

INFLUENCE OF PRE-TREATMENT ON SHRINKAGE OF FREEZE-DRIED ARCHAEOLOGICAL OAK-WOOD

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Abstract. The article presents results of the research on the shrinkage of slightly degraded, freeze-dried archaeological oak-wood (*Quercus* sp.). Before drying, samples of wood were treated with 10, 20, and 30% water solutions of PEG 300, PEG 4000, and sucrose, as well as the mixture of both polyglycols. Dimensional changes in tangential, radial, and longitudinal directions were determined immediately after freeze-drying, and then, after seasoning of the freeze-dried samples in the air at RH 44 and 70%. Shrinkage of untreated and treated freeze-dried oak-wood was considerably smaller, than that of the material, which was dried naturally (ASE_T from 49 to 97%, ASE_R from 39 to 98%).

Key words: archaeological wood, shrinkage, impregnation, freeze-drying

INTRODUCTION

Freeze-drying (lyophilisation) of porous organic materials is used mainly in food production and pharmaceutical industry [Zwetkow 1985]. This method allows for removing water without causing damage or deformation in the porous structure of material that is being dried, as well as decomposition of its chemical compounds. Regarding costs of drying lyophilisation has not been widely applied for drying sawn timber and other wood assortments. Much more frequently, this method has been used during conservation of waterlogged archaeological wood.

The first conservation activities were made on slightly decomposed wood. In order to limit the shrinkage of wood tissue and minimise the results of water freezing, before the process started, artefacts had been treated with water solution of PEG 400 [Ambrose 1970, 1990, Elmer 1973] or with t-buthanol (TBA) solution of PEG 4000 [Sawada 1978, Jespersen 1979]. Application of pre-treatment of wood with two types of polyethylene glycols was the solution to all problems appearing during freeze-drying of multiquality timber objects. Watson [1982] suggested using the mixture of PEG 400 and PEG 4000, the efficiency of which was confirmed also in case of conservation of the most degraded wood. Whereas, Cook and Grattan [1985] demonstrated usefulness of

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pre-treatment of wood with PEG 400 and PEG 3350. They also worked out the method of calculating the concentration of polyglycols for freeze-drying, depending on the degree of decomposition of wood that undergoes conservation [Cook and Grattan 1991].

In the available literature, one can find publications describing theoretical principles of waterlogged wood conservation with freeze-drying method [Jensen et al. 2002, Jensen and Schnell 2005, Jensen and Jensen 2006], as well as many articles presenting installation, parameters of the process, and objects that underwent conservation. Whereas, there are no research reports on the shrinkage of wood. In the past, influence of pre-treatment with polyethylene glycols on shrinkage of the freeze-dried archaeological wood was examined by Grattan [1989]. Comparison of dimensional stabilization in wood treated with PEG 400, PEG 1500, PEG 3350, and PEG 4000 was also made on the basis of observation of the objects that underwent conservation [Hoffmann and Fortuin 1991]. The degree of wood decomposition, its shrinkage in result of freeze-drying, and appearance of cracks were then presented only with the use of a simple point scale. In Poland, freeze-drying of untreated recent and archaeological oak-wood was researched by Matejak et al. [1997]. Whereas, Babiński [2007] examined changes in dimensions of archaeological pine-wood, treated with polyglycols and sucrose.

The research aimed at making comparison of the degree of shrinkage of slightly degraded waterlogged archaeological oak-wood, dried naturally, and with the use of freeze-drying method. The work described influence of pre-treatment with some selected chemical compounds on the degree of deformation of dried material, as well as some changes in its moisture content and shrinkage, appearing together with the changes in air humidity to the level that is characteristic of conditions of the museum's exhibition.

MATERIALS AND METHODS

The research was done on heartwood of oak (*Quercus* sp.) taken from the construction element of the house from the 13th c., coming from the excavations in Szczecin. The experimental material was characterized on the basis of the width of annual rings and percentage of late wood in samples with moisture content about 12%, as well as its maximum moisture content, conventional density and shrinkage in tangential, radial and longitudinal direction.

