

Comparing different pre-treatment methods for strongly compacted organic sediments prior to wet-sieving: a case study on Roman waterlogged deposits

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Four pre-treatment methods have been tested on strongly compacted organic sediments prior to sieving. They comprise heating, freezing, soaking in NaHCO₃ (sodium bicarbonate) and heating with 10% KOH (potassium hydroxide). The aim of the experiment was to find out which pre-treatment method facilitates the sieving process without destroying the waterlogged plant remains recovered. Several methods are already described in the literature, but only few systematic comparisons of pre-treatment methods were undertaken. Of the four techniques tested, freezing the samples prior to sieving came out as the best option; it eases sieving and has the least damaging impact on the waterlogged plant remains. In addition, it is fast, uncomplicated and does not leave any chemical waste.

Keywords: archaeobotany, subfossil plant remains, methods, waterlogged deposits, pre-treatment, sieving

Introduction

Archaeological plant macro remains are commonly recovered by wet-sieving and flotation techniques that use water to separate the plant remains from the soil. Samples are, however, frequently encountered, which are very time-consuming if not impossible to sieve due to their soil composition. These are often categorised as problem soils (Pearsall 2000) and include in particular those with a high clay content. Clay soils are notorious for their poor dispersion in water. Where plant macro remains can only be isolated from the soil once discharged from adhering soil particles, this causes a problem as they are fragile and easily damaged. It is generally advised to agitate and crush the soil as little as possible during processing; however, this is often impossible and various authors have suggested pre-treatment methods to enhance the sieving process in order to minimise manual agitation and thus damage. Table 1 summarises the pre-treatment methods for

different soil types described in the literature and/or known from archaeobotanical laboratories. While these techniques have proven to ease the process of sieving, the effects of different pre-treatment methods on the plant macro remains themselves, as indicated by Jacomet and Kreuz (1999, 115), are rarely mentioned. This is especially important for uncarbonised waterlogged remains which are often more fragile than carbonised remains.

For the following experiment, we have chosen archaeobiological samples originating from strongly compacted archaeological layers located under the current water level. Recently, while working on a Roman settlement, we experienced many difficulties in sieving such compacted sediments and needed to improve the techniques employed. The soil samples under study are characterised by their high organic content, uncharred waterlogged plant remains and a rich and diverse plant spectrum, as are common on waterlogged sites. As such we believe that this experiment has widely applicable results.

It was decided to test four existing pre-treatment techniques described in the literature and which are frequently used in archaeobotanical laboratories with the aim of evaluating their suitability for recovering

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plant material efficiently whilst causing minimal damage. In contrast to most former studies, all four methods were applied to the same archaeological soil samples, to facilitate direct comparison of the results. Similar studies were undertaken by Bending (2005) on peat deposits and modern plant material, and by de Moulins (1996) on modern charred and fossil material.

Our primary concerns were:

- how pre-treatment techniques can influence the sieving process in a positive manner, and where sieving is facilitated by pre-treatment, to identify which of the four methods is the best for processing strongly compacted organic sediments and how the results compare with untreated samples;
- the impact of the various pre-treatment methods on the uncarbonised plant macro remains. More than 98% of the material in our samples is subfossil, and many of the remains recovered are highly fragile, uncarbonised cereal remains like glumes or rachises. Will there be any visible damages to the plant macro remains as a result of those pre-treatment methods? How do the plant remains in the pre-treatment-samples differ from those in the untreated samples?

Material and methods

The samples

Three samples were selected from an assemblage of over 700 archaeobiological samples from the Roman site of *Oedenburg* at Biesheim-Kunheim, France, located in the Upper Rhine valley about 60 km North of Basel. The Roman layers are dated from the

1st to the 3rd centuries AD and the archaeological structures are under the current water level and well preserved. The authors have processed and analysed the larger bulk of these 700 samples. The samples chosen for the experiment represent our 'typical' problem samples with which we had so many problems while sieving.

