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# Material Identification and Technological Analysis of a 7<sup>th</sup>-century BC Excavated Textile from Argos, Greece

## Introduction

The name of the city of Argos is pre-Helladic (Pelasgian) and has remained the same since the beginning of the city's history. According to mythology, Argos, the son of Zeus and Niobe, was the founder of the city. It reached its peak during the Archaic period, under King Pheidon (7<sup>th</sup> century BC) (Pomeroy *et al.* 1999, 87). During that time, Argos was the centre of Argolis (today a prefecture), dominating the two major neighbouring cities, Tiryns and Mycenae. It was one of the most powerful and important Greek cities rivalling Athens and Corinth. It was caught in constant warfare with Sparta, which ended with Argos's defeat in c. 494 BC. Its refusal to fight in the Graeco-Persian Wars meant that Argos was shunned by most other city-states. It also remained neutral during the Peloponnesian War (5<sup>th</sup> century BC) between Athens and Sparta. Homer mentions Argos in the Iliad as the kingdom of Diomed and he frequently uses the alternate name Argives (inhabitants of Argos) for the Achaeans, one of the four main tribes of ancient Greeks, or Greeks in general, thus signifying the great importance the city had during that period (Kakridis 2009). The modern city of Argos is built on the site of the ancient one, which is common for the majority of Greek cities.

In April 2007, a rescue excavation run by the Hellenic Ministry of Culture – archaeologist in charge Dr Alkistis Papadimitriou – brought to light part of a cemetery of the early Archaic period (7<sup>th</sup> century BC), in the city of Argos (north-east Peloponnese). Among the oversized ceramic funerary vessels (pithoi) retrieved there was a much smaller, though very significant, copper vessel of a unique capsule shape. The two compartments of the vessel were held in place by iron pins. The copper vessel contained a substantial amount of textiles. It is a funerary vessel containing the incinerated bones of the deceased. The deceased's body would have been consumed in the pyre, the remains of the incinerated bones cleansed, wrapped in textile(s) along with fruit and placed in the vessel, which was subsequently sealed and buried in an upright position. Visual examination based on weave analysis revealed three different textiles present in the find. They were given the numbers Y1, Y2 and Y3.

Stereomicroscopy, ESEM and EDS, XRF, FTIR microscopy were applied to the textiles in order to get information on the technology and method of construction, material identification and state of preservation. More than one technique providing similar information were applied (*e.g.* EDS and XRF), since they are non-destructive, for comparison purposes.

## Material

The main volume of Y1 textile consists of a mass of folded textile of a light brown colour (Fig. 1). Numerous smaller fragments have been preserved in light brown and white colour. Some fragments adhere to the Y3 textile, the deceased's bones and the fruit offerings. Pieces of the deceased's bones and fruit were found within the folds of the main mass. The unified folded mass seemed to rest on a layer of Y3 textile (maximum dimensions of the folded mass: 280 x 300 x 110 mm). Textile Y2 consists of numerous brown, green and off-white-coloured fragments (Fig. 2). The majority of the fragments consist of multiple successive layers. Single-layered fragments adhered occasionally to the top surface of the unified mass of Y1 and on Y3 fragments (dimensions: fragments range from approx. 2mm<sup>3</sup> to 50mm<sup>3</sup>).

Textile Y3 consists of numerous dark brown, green and black-coloured fragments (Fig. 3). The majority of the fragments consist of multiple successive layers. All three textiles have been preserved in association with copper (dimensions: fragments range from approx. 2mm<sup>3</sup> to 80mm x 60mm x 60mm).

The bottom of the urn had degraded to such a degree that its contents were loosely spilled, hence they were collected in four aluminium foil trays. Tray I contained the main, more unified, volume of Y1; Trays II and III contained numerous textile fragments (Y1, Y2 and Y3) and a substantial amount of degraded organic material mixed with soil and powdered matter (?); and Tray IV contained mainly the largest preserved parts of the deceased's bones, the remains of a mineralised fruit (pomegranate), a mineralised, multilayered fragment of textile (Y3) and fragments of the degraded bottom of the copper vessel.

