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Radiocarbon dating of archaeological textiles at different states of preservation

Christina Margariti[1*], Gabriela Sava[2], Tiberiu Sava[2], Mathieu Boudin[3] and Marie-Louise Nosch[4*]

Abstract

Archaeological textiles are suitable material for radiocarbon dating as they are mainly made of organic matter, such as plant and/or animal fibres. Radiocarbon dating provides objective age estimates of archaeological finds, based on measurements of the carbon-14 isotope present in the organic matter against an internationally used reference standard. However, the quantity and quality of carbon present in the organic matter of archaeological textiles can be affected either by the conditions under which the find was preserved (such as carbonisation, chemical change of organic matter to carbon, and mineralisation, which is the gradual replacement of organic matter by metal degradation products), or by the application of certain interventive methods of conservation (such as, consolidation and the application of organic adhesives that add foreign organic matter/carbon to the textile). Six case studies of archaeological textiles dated using the carbon-14 method are presented here. Two carbonised textile finds, two which were mineralised (one treated with adhesives in the past), and a final two which come from inhumation burials (similar to the textile previously treated with adhesives). This paper includes a discussion and review of the dating method, focusing on sample selection, sample preparation, and by evaluating the efficiency of the technique on textile finds at various states of preservation. This study shows that efficiency of the technique is highly dependable on the amount of carbon present in the finds, which does not seem to be affected by carbonisation, but is greatly affected by mineralisation and the incorporation of foreign organic matter, like synthetic consolidants.

Keywords Radiocarbon dating, Textiles, Mineralised, Carbonised, Adhesives, Consolidation

Introduction

^{14}C dating is based on measurements of residual ^{14}C within the organic material analysed. The purpose of this paper is to present and discuss how ^{14}C results might be affected by the condition of the textiles, when that condition has disturbed the C content of the material analysed or when provenance information does not exist to corroborate ^{14}C results. The C of excavated textiles might also be affected at some point after excavation and during conservation treatment as their condition is often so poor it requires the use of synthetic adhesives or consolidants to retain their structure [1].

Radiocarbon dating, also referred to as carbon dating or carbon-14 dating (^{14}C), is a method that can calculate the age of materials of plant or animal origin, by measuring the amount of the radioactive isotope of carbon (C), the ^{14}C isotope, present in the material. An isotope

*Correspondence:
Christina Margariti
chmargariti@culture.gr
Marie-Louise Nosch
nosch@hum.ku.dk
[1] Directorate of Conservation of Ancient and Modern Monuments, Hellenic Ministry of Culture, Pan. Tsaldari (Peiraios) Avenue 81, 10553 Athens, Greece
[2] Department of Nuclear Physics (DFN-TANDEM), Horia Hulubei National Institute for R&D in Physics and Nuclear Engineering (IFIN-HH), Strada Reactorului 30, Magurele, Romania
[3] Royal Institute for Cultural Heritage (KIK-IRPA), Parc du Cinquantenaire 1, B-1000 Brussels, Belgium
[4] Centre for Textile Research, SAXO Institute, University of Copenhagen, Karen Blixens Plads 8, 2300 Copenhagen, Denmark

is one of two or more species of atoms of a chemical element with the same atomic number but different atomic masses [2]. Each atom has a nucleus, containing protons and neutrons, and a number of electrons orbiting around it. Since the matter of electrons is extremely small, the mass of an atom is based on the matter within the nucleus, i.e. on the number of protons and neutrons present. Isotopes of an element have the same number of protons but different numbers of neutrons [3]. Carbon has three isotopes, they all have six protons, but each isotope has six (^{12}C), seven (^{13}C) or eight (^{14}C) neutrons. The heaviest carbon isotope, ^{14}C is constantly created in the Earth's atmosphere by neutrons which are secondary cosmic rays, subsequently concerted to carbon dioxide (CO_2), and thus incorporated into living organisms, like photosynthetic plants, and then to the rest of the biosphere [4]. Once simple plants (as opposed to woody trees) are cropped, animal hair is cut, and silk filaments are produced, replenishment of ^{14}C stops. Since ^{14}C decays at a constant rate, calculations can be made to estimate when replenishment stopped, by measuring the residual amount of ^{14}C remaining in the organic matter, thus enabling dating of an excavated find [4]. This date gives the time of harvesting the plant fibres or collecting the animal fibres or filaments. However, in textiles the fibers are spun and woven into cloth, which could be used for decades or generations before the textile was deposited in a grave or discarded. The ^{14}C date of textiles therefore reflects the time of production, but not necessarily the time of consumption or final use.

The radiocarbon dating method and textiles

The radiocarbon method was first developed for dating organic compounds in mid 1940s by the physical chemist Willard Frank Libby at the University of Chicago [5]. Radiocarbon (hereafter ^{14}C) dating has become an important dating method in cultural heritage science, especially for authentication and forgery detection. The wide-spread use of the method is mainly due to the ubiquity of the organic materials found in artwork, e.g. wood, canvas, leather, vegetal fibres, etc. The most critical issues in ^{14}C dating are the sampling process, the sample size and the presence of various contaminants. Therefore, for a reliable age determination, a large enough sample mass must be used in order to obtain sufficient amount of carbon, avoid unintentionally contamination, and remove possible preservative substances from the sample. At the end of the chemical pre-treatment the samples are converted to CO_2 before being graphitised and measured in an accelerator mass spectrometer (AMS) [6]. ^{14}C dating is a destructive method, meaning that the sample collected and used cannot be used for repeated or further analyses in the future, neither is it physically fit to be re-associated with the object [7]. Radiocarbon ages are converted to calendar age equivalents by calibration according to the current calibration curve (e.g.) [8].

