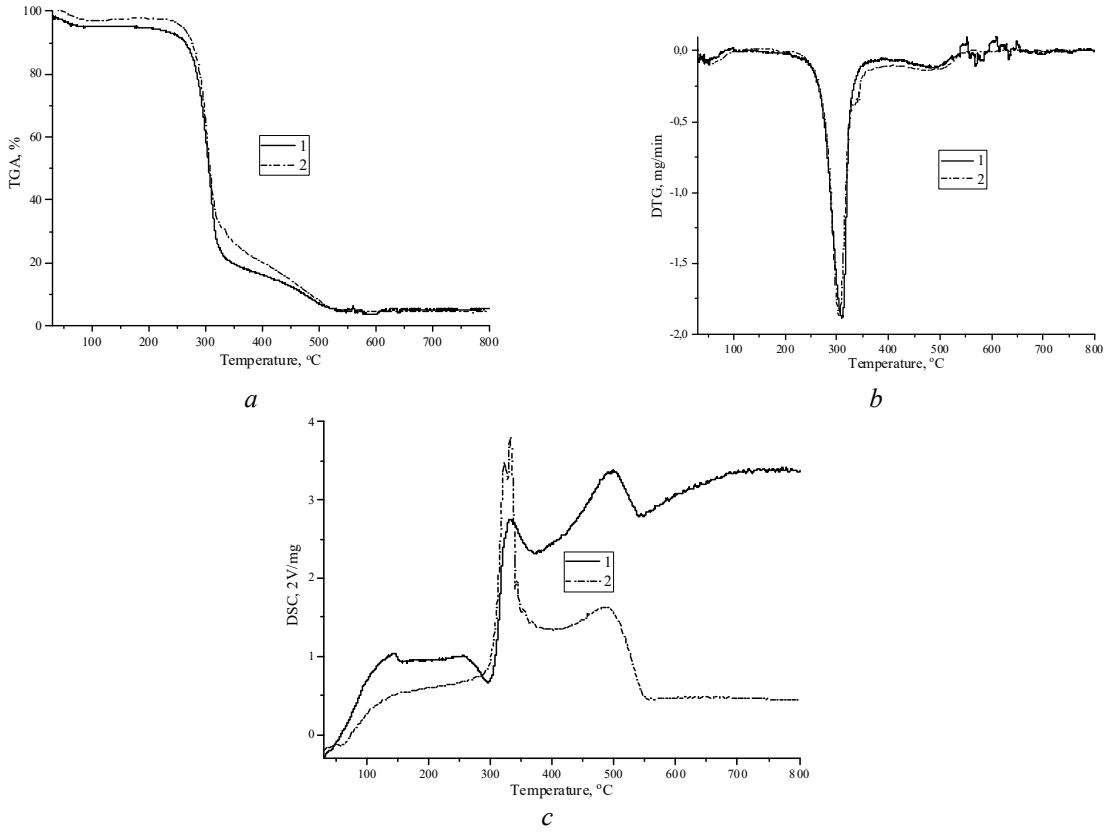
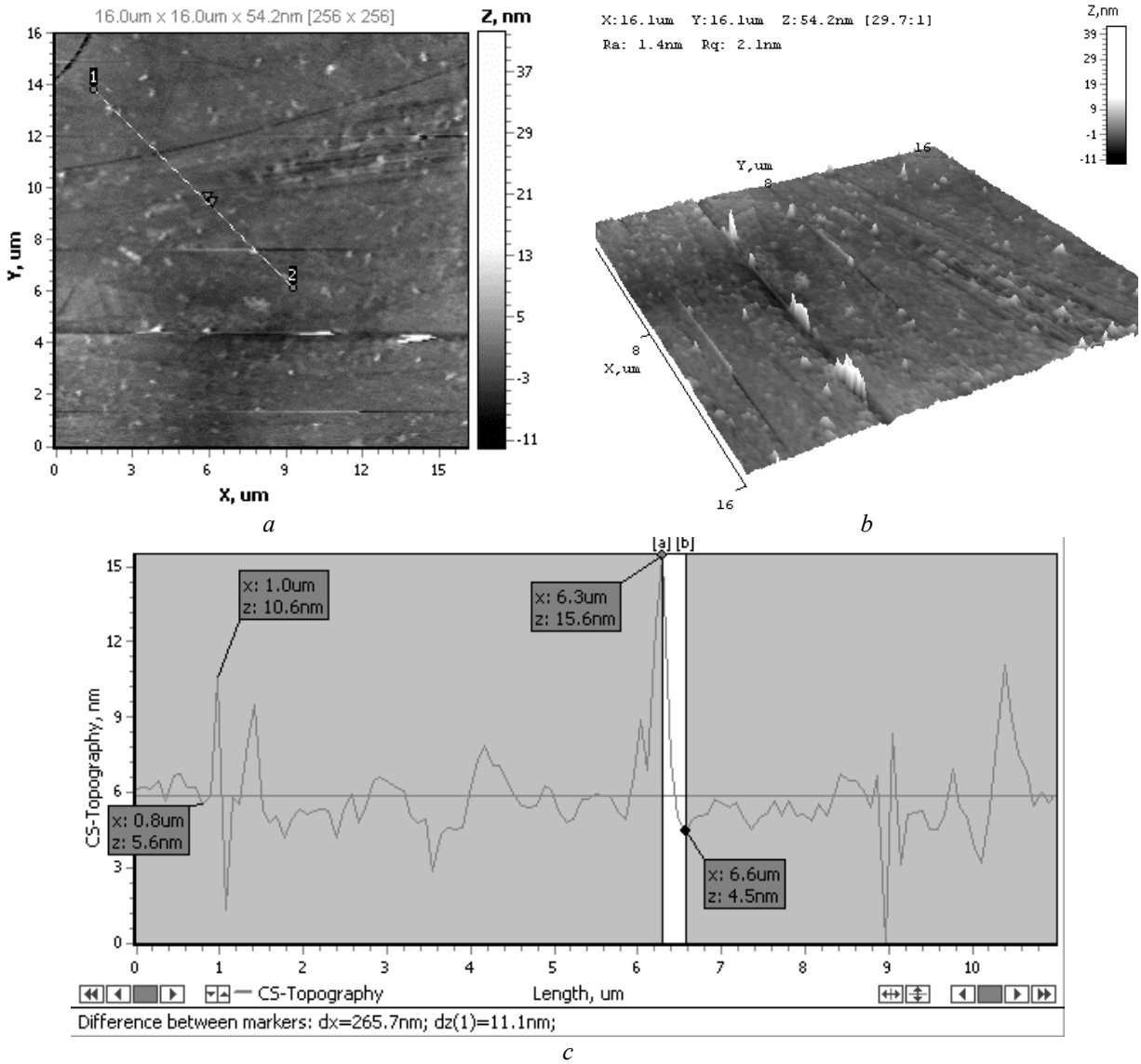


