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CHEMICAL COMPOSITION AND MECHANICAL PROPERTIES OF VARIOUS PARTS OF BIRCH WOOD

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The article compares mechanical parameters, group chemical compositions, iron content, lipid compositions before and after saponification, moisture, bulk density in three parts of *Betula pubescens* Ehrh. birch wooden parts: sapwood, false heartwood, false heartwood border. The strength properties of the false heartwood are worse than those of sapwood. The border of the false core is sometimes stronger than that of other parts. Evaluation is carried out in terms of hardness, flexural strength and compression strength. False heartwood, and even more so its border, contain more extractives extracted by ethanol, water, alkali solution, methylene chloride and less iron. The content of other components: cellulose, lignin, other polysaccharides differs, but less significantly. False heartwood border is heavier than the other wooden parts. Moisture content of the various parts decreases in the following order: false heartwood border, false heartwood, sapwood. The composition of the extractives extracted by methylene chloride is different in different parts. The composition is determined by gas-liquid chromatography-mass spectrometry before and after saponification of esters. Differences in sterols are given. An increased content of monoterpene alcohols and arylheptanoids is observed at the border. Differences in the properties of different wooden parts of a tree are explained by different amount and composition of extractives.

Keywords: Betula pubescens Ehrh., mechanical properties, chemical composition, false heartwood, false heartwood border, sapwood.

Introduction

There are not many works that deal with the differences in the chemical composition of heartwood and sapwood of deciduous species, for example [1]. At the same time, the differences in birch are not considered. One of the latest works is the comparison of cork oak parts chemical composition [2]. It turned out that the content of lignin and polysaccharides is higher in sapwood, the main differences being observed in the quantitative and qualitative composition of extractives. There are 1% more water-soluble compounds in heartwood if compared with sapwood. The content of ellagotannins is similar in heartwood and sapwood. If compared with sapwood there are almost twice more compounds soluble in ethanol in false heartwood (5.9 and 13.2% correspondingly). There are many phenolic compounds in alcohol-soluble substances, in heartwood being more than there in sapwood. Both tree wooden parts contain less than 1% of lipophilic compounds. They are represented by fatty acids in both parts. The antioxidant activity of the extracts appears to be almost the same: higher for wood core compounds and comparable to known antioxidants. The obtained results and low toxicity allows to propose the use of heartwood extracts as antioxidants.

The present article focuses on birch and to some extent is similar to the paper mentioned above. Birch is a tree species with irregular heartwood formation, so this darker part of wood is called false heartwood.

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Birch trunks examination collected in the logging area shows that at least 30% of the raw materials have a light brown false heartwood.

It is well known that birch often has false heartwood of different types. This article demonstrates the differences in the parts of birch wood that has light brown false heartwood. It is because of the color and

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not because of mechanical properties that this part of a tree is not used in a number of industries [3]. It is assumed that the formation of false heartwood occurs due to the death of tree knots and is not associated with fungi growth [4–6]. It is also assumed that the light brown false heartwood arose mainly due to external signs at the base of the tree along the longitudinal axis of the trunk. At the same time, the availability of the trunk for pathogens and oxidants through dead branches is discussed [7–9]. The staining caused is also explained by oxidation reactions of phenolic substances catalyzed by various enzymes produced by microbes present in the affected area [9].

False heartwood is formed for 10–12 years after clearing a trunk from knots [10]. But until now there has not been any consensus definite opinion on the causes for false heartwood formation in different species. Our interest in this phenomenon is due to the fact that sometimes when sawing a birch trunk with false heartwood, mechanical properties of wood at the border of false heartwood are clearly different from other parts. The border of false heartwood (BHW) is the outer part of the light brown heartwood, approximately 1.5–2 cm thick.

In addition, wood cuts left in wet state become moldy only in sapwood area, which may be due to the presence of certain protective substances and difference in moisture content. The aim of the work was to clarify the differences between mechanical properties and chemical composition of different parts of a trunk: sapwood (SW), false heartwood (HW) and the border of false heartwood (BHW).

Experimental

Four samples of *Betula pubescens* Ehrh. growing on the border of the Novgorod and Leningrad regions were selected for the analysis.The trees were about 60 years old. The cuts were made at the height of 1.5 meters from the bottom edge of the butt. All four trunks had a HW, the BHW on the cut did not differ in color from the HW. The diameter of the parts was 31 cm, the diameter of the light brown false heartwood was 13 cm.

The absolute moisture content of wood was determined after drying wood to constant weight at a temperature of 103±2 °C. Absolute moisture values was determined as the ratio of moisture removed to the mass of dried wood in percent.

