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CHOSEN PROPERTIES OF OAK WOOD IMMERSED FOR 6 MONTHS IN THE BALTIC SEA

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SYNOPSIS. Changes of physical and chemical properties, mass loss and susceptibility of oak wood to decay caused by *Basidiomycetes* fungi were assessed after wood samples had been taken out of the sea within the framework of MACHU project. Test samples of oak wood of the dimensions of 250 × 10 × 10 mm were placed in coastal waters of the Baltic Sea at the area of medieval seaport in Puck and in waters of the Gdańsk Bay at the same longitude as Orłowo (near the wreck of Swedish warship *Solen*). The samples taken out of the sea were examined visually and described. Changes in mass of the samples, their bending strength and modulus of elasticity, compression strength along the grain, ability to decay caused by *Trametes versicolor* fungus, content of mineral substances and substances soluble in water, ethanol-benzene mixture and in 1-percent aqueous solution of NaOH, content of cellulose, lignin and pentosanes as well as pH of the wood were investigated. The properties of samples taken out of the sea after 6 months of immersion were compared with the properties of control twin samples of oak wood which had not been immersed. The results obtained so far indicate that noticeable, measurable changes in oak wood immersed in sea occur already in half a year since immersion, so observation of them may be useful for protection and monitoring of underwater archaeological objects.

KEY WORDS: oak wood, Baltic Sea, strength, fungi, decay, chemical composition of wood, archaeological wood

INTRODUCTION

Due to its high density and high tannin content oak heartwood has been quite commonly used under conditions of much exposure to permanent contact with ground, water, atmospheric and biological factors (use class 4 and 5) (PN-EN 335-2 and PN-EN 350-2). The heartwood of oak in respect to rot caused by *Basidiomycetes* fungi belong to II-III classes (durable to moderately durable). It belong to class durable against *Hylotrupes bajulus*, *Anobium punctatum*, *Hesperophanes* *cinnereus* attack, moderately durable against termites, and difficult to impregnation (classes 3-4) (PN-EN 335-1 and -2, PN-EN 350-1 and -2). Because of its relatively high durability it is often used in marine environment as construction elements. In former time, oak wood was commonly used in building ships.

The sea water together with certain micro- and macro organisms such as bacteria, marine borers, fungi, algae, and insects may change the properties of wood totally or partially immersed in sea water. Wood in such conditions is exposed to many physical and biological factors causing its depreciation and degradation resulting in various stages of destruction (Kollmann 1936, Krauss and Raczkowski 1985, Archaeological wood 1990, The General... 2008, Stan i perspektywy... 2009). The reason for it consists of such phenomena as existence of a great number of overlapping factors that may have an effect on wood submerged in sea and diversity of resistance features of various wood species and even parts (sapwood, heartwood) of the same wood species. The rate of the wood destruction depends among other on the environmental conditions at the seabed resulting preservation or destroying an object located there. The following external factors are of importance: temperature and movement of water (currents), its salinity and chemical composition, oxygen content, organisms that occur in this environment and the depth to which the object is buried in the marine sediments (Cartwright and Findlay 1951, Krauss and Raczkowski 1985, Fengel and Wegener 1989, Zabel and Morell 1992, Björdal and Nilsson 2008, Sivrikaya et al. 2008, Sen et al. 2009). The activity of marine environment may lead to reduction of strength properties and changes in susceptibility of different materials to different destroying microorganisms attack. The grade of wood salinity by e.g. rock-salt (NaCl under conditions in salt mine) may also influence susceptibility of wood to fungi attack (Witomski 2008, Fojutowski et al. 2009).

