Investigation of the possibility of obtaining powdered cellulose using a non-knife method of grinding fibrous semi-finished products

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Abstract. The pulp and paper industry occupies one of the leading places among various sectors of the national economy in terms of versatility and prevalence throughout the world, as well as the need for its products for the economy of any country. In addition, with the advent of printing systems that operate at an increasingly faster speed and perform complex tasks, the requirements for the properties and quality of paper have increased. At present, powdered cellulose is a promising direction in the development of the pulp and paper industry. The article discusses the possibility of obtaining powdered cellulose with preliminary grinding on a knifeless grinding plant of the “jet-barrier” type. The morphological properties of the fiber and the degree of polymerization of powdered cellulose are given depending on the degree of grinding.

1. Introduction
In the production of packaging materials, the grinding process is of great importance. Since it affects not only the manufacturing process of the finished product, but also the properties of the fibrous materials from which it is made. The grinding operation, as a rule, is carried out in the presence of water at a fibrous mass concentration of 1 - 8% in grinding apparatus of both knife and non-knife grinding methods. When plant fibers are ground in an aqueous medium, both a purely mechanical process of changing the size and shape of fibers and a colloidal-chemical process called fiber hydration occurs. Phenomena of a mechanical nature are expressed in the shortening of fibers and their fibrillation - the separation of fibrils completely or partially from the fibers, which contributes to an increase in the outer surface of the fibers and the number of free hydroxyl groups on their surface. Hydration during milling is manifested in the swelling of hydrophilic plant fibers, which ultimately increases their ability to bind to each other with the formation of a strong leaf structure [1].

Grinding equipment can be conditionally divided into two large groups, depending on the nature of the impact on the processed pulp: knife machines and machines using knifeless processing methods. When processing in machines using knifeless methods of action, the fibers receive a developed network of interfiber bonds and are subject to much less shortening and destruction than in knife machines. The cast paper web has higher physical and mechanical properties.

Of the non-knife methods of mechanical action on the fiber, of particular interest is the grinding of fibrous semi-finished products in a combined installation of the “jet-barrier” type, in which both knife
and non-knife grinding methods can be present. To highlight the knifeless grinding method, the effect of the knife set in the installation can be excluded [2].

In devices of this type, the principle of the impact of fibers on a hard surface is implemented. The mass with a concentration of 1-3% from the circulation tank 1 is fed by the centrifugal pump 2 through the conical nozzle 3 to the obstacle 4 (figure 1).

![Figure 1. Diagrams of apparatus of the "jet-barrier" type. a - with a flat corrugated barrier; b - with a ω-shaped corrugated barrier; c - with an obstacle in the form of a rotating cylinder; 1 - tank; 2 - pump; 3 - nozzle; 4 - barrier.](image)

By repeatedly passing the cellulosic suspension, any desired degree of pulp grinding can be obtained, just as in knife grinders. The milling character of the mass is fatty, long-fiber. The milling develops in the direction of superficial fibrillation of the fibers, there is only a slight shortening of the fibers.

2. Experimental

At the Siberian State University named after M.F. Reshetnev, at the Department of Machines and Apparatus of Industrial Technologies, research is being carried out on the process of obtaining powdered cellulose. We believe that the inclusion of powdered cellulose in the composition of the paper composition will contribute to an increase in the paper-forming properties and physical and mechanical characteristics of the finished castings [3]. And it will have an additional reinforcing effect on finished paper materials, which is especially important for packaging types of paper [4]. And the intermolecular interaction of polymer components due to the convergence of fibers during compaction of the material, and the directed orientation of macromolecules will increase the printing properties of the paper, which will make it possible to apply a higher-quality pattern.

The disadvantages of many existing methods for producing powdered cellulose are unsatisfactory chemical purity, the use of aggressive solutions of inorganic acids and low environmental performance of the finished product. Analysis of the literature data also showed that research in the field of the influence of the methods of grinding the pulp on the process of obtaining powdered cellulose is practically absent.

The purpose of this study is to study the effect of various modes of a knifeless installation of the "jet-barrier" type during milling of a fibrous suspension on the process of obtaining powdered cellulose and increasing the environmental performance of the finished product.

The installation [5] consists of a mechanism for creating the operating parameters of the jet and a movable barrier; to highlight the knifeless grinding method, the effect of the knife set in the installation can be excluded (figure 2). The mechanism for creating the working parameters of the jet is a system of working and driving hydraulic cylinders. In this case, the drive cylinder has a diameter of 0.11 m, and
the working cylinder is 0.09 m. To avoid corrosion on surfaces in contact with the liquid, the parts of the working cylinder are made of steel 12X18H10T.

![Figure 2. Bladeless jet-barrier grinding plant. 1 - container, 2 - drive cylinder, 3 - working cylinder, 4 - nozzle, 5 - movable barrier.](image)

The pressure is regulated by means of the bypass valve of the pumping station of the installation, from which the system is driven. For free filling and automatic closing of the opening of the slurry supply pipe from the container to the cavity of the working cylinder, a vertically located suction valve is provided. Its main working body is a core in the form of a hollow cylinder, under the action of fluid pressure and due to the lifting force of the air volume inside the cavity, it rises up and closes with its conical part the opening of the branch pipe connecting the working cylinder with the container.