Since archaeological and heritage textiles are generally made of organic material, they can be used for ^{14}C dating, and have been subjected to this analysis on multiple occasions (e.g.) [9]. However, there are many factors that may affect ^{14}C results, such as the condition of the textile, the sample pre-treatment and the interpretation of the results. One of the most well-known example of ^{14}C dating of a textile is that of the Turin shroud which radiocarbon dating placed in the Medieval period [10, 11], a result which is being disputed regarding the accuracy of the calendar age range, the representativity of the samples, and in comparison to alternative methods like pollen analysis (e.g.) [12, 13]. The potential of textiles as suitable ^{14}C samples has been disputed in the past, when in the 1950s, ^{14}C dating results of certain Coptic textiles in the Louvre collection, gave contradictory results to the stylistic dating of the finds. ^{14}C dating was revisited many years later to identify the mistakes in the past analysis and interpretation, but the results remained in conflict with the relevant dating [14]. Other cases showed better results: ^{14}C dating of stylistically dated textiles, a Tibetan painting and an Islamic kaftan, gave date estimations in support of the stylistic dates, thus indicating that textiles can be suitable objects for ^{14}C dating [15]. This hypothesis was further corroborated by the ^{14}C of numerous textile and skin samples from Danish bogs that actually redated many assumed Late Bronze Age finds to Early Iron Age, thus providing a new tool for the interpretation of textile and skin technology [16]. Importantly, ^{14}C dating of the textiles from mummy bundles of the Chancay Culture in Peru shed light on the chronology of the Chancay and Huaura valleys that even lacked pottery seriation [17]. Similarly, in the case of Pre-Columbian and Egyptian textiles that, more often than not, lack provenance information, ^{14}C dating enabled authenticating two pre-Columbian ponchos from the Quai Branly Museum (Paris) [18], proving that a mummy was kept in the wrong coffin at the Nicholson Museum (Sydney) [19], and shedding light on the incorrect association of two Coptic textiles of the Museum of Modern Greek Culture (Athens) [20]. In addition, ^{14}C dating placed two contemporary Iron Age textile finds from different areas in Greece (Lefkandi, Euboea and Stamna, Aitolia), more than 100 years earlier than relative dating, raising issues such as the use of heirloom textiles in certain burials and disputed conventional chronologies [21–23].

Sample pre-treatment is critical to the successful application of the analysis. For example, the Acid–Base–Acid (ABA) pre-treatment of textile samples that was developed to remove contaminants from the soil or humic acids in the case of excavated finds, was shown to be detrimental for poorly preserved textiles [24]. However, in the case of traditional silk and cotton textiles from the Ryukyu Islands of Japan, acetone-ABA pre-treatment gave dates consistent with the historical record, indicating that this method of sample treatment is suitable, at least for better preserved fibres [25]. Repeated analysis and advanced pre-treatment with hexane, acetone and ethanol solvents were necessary to date excavated textiles that had previously been treated with adhesives [20]. Consequently, ^{14}C can provide crucial and particularly interesting information to textile studies, and the more applications made on textiles, the more efficient and reliable the technique will become as it adapts and improves.

The condition of archaeological textiles

Archaeological textiles have undergone burial, which is a highly destructive process for their organic matter, as it is an ideal environment for the growth of the micro-organisms that feed off organic material [26]. There are, however, certain conditions that, when established, prevent microbial growth, enabling textile preservation. Four of these conditions are examined in this paper: 1. the presence of metal salts and gradual impregnation of the organic matter by them, created by degrading metal objects in their proximity (mineralisation) (e.g.) [27, 28]; 2. extremely low and constant moisture levels (desiccation) (e.g.) [29, 30]; 3. the establishment of a micro-climate characterised mainly by a low oxygen content, and usually found in sealed tombs in Medieval churches [30]; and 4. incomplete burning, which chemically alters the fibres to a degree that they are no longer an appropriate food source for micro-organisms (carbonisation) (e.g.) [31–33].

To serve our purpose, we have selected six case studies of archaeological textiles that had undergone burial, and were dated with the ^{14}C method. Namely, two mineralised textile finds, two from inhumation burials (one preserved in an extremely dry environment, the other in one with limited oxygen), and two carbonised textiles. As an additional factor, two textiles received past treatment with synthetic adhesives: one of the mineralised textiles and one of the inhumation burials (Table 1).

In mineralisation, factors like moisture content and water movement in the burial and the proximity of the textile to metal play a crucial role regarding the amount of metal salts deposited in the fibres, as well as when this process will stop, and consequently affect the degree of mineralisation [27]. Cellulosic fibres (such as the mineralised ones selected in this study) are polymers made up of cellulose chains (of C, oxygen (O) and hydrogen (H) atoms), lying parallel to each other in crystalline regions (more robust) or tangled in amorphous regions (more prone to deterioration) [34]. A recent study showed that mineralisation primarily deteriorates the amorphous regions of cellulose but also affects the crystalline regions at a macromolecular level and even causes loss of crystallinity, and the more the fibres are mineralised the less organic compound (i.e. the matter containing the C) is preserved [35]. In inhumation burials, regardless of the prevailing conditions, both cellulosic and proteinaceous fibres undergo natural ageing, which is mainly manifested as depolymerisation or chain scission of the cellulose or fibroin (in silk) chains, though not altering the organic nature of the material [34]. In carbonisation, the increased temperatures to which the organic matter is exposed transform the major constituents like cellulose, hemicellulose and lignin (in the case of plant fibres) into aromatic compounds or inert graphite-like carbon (e.g.) [31–33]. This means that C atoms in the cellulose or protein chains join other C atoms, thus altering both the inner structure of the C atom-based polymer chains and the elemental composition of the fibres. Two of our case studies had been treated with synthetic adhesives in the past. One was consolidated with polyvinyl acetate (PVA) (No. 2 in Table 1) [36], while the other was treated with PVA as an adhesive to secure the fabric support added to the object (No. 3 in Table 1) [37]. PVA is a thermoplastic resin of polymerised vinyl acetate that contains carbon in its monomer unit (e.g.) [38, 39]. In fact, the synthetic adhesive adds foreign C to the textile fibres. To sum up, in mineralisation, the amount of C to be measured for radiocarbon dating in textile fibres, decreases; in inhumation burials and carbonisation, it is altered; while in the case of the introduction of synthetic adhesives, it increases.

