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TEXTILES AND RADIOCARBON DATING

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ABSTRACT. Natural textiles provide suitable material for radiocarbon dating. Short-lived organic matter is usually involved and, if ^{14}C dating is applied to pieces that are stylistically well dated, a better understanding of ^{14}C dating of this type of material can be gained. This study presents some examples of dating that support the stylistic dates. Repeated analyses illustrate the robustness of the standard treatment applied to the textiles.

INTRODUCTION

The presence of fibers in archaeological material is of interest to the development of weaving. The very first indication of fiber use was reported from archaeological sites as old as 30 ka BP (Kvavadze 2009). Other archaeological finds suggest that weaving has been known from the very early days of anatomically modern humans (Bar-Yosef 2011). Made of perishable organic material, very old textiles are extremely unique and preserved under special climatic and geochemical conditions (Good 2001). Nevertheless, ages as old as 9 ka BP have been obtained on old charred fragments found in the Far East (Kuzmin et al. 2012). Over millennia, weaving was developed and perfected so that textiles in addition to being everyday objects also became pieces of art. Rugs, carpets, tapestry, decorative cloths, and clothing were created, with each culture and perhaps each workshop leaving a unique style and type. Despite the fact that textiles are fragile and delicate objects, an impressive share of old textiles (up to 4000 yr old, De Moor et al. 2008) is preserved in museums and private collections. Some are studied for stylistic developments and trends of the past. Increasingly, radiocarbon dating has been applied to date textiles. The first attempts were made using conventional counting techniques. Thankfully, because the amount of material used was large and therefore quite destructive for beautiful textiles, these were rather isolated if daring examples (Van Strydonck and Bénazeth 2014). Many consider the remarkable dating of the Shroud of Turin to be a breakthrough in dating textiles (Damon et al. 1989). Despite the heated debate that followed, often beyond scientific reasoning, the fact that such a small piece of a cloth could provide such a consistent age estimate (three laboratories performed the test) demonstrated the potential of ^{14}C dating in building chronologies of precious textile objects. More studies followed that revealed the potential of ^{14}C dating textiles (Jull et al. 1996; Possnert and Edgren 1997; Rageth 2004; Van Strydonck et al. 2004; Kim et al. 2008).

Modern accelerator mass spectrometry (AMS) techniques allow dating of 1 mg of carbon and even smaller amounts to be analyzed today (Ruff et al. 2010); however, in dating art historical objects the size of the sample should be optimal to minimize the analytical error (maximize precision). AMS analysis has a standardized procedure so that reliable measurements, with multiple secondary standards, assure the high quality of data. The main challenge is sampling and sample treatment prior to AMS ^{14}C analysis. Therefore, this article presents an overview from our experience in dating functional historic textiles obtained during nearly 20 yr of collaboration. More than 100 objects, mainly silk and wool, were analyzed but the spectrum of material also included cotton, wood, and paper. From this perspective, the results obtained are of great value and justify application of ^{14}C analysis as a tool to assess and confirm dating, which would corroborate stylistic evaluations and academic affirmations.

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Often, in discussions of textile dating, the accuracy of the ^{14}C dating method is questioned. This study presents examples of repeated analyses that were performed independently (independent sampling, pretreatment, and measurement). In all cases, the results are in very good agreement (1σ) allowing calculation of a mean ^{14}C age, improving precision of the ^{14}C measurements and thereby reducing the ranges of the resulting calibrated ages.

SAMPLING TEXTILES FOR ^{14}C DATING

Over the years, textiles accounted for ~10% of all ETH laboratory analysis with wool and silk being the most frequently dated material. Occasionally, linen and cotton samples were dated, especially in the context of dating canvas.