Development of underwater archaeology was the reason to discover a lot of shipwrecks on the bed of seas and oceans. There are about 9 000 wrecks of different age in the Baltic Sea. Most of them are of wooden structure. Waterlogged shipwrecks are a rich source of knowledge about history and life of past generations. This valuable cultural heritage should be protected and saved for future. To achieve this purpose numerous investigations in various disciplines are needed. The good examples of international activity in this direction are two projects, undertaken by the Baltic countries, funded by the European Union Culture 2000 Programme:

- A shipwreck research project "Monitoring, Safeguarding and Visualizing North-European Shipwreck Sites" (MoSS 2005),
- A Maritime research project "Managing Cultural Heritage Underwater" (MACHU 2008-2010).

Within the framework of MACHU Project, Wood Technology Institute in cooperation with Polish Maritime Museum begins to work on the influence of the underwater marine environment on samples of nowadays sawn oak wood placed in proximity of archeological objects (Fojutowski et al. 2010, Pomian et al. 2010). The investigation of wood samples put close to the underwater archeological objects may not only give the information about changes in wood properties, but also the samples may play the role of bioindicators of any kind of change in the marine environment in area where they are located on the seabed. The aim of the research was to compare chosen properties of oak wood samples submerged in the Baltic Sea for 6 months with the properties of control oak wood samples not submerged in the sea water to determine changes of wood properties caused by submergence of wood in the Baltic Sea.

EXPERIMENTAL AND METHODS

The wood samples were put in investigations sites located in the coastal waters of the Baltic Sea at the area of the medieval seaport in Puck at a depth of about 2 meters and in the waters of the Gdańsk Bay at the longitude of Orłowo (near the wreck of the Swedish warship Solen) at a depth of 14 meters (Pomian et al. 2006) (Fig. 1).

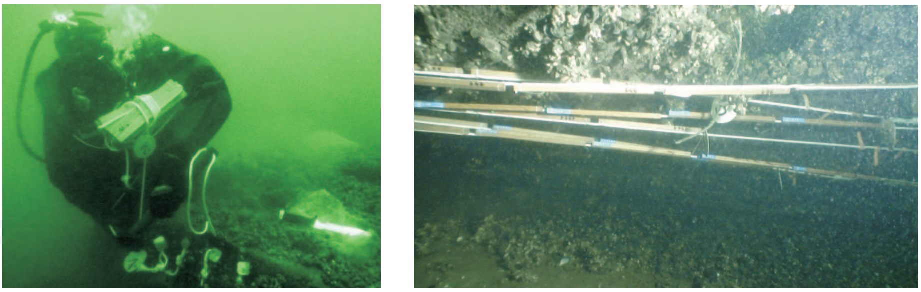

Fig. 1. The immersing of wood samples in the Baltic Sea in Puck and Orłowo sites

The research consisted of visual examination and description (documentation – photos) of the samples condition after 6 month from immersion in Baltic water, determination of sea organisms colonizing wood samples surface, investigation of changes in mass of the samples, their bending strength and modulus of elasticity, compression strength along the grain, ability to decay caused by *Trametes versicolor* fungus, content of mineral substances (ash) and substances soluble in water, ethanol-benzene mixture and in 1-percent aqueous solution of NaOH, content of cellulose, lignin and pentosanes, and wood acidity (pH). The properties of samples taken out of the sea after 6 months of immersion were compared with the properties of control twin samples of oak wood which had not been immersed.

MATERIALS

The heartwood elements 250 × 10 × 10 mm (L-T-R) of European oak wood (*Quercus robur* L.) submerged in Baltic Sea water for 6 months on the two above mentioned underwater investigations sites and the twin control wood elements, were main objects of analysis. We chose to test the twin samples in order to minimize the influence of natural differences of wood structure and properties on the results of the research. The samples were made of the selected oak flooring strip. Individual, conditioned, flooring strips of 250 × 50 × 10 mm were cut along the grain on 4 samples (sticks) of 250 × 10 × 10 mm, one of the central as a control, and the three remaining once for place in the sea water on investigations sites, each one for the different period of time (6 months, 1 year, 2 years). The sticks were marked with consecutive numbers and collected in sets of 45 items fastened to supporting lines. They were mounted on both underwater sites. Test samples were removed from investigations sites after 6 month from the date of their submerging in sea water, then described, weighed, documented (photos), cleaned from biofilm coating the surface of samples and other marine organisms, conditioned to the equilibrium moisture content under nominal conditions 20°C/65% RH and finally subjected to physical, chemical and biological examinations.