Methods

Case study 1

The Odos Thevon[1] textiles, retrieved in a 1983 rescue excavation by the Hellenic Ministry of Culture (Fig. 1a). The excavation had brought to light part of a Classical cemetery, thus the relative dating of the find is within this period (490-338 BC). These are mineralised textiles as they had been preserved in a copper alloy urn and were used to wrap the remaining bones of the deceased from the pyre burial that took place before the entombment of the urn. The textiles had been kept in storage since they were excavated, and past analyses had shown that the textiles were well preserved [27, 40]. Two samples from two different

[1] Most probably due to the rarity of textile finds In Greece, they are customarily referred to after the area they were retrieved. Recent research in the provenance of this find by C Margariti suggests it had mistakenly been referred to as the Nikaia find, in the past.

Table 1 The case studies of archaeological textiles dated with the radiocarbon method

No.	Reference name	Condition	Type of fibre	Relative dating	Pre-treatment	C measured (mg)	Radiocarbon age	^{14}C Results[a]
1	Odos Thevon [Nikaia]	Mineralised in storage	Cellulosic bast	Classical period Greece (490-338 BC)	ABA	1.1 mg	1. RICH-27497: 2427 ± 24BP	1. 95.4% probability 745BC (14.1%) 691BC 665BC (6.1%) 645BC 551BC (75.2%) 406BC
							2. RICH-27498: 2430 ± 24BP	2. 95.4% probability 746BC (15.9%) 689BC 666BC (6.7%) 644BC 564BC (72.8%) 407BC
2	Eleusis	Mineralised, adhesives, on permanent display	Cellulosic bast	Middle Classical period Greece (ca. 450 BC)	hexane–acetone-ethanol (Soxhlet) -ABA	1.1 mg	1. RICH-27499: 2445 ± 26BP	1. 95.4% probability 1505BC (95.1%) 1396BC 1332BC (0.4%) 1329BC
							2. RICH-27500: 2430 ± 26BP	2. 95.4% probability 431AD (93.2%) 607AD 625AD (2.2%) 636AD
3	MMGC Coptic tunic	Inhumation burial, desiccation, adhesives, on display (?)	Cellulosic bast	300-600 AD	RoAMS 569.73: ABA RoAMS 625.73: hexane–acetone-ethanol (Soxhlet) -ABA	1. 0.96 mg	1. RoAMS 569.73: 3169 ± 30BP	1. 95.4% probability 1505BC (95.1%) 1396BC 1332BC (0.4%) 1329BC
						2. 0.76 mg	2. RoAMS 625.73: 1527 ± 41BP	2. 95.4% probability 431AD (93.2%) 607AD 625AD (2.2%) 636AD
4	BXM excavated	Inhumation burial, desiccation, adhesives, on display (?)	1. silk	Unknown, Byzantine (?) (330-1453 AD)	ABA	1. 0.94 mg	1. RoAMS 1104.73 1064 ± 28BP	95.4% probability 949AD (77.2%) 1028AD 895AD (18.2%) 925AD 980AD (95.4%) 1040AD
			2. leather			2. 0.97 mg	2. RoAMS 1104.73 1027 ± 23BP	
5	Theva	Carbonised in storage	Cellulosic bast	Middle to Late Bronze Age Greece/ Mycenaean (?) (1750-1050 BC)	ABA	1.1 mg	RICH-27522: 3460 ± 27BP	1. 95.4% probability 745BC (14.1%) 691BC 665BC (6.1%) 645BC 551BC (75.2%) 406BC
								2. 95.4% probability 746BC (15.9%) 689BC 666BC (6.7%) 644BC 564BC (72.8%) 407BC
6	Amorgos	Carbonised from the excavation to the laboratory	1. cellulosic bast	Late Roman/ Byzantine, pottery seriation 600-800 AD	RoAMS 566.73: $CHCl_3$—ABA RoAMS 567.73: ABA	1. 0.95 mg	1. RoAMS 566.73: 1257 ± 28 BP	1. 95.4% probability 670AD (72.7%) 779AD 786AD (18.9%) 831AD 853AD (3.9%) 874AD
			2. wood			2. 0.95 mg	2. RoAMS 567.73: 1480 ± 28 BP	2. 95.4% probability 553AD (95.4%) 643AD

[a] OxCal v4.4.4; IntCal20

cellulosic bast fibre textiles were analysed (of the three textiles identified in the find). The samples were pretreated with ABA. ^{14}C dating determinations were measured using the Accelerator Mass Spectrometer (^{14}C-AMS) at KIK-IRPA. Graphitisation for the dating process was performed using the automated graphitisation system AGE III, and ^{14}C calibrations were performed using the OxCal version 4.4.4 and the IntCal20 calibration curve date [8, 41–45].

Case study 2

The Eleusis textiles retrieved in 1953 from a Classical period burial at the cemetery of Eleusis during a systematic excavation conducted by the Hellenic Archaeological Society (Fig. 1b). They had similarly been used to wrap the remaining bones from a pyre burial, and subsequently placed inside a copper alloy urn. The urn, in turn, had been placed in a stone sarcophagus along with five painted ceramic funerary vessels,

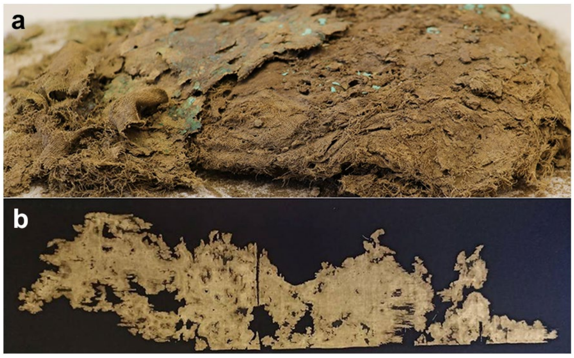

Fig. 1 Case study 1. Detail of the Odos Thevon find, maximum dimensions 240 × 160 × 40 mm **a**, and case study 2 the Eleusis find, maximum dimensions 2.2 × 0.5 m **b** (©Christina Margariti—Tina Chanialaki, Directorate of Conservation of Ancient and Modern Monuments, Hellenic Ministryof Culture)

Fig. 2 Case study 3. Late Antiquity child's tunic **a** (©Andreas Santrouzanos and Directorate of Conservation of Ancient and Modern Monuments, Hellenic Ministry of Culture), and case study 4, detail of the Byzantine & Christian Museum find **b** (©Nikos Mylonas, Byzantine & Christian Museum, Hellenic Ministry of Culture)

known as *lekythoi*, clearly situating the find in the Classical period as far as relative dating was concerned [46]. The two textiles present in the find had been consolidated in the mid-1950s with a 5% v/v solution of PVA in toluene [36] and were on permanent display at the Archaeological Museum of Eleusis, exposed to daylight for 64 years. One sample of each linen textile was analysed with the same method as in case study 1. However, in case study 2, the samples were pre-treated with hexane–acetone-ethanol (Soxhlet)—ABA, to remove the adhesive residues.

