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RADIOCARBON DATING OF EARLY EGYPTIAN POT RESIDUES

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Introduction

A number of absolute dating techniques are now used in archaeology, from dendrochronology to a variety of luminescence and radiometric methods.[1] However, radiocarbon dating remains the most effective approach for the early historic periods. This is largely because of the levels of precision achievable, but also due to the diversity of materials that can be dated, and the ease with which radiocarbon dates can be connected to specific events in the past. Radiocarbon dating can be employed on all carbon-containing materials that are biogenic in origin. Common sample types include items fashioned from plant material, such as textiles and basketry, and the remains of animal and human tissue. Radiocarbon estimates denote the time elapsed since the antecedent organism ceased exchanging carbon with its environment. For human and animal remains this is invariably taken to be the time of death, and for plants it is most commonly the time at which the material was harvested or felled.

With the advent of accelerator mass spectrometry (AMS) in the 1980s, it became possible to conduct radiocarbon analysis on samples several orders of magnitude smaller than preceding techniques.[2] Typically, AMS can produce reliable dates on as little as 10 mg of plant material and just 250 mg of whole bone powder. As a result, AMS accounts for a large proportion of the dates made on archaeological samples. No form of radiocarbon dating can, however, provide direct estimates for the age of lithic or ceramic artefacts. The principle difficulty lies in relating any datable material obtained to the manufacture or use of the object in question. In fact, carbonaceous inclusions in such materials are likely to be of geological age, and therefore beyond the 50,000 year detection limit of the technique. Consequently, there remains a disjunction between radiocarbon results and dates based on ceramic seriation. One possibility at bridging this divide comes from the radiocarbon dating of organic residues adhered to specific ceramic types. This prospect was investigated for Early Egypt by an interdisciplinary research team from the University of Oxford, University College London and Cranfield University.

The Samples

The Ashmolean Museum of the University of Oxford houses one of the world's most extensive collections of Early Egyptian ceramics. Some pieces have organic residues adhering to their surfaces. Fortunately, the chemical compositions of many of these residues have already been analysed by Serpico and White,[3] who found them to be predominantly lipids (oils and fats) of both animal and plant origin. Whether the lipids were the remains of food-

[1] See Brothwell and Pollard 2001.
[2] Hedges 1981; Gowlett 1987.
[3] Serpico and White 1996, 2000.

stuffs, offerings or traded commodities is a matter that has subsequently received attention.[4] However, such issues do not impact on their usefulness for absolute dating. For our study, the main concern was whether or not the residues were contemporaneous with their associated ceramics. If so, a radiocarbon date on the residue could be considered an absolute date for the last use of the pot. That is to say, not the time of its manufacture, but the date of its deposition in the funerary context.

The pot residues made available for our study by the Ashmolean Museum came from three of the key sites of the early Egyptian state: Naqada, Ballas and Abydos. Details of the samples taken are given in Table 1, alongside brief summaries of the compositional analysis conducted by Serpico and White.[5]

Accession No.	Site	Context	Pot Description	Likely Residue Composition	Historical Age
1895.525	Naqada	Tomb T5	Wavy-Handled (W14)	Vegetable oil or animal fat	Naqada IIC
1895.533	Ballas	Tomb 588	Wavy-Handled (W14)	Mix of vegetable oil and animal fat	Naqada IIC or IID
1896-1908 E.3158	Abydos	Tomb O; Chamber 30	Abydos ware (Levantine Import)	Vegetable oil	Reign of Djer (1st Dynasty)
1896-1908 E.4034	Abydos	Tomb O; Chamber 30	Base fragment Abydos metallic ware (Levantine Import)	Charred residue (composition unpublished)	Reign of Djer (1st Dynasty)
1896-1908 E.4065	Abydos	Tomb O	Ovoid (Nile silt clay)	Charred animal fat	Reign of Djer (1st Dynasty)
1896-1908 E.4066	Abydos	Tomb O	Ovoid (Nile silt clay)	Carbon and charred animal fat	Reign of Djer (1st Dynasty)

Table 1. The Early Egyptian pots from which samples of residue were obtained for radiocarbon dating.