Samples were also cut from parts of wood for mechanical tests (GOST 16483.10-73; GOST 16483.3-84; GOST 16483.17-81 [11–13]). Conditioning of samples was carried out in accordance with GOST 16483.0-89 [14]. A testing machine TsDM-5t.1 was used. A testing machine FM 500 "VEB Thuringer Industriewerk", GDR was also used. 3 samples were used to determine the limit of the compressive strength and limit bending. No bending tests were carried out for BHW, because for bending tests larger specimens are needed than those obtainable from BHW. 12 samples of sapwood and 9 samples of the border of heartwood were used to determine the tangential static Brinell hardness. Before the analysis wood samples were kept at constant temperature and relative humidity until equilibrium humidity was reached. The results obtained were converted into results corresponding to wood with moisture content of 12% according to the formula: $A_{12}=A_{\text{test result}}\cdot(1+0.03\cdot(W-12))$. One sample of wood had no differences in mechanical properties, moisture content, bulk density between (HW) and (BHW) and the chemical composition was not further analyzed in it.

A cylinder with a volume of 10 ml was used to determine the bulk density. Wood samples measuring 0.25–0.5 mm were prepared from corresponding parts of the wood. Moisture of the samples was 6%.

For chemical analysis, samples of 0.25–0.50 mm in size were prepared from 350–400 g wood chips taken from cuts. All work was carried out with air-dry wood. The fraction 0.25–0.50 mm was conditioned to a relative humidity of 5–6% at room temperature. The gap in time between the preparation of the sample and the determination of extractive substances did not exceed 4 weeks. Determination of lignin was carried out by the Klason method [15] after preliminary extraction with methylene chloride and then with 1% hot alkali solution. Extractives soluble in methylene chloride were determined according to standard (TAPPI T204 2011 [16]). Extractives soluble in 1% alkali solution were determined according to standard (TAPPI T 212 om-2012 R2018 [17]). The determination of the amount of substances extracted by ethyl alcohol and diethyl ether was carried out similarly. Acid-soluble lignin was determined in the filtrate as described in [18]. Mixture of nitric acid and ethanol was used for cellulose determination [19]. Before the determination extraction with methylene chloride and 1% hot alkali solution was carried out. The cellulose and lignin contents were calculated in relation to the initial plant material. Pentosans were determined via furfural bromide-bromate semi-micro method (TAPPI T223 2008 [20]). Hydrolysable polysaccharides were determined by the content of reducing substances in the hydrolysate obtained by 3 hours hydrolysis of 2.5 g of a substance with 100 ml of aqueous solution of 2% hydrochloric acid at reflux [19].

Water-soluble substances were determined by extraction in a flask with water at 90 °C (TAPPI T207 2008 [21]). Ash was determined in accordance with the standard (TAPPI T211 2016 [22]). Chromatographic analysis of the substances obtained by extraction with methylene chloride was carried out after preliminary methylation of acids or after preliminary saponification and methylation of saponification products. Extracts 0.5 g and 50 ml of 0.5 N KOH were used for the alkaline hydrolysis reaction. The mixture was boiled for 30 minutes.

The methylation reaction was carried out using diazomethane [23].

The chromatographic analysis was carried out using Agilent G2629A 6850 GC/MSD System (Agilent Technologies, Inc.) with A 5973N Series Mass Selective Detector. The ionizing energy was 70 eV. The temperatures of a separator and an ion source were 280 and 230 °C, respectively. To fractionate samples, Rxi®-5 Sil MS column (30000 × 0.18 mm ID) with a 0.10 μm (low polarity crossbond® silarylene phase; similar to 5% phenyl/95% dimethyl polysiloxane) was used. The thermostat temperature was programmed to increase from 100 to 280 °C at the rate of 5 °C per min. Subsequent exposure at the temperature of 280 °C was 30 minutes. The evaporator temperature was 270 °C. Inlet temperature was 270 °C. Flow rate of the carrier gas (helium) was 1 cm^3 per min. Dosed volume was 0.1 μL. Low molecular weight compounds were identified by comparison with the NIST 05 database. The identification of sterols and triterpenoids was also carried out in comparison with the chromatographic characteristics of the samples available in the laboratory. The standard deviation of the definitions was 0.2 after three definitions, when quantifying the content of components as a percentage of the amount of eluted compounds.

The content of iron was determined using an atomic absorption spectrometer with electrothermal atomization and Zeeman correction of nonselective absorption "MGA-915MD" with preliminary sample preparation using the mineralizer "Minotavr 2" (Lumex 2009). In total, 3 samples of different wooden parts from four trees were analyzed. The analysis was repeated 3 times.

Results and discussion

Differences in wooden parts were observed even in absolute moisture values (Table 1). Differences in moisture content of the heartwood and sapwood corresponded to the expected [24, 25]. High moisture was observed at the border between the zones (BHW) (Table 1).

The bulk weights also differed, respectively. Wood samples 0.25–0.5 mm in size were placed in a 10 ml cylinder: BHW – 2.00 g, SW – 1.64 g and HW – 1.88 g. It means that the BHW was much heavier.

