The post-treatment deterioration of marine archaeological wood – where to now?

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Abstract

As waterlogged wood is ubiquitous in excavation sites, being used for structures (ships and buildings), tools, personal effects and for decorative artefacts, this material has been the subject of most conservation research and treatment over the past few decades. The development of disfiguring white deposits on treated timbers from the Skuldelev Viking ships from Roskilde Fjord in the late 1970s was initially treated as a minor cosmetic problem. The seriousness of the situation was soon realized when more timbers were affected and the destruction of timber tissue became evident. Similar problems have also been encountered with the *Batavia* timbers from Western Australia, the Shinan ship in Korea and artefacts from the *Mary Rose* in England. Problems associated with the presence of iron corrosion products in treated, formerly waterlogged timbers were further highlighted in 2000 with the development of highly acidic regions on some *Vasa* timbers and associated artefacts. These outbreaks on the *Vasa* were thought to be related to the inability of the climate control system to keep the relative humidity in the gallery below 60%. These post-treatment developments prompted a significant period of focused research and conservation activities, this time involving multi-faceted analyses and the development of strategies to address a problem that has the potential to affect all waterlogged timbers excavated from anaerobic sites. This paper reviews the current situation regarding the role that iron species play in promoting the formation of acidic species and in catalyzing the oxidation of reduced sulfur species, cellulose and polyethylene glycol and the steps being taken to reduce post-treatment deterioration of formerly waterlogged wood. The implications for future conservation treatments of waterlogged wood are discussed.

Introduction

Waterlogged wood is ubiquitous in wet terrestrial or underwater excavation sites, being used for structures (ships and buildings), tools, personal effects and for decorative artefacts. As a result waterlogged wood has been the subject of much conservation research and treatment over the past few decades. The work has been essential because waterlogged wood may be irreversibly damaged by shrinking, cracking, warping and possibly even crumbling into fragments should it be allowed to dry in an uncontrolled fashion (Figure 1).

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Figure 1: Cross-sectional view of conserved (top) and untreated wood (bottom) showing the extent of shrinkage if highly degraded wood dries without treatment (Copyright: Western Australian Museum).

The aims of all conservation treatments should be to stabilize artefacts so that as much of the original material is preserved as possible, the dimensions of the object are retained and the causes of deterioration are ameliorated. Designated treatments should involve minimum intervention and if possible, applied treatments should be reversible or at least retreatable. Although reversibility is often quoted as an important principle for conservation treatments, it is often very difficult to achieve in practice. As long as dimensional change is avoided, the ability to retreat organic objects is enhanced if voids in structures are left unfilled, allowing future access to either solvents or alternative treatment chemicals. The importance of being able to retreat organic archaeological objects is clearly highlighted in the case of alum-treated waterlogged wood that has subsequently become highly acidic (Braovac and

Kutze *in press*). If it was not possible to retreat this wood, highly valuable archaeological objects would be lost forever.

Many different conservation treatments have been applied to waterlogged wood. Most treatments focused on preserving the wood surfaces and dimensions upon drying. From the mid 19th century to the 1960s, heated, saturated alum solutions [$KAl(SO_4)_2$] were the most commonly used treatment for waterlogged wood (Herbst 1861:174-176). The aim was for the alum to displace the water and fill the pores with a material that would solidify when cooled and dried. Unfortunately alum was not able to fully penetrate the wood, was non-uniformly distributed throughout the wood and left it prone to collapse. In addition, over time, many alum-treated objects became highly acidic and deteriorated.

In the early 1960s, the raising of the *Vasa* and excavation of the Skuldelev Viking ships from Roskilde Fjord stimulated further research into waterlogged wood treatment methods. As a result the alum treatment was abandoned and the use of polyethylene glycols (PEG) adopted as the preferred method for the conservation of waterlogged wood. For the past 45 years PEG, applied in different media and with various molecular weight and percentage compositions, has remained the most commonly used treatment for waterlogged wood.