- Sample 1 (BK99-1-352-2) is from a dark organic layer at the bottom of a large pit. The deposits were strongly compacted with macroscopically thin layers and even more compacted nodules of soil, reminiscent of dung deposits. These nodules were especially difficult to process and one was selected for the experiment.
- Sample 2 (BK39033B) comes from an archaeological layer located within a palaeochannel. It was a very organic and compacted layer and was chosen because of its very rich assemblage of cereal remains and accompanying cereal weeds.
- Sample 3 (BK14054) comes from a latrine deposit within a large pit. In contrast to the other samples, the composition of this sediment was not as compacted and consisted mainly of loam.

Plant remains from these three samples were predominantly recovered in a waterlogged state of preservation (as for the majority of the 700 samples taken on this site). Volumes of samples 1, 2 and 3 varied between 750 ml and 850 ml before sieving. All three samples were divided in five equal subsamples of approximately 150 ml volume. A grid system was used for random subsampling, as described by Van der Veen and Fieller (1982).

Table 1 Summary of the pre-treatment methods described in the literature and/or known from archaeobotanical laboratories

Pre-treatment method	Type of sediment	Time	Reference
Soaking	Loam and clay soil	1 to 24 hours	Jacomet and Kreuz (1999)
Boiling	waterlogged deposits	15 to 30 minutes	Pearsall (2000); Kenward <i>et al.</i> (1980)
Drying	any 'problem' soil	48 hours	Zibulski pers. comm.
Freeze/Thaw	clay-rich deposits	2 days	de Moulins (1996)
Sonic bath	peat	not specified	Bending (2005)
5% KOH (potassium hydroxide)	clay-rich deposits	not specified	Hellwig (1990)
5% KOH and boiling	peat	5 minutes	Grosse-Bauckmann (1986)
10% KOH	compact organic sediments	2 weeks	Behre (1983)
10% KOH and heating	clay soil	30 minutes	Ernst pers.comm.
10% HNO ₃ (nitric acid)	compact organic sediments	several days	Körber-Grohne (1967)
10% NaHCO ₃ (sodium bicarbonate)	clay soil	several hours	Pearsall (2000)
10% NaPO ₃ (sodium hexametaphosphate)	clay soil	not specified	Pearsall (2000)
mix of NH ₄ OH (ammonia) and Na ₂ CO ₃ (sodium carbonate)	clay soil	not specified	Pearsall (2000)
H ₂ O ₂ (hydrogen peroxide)	clay soil	not specified	Pearsall (2000)
Na ₂ CO ₃ (sodium carbonate)	peat	up to 5 days	Bending (2005)
NaHPO ₄ (sodium pyrophosphate)	Loam and clay soil	not specified	Bollinger and Jacomet (1981)
10% NaOH (sodium hydroxide)	peat	several hours	Birks and Birks (1980); Kenward <i>et al.</i> (1980)

Pre-treatment and sieving

Four pre-treatment methods were tested on each sample; additionally one subsample was sieved without pre-treatment. As mentioned above, the four pre-treatment methods were chosen because of their frequent use in archaeobotanical laboratories. They comprise heating (Pearsall 2000; Kenward *et al.* 1980), freezing (de Moulins 1996), soaking with NaHCO₃ (Pearsall 2000) and heating with a 10% KOH solution (Behre 1983; Ernst pers. comm.). The sediment of the 15 subsamples was immersed in water before pre-treatment.

Heating

The subsamples were topped up with water to 600 ml. They were heated on a hotplate for half an hour to a temperature of approximately 50°C and were subsequently sieved.

Freezing

The subsamples were placed in a freezer at -18°C for two days and two nights. After that, they were taken out, left overnight to defrost and sieved the next day. At the time of sieving the subsamples were completely defrosted.

Soaking with NaHCO₃ (sodium bicarbonate)

One teaspoon of NaHCO₃ was added to the subsamples. They were topped up with water to 600ml, agitated a few times and left to soak for 24 hours.

