Bio-damaged Wood Processing in Microcrystalline Cellulose Production

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Bio-damaged wood was studied as a potential raw material for the production of hydrolytic degradation cellulose products. Conditions for obtaining fine-dispersed microcrystalline cellulose (MCC) based on hydrolytic treatment of cellulose from bio-damaged wood were determined. A comparative analysis of the quantitative values of the degree of polymerization of default commercial cellulose and cellulose from damaged wood was performed. The objective of the work was to study the possibilities of obtaining MCC from bio-damaged wood possessing quantitative characteristics close to those obtained from the commercial wood, reducing the concentration of inorganic acid during the hydrolytic degradation. The experimental analysis showed that with an increase in the pulp refining degree from 15 °SR to 75 °SR, the time for the hydrolysis process decreased from 150 to 90 min, the temperature of chemical treatment decreased from 100 °C to 80 °C, and acid concentration by 0.5 N. The polymerisation degree of microcrystalline cellulose, regardless of the type of raw material, also decreased with an increase in the refining degree.

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INTRODUCTION

Deforestation for timber harvesting has long become a widespread phenomenon. This is because wood has always been an integral part of the development of civilization. It is used worldwide to generate energy and make a wide range of end products. It is in constant competition with other materials and products such as concrete, steel, ceramics, synthetic materials, glass, and fossil fuels. According to the WWF Forecast, the global demand for wood will almost triple by 2050 (Report of the World Wildlife Fund 2012). All of this can eventually lead to deforestation and, as a result, to the disruption of the natural functioning of our planet's ecological system. This is because forests not only affect the climate by increasing the level of carbon dioxide in the atmosphere, but they also have a huge impact on the environment, preventing water recycling, causing severe flooding, aquifer depletion, soil degradation, and the extinction of plant and animal species (Momzyakova 2022).

One method to solve this problem is artificial reforestation, in which trees are planted over large areas to increase the total wood stock. However, young trees take a huge amount of time to grow. At that, almost 60% of forest plantations die during their growth due to lack of care for young forests or mistakes made even when planting seedlings (Germino *et al.* 2002). Forests that then grow in such places are typically not suitable for commercial use.

Moreover, like any other resource, wood has different types of defects. Even with well-developed trees, there may be deviations that entail quite detrimental phenomena, such as dead trees or tree parts. All these deviations and damage are described as faults and result in decreased technical qualities. Faults are quality deviations in trunks, crowns, and roots of growing or dead trees that negatively affect the quality of wood and are a consequence of the following phenomena (Alekseev and Poluboyarinov 2006):

- Previous biotic diseases (damage caused by heterotrophic organisms (fungi, bacteria, viruses), by insects, *etc.*) and abiotic order (knots, mechanical damage, curvature, butt swelling, and others not related to ongoing destruction by pathological factors). All of these factors result in forest decay. Forest decay is a negative process whereby the wood fibres decompose. This phenomenon can occur even during tree growth. Its detection helps reduce the price of raw materials, as the process destroys the layers of wood and promotes the development of various diseases. Wood decay spots are characterised by changed colour and decreased strength. Affected areas decompose and after a while turn into dust (Mayorova 2008). The current levels of wood damage are very high. For example, in Siberia, the volume of wood affected by decay is about 20%;
- Difficult growth conditions due to unfavourable climatic and soil conditions. Climate is of great importance when it comes to the formation and life of forests. The composition of forests and their distribution over the earth's surface, as well as their durability and productivity, are closely related to climate. The main components (elements) of climate are light, heat, moisture, and wind. Unfavourable climatic conditions not only slow down the intensity of growth and development of forests, but also reduce their productivity and wood quality. At that, industrial and oil-production emissions into the atmosphere increase the content of sulfur oxides, fluorine, ozone, and carbon in the air. These substances combined with water vapour clouds result in acid precipitation. Acid rain disrupts the root system's ability to absorb minerals from the soil, which results in tree drying. For example, pine trees grown on rich soils have a looser type of wood, while those grown in poor conditions are denser;
- Damage by animal and plant parasites. The most widespread are such species as the Siberian silk moth, Ussuri polygraph, sawfly, pine-tip moth, and heroes (Mayorova 2008). The Ussuri polygraph is the most dangerous pest affecting coniferous trees. While other unexpected parasites infect young shoots, needles, barks, and bast layers, thereby giving the wood a rough and unaesthetic appearance, bark beetles pose a serious threat to the life of trees and are therefore considered the most dangerous pests. They are capable of destroying even large mature trees during just one season. Over the past ten years, the Ussuri polygraph has been the main cause of fir tree degradation within the Novosibirsk, Kemerovo, and Tomsk regions, as well as Krasnoyarsk Krai and Altai Krai. To date, the total area of established quarantine phytosanitary zones related to the Ussuri polygraph is 606.71 thousand hectares (these are 44 zones on the territory of 14 districts of Krasnoyarsk Krai, Russian Federation), while the area of focal points is 164.08 thousand hectares;