Thus, the BHW appeared to be a denser part of a tree. The strength of this part of the tree was also higher than the strength of HW and SW. The strength of HW was lower than the strength of SW (Table 2).

We assumed that the strength of BHW might be associated with an increased content of metals, primarily iron. But as it turned out, there was more iron and cadmium in the sapwood, through which water flowed from the ground (Table 3). Metals content was minimal in the BHW.

Table 1. Absolute wood moisture content of freshly cut trees (August)

Sample No.	Absolute wood moisture content(out of 3), %		
	SW	BHW	HW
1	59.7	123.0	87.5
2	60.0	116.7	78.3
3	57.6	115.3	70.0
4*	59.9	75.3	75.0

* One sample of wood had no differences in mechanical properties, moisture content, bulk density between (HW) and (BHW)

Table 2. Limit of compression strength along the grain, bending limit and tangential static Brinell hardness of various parts of wood in terms of 12% moisture

Mechanical test	SW	BHW	HW
Limit of compression strength, MPa	50.4±0.1	54.7±0.1*	48.0±1.5
Bending limit, MPa	112±11	–	75±1
Tangential static Brinell hardness, N/mm^2	30.3±0.7	39.4±3.5	26.5±0.9

* samples measuring 2 × 2 × 3 cm were tested, so adjacent zones were inevitably captured

Table 3. Iron and Cadmium content in different parts of birch wood

Parts of wood birch	Content Fe in the sample (mg/kg)	Content Cd in the sample (mg/kg)
HW	75±2	0.05±0.01
SW	132±2	0.06±0.02
BHW	22±1	0.05±0.01

The chemical compositions of the samples were different (Table 4). The greatest differences were observed in the amount of substances extracted with water, ethanol and alkali. It was noted that in the transition zone the activity of enzymes was observed, leading to the formation of not lignin, but other phenolic compounds [24]. Perhaps this was due to the need to carry out the protective function in the HW.

The content of substances soluble in ethanol and water corresponded to that indicated in the article [26]. The lignin and ash contents in sapwood corresponded to the value for *B. pubescens* wood [27, 28]. The high content of substances soluble in hot alkali solution was possibly associated with the dissolution of not only low molecular weight phenolic compounds, but also non-cellulosic polysaccharides. The content of other components: cellulose, pentosanes, alkali-soluble compounds of sapwood was compared with those given for silver birch. The comparison showed similar values, except for pentosans, of which there were more in silver birch (*B.verrucosa*) – 23–26% [29]. Thus, the values for sapwood were the same.

The composition of substances obtained with methylene chloride also differed (Table 5).

Table 4. Chemical composition of the anatomical elements of *Betula pubescens* Ehrh

Index	SW	BHW	HW
	Content, in % to absolutely dry raw material		
Extractive substances extractable:			
Methylene chloride	1.1±0.2	2.4±0.1	1.8±0.2
Diethyl ether	1.4±0.3	1.8±0.1	1.5±0.2
Ethanol	2.1±0.2	8.7±0.4	6.7±0.4
Hot water	2.5±0.2	6.4±0.2	4.3±0.3
1% NaOH solution	18.5±0.3	21.9±0.4	19.8±0.5
Cellulose	42.2±0.4	40.8±0.3	41.6±0.3
Pentosans	18.3±0.2	18.4±0.2	19.2±0.2
Hydrolysable polysaccharides	27.0±0.2	27.8±0.1	27.3±0.1
Lignin	19.2±0.2	20.7±0.1	20.3±0.2
Acid-soluble lignin	0.76±0.01	0.76±0.01	0.76±0.01
Ash content	0.26±0.01	0.34±0.01	0.45±0.01

Table 5. Composition of "volatile" compounds of extracts of different wooden parts of birch, obtained by extraction with methylene chloride

Retention time	Compound	SW		BHW		HW	
		Saponification					
		Before	After	Before	After	Before	After
		Content, in % to the sum of eluted compounds					
1	*2*	*3*	*4*	*5*	*6*	*7*	*8*
3.77	Borneol	Traces	Traces	0.1	0.6	<0.1	0.4
3.91	Terpinen-4-ol	Traces	Traces	<0.1	0.4	<0.1	0.2
3.98	Isomer of α-terpineol	Traces	Traces	<0.1	0.4	<0.1	0.2
4.10	Isomer of α-terpineol	Traces	Traces		0.5	<0.1	0.3
10.06	Dodecanoic acid*	–	0.3	–	0.3	–	0.2
10.51	Nonanedioic acid*	–	1.9	–	0.4	Traces	1.1
14.34	Myristic acid*	0.6	0.8	–	0.9	–	0.7
15.76	Pentadecanoic acid*	1.2	0.3	–	0.3	–	0.1
18.31	Palmitic acid*	11.4	10.9	6.1	10.0	6.6	9.3
20.30	Margaric acid*	0.8	0.9	0.6	1.0	0.7	1.1
21.30	Octadecanol-1 (Steanyl alcohol)	–	2.3	–	1.8	–	0.9
21.37	Linoleic acid*	3.7	2.3	1.4	1.8	2.3	1.5
21.54	Oleic acid*	1.2	1.5	0.2	1.4	0.6	1.5
21.61	6-Octadecenoic acid	0.6	1.5	0.1	0.9	0.3	0.6

