

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

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SYNOPSIS. The subject of the research was the shrinkage of waterlogged archaeological pinewood that underwent pre-treatment in 10-30% water solutions of PEG 300, PEG 4000, sucrose and the mixtures of polyglycols and then, it was freeze-dried. Changes in dimensions of wood samples were determined immediately after freeze-drying, and after seasoning of freeze-dried testing material at relative humidity 44 and 70%. The slightest dimensional changes were noted in case of wood treated with PEG 300 at uptake reaching the values from 22 to 37% of oven-dry wood mass.

KEY WORDS: archaeological wood, shrinkage, impregnation, freeze-drying

INTRODUCTION

Freeze-drying of wood is mainly used during conservation of artefacts coming from wet archaeological sites. Apart from getting rid of water, the basic task of each operation of that kind is to limit the shrinkage of wood tissue and appearance of characteristic deformations and cracks. Like in the case of other methods of dimensional stabilization of waterlogged wood, the archaeological objects undergo pre-treatment before drying. In some cases, applying freeze-drying method can shorten the conservation period, decrease the quantity of modifying agent, and considerably limit the use of energy necessary for heating concentrated impregnating solutions.

At the beginning, freeze-drying was performed only for slightly decomposed wood, treated with water solution of PEG 400 (AMBROSE 1970, ELMER 1973) or t-butanol (TBA) solution of PEG 4000 (SAWADA 1978, JESPERSEN 1979). Cracking and deformations appearing during freeze-drying of more degraded material or multi-quality wood were minimal, if wood was pre-treated with two types of polyglycols (WATSON 1982, COOK and GRATAN 1985).

Comparative research of shrinking of freeze-dried archaeological wood was done by GRATAN (1989). Before drying, the wood was treated with various types of polyethylene glycols. HOFFMANN and FORTUIN (1991) evaluated the efficiency of

dimensional stabilization of freeze-dried archaeological objects, treated with PEG 400, PEG 1500, PEG 3350, and PEG 4000. The degree of wood decomposition, its shrinking and cracking were presented with the use of a simple point scale.

However, in literature on museum management and conservation, one can mainly find reports presenting descriptions of freeze-dried artefacts and equipment used. Whereas, there is lack of information on observed changes in dimensions of dried objects. And most works discuss conservation of oak-wood. In the recent years, some publications have appeared, which deal with theoretical bases and modelling of the process of wood drying with the method of freeze-drying (JENSEN et AL. 2002, JENSEN and SCHNELL 2005, JENSEN and JENSEN 2006). In Poland, application of that method for conservation of waterlogged archaeological wood was discussed by Dyrka and Jagielska (1981). Matejak et al. (1997) presented some results on the shrinkage of untreated recent and archaeological oak-wood, and Babiński (forthcoming), of oak-wood that underwent treatment with polyglycols and sucrose.

The research aimed at making comparison of the degree of shrinkage of well-preserved waterlogged archaeological pinewood, dried naturally, and with the use of freeze-drying method. The work described the 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 exhibition.

MATERIALS AND METHODS

The research was done on heartwood of Scots pine (*Pinus sylvestris* L.) taken from the construction element of the building from the 17th c., coming from the excavations in Gdańsk. 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) × 50 (R) × 10 (L) mm (CS samples), and 50 (T) × 50 (L) × 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.

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

Table 1. Designations of samples and time of pre-treatments of waterlogged pinewood before freeze-drying

Designation of samples	Impregnant	Time of pre-treatment in individual solutions [weeks]				
		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 control samples (air-dried)					
Control F-D	untreated control samples (freeze-dried)					

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 3 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 5 days.

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. During freeze-drying of wood, the temperature of the ice-condenser was about -60°C. The process was finished after 48 h at the pressure being 8 Pa.

Measurements of samples 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 drying the samples to oven-dry mass at the temperature 105°C. Each measurement was taken 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 air-conditioning 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:

$$\beta = \frac{l_0 - l_1}{l_0} \cdot 100$$

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:

$$\text{ASE} = \frac{\beta_0 - \beta_1}{\beta_0} \cdot 100$$

where: ASE – anti-shrink efficiency [%],
 β_0 – shrinkage of untreated wood (Control A-D or Control F-D) [%],
 β_1 – shrinkage of treated wood [%].

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 wood used for the research. The mean maximum moisture content and the mean conventional density, being 180% and 406 kg·m⁻³, respectively, show only slight degree of decomposition of the researched material. Whereas, on the basis of all remaining statistical data (minimum and maximum value, standard deviation, variation coefficient) it can be concluded, that the degree of decomposition of wood is uniform and the properties of all research material are comparable. Linear shrinkage of archaeological wood ($\beta_T = 8.0\%$, $\beta_R = 3.2\%$, $\beta_L = 0.1\%$) reached the level comparable to that in sound pine-wood.