Case study 3

The Late Antiquity (also called *Coptic*) tunic from Egypt, from the Museum of Modern Greek Culture (MMGC) (Fig. 2a). Like other similar textiles, this object lacked information regarding provenance, while relative dating based on the style of the decoration (as widely done for dating *Coptic* textiles) placed it between 300 and 600 AD. It was obvious that it came from an inhumation burial from an area with extremely low humidity levels that enabled the preservation of the find (based on humic acid staining, very good preservation and desiccation/brittleness of the fibres). The condition of the fibres was estimated as good, as usually is the case with textiles preserved by desiccation in arid conditions that largely prevent microbial growth (e.g.) [47]. However, the tunic had been treated with soluble nylon(?) or polyvinyl acetate(?) adhesive to secure its full support with a modern fabric at some point in the mid 1960s [37]. ^{14}C analysis had to be conducted twice, since the first results indicated severe contamination by the adhesive. Samples were collected from the main linen fabric of the tunic (as opposed to the wool threads used for the decorations). The first sample (RoAMS 569.73) was pre-treated with ABA, while the second (RoAMS 625.73) with a sequence of hexane–acetone-ethanol (Soxhlet)—ABA to remove the adhesive residues. ^{14}C dating determinations were measured on the AMS (^{14}C-AMS) at IFIN-HH. Graphitisation was performed with the CHNOS Elemental Analyser/AGE III system and pressed into the AMS cathodes [42, 48]. The samples were measured in the AMS spectrometer MICADAS, normalising them to the Oxalic Acid II [48] age standard, and subtracting the ^{14}C blank levels using an unknown deep geological deposit coal. This blank was tested and compared with Merk® synthetic graphite (Carbon graphite powder, 99.9% purity). The raw data was reduced to radiocarbon ages [49] and further calibrated, in the same way as in case study 1.

Case study 4

The textile find from the Byzantine and Christian Museum (BXM) (Fig. 2b). This object was located in the museum storage with no accession number or any other information, and, to the authors' knowledge, it had never been on display or treated with foreign material. The fact that it is decorated with pure gold threads and was kept at the specific museum indicates that its relative dating would be in the Byzantine period (330-1453 AD). It most probably came from an inhumation burial, while its elaborate decoration and preservation suggest that it could have come from a sealed tomb of a prominent ecclesiastical figure.[2] Preservation of textiles in inhumation burials generally indicates that the burial space had remained largely undisturbed, hence the limited supply of oxygen and amount of soil [50]. One proteinaceous fibre sample and one leather sample (also present in the find) were analysed, both pre-treated with ABA. ^{14}C dating determinations and calibrations, and graphitisation were performed in the same manner as in case study 3.

[2] Unpublished study of 2017 of the archaeologist in charge, Maria Koutsoumpou, Ephorate of Antiquities of Cyclades, Hellenic Ministry of Culture.

Case study 5

The Theva textiles retrieved in a 1985 systematic excavation of the Hellenic Ministry of Culture in Kadmeia, the ancient citadel of Theva (Thebes), in Boeotia (Fig. 3a). The excavation revealed finds of the Byzantine, Classical and Mycenaean period. The Theva textile, a carbonised textile made of linen fibres, was found at a disturbed stratum of the Mycenaean period (13th c. BC), under a Classical period stratum [51]. The textiles had never been on display or treated with foreign material. One sample was analysed and pre-treated with ABA. ^{14}C dating determinations and calibrations, and graphitisation were performed the same as in case studies 1 and 2.

Case study 6

The Amorgos textile find [52]. This object was retrieved from a rescue excavation of the Hellenic Ministry of Culture on the Aegean island of Amorgos, it was block lifted from the excavation and brought to the conservation laboratory (Fig. 3b). The block of soil removed from the excavation contained carbonised textiles, ropes, cordage and wood, as well as stone and pottery fragments of various sizes. Pottery seriation placed it between the sixth and eighth centuries CE (see Footnote 2). One sample of cellulosic textile and one sample of wood were analysed. Since the textile sample was very fragile due to carbonisation, it was cleaned with a chloroform solution [53], and the wood sample with ABA. ^{14}C dating determinations, calibrations and graphitisation were performed in the same way as case studies 3 and 4.

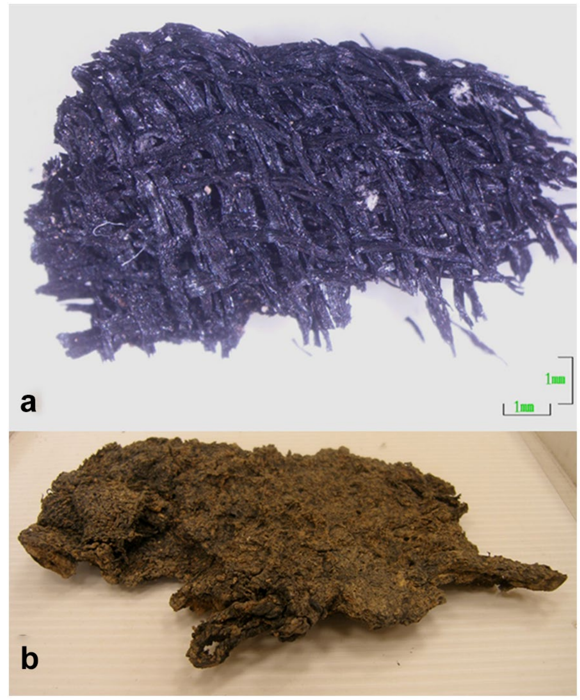

Fig. 3 Case study 5. Detail of the Theva find **a**, and case study 6 detail of the Amorgos find **b**, maximum dimensions 180 × 120 × 20 mm (©Stella Spantidaki—Christina Margariti, and Directorate of Conservation of Ancient and Modern Monuments, Hellenic Ministry of Culture)