## Methodology

Samples were chosen among the numerous loose, small-sized fragments present for each of the three textiles. In total, nine samples were removed from the find: 1) Y1 brown, 2) Y1 off-white, 3) Y2 brown, 4) Y2 green, 5) Y2 off-white, 6) Y3 brown, 7) Y3 green, 8) Y3 black, and 9) debris(?). Each of the nine samples were used for all analyses and then returned to the find intact. Visual observation revealed that some fragments carried evidence of the method of construction of the textiles. These were studied with the stereomicroscope but not chosen for further analyses.

A NIKON SM-5 stereomicroscope was used to examine the samples (magnification from 6.7 to 45 times with a 10x eyepiece). Samples were illuminated by natural sunlight. No sample preparation was necessary (Coho 1996, 73; Haerincq 2002, 248-252; Nowick *et al.* 2005, 837-842).



Fig. 1. Most of textile Y1 has been preserved in a unified, folded mass. Scale bar 50 mm (© authors).

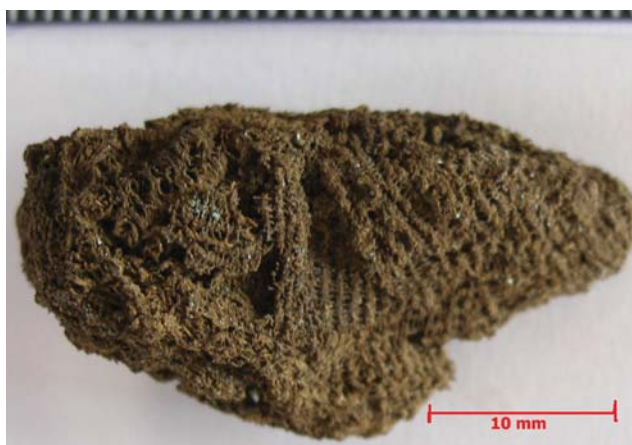


Fig. 2. Textile Y2 has been preserved in multi-layered fragments. Scale bar 10 mm (© authors).



Fig. 3. Textile Y3 has been preserved in multi-layered fragments. Scale bar 10 mm (© authors).



**Table 1. Stereomicroscopy results.**

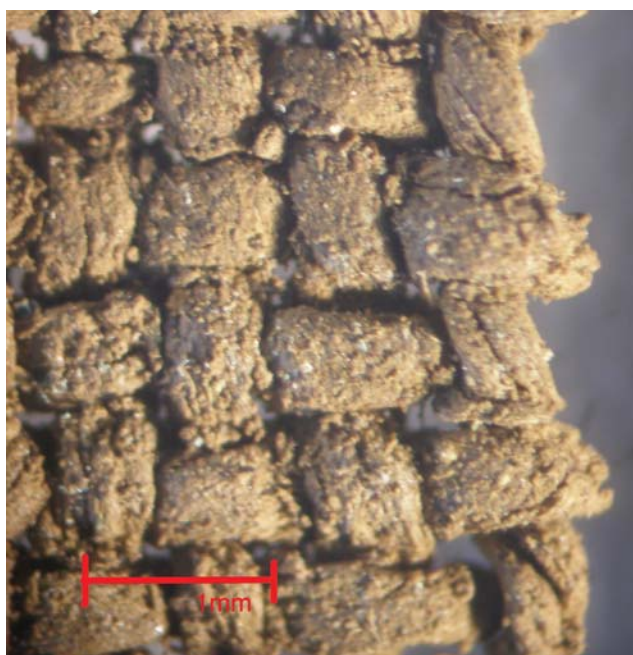
Textile	Element	Weave type	Weave count/cm <sup>2</sup>	Twist	Yarn diameter
Y1	warp	plain	16	2-ply, S	0.8± 0.2mm
	weft		16	2-ply, S	0.8± 0.2mm
	selvage	plait	22 warps x 7 wefts	2-ply, S	0.8± 0.2mm
	starting edge		6 turns/cm	2-ply(?), S	1.5± 0.2mm
Y2	warp	open plain	16	single-ply, Z	0.3± 0.1mm
	weft		48	single-ply, Z	0.1± 0.05mm
	selvage	16 warps x 48 wefts	single-ply, Z	warps 0.4± 0.1mm wefts 0.2± 0.1mm	
Y3	warp	weft-faced	7	single-ply, Z	0.8± 0.1mm
	weft		27	single-ply, Z	0.4± 0.1mm
	starting/side edge	6 warps wrapped by 1 weft	single-ply, Z		

