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Original contribution

Internal evaluation of impregnation treatment of waterlogged wood; relation between concentration of internal materials and relaxation time using magnetic resonance imaging



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ABSTRACT

The purpose of this study is to clarify the degree of impregnation resulting from treatment of internal waterlogged wood samples using MRI. On a 1.5 T MR scanner, T_1 and T_2 measurements were performed using inversion recovery and spin-echo sequences, respectively. The samples were cut waterlogged pieces of wood treated with various impregnation techniques which were divided into different concentrations of trehalose $(C_{12}H_{22}O_{11})$ and polyethylene glycol (PEG; $HO-(C_2H_4O)_n-H$) solutions. Then these samples underwent impregnation treatment every two weeks. From the results, we found that the slope of the T_1 -concentration curve using linear fitting showed the value of the internal area for PEG to be higher than the external area; internal, -2.73 ms/wt% $(R^2 = 0.880)$; external, -1.50 ms/wt% ($R^2 = 0.887$). Furthermore, the slope of the T_1 -concentration curve using linear fitting showed the values for trehalose to have almost no difference when comparing the internal and the external areas; internal, -2.79 ms/wt% ($R^2 = 0.759$); external, -3.02 ms/wt% ($R^2 = 0.795$). However, the slope of the T₂-concentration curve using linear fitting for PEG showed that there was only a slight change between the internal and the external areas; internal, 0.26 ms/wt% ($R^2 = 0.642$); external, 0.18 ms/wt% ($R^2 = 0.642$); 0.920). The slope of the T_2 -concentration curve did not show a change in linear relationship between the internal and the external areas; internal, 0.06 ms/wt% ($R^2 = 0.175$); external, -0.14 ms/wt% ($R^2 = 0.043$). In conclusion, using visualization of relaxation time T_1 , it is possible to obtain more detail information noninvasively concerning the state of impregnation treatment of internal waterlogged wood.

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1. Introduction

It is very important that we excavate materials to preserve cultural heritage for future generations. Many excavated articles are found as waterlogged wood artifacts. Waterlogged wood artifacts decay rapidly after excavation due to loss of internal moisture by drying [1]. To preserve waterlogged wood after a recovery operation, impregnation with high molecular weight material, e.g., trehalose ($C_{12}H_{22}O_{11}$) and polyethylene glycol (PEG; HO-(C_2H_4O)_n-H), is often used [2,3]. The preservation process depends on the physical parameters of the waterlogged wood (such as porosity, water content, and wet and dry bulk densities plus the density of the cell wall material) mass, volume, and sampling size also play an important role in the process [4]. After

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consideration of the characteristics, in general the process leads to impregnation in ascending order of concentration over a selected time period. Moreover, the degree of impregnation also differs according to species, condition of wood, location of excavation, and material used for impregnation. It is then important to measure quantitatively the inner impregnation or the degree of crystallization of the wood. The period of impregnation was for the most part determined from a past report [5]; this was done because complex parameters need to be considered as described above. Therefore, the process needs to be evaluated of an individual basis.

Recently archaeologists have started to use X-ray photography [6], X-ray computed tomography (X-CT) [7,8], and ultrasonic reflectoscope (US) [9] to observe the internal environments of artifacts. These techniques can also be used to non-destructively visualize the preservation process. Nuclear magnetic resonance (NMR) and magnetic resonance imaging (MRI) have also been used to be study the preservation process. One MRI study demonstrated that the determination of moisture

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Table 1

Concentration of PEG and trehalose.

PEG		Trehalose	
Weight percent (wt%)	Mole percent (mol%)	Weight percent (wt%)	Mole percent (mol%)
20 wt%	0.15 mol%	30 wt%	2.00 mol%
40 wt%	0.39 mol%	40 wt%	3.08 mol%
60 wt%	0.86 mol%	50 wt%	4.55 mol%
80 wt%	2.27 mol%	60 wt%	6.67 mol%
100 wt%	100 mol%	70 wt%	10.01 mol%

