INFLUENCE OF THE FIBROUS MASS GRINDING METHOD ON THE PROCESS OF POWDER CELLULOSE PRODUCTION

Yu. D. Alashkevich, L.V. Yurtaeva, N.S. Reshetova, R. A. Marchenko
Reshetnev Siberian State University of Science and Technology
31, Krasnoyarsky Rabochy Av., Krasnoyarsk, 660037, Russian Federation.
*E-mail: 2052727@mail.ru

The article presents results of experimental studies of cellulose properties depending on different shaped knife tooling of the disk mill used for grinding. A method for production of powdered cellulose material with characteristics close to microcrystalline cellulose is considered.

Key words: powder cellulose, grinding, knife grinding tooling, disc mill, acid, hydrolysis, fibrous mass, fiber length, fibrillation, polymerization degree.

Depending on the method of fibrous mass processing, the degree of cellulose polymerization decreases, the purity degree and the content of crystalline oriented macromolecules increases and other specific properties appear [1]. Powdered cellulose is produced by destruction of fibrous raw materials. It contains at least 92% of cellulose and has properties different from fibrous cellulose. Powdered cellulose is a white odorless powder, insoluble in water, ethanol, ether and dilute mineral acids, as well as slightly soluble in sodium hydroxide solution. Powdered cellulose is capable to swell in water, dilute acids and many solvents. Alkaline solutions lead to swelling and dissolution of the hemicellulose in its content [1]. Compared to microcrystalline cellulose, powdered cellulose has a lower crystallinity degree, lower chemical purity, contains hemicellulose and lignin.

Microcrystalline cellulose is a finely dispersed product, crystallites of cellulose microfibrils (without the amorphous part), modified natural (fibrous) cellulose powder. Depending on the initial cellulosic material and conditions for cellulose production, average particle sizes of powdered cellulose are in the range from 1 to 400 μm, the polymerization degree is in the range from 30 to 350. Microcrystalline cellulose can be produced from wood pulp, cotton fibers, reeds, cereals and sunflowers, jute and flax stems using mechanical, thermomechanical and chemical treatment of the material by precipitation of cellulose from its solutions in the powder form. The same can be also done by the radiation-chemical method. In contrast to microfibrillar, nanofibrillar and powder celluloses with a low degree of crystallinity, the microcrystalline cellulose is capable to form thixotropic gel-like dispersions in water due to a highly developed hydrophilic surface containing a large number of active hydroxyl groups. At the same time, the microcrystalline cellulose has low molecular weight [2].

Preliminary mechanical destruction of cellulose fiber using knife milling reduces duration and temperature of the reaction, allows to use less concentrated acids for further chemical treatment, as well as reduces the negative impact of the disposal acid solutions on the environment.

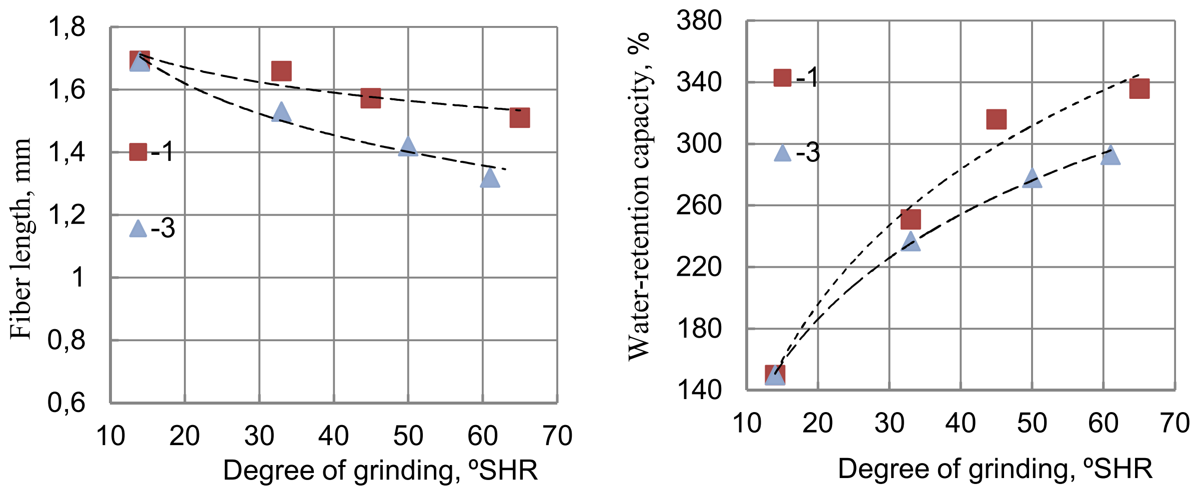
Studying the influence of differently shaped knife tooling on the powdered cellulose production process at the Department of Machines and Apparatus for Industrial Technologies, Siberian State University named after Reshetnev M.F., we treated the fibrous mass using the mechanical (knife) method applying grinding tooling with different knife profiles. A number of properties of the ground fiber suspension was determined. In order to obtain powdered cellulose, the fibrous mass was chemically treated with further determination of its viscosity and polymerization degree.