A PHILIPS XL30 Environmental Scanning Electron Microscope (magnification up to 50,000) was used. The water vapour flooding the chamber gas prevented charging of organic specimens, hence coating was not necessary. The samples were inserted into the chamber secured to aluminium stubs with double-sided adhesive tape. Accelerating voltage of the beam was kept at 15keV to minimize the risk of localised damage to the samples caused by heating. Magnification was at 400 times. No sampling preparation was necessary. The high-magnification images produced enabled identification of the fibres by their morphology (Jakes

and Sibley 1989, 239; Coho 1996, 73-76; Moulhérat *et al.* 2002, 1395; Bertrand *et al.* 2003, 388; Müller *et al.* 2004, 178-179; Garside and Wyeth 2006, 90; Joosten *et al.* 2006, 170;).

The PHILIPS XL30 ESEM was coupled with an x-ray microanalyser (EDAX CDU LEAP Detector, using the eDX© software), which allowed qualitative elemental analysis of the sample simultaneously with the microscopic imaging. Three measurements were taken from different areas of each sample. In order for the analysis to be performed, the working distance was reduced to 10 mm. Spot analysis was carried out, and a minimum of 600 counts acquired. In each case the x-ray spectrum between 0 and 14 keV was recorded. The aim of this analysis was to perform qualitative elemental analysis of the samples that would provide evidence of the type of preservation and/or enhance information for fibre identification.

As with EDS analysis, the objective of XRF examination was also to confirm the type of preservation and enhance fibre identification by qualitative elemental analysis of the samples. A BRUKER portable XRF (AXS Tracer III-V) was used for the analysis. This is a versatile instrument that offers a specified elemental range of Z=19 and above in air (and Z=11 and above in vacuum) with detection limits similar to SEM-EDS, using a rhodium anode operating at 40 kV. Samples were placed on the window of the instrument in air, and each one analysed for 60 seconds. The S1P-XRF© software was used to process the spectra, while MS Excel© was used to further process the numerical data. Although the hardware itself seems ideal for the convenient elemental analysis of all manner of textiles, the associated software proved less user-friendly. An unspecified autogain varied amongst spectra, so that the apparent counts registered for an element in one



**Fig. 4. Textile Y1 is a balanced plain weave fabric. Scale bar 1 mm (© authors).**



Fig. 5. A starting edge of Y1 in a plait shape. The weave changes from balanced plain weave to weft-faced plain weave for 1 cm width before the plait. Scale bar 1 mm (© authors).



Fig. 6. An example of a Y1 fragment where stitching is shown connecting two selvages. This could indicate that narrower strips of fabric were connected to produce a larger piece. Scale bar 1 mm (© authors).

spectrum could not be compared directly with that in a second spectrum. In an attempt to normalise the data, the spectral counts for each element were ratioed to that of rhodium in every spectrum. The Rh peak ( $L\alpha_1$  2.70 keV) arises from the primary x-rays, which are generated inside the x-ray tube and then scattered by the specimen.

FTIR spectroscopy was applied to enable fibre identification, by comparison of the characteristic peaks for the major organic polymers present in the spectra of the samples, with those of reference samples (Jakes and Sibley 1989, 240; Gillard *et al.* 1994b, 187; Chen *et al.* 1996, 222; Bertrand *et al.* 2003, 391; Edwards and Wyeth 2005, 310; Margariti *et al.* 2010, 162). The spectral range of the technique applied is expressed in wavenumbers ( $\text{cm}^{-1}$ ) from 700 to 4000  $\text{cm}^{-1}$  (Skoog *et al.* 1998, 404).

A Perkin-Elmer FTIR Spectrum One instrument attached to an optical microscope in reflectance mode was used. Samples were scanned 32 times with a resolution of 4  $\text{cm}^{-1}$ , resting on a microscope slide with a gold mirror base. The microscope aperture was adjusted to 70 x 100  $\mu\text{m}$ . Grams AI v8<sup>®</sup> software was used to process the spectra produced. No sample preparation was necessary (Margariti *et al.* 2011, 525).

## Results

### Stereomicroscopy

Stereomicroscopy clearly showed the method of construction of the fabric and the yarns (Emery, 1994) (Table 1). Textile Y1 is a balanced plain weave fabric (Fig. 4) with several technological features, such as selvages, starting edges and stitching (Figs 5, 6). It is constructed from more than one narrow piece of textile, stitched together along their selvages.