Fig. 3. TGA (*a*), DTG (*b*) and DSC (*c*) curves MCC isolated from from industrial hemp hurd (*1*) compared to flax (*2*) [23]

Thermogravimetric analysis (TGA), derivative thermogram (DTG) and differential scanning calorimetry (DSC) data depending on temperature for MCC obtained from industrial hemp hurd compared to flax are presented in Fig. 3. The DSC signal was measured together with the TGA curve. Initial weight loss of MCC from industrial hemp hurd (4.9 %), and flax (3.0 %) was observed (Fig. 3 *a*) with endothermic peaks on DTA (Fig. 3 *b*) curves at respective temperatures of 48.4, and 54.5 °C. It's due to evaporation and dehydration of adsorbed and surface water [26, 27]. This loss of mass lasts up to 120 °C. The TGA (Fig. 3 *a*), DTG (Fig. 3 *b*) and DSC (Fig. 3 *c*) curves show that cellulose samples should not be heated above 225 °C. As can be seen from the data of the TGA curve (Fig. 3 *a*), above this temperature a huge weight reduction occurs, which was attributed to the thermal degradation and complete degradation of cellulose and its conversion to char [27, 28]. According to [29, 30] the onset stage concerns the degradation, decarboxylation, depolymerisation and decomposition of glycosyl units in cellulose is believed to be due to the emission of non-combustible gases such as carbon dioxide, carbon monoxide, formic acid, and acetic acid while the second degradation stage is believed to be due to pyrolysis and emission of combustible gases. The peak decomposition temperature of the samples is visible on the derived weight loss curve (Fig. 3 *b*). The main peak decomposition temperature of MCCs is 309.2 and 304.6 °C for MCCs from industrial hemp hurd, and flax, respectively, which is in good agreement with the literature data [26, 28, 31]. This destruction process is two-stage. It's also well reflected in

the two DSC peaks shown in Fig. 3 *c* and two derived DTG peaks (Fig. 3 *b*). Final residue at 800 °C was highest for MCC from industrial hemp hurd (5.1 %), whereas MCC from flax exhibited the lowest (4.3 %) value. This is also confirmed by the values of the MCC's ash content (Table 2).

