Industrial Hemp Hurd Processing for Microcrystalline Cellulose Production and its Usage as a Filler in Paper

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This article substantiates the possibilities of replacing commercial wood with raw materials made from industrial hemp hurd (hemp-woody core) for the production of unbleached and bleached paper pulps. A comparative analysis of the mechanical characteristics of sheets of paper prepared in the Rapid-Köthen apparatus and obtained from pulp obtained from commercial wood and hemp-woody core (HWC) was undertaken. The objective of this study was to determine the effect of mechanical refining of a pulp on the production of microcrystalline cellulose (MCC) from HWC. It was shown that an increase in the pulp refining degree from 15 °SR to 83 °SR led to a decrease in the degree of polymerisation of MCC from 272 to 75, the hydrochloric acid concentration from 73 to 45.63 g/L, and the hydrolysis time from 120 min to 60 min. With the addition of 5% MCC obtained from hemp-woody core, the mechanical properties of laboratory paper sheets from HWC were improved until they met ISO 12625-4-2017 (2017) requirements for NS-2. The results obtained support using hemp-woody core for the production of MCC.

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Keywords: Hemp; Microcrystalline cellulose; Refining; Refining degree; Hydrolysis

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INTRODUCTION

The pulp and paper production process aims to obtain pulp, paper, cardboard, and other related end or intermediate products. In addition to the use of pulp for the production of cardboard and paper products, the last decade has seen an increasing interest in modified cellulose products. In most cases, modifying cellulose makes it possible to obtain materials with improved or specified properties depending on the modification method. One such modified cellulose product is powdered cellulose materials (PCM). Their unique properties help determine relevant application areas and include the following: lack of taste, ability to gel in water, water retention (typical of microcrystalline cellulose gels), large specific surface area, high sorption properties, high volume, and bulk density. The characteristics of powdered cellulose materials vary and depend on the initial cellulose materials and on production methods and conditions (Prosvirnikov 2014; Toptunov and Sevastyanova 2021). The types of powdered cellulose materials available are as follows:

- Powdered cellulose (PC) is produced by the destruction of fibrous raw materials, contains at least 92% cellulose and has properties that are somewhat different from those of fibrous cellulose;

- Microcrystalline cellulose (MCC) is a finely dispersed product, crystallites of cellulose microfibrils (without the amorphous part), modified natural (fibrous) cellulose powder;
- Microfibrillar cellulose (MFC) is a fibrillated material that contains various structural components (cellulose fibres and fragments, fibrils, and nanofibrils);
- Nanocrystalline cellulose (NCC) is a homogeneous, redispersible natural nanoparticle obtained from the crystalline regions of cellulose fibres;
- Nanofibrillar cellulose (NFC) is fibrillated cellulose material subjected to ultrafine refining.

The main characteristics of PCMs include the processing method, degree of polymerisation (DP), and particle size. The PC is obtained by mechanical processing, the DP is 1,000 and higher, and the particle size is more than 5 µm. The MCC is obtained by the chemical method (usually acid hydrolysis), the DP is less than 400, and the particle size is from 5 to 400 µm. MFC is obtained by mechanical processing, DP is from 100 to 400, and the particle size is 100 µm in length and about 1 µm in width. The NCC is obtained mainly by chemical treatment (usually more intense acid hydrolysis), the DP is less than 100, and the particle size is from 100 nm to 1 µm in length and about 2 to 50 nm in diameter. NFC is obtained by optional preliminary chemical and subsequent mechanical treatment, the DP is less than 100, and the particle size is up to several µm in length and 1 to 100 nm in width.

Analysis of literary sources has shown that no comparative evaluation of the mechanical properties of laboratory paper sheets obtained from commercial wood and the MCC prepared from hemp-woody core (HWC) has yet been made. In recent times, researchers have become interested in MCC-containing products. Microcrystalline cellulose is a finely-dispersed product of the hydrolytic destruction of cellulose. Its inclusion in the composition of materials opens up new opportunities for obtaining finished products with qualitatively and quantitatively new characteristics. MCC is traditionally obtained by treating cellulose with concentrated acids. In this case, the fibrous structure is destroyed and cellulose powder is generated.

However, enterprises that produce MCC face a number of environmental and economic problems:

- In most cases, commercial wood is used as a raw material;
- MCC production uses concentrated acids that later require proper disposal.