The starting edge is the end of a woven fabric where weaving has commenced (Hoffmann, 1964, 420). There are two main methods used for starting edges: 1) weaving, where the band is first woven and then transferred to the loom when the wefts become the warps, creating a starting border (Hald 1980, 157-158; Hoffmann 1964, 151, 154, 420), and 2) sewing or otherwise attaching, where the warps are attached to the loom from the beginning, creating a heading cord (Hoffmann 1964, 419).

Textile Y2 is a very fine fabric of open plain weave (Fig. 7), where selvages have also been kept (Fig. 8). Textile Y3 is a weft-faced plain weave fabric (Fig. 9) with certain technological features such as starting/side edges (Fig. 10). The starting/side edges have been kept. No selvages were identified.

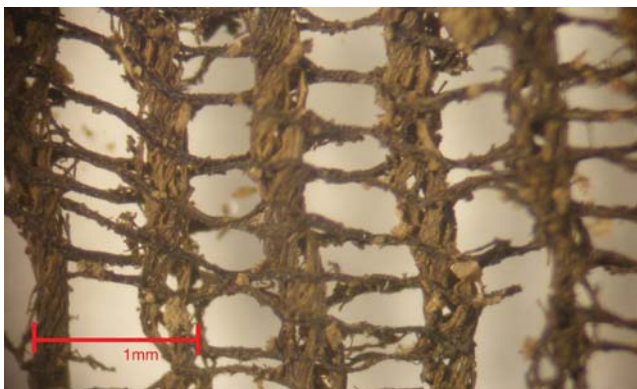


Fig. 7. Textile Y2 is a very fine fabric of open plain weave. Scale bar 1 mm (© authors).



Fig. 8. Textile Y2 selvedge. Scale bar 1 mm (© authors).

#### ESEM

ESEM examination provided some indication of the morphology and dimensions of the fibres of the three textiles. Although significantly masked by degradation products, the Y1 textile fibres (warps and wefts are the same) seem to have a cylindrical shape and average diameters 15 to 20µm, which is indicative of flax fibres (Margariti *et al.*, 2011) (Fig. 11). This is also the case for Y2 and Y3 warp fibres. The Y2 textile weft fibres seem to have pronounced striations along their length and a seemingly triangular, compact cross-section, indicative of wild silk fibres (Good 2010; Good *et al.* 2009; Sawbridge and Ford 1987; The Textile Institute 1975) (Fig. 12). The Y3 textile weft fibres seem to have been preserved as negative casts, on the inner surface of which, a pattern indicative of the epithelial scales of wool fibres was observed (Sawbridge and Ford 1987; The Textile Institute 1975) (Fig. 13). It was also confirmed that the debris present in large amounts in all three trays mainly consists of fragmented fibres.

#### EDS

In textile Y1, copper was detected, but its signal was not intense, suggesting that the Y1 textile was not in close contact with the copper vessel. In textile Y2, copper gave an intense signal in the case of certain samples, especially those taken near the selvedge areas, but was only detected in trace amounts in others. These results suggest that textile Y2 was closer to the copper vessel than textile Y1. In textile Y3 the copper signal was intense in all samples (Fig. 10), suggesting that textile Y3 was more in contact with the copper vessel than the other two textiles. Sulphur was detected in the Y3 weft sample, suggesting the presence of wool fibres (Tímár-Balászy and Eastop 1998, 48-55).

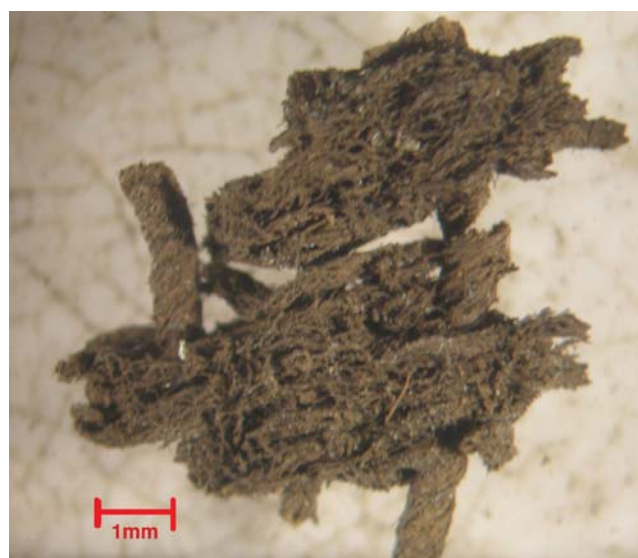


Fig. 9. Textile Y3 is a weft-faced plain weave fabric. Scale bar 1 mm (© authors).

