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PRODUCTION OF BINDER-FREE BOARDS FROM BIODEGRADED ABIES SIBIRICA WOOD

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ABSTRACT

The aim of this paper is to investigate the feasibility of obtaining binder-free wood boards from rotted wood at the final stages of biodegradation. The rot-affected wood of *Abies sibirica* harvested from a stand killed by exposure to *Polygraphus proximus Blandford* was used as a subject of research. The wood boards from rotted wood were produced by wet hot pressing. The wood pulp was prepared by hydrodynamic treatment in a rotary pulsation disperser. The obtained wood boards with a density of 800 ± 20 kg/m^3 and a thickness of 8 mm have an ultimate static bending strength of 28 MPa, modulus of elasticity (MOE) of 3.5 GPa, ultimate tensile strength perpendicular to the plate of 0.92 MPa, swelling in terms of thickness of 4.61 % in 24 hours. The boards dried after swelling tests retained 96% of their initial strength. The boards produced of biodegraded wood correspond to the EN622-3 semihard fibreboards (MB) in terms of mechanical parameters and are significantly superior in terms of water resistance.

Keywords: rotted wood; brown rot; dead wood; boards; properties; hydrodynamic treatment.

INTRODUCTION

In the context of climate change and imbalance, as well as active anthropogenic activities, there is an increasing incidence of massive mortality of forest stands. Trees die as a consequence of changes in forest growth conditions caused by droughts (Dietze and Moorcroft, 2011, Anderegg *et al.,* 2013, Anderegg *et al.,* 2015, Berdanier and Clark, 2016), forest fires (Shvidenko and Shchepashchenko, 2013, Erisov *et al.,* 2016, Wang *et al.,* 2014), widespread outbreaks of pests and diseases (Cherpakov, 2012, Tatarintsev *et al.,* 2021, Rizzo, 2003, Campbell *et al.,* 2002, Volney, 1998, Basham, 1957), harmful species invasions (Roques *et al.,* 2016, Okland *et al.,* 2019, Littell *et al.,* 2009, YuN *et al.,* 2011, Melnik *et al.,* 2018, Asner *et al.,* 2018), etc. In regions where forestry experiences massive tree mortality, felling of dead stands is practiced within the first 2-3 years. It allows mechanized harvesting at no additional cost and the use of wood almost without restrictions (Basham, 1986). Nevertheless, felling dead trees in a short period is not always possible. This may be due to the extent of stand mortality, insufficient transportation infrastructure, or a lack of sufficient production capacity for logging companies. In this case, with the increase in the age of a stand, the trunks of dead trees undergo a consistent process of destruction under the influence of physical and biological factors. The final stage of wood destruction is the development of wood-destroying fungi in it. Their activity is associated with a sharp decrease in the consumer properties of wood. Such wood is in low demand by the wood processing industry (Basham, 1984).

The destruction of coniferous wood is primarily caused by basidiomycete fungi, which result in the development of brown rot in wood (Schwarze *et al.,* 2000; Qi *et al.,* 2022; Kim *et al.,* 1996; Ruddick, 1986). It is one of the most destructive types of wood rot (Qi *et al.,* 2022; Green and Highley, 1997). The peculiarity of brown rot is that in the process of destruction under the action of fungal enzymes, first of all, polysaccharides such as cellulose and hemicellulose contained in wood cell walls are decomposed, while lignin is practically unaffected by them (Curling *et al.,* 2002, Green and Highley, 1997, Goodell and Jellison, 2001). As the wood decomposes, the porosity of the wood cell wall increases, and the destruction of the strong reinforcing cellulose framework makes it brittle—the cell wall cracks and shatters (Eriksson *et al.,* 2012).

Thus, biologically infested wood differs significantly in properties from healthy wood, which requires the development of special approaches to its processing.

