Iron Removal from Waterlogged Wood

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Table of Contents

BACKGROUND AND THEORY

Since 2003 a series of experiments using high performance iron chelators (HPIC) have been performed to investigate the prospective removal of iron compounds from Vasa wood. The initial trials with HPIC were performed as local applications on salt precipitations on parts of the hull. Not surprisingly diffusion was poor, and it was difficult rinsing out the chemicals. In subsequent experiments loose artifacts were extracted by immersion and some of the objects were later re-conserved by PEG 2000 impregnation and vacuum freeze-drying with satisfactory results.

The first part of this paper summarizes the experiences of using HPIC on *Vasa* material that faces degradation problems related to the presence of iron compounds. The second part describes how extraction with HPIC could be practically implemented. This is to facilitate the continued development of the methodology on wet archaeological wood containing high levels of iron compounds. It should be emphasized that the full long-term effects of iron extraction and rewetting of previously conserved wood is not known and should be further evaluated. However, the HPIC agents used here (DTPA and EDDHMA) are not particularly reactive and do not possess any special chemical properties more than their exceptional capacity of binding metal ions. On the other hand, the long-term effects of allowing iron compounds to remain in wood should not be underestimated. As long as free iron ions are available there is a potential risk that chemical degradation proceeds or accelerates, which in the long-term would undermine the preservation of iron-contaminated wooden artifacts.

The overall aim of these experiments was to investigate whether iron extraction is a possible way to achieve a long-term conservation of iron-contaminated Vasa wood. The following issues were raised at the start of the project:

- To what extent is it possible to extract iron compounds with HPIC from *Vasa* wood (pine and oak)?
- Are acids neutralized and polyethylene glycol (PEG) extracted simultaneously?
- How suitable are the processes of rewetting and extraction of already conserved wood in terms of its chemical and mechanical stability?

Iron related preservation concerns in archaeological wood

Iron-contaminated wood is common in archaeological contexts, especially shipwrecks, since wood is frequently found associated with iron, either as fastenings or as objects found nearby. In preservation of waterlogged wood, the presence of iron compounds and other inorganic compounds is regarded as problematic both from an aesthetic and conservation perspective. The most common types of iron compounds in waterlogged wood are various iron(III) hydroxides and oxides along with iron(II) salts and iron in reduced sulfur compounds such as pyrite and pyrrhotite. There are three main threats to waterlogged wood considered to be linked to these compounds. Firstly, inorganic compounds tend to accumulate in high levels in the surface region, which can lead to mechanical damage if the object is dried without treatment. Secondly, oxidation of iron in reduced sulfur compounds may generate acid, which can lead to hydrolysis of polysaccharides and degradation of wood. The third, and probably the most problematic threat, is that iron compounds in general are capable of catalyzing various chemical reactions and have the potential to initiate oxidative degradation. Oxidative degradation in conserved wood results in depolymerization of wood components and preservation agents (e.g. PEG) and may generate low molecular organic acids, such as formic, glycolic and oxalic acid. Thus, for the conservator, there are strong reasons to remove or inactivate iron compounds in wood before conservation.

The following generalized description of degradation associated with iron compounds can serve as a starting point when considering an iron extraction treatment[1]:

<u>Wood in the surface region</u>. Archaeological wood is initially degraded through the action of microbes, which access the wood via its surface, increasing the porosity in this region. Hence, the highest concentrations of iron, sulfur and other elements are often found here. The total content of these elements varies greatly in *Vasa* wood but they are associated with surface precipitations at levels of over 10 mg/g. A variety of compounds are commonly found depending on previous redox status: iron sulfides, iron hydroxides and sulfates, elemental sulfur and calcium sulfate (gypsum). By oxidation, migration and precipitation, these compounds can give rise to surface deposits, as described above.

<u>Interior wood.</u> Generally in *Vasa* wood, the content of iron compounds is lower below the surface but still found at significant levels. Depolymerization of cellulose and associated increase of oxalic acid has mainly been detected in wood with iron concentrations higher than 1 mg/g wood.