This installation includes two methods of grinding, it is possible to regulate the degree of influence of both knife and non-knife methods, thereby ensuring the required quality of fiber processing. There are two ways to exclude the influence of the knife set on the installation for separating the non-knife method of grinding. For this, the rotor of the knife set of the installation is left stationary, and the mass is chopped without a knife with a fixed barrier. In order to carry out non-knife grinding with a movable obstacle, the maximum gap between the rotor and stator knives is set, thus, the knife part of the mill does not affect the grinding intensity.

 Grinding of the fibrous mass in a non-knife installation, such as "jet-barrier", occurs not only due to impact, but also due to the effect of cavitation, which consists in the formation of discontinuities in some sections of the flow of a moving droplet liquid. Breaks occur in those parts of the flow where there is a significant local pressure drop as a result of pressure redistribution caused by fluid movement. For pulp refining, this effect is positive on which the degree of processing of the fiber depends.

 Due to the cavitation effect, the fibers undergo fibrillation along their axis, which results in a positive effect on the formation of mechanical properties [6, 7].

 Samples of bleached sulphate coniferous (BSC) (semi-finished product of ROP OJSC “Ilim Group”, Bratsk) were used as raw materials. The raw materials were processed by mechanical and chemical methods.

 The mechanical method was carried out on an experimental knifeless installation of the "jet-barrier" type. Technical characteristics of the installation: a nozzle with a diameter of 0.002 m, the pressure inside the hydraulic cylinder is 12 MPa, the distance from the nozzle to the obstacle is 0.1 m.

  Milling of the pulp with a concentration of 1% was carried out from 15 °SR to 75 °SR.

 Then, samples of coniferous cellulose were subjected to hydrolysis using 2.5 N hydrochloric acid in order to destroy fibrous forms of cellulose [8]. The obtained microcrystalline cellulose was subjected to grinding to a powdery state. Figure 3 shows photographs of fibers before and after hydrolysis, taken on a fiber analyzer.
To determine the intrinsic viscosity and the degree of polymerization of powdered cellulose, we used an iron-sodium complex (ISC), which is a complex of iron with sodium tartrate in a sodium hydroxide solution [9].

In the course of the research, samples were taken and the morphological properties of the fiber were investigated at various degrees of grinding before and after hydrolysis (table 1).

**Table 1. Morphological properties of fiber at different degrees of grinding.**

| °SR | Weighted average length of fibers, mkm | Width of fibers, mkm | The content of broken fibers, % | The content of the trifle by length, % | Fibrillation index, % | The content of curved fibers, % |
|-----|---------------------------------------|----------------------|---------------------------------|--------------------------------------|----------------------|--------------------------------|
| 15  | 2284                                  | 27.6                 | 41.17                           | 27.65                                | 0.84                 | 85.24                          |
| 30  | 2203                                  | 26.7                 | 30.67                           | 35.33                                | 1.277                | 69.835                         |
| 55  | 1983                                  | 25.7                 | 29.06                           | 45.69                                | 1.597                | 66.277                         |
| 75  | 1823                                  | 24.5                 | 25.46                           | 54.20                                | 1.974                | 63.195                         |
|     | Non-knife installation of the "jet-barrier" type, bleached sulfate coniferous cellulose |                     |                                 |                                      |                      |                                |
| 15  | 390                                   | 33.5                 | 60.58                           | 84.41                                | 3.21                 | 27.95                          |
| 30  | 345                                   | 35.8                 | 56.24                           | 88.41                                | 3.052                | 28.719                         |
| 55  | 325                                   | 36.2                 | 57.93                           | 89.79                                | 2.906                | 29.852                         |
| 75  | 303                                   | 36.5                 | 59.73                           | 91.59                                | 2.828                | 31.508                         |
|     | Non-knife installation of the "jet-barrier" type, powdered cellulose |                     |                                 |                                      |                      |                                |

As can be seen from the table, during mechanical processing with an increase in the degree of grinding, the weighted average fiber length and width decrease, and the content of fines along the length increases due to mechanical action on the fiber.

The fibrillation index increased during grinding from 0.84% to 1.974%, which once again confirms that the fibers undergo fibrillation along their axis due to the cavitation effect.

The morphological properties for powdered cellulose changed as follows - the weighted average length and the fibrillation index decreased. And the width, the content of broken fibers and the content of fines increase.

As a result of the study, it was found that with an increase in the degree of grinding of the fiber mass, the degree of polymerization of powdered cellulose decreases, from 260 to 95. This is explained by the fact that during mechanical processing of the fiber suspension, not only an increase in the outer surface
of the fibers and the number of free hydroxyl groups on their surface, but also the destruction of intermolecular bonds inside the cell wall of the fiber with the formation of microcracks. All this leads to an increase in the rate of the reaction of the fiber suspension with an acid and to a decrease in the degree of polymerization of powdered cellulose.

3. Conclusion
In the course of the study, the possibility of using a preliminary non-knife method of grinding fibrous materials in obtaining powdered cellulose was shown.

As a result of mechanical treatment due to chopping and fibrillation, the degree of grinding and fiber length of the cellulose suspension change, and as a result of chemical treatment, changes in the composition occur due to partial hydrolysis of cellulose and easily hydrolyzable polysaccharides.

The use of a fibrous suspension, pretreated on a knifeless grinder of the “jet-barrier” type, in the process of obtaining powdered cellulose allows the use of a less concentrated acid due to the formed microcracks inside the cell wall of the fiber. This reduces the cost of chemical treatment and reduces environmental pollution.

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