# RESEARCH



# Exploring the provenance of a Byzantine excavated assemblage of textile and leather finds by the application of instrumental analysis

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# Abstract

Stereomicroscopy, Scanning Electron Microscopy-Energy Dispersive Spectroscopy (SEM–EDS), Fourier Transform Infrared spectroscopy with Attenuated Total Reflectance probe (FTIR-ATR), High Performance Liquid Chromatography with a Photodiode Array system (HPLC–PDA), X-ray Fluorescence spectroscopy (XRF), Zooarchaeology by Mass Spectrometry (ZooMS), and <sup>14</sup>C dating were applied to an assemblage of textile and leather fragments that belong to the collection of the Byzantine & Christian Museum of Athens in Greece and lacked inventory information.The analyses applied, along with bibliographical research, yielded information on the materials, techniques and provenance of the find. The assemblage of finds consists of a gold thread embroidered silk satin, dyed with shellfish-purple; a gold strip-drawn wire embroidered silk tabby; a gold strip-drawn wire and red silk thread tapestry; a gold-thread embroidery on a tabby background; fragments of braided cord; and leather fragments. The material components could have been produced locally, in the area of the Byzantine Empire, though some of them indicate connections with India and/or China. The assemblage, dated between the mid AD 10th and mid eleventh century, was probably found in 1924 by Andreas Xygopoulos during the excavation of a tomb inside the church of Agia Sophia in Thessaloniki, Greece.

**Keywords** Byzantine textiles, Silk fibres, Gold threads, Satin, Shellfish purple, Alizarin, Leather shoes, Radiocarbon dating, Proteomics

# Introduction

Textiles are generally rare excavation finds, due to their organic nature, which is highly susceptible to the aggressiveness of the burial environment and requires specialised recovery techniques [e.g. 1; 2; 3]. This is usually the

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<sup>4</sup> Byzantine & Christian Museum, 22 Vasilissis Sofias Avenue, 10675 Athens, Greece case in the semi-arid Greek archaeological environment, which is characterised by constant fluctuations in relative humidity and temperature. Therefore, fragments of organic material recovered from excavations trigger a special research interest. The process of reconstructing information of the past from material remains requires a scientific approach [4]. Furthermore, the fundamental principle in the conservation of excavated textiles is the preservation of the material and the information this may yield [5]. The poor condition of textile finds and the fact that archaeological documentation may be missing, make the application of analytical methods and instrumental analysis necessary in order to reveal cultural and historical information [6]. In addition to this, the rarity of survival and the poor state of preservation of archaeological textile finds may lead to the preference of carrying



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out investigative instead of remedial conservation, as this could be the most appropriate method to apply, in order to retrieve and retain important information contained within the textile finds that may otherwise be lost [5, 7].

The excavated find, discussed in this paper, belongs to the collection of the Byzantine & Christian Museum (BCM) in Athens. It is in fact, an assemblage of organic finds that were brought and stored in the BCM at some, unknown, point in the past. The assemblage was kept in a wooden, portable showcase, stored probably for decades in the museum depository without any inventory number or any kind of record. It was located in the early 2000's when the collection was transferred to the new storage rooms and was given the accession number BXM 21359. Up to the point of this publication, all research into the museum's accession catalogues, its history, its acquisitions, exhibitions catalogues and publications has had no results in finding information on provenance. A brief history of the museum shows that a part of its collection was amassed by the Christian Archaeological Society (ChAE), founded in 1884, under the supervision of theologian Georgios Lambakis, secretary to Queen Olga [8]. The collection of ChAE was initially housed at the Holy Synod in Athens (1890) and was then moved and housed at the National Archaeological Museum until 1923 [9]. In the meantime, in 1914, the BCM was established by act of Law 401, and was led under its first director Prof. Adamantios Adamantiou and a Supervisory Board. It was then that the BCM started to gather antiquities and works of art through donations and acquisitions to form its collection [10]. Right after its establishment, in 1915, the first professional conservation services started in its facilities [11]. During World War I, in 1915, Allied troops landed at the port of Thessaloniki and combined to form the Army of the Orient under French commandment. Following instructions of the Ministry of Church Affairs and Public Education, Prof. Adamantiou travelled to Thessaloniki where he collected more than 1600 antiguities, which were transferred and stored or displayed at the BCM until 1994 [12]. In 1923, Georgios Soteriou took over as director of the BCM and one of his first actions was to incorporate the collection of ChAE. Soteriou curated the first exhibition of the collection in the building of the Athens Academy in 1924 and published the first collection guide. In 1926, the Greek state ceded Villa Ilissia to the BCM and its collections were transferred there. In 1930 Soteriou opened the museum's doors to the public, showing its permanent collection and published a new collection guide [13] while also providing new workrooms for the artists-conservators he had previously hired in1923 [11].

Since the finds lacked documentation and provenance information, it was decided to apply instrumental analyses in order to retrieve information on their construction, material identification and dating. At the same time it was necessary to follow the Greek Law on the protection of antiquities and cultural heritage (N. 4858/2021) that requires minimum sampling and the preference of non-destructive over destructive methods when applicable.

The small dimensions of the primary structures of textiles (i.e. fibres and threads) necessitate the use of some kind of magnification for their study. A stereomicroscope or a handheld digital microscope are the basic instruments textile researchers use to study the weave type and thread count, and the technological features of threads, such as diameter, twist direction, degree and angle [14-17, 19]. The identification of fibres demands higher magnifications than those delivered by the stereomicroscope. The Scanning Electron Microscope (SEM) is particularly useful for the identification of fibres based on the study of their morphological characteristics, since it affords very high magnifications and large depth of field [15, 17, 19, 20]. Variable Pressure SEM may operate at low vacuum or atmospheric pressure in the chamber [21, 22]. VP-SEM provides charge neutralisation, enabling non-conductive samples like textile fibres to be examined without the need of a conductive coating, thus making the analysis less invasive. SEM has even been successful in the identification of fibres preserved in poor condition [23].

SEM can support X-ray analysis when equipped with an X-Ray micro-analyser to perform Energy Dispersive Spectroscopy (SEM–EDS). SEM–EDS has been applied for the study and analysis of metal threads to determine the production method, technological properties and composition of the metal strip, as well as the type of fibre of the core [24, 25]. It can also provide information on the mordants, the metal salts used in the dyeing process to form stable complexes between the dye molecules and the textile fibres [15, 24, 26]. Mordant identification can also be accomplished by X-ray Fluorescence (XRF) a non-destructive and non-invasive technique, frequently used for the study of inorganic elements in cultural heritage objects [16, 19, 26–28].

Fourier Transform Infrared spectroscopy with an Attenuated Total Reflectance probe (FTIR-ATR) is able to identify bonds within the molecules of the substance analysed [29]. It has been broadly used for the non-destructive characterisation of textile fibres [19, 30], and even for the characterisation of different silk species [31]. It is considered suitable for fibre identification of heritage textiles, as it offers highly characteristic information, it is non-destructive, fast, and relatively easy and inexpensive [32]. However, the characteristic information offered seems to be limited in the cases of heavily degraded fibres of excavated textiles [33].

High Performance Liquid Chromatography with a Photodiode Array system (HPLC–PDA) is a destructive analysis applied for the identification of dyes and it is based on comparison with known reference compounds [25]. Although destructive, HPLC has traditionally been applied for dye analysis of archaeological and historical textiles [19, 28], most probably because it has proven to be successful even in cases where dye concentration is as low as a few nanograms [26].