#### XRF

High amounts of copper are present in all samples, especially Y2 and Y3, consistent with their preservation by copper impregnation and mineralisation. Both white samples of Y1 and Y2 appeared to have a lower copper content. However, XRF was unable to confirm fibre identification of the Y3 weft fibres (which could possibly be wool fibres as ESEM indicated). This could be in part because a larger area analysis was performed (so that a lower sulphur count would be expected), but more likely relates to the altered autogain due to the high copper content, such that signals at low energy were of much reduced intensity.



Fig. 10. Starting/side edge of textile Y3. The warps have the characteristic green colour of copper oxidation products, whereas the wefts have a brown colour. Scale bar 1 mm (© authors).

#### FTIR microspectroscopy

The textile Y1 warp and weft fibres gave similar spectra, characteristic of an organic material. Comparison of Y1 spectra with those of the references suggests a plant fibre (Fig. 14), the signature peaks of cellulose were evident, although somewhat shifted towards the higher end of the spectrum (*i.e.* 2927  $\text{cm}^{-1}$ , 1657  $\text{cm}^{-1}$ , 1162  $\text{cm}^{-1}$  and 1124  $\text{cm}^{-1}$ ). The Y2 warp and weft fibres gave almost identical spectra, which were more suggestive of proteinaceous than cellulosic fibres (Fig. 15). Although tentative, identification as silk seems more likely than wool as there are peaks in common with the copper oxohydroxide treated silk spectrum (1663  $\text{cm}^{-1}$ , and 1260  $\text{cm}^{-1}$ ). The patterns of the textile Y3 wefts are once again indicative of proteinaceous rather than cellulosic fibres, although the distinction between silk and wool is not clear (Fig. 16).

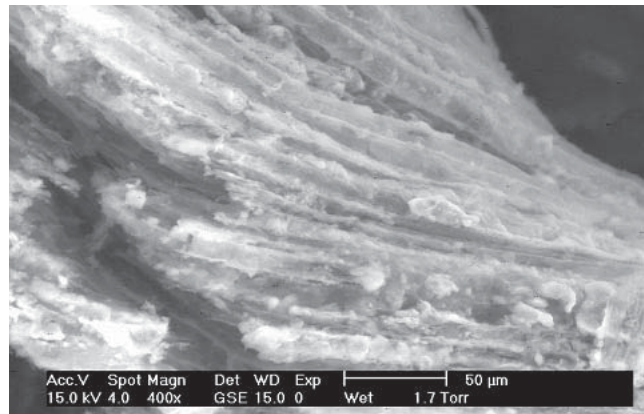


Fig. 11. Scanning electron micrograph of Y1 fibres. Fibre diameter ranges from 15 to 20  $\mu\text{m}$  (© authors).

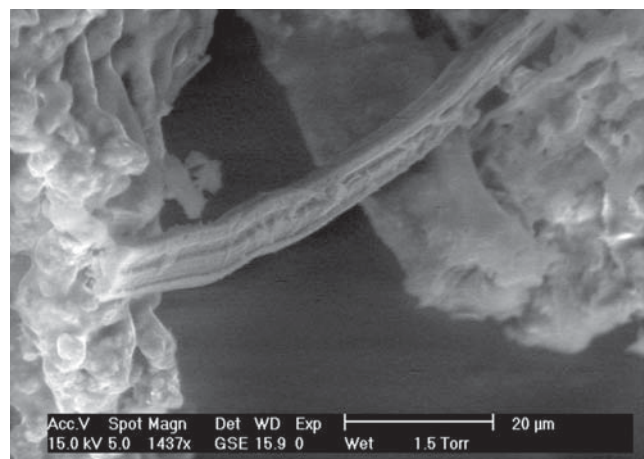


Fig. 12. Y2 weft fibres imaged in the ESEM. The fibre protruding from the degradation products has pronounced striations along its length and a seemingly triangular, compact cross-section (© authors).