In addition to PEG however, many other techniques and chemicals have been used to impregnate and consolidate waterlogged wood. These include sugars and other polysaccharides (Franguelli 1971:339-346; Imazu and Morgós 1997:235-254; Imazu and Morgós 1999:210-214; Imazu and Morgós 2002:413-428; Zhang, *et al.* 2009:253-269), melamine/formaldehyde (Haas and Muller-Beck 1960:150-158; Wittköpper 1998:277-282), radiation-induced polymerization of vinyl monomers (Braeker and Bill 1979:97-145; Munnikendam 1967:70-75; Tran, *et al.* 1990:217-234), silicone oils (Smith 2003) and acetone-rosin (McKerrell 1972:111-125). Despite the successful application of many of these techniques to waterlogged wood of varying degrees of degradation, none of these methods has overtaken PEG as the main treatment method for these archaeological materials.

Post-treatment problems have surfaced however, in PEG-treated archaeological wood. The development of highly acidic regions in the Skuldelev Viking ships (Jespersen 1989:141-152), the *Batavia* (Ghisalberti, *et al.* 2002:281-307), the *Vasa* (Sandström 2003), the Shinan ship (Kim and Yang 2004) and on artefacts from the *Mary Rose* (Wetherall, *et al.* 2008:1317-28) highlighted issues related to display environments and the complex chemical reactions that continue in waterlogged wood post-treatment. This paper examines the situation with regard to PEG-treated wood, presents the results of the latest research and discusses the implications with respect to the future conservation and research requirements of waterlogged wood.

The nature of the problem

The acid salts problem associated with PEG-treated wood was first highlighted in the late 1970s when disfiguring white deposits formed on treated timbers from the Skuldelev Viking ships recovered from Roskilde Fjord. The issue was initially treated as a minor cosmetic problem, with the powders simply brushed away and the spots camouflaged with tinted paint. The seriousness of the situation was soon realized when more timbers were affected and the destruction of timber tissue became evident (Jespersen 1989:141-152). Kirsten Jespersen's analyses identified the white powder as hydrated iron sulfate, formed by oxidation of insoluble iron sulphides (FeS and FeS_2). Astutely, Jerspersen also recommended relative humidity control as being the key to protecting treated timbers from deterioration and encouraged cooperation with chemists to find methods by which iron salts could be removed prior to impregnation treatments. Similar problems had also been encountered with the *Batavia* timbers and as a result, subsequent research focused on further refining the problem, examining whether the problem was purely chemical in nature or if microbiological activity also played a role (MacLeod and Kenna 1991:133-142).

Problems associated with the presence of iron corrosion products in PEG-treated wood were further highlighted in 2000 with the development of highly acidic regions on many *Vasa* timbers and associated artefacts. These outbreaks were thought to be related to the inability of the climate control system to keep the relative humidity in the display gallery below 60% (Sandström, *et al.* 2003). High relative humidity is also strongly linked to the development of acidity in the *Batavia* timbers when the climate control system breaks down and in the Shinan ship due to the naturally high summer humidity and temperatures in South Korea.

The Skuldelev ships, the *Batavia*, the Shinan ship, the *Vasa* and wooden artefacts from the *Mary Rose* have common problems related to the complex interactions between the wood and the assorted chemicals incorporated into the wood structure during their immersion in the marine environment and as part of their conservation treatment. Corrosion products from corroding iron objects/fastenings and sulfur produced by sulphate reducing bacteria under anaerobic conditions in the marine environment permeated the timbers prior to excavation. Recovered timbers were then treated with PEG. Storage and/or display in high relative humidity environments encouraged chemical reactions between iron salts, reduced sulfur species, PEG and degraded cellulose that led to the development of highly acidic regions in and on timbers and other wooden artefacts from these iconic shipwrecks (Figure 2). This prompted extensive scientific investigations into the complex chemistry associated with these problems and suitable treatments to reduce acidity and ameliorate further deterioration.