High performance iron chelators and principles of extraction

Within the conservation field, different methods have been applied to dissolve iron compounds from the surfaces of wet archaeological wood, e.g. hydrochloric acid, oxalate, citrate and EDTA. One difficulty with iron(III) compounds are their very low solubility. Very low pH is required to dissolve, for example, iron (III) hydroxide, but low pH is very damaging

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[1] Based on the elemental distribution in *Vasa* wood

to wood material and should be avoided. Oxalate, citrate and EDTA can dissolve some iron compounds but are too weak to affect others. Low pH will always favor the acid form of a chelating agent and thus reduce the chelating effect since it is protonated to its acid form (e.g. citric acid, Figure 1). High pH, on the other hand, will enhance the chelating effect but since the solubility of iron(III) is decreased dramatically at higher OH^--concentration, the effect is cancelled out. Thus, it is a competing equilibrium with the net result controlled/balanced by the strength of the chelating complex, the solubility of the iron compound as a function of pH, and the acid constant(s) of the complexing agent.

To extract iron compounds effectively at neutral pH therefore requires a strong iron(III) chelator. Iron(III) forms stable complexes with many multidentate ligands, especially those that can bind the iron(III) ion with a geometric fit to the ligand atoms positioned in the corners of an octahedron, such as the Fe-EDTA complex (logK = 25). But there are examples of other more powerful chelating agents, such as DTPA (logK = 29) and EDDHMA (logK = 38), (Figure 2). Often referred to as HPIC, these are stable in a very wide pH range, and can withstand hydrolysis up to pH 10 and 12, respectively. The iron-EDDHMA complex is used as a micro-nutrient in agriculture to avoid precipitation of hydroxides in alkaline soils, which can cause iron deficiency in different crops. Pure EDDHMA is not available on a commercial basis, while DTPA is a registered product that is used primarily in pulp and paper industry for complex formation and inhibition of metal ions (Fe and Mn) in various processes.

What is extraction?