Knowledge of material storage and conservation history is important for obtaining accurate ^{14}C ages. Although the standard procedure includes treatment with solvents (Figure 1), unique cases of inhomogeneous objects and samples are known. Typically, such problems are caused by repair and conservation. Therefore, sampling must avoid any fragments that cannot be assigned as original. An extreme case is fixing the original surface onto a new background, resulting in the presence of resin or glue, which often requires additional steps to the standard procedure in the laboratory preparation. This is only done when indicated in the submission form or later when the result is obviously spurious. However, this is not an advisable approach because the remains of contamination might only slightly change the ^{14}C concentration (dependent on sample size and age), therefore only slightly shifting the age of the material, and remain undetected.

Figure 1 Soxhlet apparatus at the ETH laboratory is used for treatment of textiles prior to ^{14}C dating. Solvents are boiled below the samples (yellow liquid) and condensed pure liquid refills the sample container (in the middle).

The size of sample is an important issue. Often, the amounts are sufficient and assure quality of the results. Our guidelines for sampling textiles for most accurate and precise ^{14}C ages are as follows:

1. Provide information about the storage and treatment history of the object.
2. Sample amount of 5 mg (prior to treatment, up to 50% loss) is an equivalent (50% of carbon) to 1 mg of C and in ideal cases twice the amount (for quality check). Samples smaller than that cannot undergo multiple treatments.
3. If one suspects a complicated “nature” of the object, contact the lab prior to sampling.

TREATMENT OF TEXTILES

The standard procedure of acid-base-acid (ABA) treatment has been used in ^{14}C laboratories from the early days of ^{14}C dating. The procedure appeared to be sufficient in most cases; for example, the modern treatment and dating performed on Coptic textiles gave concordant ^{14}C ages (Van Strydonck et al. 2004; Van Strydonck and Bénazeth 2014). However, depending on the textiles’ function, history, and preservation, some types of contaminants such as waxes, resin, fat of plant and animal origin, or even tar can be expected. To assure that these types of organic substances do not remain attached to the textiles, an additional step of treatment with solvents has been added (Figure 2). This so-called Soxhlet treatment involves 30-min washes in hot hexane, acetone, and ethanol (Hajdas 2008). The following ABA treatment is applied with care, i.e. dependent of the type of textile and degree of preservation time and temperature chosen (Figure 2). The textiles analyzed prior to 2011 have been combusted (950°C) in quartz tubes with an addition of CuO (oxidation) and Ag powder for purification. Graphite was prepared as described by Hajdas et al. (2004). Currently, the dry and clean textiles are burned in an elemental analyzer (EA) and pure CO_2 is transferred to the automated graphitization line AGE (for details see Wacker et al. 2010). From 1980 to 2008, AMS ^{14}C analyses at ETH were performed on the EN tandem accelerator (Bonani et al. 1987), and then using the MICADAS ^{14}C dedicated system (Synal et al. 2007).

C/N RATIO AND IDENTITY OF TEXTILES

Recently, combustion in the EA allowed for all the samples that are graphitized to have C/N ratios recorded. Our observations are similar to the data presented by Boudin et al. (2013). The first set of data pooled in the lab during 2012 on samples submitted by various customers of various origins, shows that silk and wool can clearly be identified based on their C/N value (Figure 3a).

The values obtained on 10 silk samples varied between 2.95 and 3.19 with mean value at 3.05. Values of the C/N ratio measured on 28 wool samples showed scatter between 3.42 and 3.72, with a mean value of 3.49. Other material, less frequently supplied for ^{14}C dating, such as cotton and linen has a much higher C/N ratio (Figure 3b). Values “in between” have been observed, perhaps indicating a mixture. Our observations show a potential of the textile’s C/N ratio to be not only an indicator of chemical contamination (Boudin et al. 2013) but also of a possible admixture of other textiles.

Figure 2 Treatment of textile samples using standard solvents in a Soxhlet apparatus (see Figure 1) is followed by ABA.