As the analysis of the works (Barrette *et al.,* 2015, Jouzani *et al.,* 2020, Luo *et al.,* 2010, Bowyer *et al.,* 2003, Solomatnikova *et al.,* 2011) shows, the main direction of processing of such wood is obtaining solid, liquid or gaseous fuel, as well as pulp and paper products. Nevertheless, when using this type of raw material in the production of fibrous materials, several limitations arise. Wood processing with low moisture content (less than 30%) does not allow for providing the required dimensional and qualitative characteristics of wood fibers (Lewis *et al.,* 2006). It is also noted that the penetration of wood-dyeing fungi changes the colour of wood, which creates problems with bleaching in pulp production. And when wood is infested with brown rot, it significantly reduces pulp yield (Hoeger *et al.,* 2014).

One of the most intensively developing areas of wood processing is the production of boards (Muhcu *et al.,* 2015). In classical technology for wood boards, the addition of no more than 10% rotted wood is allowed, as it reduces the mechanical properties of the material, increases the consumption of binders, and worsens the strength of adhesive joints due to a decrease in the pH of the wood (G. Nemli et al, 2018, Goncalves *et al.,* 2008, Byrne *et al.,* 2007).

Binder-free boards are of particular interest in modern conditions. The basis of such technology is the pre-treatment of wood through mechanical (Schell *et al.,* 1994; Shu *et al.,* 2021), chemical (Maloney, 1996; Widsten and Kandelbauer, 2008), or biological activation.

Biological methods of wood modification are of great interest to researchers. For example, Kerner suggest that, to obtain dry MDF boards with a density of 700 kg/m^3, chips should be pretreated with the brown rot fungus Coniophora puteana (Kerner *et al.,* 2001). This wood preparation reduces the energy required for milling in the refiner by 40%. The mechanical properties of the boards are higher, and the swelling is 60% lower compared to boards made from healthy wood.

According to the authors of the paper (Wu *et al.,* 2019), the hydrodynamic method is promising in the activation of lignocellulosic raw materials. Hydrodynamic activation of wood in a rotary pulsation disperser enables an increase in interfacial surfaces of wood particles by more than 2.5 times. It is due to the fibrillation of wood cell walls in the form of partial detachment of cellulose fibril bundles, as well as an increase in the proportion of fine fraction (size less than 20 microns) with a large specific surface area in the wood pulp. As a whole, this creates conditions for the formation of board structures without the use of adhesives due to the interphase interaction between wood particles of the activated mass (Bimestre *et al.,* 2020, Wu *et al.,* 2019, Ermolin *et al.,* 2020, Karinkanta *et al.,* 2018, Akpan *et al.,* 2021).

The purpose of this paper is to study the possibility of obtaining medium density boards from hydrodynamically activated wood of Abies sibirica affected by brown rot at the last stages of development.

MATERIALS AND METHODS

Materials

The studies were conducted on Abies sibirica wood affected by brown rot, harvested from a stand located 20 km west of the city of Krasnoyarsk. The billets were sawn from the dead trees affected by the Polygraphus proximus Blandford 20 years ago. The wood had a light brown colour. The density in the dry conditions was 245 kg/m3; the compression resistance along the fibers was 13 MPa. The uninfested Abies sibirica wood from the same region was used as a control for wood properties. In its absolutely dry state, it had a density of 365 kg/m³ and a compression resistance along the fibres of 38 MPa. The primary disintegrating of biodegraded wood was carried out using a laboratory chipping machine. The obtained wood chips were ground using a laboratory hammer mill with an 8 mm sieve diameter. The photo of the obtained particles is presented in Fig. 1. The fractional composition of the particles in the infested wood, determined by the dry fractionation method, is shown in Fig. 2.

(a) (b)

Fig. 1 Rotted wood: a – solid wood; b – disintegrated wood.

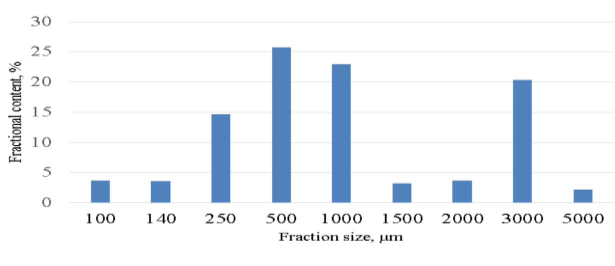


Fig.2 Fractional composition of the particles of the infested wood of Abies Sibirica.