Heating with a 10% KOH solution (potassium hydroxide)

About 15 ml KOH tablets (which equals 10% of the volume of the sample) were added to the subsamples and topped up with water to 600 ml. The subsamples were heated (at *c.* 50° C) in solution for half an hour on a hotplate under a chapel, and were stirred

occasionally. After heating the subsamples were immediately sieved.

All samples, including the untreated ones, were sieved (at 4 mm, 1 mm and 0.35 mm) using 'semi-flotation' as described by Hosch and Zibulski (2003), which is the same as 'wash-over', previously described by Kenward *et al.* (1980).

Data analysis

To measure the effects of pre-treatment on the plant remains, the fragmentation and the state of preservation of different plant species/parts in the 1 mm fraction were investigated using indices (see Tables 2 and 3 for definitions). Selection of the plant species/parts was based mainly on their abundance within the sample to ensure that comparison between subsamples of one sample is possible. Four indices were used to measure fragmentation. As plant species/parts break up in different ways, different scoring criteria were used for each (see Table 2). Five indices were used to measure preservation (after Hubbard and al Azm 1990). Once more, scoring indices were created appropriate for each plant species/part (see Table 3). The average index is calculated from the scores for each index as follows: the number of items recovered for each score (e.g. Poaceae without pre-treatment (22 items): score 1, 10 items; score 2, 4 items; score 3, 5 items; score 4, 3 items) was multiplied by this score (1 x 10 = 10; 2 x 4 = 8; 3 x 5 = 15; 4 x 3 = 12); these numbers were added up (10 + 8 + 15 + 12 = 45) and divided by the total number of items recovered (45/22 = 2.0). This final number (2.0) represents the average index. High average indices values indicate badly preserved or highly fragmented remains.

To test the statistical relationship between our results we performed a pairwise comparison of the

Table 2 Definition of the fragmentation indices

Score	Panicum	Poaceae	Cereal glumes	Cereal rachis
1	whole glume	whole caryopse	spikelet fork	3 or more segments
2	3/4 glume	3/4 caryopse	glume base with 1 glume	2 segments
3	part of glume (L)	1/2 caryopse	glume	1 segment
4	fragment	less than 1/2	fragment of glume	fragment of segment

Table 3 Definition of the preservation indices (* after Hubbard *et al.* 1990)

Score	Preservation classes*	Solanum nigrum*	Cereal glumes	Cereal rachis
1	Perfect	Epidermis perfect	All diagnostics present (keel, scar, full length of glume ...)	All diagnostics present
2	Virtually intact	Epidermis virtually intact	All but 1 present (keel, part of glume...)	All but 1 present
3	Incomplete	Epidermis incomplete	Incomplete glumes, species level	Incomplete rachis
4	Few feautres remaining	Only fragments of epidermis remaining	Few features remaining, genus level	Few features remaining
5	Gross Morphology only	Identifiable by gross morphology only	Identifiable by gross morphology only	Identifiable by gross morphology only

calculated average indices and the numbers recovered for all species. These coefficients were calculated using a Pearson's test with $\alpha = 0.01$. The number of variables (N = average scores and total numbers recovered) used is 24 for fragmentation and 18 for preservation.

Results and discussion

Results of the sieving experiment

Heating

Heating the sediment had a minor effect on the sieving process. The very compacted organic nodules in Samples 1 and 2 were broken up more easily. It was not, however, clear whether this was a consequence of the pre-treatment as they did not dissolve during cooking, but only when slightly agitated by hand while sieving. That said, the difference to untreated samples was so small as to be ignored.

Freezing

Freezing had a much more noticeable effect on the ease of sieving the samples. The organic nodules in Samples 1 and 2 were, for the most part, broken up through freezing and defrosting and passed through the sieves very quickly without much hand agitation. Significantly less time was needed to sieve these subsamples. The floated residue did still contain some clay particles, which slows down the sorting of plant macro remains, but overall it was still faster than sorting a subsample without pre-treatment.