The two vessels from Naqada and Ballas were both especially distinctive pieces obtained from highly significant contexts of the late Predynastic period. Flinders Petrie described Tomb T5 as one of largest in Naqada's elite cemetery, and noted there were no signs that it had been plundered.[6] However, the grave did include multiple individuals, and the skeletal remains were arranged in a highly unusual fashion.[7] One proposed explanation is that the tomb was in use for several generations. If this were true, the date on the lipid residue obtained by this project would still represent the last use of the pot. A less likely scenario is that intruders disturbed the context and added the vessel at a much later date. However, as this possibility cannot be fully discounted, it does contribute an additional element of uncertainty to our analysis. Both vessels, from Naqada and Ballas, belong to the Wavy-Handled series that epitomises the 'degradation of form' principle that became one of the cornerstones of Petrie's Sequence Dating method.[8] Moreover, the occurrence of this class is also regarded as diagnostic for cultural periods after Naqada IIB.[9] Tomb T5 at Naqada and Tomb 588 at Ballas are

[4] See Serpico and White 1996.
[5] Serpico and White 1996, 2000.
[6] Petrie 1896: 19.
[7] See Hoffman 1991: 116.
[8] Petrie 1901: 5.
[9] Hendrickx 2006: 78.

usually allocated to either Naqada IIC or Naqada IID.[10] As a result, the pot residues were expected to be very similar in age.

The vessels from Abydos were also excavated by Flinders Petrie in the late 19th century AD. None of the four sampled for this project is now wholly intact.[11] They were part of a corpus of pots found in a small, seemingly undisturbed cache to the northwest of Tomb O, the Royal Tomb of King Djer.[12] Djer ruled Egypt during the 1st Dynasty, so these artefacts were expected to be distinctly younger than the Wavy-Handled pots.

The Abydos cache consisted of a mixture of local wares in Nile silt clay and imports latterly determined to be of Levantine origin.[13] However, for the purpose of our chronological analysis, the provenance of the pots was secondary to the issue of whether or not their contents dated to the original burial event. One potential cause for a variance related to a fire that engulfed the tomb in antiquity, leaving most of the vessels either charred or severely burnt.[14] Such an incident could not have directly affected the radiocarbon ages of the residues themselves but it might conceivably have caused some pots to break and their contents to intermingle with surrounding organic material. Indeed, some of the pots do have organic residues adhering to their exterior surfaces, which Serpico and White[15] have suggested might be evidence of such a scenario. However, they also concluded that the many of the least damaged pots contained uncontaminated residues on their interior surfaces. For this reason, only material on the inside of the pots was sampled for this study.

Experimental

Lipid-based compounds are not regularly selected for radiocarbon dating because they are rarely preserved in sufficient quantity for analysis, and some fractions are also labile to chemical modification by their environment.[16] However, those found in the dry, sandy conditions characteristic of the cemeteries of Upper Egypt are highly likely to be free of such contamination.

The pot residues were sampled at the Ashmolean Museum. This generally entailed removing a fragment from a clump at the interior base of the pot after scraping it clear of surface debris. The consistency of the residues varied markedly from case to case. The Naqada residue exhibited the plasticity of fresh lipid material, while the Ballas sample was much grittier in nature. The Abydos residues, in comparison, were more akin to charred plant material. Approximately 50 mg samples were obtained from each vessel, wrapped in foil, and taken to the Oxford Radiocarbon Accelerator Unit (ORAU).