Extraction is a process in which a substance is removed from a matrix by means of a solvent. In the case of iron extraction, the compounds are preferably dissolved by strong chelating agents since most iron compounds have very low solubility in water. The overall extraction rate depends on the sub-processes involved in extraction: 1) diffusion of the chelating agent into the wood structure 2) dissolution of iron compounds and formation of an iron-chelate complex. 3) diffusion of the complex out of the wood to the free extraction solution where the concentration of the iron-chelate complex gradually increases (in accordance with the diffusion equation, average distance (x) for a diffusing particle at a time (t) of the expression $x = 2 (Dt /\pi)^{1/2}$). This means that the average distance of diffusion varies with the square root of the diffusion time. Practice has shown that the total amount of iron extracted is linearly proportional to the fourth root of time for the extraction process as long as both extractable iron and free chelators are available. This time dependence can be used to estimate the remaining time required to reach a certain degree of extraction (see Figure 3). Since all steps above are interlinked, the extract should be changed regularly to accelerate the whole extraction process.
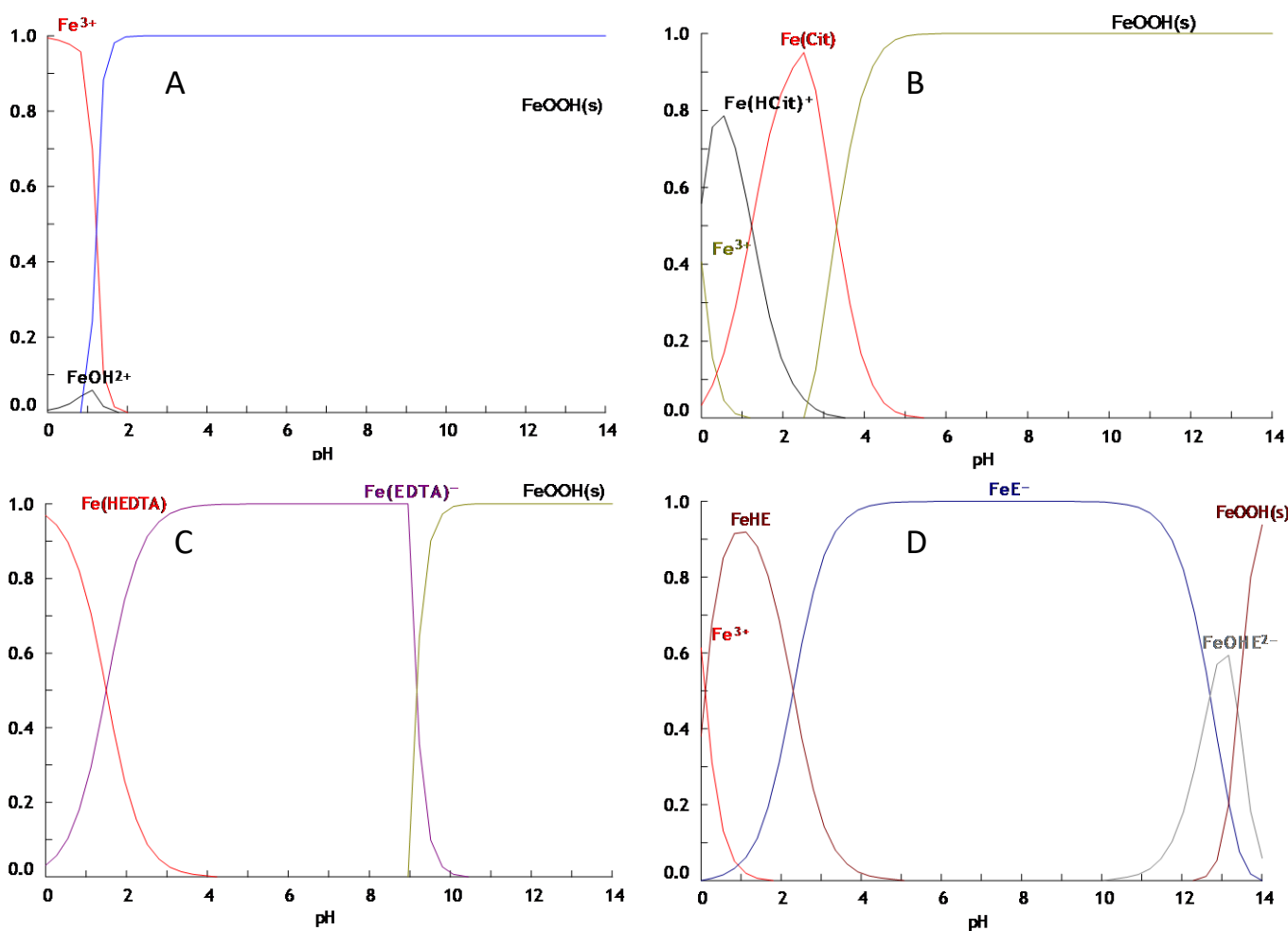
Figure 1. Stability diagrams for iron (III) in water (A) without the chelating agent and (B) with citrate, (C) EDTA and (D) EDDHMA. The diagrams show that the stronger the chelator, the more soluble the iron (III) compound. As a consequence the precipitation of iron hydroxide (FeOOH) is shifted to a higher pH.
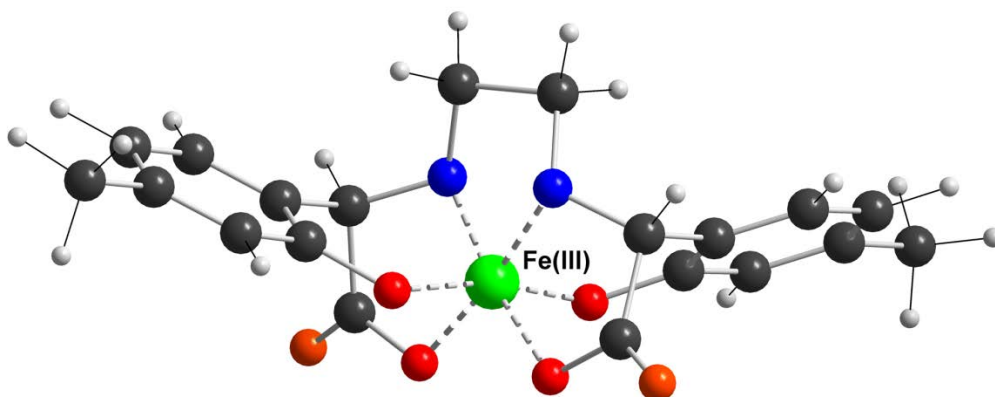