METHODS

Physical and mechanical properties

There were tested:

a) mass loss of samples resulting by immersion in the Sea, as percentage of their initial dry mass at 103 ±2°C – oven-dry method (PN-EN 13 183-1) on 10 samples for set (set = control wood or wood from Puck or wood from Orłowo),
b) wood density (PN-77/D-04 101) – 10 samples, moisture content in wood after taking out of the sea (10 samples for marine samples set) and equilibrium moisture content in wood under standard climate conditions 20°C/65% (determined by oven-dry method (PN-EN 13 183-1) on all samples under strength tests),
c) bending strength and modulus of elasticity at three-point bending (bearing spacing 120 mm – 16 samples for set (PN-77/D-04 103 and PN-77/D-04 117),
d) parallel to grain compression strength (PN-77/D-04 102) – 32 samples for set.

The oak samples (250 × 10 × 10 mm) removed from the Sea were used directly for tests of mass loss and moisture content of wood resulting from immersion of wood in sea water. The samples (180 × 10 × 10 mm) for tests of bending strength and modulus of elasticity were prepared from the removed from the sea water samples (250 × 10 × 10 mm) and from control samples (250 × 10 × 10 mm) by cutting off two 15 mm long segments from each end of the samples (250 × 10 × 10 mm). The 2 cut off outer segments were rejected, while both inner 15 × 10 × 10 mm long segments were used in tests of compression strength parallel to grain. The strength tests were done on wood samples at the state of equilibrium moisture content in 20°C/65% conditions. The statistic significance of stated changes was estimated according to standard method (PN-N-01052-03) at the α 0,05 level of significance.

Chemical composition of wood

For determination of chemical composition of wood ten orderly numbered marine samples – small stick of 250 × 10 × 10 mm dimensions (of the sets of 45 samples) and ten twin control samples were taken. The samples were dried in room conditions to constant mass, cut, milled and sifted (screened). The wood grains fraction of 0.5-1.0 mm was used for chemical analysis and fraction of 0.25-0.5 mm was used for pH determination.

Content of main and additional components of wood were determined with methods according to PROSIŃSKI (1984):

– moisture content by oven-dry method,
– content of extraction substances by Soxhlet method (ethanol:benzene 1:1),
– content of substances soluble in cold and hot water,
– content of substances soluble in 1-percent NaOH aqueous solution,
– content of Seifert cellulose,
– content of Klason lignin,
– content of pentosans by Tollens method,
– ash content (in the temperature of 600°C),
– pH determination (Gray method).