Results and discussion

The mineralised textiles, case studies 1 and 2

The relative dating of the two case studies of mineralised textiles was straightforward due to the excavation contexts, and placed both in the Classical period of Greece (490-338 BC). For case study 1, Odos Thevon, the radiocarbon age was 2427 years for one of the textiles and 2430 years for the second, with a standard uncertainty of 24 years for both. When radiocarbon age was calibrated in calendar years, the 95.4% probable result for textile 1 was 551-406 BC (with 75.2% probability), and for textile 2, 564-407 BC (with 72.8% probability) (Table 1) (Fig. 4). The overall condition of this find was very good (considering it was an excavated textile), as the fibres still retained some of their original physical and chemical properties, i.e. they were elastic and hygroscopic [27]. In general, the probable calendar dates were earlier than what was expected from relative dating. However, the latest value of the highest probable calendar date was 407-406 BC for the two textiles, which is right in the middle of the Classical period. Therefore, ^{14}C dating correctly placed Odos Thevon in the relative dating of the Classical period, and likely reduced the age range to the early to middle Classical period. Nonetheless, the gap in the highest probable calendar dates becomes more perplexing when interpreting the results. A possible reason is that the dates of interest for this case study fall within the Hallstatt radiocarbon calibration plateau (ca. 750-400 cal BC), which is a mostly flat area on the graphs that plot ^{14}C dating against calendar dates, apart from a "spike" at ca. 660 BC [41]. Measurements always calibrate to these dates, regardless of their precision [54].

For case study 2, Eleusis, the radiocarbon age was 2445 years for the first textile and 2430 years for the second, with a standard uncertainty of 26 years for both. When radiocarbon age was calibrated in calendar years, the 95.4% probable result for textile 1 was: 591-411 BC (with 59% probability), and for textile 2: 568-406 BC (with 71.5% probability) (Table 1) (Fig. 5). Similar to case study 1, the latest value of the highest probability calendar date was 411 BC for textile 1 and 406 BC for textile 2, again right in the middle of the Classical period. However, the probability percentage of these calendar dates was lower than in the Odos Thevon textiles, and especially for the first Eleusis textile sample, for which the probability was below 60%. In this case study the relative

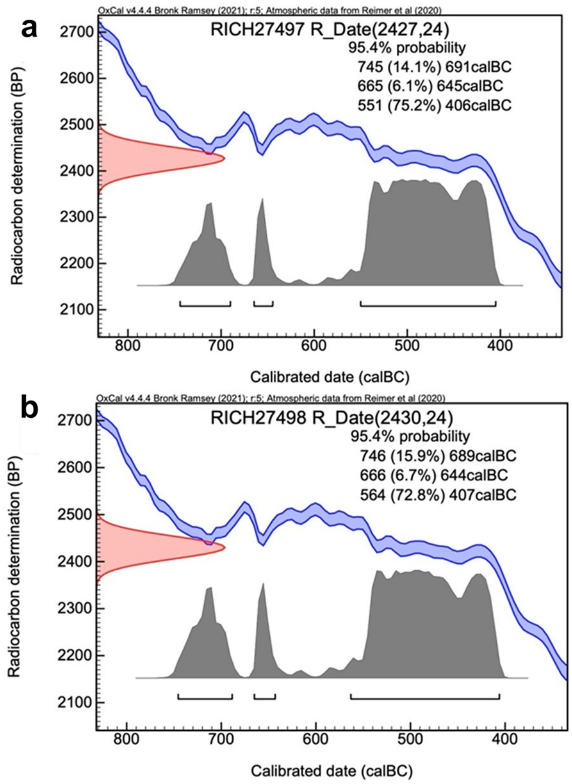

Fig. 4 Case study 1. Calibration curves of the Odos Thevon textile 1 **a** and textile 2 **b** samples (by Mathieu Boudin, KIK-IRPA)

dating of the find was more precise, locating it in the mid-fifth century BC (ca. 450 BC) due to the presence of the *lekythoi* in the sarcophagus. Based on that and on the Hallstatt plateau, ^{14}C dating correctly placed the Eleusis textiles in the early to middle Classical period. Nonetheless, the gap in the highest probability calendar dates was even larger than in the previous case study, and the probability of the closest to the relative calendar date was particularly low in one of the textiles. The overall condition of this find was medium, as the fibres were hydrophobic and brittle, but retained some elasticity when conserved in 2019. The two Eleusis textiles were found folded, and they were elastic and flexible enough to be unfolded during conservation in the 1950s, although even then it was noted that the fibres' capacity to absorb water was strikingly low [36]. It seems that the higher the degree of mineralisation, the less hygroscopic and more hydrophobic the fibres become, as a result of the organic-hygroscopic matter being replaced by the inorganic-metal salts compounds [35, 55]. Therefore, it could be assumed that the degree of mineralisation of the Eleusis textiles was higher than Odos Thevon. The Eleusis find remained on permanent display for more than six decades, exposed to sunlight. Sunlight has increased amounts of UV radiation that make it one of the main agents of decay (i.e. photodegradation for the fibres, an autocatalytic chain reaction process, and cross-linking) of organic compounds, like the textile fibres and the synthetic adhesive (e.g.) [34, 39]. Regardless of how aggressive it may be, this type of decay does not seem to interfere with the residual amount of C, as it mainly manifests in bond cleavage and chain scissions in the polymer chains, and the creation of covalent cross-links between the cellulose in the fibres and the polymer chains of the synthetic adhesives (that were present in this textile) (e.g.) [34, 39]. The higher degree of mineralisation (and the Hallstatt plateau) are possible explanations for the larger age gap, and the lower probability than the previous case study. Further experiments on radiocarbon dating of mineralised textiles at different degrees of mineralisation would be necessary to confirm or refute this preliminary observation. In addition, the presence of the polyvinyl acetate adhesive interferes with the amount of C in the sample, and has proven almost impossible to be completely removed during pre-treatment, thus making it most probably responsible for the weaker results of this case study [56, 57]. As a general remark for both case studies, the probable calendar dates were earlier than what was expected from the relative dating.

