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Preparation of Microcrystalline Cellulose Directly from Wood under Microwave Radiation

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Abstract—The influence of the composition of solutions of peracetic acid on a change in the characteristics of cellulose during the oxidative-hydrolytic destruction of aspen and pine wood under microwave radiation was investigated. Samples of the prepared cellulose were characterized by viscometry, X-ray diffraction and IR spectroscopy. It was found that the main factor determining the rate of destruction of cellulose to a low degree of polymerization values in an acid medium at the initial concentration of peracetic acid not exceeding 15% and initial concentration of hydrogen peroxide no more than 3 mol/dm^3 is the concentration of sulfuric acid. The disordering effect of microwave radiation on the crystallites of cellulose during preparation from pine wood in the presence of sulfuric acid in a concentration of 0.06 mol/dm^3 and increased duration of oxidative-hydrolytic treatment from 1 to 2 h was revealed. It was shown that the characteristics of the products obtained from aspen and pine wood by oxidative-hydrolytic treatment with 15% peracetic acid under microwave radiation, the duration of the process of 1.5–2 h and the sulfuric acid concentration of 0.3–0.7 mol/dm^3 correspond to partially oxidized microcrystalline cellulose.

Keywords: aspen wood, pine wood, peracetic acid, oxidative-hydrolytic destruction, microwave radiation, microcrystalline cellulose

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INTRODUCTION

Microcrystalline cellulose (MCC) is a material consisting of the ordered elements of the supramolecular structure of cellulose remaining after its chemical degradation to the "limiting" degree of polymerization. For many years, it has been widely and successfully used in the pharmaceutical, food, cosmetic, and other industries [1]. Conventional technology for the production of high-quality MCC is based on the use of expensive raw materials—purified cotton and wood cellulose [2, 3]. Therefore, one of the main objectives of research in the field of development of methods of MCC production is to search for conditions of chemical destruction of cheap raw materials providing the effective separation of the crystalline part of the cellulose from associated components.

As a result of numerous studies conducted by scientific teams from different countries bacterial cellulose [4, 5], tunitsin tunicates [6], cellulose for the production of paper [7] unbleached cellulose [8, 9], wood raw materials and straw [10, 11], and other waste products from agricultural and industrial crops [12–17] were suggested for MCC production. Depending on the type of cellulosic materials, different methods for its degradation were suggested: acid [4], enzymatic [18] hydrolysis, explosive autohydrolysis [10], acidic [9, 11] and alkaline oxidation [19]. The use of additive nonderivative organic compounds preventing recrystallization of disordered elementary sections of fibrils [20] was suggested in order to increase the efficiency of disintegration of fibrous cellulose structure. The positive influence of physical effects such as mechanical activation [21], ultrasonic dispersion [22] and irradiation with electromagnetic waves of microwave range [23] on MCC production was demonstrated.

In spite of the great practical importance of the results of the research, the majority of attempts at using cheap cellulose-containing raw materials did not receive a positive assessment due to the disadvantages, the main ones being the multistaged chemical treatment and the problem of obtaining a purified product. One of the most successful and promising directions of research is the search for the conditions of MCC production by oxidative and oxidative-hydrolytic degradation of lignocellulosic raw materials using such efficient delignification agents as ozone, hydrogen peroxide, and peroxyacids. Examples of studies in this area are studies of MCC production from woody and herbaceous materials, performed by the Institute of Chemistry, Komi Scientific Center of UB RAS [11, 24], the Institute of Chemistry and Chemical Technology SB RAS, and the Siberian State Technological University [21, 25–27]. An important advantage of the use of the oxidative and hydrolytic oxida-

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Table 1. The content of main components in the original wood (% of absolutely dry wood)

Wood species	Cellulose	Lignin	Hemicellulose
Aspen	48.1	21.8	26.4
Pine	49.6	28.0	16.5

tion method is the possibility of combining the processes of degradation of lignin and cellulose, reducing the number of stages and the duration of the chemical processing of raw materials in the course of MCC preparation.

In this study, in order to choose the conditions of single-stage production of MC, we investigated the effect of the composition of the solutions of peracetic acid (PAA) on changes in the characteristics of cellulose during the oxidation-hydrolytic treatment of wood under the influence of microwave radiation (MWR).