Grinding of the fibrous suspension with the concentration of 2% was carried out in a semi-industrial disk mill with 15 ºSR to 65 ºSR using a traditional tooling with eight-sector straight knives having an angle of 45° and a tooling with crescent-shaped curved knives [3, 4]. We chose the rotor speed of 1500 rpm and the knife gap of 0.1 mm as the most effective in terms of grinding duration and fiber treatment degree, based on the research previously conducted at the Department of Machines and Apparatus of Industrial Technologies [5].

Before we started the experiment, it was assumed that, all other conditions being equal, the greatest chopping effect would be achieved by the tooling with the straight knives, based on the previously determined second cutting length and contact area of rotor and stator knives. In turn, the curved knife shape will reduce the forces affecting the fiber tangentially, thus, fibrillation will prevail.

For example, the grinding degree of 70 ^{0}SR was achieved in 50 minutes using the tooling with curved shape knives, while the same was achieved in 75 minutes using the tooling with straight knives.

As can be seen from the data presented in Figure 1, when the bleached softwood pulp was mechanically treated, the considered properties changed as follows: the fiber length decreased by 20% and 30% respectively and the water-retention capacity increased by 2 times on average. The highest value of the water-retention capacity indicator is observed in bleached softwood sulphate cellulose milled by the tooling with the curved knives.



1 – curved knife tooling, softwood pulp;
3 – straight knife tooling, softwood pulp.
Fig. 1. Dependence of the fiber length and the fibrous mass water-retention capacity on the grinding duration

Thus, the data obtained as the result of the grinding degree study according to the Schopper-Riegler method, water-retention capacity and fiber length confirm that preferential fibrillation of the fibrous mass is achieved using curved knives. Due to the fibrillation increase, specific surface area of the fibers increases and a large number of cellulose hydroxyl groups are released. This will facilitate efficient penetration of the acid into the fiber structure during the hydrolysis process.

After grinding, the fibrous mass was subjected to hydrolysis in order to enhance destruction of the cellulose structure. Powdered cellulose was produced using the acidic heterogeneous hydrolysis method including preparation of a hydrolyzing solution, hydrolysis of the cellulose, washing, drying, dispersing and screening of the obtained cellulose powder.

The regulated parameters of the hydrolysis process are the temperature and acid concentration. In this regard, we studied three factors of the cellulose hydrolysis process: hydrolysis temperature (varied from 70 to 120 °C), hydrolysis duration (varied from 60 to 120 min) and hydrochloric acid concentration (varied from 2 to 4%). The hydrolysis modulus remained constant (15:1). Optimal conditions for the hydrolysis were determined using mathematical planning according to the Box's three-factor design. Input parameters of the design: hydrolysis temperature x_1 and duration x_2, concentration of the structure breaking agent x_3. Constant conditions of the experiment: hydrolysis modulus (15:1), conditions of washing, drying, grinding and screening. The output parameters: product yield s_{py}, polymerization degree s_{pd} and whiteness s_w of the powdered cellulose.