Figure 2. The iron(III) EDDHMA complex forms a very stable multidentate ligand "cage". The iron(III) (green) is surrounded by four oxygen (red) and two nitrogen (blue) and two non-bonding oxygen (orange) atoms.
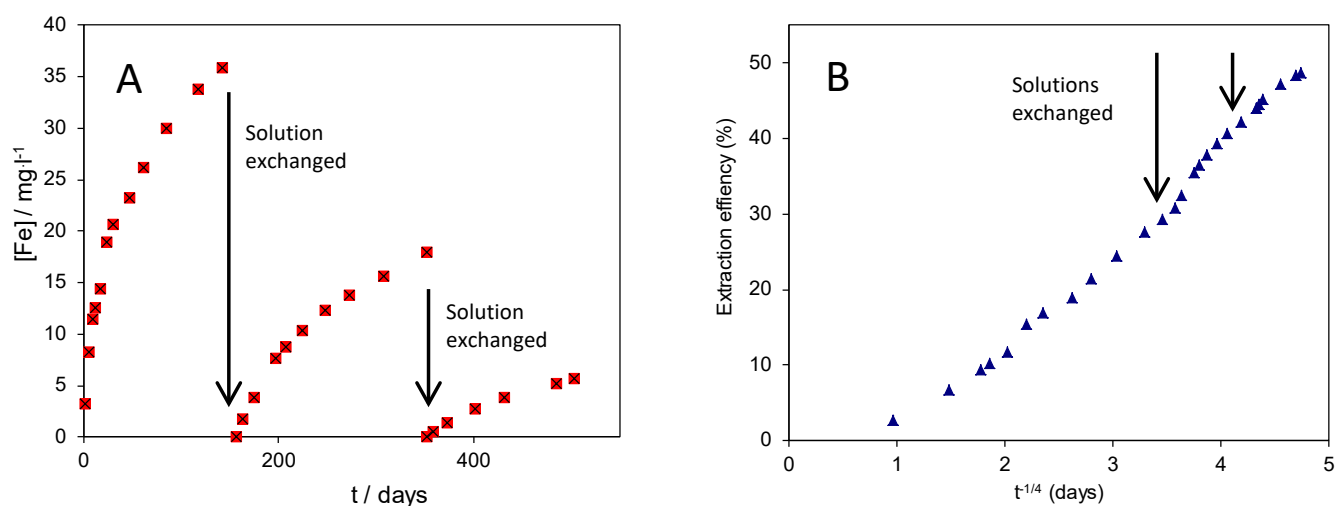
Figure 3. The concentration of iron in three consecutive extraction solutions (A) of an oak sample as determined by AAS and (B) the overall extraction efficiency for the same object as function of the fourth root of time. At the end the total amount of iron extracted was 8 mg/g corresponding to approximately 55% extraction efficiency.

SUMMARY OF RESULTS AND EXPERIENCES

The results from different extraction experiments carried out on *Vasa* wood are summarized in Table 1. The objects investigated showed a huge difference in the time needed to reach a satisfactory result (months to years). One fundamental difficulty when evaluating the impact of different parameters (e.g. type of ligand, concentration, temperature, etc.) on the extraction efficiency is that different wooden objects possess varying properties concerning iron content, type of dominant iron compounds, permeability, etc., which greatly affects the outcome of an experiment. There may also be great variability within the same object, such as different degrees of microbial degradation or iron concentration, which affects the permeability and availability of extractable iron. Partitioning of such an object into several parts in order to examine external parameters can lead to biased results. Despite these difficulties, some general conclusions concerning the external parameters can be stated, based on the experiments performed:

- High concentration of HPIC and frequent change of extraction solution provides the fastest extraction. One obvious disadvantage of this is elevated costs for chemicals and waste (see below).
- No significant difference in effectiveness between EDDHMA and DTPA were shown in the experiments, although the iron-EDDHMA complex is more stable than the iron-DTPA complex.
- pH affects the extraction efficiency of the chelator. The optimum for EDDHMA is pH 9 and above. Low initial pH can lead to precipitation of the acid form of the chelating agent (especially if the wood object displays high acidity) which therefore inhibits extraction. The optimum pH for DTPA has not been fully examined, but it does not have the same insoluble acid form as EDDHMA, which implies that DTPA can be used in neutral pH or below. However, the efficiency will be decreased in acid conditions. DTPA does not work effectively in alkaline solutions above pH 10, when competition

7

from the hydroxide complex is too strong and the iron-DTPA complex cannot be formed. The optimum pH range for DTPA is about pH 7 to 9

- The temperature of the extraction solution may influence the result, but has not been investigated sufficiently. In principle, diffusion is faster at higher temperature, but the negative effects on the wood can also be expected to be greater at higher treatment temperature, especially during a long extraction.

- Iron compounds are extracted faster and more efficiently from pine compared to oak. There are two explanations to this observation. The distribution of iron compounds differs between the two wood species. In pine, which is more porous than oak, the iron compounds are generally more concentrated in the surface region and particularly in the porous sapwood. Un-degraded heartwood of pine, which is rich in extractives, seems to be extremely impermeable preventing access of iron compounds, PEG and also HPIC. On the other hand, in oak the iron compounds have diffused deep, and therefore longer extraction time is required to remove the iron. Further studies on other species (i.e. ash and alder) are taking place.

Table 1. A survey of the extraction experiments.

Exp no.	n*	Mass (kg)	Wood species	Extraction time (months)	HPIC conc.[#] (mM)	Number of changes of extracts	Extractable iron (%)	Residual iron (mg/g)	Fe:S-ratio after extraction
1	6	1-6	Pine	30	E/D 6/14	28-42	58-98	0.3-(2)[4]	0.02-0.7
2	1	0.2	Oak	6	E, 1	2	72	0.2	5×10^{-5}
3	2[1]	0.2	Oak	17	E, 1	3	52	6	0.3/0.7
4	2	7-8	Oak	27	E, 1-10	5	24[3]	1.6[3]	0.2[3]
5	4[2]	0.2	Pine	6	E/D, 2-20	2	81-99	0.1-1	0.01-0.2
	2[1]	0.9	Oak	6	E/D, 2	2	17/46	0.4-0.7	1.0
6	6	0.3	Pine	3	D, 1	7	72	7	0.4

*n= number of objects in the experiment, #E=EDDHMA, D=DTPA. [1]One object divided in two pieces. [2] One object divided into four pieces of similar size. [3]Based on analyses of iron content in one core sample before and after extraction (0-17 mm). [4] One object had 2 mg/g residual iron, the other had 0.3-0.6 mg/g.

Effects on wood

Our experiences of the effect of iron extraction are based on experiments performed on wood that has previously been conserved with PEG and air-dried, rather than newly excavated waterlogged material. Consequently the act of re-wetting such wood may in itself cause physical damage and should not be confused with the effects resulting from the iron-extraction process. Equally, the iron-extracted material has been subsequently impregnated with PEG 2000 and freeze-dried, which may also affect the final result and our perception of the process. The general effects of the extraction process must be balanced against the long-term effects of leaving iron compounds in the wood (see further discussion below). Listed below are a number observations and conclusions from the extraction.

- Severely microbially-degraded pine was less dimensionally stable and highly sensitive to mechanical stress, indicating that the re-wetting of such material should be avoided or only be done with great caution. Oak, which was generally less microbially-

degraded, was more dimensionally stable and less susceptible to the negative effects of re-wetting.

- In addition to iron compounds, other substances were extracted, such as PEG and water-soluble salts. The high buffering capacity of the HPIC extract neutralized the acidity in the wood (Table 2). Some objects contained excess foreign matter, i.e. a mixture of old PEG, iron and sulfur compounds, salts, clay, etc.). In these cases, the extraction could be regarded both as a general cleaning of the surface region and as a specific operation to remove iron compounds.