MATERIALS AND METHODS

As a lignocellulosic feedstock to produce MCC we used chopped aspen (*Populus tremula* L.) and pine wood (*Pinus sylvestris* L.) in the form of sawdust with dimensions of 0.315–0.630 mm. The humidity of the sawdust was 5.0%. The content of the structural components in comminuted wood (Table 1) was determined according to standard procedures [28]. PAA was prepared with acetylation of hydrogen peroxide with acetic acid in the presence of catalytic amounts of sulfuric acid [29]. For MCC production we used a 15% PAA solutions with a hydrogen peroxide content of 3 M/dm^3, acetic acid 4 M/dm^3, sulfuric acid 0.06–0.7 M/dm^3. The sawdust was treated with PAA solutions under the influence of MWR with a frequency of 2450 MHz and a power 700 W at fluid module 50 and the duration 1–2 h. After treatment, the residue was separated by filtration and washed with hot (70–80°C) distilled water. For evaluation of the effect of MWR on the process of MCC production, a similar experiment was performed using convection heating at 100°C.

The average degree of polymerization (ADP) of the resulting products was determined by a viscometric method in cadoxen (GOST 25438-82). The IR spectra of the cellulose samples were recorded on a Fourier spectrometer Infralum FT-801, X-ray patterns were recorded on a diffractometer Shimadzu XRD 6000 (Tomsk Polytechnic University). The degree of crystallinity of cellulose (DC) was calculated by the integrated X-ray scattering intensity as the ratio of the sum of areas of diffraction peaks to the total area under the scattering curve minus scattering of the background [30]. The transverse dimensions of cellulose crystallites in a direction normal to a system of planes (020) were calculated according to Debye–Scherrer formula:

$$B_{020} = \frac{k\lambda}{h\cos\theta_{020}},$$

where $k = 0.9$ is the dimensionless form factor; λ the wavelength of CuK_α radiation (0.15418 nm); h the reflex half-width 020 rad; and θ_{020} the angle of diffraction from the plane system (020).

RESULTS AND DISCUSSION

Consumption of PAA (concentration of the solution 15%, fluid module 50) in an experiment for MCC production corresponded to the conditions for the efficient removal of lignin from wood [31]. For the selection of the conditions for MCC production under the influence of MWR, we investigated the effect of the duration of the oxidation-hydrolytic treatment of the wood and the composition of the PAA solutions used on the features of extracted cellulose.

The characteristics of the cellulose samples as a function of the duration of release of 15% PAA under the influence of MWR at a concentration of H_2SO_4 0.06 M/dm^3 (corresponding to the amount of H_2SO_4, introduced in H_2O_2 and CH_3COOH mixture during preparation of PAA) are shown in Table 2. For comparison, the characteristics of cellulose extracted from wood with the same PAA solution using convection heating at 100°C are also shown. For the same duration of the process (2 h), samples of cellulose produced under the influence of MWR had lower values of the yield and ADP than for convection heating. This fact indicates the intensifying impact of MWR on processes of oxidation-hydrolytic degradation of polysaccharides by components of PAA solution, and consistent with the data obtained earlier [32, 33].

Table 2. Characteristics of the cellulose, depending on the duration of extraction with 15% PAA (H_2SO_4 concentration—0.06 mol/dm^3)

Extraction duration, h	Aspen wood				Pine wood			
	yield, %	ADP	DC	B_{020}, nm	yield, %	ADP	DC	B_{020}, nm
1	51.0	560	0.62	2.5	51.2	560	0.67	2.5
1.5	49.4	440	0.64	2.7	48.6	480	0.65	2.6
2	47.2 (52.8)*	400 (540)*	0.68 (0.67)*	2.9 (2.8)*	47.5 (51.5)*	400 (520)*	0.63 (0.66)*	2.5 (3.2)*

* The characteristics of the cellulose extracted using convection heating (100°C).

Table 3. Characteristics of holocellulose extracted with 10% PAA

Wood species	Yield, %	The content of alpha-cellulose		DC	B_{020}, nm
		%[1]	%[2]		
Aspen	69.1	68.6	98.6	0.64	2.4
Pine	66.0	64.4	85.7	0.64	2.2

[1] From completely dry holocellulose; [2] based on the cellulose in absolutely dry wood.

Practically the same decrease in cellulose yield and ADP occurs after 1 h of wood treatment with PAA solution under MWR exposure and a 2 h treatment using convection heating. However, the yield of the resulting products did not reach the original content of cellulose in wood (Table 1) due to the incomplete removal of hemicelluloses. Obviously, with an H_2SO_4 concentration of 0.06 M/dm^3, MWR exposure intensifies mainly degradation processes of the lignin-carbohydrate matrix and amorphous cellulose regions without significant effect on elementary fibril regions with a more orderly organization of macromolecules. This assumption evidenced by slower reduction of APF of cellulose during the second hour of PAA treatment, failing to achieve "maximum" values characteristic for wood MCC (120–280, according to [34]). For two types of wood ADP of cellulose decreases only by 160 units and becomes equal to 400.

