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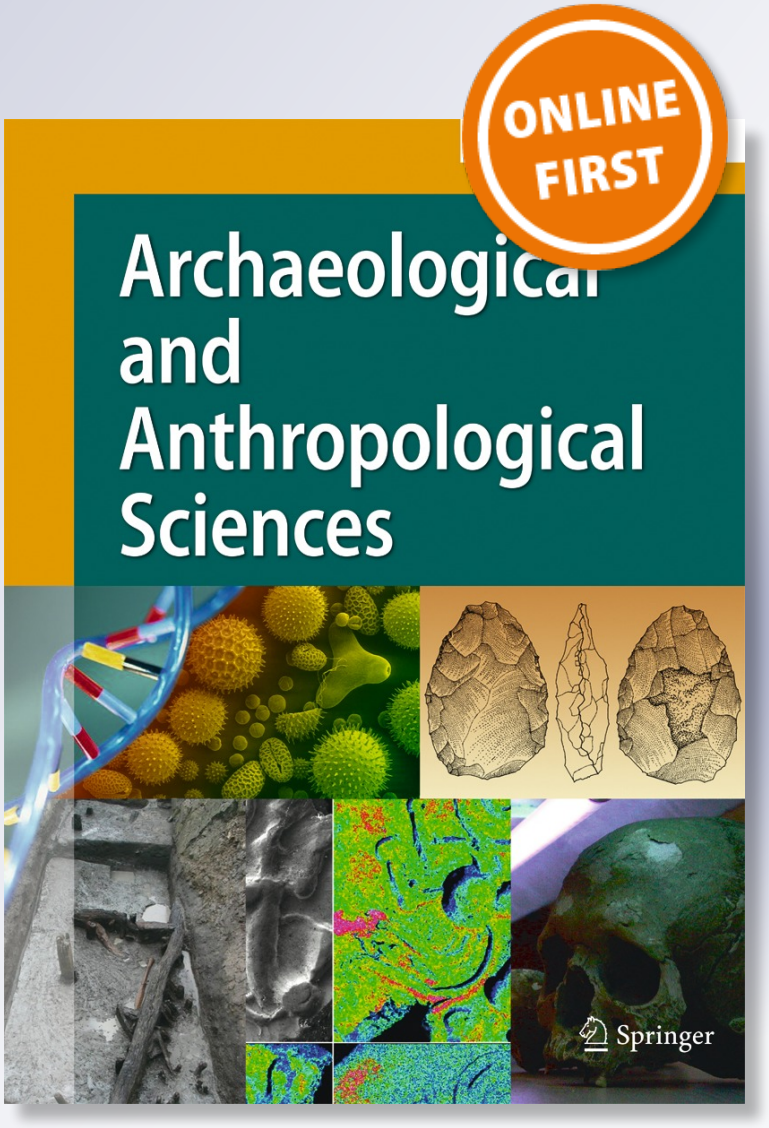

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ORIGINAL PAPER

Genuine or forged? Assessing the authenticity of a confiscated manuscript using radiocarbon dating and archaeometric techniques

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Abstract The authenticity of a confiscated parchment manuscript written in medieval Hebrew was examined using radiocarbon and archaeometric techniques. The owners of the manuscript claimed that it is old and valuable. Transmitted light showed folios of uniform thickness and opacity, while examination under ultraviolet light displayed the absence of conservation treatments. X-ray fluorescence showed the ink used was iron gall ink. On these grounds, the manuscript could be dated to the Middle Ages. However, the precision and homogeneity of the hand writing, sewing, dimensions, and margins suggest that it is a much more recent artifact. Post-bomb radiocarbon dates for the folios and threads clearly demonstrate the recent vintage of the manuscript. Biological analysis suggests that the manuscript was buried in a soil intentionally amended with animal wastes to achieve rapid aging and deterioration features.

