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WET ORGANIC ARCHAEOLOGICAL MATERIALS

Treatment of air-dried archaeological wool textiles from waterlogged environments

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ABSTRACT

Air-dried, wet archaeological wool textiles can be flat and stiff with brittle fibers, but is this a permanent collapse or can they regain their size in water? Iron Age textiles were tested comparing the width of dry fibers with the width of fibers treated in water or 70% ethanol. Both liquids expanded the fibers and the yarn increased in size, resulting in more flexible and less brittle textiles. This property was kept when the textiles were dried by stepwise dehydration in ethanol, acetone, and white spirit with a final treatment in 5% lanolin. Preliminary tests on brittle textiles can be performed on small samples to investigate if they will gain in flexibility by this method.

INTRODUCTION

When archaeological textiles are found in waterlogged sites, it is considered very important to keep them wet and cold until conservation can take place. This typically includes cleaning, stabilization, and the use of a drying procedure that will eliminate the contracting capillary forces of the water (Peacock 1992). Air drying of these textiles, depending on their degree of deterioration, can result in stiff and brittle fibers. However, it is not unusual for waterlogged archaeological textiles to have been exposed to air drying during conservation and/or storage. This is true especially for older finds treated prior to the introduction of common conservation methods like freeze-drying and dehydration. Even today, textiles may end up being air dried due to simple mistakes or by pure necessity. The latter happened during an archaeological excavation in an area near the waterfront in Copenhagen around 10 years ago (Borake 2012). The amount of waterlogged wool textiles excavated was so enormous that it became necessary to air dry some of the materials instead of using time-consuming methods such as cleaning, unfolding, and freeze-drying. Some textiles were, therefore, left unfolded and air dried, which resulted in stiff and brittle textile “packages.” Since then, some of these textiles have been treated at the School of Conservation in Copenhagen, and the results were surprising. The textiles were re-swollen in 70/30 v/v ethanol/water, unfolded, dehydrated stepwise, and impregnated in a solution of 5% lanolin in white spirit. This changed the stiff “packages” into soft and flexible textiles that could be vacuum cleaned and documented. The wool fibers had apparently not experienced the unrecoverable collapse that is often seen when waterlogged wood and leather are air dried. This was an interesting observation that gave rise to new research on re-swelling and softening of brittle and stiff archaeological wool textiles. Perhaps it was even possible to improve the condition of some of these textiles. This project focuses on investigating if a re-swelling procedure should be considered. The first thing to investigate is whether the archaeological fibers are able to absorb water and thereby soften and expand in volume. Water is well known for its softening effect on new wool, lowering its T_g from around 80°C at 45% RH to about minus 5°C in water (Wortmann et al. 2006). If the fibers do swell in water, the next thing to consider is whether or not to add a softening agent and, finally, which method should be used for the drying procedure. Some Iron Age textiles from Lønne Hede (LH)

(Demant 2007) were used for the experiment. The Lønne Hede textiles, excavated from graves, were air dried slowly in cold storage without any pretreatment, and today they appear stiff and brittle.

MATERIALS AND METHODS

Yarn samples (0.5–1 cm long) of archaeological wool textiles from the Iron Age graves from Lønne Hede were chosen for the experiment (LH yarn 1–8, Lønne Hede fragment, not registered; T×1.5 yarn 1, fragment from grave one; T×2.3E yarn 1 and 2, fragment from grave two). These textiles were not impregnated during or after excavation. They were preserved in the soil by waterlogged conditions, but the area had been drained during the previous decade, which meant that the soil was no longer permanently waterlogged when the graves were exposed (Lene B. Frandsen, personal communication, 2007). The textiles could, therefore, have been exposed to air drying even before they were excavated. The graves were block lifted and slowly air dried in cold storage. Today, the textiles are brittle and stiff, and fibers easily break off when handled.

