

Luminescence as a Relative Dating Tool: Part B – Application

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Abstract

This paper discusses the applicability of using luminescence as a *relative numerical* dating tool. Examples of when such an application may be useful include the dating of museum materials for which original external dose rate information is no longer obtainable. Without the external dose rate, it is still possible to obtain the relative ages of two or more vessels, which is particularly useful when attempting to ascertain typological sequences or chronological implications of archaeological assemblages. This paper presents a case study on determining the relative numerical age using optically stimulated (OSL) dating, carried out on a group of ancient Egyptian ceramics. This paper is preceded directly by Part A (Highcock et al., 2019) of this article, which presents a derivation of the formulae for obtaining both a relative luminescence age and the associated relative error.

Keywords: OSL dating, relative dating, museum material, Egyptian archaeology, Naqada Culture, Egyptian chronology, Predynastic Egypt, Early Dynastic Egypt, ceramics.

of relative dating by luminescence are studies of material from museum collections, for which no original sedimentary material is available for the determination of \dot{D}_{ext} ; for such (often unprovenanced) museum specimens the knowledge of relative chronological sequences is beneficial.

The companion paper (Part A, Highcock et al., 2019) of this article presents a derivation of the formulae that can be used to obtain relative ages using luminescence, as well as its associated relative error. Here, in Part B, we now present a case study, using an assemblage of wavy-handled vessels and wine jars from the Predynastic and Early Dynastic Periods of Egyptian history (c. 3300 – 3000 BC).

As discussed further in Part A, this new approach necessitates somewhat non-standard nomenclature when referring to ages. To summarise: what is generally known as a numerical age, which is given as a number of years before present or as a calendar date, we refer to as an *absolute* numerical age. What is generally known as a relative age, as determined by, for example, seriation, we continue to refer to as a relative age. The calculation presented in Part A defines a *relative numerical* age. Like a standard (absolute) numerical age, the relative numerical age is expressed as a number, and that number can be used to make quantitative statements like “this vessel is twice as old as that vessel.” Like a traditional relative age, the relative numerical age cannot (without additional evidence) be related to a number of years before present, or a calendar date.

1. Introduction

Luminescence dating can be used to determine relative ages for ceramic assemblages, even in the absence of external dose rate (\dot{D}_{ext}) measurements, which may not always be available. The most obvious example of the usefulness

2. The data set

To illustrate how OSL can be used as a relative numerical dating technique, and thus be used to further improve