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Keywords Confiscated manuscript · Deterioration features · Archaeometric analyses · Radiocarbon dating · Jordan

Introduction

Most manuscripts spanning the fifth to the fifteenth centuries AD were written on parchment (Berger et al. 1972); therefore, the physical and chemical analysis of parchment is a valuable tool for archeological, historical, and art records. In addition, physical manuscripts provide information about the techniques used in their manufacture. The chemical and physical characteristics of manuscripts and inks can be used to address questions regarding their origin and dating, and thus distinguishing authentic artifacts from forged or faked ones. Copies could be forgeries (new objects imitating something else) or fakes (altered objects that appear to be something else) (Craddock 2009). Forgeries and fakes have a long history, and the market of forged and faked antiquities has significantly increased over the past decades (Luke and Kersel 2005). Technological developments during the past few decades have advanced not only forgery skills but also the methods used for forgery detection (Bunker 1994).

Forgers work hard to reproduce ancient materials, technologies, and the appearance of the appropriate archeological period; it has become difficult to distinguish an original work from faked or forged ones using simple visual inspection (Holtorf and Schadla-Hall 1999). Before the Second World War, art historians and archeologists primarily used form, design, and iconography to identify forgeries. In the last 60 years, much work has been devoted to develop methods to determine the genuineness and correct age of an archeological artifact or artwork (Humphreys 2002; Brock 2013). These methods often focus on the dating and physical and chemical analytical techniques to determine the artifact's composition, production

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technology, and age (see Hahn 2010 for more details). This approach is also crucial for restoration or conservation work (Craddock 2009). In addition, when a forgery is detected, analytical techniques can help understand the artificial aging processes that the material was subjected to.

This study uses radiocarbon dating and various archaeometric techniques to investigate the authenticity of a confiscated parchment manuscript brought to the Archaeometry Laboratory at the Faculty of Archaeology and Anthropology at Yarmouk University, Jordan.

The manuscript

The manuscript is a hand writing resembling medieval Hebrew script but without meaning. It is similar to some fragments of the Cairo Geniza. Some isolated words can be read, but not much more. It could therefore be a magical text, abbreviations of biblical phrases, or just Nomina Barbara (Misgav, personal communication). A candlestick in the middle of a hexagonal star is drawn on the leather case enclosing the manuscript (Fig. 1a). The manuscript is divided into two booklets of 14 and 20 folios stitched by leather thread (Fig. 1a, b and c). Each folio is 23 cm long and 17 cm wide. These dimensions are different from, but within, the present international paper sizes A4 (29.7 × 21.0 cm) and A5 (21.0 × 14.8 cm). The text written on each folio is about 16 cm long and 11 cm wide. The first page of the manuscript has a drawing of a candlestick surrounded by text. The text is carefully written in two columns per page, 15 lines per column in the form of a poem (Fig. 1b). The margins of each page measures 3.5 cm on the top and bottom and 2.5 cm on the right and left. The ink colors vary between black and brown.

The manuscript folios are of thin and lightweight leather. They are fragile, showing differential aging and deterioration features, including white surface blooming, cracks, twists, bores, missing parts, dark spots, colored stains, and fading ink (Fig. 1a, b). Unfortunately, the exact context of the manuscript is unknown because it was confiscated from treasure hunters. Those in possession of the manuscript, however, claimed that it is old and valuable.