Radiocarbon dating has been applied to textiles on multiple occasions, since their organic nature makes them suitable for this type of analysis [34]. In Peru, 14C dating of the textiles from mummy bundles informed the chronology of the Chancay and Huaura valleys that lacked pottery seriation [35]. It also enabled authentication of two pre-Columbian ponchos from the Quai Branly Museum (Paris) that lacked provenance information [36]. 14C dating of leather has been a challenging issue partially because leather is rare in the archaeological record and also because its production techniques are for the most part unknown. Recent studies showed that pretreatment of leather and skin samples with XAD column chromatography purification (as compared to ABA) resulted in more accurate dating results. However, this pretreatment poses higher risks to the sample's preservation [37].

## **Materials and methods**

#### The find BXM 21359

The assemblage is preserved in a fragmentary condition and consists of numerous textile fragments, several leather fragments, a braided cord, a braided knot and minute pieces of both textile and leather. More specifically, the find consists of numerous small-sized textile fragments that form alternating layers, measuring from approx.  $2 \times 2$  mm to  $50 \times 60$  mm; the vast majority though measure less than  $0.5 \times 0.5$  mm. Leather fragments are much more limited than those of textile; four (4) of them have larger dimensions of approx.  $120 \times 40$  mm to  $80 \times 40$  mm, two (2) of approx.  $50 \times 40$  mm and numerous other smaller ones. Additionally, a large amount of pulverised organic material, such as tiny particles of fibres and leather as examined under a stereomicroscope, was isolated.

The majority of the textile fragments stand in layers. The top layer is either woven or embroidered with metal threads or strip-drawn wires [38]. There are few one-layer fragments of tabby textile. The condition of the finds was poor. Both textiles and leather are hydrophobic and brittle, unable to withstand handling without loss or breakage. The leather fragments were severely warped and delaminated. Few of the textile and leather fragments were masked by white-coloured, hard deposits. Based on its condition, it was assumed that it is an excavated find, though without any available information on the excavation site and date or any documentation of first aid or conservation treatment in the past.

According to the state of preservation and the lack of provenance information of the finds, it was decided to apply specific analytical techniques. Some of these techniques are destructive and the sample would be completely consumed by the analysis and/or could no longer be physically available to associate with the fragments [39]. For ethical reasons, it was decided to select samples from the smaller fragments, and only when necessary to cut them from the larger ones. To achieve that, all fragments were initially removed from the showcase, separated and grouped according to material, type of textile (weave type and decoration), colour and fragment size. All different kinds were then registered as Y1, Y2 ecc. up to Y8. The samples were firstly analysed with nondestructive techniques (like spectroscopy and SEM) and then were used for the other, destructive, analysis such as dye analysis, radiocarbon dating and proteomics.

## **Bibliographical research**

Research in the inventory catalogues, conservation and archaeological archives of the BCM did not provide any information on the find. Neither did the research on the available photographic archives, or guides and publications both of BCM's and ChAE's. Bibliographical research revealed an appendix in Archaiologikon Deltion publication, by Andreas Xygopoulos [40], a Byzantinist archaeologist who was the Director of the Ephorate of Antiquities of Macedonia in 1924 and collaborated with Georgios Soteriou at the BCM [13]. Xygopoulos reports the excavation of two tombs inside the church of Agia Sophia in Thessaloniki in 1924. Both tombs were built with marble slabs that had no decoration apart from one of the slabs in the second tomb (B) that bore an inscription, which was ineligible as it was not well-preserved. However, in that tomb, the bones of the body, along with textile remains, had been preserved. According to Xygopoulos, three (3) different textiles were identified: a very fine purple textile with gold-woven decoration, a coarser textile with similar gold decoration, and a very fine purple textile with gold wire embroidery. A drawing of the third textile's decoration with its design development was included in the publication [40]. The similarity of this depiction with some fragments of the finds was a first indication of the probable provenance of the find BXM 21359 (Fig. 1). Xygopoulos [40] reports that there was no evidence of the identity of the deceased and made the hypothesis that the tomb belonged to a prominent person of the Palaiologeian period (1241–1453).



**Fig. 1** Drawing by Xygopoulos of textile Y3 retrieved in 1924 from tomb B inside Agia Sophia of Thessaloniki (top), and selected fragments of the find presented in this paper mirroring the pattern shown in the drawing. © Xygopoulos 1927 (top), *Byzantine & Christian Museum* (bottom)

#### Analytical techniques applied

Visual observation, technological analysis, material identification, elemental analysis of the metal threads and fibre deposits, and radiocarbon dating were applied to shed light on the provenance of the find. Visual observation was aided by photography at 300dpi,  $6016 \times 4016$ pixels with a NIKON D750 digital camera. Larger size fragments were photographed on both sides with the camera mounted on a vertical stand. They were lit by two soft-box photography lights set at 45° angles from the stand where the fragment was placed. Study of the weave and thread measurements were recorded with a Dino-Lite AM413T digital stereomicroscope and a LEICA MZ6 stereomicroscope coupled with a BASLER Micro B 90Ris/A, 3 m N digital camera (Table 1).

Technological features and material identification of textile and leather fragments were studied by Scanning Electron Microscopy (SEM) (JEOL JSM-6510LV) in backscatter mode at 15 and 20 keV. Samples were selected from amongst the loose minute fragments of the find and not cut from the larger ones. The SEM stubs (sample carriers) were lined with a piece of double-sided carbon-coated tape and the samples were secured on them by light pressure applied locally with the tips of forceps. The samples were neither coated nor prepared by any other method. The surface and not cross-sections of the samples were analysed, to make the analysis less invasive. Energy Dispersive Spectroscopy (EDS) was applied for spot elemental analysis by an Oxford Instruments AZtec X-ray detector (coupled with the SEM).

X-ray Fluorescence spectroscopy (XRF) (NITRON XRF spectrophotometer xlt Gold) was applied to textile fibres for elemental analysis with a minimum 10 measurements per point, acquisition time 90 s, and 1.5 mm spot size. Fourier Transform Infrared (FTIR-ATR) spectroscopy was applied to enhance fibre identification. FTIR analysis (Alpha Bruker with Attenuated Total Reflectance probe) was applied with min 10 measurements per point, acquisition time 32 s, normalised power, and hundreds of  $\mu$ m (ATR probe) spot size. Two (2) loose fragments of Y1 and a 2×2 mm fragment cut from Y5 were selected as samples for XRF and FTIR-ATR.

High Performance Liquid Chromatography with a Photodiode Array system (HPLC–PDA) was applied for dye analysis. Samples from textiles Y1 (4×4 mm-loose), Y3 (3×2 mm-cut) and Y5 (2×2 mm-cut) were used for the analysis. The colourants were recovered from the fibres using acidic extraction with hydrochloric acid, followed by ethyl acetate extraction [41]. Extraction was done in 250  $\mu$ L water/methanol/37% HCl (1/1/2, v/v/v) for 10 min at 105 °C, followed by a second extraction with 500  $\mu$ L of ethyl acetate—vacuum evaporation—dissolving the residue in 30/30  $\mu$ L methanol/water from which 20  $\mu$ L was injected for analysis.

A protein-based species identification method, namely zooarchaeology by mass spectrometry (ZooMS) was applied to loose leather fragments for material identification. ZooMS investigates the differences in the sequence of collagen type I found in different genera and in some cases species. Two (2) loose fragments were selected as samples (approx.  $2 \times 3$  mm). They were pretreated/ washed with sodium hydroxide and ammonium bicarbonate and digested in the enzyme trypsin at  $37^{\circ}$  for 18 h, and subsequently analysed using a MALDI TOF Mass Spectrometer in reflector mode. Mass spectra were acquired over the m/z range 800–4000. Spectral analysis was performed by the open source, cross-platform software mMass (www.mmass.org) [42].