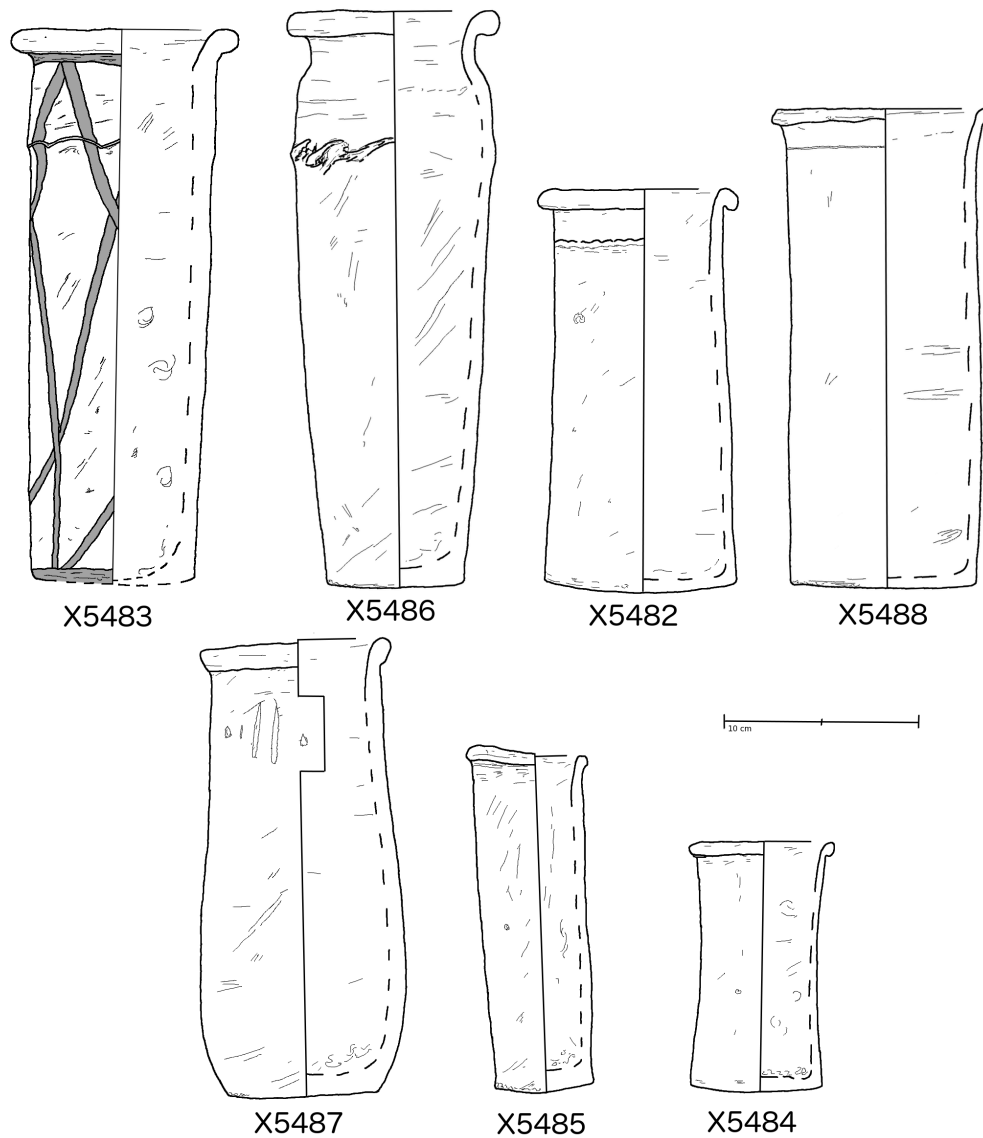


Figure 1. Wavy-handled vessels from Turah used in this study. Vessels appear in their relative chronological order (oldest to youngest left to right and top to bottom).

our understanding of the chronology of archaeological assemblages, two sets of objects were examined: a selection of seven wavy-handled vessels from Predynastic and Early Dynastic Egypt from the site of Turah (Figure 1), and three wine jars—two archaeologically complete specimens from Turah, and one sherd from Hierakonpolis, each inscribed with a *serekh* or pot mark (Figure 2).

The Turah material offered an almost complete relative sequence of wavy-handled vessels for study. This assemblage is a prime set of material on which to demonstrate how OSL can be used as a relative numerical dating technique, because, with regard to ceramic typology, it is one of the most well-understood ceramic assemblages from early Egypt. Wavy-handled vessels provided the backbone of Petrie's ceramic sequencing system, developed in the late

19th century, which is still a tool for relative dating of Egypt's earliest pottery today (Petrie, 1899, 1901). The wavy-handled vessel type is observed across Pre- and Early Dynastic Egypt, and was fundamental in defining the Naqada Culture and establishing the archaeological chronology for the Predynastic and Early Dynastic phases of Egyptian history (here we follow the Naqada Culture as defined in Hendrickx (1996, 2006); see also Kaiser (1957); Köhler (2004); Köhler & Smythe (2004); Köhler (2013) and further discussion of terminological inconsistencies in Köhler & Thalmann (2014) and Hood (2017)). It continues across several archaeological phases spanning the Predynastic and Early Dynastic Periods. While other vessel types came and went, the wavy-handled vessel continued to develop. Petrie originally based his seriation of Predynastic and Early Dynas-