Choice of methods

When hygroscopic textiles are submerged in water, the fibers will swell, the tension in the fabric will be released, and the T_g will decrease. For new wool fibers, the transverse swelling is about 16% from dry to wet state and the T_g will decrease from about 80°C at 45% RH to minus 5°C in water (Wortmann et al. 2006). When the fibers deteriorate, their swelling ability will increase or decrease depending on the chemical and morphological condition of the fibers. Measuring the transverse swelling of the fibers in water can, therefore, be an indicator of ability to improve the flexibility of the fibers and the fabric. It was decided to test water swelling as well as swelling in 70% ethanol to see if similar swelling and softening of the fibers occurred. The latter method would be preferable as the fibers were to be dried by stepwise dehydration in organic solvents. Furthermore, the antiseptic effect of ethanol is at its maximum at 70%, and, therefore, is a preferable solution if the textiles show signs of attack by fungus (Waller and Strang 1996).

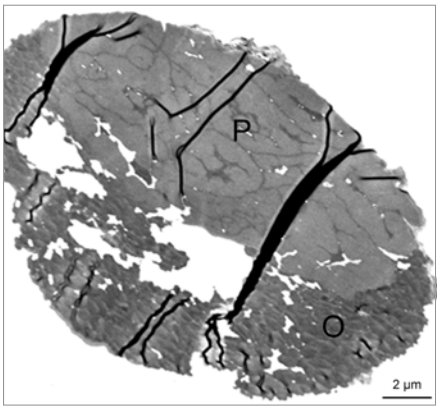

Figure 1
TEM of deteriorated Lønne Hede fiber without cuticula. Missing areas in orthocortex (O). The black lines in the paracortex (P) and orthocortex are artifacts. Photo: A.B. Scharff and L.B. Jørgensen

Stepwise dehydration in organic solvents was tested as a drying method for the textile. This method was selected because the author had previously noticed how the cross sections of the deteriorated Lønne Hede fibers appeared when they were analyzed in transmission electron microscopy (TEM). The TEM protocol had apparently had a favorable impact on the shape of the fibers, which had either regained or kept their original structure during the treatment, even though the fibers were clearly deteriorated with missing areas (Figure 1). The protocol was as follows: wool yarns were submerged in phosphate buffer (pH 7), fixated overnight in 2.5% paraformaldehyde + 2% glutaraldehyde, rinsed three times in the buffer (3 × 1-hour duration in total), stepwise dehydrated in acetone (3-hour duration in total), and finally stepwise impregnated with epoxy (Spurr) (3 × 24-hour duration). Proteins are cross linked by aldehydes, and this may be part of the reason for the stabilized structure of the wool fibers (Kiernan 2000). However, it is not known to what extent the deteriorated fibers will react with the aldehydes, and furthermore the cross-linking effect is a negative factor when attempting to improve the flexibility and thereby obtain softer wool fibers. Aldehyde treatment was, therefore, not considered in the experiments, only water treatment and stepwise dehydration in organic solvents. It was decided to use ethanol for the dehydration because it is less volatile compared to acetone and because of its antiseptic properties. The dehydration time for each concentration was set to one hour in each bath with a total of five concentrations (30, 50, 70, 96 and 99.9% ethanol) for water-swelled fibers. For the 70% ethanol-swelled fibers, only two concentrations were used (96 and 99.9% ethanol). Due to the brittle condition of the fibers, it was considered necessary to give them a final lubricating treatment. Lanolin was selected because of its good lubrication properties and because of its origin from sheep wool. It is a purified wax-like substance with good softening properties (Delacorte et al. 1971). Furthermore, the author has personal experience with its good softening properties when impregnating archaeological wool with a solution of lanolin in white spirit. Recently, the above-mentioned method was used on 17th-century archaeological waterlogged wool, which changed the stiff and brittle air-dried fabric into a soft and flexible material.

The samples for the experiment were threads that were between 0.5 and 1 cm long, and the question was therefore how it was possible to document a change in the fiber properties. The fiber width in water and 70% ethanol could be documented by simple measurements in the respective liquids. In comparison, when measuring the dry reference fibers, they should be embedded in a medium that does not change their size. By measuring and comparing the size of the cross sections of dry fibers embedded directly under vacuum in epoxy (Epofix) resin with the width of the fibers in whole mounts embedded in Euparal (R.i. 1.481), it was observed that the two methods gave similar results. Euparal was therefore used as an embedding medium for the dry reference as well as the lanolin-treated fibers. These measurements were compared with the results of fibers immersed in water and 70% ethanol. Ryder's (1981) method for documentation of wool types was used for the analyses. This method measures the width of 100 fibers, displays the results in a bar diagram and compares these as well as the calculated average and median of the measurements.