Soaking with NaHCO_3

While sieving the soda-treated subsamples no difference in processing was noted compared with the samples that did not receive a pre-treatment. A slight difference was noticed for the loamy Sample 3 but the effect was minimal. Thus while this method is often used for the processing of problem soils, e.g. with a high clay content (Pearsall 2000), it was not found to be useful for processing strongly compacted organic sediments.

Heating with a 10% KOH solution

The very compacted organic sediments of Samples 1 and 2 were broken up strongly during the heating process leaving a few, very small organic lumps. Sieving of the subsamples was clearly much faster and easier than any other of the tested pre-treatments. Furthermore, no clay particles were observed in the floated residue, the vegetative material seemed to be 'washed' thoroughly. As a result, sorting these floated residues was effortless. The treatment did, however, produce a very intense and repulsive smell and in comparison to the other pre-treatment methods, reduced the volume of organic material left after

sieving. For these reasons, it was assumed that the chemical reaction of KOH and heating has caused more than just a breaking up the compacted organic sediments.

Summary

Of the four pre-treatment methods tested, freezing and heating with KOH solution, were shown to aid the sieving process for strongly compacted sediments. In addition sorting for plant macro remains was quickened. Purely heating or soaking in a NaHCO_3 solution had little impact on the sieving process. Based on these results it was decided to abandon further investigation of these pre-treatment methods and concentrate on the two successful methods, that is freezing and KOH-heating.

Effects of pre-treatment on the waterlogged plant remains

Analysis considered diversity, fragmentation and preservation of the plant macro remains within the sub-samples of a single sample. However, the size and nature of the sub-samples meant that in some cases, where the volumes were rather small, intra-sample diversity-variation is likely to be a result of sample size. In contrast, analysis of the fragmentation and preservation of the plant macro was possible in all cases.

Only one sample (Sample 2) yielded enough suitable plant macro remains to study preservation and fragmentation so the assessment of the impact of the pre-treatment on the plant species/parts was concentrated on this sample. We emphasise that the state of preservation of this archaeological layer (origin of Sample 2) is extremely good, as was observed during excavation. It has resulted in the recovery of an abundance of organic material.

The plant species/parts selected for analysis in Sample 2 comprised *Solanum nigrum* L. seeds, *Panicum miliaceum* L. glumes, caryopses of different wild Poaceae (Gramineae), cereal glume and rachis fragments. The subsamples of Sample 2 were entirely sorted for these five plant species/parts resulting in a total of 1415 items being extracted. Cereal glumes and cereal rachises have, for this experiment, not been identified to species level as no difference was observed in the way the different cereal species reacted to the pre-treatment methods. For the record, cereal glumes comprise *Triticum spelta* L., *Triticum dicoccum* Schubler and *Triticum monococcum* L.; rachis fragments comprise *Hordeum vulgare* L. and *Secale cereale* L.

A fragmentation and a preservation value was given to cereal glumes and cereal rachis fragments.

Table 4 Summary of the fragmentation index results

		No treatment	KOH and heating	Freezing
Panicum glumes	Total number recovered	85	20	64
	Average score	3.6	3.1	3.3
Poaceae (wild grasses) caryopses	Total number recovered	22	7	36
	Average score	2.0	2.9	2.2
Cereal glumes	Total number recovered	60	50	80
	Average score	3.1	3.3	2.9
Cereal rachises	Total number recovered	321	30	573
	Average score	3.6	3.3	3.4

Table 5 Summary of the preservation index results

		No treatment	KOH and heating	Freezing
<i>Solanum nigrum</i> seeds	Total number recovered	20	26	21
	Average score	3.2	2.5	2.8
Cereal glumes	Total number recovered	60	50	80
	Average score	3.7	4.5	2.8
Cereal rachises	Total number recovered	321	30	573
	Average score	3.8	4.7	3.5

Panicum miliaceum glumes and wild Poaceae caryopses were only given a fragmentation index whereas *Solanum nigrum* seeds were only given a preservation index (Tables 2 and 3). The attribution of fragmentation and preservation indices to the different species/plant parts was chosen as objectively as possible. The average indices of the above listed plant species/parts were calculated (*infra*). Table 4 summarises the results for the fragmentation indices, Table 5 the results for the preservation indices.

