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RESEARCH ON DIMENSIONAL STABILITY IN WATERLOGGED ARCHAEOLOGICAL WOOD DRIED IN A NON-COOLED VACUUM CHAMBER CONNECTED TO A LABORATORY FREEZE-DRYER

The article presents changes in the dimensions of well-preserved, waterlogged archaeological oak and pine wood, untreated and treated with PEG 300, after drying in the air and in vacuum conditions. The effectiveness of wood conservation was evaluated on the basis of wood shrinkages in tangential and radial directions, cross-section shrinkages and anti-shrink efficiencies (ASE). The changes in the dimensions of the oak wood samples treated with a 25% solution of PEG 300 and dried in vacuum conditions were distinctly lower than the results obtained after the drying of the wood in the air. The shrinkage in the treated and vacuum-dried pine wood was lower than the shrinkage in the oak wood, but it did not differ much from the results obtained in the case of the treated wood, dried in the air.

Keywords: waterlogged archaeological wood, shrinkage, conservation, dimensional stabilisation, PEG 300, vacuum freeze-drying

Introduction

Freeze-drying is one of the most efficient methods of conserving waterlogged archaeological wood [Grattan 1989; Ambrose 1990; Unger, Schniewind, Unger 2001]. Before the freezing of an archaeological object and starting its drying in vacuum conditions, a part of the water content in the wood is replaced with polyethylene glycols (PEG) [Cook, Grattan 1985; Ambrose 1990; Jensen, Schnell 2005]. The quantity of polymer taken up by the wood to guarantee the optimum dimensional stabilisation of the treated object, depends on the type and degree of

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degradation of the dried wood. In general, the more decomposed the material, the more polyglycol it requires, and if a mixture of polyglycols is applied, a lower participation of polymer with a lower molecular weight and a bigger concentration of a compound with a greater degree of polymerisation is necessary. In practice, various types of polyethylene glycols and various concentrations of wood impregnating solutions are used before freezing and vacuum freeze-drying [Watson 1997; Chaumat et al. 2002; Schindelholtz et al. 2009]. Cook and Grattan [1991] developed a computer program, enabling the calculation of the concentration of a mixture of PEG 200 and PEG 3350, depending on the degree of degradation of the treated wood.

The research undertaken in the 21st century has proved that satisfactory results of conservation of freeze-dried wood can also depend on the temperature of freezing of treated archaeological material [Jensen, Schnell 2005; Jensen, Jensen 2006]. Taking the above into consideration, it is necessary while drying to keep the temperature of the ice condenser below -45°C, the pressure below 15 Pa, and the temperature of the dried material below the eutectic temperature of the introduced impregnating agent [Jensen et al. 2009]. Not only does this last condition require the appropriate freezing of the conserved wood, but the lower temperature of the wood also has to be maintained throughout the whole process of drying. In this case, it is necessary to equip the vacuum chamber in which the freeze-drying is performed, with a cooling system, responsible for the constant cooling of the archaeological object in order to keep its low temperature. This is related to the necessity of applying an installation, prepared especially for drying archaeological materials treated with polyglycols. Such an installation differs considerably from the more common equipment produced for the freeze-drying of food and pharmaceutical products.

In many conservation centres, waterlogged archaeological wood was earlier dried at the above-mentioned values of air pressure and temperature of ice condenser. At the same time, the frozen material was often put in chambers, which were not equipped with an installation enabling it to maintain the low temperature of the treated wood. In the last years, in Polish conservation workshops, freeze-dryers of various types have been applied in order to dry various archaeological objects (e.g. wood, leather or paper). Among them, one can find small laboratory equipment, manufactured in order to dry small samples of food and pharmaceutical products. These freeze-dryers are usually equipped with a small acrylic drying chamber, assembled over the ice condenser's chamber or a separate drying chamber connected to it. Usually, these containers are not equipped with a cooling installation, maintain the low temperature of the frozen object. According to Jensen and Schnell [2005], performing the process of freeze-drying in a non-cooled chamber in the case of archaeological wood treated with polyglycols, can lead to the collapse of cell walls – especially in the case of objects with a higher degree of decomposition. However, it is known, that the phenomenon of collapse does