Tables 3 and 4 present increase in mass of samples after treatment, uptake of impregnating agents, water contents in samples after finishing the next stages of the experiment, as well as shrinkage in CS and T samples immediately after freeze-drying, after seasoning of freeze-dried wood at the relative air humidity 44 and 70%, and after drying to oven-dry mass. As opposite to oak-wood (Babiński, forthcoming), after impregnation of pine-wood, increase in mass ΔM in all wet samples was observed. Uptake of impregnants (ranging from 10.3 to 52.2%) increased together with the increase in concentration of the modifying agent, remaining at the comparable level in case of both types of samples treated with the same solutions. Greater uptake of sucrose shall be explained with its greater density. Water con-

Table 2. Basic macroscopic characteristics and selected physical properties of pinewood

Characteristic – Property	Mean value	Minimum value	Maximum value	Standard deviation	Variation coefficient
Width of annual rings [mm]	2.26	1.42	3.21	0.46	20.39*
Percentage of latewood [%]	65.9	47.9	78.7	6.9	10.5
Maximum moisture content [%]	179.6	173.4	186.4	4.5	2.5
Conventional density [kg·m ⁻³]	406	395	417	7	1.7*
Tangential shrinkage [%]	8.0	7.3	8.9	0.6	7.5
Radial shrinkage [%]	3.2	2.7	3.6	0.3	9.4
Longitudinal shrinkage [%]	0.1	-0.2	0.2	0.1	100.0

*Value in %.

tent in the treated pinewood ranged within 93.0 and 151.7%, whereas moisture content in control samples was not less than 175%.

After two days of freeze-drying, water content in the researched samples comprised within the range from 0.6 to 3.9%. In most samples, moisture content did not exceed 2%. In case of samples treated with PEG 4000 and mixtures of PEG 300 and PEG 4000, the surface of the cross-section of wood was covered with a white layer of polyglycol, thickness of which increased with the increasing uptake of impregnating agent (PEG 4000). On the surface of dried samples, there were no visible cracks.

Tangential shrinkage of freeze-dried wood that did not undergo pre-treatment (samples Control F-D) was 6.5% (CS samples) and 5.4% (T samples), while radial shrinkage was 4.0%. Whereas, shrinkage in case of modified and freeze-dried wood was mostly slighter, ranging from 3.4 to 6.7% in tangential direction (CS samples) and from 1.5 to 5.6% (T samples), and from 1.1 to 3.3% in radial direction. As it was mentioned, the researched samples were dried to the moment they reached the moisture content lesser, than the range of equilibrium moisture content in wood that has been exposed in museums. Thus, deformations would be greater, than the shrinkage which could be expected in case of drying the testing material to the level of moisture content about 12%. After the freeze-dried samples were brought to the constant mass in the air at relative humidity 44%, tangential shrinkage of untreated wood decreased to 4.9 (CS samples) and 4.4% (T samples), and radial shrinkage decreased to 2.7%. In the same conditions, tangential shrinkage of modified wood ranged from -0.2 (small swelling) to 4.5% for CS samples, and from -0.1 to 4.2% in case of T samples, and radial shrinkage ranged from -0.1 to 2.1% (Tables 3 and 4). Even smaller shrinkage were noted after seasoning of pinewood at RH 70%. Tangential shrinkage of modified wood comprised within -0.5 and 3.8% (CS samples), and -0.8 and 3.2% (T samples), and radial shrinkage was from -0.2 to 1.7%.

The slightest deformations of wood freeze-dried and seasoned in the air at RH 44 and 70% were registered in case of samples that underwent impregnation with 20 and 30% solutions of PEG 300, at the uptake amounting to 22.1 and 24.2% (20% solution), as well as 35.8 and 37.2% of the wood oven-dry mass (30% solution). A small swelling of wood (negative values in Tables 3 and 4) took place mainly