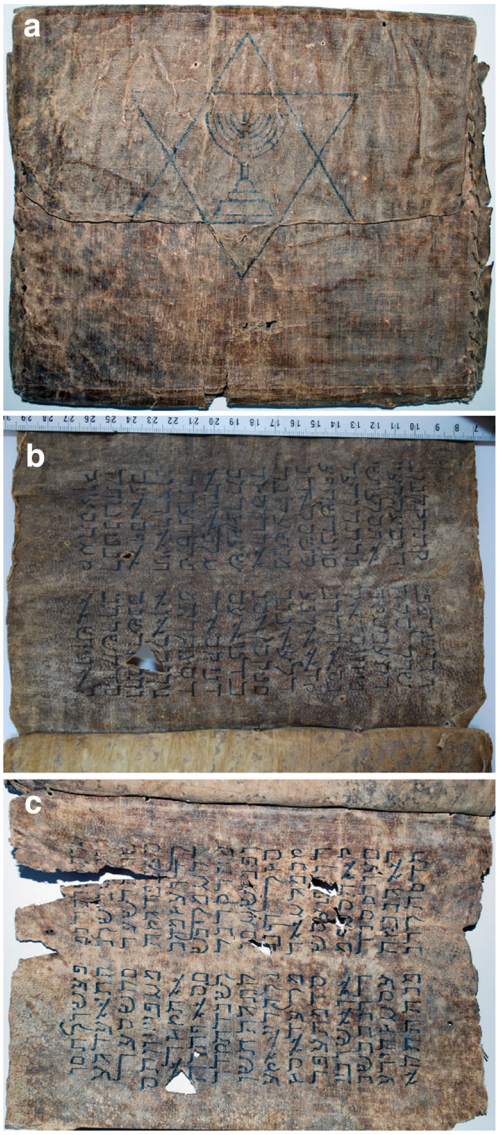

Fig. 1 **a** The case of the manuscript showing the candlestick in a hexagonal star and deterioration features (color fading, blooming, missing parts, dark spots), **b** shows dimensions of one of the manuscript folios, text lines and columns, **c** shows some deterioration features (missing parts, pores, blooming, …)

Analytical methods

The analytical techniques applied in this study are as follows:

Ultra Violet (UV) analysis: UV irradiation often reveals recent conservation treatments because new conservation materials will often make the leather surface fluoresce. The folios were irradiated using UV light (N-4K, Volt: 220, 254 nm) in a dark room.

Transmitted light analysis: transmitted light was used to reveal production marks, underwritings, and the homogeneity of folio thickness.

X-ray fluorescence (XRF) analysis: standardless semi-quantitative analysis of the ink used in writing the manuscript was performed using WD-XRF spectrometer (S4 Pioneer from Bruker). A small piece of one folio (with text) was analyzed.

Radiocarbon dating: this technique was used to date both of the folios and threads. Two small pieces, one from the folios and one from the thread, from the manuscript were collected and sent to the University of Arizona Accelerator Mass Spectrometry facility to radiocarbon date them.

A small piece (21.27 mg) was taken from a broken edge of a folio, and a small piece (8.41 mg) of the thread that bind the folios was dated to examine whether the manuscript was repaired after its production. The samples were acid-base-acid (ABA) pretreated (1 N HCl, 0.1 % NaOH, and 1 N HCl). After washing and drying, they were Soxhlet extracted with hexane, then ethanol, and finally methanol. Then, they were washed with distilled water, dried, and combusted at 900 °C with copper oxide (CuO). The measurements of ^{14}C were obtained on the produced graphite powder pressed into a target holder following the labs' procedures and calculations described in detail by Donahue et al. (1990) and Jull et al. (2002). The post-bomb radiocarbon dates were calibrated with the CALIBomb program (http://calib.qub.ac. UK/CALIBomb/frameset.html, Reimer et al. 2004) that allows correlation to the nuclear weapons testing era (post-bomb) atmospheric radiocarbon concentration. The smoothing function used is 1.0 years, which works by averaging the calibration curve over 1 year, representing the 1-year turnover time for the skin of an animal. The atmospheric bomb carbon content will therefore be averaged over that time frame. For more details about CaliBomb, see Queen's University Belfast's web site at (http://intcal.qub.ac.uk/CALIBomb/).

Biological analysis: identification of bacterial species present on the folios was performed to reveal the manuscript burial environment. Analysis was performed at the microbiology research lab, Biology Department, Yarmouk University.

Six samples were taken from different folios of the manuscript using sterile swabs (Fig. 2), inoculated on nutrient agar (NA) media, and then incubated at 30 °C for 48 h under aerobic conditions. Bacterial colonies were selected according to variations in morphological characteristics (size, shape, color, and margin). The selected bacterial colonies were sub-cultured into fresh NA media and re-streaked to insure purity of bacterial isolates. Molecular techniques were used to identify bacterial isolates to species level. Bacterial genomic DNA was extracted from the isolates as described by Chachaty and Saulnier (2000). Harvested cells of bacterial culture were lysed in 567 μl Tris-EDTA (TE) buffer, 3 μl (μl) Proteinase K (20 mg/mL), and 30 μL 25 % *w/v* SDS for 30 min at 65 °C. A hundred μl of 5 M NaCl and 80 μL of CTAB/NaCl were added to the mixture and incubated for 10 min at 65 °C. Equal volume of phenol:chloroform:isoamyl alcohol (25:24:1) was added, then the contents were mixed and centrifuged at 14,000 rpm for 5 min. The clear supernatant was extracted and transferred to a new centrifuge tube then, an equal volume of chloroform:isoamyl alcohol (24:1) was added. The sample was centrifuged again at 14,000 rpm for 5 min, and the supernatant was transferred to a new centrifuge tube. Samples were subjected to ribonuclease (RNase) treatment (1.5 mg/mL) for 30 min at 37 °C then cooled on ice, and the DNA was precipitated by the addition of an equal volume of 0 °C isopropanol. The DNA pellets were washed three times with 70 % ethanol and rehydrated in 100 μl TE buffer (pH 7.5).