End of table 5

1	*2*	*3*	*4*	*5*	*6*	*7*	*8*
22.01	Stearic acid*	9.7	13.4	7.8	9.8	9.4	0.2
23.77	Nonadecanoic acid*	1.0	1.1	1.2	1.7	1.8	2.5
25.03	Eicosanoic acid*	–	0.6	–	0.3	–	1.0
25.46	Arachidic acid*	6.9	9.0	7.8	6.2	10.1	12.4
26.70	Not identified		1.1		0.9		0.7
27.06	Heneicosanoic acid*	2.0	1.9	2.1	1.4	3.1	3.5
28.64	Behenic acid*	5.8	1.3	5.1	3.0	7.8	9.1
30.14	Tricosanoic acid*	1.0	1.3	0.6	0.8	1.1	1.7
31.60	Lignoceric acid*	1.3	1.5	0.9	0.5	1.4	2.0
32.66	Squalene	0.1	0.4	0.1	0.4	<0.1	0.2
34.38	Cerotic acid*	–	0.3	–	Traces	–	0.3
35.76	Stigmastan-3,5-diene M=394	0.1	0.7	0.2	1.6	<0.1	0.5
32.0–37.0	Peaks of arylheptanoids	8 peaks 2.9	2.0	25 peaks 45.8	10.2	12 peaks 8.9	2.9
39.20	β-Sitosterol	1.9	2.3	1.0	3.0	1.2	3.1
39.26	Stigmastanol	–	2.2	–	4.1	–	3.3
40.34	Lupeol	–	1.0	–	2.3	–	1.8
40.54	Stigmast-3,5-diene-7-one M=410	7.7	–	2.3	2.1	15.8	3.0
41.41	24-Methylene-cycloartanol	–	2.1	–	3.0	–	2.2
41.43	Citrastadienol	–	2.0	–	2.8	–	2.3
43.82	Betulinic acid*	–	4.4	–	4.7	–	3.6
46.06	Acetate betulinic acid*	11.2	–	3.8	–	6.4	–
50.50	3-Oxooleanolic acid*	2.5	1.2	0.5	1.2	0.9	1.1

*Results are shown for methyl esters

Before saponification, the ester extracts of various parts of wood differed. One wood sample had no difference between BHW and HW. The difference was that a significant number of compounds not identified by the NIST 11 database were present at the border between heartwood and sapwood (peaks from 32 to 37 minutes). The values of mass ions in mass spectrum were in the range from 300 to 356 m/z. Peaks with masses of 211 and 225 were present in almost all mass spectra of compounds. Ions with such masses are characteristic for arylheptanoids [30]. The structure of the main arylheptanoids of the HW was determined by NMR spectroscopy [31].

Heartwood is reported to be characterized by accumulation of phenolic compounds [32, 33]. Our results confirm these studies, because arylheptanoids are phenolic compounds.

In the heartwood, the amount of stigmast-3,5-diene-7-one was bigger than there in sapwood, which, apparently, accumulated as a result of β-sitosterol transformations. Analysis of data after saponification showed that only sterols β-sitosterol and stigmastanol were contained in free form in all parts of the wood.

Other sterols were found in esters. Stigmast-3,5-diene-7-one was contained only at the BHW and in the HW. The HW and the BHW were distinguished by increased content of monoterpene alcohols, which were not at all characteristic of birch. Perhaps their presence was responsible for the pungent smell of the false heartwood. These results show a significant difference in the composition of the extractive BHW from other parts of the wood.

Conclusions

Sapwood, false heartwood and the border of false heartwood of *Betula pubescens* Ehrh. differ in chemical composition and mechanical properties. Therefore, such wood has signs of juvenile. The mechanical properties of the border of the false heartwood do not always differ from the properties of the false heartwood. Static studies should be done in the future. The reasons for the change in mechanical properties of the border of false heartwood could be additional deposition of extractive substances in the cell cavities during the isolation of sapwood zone. This phenomenon was reported [24]. The increase in the amount of extractives and, first of all, phenolic compounds at the border of the false core may be due to the need to protect the central part of the trunk from damage by wood-destroying fungi. Perhaps, by increasing the density, extractives increase the mechanical properties of the wood pulp at the border of sapwood and false heartwood.

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