14C determinations of the textile (Y3 fragment,  $3 \times 4$  mm) and leather (fragment  $4 \times 4$  mm) samples were measured on the Accelerator Mass Spectrometer (AMS) at the Horia Hulubei National Institute for Research & Development in Physics and Nuclear Engineering, Romania (lab code RoAMS). Samples were pretreated with ABA, and graphitisation was performed with the system CHNOS Elemental Analyser /AGE 3 and pressed into the AMS cathodes [42, 43]. The samples were measured in the AMS spectrometer MICADAS, normalising them to the Oxalic Acid II age standard, and subtracting the 14C blank levels using an unknown deep geological

Analysis	Material	Purpose	Result
Stereo microscopy	Textile	Technological and weave analysis	a) 4 types of weave/construction: 1. Very fine and coarser tabby (Y1),* 2. Weft-faced tabby (Y6); 3. Slit (Y2) and toothed tapestry (Y4, 5); 3. Satin (Y3); Braided cord (Y8) b) 2 types of decoration: 1. Embroidery (Y3, Y7); 2. Dye (Y1, Y3)
	Metal threads	Technological analysis	2 types of metal threads: 1. Strip-drawn wire (Y1, Y2, 4, 5?); 2. Strip-twisted wire (Y3, Y6, Y7)
	Leather	Technological analysis and grain pattern analysis	Evidence of stitching/stitching holes; inconclusive but follicle distribution could be indicative of cattle leather
SEM	Textile (applied to samples from Y1-Y8)	Fibre identification	Silk (?) for the textiles, couching threads and metal thread cores. Cellulosic bast fibres for the coated lining of Y1, and the warps of Y6
SEM-EDS	Metal threads (applied to samples from Y1-Y8)	Metal analysis	2 types of metal threads: 1. gold (Y1, Y2, Y3, Y4, Y5, Y7); 2. Gold-silver alloy (Y2, Y4, Y5, Y6)
FTIR-ATR	Textile (applied to samples from Y1, Y5)	Fibre identification	2 types of fibes: 1. Silk ((Y1, Y5); 2. Cellulosic bast (Y1)
	Textile (applied to samples from Y1, Y5)	Dye analysis	Iron(II)oxide mordants (Y1, Y5)
XRF	Textile (applied to samples from Y1, Y5)	Dye analysis and identification of white colour deposits	Iron mordants; Ca salts (Y1, Y5)
HPLC-DAD	Textile (applied to samples from Y3, Y5)	Dye analysis	Alizarin (Y5); Shellfish purple (Y3); ellagitan- nins (Y1, Y3)
	Textile (applied to sample from Y1)	Material identification of black colour coating	Shellac resin (Y1)
ZooMS	Leather (applied to a sample from the leather fragments)	Species identification	Inconclusive results, indicative of the Bovine/Cervidae family
<sup>14</sup> C	Textile (applied to sample from Y3)	Dating	mid-10th to early eleventh century AD
	Leather (applied to a sample from the leather fragments)		

Table 1	Summar	y of the	results	of the	instrume	ntal a	analy	yses	applied	
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<sup>\*</sup> the letter Y was used for the different textiles as it is the initial of the Greek word for textile ( $Y\varphi a\sigma\mu a$ )

deposit coal [43]. This blank was tested and compared with Merk<sup>®</sup> synthetic graphite (Carbon graphite powder, 99.9% purity). The raw data was reduced to radiocarbon ages. 14C calibrations were performed using OxCal 4.4.4 and the IntCal20 calibration curve date [42, 44–48].

## **Results and discussion**

#### Technological analysis

Technological analysis of the textile fragments was particularly challenging. Handling was extremely difficult because of their brittleness and fragility; even minimal act with forceps and spatulas was of high risk. Studying both sides was quite perilous for the same reason; dividing different layers was unattainable and the observation of plain textile without any decoration were extremely limited. At the very beginning of the project, all fragments of substantial dimensions ( $\geq 0,5$  mm) were photographed from both sides to minimise handling. Eight (8) different textile structures were identified (7 textiles and 1 piece of cord), based on different weave types and metal thread decoration (Fig. 2). The registration numbering (Y1-8) of the different textiles types was given according to the quantity of the different types of finds and so forth (Table 2).

# Textile Y1

Textile Y1 is an embroidery with a chequered pattern, most probably a cross-shape design, formed by the alternating direction of the metal threads in small adjacent squares. The embroidery is executed with couched gold threads on a balanced background tabby of approx.  $40 \times 40/\text{cm}^2$  weave count. The gold threads seem to be strip-drawn wire, produced by hand by taking a strip of metal foil and drawing it through holes of decreasing diameters so that they curl upon themselves and the organic core [38]. The weaving threads are of  $0.1 \pm 0.02$  mm diameters with no apparent twist. The background textile is completely covered with metal thread embroidery couched by very fine threads with no twist (Fig. 3). The metal threads are of approx.



Fig. 2 Examples of fragments of the different textiles. Scale bar 10 mm. © Nikos Mylonas, Byzantine & Christian Museum

 $0.26 \pm 0.02$  mm diameter, with a metal strip of approx.  $0.27 \pm 0.01$  mm width, densely wound or curled around a thread core in a Z direction, with approx. 3 coils/ mm (the organic core was not meant to be visible). The metal strip is of  $5.3 \pm 0.2 \,\mu\text{m}$  thickness and made of gold (90.72% Au), as EDS analysis indicated (Table 2). Judging from the wavy and rounded edges of the strip, it can be assumed that it was made by wire flattened by rolling instead of a foil cut into strips, but it is too thin to draw decisive conclusions. The surface of the strip on both sides is very smooth, and shows no signs of forging [49]. The organic core is of  $90 \pm 5 \mu m$  diameter of Z twist with an approx.  $20^{\circ}$  medium twist angle [50]. The threads of the background fabric, the couching threads and the organic thread cores of the metal threads do not have distinctive characteristic features but their fine diameters of approx.  $5 \pm 1 \mu m$ , indicate silk fibres. EDS analysis of the core thread detected calcium, most probably derived from the bones of the deceased; and gold probably from the deteriorated foil of the metal thread (C K 49.62%, O K 37.67%, Ca K 1.17%, Au M 11.54%) [16]. The stratigraphy of most Y1 fragments, starting from the top layer is: A) gold thread embroidery on a tabby background fabric; B) a thicker, balanced, tabby textile used probably as a lining (approx.  $50 \times 50/cm^2$  weave count with  $0.16 \pm 0.02$  mm thread diameter of Z twist and an approx. 20° medium twist angle [50]); evidence of stitching threads/holes was detected on this textile, as well as a coating of a black coloured substance; C) leather.

