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Autor: Weber, Urs M. / Rosenthaler, Kurt J.

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Wet archaeological wood treated with sucrose

Preliminary test series

by URS M. WEBER and KURT J. ROSENTHALER

Introduction

This study was part of the European Laboratory Network (E.L.N.) Project "Sucrose" (HOFFMANN et al. 1993)[4], in which four European laboratories collaborated, coordinated by Nucléart, Grenoble, France. The preservation of waterlogged wood using sucrose has already been in practice for years in several, mainly Eastern European countries (DUMKOW and PREUSS 1990[2], KAZANSKAYA 1990[5], MORGÓS et al. 1987[7]). These authors considered the comparatively low price of sugar, its regeneratable basic materials, as well as its more or less non-polluting character as the main advantages of the sucrose method. However, neither the stabilization efficiency of sugar treatments in respect to the three anatomical directions of fibre has been studied thoroughly nor the possible biodegradation by wood-boring insects. Moreover, up to now, we have only had little experience with the disinfection of sucrose solutions which satisfy today's standards in environmental protection.

In order to increase the know-how concerning these issues, samples of as many native wood species as possible were selected by all participating laboratories and treated according to a standardized procedure.

The objectives were to

1. determine the range of application of the sucrose method in respect to the state of degradation, wood species and place of origin,
2. test low-impact antibiotic treatments on sucrose solutions,
3. study a possible impact of wood destroying organisms, especially insects, on sucrose-stabilized waterlogged wood. This third part of the study was carried out by NOLDT (1993)[8]; the somewhat astonishing as well as encouraging conclusion of his bioassays: sugar treated samples were less attractive for wood destroying termites of the genus *Reticulitermes* than untreated ones.

The present paper represents an updated as well as more comprehensive version of the Swiss part of the E.L.N. project. Unfortunately all our samples were heavily degraded, thus we were not able to evaluate the success of sucrose stabilization for less degraded wood. For more details on this issue refer to HOFFMANN et al. (1993)[4].

1. Methods and material

The methods used in this study were basically consonant with the three collaborating laboratories in Bremerhaven (Germany), Cartagena (Spain) and Grenoble (France); they are fully described in HOFFMANN et al. (1993)[4].

All samples (cross-sections and boards) were treated at room temperature (~20 °C). The initial sucrose concentration of 20% was increased in two/three steps to a final concentration of 50%/67% within 21 and 29 weeks respectively. They were dried at 70% rh for five weeks.

The procedures which deviated from the E.L.N. project directions are summarized below.

Sterilization of samples: no sterilization was executed in order to keep as close as possible to realistic procedure conditions.

Sucrose solutions: in addition to the samples preserved in solutions of 50 wt% final sucrose concentration, we stabilized a second series of samples in saturated solutions. At room temperature (20 °C) a saturated sugar solution is reached at a concentration of 67 wt% sucrose.

code	species	place of origin	age (B.C.)	water content (%)
ACE	Acer sp.	Greifensee-Böschen	~1050	~1030
ALN	Alnus glutinosa	Greifensee-Böschen	~1050	~1130
COR	Corylus avellana	Greifensee-Böschen	~1050	~1040
FAG	Fagus silvatica	Greifensee-Böschen	~1050	~ 900
FRA	Fraxinus excelsior	Greifensee-Böschen	~1050	~1170
MAL	Maloideae	Greifensee-Böschen	~1050	~ 970
QUE	Quercus sp.	Greifensee-Böschen	~1050	~ 630

Table 1

Disinfection of sucrose solutions: instead of copper ions (Cu++), which did not appear to be an effective biocide, we used isothiazolinones. All solutions were disinfected.

Drying: the samples were dried without cleaning the surplus sugar from the surface beforehand.

Analysis of wood components, hygroscopic tests: for lack of time these tests could not be carried out.

The study material originated from an excavation of a lake-dwelling settlement in the Greifensee, Switzerland. All samples were heavily degraded, the water content ranged from between 630% and 1170% (Table 1). From each species – the ideal basic form was a cylinder (20 cm long, 10 cm in diameter) – a series of 9 cross-sections (1 cm thick), 6 tangential and 6 radial boards (length: ~10 cm, width: 4–6 cm, thickness: 0.5–0.8 cm) were cut.