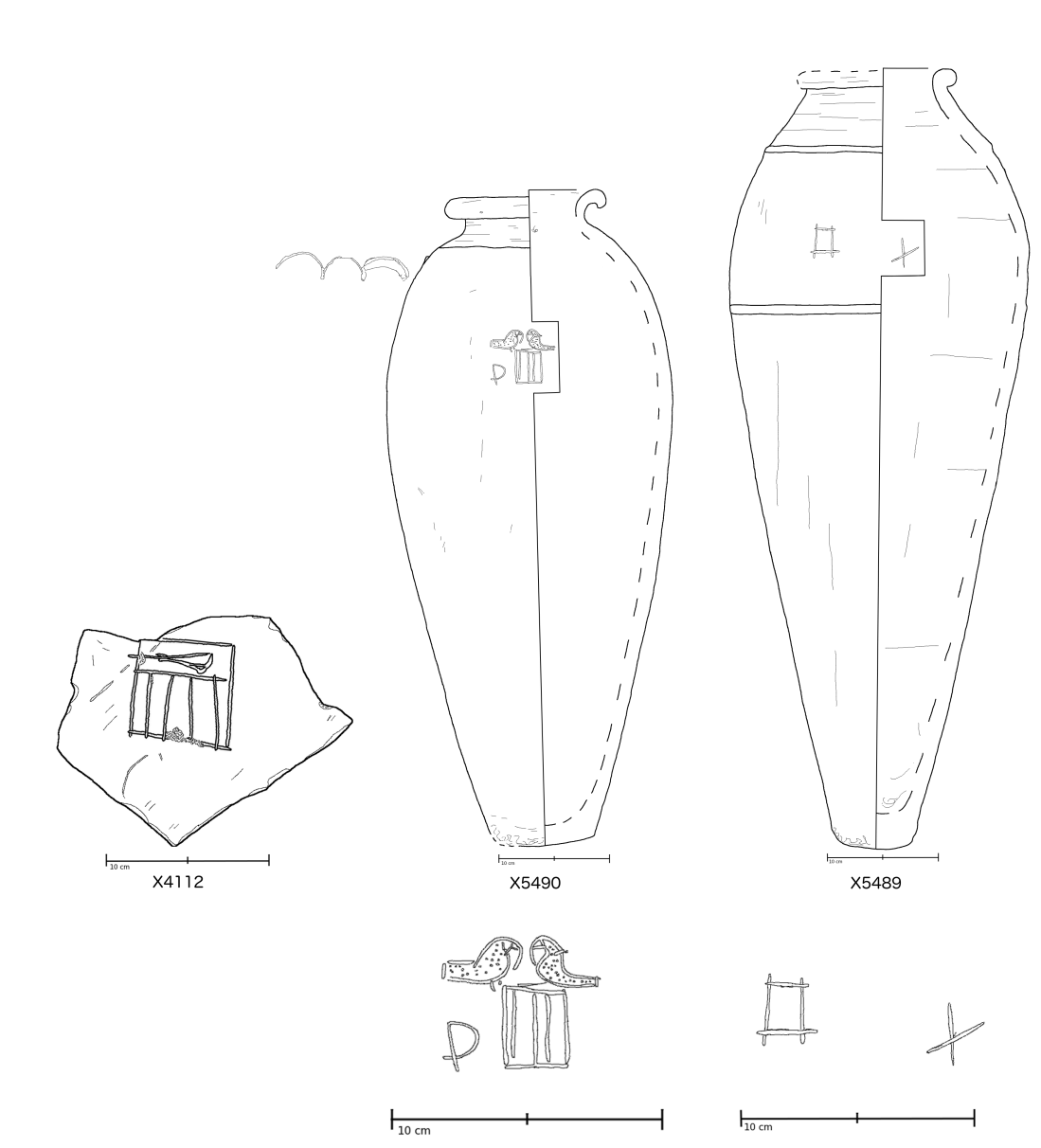


Figure 2. Wine jars used in this study. X4112, the sherd, is from Hierakonpolis; X5489 and X5490 are from Turah. Also shown at the bottom of the figure are enlarged drawings of the pot marks appearing on X5489 and X5490.

tic Egyptian ceramics on this vessel type, noting the gradual change in vessel form from the bulbous vessel with functional wavy handle, to the elongated cylindrical shape with a fine wavy decoration, which no longer served as a functional handle. Indeed, the final known development of this vessel type no longer has a wavy decoration at all, and is a smaller and rougher cylindrical form when compared to its predecessors (Köhler, 2004). The seven wavy-handled/cylindrical vessels selected for OSL analysis here represent an excellent typological assemblage of this particular ware, spanning the Naqada IIIA to Naqada IIIC2 periods.

In addition to the wavy-handled vessels, the three wine jars were also examined with luminescence. Two of these had an inscribed *serekh* (pre-fired engraved marks on the vessel in the form of a rectangle, containing a symbol/name of

historical figures). These vessels, owing to their vessel index (Köhler & Smythe, 2004), their typology and their inscribed historical information (i.e. a *serekh*, which can often be directly linked to known figures or time period within the historical chronology) (Van Den Brink, 2001, 1996), make prime candidates for illustrating the applicability of OSL as a relative numerical dating technique.

Unfortunately, with regard to the wavy-handled vessels, although it is known that all these ceramics studied here come from the site of Turah, the exact provenance of each piece was not recorded. No original depositional sediment was attached to the Turah material, and given that the site is now under military occupation, it is at present unlikely that absolute numerical luminescence dates will be obtained for this material in the future.