In the research, samples of waterlogged archaeological wood were used, dimensions of which were as follows: $50 (T) \times 50 (R) \times 10 (L) mm$ (CS samples), and $50 (T) \times 50 (L) \times 10 (R) mm$ (T samples). They were treated with the method of the bath in 10%, 10-20%, and 10-30% water solutions of PEG 300, PEG 4000, sucrose, and the two mixtures of PEG 300 and PEG 4000 for 90 weeks. Samples treated with the mixtures of polyglycols underwent the two-stage impregnation. On the first stage, wood was treated with solution of PEG 300 to which PEG 4000 was added on the next stage. Designations of untreated (control) and treated samples as well as time of impregnation in individual solutions were presented in Table 1. 2% biocide named Kemobiocide DP III was added to the initial solutions of impregnating materials as well as to water in which control samples were kept.

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In each variant specified in Table 1, 4 CS samples and 4 T samples were treated at the same time. Temperature of the solutions was maintained within the range from 15 to 20° C. When impregnation was finished, 4 pins were put in each sample in order to determine the shrinkage of wood. Prior to freeze-drying, samples which stayed in solutions or water (Control F-D) were cooled for three days at the temperature of about 3°C. When the excess impregnation material was removed from the surface of wood, the samples underwent freezing in closed polypropylene boxes at the temperature of about -27° C for five days.

Table 1. Designation of samples and time of impregnation of waterlogged oak wood before freeze-drying

Designation of samples Oznakowanie próbek	Impregnant Impregnat	Time of impregnation in individual solutions weeks Czas impregnacji w poszczególnych roztworach tygodnie							
		10%	15%	20%	25%	30%			
10% PEG 300	PEG 300	90							
20% PEG 300	PEG 300	3	3	84					
30% PEG 300	PEG 300	3	3	3	3	78			
10% PEG 4000	PEG 4000	90							
20% PEG 4000	PEG 4000	3	3	84					
30% PEG 4000	PEG 4000	3	3	3	3	78			
10% Sucrose	Sucrose	90							
20% Sucrose	Sucrose	3	3	84					
30% Sucrose	Sucrose	3	3	3	3	78			
10% PEG 300 + 10% PEG 4000	PEG 300 + PEG 4000	9		81					
15% PEG 300 + 15% PEG 4000	PEG 300 + PEG 4000	3	6		3	78			
Control A-D	untreated air-dried control samples nieimpregnowane próbki kontrolne suszone w powietrzu								
Control F-D	untreated freeze-dried control samples nieimpregnowane próbki kontrolne suszone sublimacyjnie								

Tabela 1. Oznakowanie próbek i czas impregnacji mokrego drewna dębu przed suszeniem sublimacyjnym

Freeze-drying of wood was performed in a chamber with the capacity of about 300 dm³, connected to the laboratory freeze-dryer Alpha 1-4 (Christ) with the capacity 4 kg of ice and 2-stage vacuum pump Duo 020 (Pfeiffer), with the capacity of 20 m³/h (Fig. 1). During freeze-drying of wood, the temperature of the ice-condenser was about -60° C. The process was finished after 48 hours at the pressure of 8 Pa.

Measurements of samples dimensions were taken before and after freeze-drying, immediately after taking the wood from the chamber and then, after bringing wood to the equilibrium moisture content in the air at the temperature 22°C and relative humidity (RH) 44%, in the air at the temperature 18°C and relative humidity 70% and after



Fig. 1. Equipment for freeze-drying of the organic materials in the conservation laboratory at the Archaeological Museum in Biskupin Rys. 1. Zestaw do sublimacyjnego suszenia materiałów organicznych w pracowni konserwatorskiej Muzeum Archeologicznego w Biskupinie

drying the samples to oven-dry mass at the temperature 105° C. Each measurement was made three times with calliper gauge, exact to 0.01 mm. Weighing samples was performed before and after impregnation, as well as after each stage of drying or airconditioning of wood, exact to 0.01 g.