Despite the incomplete destruction of regions of the elementary cellulose fibrils with the transitional order of macromolecules at H_2SO_4 concentration of 0.06 M/dm^3 and extraction duration of 2 h, the samples were obtained using microwave and convection heating, characterized by relatively high values of DC, corresponding to the range for MCC (0.63–0.83 according to [35]). This fact is due to the high content of the crystalline portion of cellulose in the composition of the original wood, which is indicated by DC value and the content of alpha-cellulose for holocellulose samples (Table 3) extracted by standard methods [28]. However, unlike the holocellulose from original wood, samples obtained with duration of extraction 2 h, have somewhat large values of the transverse dimensions of the crystallites. This indicates some ordering of elementary fibrils in the transverse direction, which, according to the concepts discussed in [36, 37], may be caused by the transition of cellulose into a highly elastic state during swelling and subsequent recrystallization.

Under microwave heating, prolongation of extraction from 1 to 2 h for degraded cellulose samples derived from aspen wood, is accompanied by an increase in DP and transverse dimensions of the crystallites (Table 2). For cellulose samples obtained from pine wood, longer extraction under MWR, conversely, reduced DP and the transverse dimensions of the crystallites remain almost unchanged. However, when the process was conducted using convection heating at 100°C (i.e., less severe conditions), these features of structural changes of cellulose extracted from pine wood did not occur, as evidenced by higher values of DP and transverse dimensions of the crystallites as compared with microwave heating. Obviously, when wood treatment was done using PAA and MWR, destruction of noncrystalline portions of elementary cellulose fibrils was accompanied by the partial decrystallization of ordered regions and the formation of defects on their surface.

The different character of the changes of the structural characteristics of cellulose samples extracted from aspen and pine wood under the influence of MWR, probably due to the peculiarities of the supramolecular organization of polysaccharides and their resistance to oxidation-hydrolytic degradation as part of the original wood. Comparison of DC of cellulose samples derived from aspen and pine wood as the result of extraction with the duration 1 h (Table 2), suggesting that under MWR the mesomorphic and paracrystalline regions of pine wood cellulose are less resistant to the destructive action of components of the PAA solution. The treatment of pine wood with the PAA solution in the presence of MWR for 1 h caused a significant decrease in the content of noncrystalline regions in the resulting cellulose and achievement of a maximum DC value (0.67). When the extraction time of cellulose increased from 1 h to 2 h, the influence of the reaction medium and MWR on macromolecules near the surface of crystallites, preventing their ordering in the transverse direction and accompanied by a decreased DC of cellulose, became obvious. The lack of such effects for extraction of cellulose from aspen wood can be explained by the slower destruction of mesomorphic and paracrystalline regions of elementary fibrils and, as a consequence, the lower availability of cellulose macromolecules at the surface of the crystallites for specific effect of the reaction medium and MWR.

An increased concentration of H_2SO_4 in a 15% PAA solution to 0.3 M/dm^3 or higher resulted in a more intense degradation of cellulose under microwave heating (Table 4). For the same duration of extraction (1.5 h) samples of cellulose obtained by treatment of wood with PAA solutions with higher concentrations of H_2SO_4, had a lower yield, ADP and lateral crystallite size than obtained by treatment with 0.06 M/dm^3 H_2SO_4. In addition, cellulose samples obtained with a H_2SO_4 concentration 0.3–0.7 M/dm^3

Table 4. Characteristics of the cellulose, depending on H_2SO_4 concentration in PAA solution (extraction time 1.5 h)

H_2SO_4 concentration, mol/dm^3	Aspen wood				Pine wood			
	yield, %	ADP	DC	B_{020}, nm	yield, %	ADP	DC	B_{020}, nm
0.3	45.2	260	0.64	2.2	42.7	300	0.67	2.3
0.5	42.2	200	0.69	2.2	42.2	260	0.65	2.3
0.7	37.9	120	0.67	2.2	37.9	160	0.71	2.3

characterized by higher DC values. X-ray patterns of the obtained products (Fig. 1) have two strongly overlapping diffraction maxima at 2θ = 15°–16°, corresponding to reflexes $1\bar{1}0$ and 110 of the crystal lattice of cellulose I. Changing H_2SO_4 concentration in PAA solution from 0.3 to 0.7 mol/dm^3 under microwave heating resulted in the production of cellulose samples with various degrees of degradation of macromolecules, resulting in lower ADP values, but within the interval characteristic of "marginal" DP of wood cellulose. Thus, when the H_2SO_4 concentration in PAA solution was 0.3–0.7 mol/dm^3 and the duration of extraction under MWR was 1.5 h, product characteristics corresponded to those of powdered cellulose obtained by the heterogeneous acidic hydrolysis.