Genomic DNA was used as template to amplify the 16S Ribosomal RNA (rRNA) genes using the universal primers listed in Table 1 for the 5 bacterial isolates. The polymerase chain reaction (PCR) mixture (50 μl) contains 25 μl master mix (10 mM Tris–HCl (pH 8.3), 50 mM KCl, 2 mM MgCl2, a 200-μM deoxynucleoside triphosphate and 2.5 U of Taq DNA polymerase), 2 μl from reverse and 2 μl from forward 16S rRNA universal primer, 5 μl DNA template, and 16 μl d.H_2O. Reaction mixtures were incubated in DNA thermal cycler (Xp cycler, USA) at 96 °C for 1 min and 35 cycles of PCR reaction (denaturation at 94 °C for 30 s, annealing at 45 °C for 30 s and extension at 72 °C for 1.5 min), according to Daane et al. (2001). Amplified PCR products were resolved by electrophoresis using 1.5 % Agarose gels in 1X Tris-Borate EDTA (TBE) buffer at a low voltage range of 80–90 V for 1.5–2 h. DNA was stained with ethidium bromide (EtBr) (10 mg/mL dissolved in H_2O), and the DNA fragments were visualized using a gel-doc system under UV light. Polymerase chain reactions were repeated twice to check the reproducibility of the banding shapes. A 2 kb (kilobases) DNA ladder was used as the molecular standard in order to confirm the appropriate product length. All reaction mixtures were stored at 4 °C.

Fig. 2 Sampling for biological analysis

Table 1 Universal primers of the isolated were used to amplify the 16S rRNA genes sequence

Primer name	Primer sequence
Forward eubacterial- universal primer	5′AGAGTTTGATCCTGGCTCAG3′
Reverse eubacterial-universal primer	5′ GGTTACCTTGTTACGACTT 3′

Fig. 3 A manuscript folio under transmitted light

Analysis of sequences

The purified PCR products were sequenced on both strands by Macrogen (Seoul, Korea) using the PCR primers. Sequence data was compared to the 16S rRNA gene sequences in the GenBank database using NCBI BLAST (http://blast.ncbi.nlm.nih.gov/Blast) (Purohit et al. 2003). All sequencing results for all isolates were analyzed and used to construct phylogenetic trees using Mega 4 software.

Results

The manuscript's deteriorated physical appearance, described above, suggests that it is old. On the contrary, the precision of hand writing, uniform margins, and precise spacing of the binding sewing suggest that it is a recently produced artifact. In fact, all the pages are similar in size, margin, and number of lines. Compared to the standards employed in old writings, the manuscript letters are highly consistent and have similar size and sharpness. In addition, the binding threads are very thin, cleanly cut, and the sewing holes are pierced as perfect circles.

UV fluorescence of page surfaces did not reveal any underwriting or conservation treatment. The manuscript folios under transmitted light showed uniform thickness, opacity, and scraping marks indicating skilled parchment-makers and clear deterioration features (Fig. 3). XRF analysis results showed the presence of calcium (Ca), iron (Fe), sulfur (S), potassium (K), and chlorine (Cl), while copper (Cu) and zinc (Zn) were not detected (Fig. 4).

The observations described above cannot be used to reliably distinguish between an authentic or forged manuscript, so radiocarbon dating was used to verify the age of the folios and threads.