2. Results

2.1. Impregnation and drying

The change of weight of the samples during impregnation and drying was depicted graphically (Fig. 1). Slight irregularities in the change of the weight were probably due to measurement errors, the standard deviation (st. dev.) being 0.16 g.

The weight of cross-sections increased proportionately to time except for one sample of oak which was stabilized in a 50% solution (QUE3–50%). After approximately one week, the intake of sugar ceased, whereas the above-mentioned exception took up sugar from the treatment solution during the whole period of impregnation. As a result, this sample swelled and broke into pieces. An adequate answer for this peculiar behaviour can hardly be given as all oak cross-sections were cut from the same piece of wood.

On the whole, impregnation rates of the boards did not vary significantly from those of the cross-sections, i.e. saturation of the samples with sugar was reached after one week as well. Moreover, impregnation of radial and tangential samples took about the same time. The heavy degradation of all samples possibly facilitated diffusion in any fibre direction, thus minimizing the difference between radially and tangentially cut boards. However, two boards diverged from the general course of impregnation: a radial and a tangential sample of oak which were stabilized in a 50% solution (QUE-50% tan1, QUE-50% rad1) constantly resorbed sugar up to a relative weight of nearly 160% (Fig. 1). Like the cross-section sample they swelled and decayed during the treatment. The two other oak samples, cut from the same chunk, behaved regularly.

The drying of the samples revealed no unexpected occurences. After 4 to 5 weeks (20 °C, 70% rh) all cross-sections