The textiles from the inhumation burials, case studies 3 and 4

Case study 3, the MMGC *Coptic* tunic, was lacking provenance information but was stylistically attributed to Egypt and Late Antiquity, 300-600 AD [58]. Therefore, the first analysis result that indicated the radiocarbon age at 3169 years with a standard uncertainty of 30 years, calibrated in calendar years with 95.1% probability to 1505-1369 BC, was rejected (Fig. 6). The tunic had been treated with a synthetic resin similar to the one used in the Eleusis textiles. What was very different in this case study was the fact that the resin was employed as an adhesive rather than a consolidant, i.e. a much higher percentage than 5% would have been used. Unfortunately, the exact percentage is not known since there is no written documentation of the adhesive treatment. In addition, the consolidant was lightly sprayed on the Eleusis textiles [36], whereas the adhesive would have been reactivated by heat [37]. As a result, the fibres of the tunic would have absorbed a much higher amount of synthetic resin than the Eleusis case. Case studies 2 and 4 were both treated with petrogenic adhesives, i.e. products deriving from petroleum. Similar experiments on radiocarbon dating of treated objects have shown that the higher the amount of the

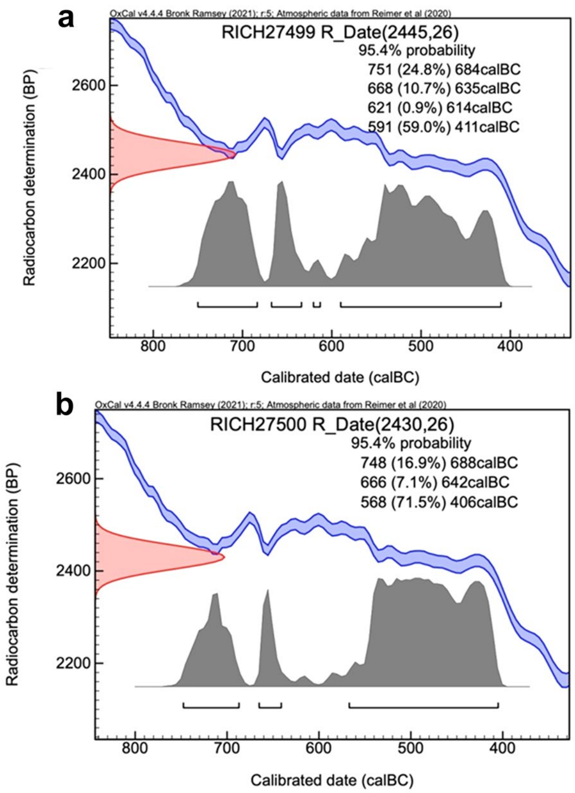

Fig. 5 Case study 2. Calibration curves of the Eleusis textile 1 **a** and textile 2 **b** samples (by Mathieu Boudin, KIK-IRPA)

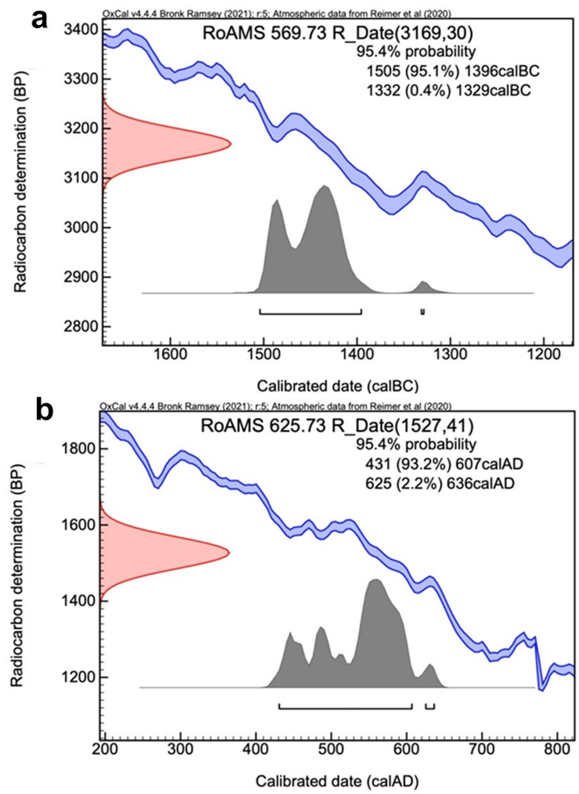

Fig. 6 Case study 3. Calibration curves of the late Antiquity child's tunic first **a** and second **b** analysis (by Gabriela and Tiberiu Sava, IFIN-HH)

adhesive, the earlier the radiocarbon dating results [56]. Furthermore, the second sample was cleansed with hexane–acetone–ethanol (Soxhlet) followed by ABA before analysis to remove as much adhesive contamination as possible [59–61]. Tests conducted on the efficiency of pre-treatment protocols for the removal of polyvinyl acetate adhesives showed that none of the protocols were able to remove this type of adhesive completely [57]. However, for the second sample, the radiocarbon age was 1527 years with a standard uncertainty of 41 years. When the radiocarbon age was calibrated in calendar years, the result for the 95.4% probable age was 431-607 AD (with a 93.2% probability) (Table 1) (Fig. 6). The relative dating of the find was based on its in-woven tapestry decoration, which is attributed to Late Antiquity tunics between the fourth and sixth centuries AD [37, 58]. ^{14}C dating was able to lower the age gap by half, placing the find in the fifth century AD. The good condition of the textile fibres in this case study (due to the preservation in arid conditions) most likely explain the high probability (93.2%—in fact the highest of all textiles tested here) of the calibrated results. As an overall remark, the probable calendar dates were much earlier than those expected from relative dating in the first sample, due to the adhesive contamination, and the fact that textile preservation in arid conditions seems to affect radiocarbon dating less than mineralisation and carbonisation.