not appear in the case of well-preserved archaeological wood, and any changes in its dimensions appear as late as during the removal of bound water. Therefore, it would be interesting to compare the dimensional changes in slightly degraded archaeological wood, dried in vacuum conditions at various temperatures of a dried material. Previous research on wood with a slight degree of decomposition showed its high dimensional stability after impregnation with polyglycols, freezing it outside the freeze-drying set and drying in the conditions of a non-cooled vacuum chamber [Babiński 2007a, 2007b; Babiński, Zborowska, Prądzyński 2011]. It is also worth mentioning, that even better results were achieved in relation to wood with a considerably worse state of preservation [Babiński 2009, 2011]. This prompted the author to do some research on dimensional changes in waterlogged archaeological wood treated with polyethylene glycol and vacuum-dried at various temperature conditions.

The research was aimed at determining the dimensional stability in well-preserved, waterlogged archaeological oak and pine wood, untreated and treated with polyethylene glycol, which underwent freeze-drying in a non-cooled vacuum chamber, connected to a laboratory freeze-dryer, then comparing the achieved results with dimensional changes in unfrozen wood, dried in the same conditions, as well as the wood dried in the air.

Material and methods

The research was undertaken on waterlogged wood drawn from historical objects that were found below groundwater level at terrestrial archaeological sites. Samples with dimensions 50 × 50 × 10 mm (T × R × L) were cut out from the oak (*Quercus* sp.) heartwood of a house beam (13th c.) in Szczecin and from the Scots pine (*Pinus sylvestris* L.) heartwood of a construction element of a building (17th c.) from Gdańsk. The wood used during the research was characterised on the basis of selected macroscopic features (width of annual rings, percentage of latewood) and physical properties (maximum moisture content, basic density, total tangential and radial shrinkage).

The research determined the dimensional changes in untreated wood, dried in the air and in vacuum conditions, wood treated with a 25% water solution of polyethylene glycol 300 (PEG 300) and dried in the air and in vacuum conditions, and wood treated with a 45% water solution of PEG 300 and dried in the air. The polyethylene glycol type and concentrations of the solutions were determined on the basis of the results of previous research [Babiński 2005, 2007a, 2007b]. The list of tested variants of treatment and drying of wood is presented in table 1.

The waterlogged archaeological wood, stabilised according to variants 4-8 was treated in water solutions of PEG 300 with concentrations: 5, 10, 15 and 20%, for 4 weeks in each solution, and then, for 8 weeks in a 25% solution.

Table 1. Variants of dimensional stabilisation of the examined archaeological wood
Tabela 1. Warianty stabilizacji wymiarowej badanego drewna archeologicznego

Variant of stabilisation *Wariant stabilizacji*		Impregnant *Impregnat*	Freezing *Zamrażanie*	Drying *Suszenie*
No. *Nr*	Designation *Oznaczenie*			
1	W-AD	- (water) *(woda)*	-	air (laboratory) *powietrze (laboratorium)*
2	W-FD	- (water) *(woda)*	+	vacuum (freeze-drying in the drying chamber) *próżnia (liofilizacja w komorze suszenia)*
3	W-FD/i	- (water) *(woda)*	+	vacuum (freeze-drying in the drying chamber, interrupted) *próżnia (liofilizacja w komorze suszenia, przerywana)*
4	25PEG-V	25% PEG 300	-	vacuum (drying chamber) *próżnia (komora suszenia)*
5	25PEG-FD	25% PEG 300	+	vacuum (freeze-drying in the drying chamber) *próżnia (liofilizacja w komorze suszenia)*
6	25PEG-FD/i	25% PEG 300	+	vacuum (freeze-drying in the drying chamber, interrupted) *próżnia (liofilizacja w komorze suszenia, przerywana)*
7	25PEG-FD/c	25% PEG 300	+	vacuum (freeze-drying in the ice-condenser chamber) *próżnia (liofilizacja w komorze kondensatora lodu)*
8	25PEG-AD	25% PEG 300	-	air (laboratory) *powietrze (laboratorium)*
9	45PEG-AD	45% PEG 300	-	air (laboratory) *powietrze (laboratorium)*

The last series of samples (variant 9) was additionally treated for 4 weeks in a 35% solution and for 8 weeks in a 45% solution. Concentrations were presented in volume percent (v/v), and while calculating and preparing them, the water content in the wood was also taken into consideration. In each variant of stabilisation, 4 samples of oak wood and 4 samples of pine wood were used, with four pins inserted in the cross-section of the wood. The uptake of polyglycol into the wood was presented in the percentage of oven-dry mass of wood.