Thermogravimetric analysis of wood

When wood is affected by wood-destroying fungi, some changes occur that significantly influence the thermal degradation processes of wood (Poletto *et al.,* 2010, Loskutov *et al.,* 2022). It predetermines the possibility of studying the physicochemical state of wood by the thermogravimetric analysis (TG/DTG). This method is widely used to study the primary polymeric components of wood, including rotted wood, and to determine the ratio of aromatic and carbohydrate components (Poletto *et al.,* 2012; Nassar, 1984).

Taking this into account, to evaluate the physicochemical changes in wood affected by rot, the thermogravimetric method (TG/DTG) was applied. Wood particles of healthy and rot-infested Abies sibirica wood were used as samples (Fig. 1).

The TG/DTG was carried out using the TG 209 F1 instrument ('NETZSCH', Germany) under the following conditions. The heating rate of samples in an oxidising atmosphere (compressed air) is 10 °C×min^{-1} from 25 to 700 °C; the flow rate of protective and purging gases is 20 ml×min^{-1}; the mass of samples of healthy wood and the wood in the final stages of rot is 3.94 and 4.27 mg respectively; Al2O3 crucible has a cylindrical shape. The calibration of the device was carried out according to the manufacturer's procedure and using standard substances from 'NETZSCH'. The data processing of the thermal analysis was carried out using the software package called 'NETZSCH. ProteusThermalAnalysis. 4.8.4.'

Hydrodynamic processing of wood

Wood processing was carried out in a laboratory hydrodynamic disperser of rotary-pulsation type (Fig. 3). The chopped wood was mixed with water at a temperature of 8-10°C in the tank (1) to a concentration of 6%. The unit was started, and the mass was repeatedly processed by passing through the processing chamber of the unit (rotor and stator) (3). The rotor speed was 2950 rpm. During the operation, axial oscillation of the rotor occurs.

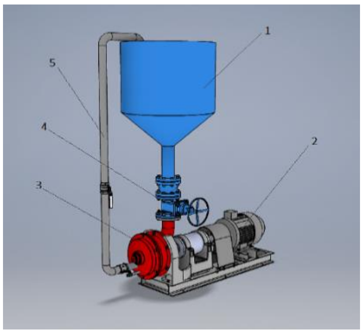


Fig. 3 General view of the experimental unit: 1 – tank; 2 – electric motor; 3 – hydrodynamic disperser; 4 – stop valve (gate valve); 5 – circulation pipe.

The fractional composition of WP mechanical wood pulp was studied using the sieve analysis method with the aid of a Retsch AS 200 control analytical screen sieve (Retsch GmbH, Haan, Germany). The weighing operation was performed using a laboratory balance with an accuracy of 0.001 g.

The morphology of wood pulp particles obtained from hydrodynamic treatment was studied using a scanning electron microscope (SEM) (Hitachi TM4000Plus, Japan).

During the hydrodynamic treatment of wood pulp, several changes occur. The dynamics of these changes depend on the type and design features of a given apparatus. Therefore, it is not reasonable to assess the results of processing by the duration. When processing fibrous materials (such as wood and cellulose), different express methods are used, which, through indirect indicators, allow us to estimate qualitative changes in the material. To assess the results of wood and cellulose milling, the water retention value (WRV) or the Schopper-Riegler degree (^{0}SR) is used (Jayme *et al.,* 1966, ISO 5267-1, 2002). Wood affected by rot, especially brown rot, is a poorly studied material. Therefore, both methods were used to assess the degree of wood processing to select the most appropriate one.

The water retention value (WRV, %) was determined according to Jaime's method (Jayme *et al.,* 1966). It represents the moisture content of the wood pulp after it has been centrifuged. The wood pulp samples were centrifuged at 3000 g for 10 minutes using the Janetzki T23 centrifuge. After that, they were dried to an absolute dry condition. The fineness was determined using a Schopper-Riegler instrument according to PN-EN ISO 5267-1 (2002).

