DOI: 10.20535/kpisn.2021.3.251456
UDC 676.166.6 + 620.3

V.A. Barbash*, O.V. Yashchenko, O.S. Yakymenko, R.M. Zakharko
Igor Sikorsky Kyiv Polytechnic Institute, Kyiv, Ukraine
*corresponding author: v.barbash@kpi.ua

EXTRACTION OF ORGANOSOLV PULP AND PRODUCTION OF NANOCELLULOSE FROM HEMP FIBRES

Received: 28 Oct. 2021. Accepted: 7 Dec. 2021.

Background. The use of cellulose and nanocellulose instead of synthetic polymers makes it possible to improve the consumer properties and environmental friendliness of composite materials. Therefore, the development of technologies for the production of organosolv pulp and nanocellulose from plant raw materials, in particular from hemp fibres, is an urgent scientific and practical problem.
Objective. The purpose of the paper is to obtain pulp from hemp fibres by the peracetic method and to study the effect of the concentration of sulfuric acid and the temperature of hydrolysis of organosolv pulp on the quality parameters of hemp nanocellulose.
Methods. Treatment of hemp fibres was performed in two stages: alkaline extraction and organosolv cooking at a temperature of 97 ± 2 °C. Nanocellulose was obtained by hydrolysis with a solution of sulfuric acid of various concentrations. The resulting nanocellulose was examined by scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FTIR), and atomic force microscopy (AFM).
Results. Organosolv hemp pulp with a residual lignin content of 0.16% and an ash content of 0.08% was obtained. The nanocellulose particles obtained from it had a transverse size of 8–36 nanometers and a length of several micrometers. Nanocellulose films had high mechanical properties: density up to 1.54 g/cm^3 and tensile strength up to 60 MPa.
Conclusions. Carrying out thermochemical treatment of hemp fibres in two stages makes it possible to obtain pulp with a minimum residual content of lignin and minerals, which is suitable for the production of nanocellulose. Nanocellulose has been successfully isolated from organosolv hemp pulp by acid hydrolysis. Subsequent ultrasonic treatment allows obtaining a stable nanocellulose gel with high mechanical properties.
Keywords: hemp fiber; organosolv pulp; nanocellulose; acid hydrolysis.

Introduction

One of the environmental problems of our time is the replacement of polymers, produced from non-renewable natural resources (oil, gas, coal), with polymers from plant materials. The use of natural polymers from plant materials is considered as an alternative to plastics and contributes to the sustainable development of society, solving environmental and economic problems in the production of consumer goods [1], [2]. The most common biopolymer on Earth is cellulose, the main component of lignocellulosic biomass. In recent decades, technologies for processing cellulose into ethers and esters, microcrystalline cellulose, oxycellulose and nanocellulose have been actively developed [3], [4].

Nanocellulose is a new class of nanomaterials, which has unique properties, such as high elastic modulus, high specific surface area, optical transparency, low thermal expansion, nanoscalability and being a renewable and biodegradable material, that enable its use in many fields [5], [6]. Nanocellulose have great potential application in various fields, such as medicine, tissue engineering scaffolds, catalysis, textiles, surface coatings, drug delivery, food packaging and green nanocomposite materials [7], [8]. Various extraction processes have been used for the production of nanocellulose but acid-hydrolysis is the most well-known, efficient, and widely used extraction method [9]. Hydrolysis allows you to easily destroy and remove the disordered and amorphous areas of cellulose fibres, which leads to the receipt

Рекомендуємо цитувати цю статтю так: V.A. Barbash, O.V. Yashchenko, O.S. Yakymenko, R.M. Zakharko, "Extraction of organosolv pulp and production of nanocellulose from hemp fibres", *Наукові вісті КПІ*, № 3, с. 83–90, 2021. doi: 10.20535/kpisn.2021.3.251456.

Please cite this article as: V.A. Barbash, O.V. Yashchenko, O.S. Yakymenko, and R.M. Zakharko, "Extraction of organosolv pulp and production of nanocellulose from hemp fibres", *KPI Science News*, no. 3, pp. 83–90, 2021. doi: 10.20535/kpisn.2021.3.251456.

of nanoparticles of cellulose. The hydrolysis process uses mineral acids such as sulfuric, hydrochloric, nitric, hydrobromic and phosphoric acids, or mixtures thereof. However, sulfuric acid is most often used for the hydrolysis of cellulose due to the short duration and relatively low temperature of the hydrolysis process, as well as the formation of a gel-like product [10]. It should be noted that many companies use acid-resistant equipment and complex systems for the regeneration of spent acid solution to produce nanocellulose by the process of cellulose hydrolysis with sulfuric acid to reduce the impact on the environment. Carrying out the hydrolysis process requires determining the values of technological parameters for each type of feedstock.