# Textile Y2

Textile Y2, a tapestry with geometric patterns, formed by alternating shiny and matte gold weft threads or, in a few areas, by rose-gold threads that create crosses with stepped edges (Fig. 2). The background weave is a tabby of approx. 60 warps  $\times$  80 wefts/cm<sup>2</sup> weave count with  $0.05 \pm 0.01$  mm warp and  $0.1 \pm 0.02$  mm weft diameters of Z twist with an approx.  $\leq 10^{\circ}$  loose twist angle (Table 2) [50]. The state of preservation of the metal thread's foil was very poor. Therefore, studying the technology and dimensions was particularly challenging. It was not possible to understand whether they were made with stripdrawn or strip-twisted gold wire and measurements are largely approximated. The shiny metal threads are of approx.  $0.1 \pm 0.02$  mm diameter, made of a metal foil rolled/wound around a most probably silk thread core. The direction of rolling/twisting and whether the foil was cut or flattened into a strip of a specific width could not be detected (Fig. 4). The metal foil is of  $1 \pm 0.05 \ \mu m$ thickness made of gold as shown by EDS (83.67% Au). The core is of a  $50\pm5$  µm diameter made up of fibres of  $3.5 \pm 0.5 \,\mu\text{m}$  diameters with no or very loose S twist (detected only in certain areas with  $a \leq 5^{\circ}$  twist angle), which are most probably silk. EDS analysis of the core detected calcium, most probably derived from the bones; silicon and aluminium were attributed to soil contamination (C K 44.30%, O K 45.48%, Al K 2.79%, Si K 2.97%, Ca K 4.45%) [16]. The rose-gold coloured metal threads appeared very similar in condition and construction to

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Textile	Construction/decoration of textile	Weave	Threads	Metal threads diameter (ø)	Metal threads construction	Metal threads composition Weight%
71	A. Couched embroidery cheq- uered pattern (crosses?) made of metal threads on a tabby back- ground fabric;	Background fabric, plain weave 40×40/cm <sup>2</sup>	Background fabric 0.1 ±0.02 mm Ø with no apparent twist, silk(?)	0.26±0.02 mm	Strip-drawn wire, strips 0.27 ±0.01 mm wide, 5.3 ± 0.2 µm thick, wound around a silk(?) core in a Z direction, with approx. 3 colls/mm	C K 6.71 O K 2.57 Au M 90.72
	B. fabric coated with shellac	Plain weave, 50 × 50/cm <sup>2</sup>	0.16±0.02 mmø with a Z twist and an approx. 20° medium twist angle	N/A	N/A	N/A
	C. leather	N/A	N/A	N/A	N/A	N/A
72	Slit tapestry of geometric pattern with silk(?) warps and silk(?) and metal thread wefts	Silk: tabby of approx. 60warps x 80wefts/cm <sup>2</sup>	Silk: 0.05 ± 0.01 mm ø silk(?) warp and 0.1 ± 0.02 mm ø weft of Z twist with an approx. ≤ 10° loose twist angle	Shiny, gold colour: 0.1 ± 0.02 mm	Strip-drawn wire of 1 ±0.05 µm thick strips wound around a silk(?) core	C K 8.54 N K 4.53 O K 3.26 Au M 83.67
				Shiny, rose gold colour: as above	As above	C K 8.54 N K 5.03 O K 2.53 Ag L 8.78 Au M 75.12
				Matte: $65 \pm 5 \mu m$	0.5 µm thick, as above	C K 14.79 O K 10.09 Au M 75.12
ß	Couched embroidery of floral motifs	4/1 satin weave (regular ?) 60 floatsx50/cm <sup>2</sup> ; the floats are dyed purple	Silk(?): float thread 0.13 $\pm$ 0.02 mm <b>Ø</b> with no apparent twist; other set of elements 0.13 $\pm$ 0.02 mm <b>Ø</b> of Z twist with an approx. $\leq$ 10° loose twist angle	0.3±0.02 mm	Strip-twisted wire of approx. 0.40 ±0.05 mm width strip, wound around a silk(?) core in a Z direction, with approx. 3 coils/mm	C K 9.30 N K 4.82 O K 5.32 Au M 80.55
74	Dovetail tapestry of geomet- ric pattern with silk(?) warps and silk(?) and metal thread wefts	Similar to Y2	Similar to Y2	Similar to Y2 matte	Gold colour, similar to Y2 matte	C K 9.23 N K 4.57 O K 5.82 Au M 80.38
				_	Rose gold colour, as above	C K 8.37 O K 6.22 Ag L 10.33 Au M 75.08
Υ5	Dovetail figurative tapestry with silk(?) warps and silk(?) and metal thread wefts	As above	As above	As above	As above (no rose gold threads detected)	As above (no rose gold threads detected)

Table 2 Technological analysis and material identification of the textile samples

Textile	Construction/decoration of textile	Weave	Threads	Metal threads diameter (ø)	Metal threads construction	Metal threads composition Weight%
46	Weft-faced fabric made of plant fibres (the wefts are metal threads)	26 wraps x 42 wefts/cm <sup>2</sup>	N/A	0.3±0.02 mm	Strip-twisted wire of approx. 0.25 mm width and 4±1 µm thickness, wound around the plant fibre core in S direction at approx. 3 coils/mm	C K 7.06 O K 5.37 Ag L 29.43 Au M 58.15
4	A. Couched embroidery of figura- tive pattern	Tabby	0.2±0.02 mm approx	Similar to Y3	Similar to Y3	C K 8.34 N K 4.62 O K 5.57 Au M 81.47
¥8	B. fabric coated with shellac(?) Braided cordage of 0.5 mm width	Similar to Y1.B N/A	Similar to Y1.B Double Z twisted threads of 0.3±0.1 mm ø	N/A N/A	N/A N/A	N/A N/A

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**Fig. 3 Top:** Scanning electron micrograph of the metal threads of textile Y1. The fine couching stitches that secured the metal thread to the background fabric are clearly visible. The thin metal foil has deteriorated in many areas exposing the organic core of the metal threads. **Bottom:** EDS spectrum of the metal foil of textile Y1, showing the foil is made of gold. © *Christina Margariti and Daphne Filiou* 

the shiny gold ones. What was markedly different was the EDS spectrum, which detected 8.78% silver (Ag) and 75.12% Au. The matte metal threads are of approx.  $65\pm5$  µm diameter, made of a metal foil rolled/wound around an organic thread core. Similarly in this case, neither the direction of rolling/twisting nor the standard width of the foil strip, could be recorded (Fig. 4). The metal foil is of approx 0.5 µm thickness and made of gold with a similar EDS spectrum as Y1 (Table 2). The core is of a  $48 \pm 2 \mu m$  diameter made up of fibres of  $3.5 \pm 0.5 \mu m$ diameters with a loose S twist with a  $\leq 10^{\circ}$  twist angle. The warp threads and the organic thread cores of the metal threads do not have distinctive characteristic features but their fine diameters indicate silk fibres. EDS analysis of the core thread detected calcium, most probably derived from the bones of the deceased and gold probably from the deteriorated foil of the metal thread (C K 14.58%, O K 11.15%, Ca K 1.09%, Au M 73.19%) [16]. The tapestry is a weft faced weave, and one of its main characteristics is that the weft is interwoven with the warp only in the areas required by the pattern [51]. If there is no connection of the different weft areas, and each weft turns back round the marginal warp of its own area, it creates a slit/



**Fig. 4 Top:** Scanning electron micrograph of an area with shiny metal threads of textile Y2. **Bottom:** Scanning electron micrograph of an area with matte metal threads of textile Y2. © *Christina Margariti* and Daphne Filiou

unjoined tapestry [50-52]. Textile Y2 is a slit/unjoined tapestry since several areas were located where the gold decoration ended and both the metal and organic adjacent threads were turned around their marginal warp (Fig. 5).