Changes in dimensions of wood from the state of maximum saturation with water (control samples) or impregnating solution (treated samples) to the state immediately after freeze-drying and to the equilibrium moisture content at the above-mentioned air parameters were determined: on CS samples – in tangential and radial direction, and on T samples, in tangential and longitudinal direction, in accordance with the following formula:

$$\beta = \frac{l_0 - l_1}{l_0} \times 100,$$

where:

 β – linear shrinkage of wood, %,

 l_0 – initial dimension of wood (in the state of maximum saturation), mm,

 l_1 – final dimension of wood (after drying), mm.

Shrinkage of impregnated and freeze-dried wood was compared with that of untreated freeze-dried samples (Control F-D) and samples dried in the air (Control A-D) with the use of ASE (anti-shrink efficiency), calculated according to the following formula:

$$ASE = \frac{\beta_0 - \beta_1}{\beta_0} \times 100,$$

where:

ASE – anti-shrink efficiency, %,

 β_0 – linear shrinkage of untreated wood (Control A-D or Control F-D), %,

 β_1 – linear shrinkage of treated wood, %.

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Uptake of impregnant was presented as the per cent ratio of the absolutely dry mass of the modifying agent that was taken up, to the absolutely dry mass of wood, determined on the basis of the mean maximum moisture content of control samples. The degree of wood impregnation was also evaluated on the basis of the change in wet mass of the sample after treatment ΔM (per cent ratio of wet mass of the sample after and before treatment), as well as water content in the sample immediately after impregnation W_{IMP} (per cent ratio of water mass to the sum of masses of absolutely dry wood and impregnating agent).

RESULTS AND DISCUSSION

Table 2 presents basic macroscopic features and some selected physical properties of the researched oak-wood. The research material is characterized with only a slight degree of decomposition of wood tissue. It is proved by the values of the maximum moisture content and conventional density, as well as the slight shrinkage in longitudinal direction.

Table 2. Basic macroscopic characteristics and selected physical properties of oak	wood
Tabela 2. Podstawowe cechy makroskopowe i wybrane właściwości fizyczne drew	na dębu

Characteristic – Property Cecha – Właściwość	Mean value Wartość średnia	Minimum value Wartość minimalna	Maximum value Wartość maksymalna	Standard deviation Odchylenie standardowe	Variation coefficient Współczynnik zmienności
Width of annual rings, mm Szerokość przyrostów rocz- nych, mm	1.11	0.73	1.78	0.21	18.63*
Percentage of latewood, % Udział drewna późnego, %	43.7	25.6	68.6	7.9	18.1
Maximum moisture content, % Wilgotność maksymalna, %	136.0	131.1	142.0	4.4	3.2
Conventional density, kg·m ⁻³ Gęstość umowna, kg·m ⁻³	494	479	506	11	2.2*
Tangential shrinkage, % Skurcz styczny, %	16.9	15.7	19.8	1.3	7.7
Radial shrinkage, % Skurcz promieniowy, %	7.6	6.9	8.5	0.6	7.9
Longitudinal shrinkage, % Skurcz wzdłużny, %	0.7	0.4	1.2	0.3	42.9

*Value in percentage.

*Wartość w procentach.

Table 3 and 4 contain comparison of the per cent changes in wood mass in result of its treatment, uptake of impregnating agents, moisture content, and shrinkage of wood immediately after freeze-drying, after seasoning of the freeze-dried samples at RH 44

Table 3. CS samples. Uptake of impregnant, water content, and tangential and radial shrinkage of oak wood

Tabela 3. Próbki CS. Wchłonięcie impregnatu, zawartość wody i skurcz drewna dębu w kierunku stycznym i promieniowym