Resistance of wood to *Basidiomycetes* fungi

The wood resistance to the rot fungi was tested using a method whose principles are presented in EN Standard [EN113]. For 16 weeks wood samples were exposed to the action of a pure culture of *Trametes versicolor* (Linnaeus ex Fries) Pilat (CTB 863 A) fungus causing white rot of wood. It is standard fungus test for white rot examinations in EN standards concerning the determination of wood and wood-based panels resistance to the *Basidiomycetes* attack and to the toxic values determination of wood preservatives too. Oak wood specimens of the dimensions of 50 × 10 × 10 mm were used as a main test and control materials (two test samples made of wood previously submerged in the sea water = marine samples and one control sample placed between them in one Kolle flask), check samples (3 marine or 3 control samples in one Kolle flask). Oak wood test samples 50 × 10 × 10 mm were cut out from the removed from the sea water samples (250 × 10 × 10 mm) and from control samples (250 × 10 × 10 mm) after rejecting the 15 mm long segments from both ends of samples 250 × 10 × 10 mm. It was main material in examinations consisting of the loss of wood mass determination as criterion of decay caused by white rot fungus. The check samples i.e. kept in a sterile (uninoculated) culture medium in conditions similar to the conditions in which samples were exposed to fungi, were used for calculation of the correction values of mass loss by changes caused by all factors but not fungus. Oak wood control samples 50 × 10 × 10 mm were also used together with control beech wood (*Fagus sylvatica* L.) sapwood samples 50 × 10 × 10 mm (one beech wood sample between two oak samples in one Kolle flask) for fungal activity control in relation to small i.e. 50 × 10 × 10 mm oak samples. Beech wood (*Fagus sylvatica* L.) samples 50 × 25 × 15 mm (two samples in one Kolle flask) were used as standard fungal activity control. The properties of beech control wood were in conformity with standard (PN-EN 113) requirements. Before the test, the samples were sterilized with steam in an autoclave (20 min, 121°C). The mean density of oak wood was: 660 ±20 kg/m^3 and beech wood: 640 ±20 kg/m^3. Mycological tests were carried out at the temperature of 22 ±1°C and 70 ±5% RH i.e. in the conditions appropriate to the decay processes of lignocelullosic material by the fungus used.

RESULTS AND DISCUSSION

Visual examination

The test samples directly after removing from the Baltic Sea were of dark colour and water-soaked in comparison with control samples (Table 1). The samples from Orłowo site were darker than those from the Puck site. The samples removed from water were still darker than control one after drying and seasoning in normal conditions 20°C/65% (Fig. 2), however the difference in colour became not so distinct. The surfaces of the samples were slippery and covered partly with gray-greenish biofilm coating. The edges were mechanically slightly rounded. The traces

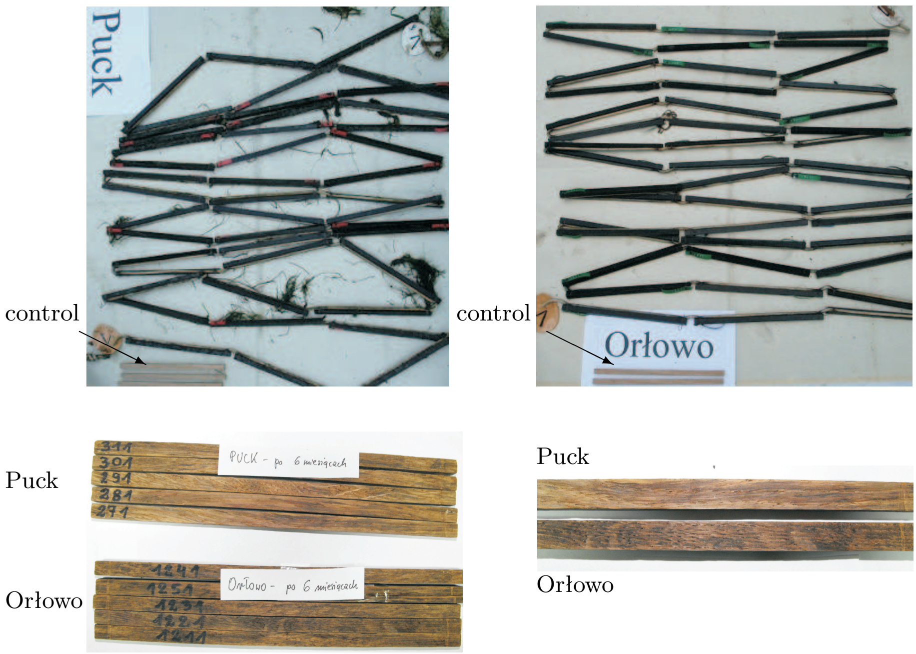

Fig. 2. The wood samples after remaining immersed in the Baltic Sea for 6 months

of seaweed were noticed on the samples from the Puck site. They did not appear on samples from Orłowo (Fig. 2). The early symptoms of *Balanus improvisus* and *Mytilus trossulus* were present on the surface of samples placed in waters in the Orłowo region.