One method of solving these problems in powdered cellulose production is to replace commercial wood with alternative raw materials, for example, with hemp-woody core (Silva 2019; Manian and Cordin 2021; Tutek and Masek 2022; Danielewicz *et al.* 2021; De Groot *et al.* 1995; Tutuş *et al.* 2016).

The difficulty in obtaining MCC from plant raw materials is that, in addition to cellulose, it contains such compounds as lignin, hemicelluloses, various extractive substances, and a small percentage of minerals. Regardless of the industry where MCC is used, it is characterised by a set of parameters that define the type of powdered cellulose material and determine the reaction of finished products to various influences (Battista 1964; Yakobsons *et al.* 1993; Kai *et al.* 2000; Tikhomirov and Bulanov 2000; Kuznetsov *et al.* 2001; Laka and Chernyavskaya 2007; Aleshina *et al.* 2014). A number of works on MCC production pay close attention to the chemical effect of acid on the pulp. Analysis of these works shows that when inorganic acids are used for MCC production, it is not always possible to minimise the acid concentration or choose the optimal process mode (Barbash *et al.* 2016; Barbash *et al.* 2018). Pulp pre-processing by mechanical refining (bars or bars-free method) can reduce the harmful effects of inorganic acids on the environment and decrease energy costs for MCC production.

The ability of refining equipment to separate various pulp into fibres, refine them, and develop certain properties can be used to obtain not only long-fibre fractions, but also MCC (Ivanov 1960; Pashinsky 1972; Kovalev 2007; Ivanov *et al.* 2017). The laboratory of the Department of Machines and Devices of Industrial Technologies of the Reshetnev Siberian State University of Science and Technology is conducting studies on the effect of the mechanical refining method of a fibrous suspension extracted from hemp-woody core on the production of paper and MCC (Kutovaya 1998; Alashkevich *et al.* 2020; Kaplev *et al.* 2022). The relevance of this study lies in the consideration of a source of raw materials alternative to commercial wood for the production of MCC to be used as a filler in paper. The subject of the study is the production of MCC from HWC.

The objective of this work was to determine the effect of the refining of a fibrous suspension on the production of MCC from hemp-woody core. At this stage, the study objectives included: obtaining unbleached pulp from industrial hemp shives; refining a fibrous suspension in a disc refiner; comparative analysis of the physical and mechanical characteristics of pulp obtained from HWC and commercial wood; chemical pulp processing; comparative analysis of the degree of polymerisation of cellulose macromolecules made of hemp-woody core and commercial wood at different refining degrees according to the Schopper-Riegler method.

EXPERIMENTAL

Samples of sulphate softwood and hardwood pulp (a semi-finished product by OAO Ilim Group, Bratsk, Russia) and hemp-woody core (*Cannabis sativa*) were of the Surskaya trademark.

The following laboratory methods were used to monitor the refining of pulp obtained from HWC: measurements of the Schopper-Riegler freeness were carried out in accordance with ISO 5267-1 (1999); mass fraction of alpha-cellulose determined according to GOST 6840-78 (1978); determining the lignin content according to GOST 11960-79 (1979); laboratory paper sheets produced in accordance with ISO 5269-1 (2005); determination of physical and mechanical characteristics, including breaking length, burst strength in accordance with ISO 2758 (2014), ISO 1974 (2012), and ISO 1924-2 (2008); determining the degree of polymerisation according to GOST 9105-74 (1974), determining brightness with ISO 2470-2 (2008).

After manual chopping, chips of 5 cm hemp-woody core samples were subjected to sulfate pulping in stainless steel ampoules with a capacity of 400 cm^3. The ampoules were placed in a glycerin thermostat preheated to the required temperature (a glycerin bath with electric heating and an agitator). Cellulose was obtained from HWC (Fig. 1) using a pulping solution of mainly sodium hydroxide and sodium sulfide (NaOH and Na_2S). Pulping was performed in a laboratory autoclave at a temperature of 165 °C for 3 h. At the end of pulping, the pulp was washed in a sieve. The solid residue on a sieve with a diameter of 3 mm round holes was considered as undefibered uncooked parts. The screened unbleached pulp yield was 38%.