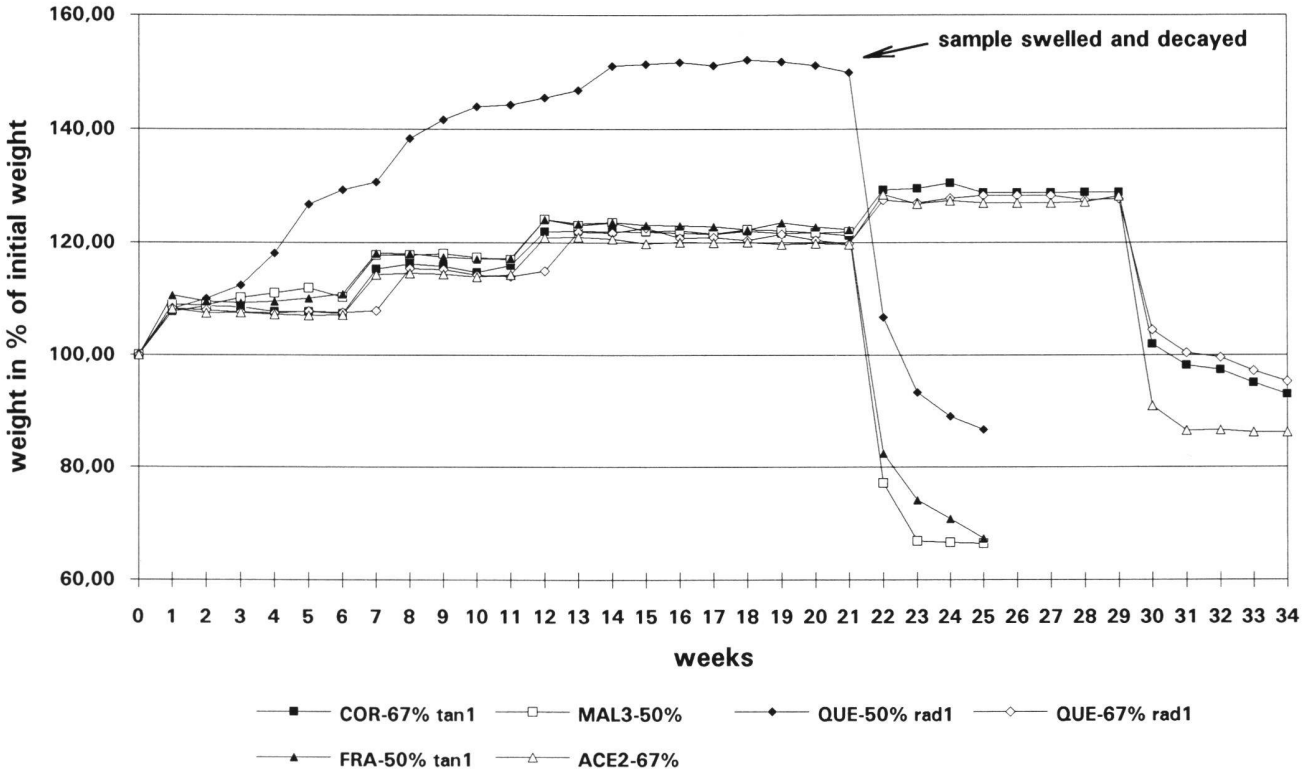

Fig. 1 Change of weight (%) of representative samples during impregnation and drying. The initial weight, measured before the impregnation, equals 100%. The drying period can easily be recognized by the abrupt decrease in weight.
COR = Corylus, MAL = Maloideae, QUE = Quercus, FRA = Fraxinus, ACE = Acer
tan = tangential, rad = radial, no supplement = cross-section