- Unreasoned human activities in forests. People pose the most significant and most diverse influence on the course of forest formation. People build factories and obtain metals that are necessary for human life, thereby emitting a large amounts of detrimental substances into the air and destroying forests. Plowing areas and grazing livestock also prevent forests from the spreading. With the rise in the population and the development of agriculture, new areas already overgrown with forest vegetation become involved in the economic turnover. Plantings are burned out, uprooted, while vacant lands are used for agriculture;
- Forest fires. There are two groups of forest fires: natural (lightning strikes, volcanic eruptions, meteorite falls, spontaneous coal and peat combustion) and anthropogenic (directly related to human activities).

To reduce the negative consequences of the above factors and improve the sanitary and forest pathological condition of forests, dead and damaged forests are sanitarily deforested in affected areas. As a result, there is an immediate problem of how to recycle damaged wood (Yurtayeva and Alashkevich 2022), which depending on the degree of damage caused to the defective trees, is attributed to semi-commercial or even wood-burning trees during enumerations in main-use cutting areas.

At the Siberian State University named after M. F. Reshetnev, the laboratory of the Department of Machines and Devices of Industrial Technologies is carrying out studies on the possibility of obtaining paper and microcrystalline cellulose with preliminary refining of fibrous semi-finished products by both cutter and non-cutter refining methods (Alashkevich 1980; Kutovaya 1998; Kaplev *et al.* 2022) from bio-damaged wood. This damage occurs in freshly harvested timber, as well as in dry and weakened trees in forests. With this, large or oval holes or grooves are visible on the timber surface. The main types of destruction are not caused by adult insects, but by their larvae, which obtain their nutrition from wood and bark.

Analysis of literary sources has shown that there are practically no studies on the use of bio-damaged wood as a raw material in the production of pulp and paper products (paper and microcrystalline cellulose).

Microcrystalline cellulose is a product of cellulose hydrolysis. Traditionally, MCC is obtained by treating cellulose with concentrated acids. In this case, the fibrous structure is destroyed and cellulose powder is formed (Battista 1964; Kai *et al.* 2000; Aleshina *et al.* 2014).

The objective of this study was to utilize softwood (fir) damaged by the Ussuri polygraph with a degree of damage of 22%; study the possibilities of obtaining MCC from bio-damaged wood possessing quantitative characteristics close to those obtained from commercial wood; and reduce the concentration of inorganic acid during the hydrolytic degradation of cellulose.

The study objectives include: obtaining unbleached pulp from bio-damaged wood; pulp processing by the hydro-mechanical method using a non-cutter jet-barrier type machine; chemical pulp processing; and comparing the polymerisation degree of cellulose macromolecules made of commercial and bio-damaged wood at different refining degrees according to the Schopper-Riegler method.

The scheme for obtaining microcrystalline cellulose of hydrolytic destruction is shown in Fig. 1.