After pulping, washing, and sorting, the pulp had a brown colour due to residual lignin, colouring matter, and adsorbed black liquor. Therefore, it was bleached with sodium hypochlorite to whiten it and remove the residual lignin. The chlorine consumption was determined depending on the residual lignin in the pulp after pulping and its moisture content (Byvshev *et al.* 2010). To reduce pulp degradation during bleaching, the pulp temperature was maintained at no higher than 40 °C for 60 min.

Fig. 1. Hemp-woody core (hurd)

One problem in obtaining fine cellulose materials by chemical means is the recycling of the used acid solutions after the hydrolysis. To reduce the harmful effects of inorganic acids on the environment and energy consumption in the production of MCC, pulp extracted from HWC was refined. The simultaneous shortening and swelling of fibres and their longitudinal splitting into fibrils will, upon further processing, contribute to an increase in the penetrating ability of the acid into the fibre structure during hydrolysis.

The 1% fibrous suspension was refined in a semi-industrial disc refiner of between 15 and 83 °SR (Fig. 2). Based on studies previously conducted at the Department, a rotor speed of 2,000 rpm and inter-cutter gap of 0.1 mm were chosen as the most effective refining duration and fibre quality, respectively (Alashkevich 1980; Nabieva 2004). Figure 3 shows a diagram of an experimental (semi-industrial) cutter-type refining machine and Table 1 shows its specifications.

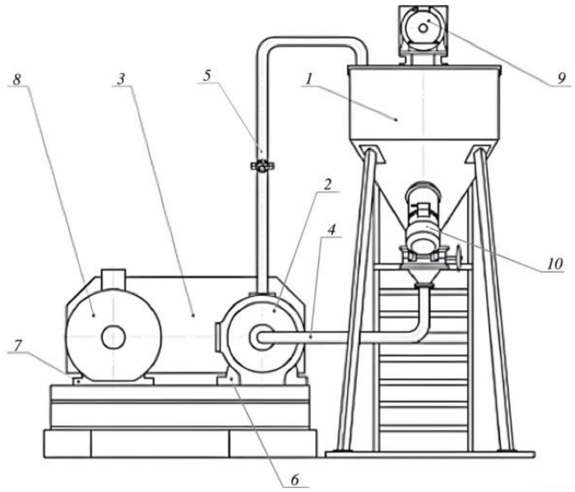
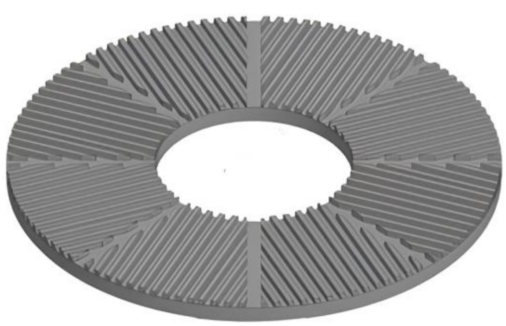


Fig. 2. Disc refiner: 1 – hydropulper; 2 – disc refiner; 3 – belt drive; 4 – discharge pipe; 5 – circulation pipe; 6 – frame; 7 – fastening; 8 – electric motor

Table 1. Specifications of the Cutter Refiner

Overall dimensions of disc refiner (length × width × height) (m)	0.95 × 0.6 × 0.8
Inter-cutter gap (mm)	0 – 6
Rotor shaft frequency (rpm)	0 – 2000
Cutter tool material	Steel 40XH
Rated engine power (kW)	22
Engine output shaft frequency (rpm)	750

The fibrous suspension was refined using a traditional eight-section tool with straight bars and an angle of 22.5° (Fig. 3), because the vast majority of refiner plate tools are designed to meet such conditions. It provides an optimal fibrillation-to-cutting ratio while reducing the energy intensity of the refining (Legotsky and Goncharov 1990; Alashkevich *et al.* 2008), which, as we expect, will lead to a decrease in the concentration of the acid used during the hydrolysis, pulp hydrolytic degradation time, and quantitative parameters of the degree of polymerisation of cellulose macromolecules during hydrolysis.



- outer and inner diameters: D = 290 mm, d = 130 mm;
- bar thickness and height: δ = 4 mm;
- h = 4 mm;
- groove width, b = 4 mm;
- second bar length, L_s =25213 m/s;
- contact area of the rotor and stator bars, F = 0.0076 m^2;
- ratio of the refiner bar surface area to the total tool surface area, F_{size}/F_{total}= 22.5%

Fig. 3. Diagram and characteristics of the bars on the refiner plate

Laboratory paper sheets of 75 g/m^2 were made on a Rapid-Köthen handsheet-forming machine, and strength properties were measured using standard methods.