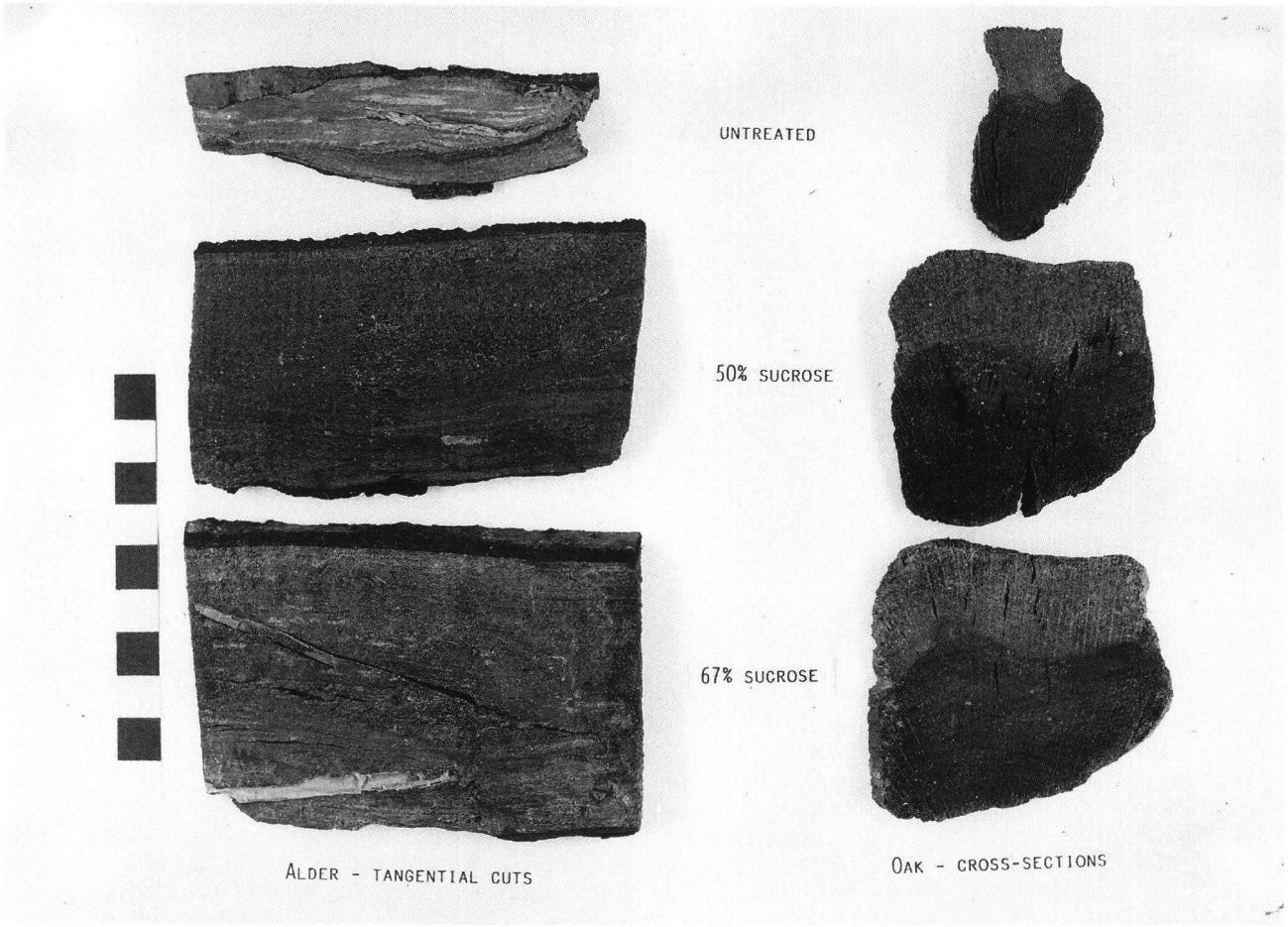

Fig. 2 Dimensions of untreated air-dried samples and samples stabilized in 50% or 67% sucrose solutions. The three comparison samples originated from the same piece of wood. Basically, the effect of sugar conservation on the stabilization of heavily degraded wood is a positive one; however, there is an obvious difference between the 50% and 67% samples.

were "dry", i.e. they did not lose anymore weight. The boards should have been dried longer because their predominating surfaces (tangential, radial) inhibited the rapid evaporation of residual water.

As a rule, dry samples are very brittle and must be handled with care.

2.2. Stabilization

Anti-shrinkage values of samples stabilized in 67% sucrose solutions were far better than those achieved in 50% solutions; this difference can be observed with the naked eye (Fig. 2).

Using 50% solutions, the average shrinkage of cross-sections after drying was 27.7% ranging from 21 to 33%; the average linear shrinkage of boards was 9.7% within a range of –16.9 to 25%. These results were far from acceptable while those achieved in 67% solutions at least partly reached the standards in conservation: the average cross-section shrinkage was 8.9% ranging from 0 to 24%. The average linear shrinkage of boards was 2.5% in the radial direction and 4.4% in the tangential direction within a range of 0 to 6.5% and 0 to 10.2% respectively.

The mean ASE (anti-shrink efficiency) of cross-sections treated in 50% solutions was 68% (62–75%) while samples treated in 67% solutions reached a mean ASE of 89% (74–100%).

Comparing the success of stabilization on the level of species, differences became evident (Figs. 3 and 4). While all cross-sections treated in 50% solutions exhibited inacceptably high shrinkage values, significant differences of shrinkage between the species were found among the 67% samples (Fig. 3). Although the water content of the untreated alder was the second highest (~1130%), cross-section shrinkage was fairly low. Yet the Maloideae samples, water content <1000%,

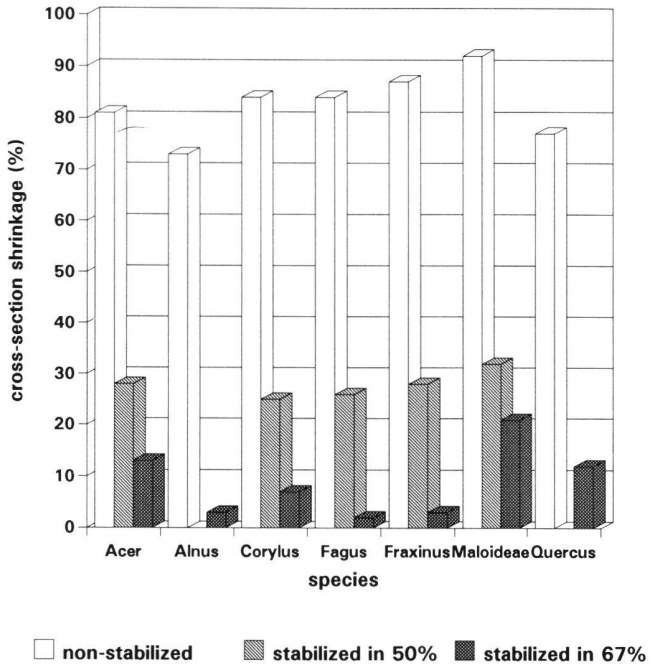

Fig. 3 Average cross-section shrinkage (%) of stabilized and non-stabilized samples at 40% rh (n = 3 per species and treatment). Due to a large number of cracks, the values of the alder and oak treated in 50% sucrose could not be calculated with satisfying accuracy; thus they were deleted.