Hot pressing of the boards

Mat formation was carried out by pouring the obtained wood pulp into a special metal mould with a mesh bottom. To dewater the mat, mechanical thrust in a cold press with a pressure of 1.0 MPa was used. After that, the boards were placed on the mesh pallets into a hot press called Fontijnepresses LabPro 1000 (Denmark). Hot pressing was carried out at a temperature of 180 °C, a specific pressure of 2.5 MPa, and a specific duration of 2 min/mm. The distance between the spacing strips corresponded to the thickness of the finished board with a format of 400 by 400 mm and a density of 800 ±20 kg/m^3. To ensure a coefficient of variation of no more than 15% in the subsequent physical and mechanical tests, five boards were produced from each type of wood pulp with varying processing times.

Testing of the boards

After pressing, the boards were kept in the laboratory for conditioning for one week. Next, the boards were cut into samples for testing. The mechanical properties of the boards were determined on the UTS-30 testing machine (Russia). The tests were carried out according to the following standards: static bending (BP), modulus of rupture in bending (MOR) (EN 310), tensile strength perpendicular to the plate IB, and swelling.

For a more detailed study of the moisture resistance of the obtained boards, further studies were conducted. The test samples were placed in a water bath and filled with water that had a temperature of (20±2) °C. The distance between the samples and the walls of the water bath was 15 mm. The water was heated to the boiling point (100°C) for (90±10) min. The duration of boiling of the samples was (120±5) min. Then, the samples were taken out of the bath, and the moisture from the surface of the samples was removed with a paper towel and placed in a drying box at (70±2) °C for (960±15) min. After drying, the samples were removed from the drying box and cooled to room temperature. To determine the tensile strength perpendicular to the plate (IBW), metal blocks were bonded with epoxy resin and tested according to EN 319 (1993).

Statistical processing

Microsoft Excel 2010 for Windows 8 was used for statistical processing. Physical and mechanical parameters were determined on at least eight samples. The mean value, standard deviation, and confidence intervals were calculated. The significance level was 0.05. All samples were tested for homogeneity of dispersion using Fisher's criterion. The significance of the differences between the samples was carried out by the Student's test.

RESULTS AND DISCUSSION

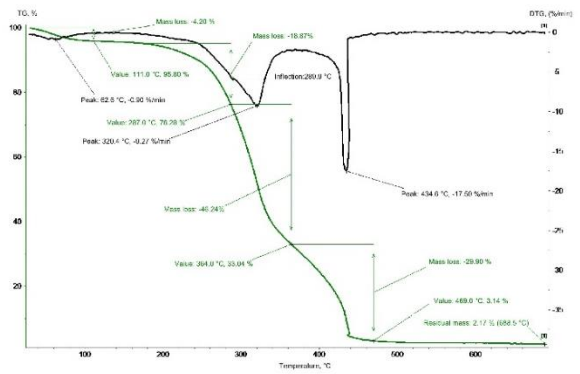
Thermogravimetric analysis

Table 1 presents experimental data obtained using the thermogravimetric method (TG/DTG) for samples of healthy and rotted *Abies sibirica* wood. The TG curve (Fig. 4a) shows four temperature ranges characteristic of the wood (Poletto *et al.,* 2012), at which there is a mass loss of the sample. In the first step of heating the wood from 20 to 115°C, water evaporation (Δm1) occurs. The decrease in sample mass at further temperatures up to 360°C is mainly caused by the thermal decomposition of hemicelluloses and the amorphous part of cellulose (Δm2). Further mass loss is attributed to the thermal decomposition of the crystalline part of cellulose (Δm3) (Nada *et al.,* 2000; Poletto *et al.,* 2010). At the last stage of heating from 406 to 518°C, the preferential thermal decomposition of lignin (Δm4) and combustion of the formed carbon occur.