After refining, the pulp was subjected to hydrolysis for enhanced destruction of the cellulose structure and to obtain microcrystalline cellulose from it through hydrolytic degradation. The quality and yield of MCC depend on the type of feedstock and the conditions of its hydrolysis – the nature and concentration of acid, temperature, and duration of the process – which was performed in a reactor.

The regulated parameters of the hydrolysis process are the refining degree according to the Schopper-Riegler method, the temperature, and the acid concentration. Because the acid concentration depends on the reaction temperature, a higher heating temperature results in a less concentrated acid solution used. In this regard, the authors studied three factors of the cellulose hydrolysis process: refining degree according to the Schopper-Riegler method (varied from 15 to 83 °SR), hydrolysis temperature (varied from 80 to 100 °C), and hydrochloric acid concentration (varied from 45.63 to 73 g/L). The hydrolysis ratio remained constant (15:1). Optimal hydrolysis conditions were determined by planning a multifactorial model experiment with normalised factor designations developed in the Statgraphics software product (Table 2). The design input parameters were acid concentration x_1, temperature x_2, duration of hydrolysis x_3, and a refining degree according to the Schopper-Riegler method x_4. The constant conditions of the experiment were hydrolysis modulus (15:1), washing, drying, refining, and screening conditions. Output parameters were the product degree of polymerisation y_1, degree of crystallinity y_2 of MCC. After hydrolysis, the resulting MCC was washed, dried, and dispersed.

Table 2. Parameters of a Full-Factor Experiment

Parameter	Designation	
	natural	normalized
Input Parameters (controlled factors)		
Acid concentration (g/L)	c	X_1
Temperature (°C)	t	X_2
Duration of hydrolysis (min)	τ	X_3
The degree of refining (°SR)	°SR	X_4
Output Pameters (controlled factors)		
Degree of polymerization	DP	Y_1
Degree of crystallinity	DC	Y_2

The dry matter content, degree of polymerization, and degree of crystallinity were determined in the obtained MCC samples, and the microstructure dimensions were determined by microscopy. Diffraction patterns (Fig. 8) were obtained using an X-ray diffractometer DRON-3 (Burevestnik Innovation Center JSC, Saint-Petersburg, Russia), with $CuK_α$ radiation, scanning 1 °/min, and scanning step 0.02°. The cellulose crystallinity index was calculated as the ratio of the sum of the peak areas of crystalline cellulose to the total area of all peaks in accordance with the methodology described in Park *et al.* (2010).

The sizes of the obtained MCC samples were measured using a high–resolution scanning microscope Hitachi SU3500 (accelerating voltage - 0.3 to 30 kV, detector - secondary and backscattered electrons). The degree of polymerization of MCC was determined in the ferruginous sodium complex in accordance with GOST 9105-74 (1974) on a capillary viscometer of the VPJ-3 type according to the method described in the work by Obolenskaya *et al.* (1985).

To study the possibility of using MCC samples as a reinforcing additive to paper, laboratory paper sheets were made with the addition of the obtained MCC samples. Paper samples were obtained from pulp extracted from HWC according to GOST 14363.4–89 (1989). The weight of the laboratory paper sheets was 2.38 to 2.40 g/m^2. The concentration of the suspension of MCC preparations added in the manufacture of paper was 5% abs. dry matter.

To assess the effectiveness of the addition of MCC to paper, the strength characteristics were measured and compared with a control sample (laboratory paper sheets without the addition of MCC).

Pulp was extracted from hemp-woody core (Fig. 4, a, b) using a pulping solution of mainly sodium hydroxide and sodium sulfide (NaOH and Na_2S). Pulping was carried out in a laboratory autoclave at a temperature of 165 °C for 3 h, the degree of sulfidity of the pulping solution 18%; hydraulic module 4.83. The cellulose yield was 38%.

(a) (b)

Fig. 4. Processing hemp-woody core: (a) unbleached pulp extracted from hemp-woody core; (b) bleached pulp extracted from hemp-woody core

RESULTS AND DISCUSSION

As a result of pulping and bleaching, before refining in the disc refiner, the pulp extracted from hemp-woody core had the following parameters: mass fraction of alpha-cellulose – 91.5%, mass fraction of lignin – 2.14%, degree of polymerisation – 580, and brightness – 87.1% (2008).