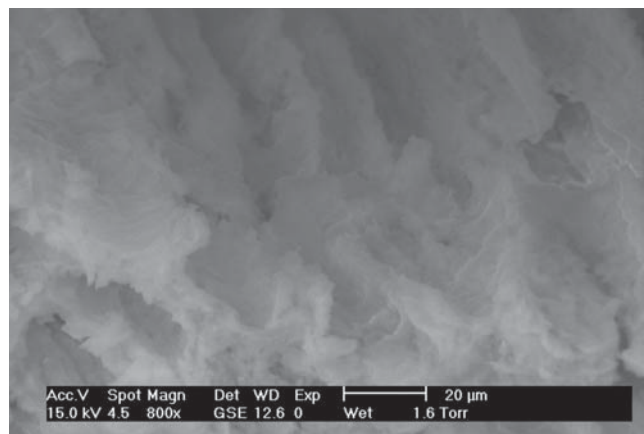


Fig. 13. Scanning electron micrograph of Y3 weft fibres. Negative casts of the fibres seem to have been preserved. A pattern indicative of the epithelial scales of wool fibres is observed (© authors).

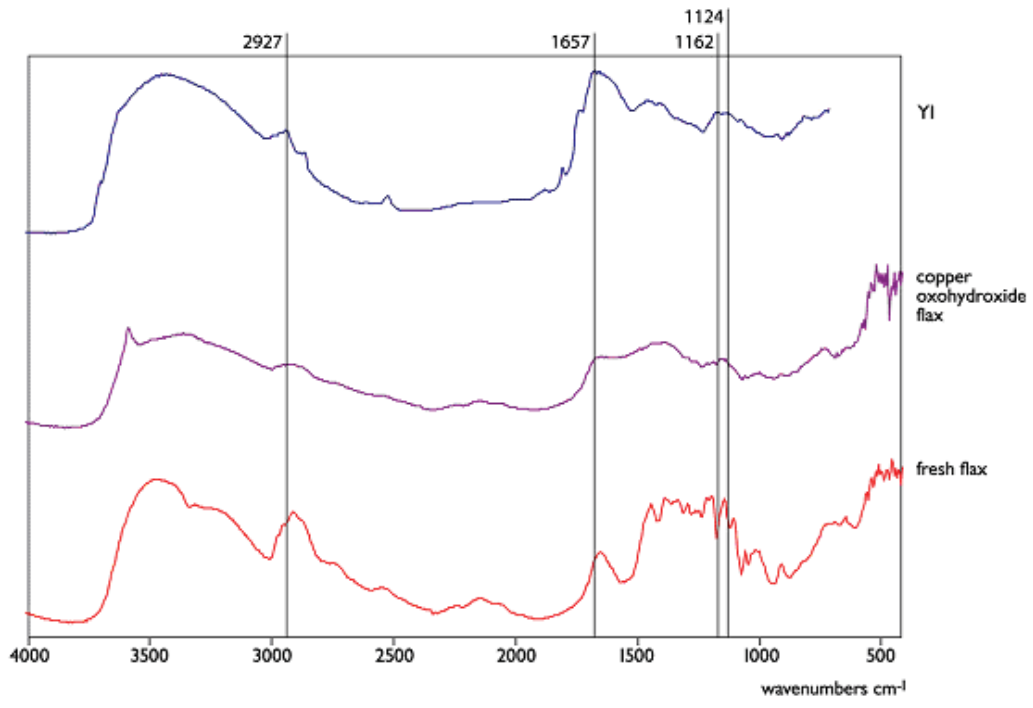


Fig. 14. FTIR microscope reflectance spectra of Y1 (top), and the reference samples: a) copper oxohydroxide treated flax (middle), b) 'fresh' flax (bottom) (© authors).