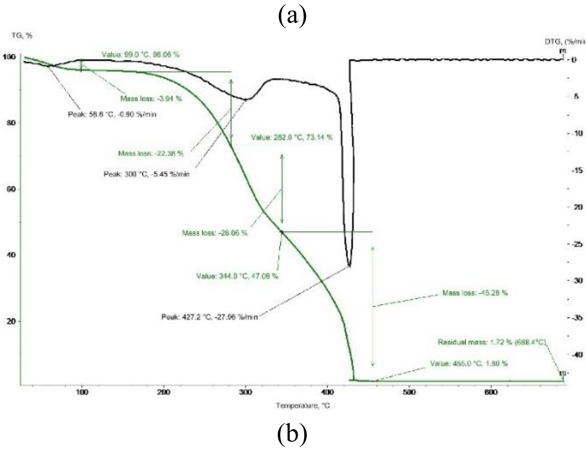
Tab. 1 Stages of thermal decomposition of wood samples.

Infestation stage	Temperature range, °C			
	Mass loss, %			
	Δm_1	Δm_2	Δm_3	Δm_4
Healthy wood	20-114	186-287	287-364	364-469
	4.23	*18.87*	*43.24*	*29.90*
Rotted wood	20-99	170-282	282-344	344-455
	3.94	*22.38*	*26.06*	*45.28*

As a result, it is found (Table 1) that the healthy and rotted wood have similar moisture contents, 4.23% and 3.94%, respectively. Herewith, the content of hemicelluloses and amorphous part of cellulose *Δm2* in the sample of the rotted wood is 18.61 % higher than that of the healthy wood. In addition, the proportion of the crystalline part of cellulose *Δm3* in the rotted wood sample is 65.92 % lower than that of the healthy wood sample. A higher proportion of lignin in the rotted wood (*Δm4*) by 51.43 % was also recorded. It should be noted that the obtained values of mass loss do not reflect the mass content of the main components of the wood but rather their proportion in the tested samples.



(a)



(b)

Fig. 4 TG/DTG thermal decomposition curves of wood.

The DTG curve obtained for healthy fir wood (Fig. 4a) is broadly consistent with data from the following studies (Loskutov *et al.,* 2022; Yang *et al.,* 2006). It is important to note that a relatively high rate of thermal decomposition of *Abies sibirica* wood (at a temperature of 320.4°C – 9.27 % / min and at a temperature of 434.6 °C – 17.50 % / min) in comparison with other conifers is mainly due to its low density. The DTG curve of the rotted wood (Fig. 3b) also exhibits two main peaks in the mass loss rate, at 5.45% /min and –26.96% /min, respectively, corresponding to temperatures of 300°C and 427°C. At almost equal temperatures, the dynamics of thermal decomposition of healthy and rotted wood differ significantly. The thermal decomposition rate of healthy wood compared to rotted wood is 70.09% higher at ~300°C. This is caused by the higher proportion of hemicelluloses, which have low thermal stability and, consequently, a high rate of mass loss. At the same time, at ~ 430°C, the rate of thermal decomposition of the rotted wood is almost twice as high as that of healthy wood. Under these conditions, the processes of thermal decomposition occur. They occur to a greater extent in the crystalline part of cellulose and lignin. However, as noted earlier, at the final stages of brown rot development, the proportion of crystalline cellulose decreases.

Additionally, lignin modification occurs. Such changes create conditions that enhance the dynamics of wood thermal decomposition (Loskutov *et al.,* 2022; Yang *et al.,* 2006; Poletto *et al.,* 2012). Thus, the results obtained confirm that the wood being studied is affected by brown rot.

Fractional composition of wood pulp

The results of the studies on the fractional composition of wood pulp (WPs) during hydrodynamic treatment are shown in Fig. 5. The proportion of the most significant fraction (more than 300 µm) remains practically unchanged during treatment. Herewith, the proportion of the fraction with the size less than 20 µm (bottom) increases. The proportion of all other fractions gradually decreases.