The evaluation of the brittleness and the flexibility of the fibers was more difficult with such small samples. It was not possible to find an objective method for measuring these properties with such short thread samples. Instead, it was decided to observe and photograph the threads while and after they were cut with a fresh razor blade in order to compare the softness and the fiber loss caused by the mechanical pressure of the blade. Furthermore, the appearance of the threads was evaluated by comparing cross sections and photographs of the yarn samples before and after treatment.

EXPERIMENTAL PROCEDURE

The swelling experiment

Each yarn sample was measured before and after swelling using Ryder's method (1981) described above, and the average swelling percentage was calculated. Whole mounts were prepared for the measurements where the dry reference fibers were embedded in Euparal, while the swelled fibers were embedded in the swelling liquid. The yarns were swelled by immersing the samples in either 70% ethanol for 24 hours or in 30% ethanol (half an hour) followed by 24-hour swelling in water. The water swelling was further improved by half an hour of vacuum treatment. The fibers' ability to bend in the liquid was examined.

The swelling and dehydration experiment

Each yarn sample was measured in its dry state before and after treatment and the average swelling percentage was calculated. All fibers in this experiment were measured in whole mounts with Euparal as the embedding medium.

Before the treatment could start, the yarn samples needed to be secured by fixing each yarn between two pieces of polyester crepeline and sewing this sandwich onto a fine-mesh metal net. Then the secured samples were swelled, as described above, and thereafter stepwise dehydrated by changing the ethanol bath every hour (30, 50, 70, 96 and 99.9 v/v% ethanol for the water-swollen samples and only 96 and 99.9% ethanol for the ethanol-swollen samples). Preparation for the lanolin treatment followed by exchanging the ethanol with white spirit (one hour in ethanol:white spirit (1:1), followed by one hour in 100% white spirit). Finally, the sample was left in a 5% lanolin solution overnight and then air dried for 24 hours in the fume hood. The softening effect of the lanolin was tested by comparing the dehydrated and lanolin-treated yarn with a yarn that was air dried just after the treatment in white spirit. The flexibility of the fibers and the appearance of the yarn were evaluated during and after the treatment, by photographing the yarn after it had been cut with a razor blade.

RESULTS

The results of the swelling show that all the samples swell from 6.6 to 20.7% in 70% ethanol and from 5.7 to 16.8% in water (Table 1).

Table 1

Average yarn widths (100 fibers) and swelling results after 24-hour swelling in 70% ethanol and water respectively

Swelling in 70% ethanol				Swelling in water			
Sample		Average	Swelling	Sample		Average	Swelling
LH	reference	18.88 ± 4.4	6.6 %	LH	reference	17.14 ± 4.6	16.8 %
Yarn 3	24 hours	20.13 ± 5.6		Yarn 4	24 hours	20.02 ± 4.3	
LH	reference	17.28 ± 3.4	20.7 %	LH	reference	18.31 ± 4.1	9.2 %
Yarn 6	24 hours	20.87 ± 3.5		Yarn 5	24 hours	20.01 ± 4.3	
Tx2.3E	reference	17.95 ± 2.4	10.4 %	Tx1.5	reference	19.34 ± 4.0	5.7 %
Yarn 2	24 hours	19.81 ± 3.5		Yarn 1	24 hours	20.31 ± 4.1	

All the samples became relatively flexible in both water and ethanol after 24 hours of swelling. It was now possible to bend them to a certain degree, but it was difficult to set exact numbers for the amount of bending that could take place without breaking the fiber, but it could be observed that the liquid had improved the fiber's flexibility compared to its dry state.

After the threads had gone through the stepwise dehydration in ethanol and a stepwise transfer to 5% lanolin in white spirit, the width of the air-dried fibers had either changed back to their original size or they had shrunk. Two of the water-swelled fibers shrank the most (5.5% to 10.9%) while the fibers in 70% ethanol only shrank from 0 to 3%. For comparison, one dry sample was immersed directly into lanolin without any pretreatment in water or ethanol. This resulted in an even larger shrinkage (12.5%) (Table 2).