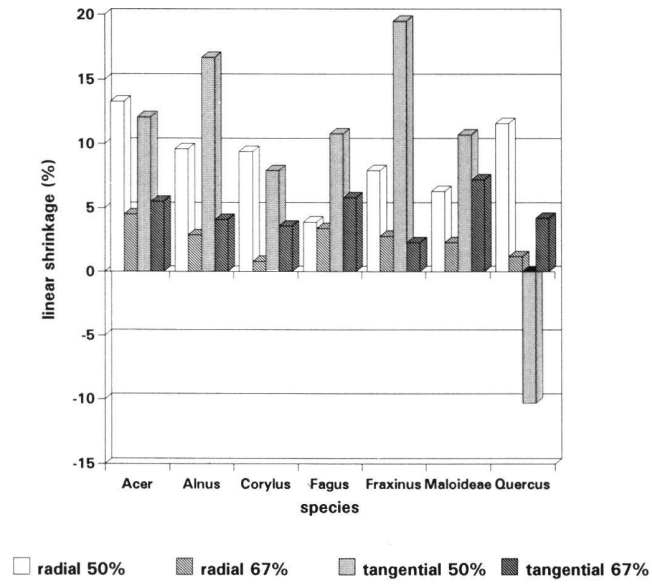

Fig. 4 Average shrinkage (%) of stabilized and non-stabilized boards in radial and tangential fibre direction (n = 2 per species, treatment, and direction).

shrank about 20%, which was the highest shrinkage value observed. The same statement can be made for the air-dried Maloideae samples. These contradictory stabilization effects can possibly be explained by differences in wood anatomy. Intercellular exchange of liquids in alderwood is probably more efficient than in Maloideae wood because the number of pits in alder vessels is higher (SCHWEINGRUBER 1990)[10], thus facilitating the permeability of sucrose. However, the very long period of impregnation should have diminished this effect.

All in all we obtained good cross-section stabilization of the alder, hazel, beech and ash, a less satisfactory stabilization of the maple and oak, and a bad one of the Maloideae samples.

Comparing the boards, the distinct difference between a 50% and 67% treatment became obvious as well (Fig. 4). Boards preserved in 50% sucrose solutions did not satisfactorily stabilize (exception: the radial beech samples). Yet, in contrast to the cross-sections, we obtained some variation between the species and anatomical cuts. The most extreme deflections, in either shrinkage or swelling, happened in a tangential direction (alder, ash, and oak). A surprisingly low shrinkage was shown by the Maloideae samples.

Those boards treated in a 67% solution achieved, as could be expected, better shrinkage values. Again, the samples with predominating tangential surfaces shrank more than those with predominating radial surfaces (except for the ash). According to WAGENFÜHR (1989)[11], the pits in dicotyledonous wood are concentrated on the radial cell walls, thus enabling an easier exchange of liquids in a tangential direction. Therefore, impregnation rates in tangential boards are lower than in radial ones. Yet by tracing the course of impregnation this theory cannot be confirmed, because the time-lapse between the first two measurements was too long. Longitudinal shrinkage can be ignored.

Observations made of the impregnated and dried samples revealed additional features such as cracks and deformations; the different stabilization efficiency of 50% and 67% sucrose solutions became apparent (Fig. 5).

The cross-sections impregnated in a 67% solution showed no cracks with the exception of the Maloideae samples. However, all cross-sections, including the 67% samples, began to deform when drying on a grating, although they were frequently turned over (Fig. 6). As a result of these deformations an unacceptable number of quite big cracks formed on the bottom of the 50% samples (Fig. 7).

The optical appearance of the boards treated in 67% sucrose was fairly good compared to the 50% samples. The number of cracks in tangential boards was higher than in radial ones in accordance with their lower ASE values. Root canals of reed (*Phragmites australis*) seemed to facilitate the initiation of cracks during the drying process. The worst results of samples preserved in 67% sucrose are listed below.

Cross-sections:

- Alder (ALN1–67%): a small piece of the sample broke off.
- Beech (all samples): two, six, and twelve small cracks formed respectively.
- Maloideae (all samples): comparatively heavy shrinkage.

One big crack per sample.