Two different methods were used to chemically prepare the residues for radiocarbon dating. The first method was applied to an aliquot of each sample. It involved the application of the ORAU's standard Acid-Base-Acid (ABA) pre-treatment procedure.[17] Briefly, this involves three steps separated by a threefold rinse with ultrapure water: acid (hydrochloric acid, 1 M, 80^{o} C, 20 min), base (sodium hydroxide, 0.2 M, 80^{o} C, 20 min), acid (hydrochloric acid, 1 M, 80^{o} C, 20 min). The fractions remaining after these aqueous pre-treatments were then freeze-dried overnight. Portions of the residues from the Naqada and Ballas pots were also pre-

[10] Hendrickx, pers. comms.; Payne 1993.
[11] See drawings in Serpico and White 1996: 131.
[12] Petrie 1901: 8–9.
[13] See Adams and Porat 1996.
[14] Petrie 1900: 9.
[15] Serpico and White 1996.
[16] See Evershed et al. 1999.
[17] Brock et al. 2010.

treated in an entirely different manner.[18] Essentially, this method just involved dissolving the residue in chloroform ($CHCl_3$), decanting the supernatant fluid, discarding the insoluble fraction, and then allowing the solvent to evaporate. Thus, for the Wavy-Handled pots, two datable fractions were obtained: one via the standard ABA pre-treatment and the other via dissolution in chloroform.

Approximately 5 mg amounts of each pre-treated sample were then combusted in an elemental analyser coupled to a mass spectrometer, which measured the stable carbon isotope ($\delta^{13}C$) values. The carbon dioxide produced was collected cryogenically, graphitised and dated at ORAU's AMS facility.[19]

Results

One of the Abydos pot residues (1896–1908 E.3158) did not produce enough carbon for AMS analysis. Upon combustion, the carbon content of the pre-treatment product was found to be extremely low (0.2% by weight). This implies the sample was largely inorganic in composition and, therefore, unsuitable for radiocarbon dating. The remaining five residues generated seven radiocarbon measurements (see Table 2). The different pre-treatment protocols applied to the Naqada and Ballas residues resulted in statistically indistinguishable results, suggesting both approaches were equally effective.

All radiocarbon measurements must be converted to calendar date ranges before any meaningful interpretation is possible. This process, known as calibration, is more complex than might initially be expected and is usually achieved by way of computer software. For this study, the radiocarbon measurements were calibrated using the OxCal program.[20] The absolute dates produced after this correction take the form of irregularly shaped probability functions. These are the dark-coloured distributions shown in Fig. 1. Calibrated radiocarbon dates, such as these, should be interpreted as follows. Firstly, radiocarbon dates are always given to a precise level of probability. In Figure 1, the 95% (or 2-sigma) probability ranges are shown. These are the ranges indicated by the square brackets beneath the distributions. The calendar dates encompassed by these brackets are 95% likely to include the true age of the sample. If the level of probability were raised, for example to 99% (3-sigma), then the ranges would expand, and *vice versa* if the probability were lowered. As is apparent in Fig. 1, the ranges are often discontinuous. Here, the interstitial calendar years are not likely to represent the true age, at this level of probability. However, for simplicity's sake, the broadest range of dates is often given, from the earliest to the latest calendar year, as is the case in Table 2.

Discussion

The results obtained on the pot residues showed excellent internal consistency and good agreement with previous radiocarbon-based and historical estimates for the cultural periods represented. For example, in his seminal assessment of the radiocarbon dates of the Egyptian Predynastic, Hassan[21] assigned the Naqada II period to 3650/3600–3300 BC. He also proposed a start date for the 1st Dynasty of approximately 3150/3100 BC. Both these estimates

[18] Based on Fowler 1985.
[19] See Bronk Ramsey et al. 2004.
[20] Bronk Ramsey 1995.
[21] Hassan 1985.