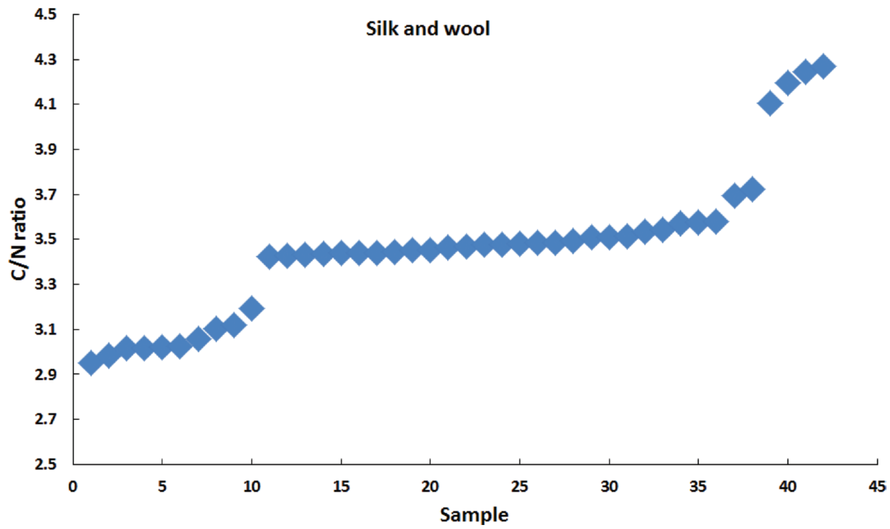

Figure 3a Combustion in the elemental analyzer prior to graphitization provides information about the material. Silk (samples 1–10) can be clearly distinguished from wool (samples 11–39). Samples 40–43 are similar to wool. Another two samples (not shown here) had C/N values of ~7.2, i.e. clearly separated from these two types of material.

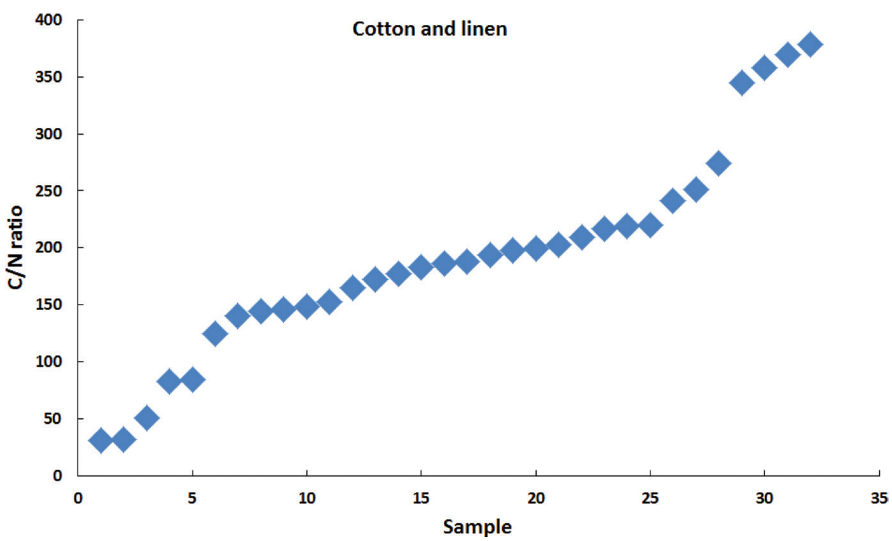

Figure 3b C/N ratios for cotton and linen. For these samples, no clear separation of type is observed and the description of samples was not always clear.

EXAMPLES OF RADIOCARBON-DATED TEXTILES

Example 1

One of the interesting studies involved the analysis of a cotton sample from the canvas of a Tibetan painting of the 15th century (Figure 4a). The goal of this dating was to prove that the painting, representing one of the most venerated Tibetan masters, Tzong khapa (AD 1357–1419), the reformer and founder of the “yellow hat” order, was painted shortly after his death, a custom with the great gurus, and not in a later period. Many more portraits would be done sometimes even centuries later due to the growing devotion and/or a cult devoted to the master.