Fragmentation

For *Panicum miliaceum* glumes and cereal rachis fragments, KOH treatment is the better method with, respectively, average indices of 3.1 and 3.3; however, the difference from 'freezing' is very small. For cereal glume fragments freezing the subsample is beneficial with an average index of 2.9; for the wild Poaceae caryopses freezing only causes a slight deterioration.

These results should, however, be interpreted with caution. The difference in the total numbers of items recovered for each plant species/part (Table 4), especially the cereal rachis fragments varies considerably. Only 30 rachis fragments were recovered from the KOH-treated sample, against 321 in the untreated sample and 573 in the frozen sample. It is clear that far fewer fragments are found in the KOH subsamples and that this is a direct result of treatment with this chemical. Characteristically the plant macro remains of the KOH-sub-sample, have a faded colour and thinner appearance (as discussed below). For that reason, it is very strongly suspected that a large amount of the uncarbonised plant macro remains has dissolved through heating with KOH. This has also

Table 6 Pearson's correlations and p values between no treatment, KOH heating and freezing based on the fragmentation of Poaceae caryopses, *Panicum miliaceum*, cereal glumes and cereal rachis, where the number of variables = 24 with $\alpha = 0.01$

	No treatment	KOH and heating	Freezing
No treatment		0.5018 0.0242	0.9608 < .0001
KOH and heating	0.5018 0.0242		0.42035 0.0650
Freezing	0.9608 < .0001	0.42035 0.0650	

been observed by Bending (2005) when using KOH for the disaggregation of peat deposits.

Considering the average fragmentation indices of the frozen and the untreated subsample (Table 4), it is obvious that uncarbonised plant macro remains have undergone the least fragmentation when frozen before sieving. In three of the four plant species/parts, it has proved the better method.

When interpreting the results from the Pearson's correlation test (see Table 6), we can infer no significant difference between the plant species/parts from the untreated and the frozen subsamples (p values < 0.0001), whereas a more significant difference is observed between the KOH and both frozen subsamples and untreated subsamples (P values = 0.0650 and 0.0242 respectively). These findings indicate a strong relationship between freezing and no treatment, while the KOH and heating method was not significantly correlated to either of the other two pre-treatment methods. This corroborates the findings from our visual analysis where the effects of



Figure 1 Difference between frozen (LEFT) and KOH-treated (RIGHT) *Triticum monococcum* spikelet fork. Photograph by G. Haldimann

KOH on the fragmentation of plant species/parts stand out against the effects of freezing and no treatment.

Preservation

Preservation indices were measured on *Solanum nigrum* seeds, cereal glume fragments and cereal rachis fragments (Table 5). The results are more explicit than those obtained from the fragmentation indices. Cereal glumes and cereal rachises are best preserved in the frozen subsamples with average indices of 2.8 and 3.5 respectively; *Solanum nigrum* seeds are best preserved in the KOH-treated subsample with an average index of 2.5.

The average preservation indices of cereal glume fragments and cereal rachis fragments clearly indicate



Figure 2 Difference between frozen (LEFT) and KOH-treated (RIGHT) *Triticum spelta* spikelet fork. Photograph by G. Haldimann



Figure 3 Difference between frozen (LEFT) and KOH-treated (RIGHT) *Hordeum* rachis. Photograph by G. Haldimann

that freezing the sample prior to sieving is the best pre-treatment method (Table 5). Since hardly any agitating by hand was necessary during sieving, most fragments of cereal chaff did not undergo much damage. On the whole, including the *Solanum nigrum* seeds, freezing gives better scores than sieving without pre-treatment.