Boards:

- Alder (ALN-67%tan1): seven cracks appeared.
- Ash (FRA-67%tan2): one edge broke off.
- Beech (FAG-67%tan1): one crack in the centre of the board.
- Beech (FAG-67%tan2): sample broke into two pieces.
- Hazel (COR-67%tan1): sample broke into three pieces.
- Hazel (COR-67%tan2): sample broke into two pieces.
- Maple (ACE-67%tan2): remarkable, unusual shrinkage. A major crack in the centre was induced by a *Phragmites* root. Sample broke into three pieces.
- Maple (ACE-67%rad2): sample broke into two pieces.
- Oak (QUE-67%tan2): sample broke into two pieces.
- Oak (QUE-67%rad2): sample broke into three pieces. The largest piece exhibited five cracks.
- Maloideae (MAL-67%tan1): sample broke into two pieces.
- Maloideae (MAL-67%tan2): one big crack in the centre; remarkable shrinkage.

On the whole, 7 of the 21 cross-sections and 12 of the 28 boards stabilized in 67% sugar solutions were obviously damaged. 11 of the 12 boards affected were tangential ones.

2.3. Disinfection of sucrose solutions

After approximately two weeks, bluish crystals began to form on the edge of the surface of the 20% sucrose solutions disinfected with Cu^{2+}-ions. One week later fermentation and the appearance of fungi was observed in both the disinfected and non-disinfected baths. Perhaps the copper ions were deactivated by complex-formations of copper ions and sugar molecules.

As soon as copper ions proved to be ineffective as a biocide, we began using isothiazolinones to disinfect the sucrose solutions. Thereafter no contamination was observed in any bath during any of the different treatments, although we did not sterilize the wood prior to the treatment. Moreover, isothiazolinones did not have a negative effect on sucrose crystallization. A specific concentration of about 15 ppm active ingredients proved to be sufficient under laboratory conditions (the pH of the baths ranged from 5 to 5.5). Yet, in practice, this dosage is usually only effective for a short time, because, for economical reasons, the volume ratio between wood and solution is usually higher than during test conditions; in addition, the probability of contamination under practical working conditions is likely to be higher. Thus, the concentration of antibiotics would have to be higher.

The activity of isothiazolinones is constantly decreasing because of consumption of the active molecules. Therefore the initial biocide dosage has to be topped up in case it drops below the level of antibiotic efficacy; an appropriate colour test is available. Since isothiazolinones contain chlorine, we are not fully convinced that this compound is as harmless to aqueous ecosystems as is commonly supposed.

2.4. Aesthetic appearance of stabilized samples

Compared to polyethylene glycol- (PEG-) stabilized archaeological wood, the appearance of the sugar-treated samples met

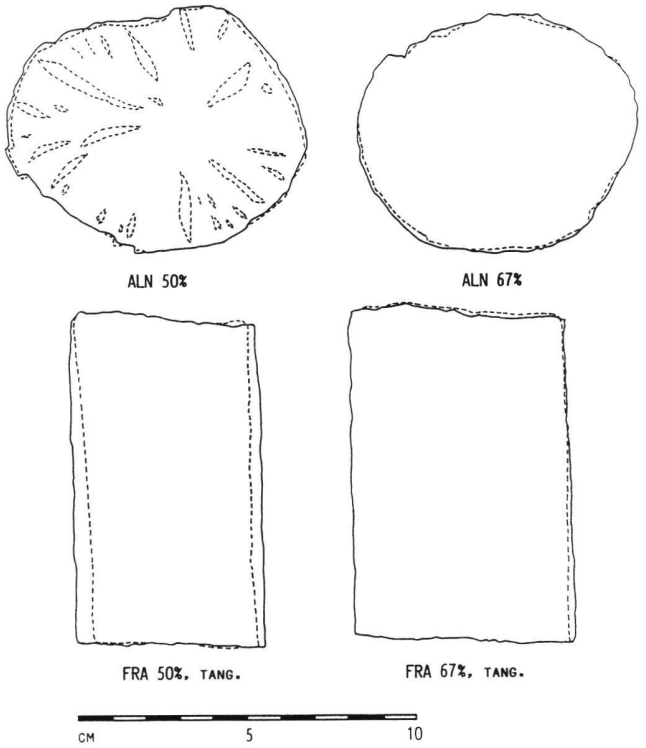

Fig. 5 Contours of samples before and after the stabilization in a 50% or 67% sucrose solution (a choice of representative samples only).
Continuous line: before stabilization
Broken line: after stabilization and drying