The radiocarbon results are presented in Table 2 and Fig. 5a, b. They revealed that both the folio and thread were "post bomb," meaning that the animal or plant from which they were derived was living in the period after atmospheric nuclear weapons testing. The radiocarbon content ($F^{14}C$) measured was higher than "modern" radiocarbon content. The calibrated dates of the folio (parchment) at ± one and two standard deviations are as follows: [cal AD 1956.07: cal AD 1956.43] 1.000; [cal AD 1955.89: cal AD 1956.63] 1.000. (1.000 means that all of the probability falls in that time frame). The calibrated dates of the thread at one and two standard deviations are as follows: [cal AD 1956.45: cal AD 1957.09] 0.945, [cal AD 2008.07: cal AD 2008.15] 0.055; [cal AD 1956.28: cal AD 1957.34] 0.769, [cal AD 2007.20 :cal AD 2008.15] 0.231.

It is interesting to note in Fig. 4 that the fraction C^{14} of the folio sample has a single intercept with the ascending limb of the radiocarbon curve, while that of the thread sample has two intercepts with the ascending and descending limbs of the curve. This indicates that the folios could have come from one time period only, while the thread has two possible dates of origin. The calibrated age of the folios falls between 1955 and 1956 at 95.4 % (2 sigma) confidence interval. The calibrated age of the thread at 95.4 % (2 sigma) falls between 1956 and 1957 (76.9 % of the probability) and between 2007 and 2008 (23.1 % of the probability). The thread sample results are permissive of either a mid-1950s date or a late 2000s date. Most likely, these results mean that either the manuscript folios were made and sewed in 1956–1957 or the manuscript was sewed (or re-sewed) in 2007–2008. In both cases, the radiocarbon dates refute the antiquity of the manuscript.

Compared to the GenBank database, the examined surfaces of the manuscript pages produced five dominant bacterial species. The five species and their identification probability are presented in Table 3. A phylogenetic tree for these species is shown in Fig. 6. The presence of *Bacillus megaterium*, *Bacillus subtilis*, *Pseudomonas fluorescens*, and *Pseudomonas aeruginosa* species is not unexpected because they mainly grow in soils, while the presence of *Escherichia coli* (*E. coli*)

Table 2 AMS 14C and calibrated dates of a folio and thread from the manuscript

AA#	Sample ID	Material	$\delta^{13}C$	F	Calibrated AD 2 sigma (95.4 %)	C^{14} age BP
101,205	Parchment	Leather	−21.9	1.0310 ± 0.0041	1955–1956	post-bomb
101,206	Thread	Leather	−22.3	1.0478 ± 0.0042	1956–1957 (76.9 %) 2007–2008 (23.1 %)	post-bomb