To obtain nanocellulose, pulp from wood is mainly used, the massive felling of which leads to a deterioration of the ecological situation. The use of harmful cooking liquors in traditional methods of delignification of wood pollutes the air space or water basins [11]. At the same time, scientists are developing more environmentally friendly methods of producing cellulose from both wood and non-wood plant raw materials [12], [13].

In this work, one of the representatives of non-wood plant raw materials, hemp fibres, widespread in Ukraine and are characterized by a high content of cellulose and hemicellulose, was used as a starting material. For the extraction of pulp from plant materials, an environmentally friendly organosolv delignification method using acetic acid and hydrogen peroxide was used. Acetic acid is used as a delignification reagent due to its relatively low cost. The use of hydrogen peroxide in pulp cooking contributes to the delignification of raw materials and an increase in its brightness. At the same time, the peroxy compounds of the cooking solution have little effect on destruction of cellulose [14].

Problem statement

Studies on the extraction of pulp suitable for the production of nanocellulose from hemp fibres using peracetic acid solutions have not been carried out. The literature also lacks data on the production of nanocellulose from hemp fibres by the process of hydrolysis of organosolv pulp with sulfuric acid. Therefore, the purpose of this work was to isolate pulp from hemp fibres by the peracetic method and to study the effect of the concentration of sulfuric acid and the temperature of hydrolysis of organosolv pulp on the qualitative parameters of hemp nanocellulose.

Methods for obtaining pulp and nanocellulose

We used the hemp fibres from the Khmelnitsky region of Ukraine after the harvest in 2020. Before research, the raw material was crushed to 2–5 mm and stored in a desiccator to maintain a constant moisture content and chemical composition. The chemical composition of hemp fibres was determined according to TAPPI standards [15]. The analyses for the chemical characterization were done in triplicate and the mean and standard deviation were calculated. All chemicals – sodium hydroxide, glacial acetic acid, hydrogen peroxide and sulphate acid were chemical grade.

The pulp was obtained from hemp fibres in two stages. At the first stage, an alkali solution was used as a preliminary treatment of plant raw materials to remove the main part of hemicelluloses and minerals and partially remove lignin. For this, the hemp fibres were extracted with a NaOH solution with concentration of 0.5 % and a consumption of 5 % from mass of absolute dry material (a.d.m.) at a temperature of 97±2 °C from 60 min to 180 min. At the second stage, to remove residual lignin and extractives, organosolv cooking was carried out using a solution of glacial acetic acid with concentration of 70 % and 35 % hydrogen peroide in a volume ratio of 7:3 at a temperature of 97±2 °C from 60 min to 180 min. These values of technological parameters were previously determined as optimal for the extraction of various representatives of non-wood plant raw materials [16]. At the end of cooking, we washed the obtained pulp with distilled water, dried in air and determined its quality indicators. The process of hydrolysis of organosolv hemp pulp was performed with solutions of sulfuric acid with concentrations of 30, 40 and 50 % at a temperature from 30 °C to 60 °C for 30 to 60 minutes. After completion of the hydrolysis process, the obtained nanocellulose was washed with distilled water using a laboratory centrifuge to neutral pH. The nanocellulose suspension was sonicated at 22 kHz for one hour to disperse the particles. The nanocellulose suspension was poured into Petri dishes to make films, which were investigated by physical and physico-mechanical methods.

Scanning electron microscopy (SEM) studies were carried out on PEM–106I (SELMI, Ukraine) microscope to observe the morphology of hemp pulps and nanocellulose films. The samples were sputter-coated with a layer of gold using the sputtering technique. Fourier transform infrared spectroscopy (FTIR) spectra of the hemp fibres, pulps and nanocellulose films were recorded on Tensor

37 Fourier-transform infrared spectrometer with a 2 cm^{-1} resolution in the 400–4000 cm^{-1} frequency range. Atomic force microscopy (AFM) was used to determine the topographic characteristics of hemp nanocellulose samples. Measurements were accomplished with Si cantilever, operating in a tapping mode on the device Solver Pro M (NT-MDT). The scanning speed and area were 0.6 line/s and 2×2 μm^2, respectively.

Results and discussion

The chemical composition of hemp fibres in comparison with the chemical composition of the most common representatives of wood are given in Table 1.

***Table* 1.** Chemical composition of hemp fibres, % from mass of a.d.m.

Raw material	Cellulose	Lignin	Extraction in		RFW*	Ash
			H_2O	NaOH		
Hemp fibres	73.9	8.8	4.2	20.2	1.9	1.6
Pine [17]	47.0	27.5	6.7	19.4	3.4	0.2
Birch [17]	41.0	21.0	2.2	11.2	1.8	0.5

**RFW – resins, fats, waxes*

As can be seen from Table 1, hemp fibres contain more than 70 % of the most valuable part of plant raw materials - cellulose, which is significantly higher than the cellulose content in wood. The lignin content in hemp fibres is 2.4-3 times less than in wood. The content of substances extracted by water in hemp fibres is in the range between deciduous and coniferous species. The content of substances extracted with 1 % alkaline solution in hemp fibres is close to the value of this indicator for softwood and almost 2 times higher than the value of this indicator for hardwood. The content of substances extracted with an alcohol-benzene mixture - resins, fats, waxes (RFW), in hemp fibres is close to the value of this indicator for hardwood and less than in softwood. In this case, hemp fibres contain more minerals (ash) than coniferous and deciduous wood. Such values of the main components of hemp fibres a priori indicate the need for lower consumption of chemicals in obtaining pulp from them compared to obtaining pulp from wood, which must be taken into account in the processes of delignification of plant raw materials and regeneration of spent cooking solution [17].