The main purpose of refining is to develop a set of properties in natural fibres, such as flexibility, elasticity, and strength. Therefore, after refining, the laboratory paper sheets were prepared from the fibrous suspension to further determine the fibre quality. Figure 5 shows the dependence of the effect of the refining duration on the increase in the refining degree on the Schopper-Riegler scale.

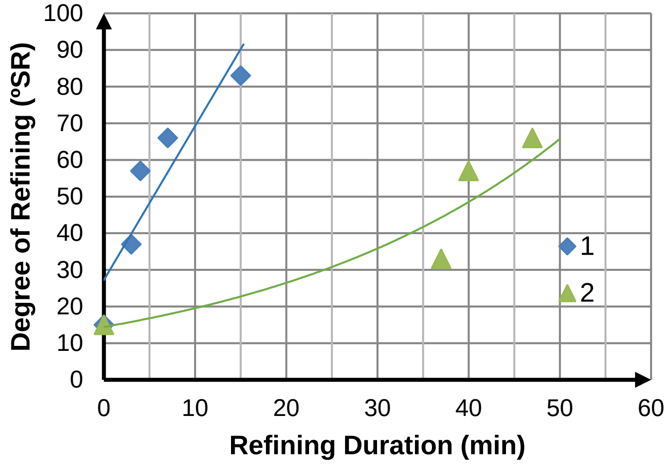


Fig. 5. Dependence of the degree of refining on the duration of refining: (1) cellulose from hemp-woody core; (2) coniferous cellulose

As shown in Fig. 5, the most intensive refining occurs in the fibrous suspension from hemp-woody core, which is explained by the difference in the structure of softwood and hardwood fibres and that of hemp-woody core (Table 3).

Table 3. Chemical Composition

Degree of Refining (°SR)	Alpha Cellulose (%)	Lignin (%)	Ash Content (%)
Coniferous cellulose			
15	87.3	4.3	0.3
38	87.3	3.9	0.2
55	87.9	3.8	0.1
78	88.2	3.6	0.1
Cellulose from hemp-woody core			
15	83.4	2.2	0.1
28	83.6	1.9	0.07
57	83.9	1.3	0.06
83	84.5	1.1	0.05

As shown in the experimental data presented in Figs. 6 and 7, after refining to higher Schopper-Riegler freeness of pulp extracted from softwood and hemp-woody core, the qualitative dependences of the studied properties (breaking length and burst strength) were identical. Meanwhile, the quantitative characteristics increased 27% for the breaking length of the laboratory paper sheets from softwood cellulose, 58% for that of the laboratory paper sheets from hemp-woody core, and 11% and 73%, respectively, for the burst strength. This is because the refining process in disc refiner is characterised by two factors: hydrodynamic and mechanical. These factors affect the fibres when the fibrous suspension passes through the working inter-bar space formed in the interface plane of the tool of the stationary stator with that of the rotating rotor.

Mineral fillers differ from vegetable fibres mainly in their greater fragility and length, as well as inability to hydrate and fibrillate during refining. For this reason, it is recommended to use special binding components to increase the strength of composites during their production. In this regard, products that use modified cellulose are now increasingly being produced (Yurtayeva and Vasilieva 2020; Vasilyeva *et al*. 2021). This is because, when included in the composition, this type of cellulose helps obtain products with improved or specified properties. Additionally, using modified cellulose in the composition of special types of paper and cardboard will open up new opportunities in the pulp and paper and other industries (Dubovy 2006; Friedman and Sorokina 2014).