The samples dried in the air (variant 1, 8 and 9) remained in the laboratory at a relative humidity of ca. 45–55% and a temperature of about 15–20°C. Before the drying of the wood in vacuum conditions, the samples were frozen (apart from variant 4) for 10 days at a temperature of –27°C. Freeze-drying was performed with the use of a set consisting of a laboratory freeze-dryer Alpha 1–4 (Christ), two-stage vacuum pump Duo 020 (Pfeiffer) and a non-cooled drying chamber with a capacity of 0.3 m^3. The samples assigned for drying in vacuum conditions were put in the drying chamber (variants 2–6) or in the freeze-dryer in ice con-

denser chamber (variant 7). The samples dried with interruptions (variant 3 and 6) were put into the non-cooled vacuum chamber for 5 hours of each day of the process, and then, they were left in the freezer (–27°C) for the next 19 hours. The temperature of the ice condenser ranged from –55°C to –65°C, and the final pressure was 5 Pa.

The dried samples (untreated wood and wood treated with PEG 300) were seasoned to obtain the equilibrium moisture content at a relative humidity 50% and the temperature of 18°C for four weeks. Changes in the dimensions of the wood were presented as tangential, radial and cross-section shrinkages, determined from the maximum moisture content to the condition immediately after drying in a vacuum and to the condition after seasoning of vacuum freeze-dried wood in the air. The reduction in the shrinkage of the wood stabilised according to variants 2–9 and then seasoned at RH 50% in relation to the shrinkage of seasoned reference samples (variant 1) was presented with the use of anti-shrink efficiency (ASE) described by Stamm [1964].

Tangential, radial and cross-section shrinkages and ASE values were calculated according to the following formulas:

$$S = \frac{D_2 - D_1}{D_2} \times 100 \tag{1}$$

where: S – linear (tangential ST or radial SR) shrinkage [%],
D_2 – dimension of the sample at the maximum moisture content [mm],
D_1 – dimension of the sample after drying and seasoning at RH 50% [mm],

$$S_{CS} = 100 - \frac{(100 - S_T)(100 - S_R)}{100} = S_T + S_R - \frac{S_T S_R}{100} \tag{2}$$

where: S_{CS} – cross-section shrinkage [%],
S_T – tangential shrinkage [%],
S_R – radial shrinkage [%],

$$ASE = \frac{S_2 - S_1}{S_2} \times 100 \tag{3}$$

where: ASE – anti-shrink efficiency [%],
S_2 – shrinkage in untreated wood, dried in the air and seasoned at RH 50% (variant 1) [%],
S_1 – shrinkage in wood stabilised according to variants 2–9 and seasoned at RH 50% [%].

The samples were weighed after each stage of the experiment and after oven-drying at a temperature of 103°C. The water content in the samples was determined immediately after the vacuum-drying and seasoning of the samples.

Results and discussion

The basic macroscopic features and selected physical properties of the archaeological wood under research are presented in table 2. Regarding the values of the mean width of annual rings and the values of maximum moisture contents and basic densities, it can be stated that in both cases, the material under research characterised a relatively slight degree of degradation. Nevertheless, the total shrinkages in the archaeological oak wood were twice as big as the shrinkages in the archaeological pine wood or shrinkages in the fresh non-degraded oak wood.

Table 2. Selected macroscopic features and physical properties of the examined archaeological wood
Tabela 2. Wybrane cechy makroskopowe i właściwości fizyczne badanego drewna archeologicznego

Feature – Property *Cecha – Właściwość*	Oak wood *Drewno dębu*	Pine wood *Drewno sosny*
Width of annual rings [mm] *Szerokość przyrostów rocznych [mm]*	1.11	2.26
Percentage of latewood [%] *Udział drewna późnego [%]*	43.7	65.9
Maximum moisture content [%] *Wilgotność maksymalna [%]*	136	180
Basic density [kg·m^{-3}] *Gęstość umowna [kg·m^{-3}]*	494	406
Total tangential shrinkage [%] *Całkowity skurcz styczny [%]*	16.9	8.0
Total radial shrinkage [%] *Całkowity skurcz promieniowy [%]*	7.6	3.2

