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Original article

Dimensional changes of waterlogged archaeological hardwoods pre-treated with aqueous mixtures of lactitol/trehalose and mannitol/trehalose before freeze-drying

Leszek Babiński *

Department of Conservation, Archaeological Museum in Biskupin, Biskupin 17, 88-410 Gąsawa, Poland

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ABSTRACT

The article presents research on changes in the dimensions of waterlogged archaeological oak and beech wood pre-treated with aqueous solutions of either a mixture of lactitol and trehalose or a mixture of mannitol and trehalose, and then vacuum freeze-dried or dried with the use of the conventional air-drying method. Uptake of impregnants, shrinkage and moisture content in wood after freeze-drying, and changes in dimensions and moisture content in all modified and dried wood samples after its seasoning in the air at relative humidity 50% and temperature of 18 °C were determined. It was shown that even at a low uptake of lactitol/trehalose or mannitol/trehalose mixture and vacuum freeze-drying, a considerable reduction in the shrinkage of the wood under research could be obtained.

1. Introduction and research aims

During conservation of waterlogged archaeological wood a part of the water filling the cell walls and lumens is replaced with an appropriate chemical compound, which reduces dimensional contraction and deformation upon drying and strengthens the structure of an object being conserved [1,2]. However, not only does the type and required amount of the impregnant applied depend on the species and the degree of wood decomposition, it also depends on the method of its drying.

Among the safest drying methods regarding waterlogged archaeological wood one can count vacuum freeze-drying [3]. It allows for a considerable reduction in wood shrinkage, cell wall collapse, deformation and cracks. Additionally, vacuum freeze-drying of waterlogged wood does not require an extensive uptake of chemicals, as in the case of the conventional air-drying method. It is also widely acknowledged that treatment of wood with solutions of low concentration decreases both consumption of the stabilising agent and the energy required to heat more concentrated solutions.

Pre-treatment of archaeological wood with an impregnant, preceding its vacuum freeze-drying, is most often performed with the use of polyethylene glycols (PEGs) [3–5]. The optimum uptake of polyglycol(s) enables high indices of dimensional stabilisation of wood to be obtained [6–10]. However, the main disadvantage of a polyglycol is the high hygroscopicity of wood treated with low molecular weight PEG (such as PEG 200, PEG 300 or PEG 400). Application of polyglycols with a lower hygroscopicity and a bigger molecular weight is possible only in the case of conservation of highly decomposed wood.

Due to the high hygroscopicity of wood treated with low molecular weight PEGs, some attention has been paid to the possibility of replacing them with sucrose or sugar alcohols. From among the numerous sugar alcohols, mainly mannitol and lactitol were used for conservation of waterlogged archaeological wood [11–14]. Wood impregnation with lactitol was followed by heat drying at 50 °C at atmospheric pressure in order to crystallize exclusively lactitol dihydrate and avoid the formation of lactitol trihydrate, which causes high volume change under crystallization and therefore may damage the weak cell walls of the degraded wood [12,13]. It was shown that wood treated with these substances is less hygroscopic and that changes in its dimensions may be small. Problems with the development of crystals of lactitol and swelling of wood that appeared during drying were solved by adding trehalose [15]. The efficiency of conservation of archaeological wood treated with mannitol and polyethylene glycol and then freeze-dried was examined by Imazu and Nishiura [16]. Results of the research performed

* Tel.: +48 52 302 50 25; fax: +48 52 302 54 20.
E-mail address: leszek.babinski@wp.pl

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by Jones et al. [17] prove that due to its relatively high freezing point mannitol could be used for dimensional stabilisation of freeze-dried archaeological wood.

The current research aimed to identify the possibility of an efficient conservation of waterlogged archaeological hardwoods with the use of pre-treatment with lactitol/trehalose and mannitol/trehalose mixtures and vacuum freeze-drying, as well as making a comparison of changes in the dimensions of wood treated in this way with the results obtained after drying the pre-treated wood with the conventional air-drying method.

2. Materials and methods

The research was done on samples of waterlogged oak wood (*Quercus* sp.) and samples of waterlogged beech wood (*Fagus silvatica* L.) cut out of construction elements drawn from terrestrial archaeological sites. The samples of oak wood came from a cottage in Szczecin (Poland), dated back to the 14th c., and the samples of beech wood from a well casing in Pfettrach (Germany), coming from the 10th c. Wood properties and dimensions of the samples used in the experiment are presented in Table 1.

Maximum moisture content, basic density and loss of wood substance were calculated according to the following equations:

$$MMC = 100\,(m_{ww} - m_{dw})\,/m_{dw}$$

where: MMC: maximum moisture content (%), m_{ww}: mass of waterlogged wood (water + wood) (g), m_{dw}: mass of oven-dried ($103 \pm 2\,^{\circ}C$) wood (g).

$$BD = m_{dw}/V_{ww}$$

where: BD: basic density (g/cm^3), m_{dw}: mass of oven-dried ($103 \pm 2\,^{\circ}C$) wood (g), V_{ww}: volume of waterlogged wood (cm^3).

$$LWS = 100\left(BD_f - BD\right)/BD_f$$

where: LWS: loss of wood substance (%), BD_f: basic density of fresh (non-degraded) wood (0.577 g/cm^3 for oak wood and 0.578 g/cm^3 for beech wood [18]), BD: basic density of archaeological (degraded) wood (g/cm^3).