Figure 4 Difference between frozen (LEFT) and KOH-treated (RIGHT) *Secale cereale* rachis. Photograph by G. Haldimann

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Again this interpretation is confirmed when performing a Pearson's correlation test (see Table 7). The p values for freezing and no treatment are lower than 0.0001, whereas the p values for KOH and freezing equal 0.2993, and the p values for KOH and no treatment equal 0.1772. Thus we can conclude from these tests that there is a significant difference between KOH on one hand and freezing or no treatment on the other, as inferred from our visual analysis.

Considering the *Solanum nigrum* seeds, while scoring the preservation indices the fading colour of the seeds, caused by KOH, was not taken into account. In addition, although seeds pre-treated by KOH were very well preserved, there was a general observation of thinner and faded epidermis, and even to some extent transparent. This fading characteristic of KOH on uncarbonised plant remains has been observed before by Kühn (1999). The impact of KOH on waterlogged plant macro remains is thus very apparent, in particular on the cereal chaff. Figs 1–4 show the difference between frozen and KOH-treated *Triticum monococcum*, *Triticum spelta*, *Hordeum vulgare* and *Secale cereale*, respectively. These images illustrate the negative effects of KOH showing that the spikelet forks and rachis fragments fade in colour, and are also partly disintegrated. Given that the number of fragments is significantly smaller in the KOH-treated subsample (see Table 5), many plant macro remains are most likely entirely dissolved. In contrast to the cereal remains, the *Solanum nigrum* seeds have not undergone much damage. Nevertheless, as described by Hartwich (1896), the epidermis of *Solanum nigrum* is rather strongly lignified which may enable it to resist the impact of KOH and heating.

Conclusions

The results of our experiment have shown that pre-treatment of strongly compacted organic sediments is valuable in aiding the recovery of waterlogged plant remains. Several authors have previously established

the positive influences of pre-treatment on 'problem soils' before (see Table 1). However, a cross-comparison of different pre-treatment methods on one sample has rarely been done, except by Bending (2005) and de Moulins (1996). For our samples, freezing, defrosting or heating the samples with KOH prior to sieving enhanced the dispersion of soil particles in water. This meant that manual agitation was less necessary during sieving; the sieving process was faster and less destructive for the plant remains.

Concerning the effects of the successful pre-treatment methods on the condition of the uncarbonised plant remains, we have found both positive and negative consequences. KOH-treatment clearly had a destructive nature with many of the uncarbonised plant remains being damaged, either in the form of faded surface colour and/or disintegration. As a result, we suggest that this pre-treatment is not used when dealing with uncarbonised waterlogged plant remains, as too much information will be lost. Of course we have tested only one application of KOH-treatment (addition of 10% KOH solution and heating the sample) and there might be other treatments where KOH is less destructive, e.g. a 5% solution and/or without heating (Bending 2005). Nevertheless this was beyond the scope of our experiment. The least intrusive effects on the uncarbonised plant remains were obtained from freezing the samples prior to sieving. In general those plant remains were best-preserved and least fragmented even in comparison with plant remains from untreated samples. In addition de Moulins (1996) has already stated that this method has minor effects on the carbonised remains.

From our experiment we, therefore, conclude: that to obtain the best retrieval and to ensure the least damage of waterlogged plant remains from strongly compacted organic sediments, it is advisable to freeze the samples in advance of sieving. It is an easy and cheap method that does not leave any chemical residues.

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Table 7 Pearson's correlations and p values between no treatment, KOH heating and freezing based on the preservational state of *Solanum nigrum* seeds, cereal glumes and cereal rachis, where the number of variables = 18 with $\alpha = 0.01$

No treatment KOH and heating Freezing		
No treatment	0.3328	0.9617
	0.1772	< 0.0001
KOH and heating	0.3328	0.2591
	0.1772	0.2993
Freezing	0.9617	0.2591
	< 0.0001	0.2993

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