The dependence of the quality indicators of hemp pulps on the duration of alkaline extraction and organosolv cooking of the pulp after alkaline treatment is presented in Table 2.

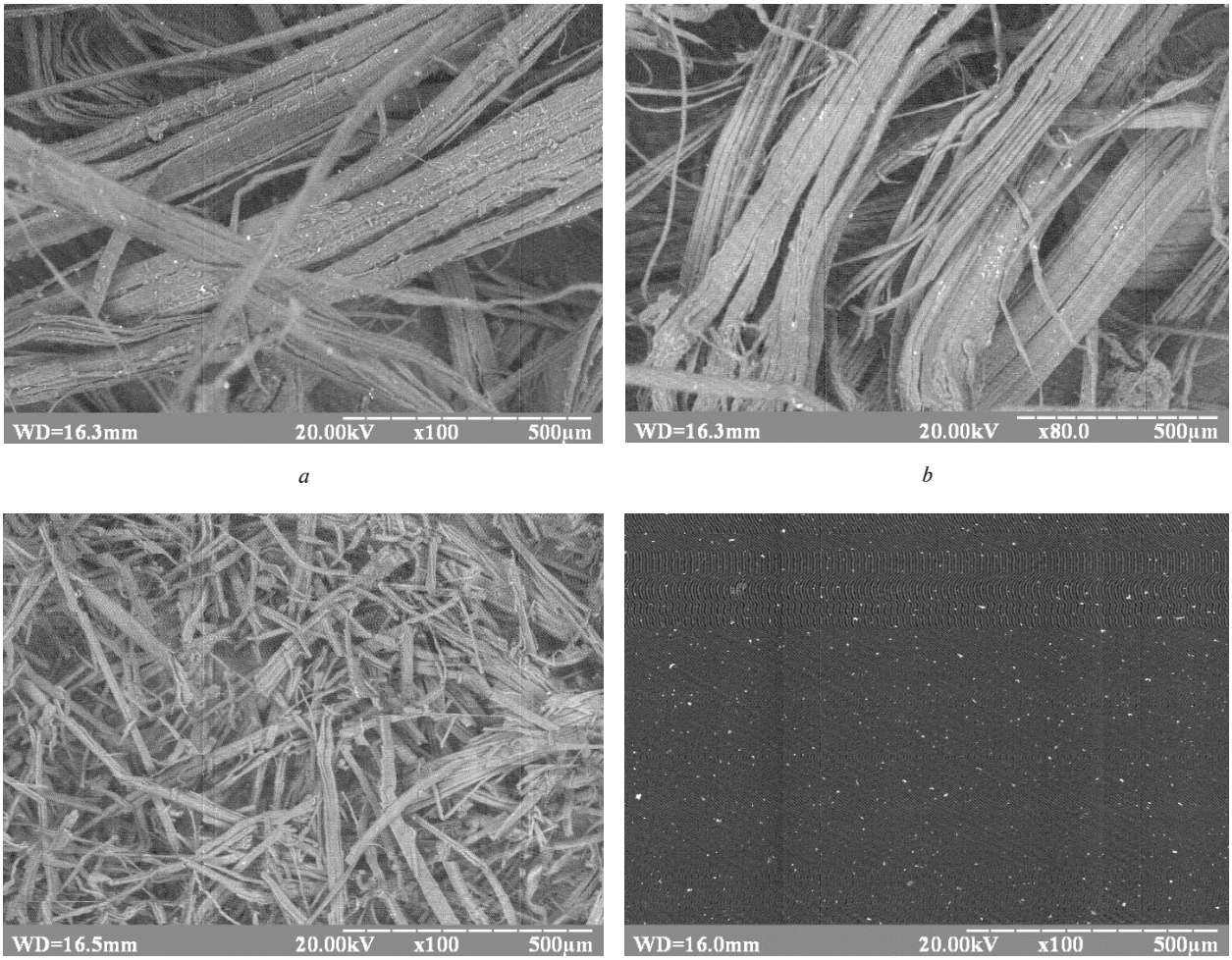
***Table* 2**. Dependence of quality indicators of hemp pulps on the duration of alkaline extraction and organosolv cooking, % from mass of a.d.m.