Case study 4, the BXM excavated textile find, completely lacked provenance information. Based on certain technological characteristics and decorative elements, the hypothesis was that it was a Medieval—Byzantine for Greece—find from a tomb burial (330-1453 AD).[3] Regrettably, no information on past display or conservation treatment was found. ^{14}C dating results indicated the radiocarbon age at 1064 years with a standard uncertainty of 28 years. When the radiocarbon age was calibrated in calendar years, the 95.4% probable result was 949-1028 AD (with a 77.2% probability). ^{14}C dating results for the leather indicated that the radiocarbon age was 1027 years with a standard uncertainty of 23 years. When radiocarbon age was calibrated in calendar years, the 95.4% probable result was 980-1040 AD, strengthening the likelihood of a date from mid-tenth to early eleventh century AD (Table 1 and Fig. 7). ^{14}C dating was thus

[3] The analyses and publication of this find are at the time of writing still in progress.

able to corroborate the hypothesis that this was a Byzantine find, and, in fact, placed it within a very narrow period, the Macedonian era (862-1056 AD), named after the Macedonian dynasty ruling at that time, the second longest dynasty in Byzantine History [62]. The Macedonian era, sometimes culturally referred to as the Macedonian renaissance, is considered the golden age of the Byzantine Empire as art, literature and culture flourished [63, 64]. Therefore, the more precise ^{14}C date of this case study contributes to the understanding of the technology in the construction and decoration of this find. As this case shows, generally speaking, the process of inhumation burial did not seem to greatly affect ^{14}C results. In addition, the leather sample gave results of higher probability than the textile (although both were of proteinaceous nature). The more robust structure of the leather compared to the very fine silk textile, was most probably responsible for a better preservation of the former.

The carbonised textiles, case studies 5 and 6

For case study 5, Theva, the radiocarbon age was 3460 years with a standard uncertainty of 27 years. When radiocarbon age was calibrated in calendar years the 95.4% probable result was: 1881-1732 BC (with a 81.6% probability) and 1732-1690 (with a 13.9% probability) (Table 1) (Fig. 8). The overall condition of this find was estimated as medium, since there was enough material left, and it had been kept in storage, but it was uncertain whether it had received any consolidation treatment, as no relative written documentation was found. Due to its fragile nature, the find was block-lifted from the excavation site [65]. In past years, at least in Greece, consolidation of sensitive organic finds before block-lifting was common practice. It is therefore possible that the Theva textiles were consolidated with a polyvinyl acetate resin, like Mowilith®50. However, FTIR spectra of the textile (collected in reflectance mode) [66] did not show any of the bands related to the -C=O stretching vibration associated to acetate groups (1729, 1433, 1370 cm-1, collected by ATR-FTIR) [67, 68]. In general, the probable calendar dates for Theva were earlier than what was expected from the relative dating. However, the latest value of the highest 95.4% probability was 1732 BC, which lies at the earliest stage of the Mycenaean period (c. 1700–1100 BC). Judging from the results in case studies 2 and 3, with adhesives, the earlier probable calendar dates could be attributed to past treatment with a synthetic resin [56]. Nonetheless, ^{14}C dating was able to place the find in the Late Bronze Age for Greece (Mycenaean) rather than in the Classical period, which was the main archaeological concern, as the stratum where it was found had been disturbed.

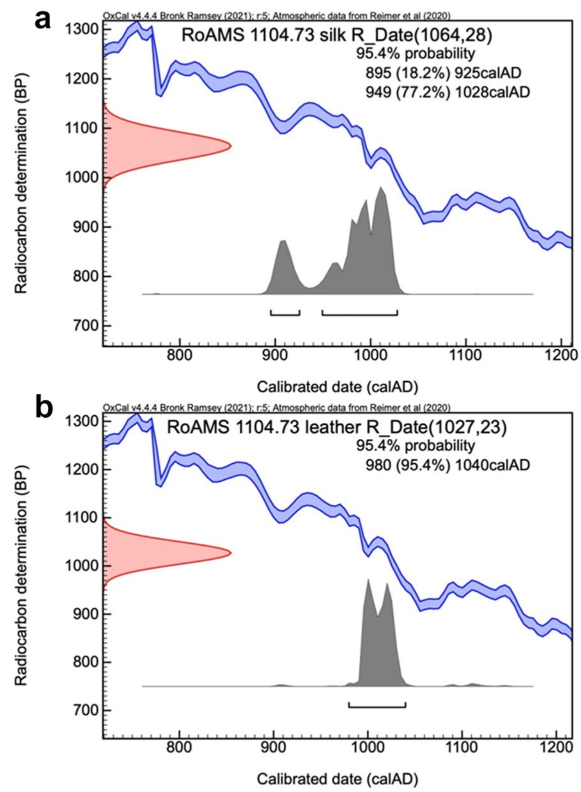

Fig. 7 Case study 4. Calibration curves of the textile **a** and leather **b** samples from the Byzantine & Christian Museum find (by Gabriela and Tiberiu Sava, IFIN-HH)

For case study 6, Amorgos, the radiocarbon age of the textile sample was 1257 years with a standard uncertainty of 28 years. When the radiocarbon age was calibrated in calendar years, the 95.4% probable result was 670-779 AD (with a 72.7% probability). The radiocarbon age of the wood sample was 1480 years with a standard uncertainty of 28 years. When radiocarbon age was calibrated in calendar years, the 95.4% probable result was 553-643 AD (Table 1) (Fig. 9). The overall condition of this find was considered good, since there was a large quantity of material left, it had been transferred from the excavation site straight to a conservation laboratory, and it had not received any treatment with synthetic resins. Based on all associated finds retrieved (like ropes and certain stone fragments), it was suggested that the textiles were sails and the wood fragments were parts of the masts and boat haul [52]. The earlier highest probability calendar date for the textile and the later date for the wood were calibrated to the seventh century AD, with a possible interpretation that this is the date of the find. However, pottery seriation indicated they belonged to both the seventh and eighth