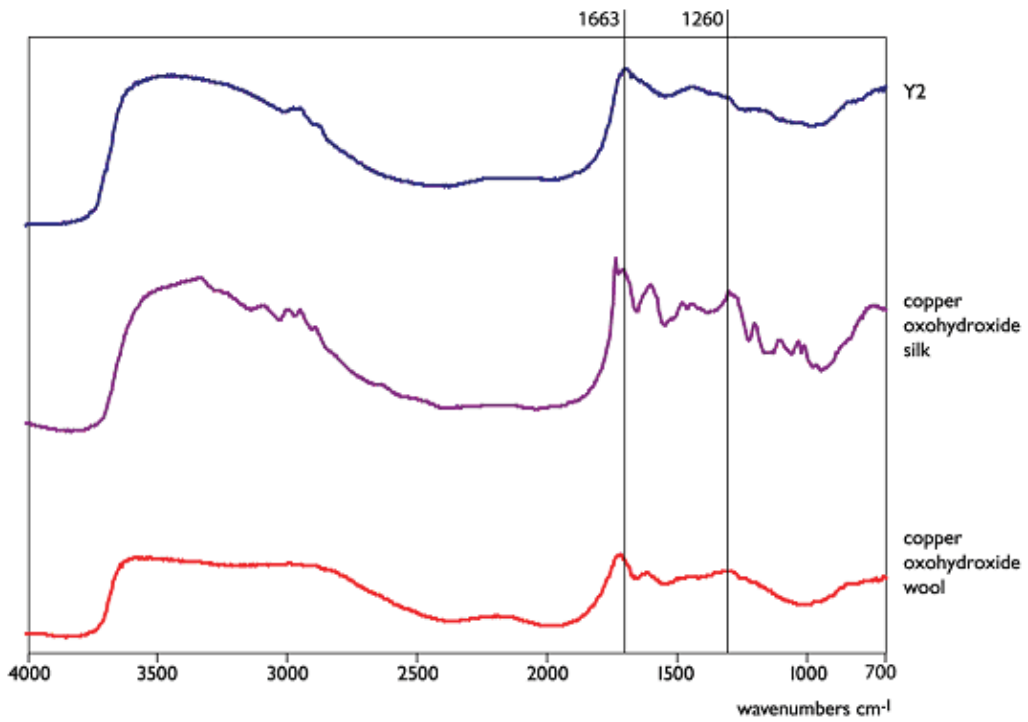


Fig. 15. FTIR microscope reflectance spectra of Y2 (top), and the reference samples: a) copper oxohydroxide silk (middle), b) copper oxohydroxide wool 1 (bottom) (© authors).

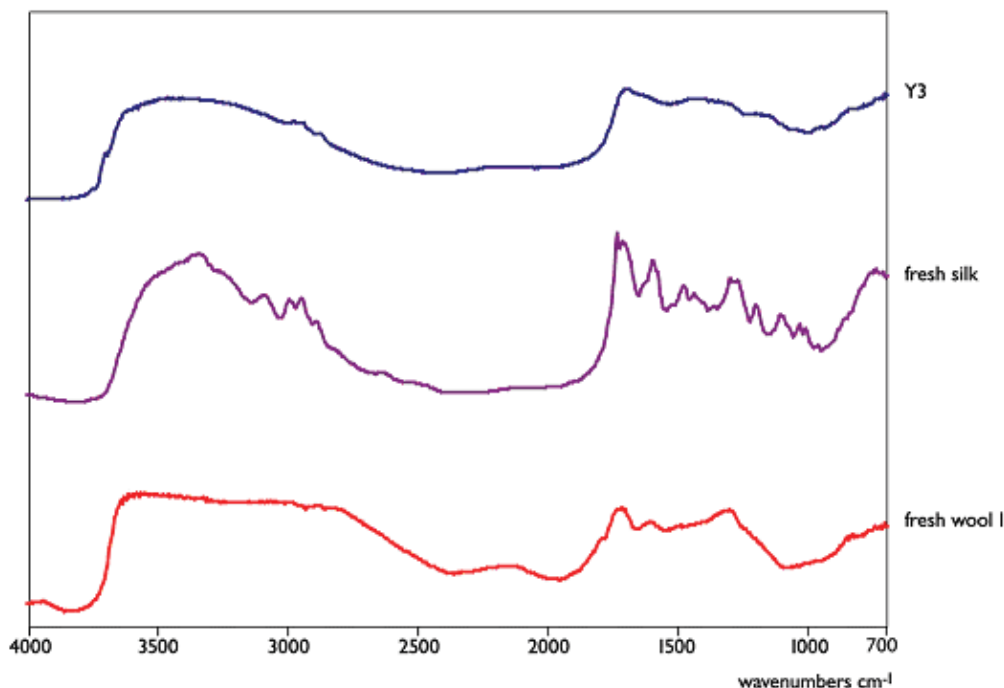


Fig. 16. FTIR microscope reflectance spectra of Y3 (top), and the reference samples: a) 'fresh' silk (middle), b) 'fresh' wool (bottom) (© authors).