#### Textile Y3

Textile Y3 is an embroidery with a design of repetitive circular medallions that enclose alternately a floral motif and a four-leaf cross. This design resembles the one drawn by Xygopoulos (Figs. 1, 2) [40]. The embroidery is executed with couched gold metal threads on a float weave background textile, made of bluish-purple floats on brown threads [50]. The background textile is a 4/1 float weave, where 1 thread of the elements of one direction (warps or wefts) passes over (floats) 4 threads and under 1 thread of the other direction's set of elements (Fig. 6). The diagonal alignment of the floats and the binding points (pass under) are always single but intermittent, thus indicating it is a 4/1 or 5 ends



**Fig. 5** Detail of textile Y2 slit tapestry fragment, showing an example of a slit between the gold decoration and the main fabric. Scale bar 0.5 mm. © *Christina Margariti and Daphne Filiou* 



**Fig. 6** Detail of the 4/1 satin background fabric of the Y3 embroidery, where 1 thread of the bluish-purple elements passes over 4 threads and under 1 thread of the set of brown elements. Scale bar 1 mm. © *Christina Margariti and Daphne Filiou* 



**Fig. 7** Scanning electron micrograph of the metal threads of textile Y3. The fine couching stitches that secured the metal thread to the background fabric are clearly visible. The metal foil is wound around the organic core in Z direction. © *Christina Margariti and Daphne Filiou* 

satin weave fabric [50, 51]. However, due to the poor condition it was not possible to determine whether it is a warp or weft-faced and whether it is a regular or irregular satin. The back of the textile has a diagonal effect, therefore it is probably a regular satin, but more areas would be necessary to measure for a conclusive result [50]. The weave count is approx. 60 floats  $\times$  50/  $cm^2$  with aprox.  $0.13 \pm 0.02$  mm float thread diameters with no apparent twist, and a bluish-purple colour; and 0.13±0.02 mm the other set of elements' diameters of Z twist with an approx.  $\leq 10^{\circ}$  loose twist angle, and a brown colour. Similarly to textile Y1, couching stitches made with a group of very fine threads with no twist, have been used to secure the gold threads of the embroidery to the satin background. The metal threads made with a strip-twisted gold wire [38] densely wound around the organic core are of approx.  $0.3 \pm 0.02$  mm diameter. The foil was flattened or cut in approx.  $0.40 \pm 0.05$  mm width strips, wound around a thread core in a Z direction, with approx. 3 coils/mm (Fig. 7, Table 2). The metal foil is of  $5 \pm 1 \mu m$  thickness and made of gold (Au 80.55%) as EDS analysis indicated. The organic core is of  $80 \pm 10 \ \mu m$  diameter of Z twist with an approx. 20° medium twist angle [50]. EDS analysis of the core detected calcium and phosphorus, most probably derived from the bones; as well as aluminium and silicon from soil contamination (C K 46.57%, O K 44.51%, Al K 1.62%, Si K 1.73%, P K 1.56%, Ca K 4.19%) [16]. The threads of the background textile, the couching stitches and the organic cores of the metal threads do not have distinctive characteristic features and are of very fine diameters of approx.  $5 \pm 1 \mu m$ , indicative of silk fibres.

# Textiles Y4 and Y5

Textile Y4 is a tapestry of a geometric pattern that forms rectangles in a set of three with a diagonal layout. It is executed in gold thread and in certain cases rose-gold coloured metal threads (Fig. 2). In both cases, the rectangles are of approx. 1.5×3 mm dimensions. The background weave and the metal threads are very similar to the fabric and the matte metal threads of Y2 (Table 2). However, it is not a slit tapestry since the wefts of adjacent areas of gold pattern and background weave seem to turn back alternately around the same warp, which is their common boundary, making Y4 a toothed tapestry [51]. The state of preservation was also similar, so the direction of rolling/twisting could not be detected. The metal foil of the gold threads is made of Au, while the EDS spectrum of the rose-gold threads showed 10.33% silver (Ag) and 75.08% Au. EDS analysis of the organic threads detected calcium and phosphorus, most probably



**Fig. 8** Detail of textile Y5 toothed tapestry fragment, showing an example of the wefts between the gold decoration and the main fabric wound around a shared warp. Scale bar 1 mm. © *Christina Margariti and Daphne Filiou* 



Fig. 9 Top: Scanning electron micrograph of the metal threads of textile Y6. Bottom: EDS spectrum of the metal foil of textile Y6, showing the foil is made of gold and silver. © *Christina Margariti and Daphne Filiou* 

derived from the bones of the deceased; aluminium and silicon from soil contamination; sulphur that could be indicative of silver corrosion; iron that could be attributed to dye mordants; and silver from the metal foil (C K 31.29%, O K 45.10%, Mg K 0.43%, Al K 2.28%, Si K 4.56%, P K 3.06%, S K 1.20%, K K 0.68%, Ca K 4.66%, Fe K 5.56%, Ag L 1.58%) [18, 25].

Textile Y5 is a figurative patterned tapestry, made with similar metal threads and on a similar background weave as textile Y4. In this case no rose-gold coloured metal threads were detected. EDS analysis of the organic threads was in agreement to previous results and detected calcium and phosphorus, as well as aluminium and silicon (C K 40.84%, N K 9.65%, O K 43.56%, Mg K 0.26%, Al K 0.63%, Si K 3.09%, P K 0.79%, S K 0.31%, Cl K 0.10%, K K 0.08%, Ca K 0.65%, Fe K 0.14%). This is not a slit tapestry, as slits were not detected in the areas where the gold wefts meet the adjacent threads. The condition of the fragments was very poor and the dimensions of the threads were so fine that the magnifications offered by the stereomicroscope were not optimal for technological analysis. Nevertheless, adjacent wefts seem to turn back alternately around the same warp, making also Y5 a toothed tapestry [51](Fig. 8, Table 2). Some textile fragments had one layer of Y4 on one side and a layer of Y5 on the other. Observation under the microscope showed that in these cases, the fragments were connected along the edge, which was and folded, indicating that Y4 and Y5 were originally part of the same textile.

### Textile Y6

Textile Y6 is a weft-faced textile of 26 wraps × 42 wefts/ cm<sup>2</sup>. The wefts are rose-gold coloured metal threads of approx.  $0.3 \pm 0.02$  mm diameter, made with a striptwisted gold wire [38] densely wound around the organic core. The metal foil is of approx. 0.25 mm width,  $4\pm1$  µm thickness strips, wound around the thread core in most probably an S direction at approx. 3 coils/mm (Fig. 9). EDS analysis of the metal foil detected 29.43% Ag and 58.15% Au (Fig. 9, Table 2). The core is made of S twisted threads at an approx. 30° tight twist angle [50], of  $0.16 \pm 0.03$  mm diameter with  $6 \pm 1 \mu$ m fibre diameter. The fibres of the core bear no distinguishing morphological features and might be silk. EDS analysis of the core threads detected calcium and phosphorus, most probably derived from the bones; sulphur that could be indicative of silver corrosion; and iron that could be attributed to dye mordants (C K 46.38%, O K 43.12%, Na K 0.18%, Mg K 0.18%, Al K 0.43%, Si K 1.35%, P K 0.56%, S K 2.52%, Cl K 0.10%, K K 0.11%, Ca K 4.69%, Fe K 0.37%) [18, 25]. The warps are generally covered by the metal thread wefts (Fig. 2). However, parts of the warp seen at the SEM showed it is made of 0.14±0.02 mm diameter threads Z twisted at an approx. 30° tight twist angle [50], with  $12\pm4 \mu m$  fibre diameters. The fibres are in a very poor condition and very small areas are visible due to the type of the weave. Nevertheless, they seem to have nodular thickenings along their length that are indicative of cellulosic bast fibres (e.g. flax). As shown in Fig. 2, Y6



Fig. 10 Leather fragments bearing evidence of stitching and stitching holes. © Nikos Mylonas, Byzantine & Christian Museum

connects with what is probably Y2, hence Y2 and Y6 are part of the same tapestry.

# Textiles Y7 and Y8

Textile Y7 is an embroidery of figurative pattern on a, most probably, tabby background textile (Fig. 2, Table 2). The preserved quantity of Y7 is particularly small and the weave type could not be identified further. The metal threads seem similar to those of Y3, but on the back side of the larger fragment there is a layer of black coloured coating as in textile Y1. Textile Y8 is a piece of braided cord of 0.5 mm width, made of double Z twisted threads of  $0.3 \pm 0.1$  mm diameters (Fig. 2). A knotted cord fragment has also been preserved.