Duration of treatment, min	Yield, %	Residual lignin content, %	Ash content, %
60	82.6* 76.6**	8.3 0.36	0.24 0.10
120	76.6 67.4	7.9 0.31	0.18 0.09
180	75.4 64.5	6.6 0.16	0.13 0.08

*- after alkaline extraction
**- after organosolv cooking

Table 2 shows that increasing the duration of contact of hemp fibres with alkaline solution at elevated temperature leads to a decrease in the numerical values of the yield and residual content of lignin and ash in hemp pulp. For cooking organosolv hemp pulp used pulp treated with NaOH solution at a consumption of 5 % from a.d.m. for 180 minutes. As can be seen from Table 2, increasing the duration of organosolv cooking naturally reduces the yield of pulp and the residual content of lignin and minerals. The process of delignification of plant raw materials occurs due to the cleavage of α- and β- ether bonds of lignin under the action of electrophilic particles of hydroxonium H_3O^+, formed by peroxyacids in an acidic environment. The hydroxonium cation promotes the reactivity of lignin, the cleavage of ether bonds, the reduction of the molecular weight of lignin and its conversion into a cooking solution. The resulting organosolv hemp pulp has an insignificant residual content of lignin and ash and can be used for further chemical processing, in particular, to obtain nanocellulose.

The process of extraction of non-cellulosic components from plant raw materials is confirmed by SEM data (Fig. 1). As can be seen from the above data, alkaline treatment leads to remove the main part of hemicelluloses and minerals and partially remove lignin (Fig. 1b). Carrying out further peracetic cooking of pulp leads to remove residual lignin, extractives, and shortening of the sizes of fibres (Fig. 1c). Further treatment of organosolv pulp with sulfuric acid solutions leads to the rupture of 1–4 glycosidic bonds between the pyranose units of the cellulose macromolecules, to the dissolution of the amorphous part of cellulose, reducing the degree of polymerization and fibre size to nanoparticles (Fig. 1d).



a *b* *c* *d*

Fig. 1. SEM images of hemp fibres (*a*); pulp after alkali treatment (*b*); pulp after peracetic cooking (*c*) and hemp nanocellulose (*d*)

The change of chemical composition of hemp fibres in the process of its thermochemical treatment was confirmed by infrared spectroscopy. Fig. 2 shows the Fourier IR spectra of hemp fibres, pulps after alkaline treatment and cooking. All spectra are characterized by a wide bandwidth in the region of 3000–3800 cm^{-1}, which corresponds to stretching vibrations of hydroxyl groups included in intramolecular and intermolecular hydrogen bonds. The bands in the area of 3000–2800 cm^{-1} correspond to the asymmetric and symmetric stretching vibrations of the $–CH_2$, –CH and hydroxyl groups [18]. Their number naturally decreases in the course of thermochemical processing of raw material, as evidenced by a decrease in intensity peaks in this area. In the Fourier IR spectra of hemp pulps after stages I and II (spectra 1 and 2 in Fig. 2), in comparison with the spectrum of the initial plant raw material, a decrease in the intensity of bands in the region of 1500–1800 cm^{-1}, which characterize the bands of stretching vibrations of double bonds.

Vibration bands in the 1740 cm^{-1} region indicate the presence of a carbonyl group characteristic of hemicelluloses. As can be seen from Fig. 2, alkaline treatment significantly removes hemicelluloses from the cellulose composition, but subsequent organosolv cooking increases the amount of carbonyl groups due to oxidation by hydrogen peroxide. A decrease in the intensity of vibrations in region of 1700 cm^{-1}, which is characteristics of aromatic compounds – residual lignin, indicates a removal of lignin from plant material and pulp during their thermochemical treatments (spectra 3 in Fig. 2). In the region 1200–1450 cm^{-1} absorption bands are located due to bending vibrations of the angles and bonds of the CH_2OH group at C6 of cellulose. The band in the region of 1430 cm^{-1} are due to the deformation vibrations of the CH_2 groups, the bands at 1360 and 1340 cm^{-1} are due

to deformation vibrations of hydroxyl groups. The band at 1160 cm^{-1} is due to the asymmetric vibrations of the C–O bonds, while the band at 1060 cm^{-1} corresponds to the vibrations of the C–O–C bridge of the glucopyranose ring of cellulose [19]. The increase of the intensity of the bands in the region of 1250, 1500 i 1700 cm^{-1} demonstrates the efficiency of removal of lignin and noncellulose components from the plant feedstock in the investigated sequence of thermochemical treatments.