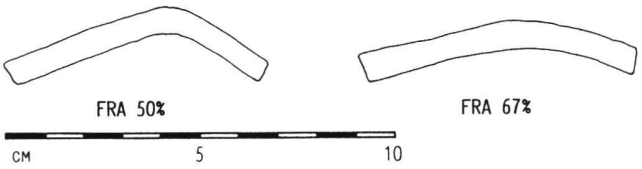

Fig. 6 After drying: typical deformations of cross-sections treated in 50% or 67% sucrose solutions.

aesthetic demands, i.e. the characteristic colour and structure of the wood usually could be recognized. The colours of the beech, maple and Maloideae were hard to tell apart, yet they still could be identified by their wood structure. The surface of the samples felt dry. No sample showed sucrose crystallization on the surface, although they had not been cleaned after the impregnation.

3. Discussion

A major handicap in the use of small samples for studying the stabilization of waterlogged wood is possibly the destruction of the internal wood structure, i.e. the disturbance of the axiom of constant tension (MATTHECK 1992[6]). Better said, more realistic results could probably be obtained by means of

Fig. 7 The two sides of a cross-section stabilized in a 50% sucrose solution. The concave bottom side shows a number of major cracks while only a few are visible on the convex upper side.

using wood samples of an adequate, more or less "natural" ratio between the three anatomical directions. It goes without mentioning that it is often impossible to get samples of a larger size.

In contrast to results published by DUMKOW and PREUSS (1990)[2], GROSSO (1981)[3], MORGÓS et al. (1987)[7], and PARRENT (1985)[9], we cannot recommend a final sucrose concentration of 50% (g/g) for heavily degraded wood, at least not for unheated use; the aquired ASE of 68% on an average is simply not acceptable. However, an average ASE of 89% obtained in 67% (g/g) sucrose solutions can be noted as fairly good. For the stabilization of less degraded wood lower concentrations are probably sufficient. In our laboratory recently, a water tube (4.5 m × 0.1 m^2, *Pinus silvestris*, u_{max} ~160%) was successfully preserved in a 40% sucrose solution.

Partly contradictory stabilization effects of differently degraded wood species leads to the presumption that the water content of untreated archaeological wood does not provide enough information to thoroughly depict the grade of degradation. Thus, wood chemical analysis must be carried out in order to determine the percentage of cellulose and lignine. In addition, the wood structure of a species must be taken into account, if the pathways of diffusion are to be understood. It can be assumed that the divergent stabilization capacity of tangential and radial boards is caused by different intercellular exchange rates due to variations in cell construction. Yet the above-mentioned destruction of the internal equilibrium of tensions by cutting a trunk into a number of thin pieces might explain this discrepancy as well. More investigation is necessary to clarify the pathways of diffusion as well as the influence of specific wood structures.

The different behaviour of the seven species we worked with did not come as too much of a surprise, although this could have been implied by the more or less equal grade of degradation.

29 weeks of impregnation (up to 67% sucrose) proved to be sufficient to stabilize the samples; however, tests should be conducted with larger objects in order to optimize and/or minimize the duration of impregnation.

Isothiazolinones proved to be an effective biocide to disinfect sucrose solutions. Still, further investigations should be initiated in order to find cheaper and, from an ecological point of view, less questionable substances. Initial experience with benzoic acid was gained by BECKER et al. (1991).[1] A series of sterilization tests comparing the effects of a variety of organic and inorganic compounds on different sucrose solutions is presently being conducted at the Swiss National Museum in collaboration with the Swiss Federal Laboratories for Materials Testing and Research (EMPA).

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SUMMARY

Samples from seven species of heavily degraded waterlogged archaeological wood (u_{max} = 630–1170%) were preserved in unheated sucrose solutions up to 50 wt% and 67 wt% respectively. On the whole, an acceptable stabilization was only obtained in the 67% solutions, average residual cross-section shrinkage being 8.9% within a range of 0 to 24%, corresponding to an anti-shrink efficiency (ASE) of 89% (74–100%). The average ASE for cross-sections treated in 50% solutions was only 68% (62–75%). Tangential boards exhibited a more intensive shrinkage than radial ones. The stabilization of the seven species was successful in varying degrees; the best results were obtained for alder, the worst ones for *Maloideae* samples. After drying, the samples looked quite natural, i.e. the specific colour and structure of the wood could be identified. Disinfection of the sucrose solutions with Cu^{2+}-ions proved to be ineffective. Using isothiazolinones (~15 ppm active ingredients) microbiological contamination was no longer observed.