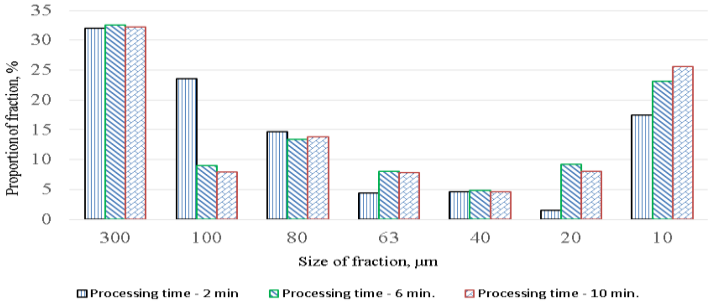
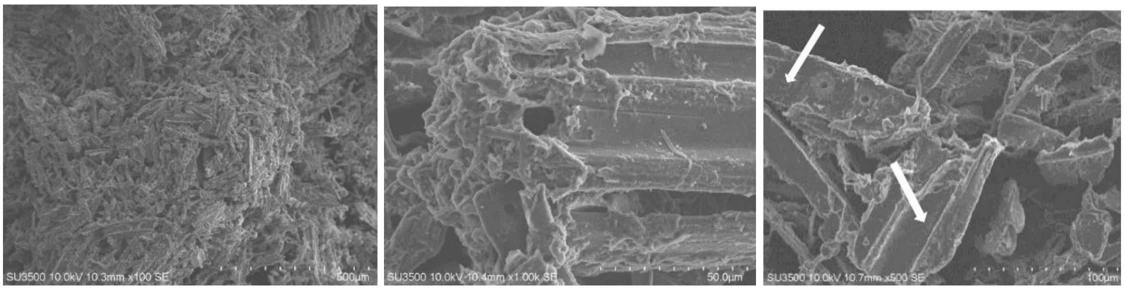


Fig. 5 Change of fractional composition in the process of hydrodynamic treatment.

SEM images

The analysis of the SEM images of WPs obtained through hydrodynamic treatment of biodegraded wood reveals the following. The mass has a homogeneous structure without significant components (Fig. 6a.) The particles retain a cellular structure. It is necessary to pay special attention to the nature of fracture. Previously, we noted that the hydrodynamic treatment of uninfested wood exhibits strongly marked fibrillation, characterized by partial delamination of the end sections of particles into bundles of fibrils, as well as the formation of ribbon-shaped particles (Ermolin et al., 2019). In the particles of infested wood, there are only rudiments of such delaminations (Fig. 6b). The surfaces of many wood particles are smooth, which is characteristic of a brittle fracture (Fig. 5c). This pattern of the destruction of the wood affected by brown rot is most likely due to the decomposition of the strong reinforcing cellulose frame (Eriksson et al., 2012). The images in Figures 6b and 6c clearly show that the inner layer of S3 cell walls is not destroyed by fungi (indicated by arrows). This confirms the data (Liese, 1970, Wilcox, 1968) that this layer is more resistant to brown rot enzymes than the S2 inner layer.



(a) (b) (c)

Fig. 6. SEM images of activated mass at 70 ^{0}SR from rotted Abies sibirica.

The change in the WRV index during the hydrodynamic treatment process is shown in Fig. 7a. For the first 2 minutes of treatment, there is a sharp increase in this index, from 84% to 245%. After that, a decrease in the growth rate is observed. For the next 2 minutes, the index increases by up to 266%. Then it decreases and stabilizes at a level of 220%.

The pattern of 0SR (Fig. 7b) changes significantly during the treatment process. Initially, there is also intensive growth. During the first two minutes, it increases from 14 to 50. Then, the growth rate slows down significantly, and a monotonous increase occurs.