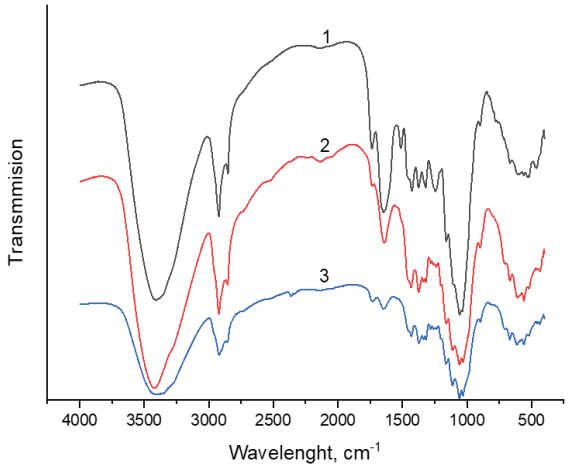


Fig. 2. FTIR Spectra of hemp fibres (1), pulp after alkali treatment (2) and organosolv hemp pulp (3)

As a result of the process of hydrolysis of organosolv hemp pulp with sulfuric acid solutions, a transparent nanocellulose suspension was obtained, which remained stable for several months (Fig. 3).

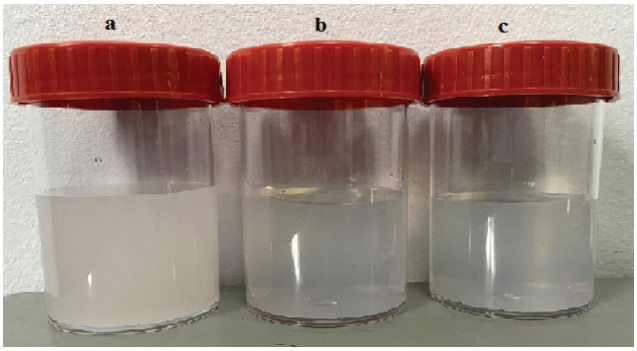
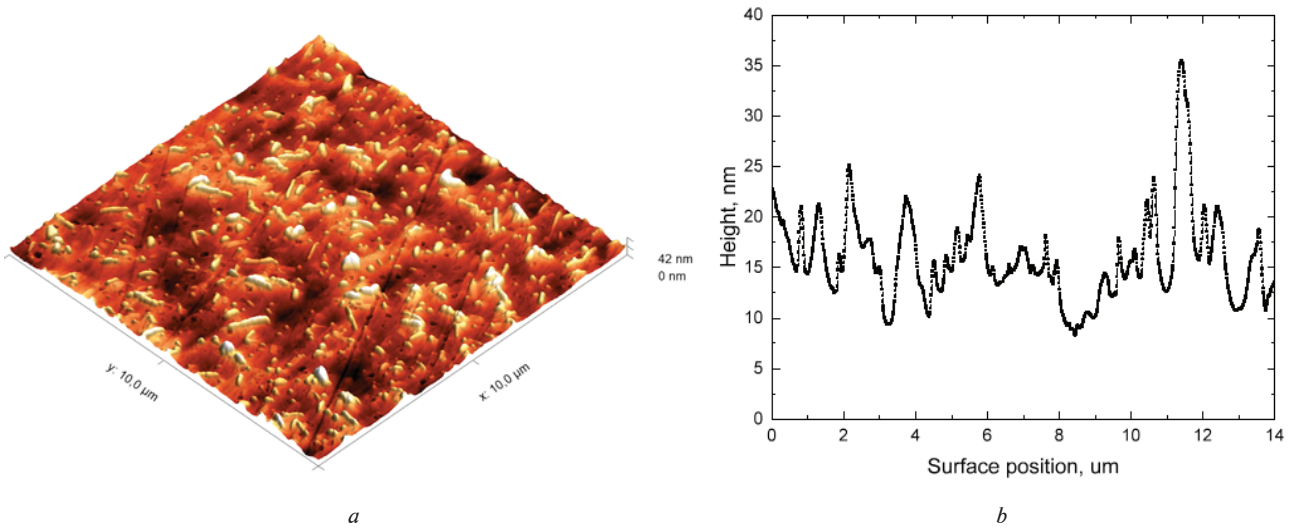


Fig. 3. Photographs of vials with hemp nanocellulose suspensions: *a* without ultrasonic treatment; *b* after ultrasonic treatment and *c* after 3 months of storage

Nanosize of hemp nanocellulose particles confirmed by AFM method in the tapping mode (Fig. 4).

As can be seen from the data in Fig. 4, the cross section of the cellulose nanoparticles ranges from 8 to 36 nm. The following values of the sizes of nanoparticles of hemp nanocellulose are within the sizes of nanocellulose particles obtained by hydrolysis of organosolv pulps from other representatives of non wood plant raw materials, for example: 16–20 nm for wheat straw, 10–28 nm for kenaf, 10–18 nm for miscanthus, 20–60 nm for flax fibres [16]. As shown in Fig. 4 *a*, nanocellulose particles aggregated and interlaced.



a *b*

Fig. 4. AFM image of a nanocellulose film in height (*a*); lateral size of nanoparticles (*b*)

Dependences of physical and mechanical parameters of hemp nanocellulose films on the main technological parameters of the hydrolysis process are shown in Fig. 5 and 6.