As a result of optimization, hydrolysis (chemical treatment) of the cellulose samples after grinding was carried out as follows, all other conditions being equal: temperature t = 85 ºC with 2.5% hydrochloric acid HCl using a hydrolysis unit for 75 minutes. The polymerization degree was determined based on viscosity of the iron-sodium solution [6, 7].

Analysis of the obtained data showed that acid hydrolysis decreases the polymerization degree of powdered cellulose, regardless of its type, with an increase in the grinding degree of the treated fibrous mass. The effect was observed in the samples from bleached sulphate softwood cellulose by an average of 0.4% and in the samples from bleached sulphate hardwood cellulose by 0.3%. Thus, the polymerization degree for the samples from bleached sulphate softwood with an initial grinding degree of 15 ºSR equaled to 173, and with the final grinding degree of 65 ºSR it equaled to 115. For the samples of bleached sulphate hardwood the values were 270 and 216 respectively.

Thus, the study shows the possibility to use preliminary knife grinding of fibrous materials to produce powdered cellulose with the characteristics similar to microcrystalline cellulose.

Acknowledgments
The work was carried out under the Government Order issued by the Ministry of Education and Science of Russia for "Deep Conversion of Plant Material" by the Research Laboratory team under the project "Technology and Equipment for Plant Biomass Chemical Processing" (subject number: FEFE-2020-0016).

References

1. Miller J (2017). Nanocellulose Producers, Products, and Applications: A Guide for End Users. TAPPI, 160 p.
2. Miller J (2018). Nanocellulose Challenges and Opportunities: End User Perspectives. TAPPI, 75 p.
3. Alashkevich Yu D, Kovalev V I, Kharin V F, Mukhachev A P Siberian State Technological University, Russian Federation. Razmalyvayushchaya garnitura (Tekst) [Grinding tooling (Text)]. MPK51 D21D1/30, B02C 7/12, 2006110647/12, decl. 03.04.2006, publ. 10.10.2007, bul. No. 28, 8 p., pat. 2307883.
4. Alashkevich Yu D, Kovalev V I, Pakhar E. E, Nesterov D V / Patent na izobreteniye No. 2365695 Razmalyvayushchaya garnitura [Patent for invention 2365695 Grinding tooling]. MPK D21D1/30 (2006.01) B02C7/12 (2006.01).
5. Shurkina V I, Yurtaeva L V, Marchenko R A, Fedorova O N, Alashkevich Yu D (2017) Investigation of the fibrous mass properties using a tooling with curve shaped knives [Issledovaniye svoystv voloknistoy massy pri ispolzovanii garnitury s krivolineynoy formoy nozhey]. Reshetnevskiye chteniya: materialy XXI mezhdunarodnoy nauchno-prakticheskoy konferencii, posvyashchennoy pamyati generalnogo konstruktora raketno-kosmicheskikh sistem akademika M. F. Reshetneva, Krasnoyarsk, 02–11 noyabrya 2017, ch. 2 [Reshetnev readings: materials of the XXI international scientific and practical conference dedicated to the memory of the general rocket and space systems designer and academician Reshetnev M.F., Krasnoyarsk, 02–11 November 2017, part 2]. Krasnoyarsk, Izd-vo SibGU im. M.F. Reshetneva, 2017, part 2, pp. 169-171.
6. Ardizzone S (1999). Microcrystalline cellulose powders: structure, surface features and water sorption capability. Springer Series: Cellulose, 1 (6), pp. 57-69.
7. Obolenskaya A V, Shchegolev V P, Akim G L and others (1985). Prakticheskiye raboty po khimii drevesiny i tsellyulozy [Practical works on chemistry of wood and cellulose]. Moscow, Lesn. prom-t, 412 p.