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

Designation of samples	ΔM [%]	Uptake [%]	Water content [%]				Shrinkage of wood [%]							
			W_{IMP}	W_{FD}	W_{44}	W_{70}	immediately after freeze-drying		seasoned at RH 44%		seasoned at RH 70%		oven-dried at 105°C	
							β_T	β_R	β_T	β_R	β_T	β_R	β_T	β_R
Control A-D			176.0		8.8	10.6			5.5	2.0	5.0	1.8	8.0	3.2
Control F-D			175.5	1.1	8.5	11.4	6.5	4.0	4.9	2.7	4.1	2.2	7.2	3.8
10% PEG 300	100.5	10.3	151.5	1.5	7.4	11.9	6.7	2.6	4.5	1.4	3.3	0.8	6.2	1.9
20% PEG 300	101.2	24.2	124.9	2.0	8.1	14.6	4.8	1.8	1.4	0.3	0.1	-0.1	2.1	0.3
30% PEG 300	102.0	35.8	107.4	2.7	9.6	17.8	3.4	1.1	-0.2	-0.1	-0.5	-0.2	0.6	0.0
10% PEG 4000	100.6	13.1	145.6	0.6	7.6	10.4	6.5	3.3	4.6	2.1	3.8	1.7	6.6	3.0
20% PEG 4000	101.3	26.1	121.7	0.7	6.9	9.6	5.9	2.8	4.2	1.7	3.4	1.3	5.8	2.4
30% PEG 4000	102.8	44.0	97.0	0.6	6.1	8.7	5.0	2.4	3.3	1.5	2.5	1.1	4.4	2.0
10% Sucrose	101.8	15.0	144.4	1.0	7.6	11.6	5.6	2.8	3.9	1.7	2.8	1.2	5.7	2.6
20% Sucrose	104.0	31.4	118.5	1.6	7.1	10.9	6.0	2.9	3.6	1.5	2.5	1.0	5.3	2.3
30% Sucrose	106.6	48.7	98.0	2.1	6.7	9.9	5.7	2.4	3.1	1.0	2.0	0.6	4.7	1.8
10% PEG 300 + 10% PEG 4000	100.9	24.0	124.6	1.6	7.1	11.5	5.4	2.1	3.3	1.0	2.0	0.4	4.5	1.2
15% PEG 300 + 15% PEG 4000	101.4	33.2	110.1	1.5	6.5	11.5	4.7	1.9	2.4	0.8	0.7	0.0	3.0	0.7

Explanation of abbreviations in Tables 3 and 4:

ΔM – per cent ratio of the mass of samples after and before pre-treatment,

W_{IMP} – water content in the samples immediately after pre-treatment,

W_{FD} – water content in the samples immediately after freeze-drying,

W_{44} – water content in the freeze-dried samples seasoned at RH 44%,

W_{70} – water content in the freeze-dried samples seasoned at RH 70%,

β_T – tangential shrinkage,

β_R – radial shrinkage (only in Table 3),

β_L – longitudinal shrinkage (only in Table 4).

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

Designation of samples	ΔM [%]	Uptake [%]	Water content [%]				Shrinkage of wood [%]							
			W_{IMP}	W_{FD}	W_{44}	W_{70}	immediately after freeze-drying		seasoned at RH 44%		seasoned at RH 70%		oven-dried at 105°C	
							β_T	β_L	β_T	β_L	β_T	β_L	β_T	β_L
Control A-D			182.2		9.0	10.7			6.2	-0.1	5.7	-0.1	8.9	0.0
Control F-D			180.4	1.1	8.8	11.6	5.4	0.7	4.4	0.3	3.5	0.2	6.8	0.4
10% PEG 300	100.6	10.4	151.7	1.3	7.3	12.0	5.2	0.2	3.5	0.1	2.1	0.0	5.3	0.1
20% PEG 300	101.8	22.1	130.2	2.0	7.8	14.7	2.5	0.1	0.1	0.0	-0.6	0.0	0.5	0.0
30% PEG 300	102.8	37.2	106.7	3.1	8.9	17.6	2.0	0.1	-0.1	-0.1	-0.8	-0.1	0.8	-0.1
10% PEG 4000	100.7	12.0	148.6	1.0	7.4	10.7	5.6	0.3	4.2	0.1	3.2	0.1	6.1	0.2
20% PEG 4000	102.8	29.8	118.7	0.8	6.5	9.7	4.4	0.3	3.1	0.1	2.2	0.0	4.3	0.2
30% PEG 4000	103.3	44.1	97.9	1.5	5.5	8.8	4.3	-0.1	3.4	-0.2	2.2	-0.2	4.5	-0.1
10% Sucrose	102.4	14.6	147.0	0.9	7.7	11.8	5.1	0.2	3.6	-0.1	2.5	0.0	5.6	0.1
20% Sucrose	104.6	30.2	121.9	2.5	7.6	11.8	4.3	0.2	3.0	0.0	1.8	-0.1	4.6	0.1
30% Sucrose	106.4	52.2	93.0	3.9	7.3	10.8	1.5	0.1	1.7	0.0	0.7	0.0	3.2	0.1
10% PEG 300 + 10% PEG 4000	101.4	21.6	130.5	1.6	7.0	11.8	3.9	0.1	2.2	0.0	0.9	0.0	3.2	0.1
15% PEG 300 + 15% PEG 4000	101.8	32.0	112.9	1.7	6.4	11.8	3.6	0.2	1.8	0.0	0.4	0.0	2.9	0.1

Explanation of abbreviations – see Table 3.