In result of treatment, the uptake of PEG 300 by the oak wood ranged from 29.0 to 30.9% of oven-dry mass of wood. Only in the case of samples dried in the air (variant 25PEG-AD), the quantity of polyglycol taken up by the wood was slightly lower (27.5%). In turn, after treatment of the samples with a 45% solution of polyglycol, the uptake of the impregnating agent was 55.8% of oven-dry mass of wood. Due to a higher maximum moisture content (resulting from the lower density and higher porosity of wood), the uptake of PEG 300 in the case of the pine wood samples was from 40.5 to 42.3%. The lower content of polyglycol was noted again in the wood assigned for drying in the air (37.3%). After impregnation of the pine wood samples with a more concentrated solution, the content of a polymer in the wood increased to 72% of oven-dry mass of wood.

After treatment with 25% PEG 300, the water content in the oak wood samples decreased to a level of about 73–77%, and after the treatment of the wood with a 45% solution of polyglycol, to about 40%. In the case of the pine wood

samples, the water content in the wood immediately after treatment was about 95–101% (25% solution) and 60% (45% solution), respectively.

Table 3 presents the water content in the wood determined directly after finishing the tested drying processes (W_D) and after the seasoning of the wood in the air at a relative humidity of 50% and a temperature of 18°C (W_{50}). Both in the case of the oak wood and pine wood, the water content in the samples immediately after vacuum drying (W_D) was considerably lower, than the equilibrium moisture content in the wood that did not undergo treatment (variants 2 and 3) and the treated wood (variants 4–7), seasoned after drying to the constant mass (W_{50}). The water content determined immediately after the drying of the samples in vacuum conditions was higher in the case of the oak wood, than in the pine wood dried in the same conditions. It is particularly distinct in the case of variants 6 and 7. This can be explained by the higher density of the oak wood. The moisture content in the samples that did not undergo treatment was only slightly higher after seasoning in the case of the oak wood, and in the case of the treated samples (variants 4–7 and 8–9), it was considerably lower for the oak wood, than for the pine wood. This resulted from the lower uptake of hygroscopic PEG 300 in the oak wood samples, characterising a lower maximum moisture content and higher basic density.

Table 3. Water content in untreated wood and wood treated with PEG 300 after finish of drying (W_D) and/or after finish of seasoning of the samples in the air at a relative humidity of 50% and a temperature of 18°C (W_{50})
Tabela 3. Zawartość wody w drewnie nieimpregnowanym i drewnie impregnowanym PEG 300 po zakończeniu suszenia (W_D) ***i/lub po zakończeniu sezonowania próbek w powietrzu o wilgotności względnej 50% i temperaturze 18°C*** (W_{50})

Variant of stabilisation *Wariant stabilizacji*		Oak wood *Drewno dębu* [%]		Pine wood *Drewno sosny* [%]	
No. *Nr*	Designation *Oznaczenie*	W_D	W_{50}	W_D	W_{50}
1	W-AD		8.0		7.8
2	W-FD	0.5	7.4	0.1	7.3
3	W-FD/i	1.9	7.6	0.3	7.2
4	25PEG-V	1.2	7.7	1.2	8.9
5	25PEG-FD	1.3	7.9	1.4	9.1
6	25PEG-FD/i	6.1	8.3	1.7	9.7
7	25PEG-FD/c	5.1	7.9	2.5	9.3
8	25PEG-AD		7.9		8.8
9	45PEG-AD		9.0		11.1

Table 4 presents the shrinkages of wood, determined immediately after the vacuum-drying process. Tables 5 and 6 list the shrinkages of oak wood and pine

wood after drying in the air or in vacuum conditions, and then, seasoning in the air at a relative humidity of 50% and temperature of 18°C, and corresponding ASE values related to the shrinkage in the untreated samples, dried in the air (variant 1).