Figure 2. Acid-affected region on a hull timber of the Shinan ship (Copyright: National Research Institute of Maritime Cultural Heritage, Korea).

Iron compounds found on and inside acid-affected timbers include reduced and oxidized species, specifically pyrite, goethite, melanterite, rozenite, jarosite, natrojarosite, magnetite, siderotil and siderite (Cha *in press*; Ghisalberti, *et al.* 2002:281-307; Jespersen 1989:141-152; MacLeod and Kenna 1991:133-142; Sandström, *et al.* 2003). Acid levels in iron-rich areas range from pH[4] 0 to 3.5. Although only very low levels of pyrite were detected in *Vasa* timbers, relatively high levels of sulfur were found, predominantly in the form of elemental sulfur and thiols[5] of the cysteine[6] type (Sandström, *et al.* 2003). In *Batavia* timbers however, higher levels of pyrite and only small quantities of elemental sulfur were found (MacLeod and Kenna 1991:133-142).

The development of acid salt problems focused attention on the role that iron plays in promoting the formation of acidic species and in catalyzing the oxidation of reduced sulfur species, cellulose and PEG. Catalysed oxidation of reduced sulfur species, such as pyrite and elemental sulfur, leading to the production of sulfuric acid, was initially thought to be one of the main degradation mechanisms occurring in treated archaeological timbers (Ghisalberti, *et al.* 2002:281-307; Sandström, *et al.* 2003). While acidic hydrolysis of cellulose and hemicelluloses undoubtedly

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[4] pH – negative $\log_{10}$ of the hydrogen ion concentration.

[5] thiol - an organosulfur compound that contains a –C–SH or R–SH group where R represents an alkane, alkene, or other carbon-containing group of atoms.

[6] Cysteine – a thiol-containing α-amino acid.

contributes to deterioration of treated timber, subsequent studies demonstrated that there was less degradation in organically bound sulfur-rich areas of the *Vasa* and higher levels of degradation in low sulfur, high iron regions (Almkvist 2008). These findings indicated that organically bound sulfur was acting as an anti-oxidant and reducing the catalytic effects of iron compounds in these areas.

In addition to the contribution to acid development by the oxidation and hydrolysis of iron salts and the production of sulfuric acid, iron species have also been implicated in free radical reactions, the acid hydrolysis of cellulose and other species, the oxidative depolymerisation[7] of cellulose and PEG (Almkvist 2008; Elding *in press*; Godfrey, *et al. in press*; Lindfors, *et al.* 2007:57-63), the depolymerisation of hemicellulose and the formation of increased amounts of low molecular weight organic acids (formic, oxalic, acetic and glycolic acids) in sub-surface *Vasa* timber sections that have high iron to sulfur ratios (Almkvist 2008). While acetic acid generation can be directly linked to hemicellulose breakdown, the presence of greater amounts of formic acid in PEG-rich areas indicate that oxidative degradation of PEG is the major source of this acid (Almkvist 2008; Godfrey, *et al. in press*). It became clear that the situation in iron-impregnated, PEG treated wood is far more complex than originally thought, with more than just sulfuric acid contributing to the acidity of the timbers.

Where to now?

The increased knowledge regarding post-treatment deterioration of waterlogged wood necessarily imposes on cultural heritage professionals the need to carefully consider their actions with regard to the management of this valuable cultural resource. Some of the questions facing heritage management professionals include:

- What can be done to minimise further deterioration in archaeological wood that is already in a treated, dry state?
- If timbers are excavated with the intent of conservation, how should current treatment regimes be altered to minimise on-going post-treatment deterioration?
- For already excavated timbers for which conservation treatments are not yet complete, can treatment regimes be modified to take into account recent research developments?
- Should timbers continue to be excavated for study and conservation and if excavation is undertaken, is study, documentation and then re-burial an appropriate preservation management strategy?