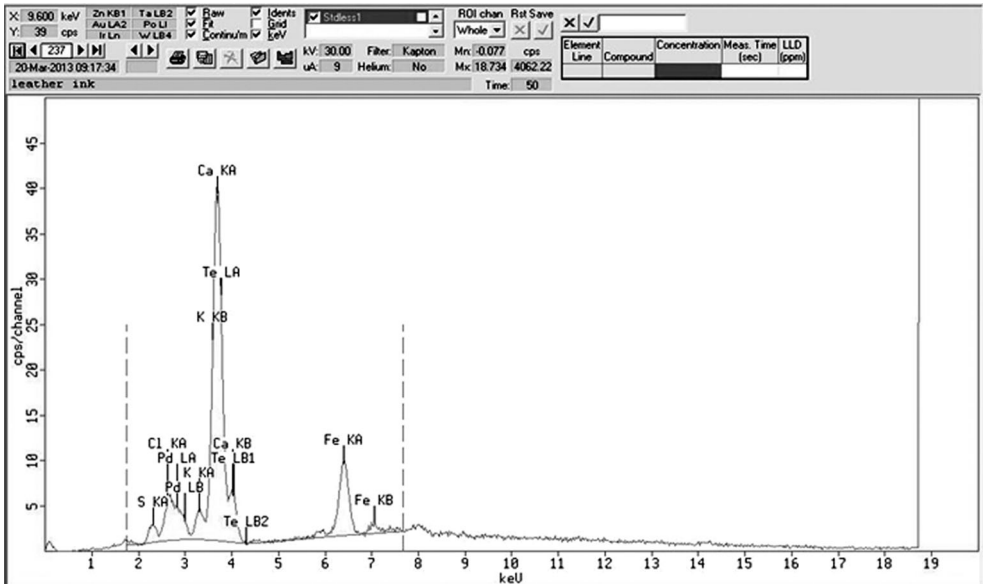

Fig. 4 XRF results of the ink

specie is surprising because it almost exclusively has a fecal origin and grows in the lower intestine of warm-blooded animals (APHA 1992; Berg 2004). The presence of *E. coli* may indicate that the manuscript was exposed to a fecal contamination at a certain stage of its burial in soil. Soils contain a large number of microorganisms that survive for different periods of time depending on their species and burial environments, i.e. soil contents and properties (pH, salinity, humidity, etc). The major group of microorganisms that reside in soil for a long period of time is the Gram positive spore forming bacteria which include *Bacillus*, *Clostridium*, and *Actinobacillus*. On the contrary, Gram negative spore forming bacteria which include *E. coli* could be present in the soil accidentally but for a short period of time.

Discussion and conclusion

Regular use of parchments started during the Hellenistic period, especially in the Middle East and Europe (Craddock 2009). To make a parchment, the skin of young sheep, cow, or deer, consisting of a semi-solid matrix of collagen undergoes a series of treatments (Reed 1972). The final untanned product is a fine, thin and stable dermal layer (Dolgen et al. 2007). Folios of the parchment under investigation are of thin and lightweight leather which might indicate the use of deer skins. Human, biological, chemical, and environmental aging factors (single or combined) may cause physical and chemical deterioration of parchment. These include (among others) handling, reading, macro and micro-organism attack, acids, humidity, light, elevated temperature, and atmospheric pollutants (Dolgen et al. 2007; Marengo et al. 2011).

Table 3 Identified bacterial isolates based on 16S rRNA analysis

Isolate code	Closest relative species	Identity (%)
ARH1	*Escherichia coli*	99
ARH2	*Pseudomonas fluorescens*	98
ARH3	*Pseudomonas aeruginosa*	99
ARH4	*Bacillus subtilis*	97
ARH5	*Bacillus megaterium*	98

The radiocarbon dates of the folios and thread are clearly very young and point to a forged manuscript made with parchments and threads of recent origin. The manuscript exhibits features of great age and deterioration that normally need a long time to form. In order to explain the relatively rapid development of the deterioration features, bacterial species analysis was performed. In contrast to the four soil-derived species, *E. coli* is most likely derived from soils contaminated with animal feces. Animal wastes accelerate the deterioration process of organic materials including parchments (Parodi 2015). Therefore, we conclude that the manuscript was buried in a soil deliberately contaminated with animal waste to accelerate its deterioration and appear old.

Different kinds of ink were used in antiquity. The earliest ink is the carbon ink known from the third century B.C. Carbon ink was produced by mixing soot containing pure carbon and oxidized materials with water and gum to keep the carbon in suspension (Duval and Guicharnaud 2004; Smith 2009; Sharma et al. 2014). Carbon ink was followed by the use of iron gall ink during the Middle Ages. By the end of the late Middle Ages, iron gall ink was the primary ink in use (Kolar et al. 2006). Iron gall ink was primarily made of gallotannic acid (oak galls), iron sulfate (green vitriol), and sometimes also copper sulfate (blue vitriol), gum (binder), and water (Hahn et al. 2004). In some cases, green and blue vitriol were