## Discussion

The application of stereomicroscopy was useful in identifying the method of construction of the three textiles and their yarns. Selvages and starting edges have been retained. It is constructed from more than one narrow piece of textile, stitched together along their selvages. Textile Y2 is a very fine fabric of open plain-weave. Selvages have also been retained. Textile Y3 is a weft-faced plain-weave fabric with evidence of starting/side edges, but none of selvages.

The ESEM provided much higher magnification and allowed examination of the whole surface of the samples. It therefore provided some indication on fibre identification and much information on the characterisation of the condition of the fibres. Textile Y1 (both warps and wefts) and the warps of textiles Y2 and Y3 are possibly made of cellulosic, probably bast fibres. The wefts of textile Y2 could be made of wild silk fibres, and the wefts of textile Y3 were possibly made of wool. A more comprehensive reference collection would be necessary for conclusive fibre identification. The outcomes of ESEM-EDS and XRF were consistent and characterised the type of preservation. Both techniques showed that copper and iron were mainly responsible for the preservation of the Argos find.

ESEM-EDS further aided fibre identification in the case of the Y3 wefts, by detecting sulphur, which is indicative of wool fibres. Sulphur could, of course, arise from a source of contamination; nevertheless, it was only detected in the Y3 weft fibres and in no other fibres or the debris.

FTIR microscopy provided information on the characterisation of the condition of the textiles by indicating that they were heavily masked and/or impregnated by degradation products but there were organic remains still present. Fibre identification results were inconclusive, yet consistent with the ESEM results, thus complementing fibre identification conclusions.

In general, the results from all techniques were consistent. Consistency was very important since no one technique was sufficient to provide reliable results, especially as far as fibre identification was concerned. For example, ESEM analysis indicated textile Y1 was made of cellulosic, probably bast fibres, which was further suggested by FTIR analysis. Similarly, ESEM revealed a pattern reminiscent of wool epithelial scales in Y3 weft fibres, which was consistent with the detection of sulfur by ESEM-EDS analysis.



### Conclusion

The information drawn from these analyses could be used to better illustrate the way the textiles had been placed in the funerary vessel. A significant number of Y3 fragments are green-coloured, which, as analysis showed, means that they are impregnated/masked by copper degradation products. In addition, the Y3 fibres seem to be mainly preserved as negative casts. Iron, which was present in the vessel, is generally responsible for the creation of negative casts of textile fibres (Gillard *et al.* 1994a, 133, 137-138; Coho 1996, 70; Janaway 1989, 21). These results indicate that Y3 was the textile most likely to have been in direct contact with the degrading metals of the funerary vessel, and therefore it is reasonable to deduce it must have been the textile laid at the bottom of the copper vessel.

The main mass of Y1 consists of multiple layered folds of cloth, which also contain the fruit offered and the bones of the deceased. There are very few Y1 fragments impregnated with green copper degradation products, hence this textile must have not been in direct contact with the funerary copper vessel. Therefore, Y1 is most likely to have been the textile used to wrap the both the deceased's bones remaining from the pyre (and possibly ashes) and the fruit offered at its funeral, and then placed on Y3.

The Y2 single-layer fragments were detected adhered to the top surface of the Y1 unified mass. On the other hand, three-dimensional Y2 fragments, consisting of multiple single layers, have been found around the edges of the Y1 unified mass. The bottom side of several of them, along with certain selvedge fragments, had a bright green colour, indicative of impregnation and hence direct contact with the copper corrosion products. Therefore, the fine, semi-transparent Y2 textile may have been laid on top of Y1 to cover it, the remaining width, gathered around the edges of the folded Y1, coming in contact with the sides of the copper funerary vessel.

Although the quality of these analytical results might be hindered by the poor condition of the specimen and/or limited sources of reference, the combination of the results from all techniques applied revealed information on the find that should be useful both to the conservator and the archaeologist and textile historian.

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