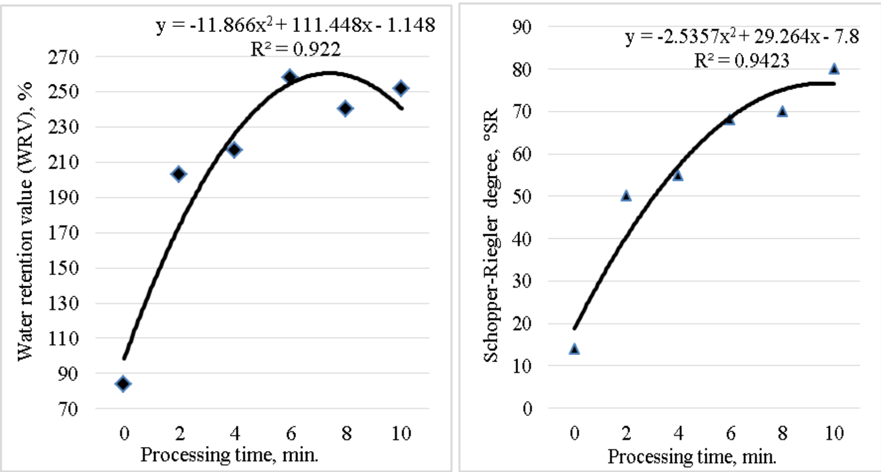


Fig.7 The change of the WRV and ^{0}SR indices in the process of hydrodynamic treatment.

The results of studying the influence of processing time on the mechanical properties of the boards are presented in Fig. 8. The ultimate static bending strength increases with increasing processing time. Up to the sixth minute, it intensifies further. Then the growth slows down. The ultimate tensile strength perpendicular to the plate (Fig.8a) increases monotonically with increasing processing time.

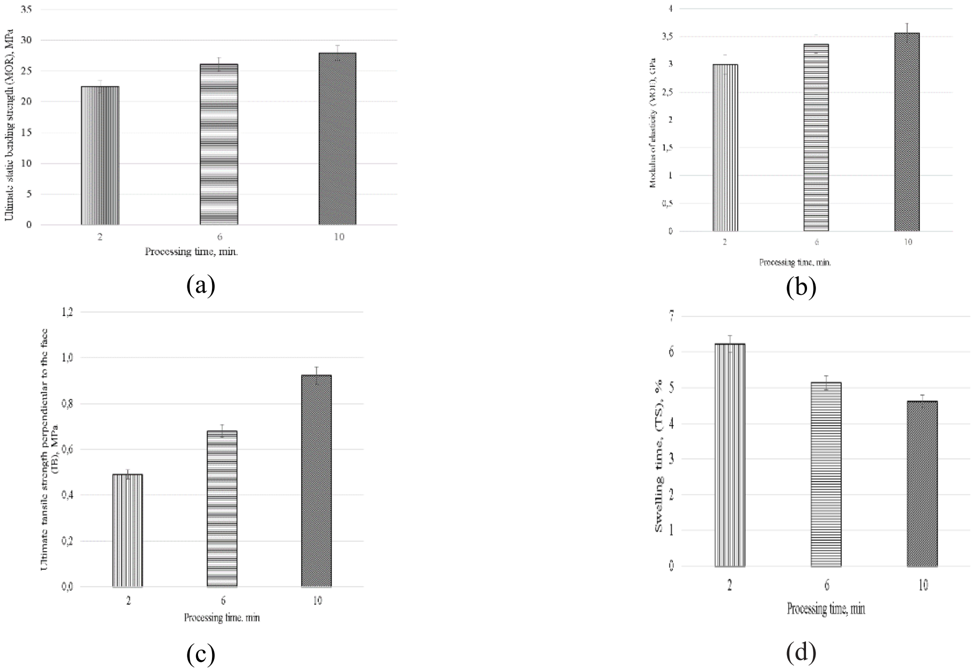


Fig. 8 Properties of boards depending on processing time.

The swelling of the boards (TS) is illustrated in Fig. 8d. The swelling value decreases with increasing processing time. In general, this indicator has low values in the obtained boards. It should be noted that even at the minimum processing time of 2 minutes, the value is much lower than that allowed by EN 622-3:2009 for the MBH.HLS fiber boards.

When studying the moisture resistance of the boards by boiling them in water, it was obtained that the boards did not fracture. After drying, they had a sufficiently high strength, which also depended on the processing time (Fig. 9). The appearance of the samples remained unchanged. During the drying process, the dimensions of the samples practically returned to their original values. The residual swelling does not exceed 0.51 %.