Table 5. ASE values for pre-treated and freeze-dried pinewood, seasoned at RH 44 and 70%, determined in relation to the shrinkage of untreated air-dried wood (Control A-D samples)

Designation of samples	ASE [%]							
	CS samples				T samples			
	RH 44%		RH 70%		RH 44%		RH 70%	
	T	R	T	R	T	L	T	L
10% PEG 300	18.2	30.0	34.0	55.6	43.5	200.0	63.2	100.0
20% PEG 300	74.5	85.0	98.0	105.6	98.4	100.0	110.5	100.0
30% PEG 300	103.6	105.0	110.0	111.1	101.6	0.0	114.0	0.0
10% PEG 4000	16.4	-5.0	24.0	5.6	32.3	200.0	43.9	200.0
20% PEG 4000	23.6	15.0	32.0	27.8	50.0	200.0	61.4	100.0
30% PEG 4000	40.0	25.0	50.0	38.9	45.2	-100.0	61.4	-100.0
10% Sucrose	29.1	15.0	44.0	33.3	41.9	0.0	56.1	100.0
20% Sucrose	34.5	25.0	50.0	44.4	51.6	100.0	68.4	0.0
30% Sucrose	43.6	50.0	60.0	66.7	72.6	100.0	87.7	100.0
10% PEG 300 + 10% PEG 4000	40.0	50.0	60.0	77.8	64.5	100.0	84.2	100.0
15% PEG 300 + 15% PEG 4000	56.4	60.0	86.0	100.0	71.0	100.0	93.0	100.0

Table 6. ASE values for pre-treated and freeze-dried pinewood, seasoned at RH 44 and 70%, determined in relation to the shrinkage of untreated and freeze-dried wood (Control F-D samples)

Designation of samples	ASE [%]							
	CS samples				T samples			
	RH 44%		RH 70%		RH 44%		RH 70%	
	T	R	T	R	T	L	T	L
10% PEG 300	8.2	48.1	19.5	63.6	20.5	66.7	40.0	100.0
20% PEG 300	71.4	88.9	97.6	104.5	97.7	100.0	117.1	100.0
30% PEG 300	104.1	103.7	112.2	109.1	102.3	133.3	122.9	150.0
10% PEG 4000	6.1	22.2	7.3	22.7	4.5	66.7	8.6	50.0
20% PEG 4000	14.3	37.0	17.1	40.9	29.5	66.7	37.1	100.0
30% PEG 4000	32.7	44.4	39.0	50.0	22.7	166.7	37.1	200.0
10% Sucrose	20.4	37.0	31.7	45.5	18.2	133.3	28.6	100.0
20% Sucrose	26.5	44.4	39.0	54.5	31.8	100.0	48.6	150.0
30% Sucrose	36.7	63.0	51.2	72.7	61.4	100.0	80.0	100.0
10% PEG 300 + 10% PEG 4000	32.7	63.0	51.2	81.8	50.0	100.0	74.3	100.0
15% PEG 300 + 15% PEG 4000	51.0	70.4	82.9	100.0	59.1	100.0	88.6	100.0

after seasoning of samples at RH 70%. It resulted from higher hygroscopic ability of PEG 300 and increase in moisture content in samples (W70), even to 17.8% (Table 3). In all remaining cases, moisture of impregnated samples did not exceed 12%.

ASE values determined in relation to wood that did not undergo freeze-drying (Control A-D) were presented in Table 5, and in relation to freeze-dried wood that did not undergo pre-treatment (Control F-D), in Table 6. ASE values ranging within 0 and 100% determine decrease in shrinkage of wood in comparison to the shrinkage of control samples; whereas values exceeding 100% mean the swelling of wood. The highest degree of dimensional stability in the tested wood was noted in

case of samples that were treated with 20 or 30% solution of PEG 300. In relation to the wood that did not undergo freeze-drying, ASET values ranged between 74.5 and 114.0%, whereas, ASER values, between 85.0 and 111.1%. In both cases (20 and 30% PEG 300), ASE calculated in relation to freeze-dried unmodified wood appeared to be similar (Table 6). Advantages resulting from pre-treatment of wood with PEG 300 can also be observed after two-stage treatment (especially with 15% PEG 300 + 15% PEG 4000).

CONCLUSIONS

1. Shrinkage of slightly degraded, freeze-dried archaeological pine heartwood which was then seasoned in the air with RH 44 and 70% is not considerably lower, than the shrinkage of wood that was dried in a natural way, in an air-conditioned room.
2. Shrinkage of well-preserved archaeological pine heartwood that was treated with PEG 300, PEG 4000 or sucrose before freeze-drying is smaller, than the shrinkage of untreated wood.
3. The smallest changes in dimensions of freeze-dried archaeological pinewood with the conventional density about $406 \text{ kg}\cdot\text{m}^{-3}$ shall be expected, if the conserved object undergoes pre-treatment with PEG 300 and the uptake of polyglycol is from about 22 to 37% of oven-dry wood mass.

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