The concentration of H_2SO_4 in a 15% PAA solution from 0.3 mol/dm^3 or higher provides MCC samples with lower values of the transverse dimensions of the crystallites than cellulose extracted from wood with same length of process and H_2SO_4 concentration 0.06 mol/dm^3 (Table 2). Along with higher DP of MCC samples, this feature of the process suggests that an increased H_2SO_4 concentration under MWR accelerated hydrolytic degradation of cellulose macromolecules in mesomorphic and paracrystalline regions of elementary fibrils and, consequently, reduced the possibility of the recrystallization with formation of defective crystal structures. At the same time, for the same duration of MWR, with extraction under MWR, an increase in H_2SO_4 concentration in PAA solution within 0.3–0.7 mol/dm^3 was not accompanied by a reduction of the transverse sizes of the crystallites, indicating that the intensification of the degradation of macromolecular chains occurred primarily in the longitudinal direction of the crystallites (i.e., at the ends). Thus there is no strict dependence between the changes of yield values and DP of MWR samples when the H_2SO_4 concentration is increased. This lack of correlation between the amount of removed material and changes of DP value during the hydrolysis of wood cellulose was noted by the authors of study [38] and, in their opinion, it is associated with the destruction of the crystallites themselves.

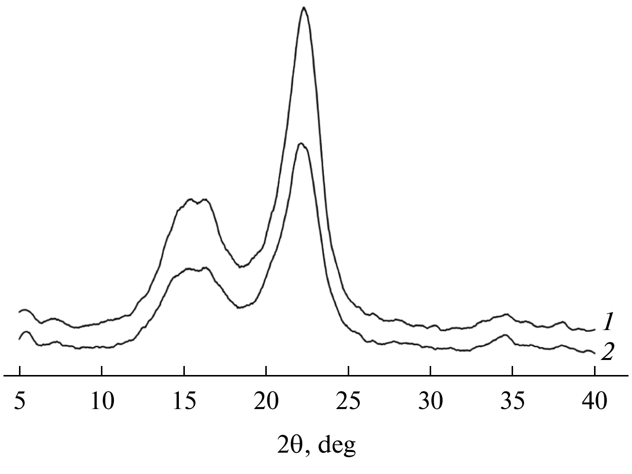

Fig. 1. X-ray patterns of MCC extracted from aspen wood (*1*) and pine wood (*2*) (H_2SO_4 concentration 0.3 mol/dm^3, extraction time 1.5 h).

The data that resulted from treatment of the wood with 15% PAA solution with a H_2SO_4 concentration of 0.3–0.7 mol/dm^3 do not contradict the assumption made above about partial decrystallization of ordered regions of cellulose under conditions of microwave heating. For a H_2SO_4 concentration mol/dm^3 in PAA solution decrease in DC of cellulose during the second hour of extraction from pine wood (Table 2) convincingly explained by partially disordered crystallites. Increased concentration of H_2SO_4 in PAA solution to 0.3–0.7 mol/dm^3 resulted in a more intense destruction of noncrystalline supramolecular structures, preventing their accumulation and providing high DC values of obtained products. The stability of the transverse dimensions of the crystallites at significantly reduced sample ADP values of MCC samples, caused by increased concentration of hydrolysis agent, indicates that the process of decrystallization mainly take place longitudinally in crystallites from the ends. Obviously, first of all, it is associated with a different resistance of bonds, fixing the position of glucopyranose units along three directions of the crystal lattice. This assumption about partially disordered crystallites is also evident from the increased content of the readily hydrolyzed fraction in the cellulose exposed to MWR, established by the authors [39].

The characteristic absorption bands of phenylpropane units of lignin (1605–1593, 1515–1495 and 1470–1460 cm^{-1}) are not present on the IR spectra of the obtained MCC samples (Fig. 2). This indicates the occurrence of deep oxidation reactions of phenolic structures and effective diffusion of delignification