- After extraction, structural details appeared and the color was changed from black-brown to bright hues similar to fresh timber (Figure 4-5).

- The intense red color of the iron-EDDHMA complex could be removed after successive rinses of the object in water.

- The effects of the chemicals on the cellulose (the degree of polymerization) were relatively small or in some cases negligible in treatments at moderately alkaline pH (<9). The effects on other wood components (both lignin and hemicellullose) can be expected to be greater at a pH>9, but have not been investigated. A strict pH limit for significant depolymerization has also not been investigated. It is worth noting that degradation of cellulose in some investigated objects was significantly high before the extraction and so the effects that could be attributed to the extraction itself were hard to determine.

Long-term assessment

To evaluate the long-term effect of iron extraction, the first objects that have gone through both extraction (2003-2005) and re-conservation (2006) have been followed up after five years. The analytical protocol comprises visual inspection, pH measurements, and an analysis of organic acids and the PEG quality, Table 2. The results from the 2012 evaluation showed no signs of acidification or PEG alteration.

Table 2. Analyses of pH and PEG content on samples that were extracted 2003-2005 and re-conserved 2006

Sample no.	Depth (mm)	pH* (2003)	pH# aqueous extract (2005)	pH# aqueous extract (2011)	PEG* (%) (2005)	PEG(%) (2011)
65759	0-5	1-2	6.7	6.4	0.2	15
65760	0-5	2-3	8	7.1	-	20
65761	0-7	1-3	6.4	7,0	0.3	18
65762	0-5	3	8	6.9	-	17

*on the surface by indicator paper
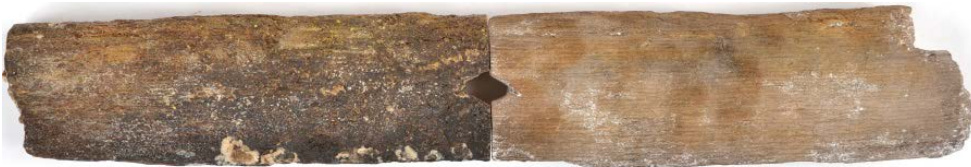
#analyses on the entire core

Figure 4. The appearance of two object before (left) and after (right) three months extraction in DTPA followed by PEG-impregnation and freeze-drying.

Figure 5. The appearance of a pine object before (large piece at upper side and bottom) and after (smaller corresponding pieces) 30 months extraction in EDDHMA followed by PEG-impregnation and freeze-drying. The total amount of iron extracted was 13 mg/g corresponding to approximately 100 g iron compounds and >90% extraction efficiency.

General conclusions and recommendations

For the conservator, there are strong reasons to remove or inactivate iron compounds in wood before conservation due to the negative effects associated: catalyzed oxidative degradation, acid hydrolysis and migration of salts. Extraction of iron compounds from wood is possible but very time consuming according to experiments performed on Vasa wood. Removing inorganic compounds from the wood is desirable as the surface regains more of its original texture and color. In the case of highly degraded surface wood, the risks of re-wetting and removal of PEG may not always be appropriate. The adverse effects, such as pressure damage during handling, strength loss and possible defibration must also be considered.

Arguments for and against extraction treatment should be discussed in terms of the negative effects of such treatment and expected iron-initiated degradation if no extraction is carried out. Knowledge of chemical degradation and effects of extraction in the long-term are still limited, which complicates the decision of whether or not to re-conserve already treated objects. However, as long as free iron ions are available there is a potential risk that chemical degradation proceeds, which in the long-term would undermine the preservation.

Although the results obtained are promising, further research and trials must be carried out and long-term monitoring of iron-extracted wood must also continue. For newly excavated waterlogged wood, the situation is simpler, since an aqueous extraction treatment could be implemented in conjunction with or as part of the necessary conservation.

EXTRACTION IN PRACTICE

Analysis of wood

Before extraction with HPIC is undertaken, the state of degradation and iron content should be established. In many cases the presence of iron compounds is obvious visually and no special analyses are needed. As described above, surface wood with more than 10 mg iron and sulfur per gram wood is associated with salt precipitation, and interior wood with more than 1 mg iron per gram wood often displays depolymerisation and/or acidification.