Table 4. Tangential, radial and cross-section shrinkages (S_T, S_R, S_{CS}) in untreated and treated oak wood and pine wood dried in vacuum conditions
Tabela 4. Skurcze styczne (S_T)***, promieniowe (S_R) i przekroju poprzecznego*** (S_{CS}) ***nieimpregnowanego i impregnowanego drewna dębu i drewna sosny suszonego w warunkach próżni***

Variant of stabilisation *Wariant stabilizacji*		Oak wood *Drewno dębu* [%]			Pine wood *Drewno sosny* [%]		
No. *Nr*	Designation *Oznaczenie*	S_T	S_R	S_{CS}	S_T	S_R	S_{CS}
2	W-FD	6.7	4.1	10.5	6.1	3.4	9.3
3	W-FD/i	6.6	3.8	10.1	6.3	3.6	9.7
4	25PEG-V	4.4	1.6	5.9	3.3	0.9	4.2
5	25PEG-FD	4.2	1.5	5.6	2.8	0.7	3.5
6	25PEG-FD/i	3.9	1.2	5.1	2.8	0.6	3.4
7	25PEG-FD/c	3.3	1.2	4.5	3.4	1.1	4.5

Table 5. Tangential, radial and cross-section shrinkages (S_T, S_R, S_{CS}) and anti-shrink efficiencies (ASE_T, ASE_R, ASE_{CS}) in untreated and treated oak wood dried according to tested variants and seasoned in the air at a relative humidity of 50% and a temperature of 18°C
Tabela 5. Skurcze styczne (S_T)***, promieniowe*** (S_R) ***i przekroju poprzecznego*** (S_{CS}) ***oraz wskaźniki stabilizacji wymiarowej*** (ASE_T, ASE_R, ASE_{CS}) ***nieimpregnowanego i impregnowanego drewna dębu suszonego według testowanych wariantów i sezonowanego w powietrzu o wilgotności względnej 50% i temperaturze 18°C***

Variant of stabilisation *Wariant stabilizacji*		Shrinkage *Skurcz* [%]			ASE *ASE* [%]		
No. *Nr*	Designation *Oznaczenie*	S_T	S_R	S_{CS}	ASE_T	ASE_R	ASE_{CS}
1	W-AD	12.8	4.3	16.5			
2	W-FD	5.6	3.2	8.6	56.3	25.6	47.8
3	W-FD/i	5.6	3.1	8.5	56.3	27.9	48.3
4	25PEG-V	1.4	0.4	1.8	89.1	90.7	89.1
5	25PEG-FD	1.3	0.4	1.7	89.8	90.7	89.7
6	25PEG-FD/i	1.7	0.4	2.1	86.7	90.7	87.3
7	25PEG-Fd/c	0.9	0.3	1.2	93.0	93.0	92.7
8	25PEG-AD	6.6	1.2	7.7	48.4	72.1	53.2
9	45PEG-AD	1.4	0.1	1.5	89.1	97.7	90.9

Table 6. Tangential, radial and cross-section shrinkages (S_T, S_R, S_{CS}) and anti-shrink efficiencies (ASE_T, ASE_R, ASE_{CS}) in untreated and treated pine wood dried according to tested variants and seasoned in the air at a relative humidity of 50% and a temperature of 18°C

Tabela 6. Skurcze styczne (S_T)***, promieniowe*** (S_R) ***i przekroju poprzecznego*** (S_{CS}) ***oraz wskaźniki stabilizacji wymiarowej*** (ASE_T, ASE_R, ASE_{CS}) ***nieimpregnowanego i impregnowanego drewna sosny suszonego według testowanych wariantów i sezonowanego w powietrzu o wilgotności względnej 50% i temperaturze 18°C***

Variant of stabilization *Wariant stabilizacji*		Shrinkage *Skurcz* [%]			ASE *ASE* [%]		
No. *Nr*	Designation *Oznaczenie*	S_T	S_R	S_{CS}	ASE_T	ASE_R	ASE_{CS}
1	W-AD	5.2	1.8	6.9			
2	W-FD	4.6	2.5	7.0	11.5	-38.9	-1.2
3	W-FD/i	4.8	2.6	7.3	7.7	-44.4	-5.4
4	25PEG-V	0.3	-0.1	0.2	94.2	105.6	97.1
5	25PEG-FD	0.1	-0.1	0.0	98.1	105.6	100.0
6	25PEG-FD/i	-0.1	-0.1	-0.2	101.9	105.6	102.9
7	25PEG-FD/c	0.1	-0.1	0.0	98.1	105.6	100.0
8	25PEG-AD	-0.3	-0.1	-0.4	105.8	105.6	105.8
9	45PEG-AD	-0.5	-0.1	-0.6	109.6	105.6	108.7

As previously mentioned, the water content in all the samples immediately after they had been dried in vacuum conditions was distinctly lower, than the corresponding equilibrium moisture content of the wood noted after the stage of its seasoning (table 3). The considerable degree of drying of the samples, reached in vacuum conditions, was related to the higher shrinkages of wood (table 4), rather than the values obtained after the seasoning of the dried wood (tables 5 and 6, variants 2–7). The biggest differences appeared in the case of the samples treated with a 25% solution of PEG 300 (variants 4–7).