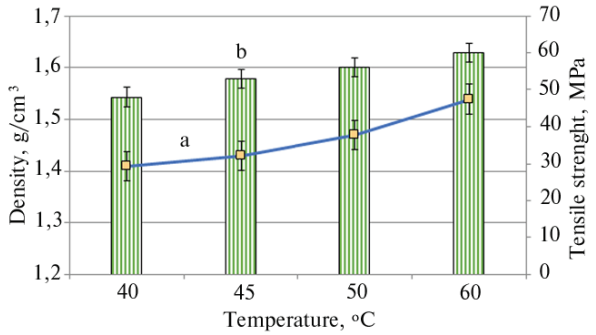


Fig. 5. Dependences of density (*a*) and tensile strength (*b*) of nanocellulose films on the temperature of the process at an acid concentration of 50%

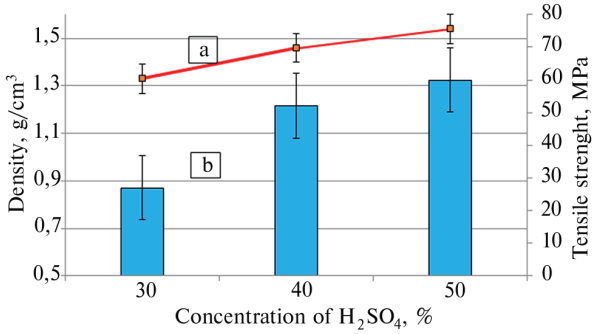


Fig. 6. Dependences of the density (*a*) and tensile strength (*b*) of nanocellulose films on the concentration of H_2SO_4 at a temperature of 60 °C

As can be seen from Fig. 5 and 6 data, increasing the acid concentration and temperature increases the density and tensile strength of nanocellulose films, which can be explained by the formation of stronger bonds between cellulose nanoparticles. The obtained hemp nanocellulose can be used as a reinforcing additive in the production of paper and cardboard [20], as a component of flexible energy storage electronic products, supercapacitors, lithium-ion batteries and solar cell, in the composition of epoxy materials [21], in the production of thermoelectric materials [22], as biodegradable flexible transparent films for ultraviolet and high energy visible light protection [23], as membranes for water purification [24].

Conclusions

1. Thermochemical treatment of hemp fibres by alkaline extraction and organosolv cooking for 180 minutes allows obtaining pulp with a minimum residual content of lignin (0.16%) and minerals (0.08%), suitable for the production of nanocellulose.

2. The process of extracting non-cellulosic components from hemp fibres with a change in the structure of hemp pulp was confirmed by the SEM data. Removal of hemicelluloses and lignin during the thermochemical treatment is confirmed by FTIR measurements.

3. It was shown that an increase in the temperature of hydrolysis and concentration of sulfuric acid increases the density and tensile strength of nanocellulose films due to the formation of stronger bonds between cellulose nanoparticles, which is confirmed by the AFM data.

4. Obtaining result promotes the effective utilization of lignocellulosic fibres as a renewable source of nanocellulose for the production of bionanomaterials for diversified applications.

References

[1] Z. Schlanger, "The problem with turning to paper after the EU's plastic ban", *Quartz*, Mar. 2019. [Online]. Available: https://qz.com/1582520/the-problem-with-turning-to-paper-after-the-eus-plastic-ban.

[2] L. Jasmania, and W. Thielemans, "Preparation of nanocellulose and its potential application", *Int. J. Nanomater. Nanotechnol. Nanomed.*, vol. 4, no. 2, pp. 14–21, Oct. 2018, doi: 10.17352/2455-3492.000026.

[3] H. Shaghaleh, X. Xu, and S. Wang, "Current progress in production of biopolymeric materials based on cellulose, cellulose nanofibers, and cellulose derivatives". *RSC Adv.*, vol. 8, no. 2, pp. 825–842, 2018, doi: 10.1039/C7RA11157F.

[4] A. Naderi and T. Lindström, "Rheological Measurements on Nanofibrillated Cellulose Systems: A Science in Progress" in *Cellulose and cellulose derivatives: synthesis, modification and applications,* M. I. H. Mondal, Ed. NY, USA: Nova Science Publishers, 2015, pp. 187-204.

[5] C. Yadav *et al.*, "Plant-based nanocellulose: A review of routine and recent preparation methods with current progress in its applications as rheology modifier and 3D bioprinting", *Int. J. Biol. Macromol.* , vol. 166, pp. 1586–1616, Jan. 2021, 10.1016/j.ijbiomac.2020.11.038.

[6] M. K. Bakri, M. R. Rahman, and A. Kakar, "Current and Past: Nanocellulose and Nanocomposites and Its Future Applications", *Academia Letters*, Article 1483, Jul.2021, doi: 10.20935/AL1483.

[7] M. Alavi, "Modifications of MCC, NFC, and NCC for antimicrobial and wound healing applications", *e-Polymers*, vol. 19, no.1, pp. 103–119, May 2019, doi: 10.1515/epoly-2019-0013.