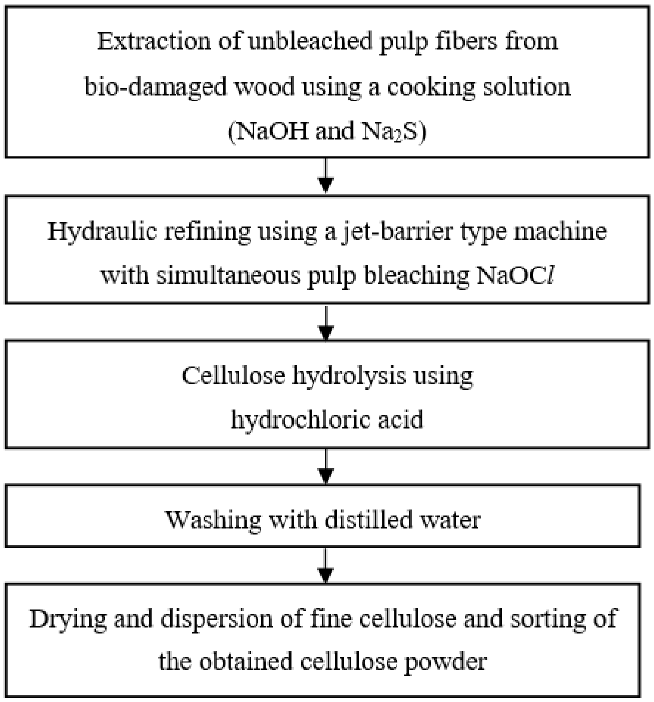


Fig. 1. Scheme of obtaining microcrystalline cellulose

EXPERIMENTAL

The following laboratory methods were used to monitor the refining of pulp obtained from bio-damaged wood:

- Measurements of the refining degree by °SR were carried out in accordance with ISO 5267-1 (1999);
- Mass fraction of alpha-cellulose determined according to GOST 6840-78 (1978);
- Castings produced in accordance with ISO 5269-1 (2005);
- Determination of physical and mechanical characteristics, including breaking length, bursting strength in accordance with ISO 5270 (2012), ISO 1974 (2012), and ISO 1924-2 (2008).
- Polymerisation degree determined in accordance with GOST 9105-74 (1974).

Because the kraft pulping method is currently not only the dominant alkaline pulping method when using wood as a raw material, but also the most important technique for the production of pulp, the method described in this work was used to extract pulp from bio-damaged wood with a pulping solution with its main components as sodium hydroxide and sodium sulfide (NaOH and Na_2S). Pulping was carried out in a laboratory autoclave at a maximum temperature of 170 to 171°C for 3.5 h, liquid module – 5, and degree of sulfidity of the cooking liquor – 20.8%. To extract pulp, the resulting semi-finished product was washed and screened in a laboratory blow tank. The pulp yield after cooking was 43%, and the alpha cellulose content was 84.5%.

The problem in obtaining powdered cellulose materials using chemical means is the recycling of used acid solutions after the hydrolysis. To reduce the harmful effects of inorganic acids on the environment and energy consumption for obtaining microcrystalline cellulose, pulp extracted from bio-damaged wood was subjected to hydro-mechanical method of processing.

The hydro-mechanical method of processing the obtained pulp included the refining and bleaching of a fibrous suspension with a concentration of 2% on an experimental non-cutter refining jet-barrier-type machine (Lahno *et al.* 1990) (Figs. 2 and 3) from 15 °SR to 75 °SR with parameters selected on the basis of studies previously conducted at the Department (Kaplyov *et al.* 2021; Yurtayeva *et al.* 2021; Alashkevich *et al.* 2023): operating pressure 13 MPa, distance from the nozzle to the barrier 0.2 m, and nozzle taper angle 45°.

Since the hemicelluloses and lignin in chemically processed pulp reduce the quality of the finished products, the fibrous suspension was subjected to bleaching during refining. Sodium hypochlorite (NaOCl), hydromodule 1:22, was used as the bleaching agent.

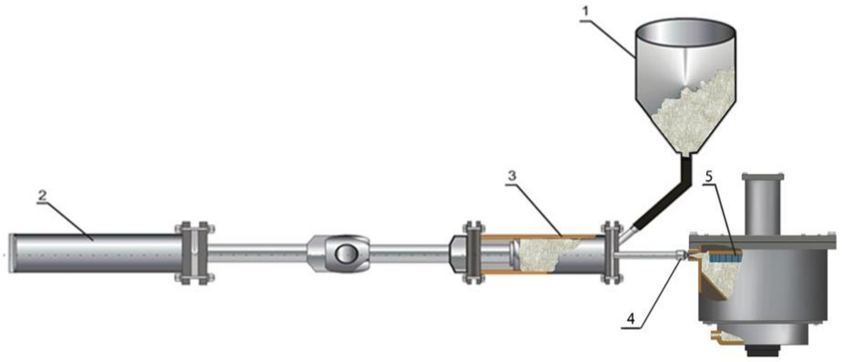


Fig. 2. Bladeless jet-barrier refining plant: 1 – container; 2 – drive cylinder; 3 – working cylinder; 4 – nozzle; 5 – movable barrier