The painting, of exquisite workmanship, fine detail, and corresponding stylistically to a renaissance period of the arts in west Tibet, would suggest a date of 15th–16th centuries AD, i.e. close to the master's death. Calibration of the ^{14}C age obtained on the canvas provided multiple calendar age intervals from 14th/15th and early 15th century AD, suggesting a period rather close to or around the

Figure 4a Painting of Tzong khapa (AD 1357–1419) sampled for ^{14}C dating.

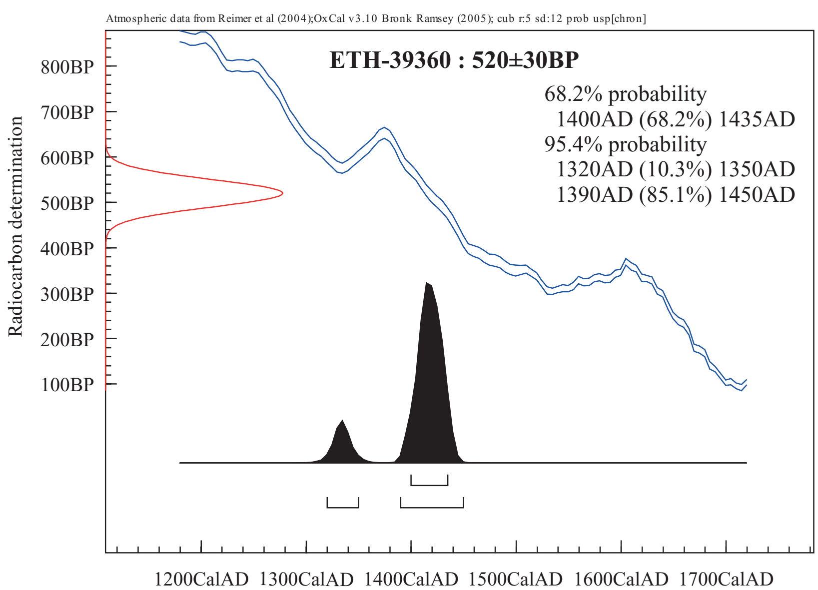

Figure 4b ^{14}C dating results of cotton date the canvas to AD 1390–1450. Calibration was performed as for the original data output, i.e. using OxCal v 3.10 (Bronk Ramsey 1995) and the IntCal04 data set (Reimer et al. 2004).

time of the death of Tzong khapa (AD 1419) (Figure 4b), which agrees with stylistic observations and supports the proposed assumption.

Example 2

The effectiveness of the treatment method applied to typical samples is best illustrated when independent samples are analyzed. Multiple sampling was performed on an Islamic caftan with birds in kufic inscription originating from Central Asia and created in 10th–12th century.

Table 1 Results of dating performed on 2 independent samples of silk (No. 74) and cotton (No. 75) from the caftan.

Sample code	^{14}C age BP	±1σ	Calendar age (2σ)	C/N
No. 74	934	26	AD 1020–1160	3.10
No. 75	958	26	AD 1020–1160	32.25
R_Combine 74 & 75	946	18	AD 1020–1160	

This Islamic caftan, made of silk (No. 74) and lined with cotton (No. 75), was analyzed to assess if the manufacture and the lining were contemporary and also because it should be customary to double-check different materials in dating textiles. For example, a difference in age is possible between the age of the thread of embroidery and that of the cloth bearing the embroidery. In the case of the Islamic caftan, both materials were contemporary and the age was in agreement with stylistic assessment. This example of improved ^{14}C precision (mean value) did not improve the calendar age precision, however (Figure 5 and Table 1). The ^{14}C age is centered on the age plateau at 940 BP and 2σ calendar age range remains as for the lower ^{14}C precision, illustrating the limitation of ^{14}C dating.