Physical and mechanical properties

The mean mass loss of the oak wood samples from the investigation site Puck remaining submerged in the Baltic Sea for 6 months was 6.8% but mean mass loss of oak wood samples from the investigation site Orłowo was not so high coming to 3.8% (Table 1). The determined mass loss may be assessed as rather large taking into account short period of wood samples remaining in water. The moisture content of wood resulting from submerging the wood in sea water increased to over 100% (Table 1). It could be expected because of small thickness of wood samples (cross section 10 × 10 mm).

Table 1. Mass loss and moisture content of oak wood samples after removing from underwater investigations sites Puck and Orłowo where the samples were staying for 6 months

Investigation site	Value	Mass loss of samples as result of staying immersed in Baltic Sea [%]	Moisture content after removing samples from water [%]
Puck	minimum	6.3	84.9
	mean	6.8	110.9
	maximum	7.7	143.0
Orłowo	minimum	3.3	82.8
	mean	3.8	115.0
	maximum	4.3	145.2

Equilibrium moisture content of wood (Table 2) increased very distinctly from around 9% for control wood to the level of about 13% for wood, which was submerged in sea water. It indicated a strong increase of wood hygroscopicity. The changes in density of wood tested for bending strength (Table 2) in comparison to control wood were not very high – amounted to minus 5% for wood samples of the Puck marine site to plus 2% for samples of Orłowo. Bending strength of wood decreased, for wood of the Puck marine site even to the level of 77% and of 87% of the value characterising natural wood, for wood of Orłowo. MoE at bending for wood samples of Puck were also smaller than this of natural wood rising 74% and 93% respectively. Parallel to grain compression strength of the tested wood decreased to the level 77% – Puck and 85% – Orłowo, similar to the changes of bending strength.

The stated changes were statistically significant on the α 0,05 level of significance, apart from density changes – Orłowo region.

Table 2. The main physical and mechanical properties of oak wood remaining immersed in the Baltic Sea for 6 months on the investigations sites Puck and Orłowo

Investigation site	Tested property	Statistical values	Control	Immersed in water for 6 months
Puck	equilibrium moisture content for 20°C/65%RH [%]	x_{mean}	9.1	12.8
		x_{min}	8.8	12.5
		x_{max}	9.4	13.3
		s	0.17	0.22
Orłowo		x_{mean}	9.0	13.0
		x_{min}	8.6	12.5
		x_{max}	9.5	13.5
		s	0.24	0.28
Puck	density by bending strength γ_{gs} [kg/m^3]	x_{mean}	680	644
		x_{min}	593	521
		x_{max}	767	744
		s	53	63
Orłowo		x_{mean}	668	679
		x_{min}	553	555
		x_{max}	801	799
		s	63	66
Puck	bending strength R_{gs} [N/mm^2]	x_{mean}	119.3	91.4
		x_{min}	80.5	51.3
		x_{max}	161.9	123.9
		s	26.4	18.8
Orłowo		x_{mean}	114.1	99.7
		x_{min}	58.3	56.1
		x_{max}	164.3	125.6
		s	29.0	21.2
Puck	modulus of elasticity by bending E_{gs} [N/mm^2]	x_{mean}	9 757	7 235
		x_{min}	7 767	3 838
		x_{max}	12 750	10 200
		s	1 504	1 437
Orłowo		x_{mean}	9 325	8 721
		x_{min}	5 317	4 620
		x_{max}	12 460	11 900
		s	2 025	2 019
Puck	compression along the grain R_{sc} [N/mm^2]	x_{mean}	62.2	47.6
		x_{min}	52.8	30.7
		x_{max}	80.2	58.3
		s	7.3	6.9
Orłowo		x_{mean}	61.7	52.3
		x_{min}	41.0	33.9
		x_{max}	79.8	64.8
		s	10.2	8.5

x_{mean} – mean value, x_{min} – minimum, x_{max} – maximum, s – standard deviation.