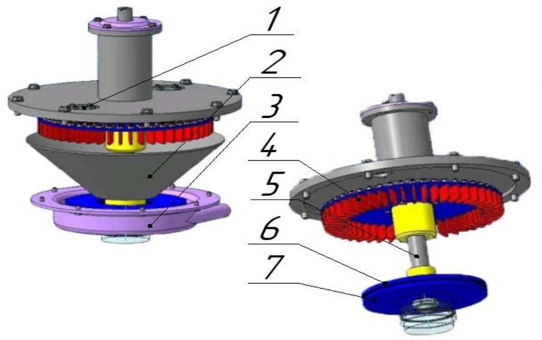


Fig. 3. Combined refining node: 1 – combined refining node body; 2 – cone; 3 – knife-based refining chamber; 4 – turbine; 5 – shaft; 6 – tackle stationary disc; 7 – tackle movable disc

When refining plant fibres in an aqueous medium, the following processes occur: Mechanical processing results in changes in the size and shape of fibres, with their longitudinal splitting into fibrils. Colloid chemical effects tend to increase the number of free hydroxyl groups on the fibre surface.

Pulp refining in a non-cutter machine, such as a jet-barrier-type one, takes place due to not only the impact, but also the cavitation effect, which consists in the formation of discontinuities in some sections of the flow of a moving droplet fluid (Fig. 4). The discontinuities occur in those parts of the flow where there is a noticeable local pressure drop resulting from pressure redistribution due to fluid movement. For pulp refining, this effect positively affects the degree of fibre processing.

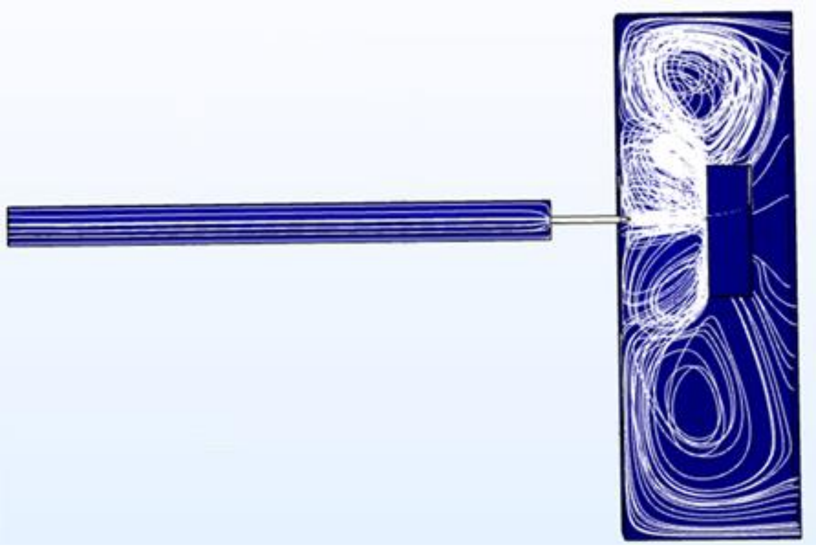


Fig. 4. Impact of a fibrous suspension on an obstacle in a "jet-barrier" type installation

The cavitation effect makes fibres undergo fibrillation along their axis, resulting in a positive effect on the formation of mechanical properties (Kaplyov 2019). Moreover, the increased outer surface of the fibres is most influenced by the flow rate of the jet from the nozzle to the barrier. Marchenko (2006) found that it affects the impact force of the jet on the barrier and the magnitude of the tangential stresses arising when the jet spreads over the barrier, as well as the wave nature of the jet movement, which, in turn, determines the effect of ultrasonic cavitation at the point of contact of the jet with the barrier.

In experimental studies, the jet velocity was determined by Eq. 1,

$$v = \frac{V}{F \times T} \tag{1}$$

where V is the volume of the suspension (m^3), F is the cross-sectional area of the nozzle (m^2), and T is the flow time of the suspension from the cylinder (s).