In the case of the oak wood samples (table 5), shrinkages in the untreated and vacuum-dried wood (variants 2 and 3), as well as the wood treated with a 25% solution of PEG 300 and dried in the air (variant 8) were, after the stage of seasoning, lower by approximately 50% in comparison with the shrinkage in the reference samples (variant 1). Then, the anti-shrink efficiency values in the tangential direction (ASE_T) and cross-section (ASE_{CS}) were about 50%, and in the radial direction (ASE_R), below 30% (variants 2 and 3), or more than 70% (variant 8). Considerably lower changes in the initial dimensions of the waterlogged wood were noted in all the samples treated with a 25% solution of PEG 300 and dried in vacuum conditions. This referred equally to wood that underwent interrupted and uninterrupted freeze-drying, as well as to the samples which were

not frozen (variant 4). Shrinkages determined during these tests were then about 10 times lower than shrinkages in untreated reference samples, dried in the air. Similar values were also obtained in the case of the wood which was treated with a 45% solution of polyglycol before drying in the air. In both cases, ASE values reached about 90% and they were considerably higher than the values registered by Jones et al. [2009] after freeze-drying of archaeological wood treated with sugar alcohols. Such a high degree of dimensional stabilisation of wood points to the possibility of applying the tested methods of drying for the conservation of small, well-preserved oak wood archaeological objects. The smallest dimensional changes were registered in the case of the wood that underwent freeze-drying in the ice condenser chamber (variant 7). Putting the samples in the coolest place of the system resulted in being able to maintain a lower temperature of the frozen solution of polyglycol than in the case of a non-cooled drying chamber. In spite of the increase in the temperature of the samples during the drying of the wood in non-cooled chamber conditions, the results obtained in the case of the samples dried after freezing, and even without previous freezing (variant 4) did not differ much from the best variant of stabilisation. At the same time, at the comparable shrinkages of wood, the tested variants of vacuum-drying differed in the time of the process. In the case of the samples dried in the neighbourhood of the ice condenser (variant 7), the time of the process was 7 days. After 3 days, which were enough to have the wood dried uninterruptedly in the non-cooled chamber, the water content in the samples, which were placed in the coolest place of the system was still 40%. The shortest time of drying in vacuum conditions was noted in the case of the samples dried in an interrupted process (variant 6) in which the total time of remaining of wood in decreased pressure conditions was only 25 hours. It can be extremely significant regarding the costs of the process, especially during the drying of objects of a bigger size, requiring a longer drying time. Interrupting the process and decreasing the temperature of the solution of the treating agent between the next stages of the drying of the wood in vacuum conditions can positively influence the results of the dimensional stabilisation of objects that undergo the conservation process. It shall be supposed that this will play a more important role in the case of areas, which are characterised by a higher degree of decomposition of the wood tissue. However, this would require some tests on the material showing a more much advanced degree of degradation of cell walls.

Shrinkages in the untreated reference pine wood samples were considerably lower (at least by half) than the shrinkages in the oak wood. The drying of the untreated pine wood samples in vacuum conditions (variants 2 and 3) did not result in the lowering of cross-section shrinkage (table 6). However, bigger differences were observed in the case of linear shrinkages. While ASE values in the tangential direction shaped at the level of about 10%, ASE values in the radial direction reached negative values (about –40%). This points to a slight decrease in tangential shrinkage and at the same time, an increase in radial shrinkage in compari-