			Water content Zawartość wody %				Shrinkage of wood Skurcz drewna %							
Designation of samples Oznakowanie próbek	ΔM %	Uptake Wchło- nięcie %	WIMP	W _{FD}	W44	W ₇₀	immediately after freeze- drying bezpośrednio po liofilizacji		seasoned at RH 44% sezonowane- go przy RH 44%		seasoned at RH 70% sezonowane- go przy RH 70%		oven-dried at 105°C wysuszonego w 105°C	
							β_{T}	β_R	β_{T}	β_R	β_{T}	β_R	β_{T}	β_R
Control A-D			133.9		10.2	11.3			14.7	5.9	14.1	5.6	16.9	7.6
Control F-D			133.1	1.4	9.7	12.3	6.3	5.2	5.0	4.0	4.4	3.5	6.9	5.5
10% PEG 300	100.2	10.5	112.5	1.7	8.1	11.8	6.3	3.8	5.0	2.9	4.1	2.2	6.7	3.7
20% PEG 300	100.8	18.6	99.1	2.3	7.7	12.9	5.3	2.6	3.3	1.3	2.0	0.6	5.0	1.7
30% PEG 300	101.6	30.5	82.3	2.5	8.0	14.5	4.7	2.0	1.7	0.4	0.4	0.1	3.3	0.8
10% PEG 4000	99.7	3.3	126.1	1.2	9.6	12.3	7.0	4.8	5.5	3.5	5.0	3.2	7.5	5.1
20% PEG 4000	98.4	10.0	109.3	1.3	9.1	11.7	7.6	4.8	6.2	3.6	5.5	3.1	8.1	5.0
30% PEG 4000	95.9	21.2	85.1	1.2	7.8	11.0	7.0	4.0	5.4	2.9	4.6	2.3	6.4	3.7
10% Sucrose	101.0	11.2	112.5	1.7	8.9	12.3	6.4	4.2	5.1	3.2	4.3	2.6	6.8	4.2
20% Sucrose	102.7	22.4	96.4	2.5	8.4	12.0	6.3	3.9	4.8	2.7	3.6	1.9	6.1	3.4
30% Sucrose	104.3	36.5	79.0	4.6	8.4	12.4	7.0	3.5	5.1	2.3	3.3	1.3	5.8	2.7
10% PEG 300 + 10% PEG 4000	98.6	14.8	101.2	2.0	8.0	12.1	6.7	3.7	5.2	2.6	4.2	1.9	6.8	3.3
15% PEG 300 + 15% PEG 4000	99.0	20.7	91.9	2.1	7.3	12.2	5.9	2.8	4.0	1.8	2.6	1.0	5.2	2.0

Objaśnienie skrótów w tabeli 3 i 4: ΔM – procentowy stosunek masy próbek po i przed impregnacją, W_{IMP} – zawartość wody w próbkach bezpośrednio po impregnacji, W_{FD} – zawartość wody w próbkach bezpośrednio po liofilizacji, W_{44} – zawartość wody w próbkach liofilizowanych i sezonowanych przy RH 44%, W_{70} – zawartość wody w próbkach liofilizowanych i sezonowanych przy RH 70%, β_T – skurcz styczny, β_R – skurcz promieniowy (tylko w tabeli 3), β_L – skurcz wzdłużny (tylko w tabeli 4).

and 70% and after drying the treated and freeze-dried wood to obtain absolutely dry mass. Table 5 and 6 shows the values of ASE for treated wood, brought to the equilibrium moisture contents at RH 44 and 70%.

Uptake of impregnating agents by oak-wood increased with the increase in final concentration of the solution. As it should be expected, higher values were obtained in case of CS samples and wood treated with compounds of lower molecular weight (PEG 300, sucrose). Greater density of sucrose (about 1.6 g·cm⁻¹) than density of polyglycols

Table 4. T samples. Uptake of impregnant, water content, and tangential and longitudinal shrinkage of oak wood

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Tabela 4. Próbki T. Wchłonięcie impregnatu, zawartość wody i skurcz drewna dębu w kierunku stycznym i wzdłużnym