The jet flow rate was determined for the suspension (1) based on measurements of the suspension flow time from the working cylinder at pressure 13 MPa and concentration 1 kg/m^3. For example, in the current study, with the nozzle diameter 0.002 m, the pressure in the working cylinder 13 MPa, the concentration 1 kg/m^3, and 60 °SR flow time of the suspension amounts to 13.8 s. Nozzle cross-sectional area was calculated according to Eq. 2:

$$F = \frac{\pi \times 0.002^2}{4} = 3.14 \times 10^{-6} \tag{2}$$

The jet flow rate is important and was calculated according to Eq. 3:

$$v = \frac{0.005}{3.14 \times 10^{-6} \times 13.8} = 115.4 \text{ m/s} \tag{3}$$

After refining, the pulp was subjected to hydrolysis to enhance the destruction of the cellulose structure and obtain microcrystalline cellulose from it by hydrolytic degradation. Figure 1 shows the scheme for obtaining microcrystalline cellulose with preliminary refining of a fibrous suspension.

The regulated parameters of the hydrolysis process are temperature and acid concentration. Because the acid concentration depends on the reaction temperature, a higher heating temperature results in a less concentrated acid solution that should be used. In this regard, three factors of the cellulose hydrolysis process were studied: hydrolysis temperature (varied from 70 to 120 °C), hydrolysis duration (varied from 60 to 120 min), and hydrochloric acid concentration (varied from 1.5 to 3%). The hydrolysis ratio remained constant (15:1). Optimal conditions for the hydrolysis were determined using mathematical planning according to the Box design for 3 factors. Design input parameters: hydrolysis temperature x1, duration x2, and destruction agent concentration x3. Constant conditions of the experiment: hydrolysis modulus (15:1), washing, drying, refining, and screening conditions. Output parameters: product yield yв, polymerisation degree yp.d, and whiteness yb of microcrystalline cellulose.

The cellulose molecule consists of a supramolecular structure (amorphous and crystalline sections) and hemicelluloses (Fig. 5).

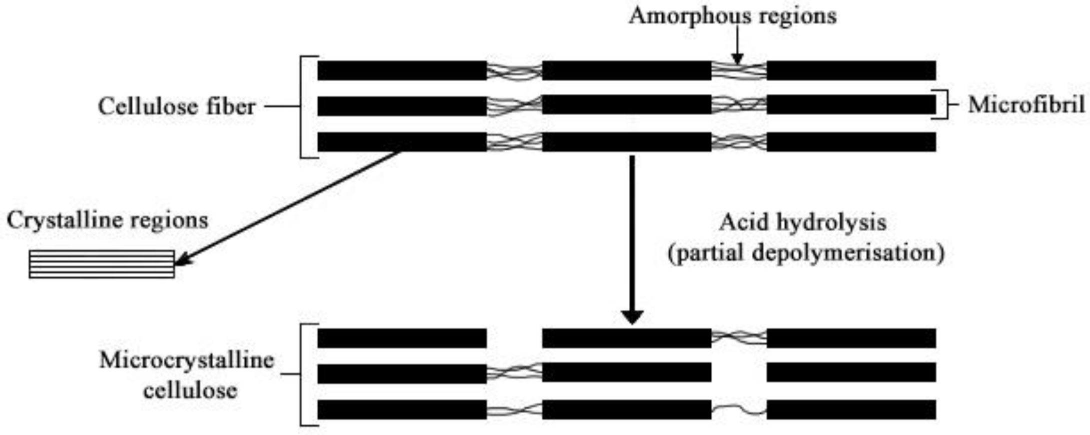


Fig. 5. Chemical destruction of cellulose

The hydrolysis (chemical treatment) of cellulose samples after refining was carried out using 1.25 to 2.0 N hydrochloric acid to destroy their fibrous structure. During cellulose hydrolysis, amorphous regions are destroyed first, cellulose loses its fibrous structure and turns into hydrocellulose, *i.e.*, a mixture of the original cellulose with hydrolysis products of different polymerisation degrees. Hydrocellulose had a reduced polymerisation degree but a higher crystallinity degree compared to its original version. Before obtaining hydrocellulose, hydrolysis proceeded rapidly (at a high speed) to a temperature of 100 °C. Cellulose microfibrils broke down into individual crystallites. Thereafter, the reaction slowed down sharply and the maximum polymerisation degree was reached. The polymerisation degree expresses the average length of cellulose chains. After hydrolysis, the resulting microcrystalline cellulose was washed, dried, and dispersed.