son to the shrinkage in the reference samples. Differences between the shrinkage in the vacuum-dried wood and wood dried in the air were, however, not so drastic, as this could be assumed on the basis of ASE values. Nevertheless, tangential shrinkage in the case of the samples dried in vacuum conditions was about 0.4-0.6% lower, and radial shrinkage, about 0.7–0.8% higher, than in the case of material dried in the air. This meant, that the proportion between both shrinkages was in the case of the vacuum-dried samples more similar to the proportion accepted for fresh wood, whereas changes in the dimensions of the samples treated with polyglycols and vacuum-dried (variants 4–7) were very slight in both anatomic directions. In most cases, a small swelling of the wood in relation to its dimensions in waterlogged conditions was noted (negative values in table 6). The dimensional changes ranged mainly from 0.1 (shrinkage) to –0.1% (swelling). Also the wood treated with a 25% solution of PEG 300 and dried in the air did not show any considerable dimensional changes. Only the slight swelling of the samples in the tangential direction was noted (–0.3%). This swelling increased when the wood treated with a more concentrated solution of polyglycol was dried (variant 9), whereas an increase in the swelling of the wood in the radial direction was not observed. A comparison of the results obtained for the wood treated with a 25% solution of PEG 300 and dried in vacuum conditions (variants 4–7) and in the air (variant 8) points to a similar level of dimensional stabilisation in the tested pine wood, independent of the conditions in which it was dried.

Conclusions

On the basis of the results of the research, the following conclusions have been drawn:

1. The dimensional changes in well-preserved, waterlogged archaeological oak and pine wood, pre-treated with PEG 300 and freeze-dried mainly depend on the quantity of the polymer taken up by the wood, and less, on the temperature of the dried object.
2. The set consisting of a freeze-dryer and non-cooled drying chamber can be used for the conservation of small, well-preserved archaeological wood objects, pre-treated with PEG 300.
3. Interrupting the vacuum freeze-drying of frozen archaeological wood, treated with PEG 300, does not have much influence on the effectiveness of its conservation (dimensional stabilisation), but it may shorten the working time of the freeze-dryer.
4. Comparable dimensional changes, as in the case of the wood modified with a 25% solution of PEG 300 and vacuum-dried, can be obtained after treatment of pine wood with a 25% solution of PEG 300 and treatment of oak wood with a 45% solution of PEG 300, and then drying in the air.

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BADANIE STABILNOŚCI WYMIARÓW DREWNA ARCHEOLOGICZNEGO SUSZONEGO W NIECHŁODZONEJ KOMORZE PRÓŻNIOWEJ POŁĄCZONEJ Z LIOFILIZATOREM LABORATORYJNYM

Streszczenie

W wielu ośrodkach konserwatorskich przyjmuje się, że drewno archeologiczne impregnowane poliglikolami etylenowymi (PEG) i suszone liofilizacyjnie wymaga nie tylko odpowiedniego wchłonięcia impregnatu, ale także stałego utrzymywania niskiej temperatury zamrożonego obiektu. Wiąże się to z koniecznością stosowania instalacji z chłodzoną komorą suszenia, zaprojektowaną do konserwacji materiałów nasyconych poliglikolami. Celem przeprowadzonych badań było porównanie stabilności wymiarów nieimpregnowanego i impregnowanego dobrze zachowanego drewna archeologicznego, które poddano suszeniu w warunkach próżni w niechłodzonej komorze połączonej z liofilizatorem i w chłodzonej komorze liofilizatora oraz zmian wymiarów drewna, niezamrożonego, suszonego w próżni i w powietrzu.

Badania wykonywano na próbkach mokrego drewna archeologicznego o wymiarach 50 × 50 × 10 mm (T × R × L) wycinanych z twardzieli dębu (*Quercus* sp.) i twardzieli sosny zwyczajnej (*Pinus sylvestris* L.). W pracy określano zmiany wymiarów drewna nieimpregnowanego suszonego w powietrzu i w warunkach próżni, drewna impregnowanego 25% wodnym roztworem PEG 300 i suszonego w powietrzu i w próżni oraz drewna impregnowanego 45% roztworem PEG 300 i suszonego w powietrzu według wariantów impregnacji i suszenia podanych w tabeli 1. Próbki suszone w próżni zamrażano (poza wariantem 4) w temperaturze –27°C. Suszenie przeprowadzano w niechłodzonej komorze połączonej z liofilizatorem Alpha 1–4 (Christ) i dwustopniową pompą próżniową Duo 020 (Pfeiffer). Próbki wysuszone w powietrzu i w próżni sezonowano do wilgotności równowagowej przy względnej wilgotności powietrza 50% i temperaturze 18°C. Zmiany wymiarów drewna przedstawiono jako częściowy skurcz styczny, promieniowy i przekroju poprzecznego od stanu maksymalnego nasycenia wodą do stanu bezpośrednio po suszeniu w próżni i do stanu po sezonowaniu próbek w powietrzu. Porównanie skurczów sezonowanego drewna w stosunku do skurczów nieimpregnowanych próbek kontrolnych suszonych w powietrzu przeprowadzono przy pomocy odpowiednich wskaźników stabilizacji wymiarowej ASE.