Conserved, dry wood

It is clear that although waterlogged timbers initially appear to be stable after treatment with PEG, deterioration may continue long after the timbers have been dried. As low molecular weight PEGs are hygroscopic[8] their presence in wood, combined with elevated relative humidity levels, enables sufficient moisture to be retained for chemical reactions to continue in the ‘dry’ archaeological wood. For already treated archaeological wood there are limited options to minimize further deterioration, all of which come at a significant cost. These options depend on the

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[7] Depolymerisation – a reaction which leads to the break up of polymer chains into smaller units.

[8] Hygroscopic – readily absorbs moisture from the air

condition, size and state of the wood - are they part of a hull structure for instance? In this latter case, as typified by timbers in the *Vasa*, post-PEG treatment options are extremely limited due to the restricted access to individual timbers in the multi-layered hull structure. More options are available however for the timbers in the *Batavia* hull reconstruction as each timber can be individually removed and re-treated if necessary.

As first muted by Jespersen (1989:141-152), environmental control of the storage/display environment, primarily by a reduction in the ambient relative humidity, is critical to the mitigation of deterioration mechanisms. While the capital cost for humidity control systems and the on-going running costs are very high, for large objects such as complete hull structures, this may be the only option available to minimise further degradation. The *Vasa* Museum installed a new air conditioning system in 2004, reducing the relative humidity in the display environment from upper levels of 65 – 70 % to a target range of 51 – 59 %. The effectiveness of this change has been reflected in a dramatic decrease in the number of acidic outbreaks that have occurred following this change in display conditions (Hocker, *et al.* 2009:469-481). By comparison, the conditions in the *Batavia* display gallery are currently maintained at 47 – 57 %, reduced from 50 – 60 %, in an attempt to reduce the incidence of acidic outbreaks.

Conservation professionals continue their efforts to determine the best treatment options for recovered waterlogged wood and to examine methods by which already treated wood can be better preserved. In order to minimise the post conservation problems associated with treated timbers, it is necessary to examine the kinetics of deterioration processes, the rate of oxygen consumption in treated timbers, and the environmental requirements that are needed in order to minimise deterioration processes. Research is continuing in all of these directions with recent research shedding further light on aspects of deterioration processes including PEG degradation (Almkvist and Persson 2005:269-278; Mortensen, *et al.* 2005:261-267; Mortensen *et al. in press*), oxygen consumption and measurement in conserved archaeological wood (Matthiesen and Nordvig *in press*) and the effects of consolidants on moisture absorption (and hence on the rates of chemical reactions) in conserved wood (Jensen, *et al. in press*). These and more studies are needed in order to fully understand the nature of the problem and the best way(s) to reduce degradation of treated archaeological wood.

Treatment of excavated archaeological wood

Despite research into alternative impregnation and consolidation regimes, PEG impregnation is still the most widely used treatment method for waterlogged wood. Disadvantages of its use however, include its cost, surface finish (dark, waxy and unnatural colour unless carefully cleaned) and its hygroscopicity that necessitates careful control of the relative humidity of storage and display environments for PEG treated wood.

PEG is viewed by some as being only suitable for short-term stabilization because of its degradation over time (Christensen, *et al.* 2010:1-7). Mikkel Christensen also considers that research is needed in order to develop a more appropriate consolidant that leaves ‘an airy structure in order to allow re-treatment in the future’ and that modern materials science should be applied to develop more durable impregnation regimes, possibly involving ‘foaming a polymer’, a combination of nanoparticles with a polymeric ‘spider web’, a framework using biomimetic materials such as an artificial lignin or via biomineralisation with an inorganic skeleton (Christensen 2010:1-7).

Until such time as a more appropriate consolidant is developed however, PEG use is likely to continue. If this is the case then it is critical that any treatment regime include steps to remove iron compounds from waterlogged wood. The extent of iron removal needed to minimise continuing degradation of polysaccharides and PEG is still to be determined. Research efforts should therefore be made to determine if there is a threshold concentration of iron at which these degradation mechanisms are of minimal impact. Unfortunately the complex nature of deteriorated wood components, incorporated chemicals from the burial environment and the differing grades, concentrations and purities of PEG used to treat archaeological wood, complicate research efforts in this area.