To determine the polymerisation degree of the microcrystalline cellulose, a complex compound of hexasodium tristartrate, or the so-called ferruginous sodium complex (FSC), was used, which is a complex of iron with sodium tartrate in a sodium hydroxide solution (Obolenskaya *et al.* 1985; Vasyutin and Kanavellis 1968).

RESULTS AND DISCUSSION

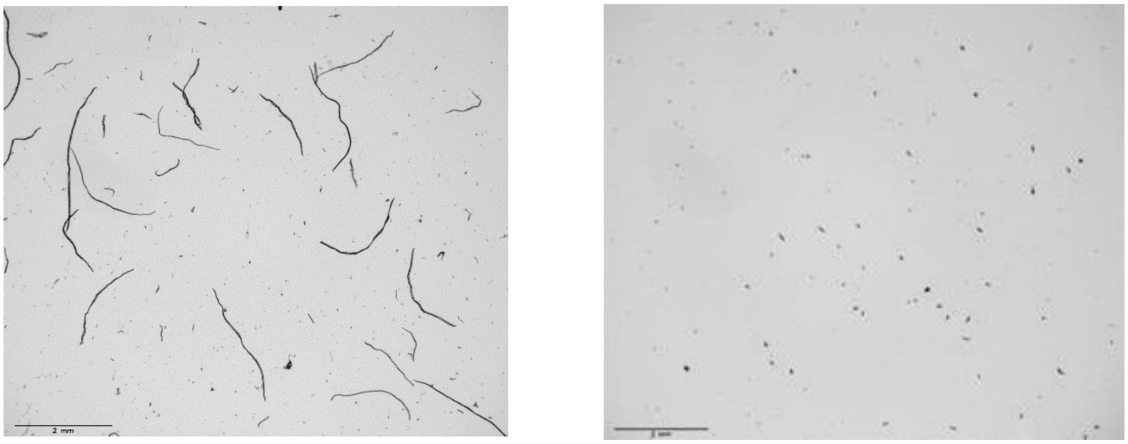
Table 1 contains experimental data on the mechanical strength properties of finished castings at the refining degree on the Schopper-Riegler scale after refining on a non-cutting refining jet-barrier-type machine when compared to the above indicators in accordance with GOST 11208-82 (1982) for kraft unbleached wood pulp (softwood) of various grades (Table 1).

Table 1. Physical and Mechanical Indicators

Degree of Grinding on the Shopper-Rigler Scale (°)	Lignin (%)	Alpha cellulose (%)	Weighted average length of fibers (mc)	Breaking Length (km)	Pressure Resistance (kPa)	Tear Resistance (gs)
15	5,12	84,5	2203	3,85	153	700
60	2,03	83,4	1823	10,52	517	1000
NS-1 (60)	-	-	-	9,10	470	830
* NS-1 – GOST 11208-82 indicators with a grinding degree of 60 °C						

Table 1 shows that with an increase in the pulp refining degree, the quantitative values of the mechanical properties of castings from bio-damaged wood with a refining degree 60 °SR on the Schopper-Riegler scale were slightly higher than those of GOST 11208-82 at the same refining degree, unlike castings at the refining degree 15 °SR.

Therefore, the fibrous suspension obtained from bio-damaged wood and passed the refining stage can be recommended for the manufacture of high-strength technical and packaging paper; moisture-resistant base paper for abrasive cloths, telephone and cartridge paper, ODP-35 and ODPN-28 waxed paper bases, and for other special types of paper.



(a) (b)

Fig. 6. Photos of cellulose fibers: (a) before hydrolysis, (b) after hydrolysis

Microcrystalline cellulose was obtained after refining the fibrous suspension at a refining degrees of 15, 30, 60, and 75 °SR. Figure 6 presents cellulose fibres made using a fibre analyser at a refining degree of 60 °SR and after hydrolysis, illustrating the difference between the sizes of natural fibres and hydrolysed ones. Thus, for example, with an increase in the refining degree, the weighted average length of fibres and their width decreased 14%, while the content of fines along the length and the fibrillation index increased by 2 times. Moreover, the nature of the change in the morphological properties for microcrystalline cellulose is as follows – the weighted average length and the fibrillation index decreased 48% and 16%, respectively, while the fines content increased 30%.