content of wood was comparable in accuracy and reliability to gravimetric methods, however, waterlogged wood was not studied [10]. Another study reported on the preservation process used for archaeological waterlogged wood samples by PEG impregnation using ¹³C NMR spectroscopy [11]. Here, it was found that at high PEG concentrations the polymer accumulates in the remaining free volume with more but less tightly bound molecular interactions with the residual wood components. The other study focused on 1-D ¹H NMR profile analysis of diffusion of D₂O in archaeological wood [12]; from this study it is notable that the diffusion was highest in the longitudinal orientation with respect to the wood structure and successively smaller in the tangential and radial directions. Consequently, using MRI, it is predicted that a higher contrast image can easily be acquired for nonmetallic samples when compared to the other methods. These evaluation methods did not attempt to the directly achieve visualization of the degree of impregnation of a high molecular weight material. Additionally, signal intensity (SI) analysis would not only result in absolute, but relative evaluation because of various systematic factors, e.g., receiver gain, radio frequency, and setting of subject for inhomogeneity [13]. Thus, we focused on the physical value of relaxation time using the nuclear magnetic resonance phenomenon.

The objective of our study was to quantitatively evaluate the degree of impregnation of high molecular weight material, e.g., trehalose and PEG impregnated into waterlogged wood using MRI.

2. Materials and methods

2.1. Subject

The sample used in this study was waterlogged Abies wood in the family Pinaceae. The excavated wood was cut in half. Next, the wood was divided into seven samples. Each part was impregnated with different concentrations of PEG (HO-(C₂H₄O)_n-H, 3100 g/mol, PEG#4000, NOF Corp., Tokyo, Japan) and trehalose [C12H22O11,378 g/mol (dihydrate), TREHA, Hayashibara Corp., Okayama, Japan]. The study date was set at 14 weeks. Each sample was impregnated every two weeks at different starting points. At each time point, images of the wood were acquired. For the final preparation, PEG mixed with water at 20, 40, 60, 80, 100 weight percent (wt%), and after drying treatment, and trehalose mixed with water at 30, 40, 50, 60, 70 wt% and after drying treatment, respectively. Here, molecular concentration wt% is mainly described in this paper; in many cases, scientists of conservation science use this denotation when mixing high molecular weight material. Table 1 shows each concentration wt% and mol% for PEG and trehalose used in this study. This study evaluated the images at the final time point. Fig. 1 shows a photograph and a schematic of each part of wood at different concentration of PEG and trehalose.

2.2. MR imaging

All waterlogged wood studies were performed on a 1.5 T MR scanner (Vantage, Toshiba Medical Systems Corp., Otawara, Japan). T_1 measurements were performed using a multiple inversion recovery (IR) method. The imaging parameters were inversion time (TI), 15, 30, 45, 60, and 75 ms; echo time (TE), 12 ms; repetition time (TR), 8485 ms; field of view (FOV), 300 mm; matrix size, 256 × 256; slice thickness, 5 mm; slice interval, 12 mm. T_2 measurements were performed using a multiple spin-echo (SE) method. The imaging parameters were TE, 15, 30, 45, 60, and 75 ms; TR, 12,120 ms; FOV, 300 mm; matrix size, 256 × 256; slice thickness, 5 mm; slice interval, 12 mm. These slice orientations were identical axial slices.

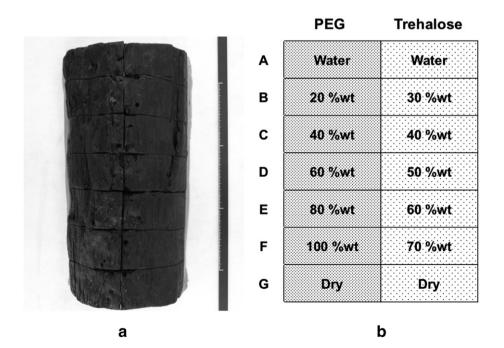


Fig. 1. Photograph and schematic of parameters for waterlogged wood in this study. The sample was waterlogged wood of species Abies in the family Pinaceae. (a) The photograph shows an overview from the top. A waterlogged wood was cut into 14 segments. (b) A schematic shows the breakdown of 14 segments after impregnation treatment. Each segment was impregnated with different concentration of PEG or trehalose solutions (except of A section). Here, 'Dry' means after drying treatment.

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