## Leather

The leather fragments were severely distorted, delaminated, and brittle and could not withstand flattening. Two of the fragments have a pointed, almond-shape form and almost all leather fragments bear evidence of stitching holes (of approx. 1 mm diameter occurring every 4 mm) along their edges (Fig. 10). A possible interpretation of the leather fragments, based on the shape and evidence of stitching could be that they are the remains of shoes, but not enough technological features have been preserved to ascertain this hypothesis [53, 54]. The condition



Fig. 11 FTR-ATR spectra of the textile samples analysed. From the top: Y1 embroidery background fabric is the blue spectrum; Y2 geometric tapestry background fabric is the black spectrum; Y1 support fabric with black coating is the red spectrum. © *Eirini-Chrysanthi Tsardaka, Stamatis Amanatiadis, Georgios Karagiannis, Ormylia Foundation- Art Diagnosis Center* 

of the leather did not allow for conclusive species identification from grain pattern analysis. However, in one of the fragments, a smooth grain pattern was detected with what seemed to be regularly distributed follicles,which are indicative of cattle leather [42]. Similarly, it was not possible to identify the species of the leather by proteomics analysis, which was applied twice, most probably because of the poor state of preservation of the collagen.

## FTIR-ATR

FTIR-ATR spectra of the Y1 background fabric (chequered pattern embroidery) and Y5 background weave (figurative patterned tapestry) showed band assignments at 3274 cm-1 v(N-H) bending free and H-bonded, assigned to amide bonds, 1622 cm - 1 v(C = O) from carbonyl stretching assigned to amide I, 1517 cm – 1  $\delta$ (N–H) bending assigned to amide II, and 1230 cm -1 v(C-N) stretching to amide III, in peptide bonds (-CONH-) that link the aminoacids of proteins together, indicating the presence of proteinaceous/animal fibres (Fig. 11, Table 1) [55]. Taking into account the SEM results showing fibres with very fine diameters (around 5  $\mu$ m) and the absence of any characteristic morphological features, like scales (characteristic of wool fibres), it was assumed that the majority of the fibres used for the textiles and threads of BXM 21359 were silk fibres. The sample from the coated lining, present in Y1 fragments, exhibited band assignments at 1365 cm – 1  $\delta$ (C–H), 1315 cm – 1  $\delta$ (CH2), and 1029 cm -1 v(C-OH), arising from the polysaccharide components (i.e., cellulose). The band at 1545 cm-1 could be assigned to v(C=C) aromatic in-plane, indicative of the presence of lignin, suggesting that the fibres, are most probably cellulosic bast fibres (like flax, hemp or nettle) [55].]. The band at 987 cm – 1 could be assigned to iron(II)oxide vibrations and is in accordance with the XRF results presented below [56-58]. The band at 1029 cm - 1 (stretching of C-O in a hydroxyl group) could be assigned to the bone mineral hydroxyapatite,

#### Table 3 HPLC analysis results

Sample

 $\lambda$  (nm)

since as shown below, phosphorus and calcium were identified by XRF [16].

## **HPLC** analysis

HPLC detected traces of alizarin (1, 2-dixydroxyanthraquinone) on the background weave of Y5 (figurative tapestry) (Table 3). This anthraquinone compound indicates dyeing with the roots of a red dye source from the Rubiaceae family, such as, for example, cultivated European madder (Rubia tinctorum L.) (Table 1). The madder plant is a native of the Eastern Mediterranean and Southern Europe, where the root is called alizari (from the Arabic al-'usara: juice). The roots would be harvested in the autumn from at least two year old plants to provide a dye with a more intense crimson shade [59, 60]. The background fabric of Y3 embroidery (satin textile), was in such poor condition that it was not possible to separate the bluish-purple float threads from the brown ones. On this sample HPLC showed: a) brominated indigoids, 6-monobromoindigotin (major) and 6,6'-dibromoindigotin, as well as indigotin and isatin, indicative of shellfish purple; b) ellagic acid, pointing to the use of ellagitannins. Shellfish purple is a violet dye ranging in shade from purplish-red to violet-blue, varying according to the shellfish species used. It is developed by the enzymatic hydrolysis of colourless precursors of colourants present in the hypobranchial gland of purple-giving molluscs, followed by aerobic and light oxidation [59, 61]. According to Pliny the Elder (Natural History IX: 33) molluscs should be harvested in late autumn or late winter, because in egglaying periods, like spring, their secretions would have decreased colourant strength. Ellagitannins are hydrolysable polyphenols of vegetable origin. In dyeing, they were used as they form insoluble compounds with the metallic mordants and the dyestuffs. In addition, since they are associated with many plant colourants, they can act both as mordants and contributors to the final

Tapestry background weave (Y5)	14,808/01	EtAc	Traces of alizarin	255
Embroidery background fabric (Y2)	14,808/02	EtAc	61 ellagic acid, 12 6-monobromoindigotin, 11 6,6'-dibro- moindigotin, 2 indigotin, 13 U1* (rt16,1 min, 405 nm), 3 U2 (rt9,3 min, 390 nm), 1 U3 (rt19,3 min, 395 nm) 41 6-monobromoindigotin, 37 6,6'-dibromoindigotin, 11 indigotin, 11 isatin	255 288
Support fabric of Y1, coated	14,808/03	EtAc	98 ellagic acid, 2 U4 (rt20,1 min, 455 nm) U4 (rt20,1 min, 455 nm) and several unknown compounds with maxi- mum absorbance of 450/455 nm	255 450

Extr

Substances identified

\* U stands for unidentified substance; rt stands for retention time, the time taken for the solute dyestuff to pass through the chromatography column

KIK/IRPA code

colour of the dye, producing a range of colours from yellow, to red and violet, depending on the plant source [59]. Thus, HPLC results indicate that the bluish-purple floats of the background fabric of the embroidery were dyed with shellfish purple and the other elements with a dye including ellagitannins, since tannins have been widely used in dyeing silk. However, the ellagic acid detected could be a migration from the leather fragments, since tannins have also been used for the production of leather from skins and hides [59, 62, 63].

The coated lining of Y1 was also analysed with HPLC (Table 3). It contained a large amount of ellagic acid, meaning that it may have been dyed with ellagitannins or/and these tannins migrated from the leather. In addition, very small amounts of several compounds with maximum absorbance of 450/455 nm were detected, with, as the major one, a compound close to erythrolaccin at retention time of 20,1 min. These compounds refer to shellac resin, the black substance coating the fibres, which was likely used as an adhesive. Based on the stratigraphy of many Y1 fragments, it was assumed that the chequered pattern gold embroidery was executed on a

sheer fabric, lined with a thicker, support fabric and then adhered to the leather. According to this hypothesis, the ellagic acid detected in the fragment in a large amount had migrated from the leather to the fibres rather than being present in a dye source.

Three unknown compounds (named U1, U2 and U3) were detected at retention times of 9.3, 16.1 and 19.3 min with maximum absorbances of 390, 405 and 395, respectively (Table 3). Although not identified, the three compounds have previously been detected together in archaeological samples. However, it is not clear whether they derive from a biological dye source or are due to the archaeological burial environment.