[8] S. Naz, J. S. Ali, and M. Zia, "Nanocellulose isolation characterization and applications: a journey from non-remedial to biomedical claims", *Bio-Design and Manufacturing,* vol. 2, no. 3, pp. 187-212, Aug. 2019. doi: 10.1007/s42242-019-00049-4.

[9] S. Beck-Candanedo, M. Roman, and D. G. Gray, "Effect of reaction conditions on the properties and behavior of wood cellulose nanocrystal suspensions", *Biomacromolecules*, vol. 6, no. 2, pp.1048–1054, 2005, doi: 10.1021/bm049300p.

[10] Y. Habibi, "Key advances in the chemical modification of nanocelluloses", *Chem. Soc. Rev.*, vol. 43, no. 5, pp.1519–1542, 2014, doi: 10.1039/C3CS60204D.

[11] F. T. Seta *et al.*, "Preparation and characterization of high yield cellulose nanocrystals (CNC) derived from ball mill pretreatment and maleic acid hydrolysis", *Carbohydrate Polymers,* no. 234, pp. 115942–11612, Apr. 2020, doi: 10.1016/j.carbpol.2020.115942.

[12] I. Deykun, V. Halysh, and V. Barbash, "Rapeseed straw as an alternative for pulping and papermaking", *Cellulose Chem. Technol.*, vol. 52, no. 9–10, pp. 833–839, 2018.

[13] A. Ab. Latif, S. Harun, M. S. Sajab, M. Markom, and J. Md Jahim , "Ammonia-based pretreatment for ligno-cellulosic biomass conversion – an overview", *J. Eng. Sci. Technol. Rev.*, vol. 13, no. 6, pp. 1595–1620, 2018.

[14] Q. Lyu, X. Chen, Y. Zhang, H. Yu, L. Han, and Weihua Xiao, "One-pot fractionation of corn stover with peracetic acid and maleic acid", *Bioresource Technology,* vol. 320, pp. 124306–124313, 2021, doi: 10.1016/j.biortech.2020.124306.

[15] TAPPI Test Methods, Tappi Press, Atlanta, US, 2004.

[16] V. Barbash, and O. Yashchenko, "Preparation, properties and use of nanocellulose from non-wood plant materials", in *Novel Nanomaterials*", K. Krishnamoorthy, Ed. IntechOpen, pp. 1–23, 2020, doi: 10.5772/intechopen.94272.

[17] G. A. Smook, "Handbook for Pulp & Paper Technologists". 3rd ed., Angus Wilde Publications, 425 p, 2003.

[18] N. A. Rosli, W. H. W. Ishak, and I. Ahmad, "Eco-friendly high-density polyethylene/amorphous cellulose composites: Environmental and functional value", *J. Clean. Prod.*, vol. 290, pp. 125886–12594, Mar. 2021, doi: 10.1016/j.jclepro.2021.125886.

[19] R. A. Ilyas, S. M. Sapuan, M. R. Ishak, and E. S. Zainudin, "Effect of delignification on the physical, thermal, chemical, and structural properties of sugar palm fibre", *BioResources*, vol. 12, no. 4, pp. 8734–8754, 2017.

[20] V. A. Barbash, O. V. Yashchenko, A. S. Gondovska, O. S. Yakymenko, "Nanocellulose from reed stalks to improve the properties of paper for packaging food products", *KPI Sci. News*, no. 2, pp. 90-96, 2021, doi: 10.20535/kpisn.2021.2.236944.

[21] D. Starokadomsky et al., "Resistance and strength of bio-compatible epoxy-cellulose composites, as a function of concentration and dispersity of cellulose filler", *Biomed J. Sci & Tech Res*, vol. 31, no. 3, pp. 24211–24218, Oct. 2020, doi: 10.26717/BJSTR.2020.31.005103.

[22] N. P. Klochko *et al.*, "Efficient biodegradable flexible hydrophobic thermoelectric material based on biomass-derived nanocellulose film and copper iodide thin nanostructured layer", *Solar Energy*, vol. 212, pp. 231–240, Dec. 2020, doi: 10.1016/j.solener.2020.10.081.

[23] N. P. Klochko *et al.*, "Biodegradable flexible transparent films with copper iodide and biomass-derived nanocellulose for ultraviolet and high energy visible light protection", *Solar Energy*, vol. 220, pp. 852–863, May 2021, doi: 10.1016/j.solener.2021.04.014.[24] S. P. Espнndola, M. Pronk, J. Zlopasa, S. J. Picken, and M. C, M. Loosdrecht., "Nanocellulose recovery from domestic wastewater, *J. Clean. Prod.*, vol. 280, p. 124507, Jan. 2021, doi: 10.1016/j.jclepro.2020.124507.