Material

Extraction of wooden objects should take place in suitable containers, which essentially means that the object should fit and that the total volume of solution does not become unnecessarily large. The container material can be plastic or stainless steel and covered to avoid evaporation. The object should be placed on a support so that the extraction solution may circulate all around the object. The bath should also be provided with equipment for stirring or circulation, e.g. a pump used for an aquarium. The container should be equipped with a drain or pumping device for emptying.

Preparation of solutions

Depending on what is most practical, preparation of the extraction solution and immersion of the object can be done in two different ways:

1) Mix the solution directly into the bath to a pre-determined volume and then immerse the object.

2) If the object is already placed in a bath filled with water, a concentrated solution is added to achieve a pre-determined final concentration and total volume.

Both approaches require that a more concentrated solution of the chelating agent is added to a larger volume of water. A pH adjustment can be made directly in the bath before the object is lowered (case 1) or by a rough adjustment of the pH of the concentrated solution followed by final adjustment of the bath (case 2).

The general formula $c_1 \times V_1 = c_2 \times V_2$ is used for calculating the solution volume, where c_1 is the concentration of HPIC to be added, c_2 the desired final concentration in the bath and V_2 the total volume after addition and possible dilution. The added volume of the concentrated HPIC is then calculated by $V_1 = c_2 \times V_2/c_1$.

> *Example 1: Solution preparation*
>
> Suppose we want a 10 mM solution (= 0.01 mol/L) of DTPA in a final volume of 100 liters. DTPA can be purchased in a concentrated sodium salt in aqueous solution (40%) with a density of about 1.25 kg/L.
>
> First calculate the molarity: 40% means about 400 g per kg DTPA solution, or 500 g per liter of solution after density correction. Since the molar mass of the DTPA salt is 503 g/mol, the concentration of the solution is calculated as: 500 g/L divided by 503 g/mol = 1.0 mol/L.
>
> Then use the formula above to calculate how much DTPA to add: V_1 = 0.010 M x 100 liters/1.0 M = 1.0 L. 1.0 L of 40% DTPA and 100 L water is added to the bath to achieve a 10 mM solution.

Adjustment of pH

The pH of the solution is monitored with pH meter or indicator paper. If the solution's pH must be adjusted, this should be done with small portions of strong acid or strong base, preferably hydrochloric acid or sodium hydroxide with a concentration of 0.5-2 mol/L. Less concentrated stock solutions of acid or base are also possible to use, but larger volumes will be consumed, which will affect the total volume significantly. The HPIC, such as DTPA and EDDHMA, are multivalent acids and since pH adjustment will be made in their buffer region, pH will change only slowly during the addition. The approximate amount of acid or base needed to reach a certain pH can be calculated (see example 2). Normally, the adjustment is done without major problems, if the adjustment is made in initially small steps with a strong acid or base. Both acid and base should be available to adjust to the desired pH. Appropriate precautions should be taken while handling strong acids and bases (protective goggles and gloves!). In Example 1 above (dilution of 40% DTPA), pH is preferably adjusted in the diluted final solution. Measurement of pH in concentrated solutions can be misleading, and it is also more difficult to adjust pH. If it is desired to adjust the pH before addition of the concentrated chelator to the bath, an intermediate adjustment can be done. Dilute the chelator 1:10 to make

the adjustment in this solution before adding to the bath. A final control and adjustment of pH directly in the bath should also be made.

Example 2: Rough estimation of the volume of acid needed for pH adjustment of alkaline DTPA.

Suppose we have the final diluted solution in example 1 (100 L 10 mM DTPA) with pH=11. We want pH 9, i.e. a decrease by two units. pK_{a5} and pK_{a4} of DTPA is 10.6 and 8.6, respectively, thus also a two pH-step. This means that we must provide one equivalent acid to protonate one equivalent DTPA two pH steps. In addition, the free hydroxide concentration (pH=11, $[OH^-]$= 0.001 M) must also be neutralized.