			Water content Zawartość wody %				Shrinkage of wood Skurcz drewna %							
Designation of samples Oznakowanie próbek	ΔM %	Uptake Wchło- nięcie %	W _{IMP}	W _{FD}	W44	W ₇₀	immediately after freeze- drying bezpośrednio po liofilizacji				seasoned at RH 70% sezonowane- go przy RH 70%		oven-dried at 105°C wysuszonego w 105°C	
							β_{T}	$\beta_{\rm L}$	β_{T}	β_L	β_{T}	β_L	β_{T}	$\beta_{\rm L}$
Control A-D			137.6		10.5	11.0			12.0	0.6	11.9	0.4	14.3	0.7
Control F-D			132.9	2.7	10.2	13.0	4.7	0.9	4.0	0.5	3.4	0.3	6.2	0.7
10% PEG 300	98.5	9.8	109.9	6.0	8.5	12.1	4.9	0.6	4.9	0.3	4.0	0.1	6.6	0.4
20% PEG 300	98.5	14.3	101.6	18.1	7.7	13.2	3.6	0.3	5.9	0.3	4.0	0.1	7.1	0.4
30% PEG 300	97.4	26.1	80.7	19.2	8.1	14.9	3.3	0.2	5.9	0.1	3.2	0.0	7.4	0.2
10% PEG 4000	99.9	0.1	134.1	2.2	9.9	12.8	4.8	0.7	4.0	0.2	3.4	0.4	6.1	0.6
20% PEG 4000	94.8	3.9	113.6	5.8	9.6	12.5	6.1	0.6	6.1	0.4	5.5	0.3	8.0	0.6
30% PEG 4000	87.2	10.2	85.2	10.2	8.7	12.3	5.2	0.3	5.8	0.3	5.0	0.2	7.3	0.5
10% Sucrose	100.1	7.6	117.8	5.3	9.6	13.1	5.2	0.6	5.1	0.4	4.3	0.2	7.1	0.6
20% Sucrose	100.3	16.5	101.4	10.2	9.1	13.1	4.8	0.5	5.2	0.3	4.0	0.2	6.6	0.4
30% Sucrose	99.5	22.0	90.8	19.3	9.4	14.0	3.7	0.2	5.3	0.2	3.5	0.0	5.8	0.3
10% PEG 300 + 10% PEG 4000	96.6	11.2	103.4	7.4	8.3	12.5	5.5	0.4	5.8	0.3	4.6	0.1	7.3	0.4
15% PEG 300 + 15% PEG 4000	96.0	18.0	90.3	12.4	7.1	12.2	5.1	0.2	6.5	0.2	4.8	0.0	7.2	0.2

Explanation of abbreviations - see Table 3.

Objaśnienie skrótów – patrz tabela 3.

(about 1.1 g·cm⁻¹) shall not be underestimated, as well. With the increase in uptake, moisture content in the treated sample decreased. W_{IMP} values stated in Table 3 and 4 comprised within the range from 79.0 to 134.1%. During the long impregnation period, dehydration of slightly degraded wood tissue was observed. It is proved by clear lowering of the per cent ratio of wet wood mass after and before treatment (ΔM values below 100% in Table 3 and 4). It could be observed in case of T samples, but also after impregnation of some CS samples (especially those treated with PEG 4000).

Moisture content in freeze-dried untreated CS samples was 1.4%. Apart from wood treated with 30% solution of sucrose (samples with the greatest uptake), moisture content of treated CS samples was similar (W_{FD} from 1.2 to 2.5%). Whereas, moisture content in T samples was 2.7% for untreated wood and from 2.2 to 19.3% in case of treated samples. The samples with the greatest uptake and smallest moisture content (W_{IMP}), treated with the most condensed solutions dried most slowly.

Table 5. ASE values for pre-treated and freeze-dried oak wood, seasoned at RH 44 and 70%, determined in relation to the shrinkage of untreated air-dried wood (Control A-D samples) Tabela 5. Wartości ASE dla impregnowanego drewna dębu wysuszonego sublimacyjnie i sezonowanego przy względnej wilgotności powietrza 44 i 70%, określane w stosunku do skurczu drewna nieimpregnowanego suszonego w powietrzu (próbki Control A-D)