Accession No.	Pre-treat-ment Method	$\delta^{13}C$ (PDB)	Radiocarbon (^{14}C) Measurement		Calibrated Date (95% probability)		Lab No. (OxA-)
			Date (^{14}C Yrs BP)	Error (^{14}C Yrs)	From (BCE)	To (BCE)	
1895.525	ABA	-25.0	4577	35	3498	3106	25417
	CHCl3	-29.2	4543	31	3366	3103	X-2446-41
1895.533	ABA	-24.3	4625	31	3515	3349	26090
	CHCl3	-24.2	4582	29	3498	3119	X-2473-57
1896–1908 E.4034	ABA	-25.4	4344	32	3081	2896	25595
1896–1908 E.4065	ABA	-23.1	4307	33	3014	2884	26044
1896–1908 E.4066	ABA	-24.7	4397	29	3097	2917	26091

Table 2. The radiocarbon dates produced on the Early Egyptian pot residues. The Naqada (1895.525) and Ballas (1895.533) samples were pre-treated using two different protocols, with both approaches producing statistically indistinguishable results. Carbon stable isotope (δ13C) values are used in the calculation of radiocarbon dates and are conventionally published alongside all results.

match our new results precisely. Later radiocarbon dating at Umm el-Qaab[22] on various contexts of the late Predynastic and 1st Dynasty also supported the time ranges proposed by Hassan. Indeed, Hendrickx's[23] recent review implies that Hassan's dates remain the best guidelines for the absolute age of the formative period. Such independent corroboration of our results strengthens the likelihood that the residues we dated were indeed contemporaneous with the original deposition events. The anticipated offset between the Predynastic and Early Dynastic vessels is clearly evident in Fig. 1. Nonetheless, the four radiocarbon results produced on the Wavy-Handled pots were disappointingly broad, as a result of the reference data against which the calibration adjustment is made over this time period. Because the pairs of results on each Wavy-Handled pot amount to duplicate age estimates on the same sample, it is permissible to average them. This does not dramatically change the date ranges for the Naqada residue (3482–3110 BC, 95% probability, compare Table 2), but it does improve the precision of the Ballas sample (3499–3344 BC, 95% probability), and provides some suggestion that this residue is in fact the older of the two. Such a distinction, however, could not be made definitively without obtaining further radiocarbon measurements.

The three results produced on the Abydos samples are statistically identical, supporting the belief that the vessels were interred at the same time. Indeed, the measurements are consistent with the pot contents dating from a single calendar year. If this were true and the organic materials fresh at the time of deposition, it would be possible to average the three results, reducing the associated date range to just 111 years (3020–2909 BC, 95%). Nonetheless, the results still convincingly allocate the Tomb of Djer, second king of the 1st Dynasty, to the turn of the 3rd millennium BC, a reference point of considerable value in its own right.

One further technique that could be used to enhance the precision of both the Predynastic and Early Dynastic results is Bayesian statistical modelling.[24] But for any significant improvement to be achieved, such analysis would require additional samples and several more chronometric dates. Moreover, it would need to be possible for chronological relationships between the samples to be defined on the basis of established historical or archaeological evidence.

[22] Görsdorf et al. 1998.
[23] Hendrickx 2006: 92.
[24] See Bronk Ramsey 2009.