The waterlogged wood samples were immersed in 5% aqueous solutions of the mixture of lactitol and trehalose in the ratio 9:1 (w/w) (4.5 g lactitol + 0.5 g trehalose + 95 g water) or the mixture of mannitol and trehalose in the ratio 9:1 (w/w) (4.5 g mannitol + 0.5 g trehalose + 95 g water). Concentrations of the solutions were increased every three weeks by 5%. In the final stage of the impregnation, the tested samples were treated with the following solutions of lactitol/trehalose (LT) mixture: 10% (impregnation marked as LT10), 20% (LT20), 30% (LT30) (two series of samples), and 45% (LT45). The final concentrations of the solutions of mannitol/trehalose (MT) mixture were: 10% (impregnation marked as MT10), 20% (MT20), and 25% (MT25) (two series of samples). Tested variants of wood conservation are summarised in Table 2. Due to the lower solubility of mannitol, 30 and 45% aqueous solutions of mannitol/trehalose were not prepared. In each variant, four wood samples with four stainless steel pins inserted in the dominant surface of each sample were treated. All the samples were treated at the ambient temperature (about 18 °C) for 30 weeks – independent of the time of obtaining the assumed final concentration. The solutions were prepared taking into consideration the quantity of water in the container with the samples and the water content in the samples.

The samples treated with solutions of the final concentration ranging from 10 to 30% (LT10, LT20, LT30, MT10, MT20, and MT25) underwent freezing for 5 days at the temperature −27 °C, and then they were vacuum freeze-dried (drying marked as FD; Table 2) for 4 days. The final air pressure in the vacuum chamber was 5.5 Pa.

The remaining samples (second series of samples treated with 30% solution of the mixture of lactitol and trehalose, samples treated with 45% solution of the mixture of lactitol and trehalose, second series of samples treated with 25% solution of the mixture of mannitol and trehalose as well as the untreated control samples) were dried in the air (drying marked as AD; Table 2) at relative humidity (RH) about 60% and temperature of about 18 °C.

After drying (FD or AD), all samples were seasoned to a constant mass (equilibrated) in the air at the relative humidity 50% and the temperature of 18 °C. The examined samples were weighed and the distances between the pins were measured before impregnation, after freeze-drying, and after wood equilibration, with an accuracy of 0.01 g and 0.01 mm, respectively.

After equilibration, all tested wood samples were oven-dried to constant mass at a temperature of $103 \pm 2\,^{\circ}C$ in order to determine their uptakes of impregnants and moisture contents. The uptakes and moisture contents in the impregnated wood directly after finishing of freeze-drying and then after seasoning of the samples were calculated according to the following equations:

$$U = 100\,m_i/m_{dw} = 100\,(m_{dw+i} - m_{dw})\,/m_{dw}$$

$$m_{dw} = m_{ww}/\,(MMC + 1)$$

where: U: uptake of impregnant (%), m_i: mass of impregnant (g), m_{dw}: mass of oven-dried ($103 \pm 2\,^{\circ}C$) wood (g), m_{dw+i}: mass of oven-dried ($103 \pm 2\,^{\circ}C$) wood treated with impregnant (g), m_{ww}: mass of waterlogged wood (g), MMC: maximum moisture content in wood (determined on control sample) (g/g).

$$MC = 100\,m_{wat}/m_{dw+i}$$

where: MC: moisture content in wood treated with impregnant (%), m_{wat}: mass of water in wood treated with impregnant (g), m_{dw+i}: mass of oven-dried ($103 \pm 2\,^{\circ}C$) wood treated with impregnant (g).

Changes in the dimensions in the main anatomical directions of the wood samples were determined on the basis of measurements of the distances between four pins inserted in each sample and presented as a shrinkage of wood from the condition of the maximum moisture content to the condition immediately after freeze-drying, and to the condition of reaching equilibrium moisture content at the relative humidity of 50% and the temperature of 18 °C. Shrinkage of wood was determined in the tangential (samples CS and samples T), radial (samples CS) and longitudinal (samples T) directions, in accordance with the following equation:

$$\beta = 100\,(l_0 - l_1)\,/l_0$$

where: β: linear shrinkage of wood (%), l_0: initial dimension of wood (in waterlogged condition) (mm), l_1: final dimension of wood (after freeze-drying or equilibration) (mm).

Shrinkage of treated and untreated wood, determined at RH = 50% and T = 18 °C, were compared with the use of Anti-Shrink Efficiency (ASE), calculated according to the following equation:

$$ASE = 100\left(\beta_0 - \beta_1\right)/\beta_0$$

where: ASE: anti-shrink efficiency (in the tangential: ASE_T, radial: ASE_R or longitudinal direction: ASE_L) (%), β_0: linear shrinkage of untreated wood (%), β_1: linear shrinkage of treated wood (%).

3. Results

The uptake of impregnants in oak and beech wood is presented in Table 3. The quantity of lactitol/trehalose or mannitol/trehalose mixture penetrated in the wood increased together with the increase in concentration of the impregnating solution.