Designation –	ASE, %											
		CS samples	– próbki C	S	T samples – próbki T							
Oznakowanie próbek	RH	44%	RH	70%	RH	44%	RH 70%					
protect	Т	R	Т	R	Т	L	Т	L				
10% PEG 300	66.0	50.8	70.9	60.7	59.2	50.0	66.4	75.0				
20% PEG 300	77.6	78.0	85.8	89.3	50.8	50.0	66.4	75.0				
30% PEG 300	88.4	93.2	97.2	98.2	50.8	83.3	73.1	100.0				
10% PEG 4000	62.6	40.7	64.5	42.9	66.7	66.7	71.4	0.0				
20% PEG 4000	57.8	39.0	61.0	44.6	49.2	33.3	53.8	25.0				
30% PEG 4000	63.3	50.8	67.4	58.9	51.7	50.0	58.0	50.0				
10% Sucrose	65.3	45.8	69.5	53.6	57.5	33.3	63.9	50.0				
20% Sucrose	67.3	54.2	74.5	66.1	56.7	50.0	66.4	50.0				
30% Sucrose	65.3	61.0	76.6	76.8	55.8	66.7	70.6	100.0				
10% PEG 300 + 10% PEG 4000	64.6	55.9	70.2	66.1	51.7	50.0	61.3	75.0				
15% PEG 300 + 15% PEG 4000	72.8	69.5	81.6	82.1	45.8	66.7	59.7	100.0				

After the process of freeze-drying, on the surface of the cross-section of wood treated with PEG 4000 one could observe a white layer of polyglycol. Its amount increased with the increase in PEG 4000 uptake. Wood cracking appeared only on the surface of two T samples (treated with PEG 4000 and sucrose), as well as all T samples, freeze-dried without any pre-treatment (Control F-D samples).

Shrinkage of freeze-dried untreated wood was 6.3% in tangential and 5.2% in radial direction for CS samples, and 4.7% in tangential and 0.9% in longitudinal direction for T samples. Shrinkage of treated wood was slightly smaller or slightly greater than the above-mentioned values. It depended on the type and uptake of modifying agent (Table 3 and 4). Regarding big differences in moisture content in individual batches of dried T samples it was recognised, that making comparison of wood dimensions immediately after freeze-drying is not very objective. Same time, the majority of samples (mainly CS samples) was dried definitely below the values of equilibrium moisture content of wood staying in conditions of the museum's exhibition. Whereas, small quantity of samples the moisture content of which exceeded the value of about 12%, required further drying. Making comparison of the shrinkage of treated wood with that of both control samples was thus performed after seasoning of wood at relative air humidity 44 and 70%.

Shrinkage of freeze-dried untreated wood (Control F-D samples), and then, seasoned in controlled conditions at RH 44 and 70%, was considerably lower, than shrinkage of

Table 6. ASE values for pre-treated and freeze-dried oak wood, seasoned at RH 44 and 70%, determined in relation to the shrinkage of untreated and freeze-dried wood (Control F-D samples) Tabela 6. Wartości ASE dla impregnowanego drewna dębu wysuszonego sublimacyjnie i sezo-nowanego przy względnej wilgotności powietrza 44 i 70%, określane w stosunku do skurczu drewna nieimpregnowanego suszonego sublimacyjnie (próbki Control F-D)

Designation of samples	ASE, %										
		CS sample	s – próbki C	S	T samples – próbki T						
Oznakowanie próbek	RH	44%	RH	70%	RH	44%	RH 70%				
proteck	Т	R	Т	R	Т	L	Т	L			
10% PEG 300	0.0	27.5	6.8	37.1	-22.5	40.0	-17.6	66.7			
20% PEG 300	34.0	67.5	54.5	82.9	-47.5	40.0	-17.6	66.7			
30% PEG 300	66.0	90.0	90.9	97.1	-47.5	80.0	5.9	100.0			
10% PEG 4000	-10.0	12.5	-13.6	8.6	0.0	60.0	0.0	-33.3			
20% PEG 4000	-24.0	10.0	-25.0	11.4	-52.5	20.0	-61.8	0.0			
30% PEG 4000	-8.0	27.5	-4.5	34.3	-45.0	40.0	-47.1	33.3			
10% Sucrose	-2.0	20.0	2.3	25.7	-27.5	20.0	-26.5	33.3			
20% Sucrose	4.0	32.5	18.2	45.7	-30.0	40.0	-17.6	33.3			
30% Sucrose	-2.0	42.5	25.0	62.9	-32.5	60.0	-2.9	100.0			
10% PEG 300 + 10% PEG 4000	-4.0	35.0	4.5	45.7	-45.0	40.0	-35.3	66.7			
15% PEG 300 + 15% PEG 4000	20.0	55.0	40.9	71.4	-62.5	60.0	-41.2	100.0			