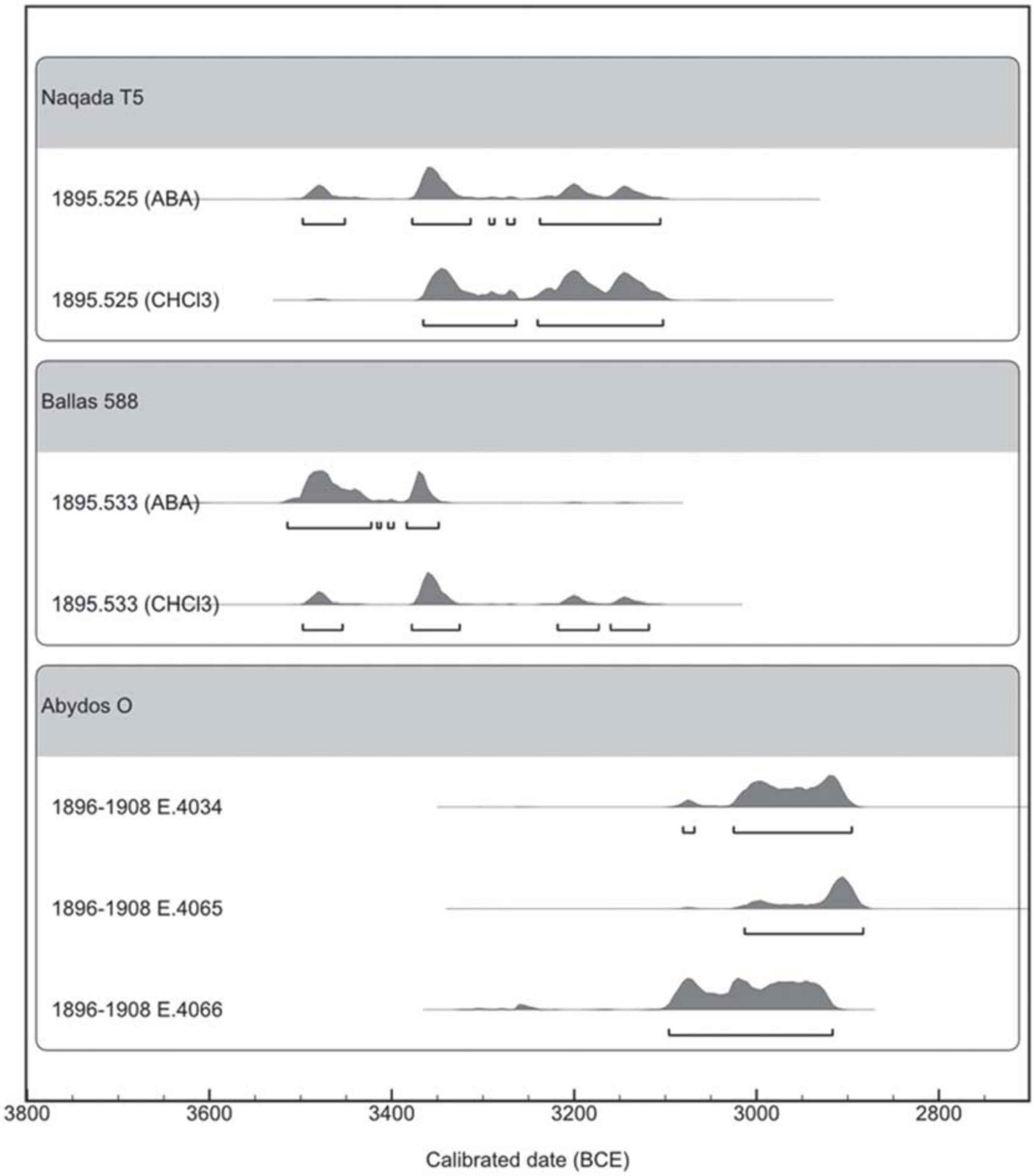

Fig. 1. The calibrated date ranges (square brackets, 95% probability) for the seven pot residues. The full extent of the date ranges is given in Table 2.

Nonetheless, the unmodelled dates presented here clearly demonstrate that pot residues can be used as good dating proxies for both individual vessels, and for their broader archaeological contexts.

Conclusions

This research sought to establish whether radiocarbon dating could be used to produce absolute dates for Early Egyptian ceramic artefacts by analysing organic residues adhered to their interior surfaces. Five vessels from three of the key sites of Egypt's formative period were dated and the results were both highly coherent and commensurate with historical estimates. This research points the way for further analysis of such pot residues, which may provide the most direct means by which the pottery-based chronologies for Early Egypt can be anchored to the absolute time-scale.

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Radiocarbon dating of Early Egyptian pot residues

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