### **XRF** analysis

The same samples analysed with FTIR-ATR were analysed with XRF. The main outcome of the XRF analysis was the detection of iron (Fe), which is in agreement with the FTIR-ATR results where a band assigned to iron(II) oxide was detected (Fig. 12, Table 1). In Y1 background fabric, 3.60% Fe was detected; in Y5 background weave, 21.57%; and in Y1 lining fabric, 5.269%. Since no iron



Fig. 12 XRF spectra and percentages of the elements detected on the textile samples analysed. Y1 embroidery background fabric is the blue spectrum; Y2 geometric tapestry background fabric is the black spectrum; Y1 support fabric with black coating is the red spectrum. © *Eirini-Chrysanthi Tsardaka, Stamatis Amanatiadis, Georgios Karagiannis, Ormylia Foundation-Art Diagnosis Center* 

was detected by EDS analysis in the metal threads, it was hypothesised that the iron detected in the textiles might point to the use of iron mordants for the procedure of dyeing of the textiles. HPLC analysis detected alizarin in Y5, indicating that the textile was dyed with madder. Different metallic salts have been used since ancient times as a pretreatment of textiles to be dyed as a means to create stronger bonds between the dye and fibre molecules. Stronger bonds can be formed due to the geometry of metallic salts that present one or more sites of chelation for the dye molecules [59]. Based on mentions in the Leiden and Stockholm papyri, iron mordants (mentioned as 'mordant drugs') seem to have been known in the Eastern Mediterranean as early as the beginning of the Common Era, and the iron mordants most commonly used were ferrous—iron(II)sulphate and iron acetate [59]. Iron (II) sulphate was commonly used in antiquity in the Mediterranean basin to darken and intensify the violet tones of dyes imitating true shellfish purple, and iron acetate to darken red dyes and to obtain dark purplish-reds and violet tones [59]. Iron mordants also improve the light-fastness of madder dyed textiles [60]. The detection of calcium (Ca) in the textiles was attributed to salt migration from other sources, such as the bones of the body, marble and/or soil of the tomb. Calcium (Ca) was detected at 14.63% in Y1 background fabric, 34.97% in Y5 background weave, and 29.93% in Y1 lining fabric. The detection of phosphorus (P) was also attributed to the bones of the body and was detected at 15.57% in Y1 background fabric, 19.56% in Y5 background weave, and 0.83% in Y1 lining fabric [16]. Gold (Au) at 22.31% was also detected at Y1 background fabric, which is the background fabric of the gold embroidery. The presence of aluminium (Al, at 1.77% in Y1 background fabric, 2.06% in Y5 background weave, and 1.56% in Y1 lining fabric) and silicon (Si, 4.98% in Y1 background fabric, 3.97% in Y5 background weave, and 10.31% in Y1 lining fabric) was attributed to soil contamination.

## **Radiocarbon dating**

The radiocarbon age of the textile sample was 1064 years with a standard uncertainty of 28 years. When calibrated in calendar years, the 95.4% probable result was





**Fig. 13:** <sup>14</sup>C calibration curves of **a** the textile and **b** the leather sample. © *Gabriela Sava, Tiberiu Sava, Horia Hulubei National Institute for R&D in Physics and Nuclear Engineering (IFIN-HH)* 

949–1028 (with 77.2% probability). The radiocarbon age of the leather sample was 1027 years with a standard uncertainty of 23 years. When calibrated in calendar years, the 95.4% probable result was 980–1040. <sup>14</sup>C results placed the find between AD mid-10th to early eleventh century (Table 4) (Fig. 13) [64]. This analysis confirmed the hypothesis that BXM 21359 is a Byzantine find, and in fact placed at the Macedonian era (862–1056), named after the Macedonian dynasty in rule at that time, the second longest dynasty in Byzantine

Table 4 <sup>14</sup> C dating results of the textile and leathe	r samples analys	ed
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Sample	Pre-treatment	C measured (mg)	Radiocarbon age	<sup>14</sup> C Results <sup>1</sup>
Textile	ABA	0.94 mg	RoAMS 1104.73 1064±28BP	95.4% probability 949AD (77.2%) 1028AD 895AD (18.2%) 925AD
Leather	ABA	0.97 mg	RoAMS 1104.73 1027±23BP	980AD (95.4%) 1040AD

<sup>1</sup> OxCal v4.4.4; IntCal20

History (Table 1) [65]. The Macedonian era, sometimes culturally referred to as the Macedonian renaissance, is considered a golden age of the Byzantine Empire as art, literature and culture flourished [66].

## Discussion of the results

The identification of silk fibres in the textiles would be expected of a find from this period. By the middle 10th c., precious textiles were directly associated with Byzantine culture and were considered elements of civilisation [67]. Silk was possibly the most characteristic feature of the most valued Byzantine fabrics and garments that were important economic assets frequently exploited for political reasons, diplomacy, and even in exchange for the provision of military and naval support to the Byzantines by their Italian, Russian or Bulgar neighbours [68-70]. The most elaborate and precious fabrics and garments were used to separate the church from the state and the state from the society [71], and functioned as social signifiers [72]. Imperial silk production factories, as well as flax (linen) and wool mills were well-established by the end of the sixth century. However, by the 10th c., workshops located in many urban centres of the Byzantine Empire, including Thessaloniki, would have used Chinese imported silk and locally produced material for the production of silk textiles [73, 74]. The identification of the satin weave however, is of greater interest. The invention of satin took place in China during the Song dynasty (960-1279) [75]. Gold patterned Chinese silks on satin weave background and satin damasks, preserved in church treasuries of Europe come from a later period, the Yuan dynasty period (1279-1368) [75]. It is not clear when the satin weave was first produced in Europe. According to the American Textile History Museum, the first western country to produce satin was Italy in the twelfth century, and it spread throughout Europe in the fourteenth century [76]. The earliest known silk-weaving workshops established in Italy were in Sicily (1130-54) and Palermo (1147), and Byzantine weavers captured in the Peloponnese (South Greece) served there. However, the earliest certified silk fabric produced in Palermo is a twill weave fabric [75]. Since the Y3 satin textile of find BXM 21359 is dated to 949-1028 with a 77.2% probability it could indicate ties and/or trade routes with China and/or the East.

The technique of twisting a gold strip around a fibrous core had already been used since the late Roman period and would have been executed by the use of a spindle rolled manually, or on the thigh [77]. Byzantium was famous for its woven textiles decorated with gold metal threads since the 6th c. [78]. Some of them were even woven entirely of gold (*holou chryshyphanta*) [71], or embroidered, gilded or appliquéd with gold [79]. According to Muthesius [71], surviving Byzantine couched embroidery, using gold thread, usually employs a silver gilt strip wound upon a twisted silk core. Gilt silver threads were already used before the 10th c. [80]. However, this is not the case with BXM 21359; in the majority of the metal threads of the find, EDS analysis detected only gold. In addition, two different types of gold threads were identified: A) a thread that consists of a thin strip of gold wound around an organic core (silk) [77] and B) a strip drawn-wire that forms a wire with a helical seam that is in practice a tube containing the organic core [38]. The second type of thread may refer to chrysosôlênokentêtos indicating a rare find of that period, and an owner of a particularly high status, similar to the blue silk royal tunic of Roger II of Sicily [81]. The high status of the owner is also supported by the presence of tapestries amongst the textiles. It is particularly time consuming to weave tapestry textiles, since even nowadays the technique can only be accomplished by hand, hence they are generally esteemed as symbols of wealth and high status [82]. The figurative embroidery identified would be expected in textiles dated to the Macedonian dynasty. During that period arts flourished specifically in embroideries, and figurative representations were established, which would be used for the decoration of ecclesiastical textiles during the 13th c. [83]. Therefore, the find BXM 21359 could have come from the burial of a member of the highest ranks of the clergy.