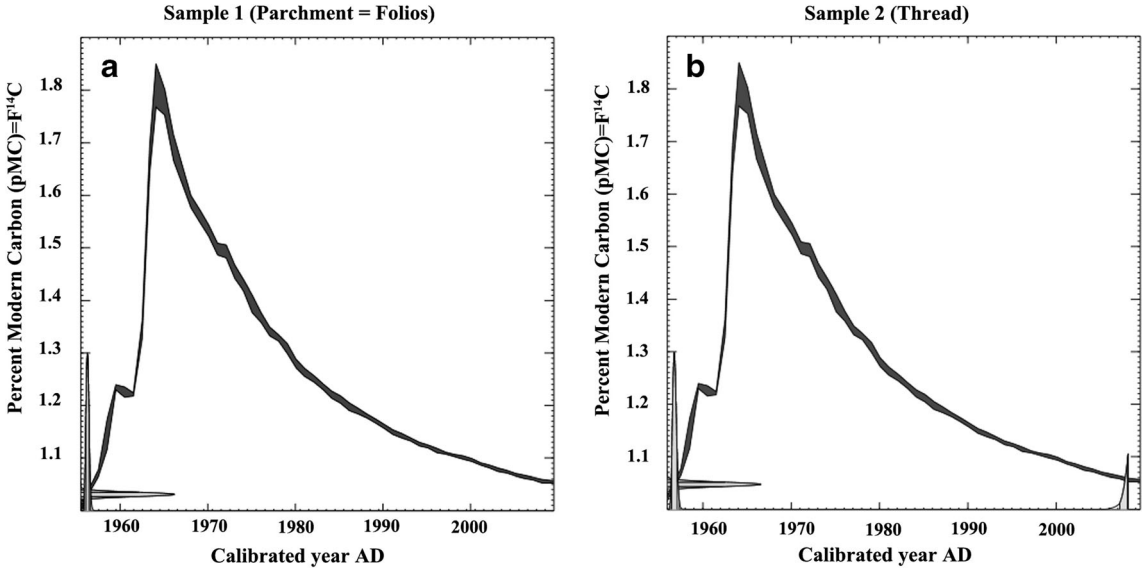

Fig. 5 Calibrated ages AD of **a** the folios showing one intersection in the ascending limb, **b** the thread showing one intersection with the ascending limb and one with the descending limb (two possible dates)

mixed with zinc sulfate (white vitriol) (Thompson 1956; Burguad et al. 2008; Burgio et al. 2010). XRF results reveal the main components of the iron gall ink that was used in writing this manuscript. Fe originates from the vitriol ore and K mainly from the gallnuts. It is likely that copper and zinc were not detected because of the sample's small size or perhaps blue and white vitriol were not added to the ink recipe for this manuscript. Because most iron gall inks turn brown over time, the brown ink on the manuscript does not indicate an aged iron gall ink. The calcium in the XRF spectrum might come from the soil or the tanning processes.

We conclude that the manuscript is forged. It was very likely buried in soil amended with animal wastes to accelerate the aging process. Parchment that is less than 60 years old was used to produce the manuscript, and thread of the same age (or only 6 years old) was used to bind the folios. The forgers of this manuscript intended to produce a document with the appearance of a medieval manuscript. Although they used modern materials, they also used medieval Hebrew script and iron gall ink, common in the Middle Ages, in the construction of the artifact. Aging accelerants were used to impart an appearance of age.

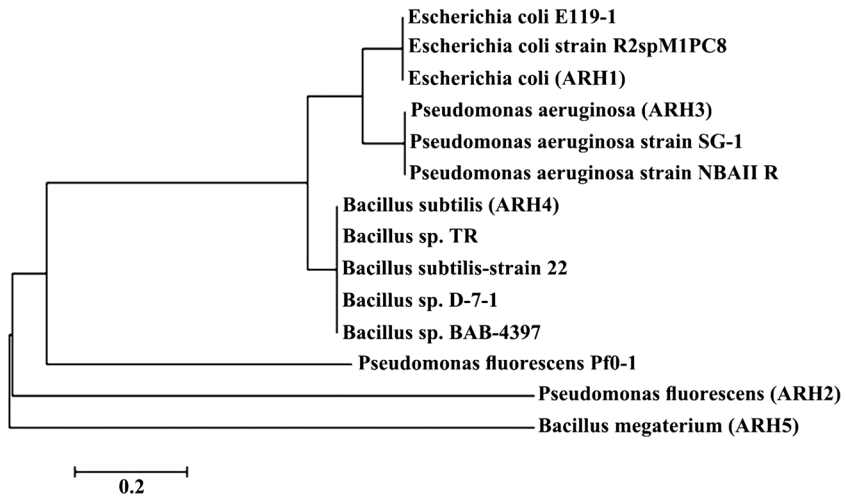

Fig. 6 Phylogenetic tree for bacterial species isolated from the manuscript. The evolutionary distance = 0.2. The evolutionary history was inferred using the Minimum Evolution method. The optimal tree with the sum of branch length = 3.23668491 is shown. The percentage of replicate trees in which the associated taxa clustered together in the bootstrap test (500 replicates) is shown next to the branches. The tree is drawn to scale, with branch lengths in the same units as those of the evolutionary distances used to infer the phylogenetic tree. The neighbor-joining algorithm was used to generate the initial tree. Phylogenetic analyses were conducted in MEGA4

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