samples which were dried in the air only (Control A-D samples). So, the shrinkage of freeze-dried wood in tangential direction (β_T from 3.4 to 5.0%) was about three times, and in radial direction ($\beta_{\rm R}$ equal to 3.5 and 4.0%) about one and a half times lower. Very clear decrease in shrinkage of wood was observed also in case of the samples that were treated before freeze-drying. Then, ASE_T values ranged from 49.2 to 97.2%, and ASE_R values, from 39.0 to 98.2% (Table 5). Whereas, differences between changes in dimensions of freeze-dried untreated and treated wood were not so big. At the same time, tangential shrinkage of pre-treated CS and T samples was only slightly bigger, than shrinkage of samples that were not pre-treated (Control F-D). Worse results are represented with negative ASE values in Table 6. Only in case of CS samples with greater uptake of PEG 300, distinct lowering in tangential shrinkage to the level of 1.7 and 0.4% could be observed. Whereas, radial shrinkage of treated wood was always lower, than the shrinkage of Control F-D samples. Similar to the previous situation, the most satisfying ASE values (90.0 and 97.1%) were obtained in case of wood that was treated with the most condensed solution of PEG 300. They corresponded with the shrinkage of wood being 0.4 and 0.1%. Comparable level could be observed also in case of the shrinkage of oak-wood in longitudinal direction (Table 4).

CONCLUSIONS

1. Shrinkage of slightly degraded oak heartwood with the maximum moisture content about 136% and the conventional density about 490 kg·m⁻³ which underwent freeze-drying and then, it was seasoned at RH 44 and 70% is distinctly lower, than the shrinkage of wood dried only in the air-conditioned room.

2. Shrinkage of well-preserved archaeological oak-wood, which was treated with polyethylene glycols or sucrose, and then, freeze-dried and seasoned in air at RH 44 and 70%, can be lower or higher, than the shrinkage of untreated oak-wood, freeze-dried and seasoned in the same conditions. It depends on the type and quantity of a modifying agent, that was absorbed by the wood during its impregnation.

3. Time of freeze-drying of archaeological oak-wood, treated with polyethylene glycols or sucrose increases together with the increase in the quantity of impregnating agent, that is taken up.

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REFERENCES

- Ambrose W.R., 1970. The freeze-drying of swamp degraded wood. In: Conservation of stone and wooden objects. IIC, London, 53-57.
- Ambrose W.R., 1990. Application of freeze-drying to archaeological wood. In: Archaeological wood. Properties, chemistry and preservation. Eds R.M. Rowell, R.J. Barbour. Advances in Chemistry, Series 225. Am. Chem. Soc. Washington DC, 235-263.
- Babiński L., 2007. Influence of pre-treatment on shrinkage of freeze-dried archaeological pinewood. Folia For. Pol. Ser. B, 38, 3-12.
- Cook C., Grattan D.W., 1985. A practical comparative study of treatments for waterlogged wood. Part 3. Pretreatment solutions for freeze-drying. In: Waterlogged wood. Study and conservation. Proceedings of the 2nd ICOM Waterlogged Wood Working Group Conference, Grenoble 1984. Eds R. Ramiére, M. Coraldelle. Centre d'Etude et de Traitement des Bois Gorges d'Eau, 219-240.
- Cook C., Grattan D.W., 1991. A method of calculating the concentration of PEG for freezedrying waterlogged wood. In: Proceedings of the 4th ICOM Group on Wet Organic Archaeological Materials Conference, Bremerhaven 1990. Ed. P. Hoffmann. ICOM Bremerhaven, 239-251.
- Elmer J.T., 1973. Gefriertrocknung neolithischer Gewebe und Geflechte. Arbeitsblätter für Restauratoren 1, Gruppe 8, 17-22.
- Grattan D.W., 1989. International comparative wood treatment study. In: Conservation of wet wood and metal. Proceedings of the ICOM Conservation Workings Groups on Wet Organic Archaeological Materials and Metals, Fremantle 1987. Eds I.D. Mac Leod, D.W. Grattan. West. Austr. Museum Perth, 163-191.
- Hoffmann P., Fortuin G., 1991. An evaluation study on the freeze-drying of waterlogged wood. In: Proceedings of the 4th ICOM Group on Wet Organic Archaeological Materials Conference, Bremerhaven 1990. Ed. P. Hoffmann. ICOM Bremerhaven, 331-357.