Chemical composition of wood

It was found that oak wood immersion in water of the Baltic Sea for the period of 0.5 year caused changes in the amount of substances soluble in water and organic solutions as well as in substances soluble in 1-% NaOH aqueous solution. Substances soluble in ethanol-benzene mixture decreased from 3.13% for control wood to 1.85% for immersed wood in Puck and from 3.37% for control wood to 2.16% for immersed wood in Orłowo. The amount of substances soluble in cold water decreased about 1% (from ~6% to ~5%) and in hot water about 2% (from ~11% to ~9%) for the samples immersed in both sites (positions). Substances soluble in 1%-NaOH aqueous solution decreased by about 2% during half a year of oak samples immersion in the Baltic Sea. The amount of main components of wood (cellulose and lignin), as well as pentosanes, did not significantly change during half a year immersion of the tested oak wood samples in the Baltic Sea.

Table 3. Changes of chemical composition in oak wood samples immersed in the Baltic Sea water test areas in Puck and Orłowo

Tested property	Oak wood samples			
	Puck		Orłowo	
	control (not immersed)	immersed for 6 months	control (not immersed)	immersed for 6 months
	% of oven-dry wood			
Moisture content	7.99	9.53	7.65	9.66
Substances soluble in:				
Ethanol-benzene	3.13	1.85	3.37	2.16
Cold water	6.08	5.11	5.69	5.08
Hot water	10.67	8.87	10.45	8.81
1% NaOH	23.38	21.49	23.65	21.75
Ash content	0.22	1.27	0.22	1.26
Seifert's cellulose	37.87	37.29	37.85	37.21
Klason lignin	25.70	25.94	26.00	26.31
Pentosanes	20.59	21.07	21.17	21.73
pH	3.95	6.00	3.98	5.84

However, the significant differences were observed in pH value and in ash content (mineral substances). Oak samples after 0.5 year immersion in sea water contained 5.8 times more mineral substances (1.27% in Puck and 1.26% in Orłowo) in comparison with control samples (0.22%). It is a well known effect of elements migration from soil and water to wood deposited in natural conditions especially for old archaeological objects (Archaeological wood 1990, Wróblewska 1999, 2007, Zborowska et al. 2004, 2009).

The acidity of immersed oak samples increased during half a year from pH = 3.95 to pH = 6.00 in Puck and from pH = 3.98 to pH = 5.84 in Orłowo. Increase in pH value of waterlogged archaeological oak wood recovered after hundreds of years was observed in former work by Wróblewska (2007). Analysis of oak samples immersed for half a year in the Baltic Sea revealed that processes of pH changes, as well as mineralization of wood, start in such a short time as half a year.

Resistance of wood to *Basidiomycetes* fungi

The results of the loss in mass of wood caused by *Basidiomycetes* fungus and the external appearance of wood samples after fungus activity are shown in Table 4 and Figure 3. The mass loss of beech wood caused by *Trametes versicolor* (*Tv*) fungus

Table 4. The test results of resistance to 16 weeks exposition to *Trametes versicolor* fungus attack of oak wood samples immersed previously for 6 month in the Baltic Sea

Samples exposed to fungus attack	Orłowo	Puck
	Trametes versicolor	
	mass loss of samples [%]	
Marine – oak samples 50 × 10 × 10 mm (from specimens immersed for 6 months in the Baltic Sea)	12,6	17,7
Control to marine – oak samples 50 × 10 × 10 mm tested together with marine samples in the same Kolle flask	0,6	0,6
Virulence control – oak samples 50 × 10 × 10 mm	0,2	
Virulence control – beech samples 50 × 10 × 10 mm	29,2	
Virulence control – beech samples 50 × 25 × 15 mm	13,8	
Samples not exposed to fungus attack	samples on sterile nutrient	
Check to marine samples – oak samples 50 × 10 × 10 mm (from specimens immersed for 6 months in the Baltic Sea)	0,13	0,23
Check to control – oak samples 50 × 10 × 10 mm	0,12	0,27