В.А. Барбаш, О.В. Ященко, О.С. Якименко, Р.М. Захарко

ЕКСТРАКЦІЯ ОРГАНОСОЛЬВЕНТНОЇ ЦЕЛЮЛОЗИ ТА ОДЕРЖАННЯ НАНОЦЕЛЮЛОЗИ З ВОЛОКОН КОНОПЕЛЬ

Проблематика. Застосування замість синтетичних полімерів целюлози та наноцелюлози дає можливість покращити споживчі властивості та екологічність композитних матеріалів. Тому розробка технологій отримання з рослинної сировини, зокрема волокон конопель, органосольвентної целюлози та наноцелюлози є актуальною науково-практичною проблемою.

Мета дослідження. Отримати целюлозу з волокон конопель пероцтовим методом та вивчити вплив концентрації сульфатної кислоти та температури гідролізу органосольвентної целюлози на показники якості конопляної наноцелюлози.

Методика реалізації. Обробку волокон конопель виконували в дві стадії: лужна екстракція й органосольвентне варіння за температури 97 ± 2 °С. Наноцелюлозу отримували шляхом гідролізу органосольвентної целюлози розчином сульфатної кислоти різної концентрації. Отриману наноцелюлозу досліджували методами скануючої електронної мікроскопії (СЕМ), інфрачервоної спектроскопії з Фур'є перетворенням (FTIR), атомною силовою мікроскопією (AFM).

Результати дослідження. Отримано органосольвентну конопляну целюлозу із залишковим вмістом лігніну 0,16 % і зольністю 0,08 %. Одержані з неї частинки наноцелюлози мали поперечний розмір 8–23 нанометрів і довжину до декількох мікрометрів. Наноцеллюлозні плівки мали високі механічні показники: щільність до 1,54 г/см^3 і міцність на розрив до 60 МПа.

Висновки. Проведення термохімічної обробки волокон конопель у два етапи дає можливість отримати целюлозу з мінімальним залишковим вмістом лігніну та мінералів, яка придатна для виробництва наноцелюлози. Наноцелюлоза була успішно виділена з органосольвентної конопляної целюлози за допомогою процесу кислотного гідролізу. Подальша ультразвукова обробка дає можливість отримати стабільний наноцелюлозний гель з високими механічними властивостями.

Ключові слова: волокно конопель; органосольвентна целюлоза, наноцелюлоза, кислотний гідроліз

В.А. Барбаш, О.В. Ященко, О.С. Якименко, Р.М. Захарко

ЭКСТРАКЦИЯ ОРГАНОСОЛЬВЕНТНОЙ ЦЕЛЛЮЛОЗЫ И ПОЛУЧЕНИЕ НАНОЦЕЛЮЛОЗЫ ИЗ ВОЛОКОН КОНОПЕЛЬ

Проблематика. Применение вместо синтетических полимеров целлюлозы и наноцеллюлозы дает возможность улучшить потребительские свойства и экологичность композитных материалов. Поэтому разработка технологий получения из растительного сырья, в частности из волокон конопли, органосольвентной целлюлозы и наноцелюлозы является актуальной научно-практической проблемой.

Цель исследования. Получить целлюлозу из волокон конопли перуксусным методом и изучить влияние концентрации серной кислоты и температуры гидролиза органосольвентной целлюлозы на показатели качества конопляной наноцеллюлозы.

Методика реализации. Обработку волокон конопли производили в две стадии: щелочная экстракция и органосольвентная варка при температуре 97 ± 2 °С. Наноцеллюлозу получали путем гидролиза раствором серной различной концентрации. Полученную наноцеллюлозу исследовали методами сканирующей электронной микроскопии (СЭМ), инфракрасной спектроскопии с Фурье-превращением (FTIR), атомной силовой микроскопией (AFM).

Результаты исследования. Получена органосольвентная конопляная целлюлоза с остаточным содержанием лигнина 0,16 % и зольностью 0,08 %. Образовавшиеся в процессе гидролиза частицы наноцеллюлозы имели поперечный размер 8–23 нанометров и длину до нескольких микрометров. Наноцеллюлозные пленки обладали высокими механическими свойствами: плотностью до 1,54 г/см^3 и прочностью на разрыв до 60 МПа.

Выводы. Проведение термохимической обработки волокон конопли в два этапа позволяет получить целлюлозу с минимальным остаточным содержанием лигнина и минералов, которая пригодна для производства наноцеллюлозы. Наноцеллюлоза была успешно выделена из органосольвентной конопляной целлюлозы посредством процесса кислотного гидролиза. Последующая ультразвуковая обработка позволяет получить стабильный наноцеллюлозный гель с высокими механическими свойствами.

Ключевые слова: волокно конопли; органосольвентная целлюлоза; наноцеллюлоза; кислотный гидролиз.

Рекомендована Радою
хіміко-технологічного факультету
КПІ ім. Ігоря Сікорського

Надійшла до редакції
28 жовтня 2021 року

Прийнята до публікації
7 грудня 2021 року