Fig. 4 *a* shows a 2D image of the MCC surface with a cross-section line. The particles are distributed throughout the scan, while there are no clusters of particles and their agglomerates. This is clearly visible in 2D (Fig. 4 *a*) and 3D (Fig. 4 *b*) images. The surface roughness is 1.4 nm. The analysis of the cross-sectional profile (Fig. 4 *c*) on the 2D image (Fig. 4 *a*) indicates the nanosize of the particles (the height of the elements ranges from 5.0 to 11.1 nm).



a *b* *c*

Fig. 4. 2D (*a*) and 3D (*b*) images of surfaces with shown cross-section lines (*a*) and the profile of cross-section lines (*b*)

If the size of this scan is reduced to 10.4×10.4 μm (Fig. 5 *a*), it is possible to determine more clearly the sizes of the relief particles along the selected section line, and their shape on the 3D image (Fig. 5 *b*). The last one shows that the particles are pyramids of different configurations. The surface roughness is *R*a = 1.3 nm. Analysis of the profile of the cross-section lines (Fig. 5 *c*) shows that the heights of the particles range from 2.6 to 4.5 nm.

It was found that the obtained MCC samples from industrial hemp hurd had a specific surface

area of 2.6 m^2/g, a pore volume of 0.008 cm^3/g, and a pore diameter of 3.6 nm, which indicates an MCC's non-porous structure. These are in good agreement with the literature data [32, 33].