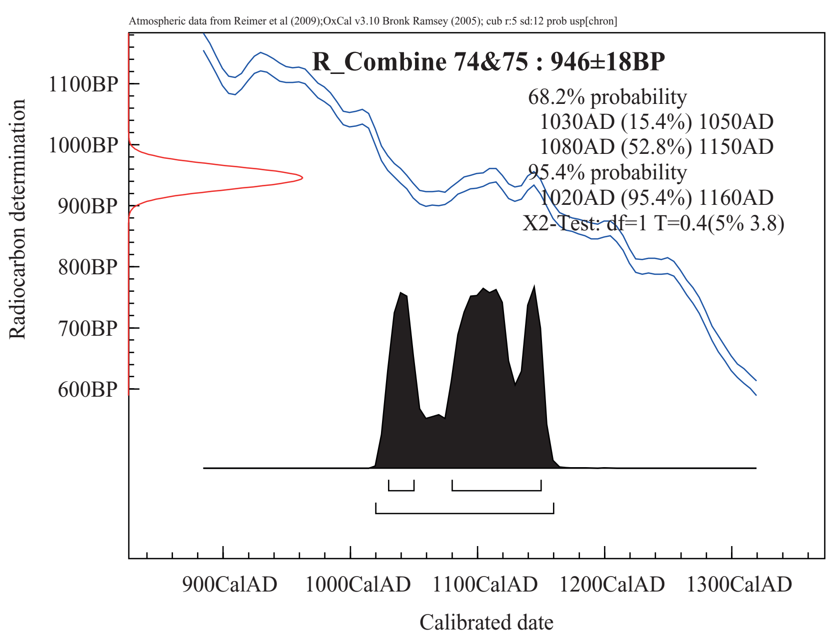

Figure 5 Calibration of combined results for caftan dating results in the period AD 1020–1160, similar to the range (2σ level) obtained for each sample separately. Calibration was performed as for original data output, i.e. using OxCal v 3.10 (Bronk Ramsey 1995) and the IntCal09 data set (Reimer et al. 2009).

SUMMARY

The development of ^{14}C dating textiles has reached a point that very precious objects can be studied successfully and the ^{14}C age thus adds important information. The sampling of unusual objects should be performed with the help and/or supervision of a ^{14}C laboratory. As illustrated in this study, a close collaboration of textile specialist and dating laboratory provides satisfactory results.

REFERENCES

Bar-Yosef O. 2011. Climatic fluctuations and early farming in West and East Asia. *Current Anthropology* 52(S4):S175–S193.

Bonani G, Beer J, Hofmann H, Synal H-A, Suter M, Wölfli W, Pfleiderer C, Junghans C, Münnich KO. 1987. Fractionation, precision and accuracy in ^{14}C and ^{13}C measurements. *Nuclear Instruments and Methods in Physics Research B* 29(1–2):87–90.

Boudin M, Boeckx P, Buekenhoudt A, Vandenabeele P, Van Strydonck M. 2013. Development of a nanofiltration method for bone collagen ^{14}C AMS dating. *Nuclear Instruments and Methods in Physics Research B* 294:233–9.

Bronk Ramsey C. 1995. Radiocarbon calibration and analysis of stratigraphy: the OxCal program. *Radiocarbon* 37(2):425–30.

Damon PE, Donahue DJ, Gore BH, Hatheway AL, Jull AJT, Linick TW, Sercel PJ, Toolin LJ, Bronk CR, Hall ET, Hedges REM, Housley R, Law IA, Perry C, Bonani G, Trumbore S, Woelfli W, Ambers JC, Bowman SGE, Leese MN, Tite MS. 1989. Radiocarbon dating of the Shroud of Turin. *Nature* 337(6208):611–5.

De Moor A, Verhecken-Lammens C, Verhecken A. 2008. *3500 Years of Textile Art*. Antwerp: The Collection ARTheadquARTers. p 104–5.

Good I. 2001. Archaeological textiles: a review of current research. *Annual Review of Anthropology* 30: 209–26.