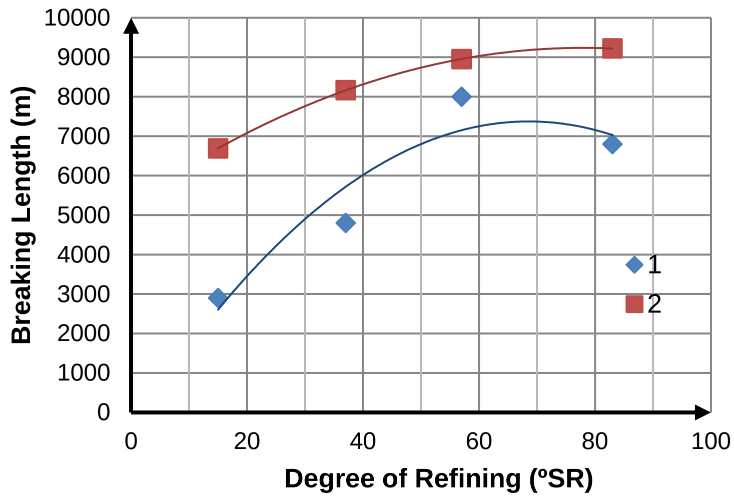


Fig. 6. Dependence of the breaking length on the degree of refining: (1) cellulose from hemp-woody core; (2) coniferous cellulose

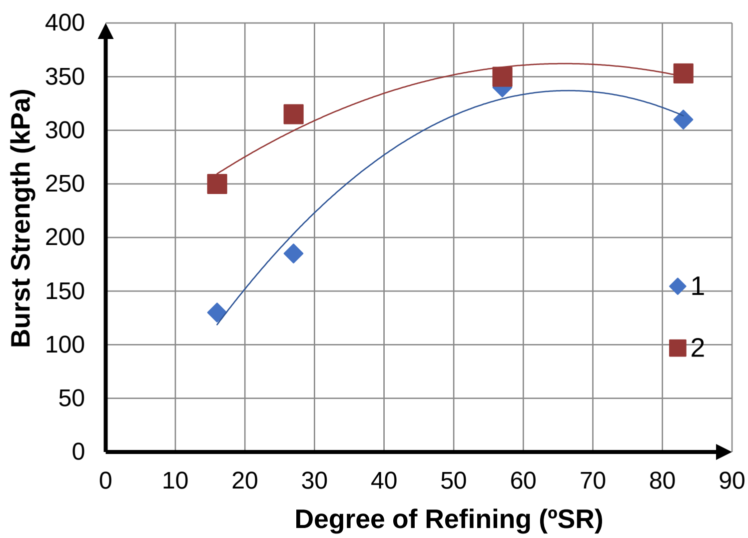


Fig. 7. Dependence the burst strength on the degree of refining: (1) cellulose from hemp-woody core; (2) coniferous cellulose

The most effective conditions for the chemical treatment of annual plants and the characteristics of the obtained MCC samples from hemp-woody core and wheat straw in comparison with Avicel standard FAO UN MCC 460 i are shown in Table 3.

It is established (Table 4) that the indicators of the obtained MCC samples correspond to those for Avicel microcrystalline cellulose according to the FAO UN MCC 460 i standard. Hydrolysis of cellulose, which was pre-refining in an aqueous medium, significantly reduced the "rigidity" of the hydrolytic degradation process.

The decrease in the degree of polymerisation of microcrystalline cellulose with an increase in the pulp refining degree is explained by the fact that the refining of a fibrous suspension not only increases the outer surface of the fibres and the number of free hydroxyl groups on their surface, but also destroys intermolecular bonds inside the fibre cell wall, thereby causing microcracks. All this leads to an increase in the rate of acid reaction with the fibrous suspension and a significant decrease in the degree of polymerisation of microcrystalline cellulose (Alashkevich *et al.* 2023).

Table 4. Values of MCC Output Parameters

Technological Parameters of Hydrolysis	Degree of Polymerization	Degree of Crystallinity	Yield (%)	Brightness (%)	Bulk density (g/mL)
73 g/L; 15 °SR; 120 min; 100 °C	272	0.63	96.4	84.0	0.244
73 g/L; 28 °SR; 110 min; 95 °C	190	0.64	95.9	85.0	0.275
58.4 g/L; 57 °SR; 100 min; 90 °C	158	0.67	95.6	86.0	0.305
54.75 g/L; 67 °SR; 80 min; 85 °C	120	0.70	95.4	88.0	0.308
45.63 g/L; 83 °SR; 60 min; 85 °C	75	0.71	95.1	86.0	0.315
Avicel standard FAO UN MCC 460 i	265	0.63–0.82	–	90.0	0.280-0.360

The analysis of the X-ray diffractograms of the MCC showed that with an increase in the degree of refining, the degree of crystallinity of cellulose increased (Fig. 8).