For the microcrystalline cellulose (Fig. 7), the yield was 95%. A sieve analysis according to GOST 3584-73 (1973) showed that particles smaller than 100 microns in size were 92.4%; crystallinity index according to X-ray phase analysis – 0.72; whiteness – 86.3%; and bulk density – 308 kg/m^3.

Fig. 7. Photos of microcrystalline cellulose

The experimental analysis showed that with an increase in the pulp refining degree from 15 to 75 °SR, the time for the hydrolysis process decreased from 150 to 90 min; the temperature of chemical treatment decreased from 100 to 80 °C; and acid concentration decreased by 0.5 N. The polymerisation degree of microcrystalline cellulose, regardless of the type of raw material, also decreased with an increase in the refining degree.

Figure 8 shows the dependence of the polymerisation degree of microcrystalline cellulose on the bio-damaged wood pulp refining degree when compared to pulp extracted from commercial softwood and hardwood species.

As can be seen from Fig. 8, regardless of the type of cellulose, with an increase in the pulp refining degree, the polymerisation degree of a microcrystalline cellulose decreased. Thus, at a freeness value of 15 °SR, the quantitative values of RD for microcrystalline cellulose value of 60 °SR were 17% higher, and at a freeness value of 75 °SR, the quantitative values of polymerisation degree practically did not differ. This is explained by the fact that the refining of the 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 with the formation of 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 the microcrystalline cellulose. For the quantitative polymerisation degree values of microcrystalline cellulose obtained from hardwood at a refining degree of 75 °SR, as can be seen from the graph, they were significantly higher than those of softwood. This is due to the difference in the structure of softwood and hardwood fibres.

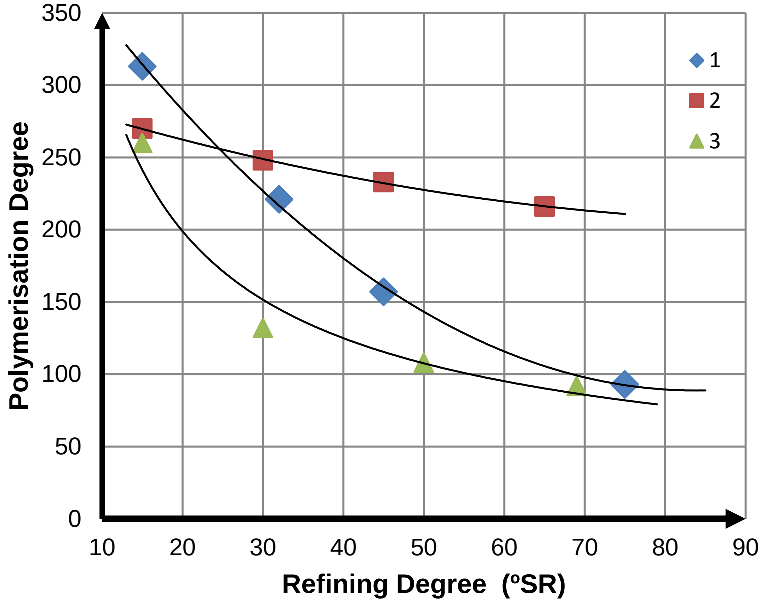


Fig. 8. Dependence of the polymerisation degree of microcrystalline cellulose on the pulp refining degree: (1) Cellulose extracted from bio-damaged wood; (2) Cellulose extracted from hardwood; (3) Cellulose extracted from softwood

CONCLUSIONS

1. It was shown that the production of microcrystalline cellulose from bio-damaged wood partially balanced the consumption of commercial wood and recycled bio-damaged wood;
2. It was revealed that microcrystalline cellulose can be obtained from bio-damaged wood with characteristics close to those of commercial wood;
3. Using a fibrous suspension preprocessed with a non-cutting refining jet-barrier-type machine can reduce costs (acid concentration, processing time and hydrolysis temperature) for further chemical treatment in obtaining microcrystalline cellulose.

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