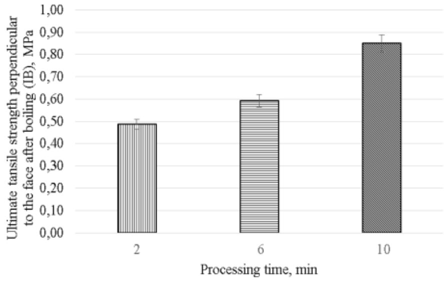


Fig. 9 Ultimate tensile strength perpendicular to the plate after boiling in water for 2 hours.

It follows from the results obtained that during processing, qualitative changes gradually occur in the wood pulp, resulting in improved board properties. The value of 0SR can most adequately assess these changes. As the 0SR increases, the properties of the boards also increase. Therefore, this rapid method can be used to determine the results of hydrodynamic treatment of wood. The patterns of changes in water retention value and board properties do not coincide. In earlier studies using uninfested wood, it was found that this parameter could be used to assess the processing effect (Ermolin *et al.,* 2019). The difference in the patterns is because processing biodegraded wood produces particles of a different shape, specifically shorter particles. Also, the size of fine-fraction particles is significantly smaller. Therefore, it is not reasonable to use the WRV index when processing the rotted wood.

The closest analogue of these boards is MB. Comparing the properties of the boards with the requirements of EN 622-5:2009, the following can be noted. The boards obtained from rotted wood are fully compliant with the requirements for non-load-bearing boards in both dry and wet conditions. The indicator that limits the use of the obtained boards as load-bearing is bending strength. The tensile strength perpendicular to the plate and the swelling in thickness meet the requirements for this type of board.

One of the advantages of boards made of rotted wood is high moisture resistance. The residual tensile strength perpendicular to the plate after boiling in water varies from 0.49 MPa to 0.85 MPa. According to the standard, this index should be not less than 0.15 MPa. Thus, its values are many times higher than the norms. Herewith, as already mentioned, the boards have minimal residual swelling.

Studies have shown that wood with low mechanical properties resulting from brown rot infestation can be used to produce relatively strong and moisture-resistant binder-free boards. The evident reason for the high values of physical and mechanical properties of the boards is a different mechanism of their structural formation. This requires specific analysis.

Since no binder is used, the structure of these boards is formed by interfacial interactions between the wood particles. During the hydrodynamic treatment process, the size of the wood particles decreases, increasing the area of interfacial surfaces and creating prerequisites for the formation of a bonded structure. The nature of the interfacial interactions is determined by the material properties. This wood contains much more lignin with altered properties (Kirk, 1975, Goodell, 2003). In particular, it contains numerous phenolic hydroxyl groups and carboxyl groups. This suggests the possibility of hydrogen bonding in the contact zone of wood particles. In addition, it was observed that pretreating wood under artificial conditions with brown-rot enzymes allows to produce binder-free fibreboards with high mechanical properties. The reason for the increase in adhesion, as suggested by the authors of the works (Felby *et al.,* 2004; Widsten *et al.,* 2002), is that interfibre covalent bonds form between the structural elements of modified lignin in the contact zone during the hot pressing of the boards. The exact process can also occur between the wood particles of the activated mass. In sum, this results in a sufficiently strong board structure. The high moisture resistance of the boards can also be explained by the formation of covalent bonds.

CONCLUSION

Wood affected by brown rot is a significant raw material resource that requires finding ways to utilize it efficiently. The processes that occur in wood under the influence of wood-destroying fungi, along with the deterioration of various properties, create prerequisites for obtaining qualitatively different types of materials. The boards obtained in this work have significantly higher moisture resistance compared to traditional wood boards. Herewith, moisture resistance is achieved not by introducing modifying additives into the boards, as is the case with MB. It is caused by the changes that occur during rotting as a result of hydrodynamic processing. Studying the nature of these changes may reveal some ways to improve board properties.

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