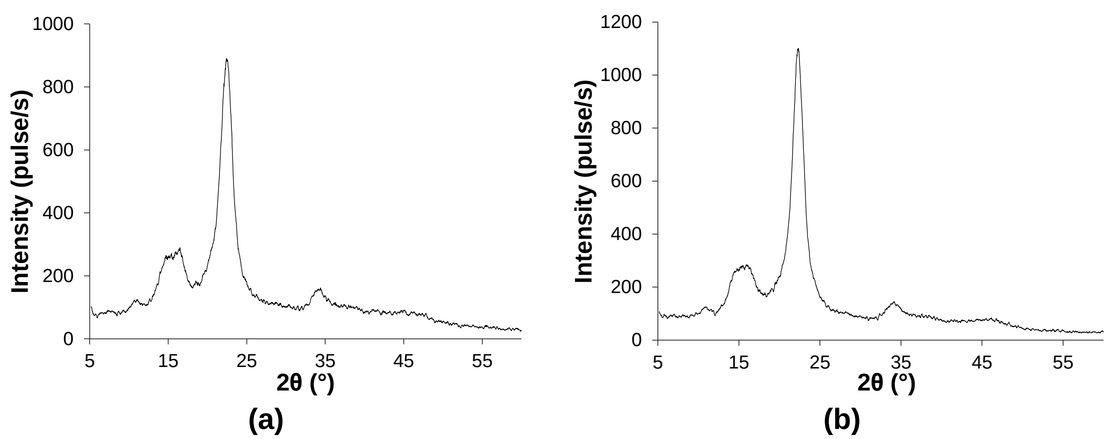


Fig. 8. Diffractogram of microcrystalline cellulose: (a) at 15 °SR; (b) at 83 °SR

Table 5 shows the experimental results of the individual mechanical properties of finished laboratory paper sheets from the refining degree on the Schopper-Riegler scale. The results were obtained from hemp-woody core with and without MCC (at the 5% level) after refining in a disc refiner.

Table 5. Physical and Mechanical Parameters Laboratory Paper Sheets from Hemp-woody Core

Degree of Refining on the Shopper-Rigler Scale	Breaking Length (m)		Burst Strength (kPa)		Tear Resistance (mN)	
	pulp	pulp with MCC	pulp	pulp with MCC	pulp	pulp with MCC
15	2800	3300	121.2	131.0	184	201
38	4900	5050	180.1	197.7	379	402
57	8000	8400	385.4	402.0	642	660
65	7300	7652	310.5	327.4	651	674
NS-3 (60)	7800		-		640	
NS-2 (60)	8200		-		770	

Note: MCC is an additive of microcrystalline cellulose
NS-2, NS-3 are ISO 12625-4-2017 (2017) parameters with a refining degree of 60 °SR

As can be seen from the table, with an increase in the pulp refining degree, the mechanical properties of laboratory paper sheets from hemp-woody core at a refining degree of 57 ºSR on the Schopper-Riegler scale complied with ISO 12625-4-2017 (2017) for NS-3 (for moisture-resistant paper, base paper for the inner layers of decorative laminated plastic, cardboard for end caps of filter elements). When adding 5% MCC, they corresponded to the indicators of ISO 12625-4-2017 (2017) for HC-2 (for bag paper, light-proof paper, paper for textile cartridges and cones, adhesive tape base, for smooth layers of cardboard, cardboard box, waterproof, upholstery, cushioning, shoe and other types of paper and cardboard).

CONCLUSIONS

1. Hemp-woody core (hemp hurd) was successfully used as the source of cellulose for the production of microcrystalline cellulose (MCC). A sequence of alkaline pulping, bleaching, mechanical refining, and acid hydrolysis were employed in the conversion of the hemp-woody core to MCC.
2. When MCC from hemp-woody core was included in a fibrous suspension obtained from hemp-woody core, a semi-finished product pulp obtained from hemp-woody core could be produced. The properties were judged favorable for the production of sack and lightproof paper, paper for textile cartridges and cones, bases for adhesive tapes, for smooth cardboard layers, box, waterproof, panel, packing, leather cardboards, and other types of paper and cardboard.
3. Refining the fibrous mass of hemp-woody core in an aqueous medium before the hydrolysis process increased its reactivity by 35%, which subsequently sped up the process of preparing MCC.
4. The use of cellulose pretreated on a disc refiner can make it possible to reduce the concentration of acid during chemical treatment by 1.6 times.

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