The identification of shellfish purple further supports the above. Shellfish purple was always a colour of great significance, associated with luxury and the highest ranks of the society. It is also known as 'royal purple', as it first appeared on a 13th c. BC Linear B tablet from Knossos, Crete; 'divine purple' and 'imperial purple', as it appears in 3rd c. written sources; 'adoration purple', since Diocletian instituted a relevant, mystical ritual performed at imperial audiences [59]. Restrictions were in effect in the trade and use of the textiles manufactured in the Imperial workshops, also controlling the supply of silk, purple dye and gold embroidery until 1204, when Constantinople was captured at the Fourth Crusade [84]. The production and consumption of silks dyed with shellfish purple, was by law an Imperial monopoly during the Macedonian era, as included in the Basilika (contemporary Law Code) [69]. In the *Book of the Prefect*, a list of regulations concerning collegia and private guilds of Byzantium, it is mentioned that higher ranks of the military were allowed three categories of superior purple dye, while the lower ranks cheaper purples; wealthy citizens were allowed to produce silk textiles at home for domestic consumption; and ordinary citizens were allowed to include scraps of purple silk decoration to their tunics [70, 72].

Shellac is a natural resin secreted by the Indian insect *Kerria lacca*, also found in Thailand, Myanmar and South China [85]. Apparently, the origins of the introduction of shellac in Europe are in the late 16th c., almost five centuries later than the date of the find [85–87]. Therefore, similarly to the satin weave, the presence of shellac in BXM 21359 could indicate ties and/or trade routes with China and/or the East.

## Conclusion

The application of instrumental analysis, combined with bibliographical research, was successful in placing the find BXM 21359 into context. Stereomicroscopy, SEM–EDS, FTIR-ATR, HPLC–DAD, XRF, ZooMS and <sup>14</sup>C, enabled material identification and dating of the textile and leather fragments. The assemblage BXM 21359 dated in the mid 10th c.-early 11th c. consists of a gold thread embroidered textile, a gold thread tapestry, and a gold-thread embroidered textile decorating a leather object/s, probably shoes.

Four (4) different kinds of tapestry were identified, the Y2, Y4 and Y6 textiles with geometric patterns, and the Y5 with figurative patterns. Physical evidence of connection was found between Y2-Y6 and Y4-Y5 fragments. All the tapestries have combined a gold thread decoration with a very fine, tabby silk background weave that at least for the Y4-Y5 fragments, was dyed with madder and probably had a red colour. The tapestry decoration was created by the use of two types of gold threads (shiny and matte) and one type of gold and silver alloy threads, while both the slit and toothed tapestry techniques were detected. Pieces of a braided cord and a knot were also identified, but no physical evidence to its connection with the textiles or the leather has been preserved. Similar finds of leather remains, presumably shoes, and braided cords, at the level of the breast of the body, have been found in a later burial, dated in the 14th c. during the excavation of a tomb at the church of Christ The Saviour [88]. An hypothesis would be that all tapestry fragments were originally part of one garment, that could be a vestment fastened across the chest with the braided cord. In that case, this 'vestment' could be connected to the first very fine purple textile with gold-woven decora*tion*, identified by Xygopoulos [40].

Three (3) embroidered textiles were also identified, Y1 with a chequered pattern, Y3 with medallions containing floral and cross motifs and Y7 with a figurative pattern. They are all embroideries with gold threads couched on the surface of the textile. Y1 embroidery was executed with gold threads couched on a very fine silk, tabby background tabby, that was stitched on a thicker tabby textile of a cellulosic bast fibre, which was in turn adhered to the leather with shellac resin. Y1 could be related to

the second, *thicker, with dense gold decoration forming crosses* textile, reported by Xygopoulos [40]. Y3 was executed on a silk satin background textile with floats dyed with shellfish purple, while the other set of threads were undyed or possibly dyed with ellagitannins. Y3 could be linked to the third, *possibly outerwear, very fine purple textile, embroidered with gold wire* described and drawn by Xygopoulos [40].

In the case of Y7 embroidery, the back side of the larger fragment was coated with a substance similar to the shellac adhesive found in Y1. However, no physical evidence exists to indicate textiles Y1 and Y7 were originally connected.

The few and in poor condition leather fragments cannot assist in drawing conclusions on whether they are indeed remains of shoes. However, the wavy edges with holes spaced in regular distance (every 4 mm) show signs of a stitched leather object that comes closer to a shoe rather than a book binding, a belt, a glove or else. If they were shoes, they would most probably have been pointed leather shoes decorated with gold embroidery, similar to shoes of the BCM collection (BXM 506, BXM 507) [89] and to later 15th c. finds from the church of Agia Sophia in Mystras, Greece [90].

By comparison to Xygopoulos' publication [40], the assemblage of BXM 21359 could be the one of the burial in tomb B, inside the church of Agia Sophia in Thessaloniki, excavated in 1924. The presence of silk and gold textiles in the tomb could be explained by the common practice of entombing the deceased in precious garments, layered on top of the customarily accepted pure white cloth [74]. On the other hand, the world events of the time period of the excavation in Thessaloniki could justify the transportation of the finds to Athens in order to be safeguarded. Transportation could also have taken place so that the find received a conservation treatment since BCM had already established a conservation laboratory and artists-conservators were already employed in its workforce. The customised, portable, wooden showcase, where the assemblage was kept, and the fact that no bones were stored in it, could indicate that it was mounted to be displayed at the exhibition of the BCM presented for the first time at the Athens Academy, in 1924 by Soteriou. The above hypotheses could be supported by the resemblance of one of the finds with the detailed drawing of Xygopoulos. However, until now, no trace in the BCM's conservation archive nor the exhibition catalogues or inventory records has been found.

In any case, instrumental analysis showed that BXM 21359 is an important Byzantine assemblage of textiles and leather of the mid-10th to mid-11th c.. Silk was the most characteristic feature of the precious textiles directly associated with the 10th c. Byzantine culture

[70], and the silk used for BXM 21359 could have been produced locally in Thessaloniki [74] or imported from China [73]. The identification of the satin weave further supports the hypothesis that at least textile Y3 was imported or somehow brought to Thessaloniki from China. In addition, the presence of shellac used for the construction of the leather objects could be indicative of a trade route between Thessaloniki and India or China [85-87]. Therefore, the provenance of BXM 21359 seems to have had ties with the East. The burial could have been of a person of particularly high status based on the fact that silk, gold and shellfish-purple dye, were used for the construction of their garments. A practice that by the 10th c., was not exclusive to the Emperors, but also used by the clergy or civilians of high rank or office [70]. The clergy were interred inside the nave while civilians were buried in the narthex [88]. The find published by Xygopoulos was found in the chancel of the church [40]. Most probably the assemblage BXM 21359 belonged to a person in the higher ranks of the clergy, and could originate from the burial in the church of Agia Sophia in Thessaloniki that was the burial area of Bishops from the 9th to the 13th c. [88, 91].

Textiles and textile related material are important carriers of past information due to the plethora of materials used for their construction (e.g. fibres, dyes, metals, adhesives). Archaeological textiles are generally made of organic materials, which are highly susceptible both to the processes of burial and the abrupt environmental condition changes occurring upon excavation. Therefore, the application of instrumental analysis, with an ethical approach ensuring the preservation of material for future study, analysis, and even display, is of utmost importance.

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#### Author contributions

CM and DF conceived the project; organised the analysis, studied the textiles and performed steremicroscopy and SEM–EDS analysis; GS performed 14C analysis and wrote the relevant part in the paper; IVB performed HPLC–PDA analysis and wrote the relevant part in the paper; CM and DF wrote the paper.

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#### Data availability

Raw data have not been uploaded in any repository.

#### Declarations

#### **Competing interests**

The authors declare no competing interests.

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