ZUSAMMENFASSUNG

Stark abgebaute Proben archäologischen Nassholzes von sieben Baumarten (u_{max} = 630–1170%) wurden in ungeheizten wässrigen Lösungen von 50% (g/g) beziehungsweise 67% (g/g) Sucrose konserviert. Eine akzeptable Stabilisierung wurde nur in den 67prozentigen Lösungen erreicht, bei einem Querschnittsschwund von 8,9% (0–24%), entsprechend einem Anti-Schwindungsvermögen (= anti-shrink efficiency, ASE) von 89% (74–100%). Das durchschnittliche Anti-Schwindungsvermögen für Querschnittsproben, die in 50%-Lösungen konserviert wurden, betrug nur 68% (62–75%). Tangentiale Proben zeigten einen stärkeren Schwund als radiale. Der Sta-

bilisierungserfolg für die sieben Holzarten war unterschiedlich; die besten Resultate wurden mit Erle *(Alnus sp.)*, die schlechtesten mit Kernobst *(Maloideae)* erreicht. Nach der Trocknung erschienen die Proben mehr oder weniger natürlich, d.h., die holzspezifische Farbe und Struktur war sichtbar. Die Desinfektion der Zuckerlösungen mit Kupfer (Cu^{2+}) -Ionen war nicht erfolgreich. Nach der Beigabe einer Isothiazolinon-Lösung (~15 ppm aktive Substanzen) wurde keine mikrobielle Kontamination der Lösungen mehr beobachtet.

RÉSUMÉ

Des échantillons de sept types de bois archéologiques gorgés d'eau fortement dégradés (u_{max} = 630–1170%) ont été conservés dans des solutions aqueuses non chauffées de saccharose à 50% (g/g) et à 67% (g/g). Une stabilisation acceptable n'a été obtenue que dans les solutions à 67%, par un retrait moyen de la coupe transversale de 8,9% (0–24%), correspondant à une capacité antiretrait (= anti-shrink efficiency, ASE) de 89% (74–100%). La capacité antiretrait moyenne des échantillons conservés dans les solutions à 50% n'était que de 68% (62–75%). Sur les échantillons transversaux on a observé un retrait plus important que sur les échantillons radiaux. Des valeurs de stabilisation variables ont été relevées sur les sept types de bois, les meilleurs résultats ayant été obtenus avec le bois d'aune (Alnus sp.), les moins bons avec le bois de *Maloideae.* Après dessiccation, les échantillons avaient retrouvé un aspect plus ou moins naturel, la couleur et la structure spécifiques du bois étant visibles. La désinfection des solutions de saccharose par des ions de cuivre (Cu^{2+}) n'a donné aucun résultat. L'adjonction d'une solution de isothiazolinone (~15 ppm substances actives) a permis d'éliminer toute contamination microbienne.

RIASSUNTO

Un campionario di sette tipi di legno archeologico saturo d'acqua (u_{max} = 630–1170%) fortemente degradati è stato conservato in soluzioni acquose non scaldate al 50% (g/g), risp. al 67% (g/g) di saccarosio. Una stabilizzazione accettabile è stata raggiunta solo nelle soluzioni al 67%, con un ritiro della sezione trasversale del 8,9% (0–24%) corrispondente ad una capacità di resistenza al ritiro (= anti-shrink efficiency, ASE) del 89%. La capacità media di resistenza al ritiro di ogni campione di sezione trasversale conservato in soluzioni al 50% era pari solo al 68% (62–75%). Il ritiro riscontrato nei campioni trasversali è superiore a quello registrato nei campioni radiali, mentre la stabilità dei sette tipi di legno è risultata variabile. I risultati migliori sono stati ottenuti con il legno d'ontano (Alnus sp.), quelli meno soddisfacenti con il legno di *Maloideae.* Dopo l'essiccazione, i campioni avevano riacquistato un aspetto più o meno naturale ed erano nuovamente visibili il colore e la struttura specifici del legno. La disinfezione della soluzione di saccarosio con ioni di rame (Cu^{2+}) si è rivelata inutile. L'aggiunta di una soluzione di isotiazolinone (~15 ppm sostanze attive) ha consentito di eliminare qualsiasi contaminazione microbatterica delle soluzioni.