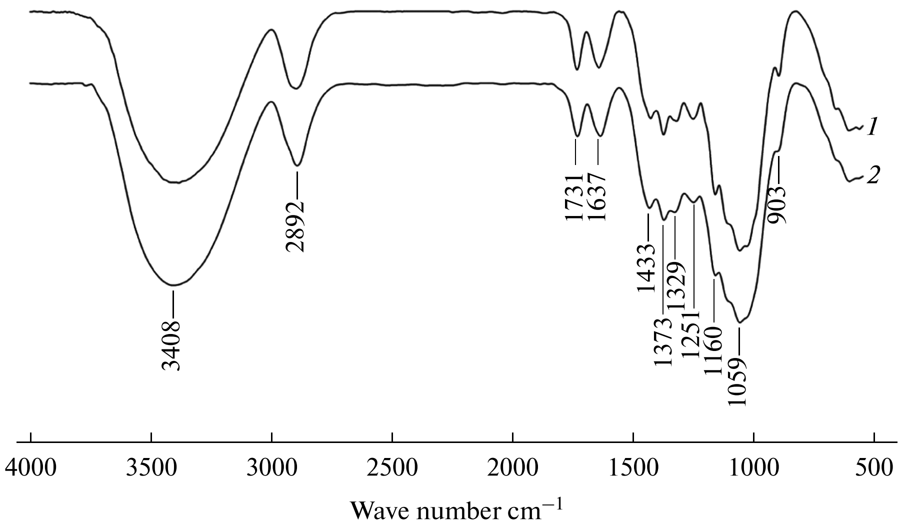

Fig. 2. IR spectra of MCC extracted from aspen wood (*1*) and pine wood (*2*) (H_2SO_4 concentration 0.3 mol/dm^3, extraction time 1.5 h).

products in solution under experimental conditions. The position, shape and intensity ratio of the bands in the IR spectra of MCC samples are similar to that described in [40] spectra of wood cellulose, extracted according to Kurschner-Hoffer method. The nature of absorption in the 1400–1300 cm^{-1} region is due to the structural features of obtained MCC samples. In contrast to spectra of unhydrolyzed residues of cotton and wood cellulose, described in [41], the spectra of extracted MCC samples is characterized by the absence of the band at 1360 cm^{-1} and lower intensity and overlapping bands at 1340 and 1320 cm^{-1}. The presence of the band at 1731 cm^{-1} corresponding to the valence vibrations of carbonyl group, along with the features discussed above, gives grounds to assume the partial oxidation of CH_2OH-groups of glucopyranose units of cellulose with peroxide compounds. As shown for MCC extraction by oxidation-hydrolytic method [42], the absorption at 1730 cm^{-1} appears as a result of the modification of the surface of the crystallites by carboxyl groups.

An analysis of the results leads to the conclusion that under the conditions of the experiment the main factor determining the rate of decline of ADP of cellulose to a “limit” value is the H_2SO_4 concentration. This indicates the predominant role of a hydrolytic degradation over the oxidative destruction in the process of paracrystalline regions of elementary cellulose fibrils during the treatment of the wood with 15% PAA in the presence of MWR. According to current theories [43–45] in an acidic medium the main focus of oxidative reactions involving PAA and H_2O_2 is the process of delignification and the destruction of polysaccharides causing the occurrence of a side process, the hemolytic decomposition of peroxide bonds. Even with relatively high initial concentrations of PAA (15%) and H_2O_2 (3 mol/dm^3) the oxidation-hydrolytic treatment of wood in the presence of MWR for 2 h did not lead to a decrease in ADP of cellulose to “limit” values. Obviously, the highest contribution of the oxidative conversion of cellulose is made to the destruction of the amorphous regions of the elementary fibrils. When the concentration of peroxide compounds decreased, the role of reactions with their involvement in reducing ADP of cellulose becomes less significant. At H_2SO_4 concentration of 0.06 mol/dm^3 hydrolytic degradation of cellulose under MWWR was not sufficiently intensive to provide rapid disintegration of mesomorphic and paracrystalline portions of elementary fibrils. Production of MCC from wood for 1.5–2 h is only possible by increasing the concentration of H_2SO_4 to 0.3–0.7 mol/dm^3 in 15% PAA solution.

CONCLUSIONS

1. In acidic media with initial PAA concentrations not higher than 15% and a H_2O_2 concentration not higher than 3 mol/dm^3, the oxidative conversions of the structural components of wood under MWR are not the main influence on the destruction of paracrystalline regions of the elementary fibrils of cellulose.

2. Under relatively mild conditions of hydrolytic degradation (H_2SO_4 concentration 0.06 mol/dm^3) a disordering effect of MWR on cellulose crystallites was revealed with an increased duration of its extraction from the pine wood by a 15% PAA solution from 1 to 2 h.

3. The conditions for the single step preparation of partially oxidized MCC by oxidative- hydrolytic treatment of wood under the influence of MWR were suggested: PAA concentration 15%, H_2SO_4 concentration 0.3 to 0.7 mol/dm^3, fluid module 50, and the duration of the process 1.5 to 2 h.

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