Hajdas I. 2008. The radiocarbon dating method and its applications in Quaternary studies. *Quaternary Science Journal - Eiszeitalter und Gegenwart* 57:2–24.

Hajdas I, Bonani G, Thut H, Leone G, Pfenninger R, Maden C. 2004. A report on sample preparation at the ETH/PSI AMS facility in Zurich. *Nuclear Instruments and Methods in Physics Research B* 223–224:267–71.

Jull A, Donahue D, Damon P, Orna M. 1996. Factors that affect the apparent radiocarbon age of textiles. In: *Archaeological Chemistry*. ACS Symposium Series, Volume 625. Washington, DC: American Chemical Society. p 248–53.

Kim K, Southon J, Imamura M, Sparks R. 2008. Development of sample pretreatment of silk for radiocarbon dating. *Radiocarbon* 50(1):131–8.

Kuzmin Y, Keally C, Jull A, Burr G, Klyuev N. 2012. The earliest surviving textiles in East Asia from Chertovy Vorota Cave, Primorye Province, Russian Far East. *Antiquity* 86(332):325–37.

Kvavadze E. 2009. 30,000-year-old wild flax fibers (vol 325, pg 1359, 2009). *Science* 326(5951):366.

Possnert G, Edgren T. 1997. Radiocarbon dating of textiles. In: Edgren T, Jungner H, editors. *Proceedings of the VII Nordic Conference on the Application of Scientific Methods in Archaeology*. Helsinki: Finnish Antiquarian Society. p 41.

Rageth J. 2004. Radiocarbon dating of textiles. *Orientations* 35(4):57–62.

Reimer P, Baillie M, Bard E, Bayliss A, Beck J, Bertrand C, Blackwell P, Buck C, Burr G, Cutler K, Damon P, Edwards R, Fairbanks R, Friedrich M, Guilderson T, Hogg A, Hughen K, Kromer B, McCormac G, Manning S, Ramsey C, Reimer R, Remmele S, Southon J, Stuiver M, Talamo S, Taylor F, van der Plicht J, Weyhenmeyer C. 2004. IntCal04 terrestrial radiocarbon age calibration, 0–26 cal kyr BP. *Radiocarbon* 46(3):1029–58.

Reimer PJ, Baillie MGL, Bard E, Bayliss A, Beck JW, Blackwell PG, Bronk Ramsey C, Buck CE, Burr GS, Edwards RL, Friedrich M, Grootes PM, Guilderson TP, Hajdas I, Heaton TJ, Hogg AG, Hughen KA, Kaiser KF, Kromer B, McCormac FG, Manning SW, Reimer RW, Richards DA, Southon JR, Talamo S, Turney CSM, van der Plicht J, Weyhenmeyer CE. 2009. IntCal09 and Marine09 radiocarbon age calibration curves, 0–50,000 years cal BP. *Radiocarbon* 51(4):1111–50.

Ruff M, Fahrni S, Gäggeler HW, Hajdas I, Suter M, Synal H-A, Szidat S, Wacker L. 2010. On-line radiocarbon measurements of small samples using elemental analyzer and MICADAS gas ion source. *Radiocarbon* 52(4):1645–56.

Synal H-A, Stocker M, Suter M. 2007. MICADAS: a new compact radiocarbon AMS system. *Nuclear Instruments and Methods in Physics Research B* 259(1):7–13.

Van Strydonck M, Bénazeth D. 2014. Four Coptic textile from the Louvre collection ^{14}C redated after 55 years. *Radiocarbon* 56(1):1–5.

Van Strydonck M, De Moor A, Bénazeth D. 2004. ^{14}C dating compared to art historical dating of Roman and Coptic textiles from Egypt. *Radiocarbon* 46(1):231–44.

Wacker L, Němec M, Bourquin J. 2010. A revolutionary graphitisation system: fully automated, compact and simple. *Nuclear Instruments and Methods in Physics Research B* 268(7–8):931–4.