One equivalent: 100 L x 10 mM = 1 mol
pH reduction/hydroxide neutralization: 100 L x ($[OH^-]_{pH11}$ - $[OH^-]_{pH9}$) = 100 x (0.001 to 0.00001) ≈ 0.1 mol
The total amount of acid to add is approximately 1.1 mol. If 2 M HCl is available, a total of at least 0.5 liters is required to decrease pH.

After initial preparation and pH adjustments, the extraction will proceed. During extraction, acids will be neutralized (pH decreases) but also iron compounds that contain hydroxide as a counter-ion will be dissolved (pH increases). pH should therefore be monitored and adjusted regularly.

Replacement of solutions

Iron extraction is more efficient at high concentrations of the HPIC and at low concentrations of iron complexes in the extraction solution. When the concentration of chelated iron increases in the solution, the driving force and speed of diffusion will slow down. This means that the extraction solution should be replaced as soon as the concentration of the complex has increased significantly. As a rule of thumb, the extraction should be changed before the complex has reached half the initial concentration of HPIC. This is illustrated in Figure 3 The extraction rate is highest at the beginning of the treatment when easily accessible iron compounds in the surface region are released. It is therefore beneficial to change extraction solution more frequently during the first months. To keep control over the process and to be able to make the correct decisions, the iron content in the extract must be analyzed and this should be done at least before each solution change. Several methods are possible, see below.

Example 3. How much iron can be chelated by one single extraction solution?

The total amount of DTPA added in example 1 above is 1 mol. Since every DTPA molecule is capable of binding one iron ion, 1 mol iron ions will be chelated at equilibrium. For example, 1 mol iron ions corresponds to over 100 g iron(III) hydroxides (molar weight ca. 100 g/mol). However, to remove 1 mol iron compounds effectively (i.e. in the most time-efficient way) the solution must be changed several times as stated above.

Atomic absorption spectroscopy (AAS)

Determination of total concentration of iron in aqueous solutions with AAS is relatively simple and can provide a very accurate result. Normally, there is no need to dilute the sample. An appropriate wavelength is chosen as described by the instrument manufacturer. Calibration solutions are prepared from a standard solution of iron (1000 mg/L) which is diluted with an aqueous solution of HPIC of the same concentration as that used in the extraction. Commercial laboratories can analyze metal concentrations in water with high resolution ICP-AES (Inductively Coupled Plasma Atomic Emission Spectroscopy) directly after dilution or digestion.

Spectrophotometry

There are other methods in the literature that use spectrophotometry to determine iron content in water solutions. The problem is that most of them apply colored iron chelators that are weaker than DTPA and hence are useless. The iron (III) ions and EDDHMA forms a very strong red-colored complex with an absorption maximum at 490 nm. A rough determination can be made by measuring the absorbance and dividing by the extinction coefficient of the complex (ε = 4500 $M^{-1}cm^{-1}$). Corresponding complexes with DTPA are much weaker in color and an accurate determination cannot be done at the levels normally used in extraction.

When is the extraction complete?

The question of when an extraction should be ended is difficult to assess. Obviously, the goal is to extract as much as possible of the iron compounds in a wooden object, but a complete extraction is not possible to achieve. This is because the extraction rate decreases gradually, as more and more inaccessible and poorly soluble iron compounds are being extracted, as shown clearly in Figure 3. By extrapolating the potential extractable iron, a rough idea of how much time it will take to extract a certain amount of iron can be estimated, see Figure 3B. Even though not all iron is extracted it is likely that iron ions become chelated and their ability to act as catalysts in degradation reactions is hampered.

Waste and environmental issues

Concerning the toxicity and environmental impact of DTPA and EDDHMA, these seem to be relatively small although not completely studied. Large amounts (hundreds of tons) of EDTA and DTPA are released from the pulp and paper industry and municipal sewage treatment plants annually. Direct effects have not been observed, but the long-term effects are not known. Around the mills accumulation of DTPA has been found in the sediments. The iron-EDDHMA complex is used as a micronutrient on alkaline soils in horticulture and agriculture without restrictions. The assessment is therefore that small-scale extraction of waterlogged wood produces relatively small amounts of waste with a limited environmental effect. This is especially true if the substances are distributed as complexes with iron ions.

Further Reading

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