Table 2

Average yarn widths (100 fibers) and swelling results (% of average dry width) of the experiments

Dehydrated from 70% ethanol				Dehydrated from water			
Sample		Average	Swelling	Sample		Average	Swelling
LH	reference	18.85 ± 4.3	0.5 %	LH	reference	18.31 ± 4.1	- 1.6%
Yarn 1	5% lanolin	18.95 ± 4.2		Yarn 5	5% lanolin	18.02 ± 3.6	
LH	reference	19.65 ± 4.1	0 %	Tx2.3E	reference	17.23 ± 2.9	- 5.5 %
Yarn 2	5% lanolin	19.65 ± 4,6		Yarn 1	5% lanolin	16.28 ± 3.6	
Tx2.3E	reference	17.95 ± 2.4	- 3 %	Tx2.3E	reference	17.95 ± 2.4	4,7
Yarn 2	5% lanolin	17.41 ± 3.3		Yarn 2	5% lanolin	18.80 ± 3.2	
				Tx1.5	reference	19.34 ± 4.0	- 10.9 %
				Yarn 1	2.5% lanolin	17.24 ± 4.1	
LH	reference	18.30 ± 3.8	3.4 %	LH	reference	17.65 ± 3.4	2.8 %
Yarn 7	+ acetone 5% lanolin	18.93 ± 3.7		Yarn 8	+ acetone 5% lanolin	18.14 ± 4.1	
Yarn immersed directly in 2.5% lanolin without pretreatment							
Tx1.5					reference	19.34±4.0	- 12.5 %
Yarn 1					Yarn immersed in 2.5% lanolin without pretreatment	16.93±3.6	

The lanolin in white spirit clearly improved the flexibility of the fibers when it was used on swollen and dehydrated fibers, whereas its softening effect was much less when it was used directly on dry fibers. When swollen yarns were dehydrated and air dried before the lanolin treatment, the yarns returned to a more brittle condition.

The lanolin-treated yarns seemed to have become more relaxed and open in structure, something that was best observed in cross sections (Figures 2–3). When these yarns were cut after treatment, they clearly demonstrated a change in flexibility. The fibers did not break as they did before treatment (Figures 4–5).

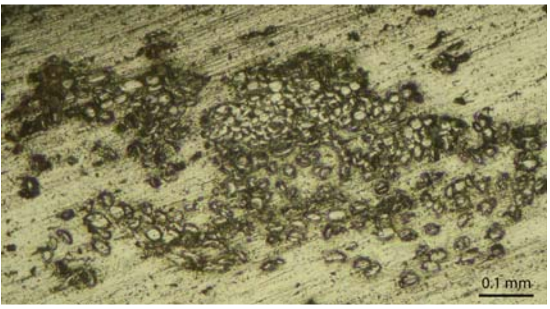

Figure 2

Cross section of Tx2.3E yarn 1 before treatment. Photo: A.B. Scharff

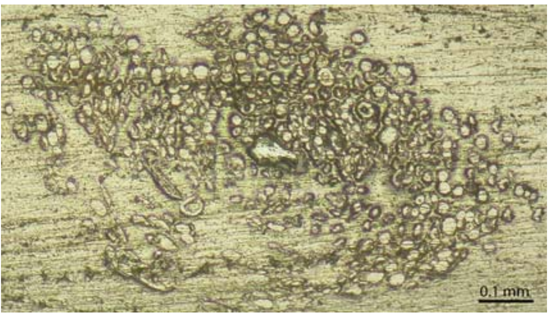

Figure 3

Cross section of Tx2.3E yarn 1 after swelling in 70% ethanol, stepwise dehydration, and treatment with lanolin in white spirit. Photo: A.B. Scharff

DISCUSSION

The swelling results show that it is possible to make the fibers swell in both water and 70% ethanol, but the degree of swelling varies. This variation could reflect a different degree of deterioration, but it could also be related to the measuring process. Some problems occurred when measuring the fibers, especially in 70% ethanol. The liquid evaporated and air bubbles caused the fibers to float, making it difficult to avoid re-measuring the same fibers. Therefore, the swelling results for ethanol and water mounts are not as precise as the measurements done in permanent mounts. Based on these varying results, it is not possible to conclude whether it is better to use 70% ethanol or water for the swelling. It could, perhaps, be possible to improve the accuracy of the water and ethanol-swelled fibers by mounting them in a water-based, permanent mounting medium (Aquatex), but this first needs to be tested.