3 samples attacked by *Tv*, control oak between marine

control beech wood between control oak wood samples

control oak wood between marine oak wood samples

Fig. 3. The samples after 16 weeks exposition to *Trametes versicolor* fungus

(Table 4) shows that activity of the fungus seemed to be not very great for standard size 50 × 25 × 15 mm samples because the mass losses reached near 14, but for 50 × 10 × 10 mm wood samples, which were tested together with control oak wood samples, the mass loss amounted about 29%. The control samples 50 × 10 × 10 mm made of oak wood undergo the fungus activity, in one Kolle flask together with beech wood, was decayed only to a very small extent – mass losses 0.2%. The result indicates and confirms the high resistance of oak wood heartwood to rot caused by *Basidiomycetes* fungi (Cartwright and Findlay 1951, EN 350-2, Ważny 1959, 1960). In marine oak wood samples (previously submerged in sea water) however, *Tv* fungus caused greater mass losses, 17.7% at the Puck site and 12.6% at Orłowo site respectively. These data imply the possible increase of oak wood susceptibility to rot caused by *Tv* fungus as result of submerging it in the sea water. Unexpected result is at the same time the increase of decay grade of control oak wood by the condition of rot testing (from 0.2% in test in one Kolle flask with beech wood to the level of about 0.6% in test in one Kolle flask with marine oak wood samples). It seems to indicate the peculiar kind of stimulation of the fungus action or preference of fungus in decay control beech wood than control oak when tested together in one Kolle flask. Similar phenomenon was stated during the tests of the same sets of oak samples to *Coniophora puteana* (*Cp*) fungus (Fojutowski et al. 2010), although other present unknown interactions and circumstances related with the material differences of the tested wood samples should be considered. Ważny (1959) using *Cp* and *Serpula lacrymans* fungi during 4 month tests, in comparison to our results, has got ca. 5% mass losses of oak wood samples 20 × 20 × 20 mm. The matter requires further investigations. The state of wood attacked by *Tv* fungus presented in Figure 4 (strong growth of the fungus) indicates that in favourable conditions further progress in wood decay is possible. On significantly reduced resistance of oak wood against *T. versicolor* after its exposition to sea water may affect leaching from wood of tannins and other chemical compounds and/or increase of microelements introduced to the wood with sea water. Synergistic effect of sea microorganisms or their secretions may be taken into consideration too.

CONCLUSIONS

It was found that wood immersion in the water of the Baltic Sea for a period of half a year may give the following results in the 10-milimetre-thick zone of wood: decrease in bending strength, in modulus of elasticity and in compression strength along the grain of wood (all of which are connected with mass loss), increase in moisture content and hygroscopicity, significant increase in susceptibility of wood to decay caused by *Basidiomycetes* fungi – white rot fungi – e.g. *Trametes versicolor*, clear change of wood colour and surface structure, increase in content of mineral substances, increase in pH value, decrease in content of substances soluble in water, ethanol-benzene mixture and in 1-percent aqueous solution of NaOH.

The reduction of resistance of oak wood to *T. versicolor* after its exposition in sea water requires further investigation in order to obtain good explanation taking into account such phenomena as e.g. leaching of some chemical constituents (tannins, waxes, dyes, polysaccharides etc.) and/or increase in microelements introduced into wood with sea water, synergistic effect of sea microorganisms or their secretions. Some changes of wood properties may depend on depth of wood immersion in sea water.

Balanus improvisus and *Mytilus trossulus* may growth on the surface of samples placed in sea waters.

The results obtained so far indicate that noticeable, measurable changes in oak wood immersed in sea occur already in half a year since immersion starts, so observation of them may be useful for protection and monitoring of underwater archaeological objects.

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