In the early 1990s preliminary experiments on the extraction of iron corrosion products from degraded, iron impregnated waterlogged wood found that the use of sodium dithionite in conjunction with diammonium citrate and PEG solutions, at neutral pH, was very effective in removing iron minerals from iron impregnated timber whilst successfully stabilising the wood structure (MacLeod, *et al.* 1991:119-132; MacLeod, *et al.* 1994:199-211). More recently, high performance iron chelators, diethylenetriamine pentaacetic acid (DTPA) and ethylenediimino-bis (2-hydroxy-4-methylphenyl) acetic acid (EDDHMA), have been successful in removing iron corrosion products from acid-affected, PEG treated waterlogged wood (Almkvist and Persson 2006:678-684). Potential problems with the latter treatment however, include degradation of hemicelluloses due to the alkaline nature of the chelating solution[9] and post-treatment removal of the rich red colour that the iron-EDDHMA complex imparts to the wood. While research continues in this area, a comparison of the sodium dithionite/ammonium citrate/PEG treatment with both the DTPA and EDDHMA treatments, found that the former solution was significantly more effective in removing iron from waterlogged wood (Richards *et al. in press*).

Reburial of waterlogged wood

Excavation, study, documentation and reburial of waterlogged, archaeological wood may be a valid conservation management option for some, though not all, of these problematic materials. Reburial of excavated archaeological materials has long been used as a management tool, particularly in cases where large numbers of similar artefacts have been recovered, where the conservation budget is insufficient to allow the treatment of all objects, where the display or further research value of objects is limited or when sufficient archaeological information can be obtained from the objects without the necessity of a full conservation treatment (Bergstrand, *et al.* 2005:9-39; Godfrey, *et al.* 2004:343-351; Manders, *et al.* 2008:179-203).

Depth of burial has been found to be a very important parameter when relating the degree of wood decay to the local environment. Many authors have shown that the extent of biological degradation of wood decreases considerably with burial depths greater than 50cm (Björdal and Nilsson 2002:235-244; Gregory 1999:78-83). However, recent studies have indicated that to decrease degradation of wood to almost negligible levels the depth of burial may need to be significantly greater than

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[9] Chelating solution – a solution containing a chemical that binds strongly to a metal, forming a soluble, complex molecule.

50cm (Bergstrand and Nyström Godfrey 2007:207-224; Nyström Godfrey, *et al.* 2011; Richards, *et al.* 2009:113-160).

So based on past and current research, long-term preservation of reburied wood requires burial depths of over 50cm, in an environment which is sub-oxic to anoxic, is strongly reducing in nature with near neutral pH and dominated by anaerobic chemical and microbiological processes (sulphate reduction and methane production). In addition, the porosity of the reburial sediment should be low and possess almost negligible levels of interstitial organic materials such as dead seagrass, other dead/decaying organic matter or organic pollutants. More importantly, the area in which the material is reburied needs to be stable and not subjected to significant sediment movement through excessive water movement, such as strong current flow, large tidal ranges and frequent storm events (Nyström Godfrey, *et al.* 2011).

Conclusions

Although the guidelines for the conservation and preservation of underwater cultural heritage, developed by UNESCO in 2001, strongly endorse the in situ preservation of underwater cultural heritage sites and artefacts (UNESCO 2001), wood will continue to be excavated from at least some underwater sites. Port developments, underwater pipeline construction and dredging operations for example, often interact with shipwreck or other submerged archaeological sites. In these instances, excavation and either documentation, conservation or reburial, even at an alternative site may need to be considered. More research is needed with regard to the development of durable conservation treatments, for the amelioration of deterioration processes in already treated wood and in the development of protocols and guidelines for the effective preservation of waterlogged wood by reburial.

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