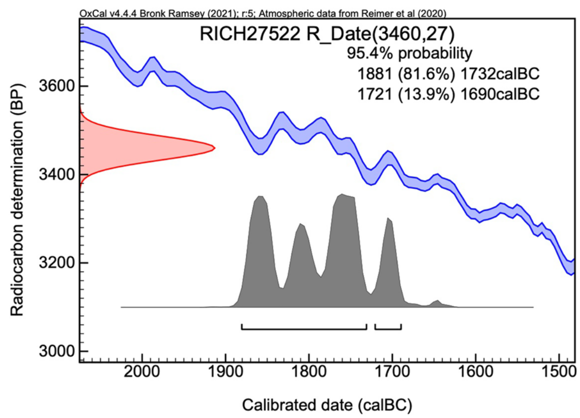

Fig. 8 Case study 5. Calibration curve of the Theva sample (by Mathieu Boudin, KIK-IRPA)

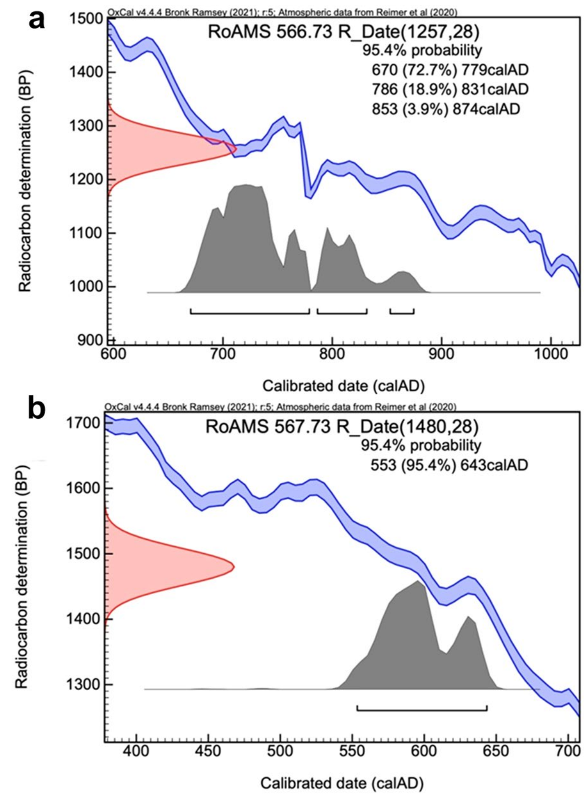

Fig. 9 Case study 6. Calibration curves of the textile **a** and wood **b** samples from the Amorgos find (by Gabriela and Tiberiu Sava, IFIN-HH)

centuries AD (600-800 AD). Therefore, another possible interpretation could be that the wooden parts of the boat were older, while the textile sails had, at some point, been replaced with new ones. A general remark for these case studies is that carbonisation of organic finds does not seem to greatly affect radiocarbon dating. Similar to case study 4, the wood sample (again, a more robust material) gave better results.

Conclusions

^{14}C dating can be a particularly useful tool for textile studies, since it has shown to corroborate relative dating, as in case studies 1 and 2; to reduce large age gaps in relative dating, as in case study 3; to put into context material science and technological information, as in case study 4; to shed light on ambiguous archaeological information, as in case study 5; and to bring together formerly unconnected archaeological information, as in case study 6.

Nonetheless, its performance and results are dependent on the state of preservation of the textiles analysed, and in certain cases, they can affect the dating in major ways. In the case of mineralised textiles, the degree of mineralisation is crucial to the ^{14}C dating results, and, as case studies 1 and 2 indicated, the higher the degree of mineralisation, the lower the reliability of the results. A hindrance of paramount importance to ^{14}C is the presence of synthetic resins in the textiles, as shown in case studies 3 and 4. Based on the performance of those finds, the higher the amount of resin in the fibres, the higher the probability of unsuccessful analysis. Additionally, the results contaminated from a petrogenic resin seem to give much earlier calibrated dates than expected [59]. In general, the effects of inhumation burial and carbonisation do not seem to interfere with ^{14}C dating results. However, radiocarbon dating results on textiles might be of lower quality compared to those achieved on more robust organic finds (like leather and wood), which are more likely to be better preserved in the same context.

Radiocarbon dating is a destructive and costly technique, hence the samples tested will be completely consumed by the analysis, and it generally requires a substantial budget [69]. It is also important to remember that the date of the textile tested (or for that matter, the date of any organic find dated by radiocarbon analysis) is not necessarily the date of the site it originated from [70].

Appropriate sampling and pre-treatment are essential to the successful outcome of the analysis. When samples are removed from textiles in very poor states of preservation (e.g. heavily mineralised) the amount of the sample removed should probably be larger than a sample taken from a non-mineralised textile, to ensure enough C is left after pre-treatment to enable reliable measurements. In addition, when it is considered necessary to treat a textile with resins, samples for ^{14}C dating should be collected before the treatment. Certain types of resins cannot be completely removed even when following advanced cleaning protocols [57]. Furthermore, although conservators traditionally have used reversible materials, the poor state of preservation of the artefacts often means that the material cannot withstand the reversibility process. In principle, the textiles in the worst condition are generally consolidated and/or treated with adhesives (e.g.) [71]. The conservation process offers a unique sampling opportunity, given that throughout the procedure edges, back sides, as well as areas with significant loss or damage (areas from which it is ethically appropriate to remove samples) become fully accessible. Also, the necessary handling during a conservation treatment unavoidably disassociates small samples from objects that conservators should carefully collect, store and label for future analysis. Proper labelling and storage of the collected samples is compelling to ensure the research questions will be correctly answered by the analysis. Good collaboration and close contact between archaeologists, conservators and scientists ensures the most successful results, as similarly pointed out in previous relevant publications (e.g.) [15].

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Author contributions
CM and MLN conceived the project; CM conceived the paper outline, organised the analysis, conserved the finds in collaboration with colleagues; GS, TS, MB performed the analyses; CM, GS, TS, MB, MLN wrote the paper. All authors read and approved the final manuscript.

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Declarations

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