The drying and softening procedure using ethanol and lanolin in white spirit improved the flexibility of the fibers and made them less brittle, but at the same time the fibers sometimes decreased in size compared to their dry state (Table 2). This could be related to the dehydration process, because it was observed that something happened approximately half an hour after the yarn had entered the ethanol/white spirit mixture (1:1). At first, the liquid became unclear and then globules emerged on the surface. Possibly it was water that was squeezed out of the fibers. Maybe the transfer from ethanol to white spirit was too harsh. When acetone was included in the dehydration process, the liquid never became unclear and the average fiber measurements increased by a few percentage points after drying (Table 2). This suggests that a dehydration process should include acetone in the stepwise dehydration from ethanol to white spirit.

The swelling procedure is crucial for improving the properties of the fibers. This was shown by comparing the dehydrated and lanolin-treated fibers with dry fibers that had been immersed directly in the lanolin solution. The latter only slightly improved the flexibility and the fibers were still brittle when cut. Furthermore, the fibers were shrunk by the treatment (Table 2).

After treatment, the improved flexibility and the reduced brittleness of the yarns seem to be the same for water-swelled and 70% ethanol-swelled samples. It is, therefore, preferable to start the swelling with 70% ethanol, because it will minimize the number of dehydration baths, and thereby the time and the mechanical effect of the transfer from one bath to the next. Furthermore, in most cases, the ethanol-swelled fibers did not decrease in width after the treatment.

The lanolin clearly improves the flexibility of the wool fibers, especially when used on swollen and dehydrated fibers. However, it needs to be investigated whether this is a long-lasting effect. Delacorte et al. (1971) have previously carried out tests with lanolin emulsion on wool, which showed that the improved softening effect of the lanolin had disappeared after a three-week period at 70°C with alternating RH (35% and ≥ 90%). These are, of course, extreme conditions compared to the preferred storage conditions for archaeological textiles, but apparently the effect of the lanolin may change over time. However, Delacorte et al. (1971) also showed that the treated fabric did not seem to have suffered greater material deterioration upon aging than the untreated blank. Finally, lanolin is a waxy substance that enhances the tendency of the wool to collect dust (Delacorte et al. 1971), and treated textiles must therefore be properly shielded to avoid accumulation of unwanted materials on the textile surface.

It is important to consider the mechanical damage and fiber loss that may occur when the textiles are transferred from one bath to the next. Yarns that were not secured prior to the treatment clearly lost fibers, and sometimes the yarns split up totally during the process. This was, however, eliminated by securing the yarns between polyester crepeline and stabilizing this sandwich with metal net. Furthermore, the mechanical

Figure 4
Lønne Hede fragment illustrating the brittle character of the textile. Sample LM yarn 2 is the horizontal yarn in the middle. Photo: A.B. Scharff

Figure 5
Lønne Hede sample LM yarn 2 after swelling in 70% ethanol, dehydration in ethanol, and treatment in 5% lanolin

damage was minimized by exchanging the liquid instead of moving the sample from one bath to the next.

CONCLUSION

This project shows that it was possible to re-swell the dry and brittle Lønne Hede fibers and improve their flexibility. The fibers exhibit a varying degree of swelling in both water and 70% ethanol, which means that it is not possible to highlight one swelling method above the other. They both seem to work fine as swelling agents. Stepwise dehydration with ethanol, white spirit, and a final impregnation with lanolin enhance the flexibility of the Lønne Hede fibers, but the fibers sometimes end up smaller in width than they were before the treatment. This shrinkage appears to be avoided if acetone is added to the dehydration procedure. However, further work is needed to confirm this. The effect of the method can be tested on a small yarn sample. If it swells in 70% ethanol, there is reason to believe that the above-mentioned treatment will increase the flexibility of the fibers and improve the flexibility of the textiles.

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