Podstawowe cechy makroskopowe i właściwości fizyczne badanego drewna podano w tabeli 2. Po impregnacji próbek wchłonięcie PEG 300 do drewna poddawanego suszeniu w próżni wynosiło od 29,0 do 30,9% (dąb) oraz od 40,5 do 42,3% (sosna) s.m. drewna. Zawartość wody w drewnie dębowym zmniejszyła się do 73–77%, a w drewnie sosnowym do 95–101%.

Po suszeniu drewna w próżni zawartość wody w próbkach była niższa niż równowagowa wilgotność drewna nieimpregnowanego i impregnowanego, suszonego w próżni a następnie sezonowanego (tabela 3). Znaczny stopień wysuszenia próbek w próżni łączył się z większym skurczem drewna (tabela 4) niż wartości uzyskane po sezonowaniu wysuszonego drewna (tabela 5 i 6).

W przypadku próbek dębowych skurcze drewna nieimpregnowanego i suszonego w próżni oraz skurcze drewna nasyconego 25% PEG 300 i suszonego w powietrzu były po etapie sezonowania o około połowę mniejsze w porównaniu ze skurczami próbek kontrolnych (nieimpregnowanych i suszonych w powietrzu). Wskaźniki zmniejszenia skurczu ASE w kierunku stycznym i skurczu przekroju poprzecznego wynosiły wówczas około 50%, a w kierunku promieniowym poniżej 30% (wariant 2 i 3) lub ponad 70% (wariant 8). Zdecydowanie mniejsze zmiany początkowych wymiarów mokrego drewna odnotowano dla wszystkich próbek nasyconych 25% PEG 300 i suszonych w próżni. Dotyczyło to w równym stopniu drewna suszonego liofilizacyjnie w sposób ciągły i przerywany, jak i próbek niezamrożonych (wariant 4). Oznaczane w badaniach skurcze były wówczas około 10-krotnie mniejsze niż skurcze nieimpregnowanych próbek kontrolnych wysuszonych w powietrzu.

Zmiany wymiarów próbek sosnowych impregnowanych poliglikolem i suszonych w próżni były bardzo niewielkie w obydwu badanych kierunkach anatomicznych. W większości przypadków odnotowano nieznaczne spęcznienie drewna w stosunku do jego wymiarów w stanie mokrym (wartości ujemne w tabeli 6). Zmiany wymiarów próbek zawierały się głównie w przedziale od 0,1 do –0,1%. Drewno impregnowane 25% PEG 300 i suszone w powietrzu nie wykazywało także większych odkształceń wilgotnościowych. Porów-

nanie wyników uzyskanych dla drewna impregnowanego 25% PEG 300 i suszonego w próżni lub w powietrzu wskazuje na podobny poziom stabilizacji badanego materiału – niezależnie od warunków jego suszenia.

Przeprowadzone badania wykazały, że wysoka stabilność wymiarów dobrze zachowanego drewna archeologicznego zależy głównie od optymalnej ilości wprowadzonego środka modyfikującego, a w mniejszym stopniu od temperatury drewna suszonego w próżni. Porównywalną stabilność wymiarów jak w przypadku drewna modyfikowanego 25% PEG 300 i suszonego w próżni można uzyskać po jego wysuszeniu w powietrzu przy takim samym wchłonięciu poliglikolu do drewna sosny i około dwukrotnie większym wchłonięciu do drewna dębu. Zestawy składające się z liofilizatora i niechłodzonej komory suszenia mogą być wykorzystywane do konserwacji małych dobrze zachowanych archeologicznych obiektów drewnianych impregnowanych poliglikolami.

Słowa kluczowe: drewno archeologiczne, skurcz, konserwacja, stabilizacja wymiarowa, PEG 300, suszenie liofilizacyjne