- Jensen P., Jensen J.B., 2006. Dynamic model for vacuum freeze-drying of waterlogged archaeological wooden artifacts. J. Cult. Herit. 7, 156-165.
- Jensen P., Schnell U., 2005. The implications of using low molecular weight PEG for impregnation of waterlogged archaeological wood prior to freeze drying. In: Proceedings of the 9th ICOM Group on Wet Organic Archaeological Materials Conference, Copenhagen 2004. Eds P. Hoffmann, K. Strætkvern, J.A. Spriggs, D. Gregory. ICOM Bremerhaven, 279-308.
- Jensen P., Jørgensen G., Schnell U., 2002. Dynamic LV-SEM analyses of freeze drying processes for waterlogged wood. In: Proceedings of the 8th ICOM Group on Wet Organic Archaeological Materials Conference, Stockholm 2001. Eds P. Hoffmann, J.A. Spriggs, T. Grant, C. Cook, A. Recht. ICOM Bremerhaven, 319-331.
- Jespersen K., 1979. Conservation of waterlogged wood by use of tertiary buthanol, PEG and freeze-drying. In: Conservation of Waterlogged Wood. International Symposium on the Conservation of Large Objects of Waterlogged Wood. Ed. L.H. de Vries-Zuiderbaan. Gov. Print. Publ. Office Hague, 69-76.
- Matejak M., Niemz P., Jarczyk A., 1997. Liofilizacyjne suszenie drewna [Freeze-drying of wood]. In: Suszenie drewna. Wyd. SGGW Warszawa, 41-44 [in Polish].
- Sawada M., 1978. Conservation of waterlogged wooden materials from the Nara Palace Site. In: Proceedings of the 1st International Symposium on the Conservation and Restoration of Cultural Property – Conservation of Wood. National Research Institute of Cultural Properties Tokyo, 49-58.
- Watson J., 1982. The applications of freeze-drying on British hardwoods from archaeological excavations. In: Proceedings of the ICOM Waterlogged Wood Working Group Conference, Ottawa 1981. Eds D.W. Grattan, J.C. McCawley. ICOM Ottawa, 237-242.
- Zwetkow Z.D., 1985. Vakuumgefriertrocknung. VEB Fachbuchverlag Leipzig.

WPŁYW WSTĘPNEJ IMPREGNACJI NA KURCZENIE SIĘ WYKOPALISKOWEGO DREWNA DĘBU SUSZONEGO SUBLIMACYJNIE

Streszczenie. W artykule przedstawiono wyniki badań nad kurczeniem się nieznacznie zdegradowanego wykopaliskowego drewna dębu (*Quercus* sp.) suszonego sublimacyjnie. Przed suszeniem, próbki drewna poddano impregnacji w 10-, 20- i 30-procentowych wodnych roztworach PEG 300, PEG 4000 i sacharozy oraz w mieszaninie obydwu poliglikoli. Zmiany wymiarów drewna w kierunku stycznym, promieniowym i wzdłużnym określano bezpośrednio po liofilizacji, a następnie po sezonowaniu wysuszonych sublimacyjnie próbek – w powietrzu o wilgotności względnej wynoszącej 44 i 70%. Skurcz nieimpregnowanego i impregnowanego drewna dębu suszonego sublimacyjnie był wyraźnie mniejszy niż skurcz materiału suszonego w sposób naturalny (ASE_T od 49 do 97%, ASE_R od 39 do 98%).

Słowa kluczowe: drewno wykopaliskowe, skurcz drewna, impregnacja, suszenie sublimacyjne

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