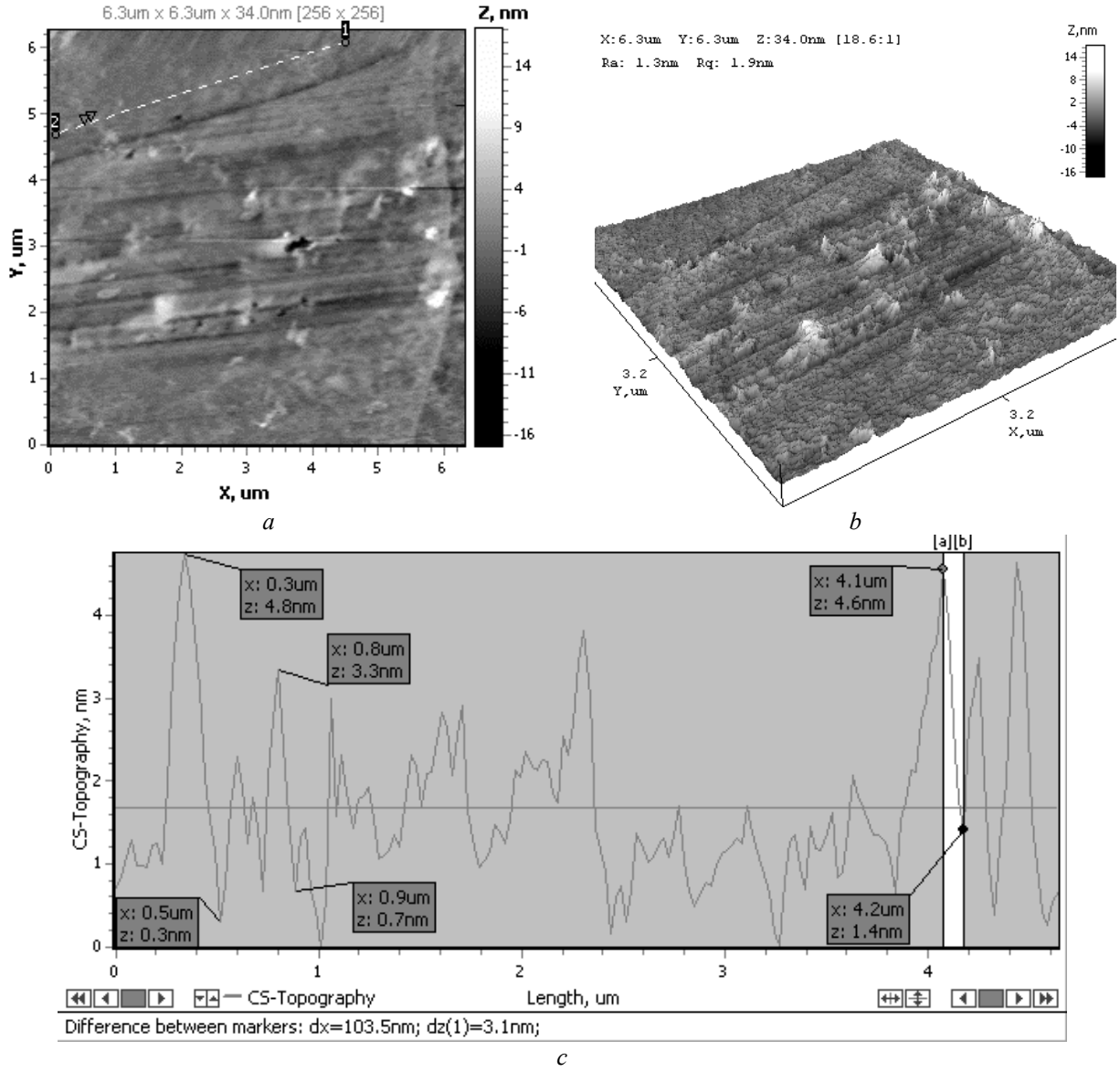


Fig. 5. 2D (*a*) and 3D (*b*) images of the surface with a cross-sectional line and profile (*c*)

CONCLUSION

The possibility of obtaining MCC from a hemp hurd by the method of organic solvent cooking has been studied. It was found that the yield of the cellulose product from hurd is 15 % (wt.) lower than from flax. However, hemp MCC contains 0.8 % less lignin than flax. It was shown that MCC from hemp hurd compared to flax has a lower degree of polymerization (180 vs. 277), which in turn provided a higher crystallinity index (0.88). Additional confirmation of the quality of the obtained cellulose is the comparison of the FTIR spectra of the studied samples compared to the industrial sample. Analysis of AFM scans shows that particle heights range from 2.6 to 11.1 nm. In this way, it was found that it is possible to obtain MCC from a hemp hurd using the method of organic solvent cooking.

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Структурні та морфологічні особливості мікрокристалічної целюлози із костри технічної коноплі

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Нині існує підвищений інтерес до вирощування конопель, а також до продуктів з неї. Основні дослідження спрямовані на використанні насіння та волокон. Водночас костру, яка залишається, пропонується використовувати для мульчування, виготовлення утеплювачів та підстилок для тварин. З огляду на високий вміст целюлози в її складі при низькому вмісті неорганічних складових вона може бути перспективною сировиною для отримання мікрокристалічної целюлози (МКЦ). Тому метою нашої роботи було отримання МКЦ з костри коноплі, встановлення її фізико-хімічних характеристик та порівняння їх з показниками МКЦ, отриманої раніше з іншої луб'яної культури льону. Для досліджень використовували повітряно-суху конопляну костру – відходи після вилучення волокна з технічних конопель. Вона має такі характеристики: вологість 8 %, частка органічних компонентів до сухої маси 97.3 % (целюлози – 48.4, геміцелюлози – 25.8, лігніну – 20.9 % мас.) та неорганічних компонентів – 2.7 %. Для одержання мікрокристалічної целюлози конопляну костру піддавали органо-сольвентному варінню. За допомогою методів низькотемпературної адсорбції-десорбції азоту, XRD, XRF, FTIR-ATR, AFM, TGA та DSC досліджено структуру та морфологію МКЦ. Встановлено, що органосольвентним методом варіння можна отримати МКЦ з виходом 83.2 %. Отриманий продукт був білою речовиною без смаку та запаху з 96.9 % органічних компонентів (включаючи 98.5 % целюлози 1.5 % лігніну) та 3.1 % неорганічних компонентів (включаючи 91.4 % SiO_2). Методом XRD підтверджено наявність кристалічної складової в отриманому МКЦ за рахунок наявності інтенсивності пікового рефлексу в області $2\theta = 22–23°$, що відповідає площині 002 кристалічної ґратки природної целюлози I. За цими даними розраховано індекс кристалічності – 0.88. Спектр FT-IR зразка показує типові функціональні групи, що відповідають МКЦ. На термограмах (TGA) є дві чіткі області втрати маси. Встановлено, що отримані зразки мають питому поверхню 2.6 $м^2/г$ та діаметр пор 3.6 нм, що свідчить про непористу структуру МКЦ. Метод AFM показує, що частинки розподілені по всьому скану, при цьому відсутні скупчення частинок та їхні агломерати, висота елементів яких коливається від 5.0 до 11.1 нм. Шорсткість поверхні Ra = 1.3–1.4 нм.

***Ключові слова**: мікрокристалічна целюлоза, костра коноплі, льон, рельєф поверхні*

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