Table 1. Relative ages of vessels from Turah and Hierakonpolis. Relative ages of the wavy-handled/cylindrical vessels and the wine jars from Turah and Hierakonpolis. The table shows the equivalent dose (D_e) and the total internal dose rate (\dot{D}_{int}) for both coarse grain (CG) and fine grain (FG) material as available. Also shown are the relative numerical ages of each pot, relative to a reference vessel (X5486, see text), again for CG and FG. Finally, the table also shows a combined age, which is either the CG or FG age if only one is available, or a combination of both CG and FG results made by using kernel density estimation. It is this combined age which is displayed in Figure 3. Also given is the ratio N of accepted aliquots to total aliquots. Element concentrations used to determine \dot{D}_{int} are available in Hood (2017). The following rejection criteria were applied: test dose error: $\leq 20\%$; recycling ratio: $\leq 20\%$; recuperation: $\leq 5\%$; IRSL/OSL ratio: $\leq 15\%$ (Note that these rejection criteria are slightly higher than ‘standard’ rejection criteria. This is owing to MET sampling producing very few aliquots for some samples and therefore in order to work with these samples a more flexible rejection criteria was implemented. Further details can be found in Hood (2017)). Uncertainties in D_e are calculated, using the central age model, from non-rejected aliquots; uncertainties in \dot{D}_{int} , which incorporate the uncertainty in elemental analysis, were determined by DRAC (Durcan et al., 2015); uncertainty in the relative ages is calculated using the uncertainties in D_e , the uncertainties in \dot{D}_{int} , and the uncertainty that results from the relative age formula as detailed in Part A (Highcock et al., 2019).

| Site | Vessel | N | | D_e (Gy) | | \dot{D}_{int} (Gy/ka) | | Date Relative to X5486 (expressed as a ratio) | | Combined Date Relative to X5486 (as a ratio) |
|-------|--------|-------|-----|------------------|------------------|-------------------------|-----------------|--|-----------------|--|
| | | CG | FG | CG | FG | CG | FG | CG | FG | |
| Turah | X5482 | N/A | 6/6 | N/A | 12.05 ± 0.61 | N/A | 1.68 ± 0.07 | N/A | 1.18 ± 0.09 | 1.18 ± 0.09 |
| Turah | X5484 | 1/8 | N/A | 8.09 ± 1.68 | N/A | 1.38 ± 0.07 | N/A | 0.86 ± 0.19 | N/A | 0.86 ± 0.19 |
| Turah | X5486 | 3/7 | 6/6 | 9.60 ± 0.78 | 11.27 ± 0.40 | 1.41 ± 0.08 | 1.84 ± 0.07 | 1.00 ± 0.11 | 1.00 ± 0.07 | 1.00 ± 0.06 |
| Turah | X5488 | 3/6 | N/A | 10.17 ± 1.36 | N/A | N/A | 1.63 ± 0.06 | N/A | 1.02 ± 0.15 | 1.02 ± 0.15 |
| Turah | X5489 | 2/14 | 5/6 | 8.73 ± 0.32 | 10.05 ± 0.25 | 1.50 ± 0.08 | 1.81 ± 0.07 | 0.85 ± 0.06 | 0.91 ± 0.06 | 0.88 ± 0.04 |
| Turah | X5490 | N/A | 6/6 | N/A | 9.81 ± 0.69 | N/A | 1.63 ± 0.06 | N/A | 0.98 ± 0.09 | 0.98 ± 0.09 |
| HK | X4112 | 11/18 | N/A | 7.43 ± 0.48 | N/A | 1.65 ± 0.10 | N/A | 0.66 ± 0.06 | N/A | 0.66 ± 0.06 |

Far more is known about the provenance of the Hierakonpolis sherd than the vessels recovered from Turah. Owing to the nature of the *serekh* inscription, which possibly exhibits the name of Narmer, arguably Egypt’s first Pharaoh, a rather detailed discussion of its find-spot was included in its publication (Garstang, 1907: 135, Pl. III; cf. Adams, 1995: 123–124). Unfortunately, no original depositional material was attached to this sherd either.

3. Methodology

All OSL samples were collected using the minimum extract technique (MET) sampling protocol, specifically designed for use on museum materials (Hood & Schwenninger, 2015). Sample preparation was done in subdued lighting conditions following standard coarse grain and fine grain sample preparation (Hood & Schwenninger, 2015; Hood, 2017). OSL measurements were carried out on a Risø automated DA-15 luminescence reader. The SAR protocol (Murray & Wintle, 2000) was used in combination with a post-IR blue measurement so that any IRSL signal present owing to feldspar contaminants would be removed by IR stimulation carried out before measuring the OSL signal during each SAR cycle (Banerjee et al., 2001; Mauz & Lang, 2004). An IRSL/OSL depletion ratio of $\leq 15\%$ was used as a rejection criterion.

Optical excitation was achieved by the use of filtered blue diodes (410–510 nm emission), and infrared stimulation using IR diodes. Luminescence signals were detected in the UV spectrum by an EMI 9635Q bialkali PMT, fitted with a 7.5 mm Hoya U340 glass filter (Riso, 2007). Sample irradiation was done using a sealed ^{90}Sr beta source at a rate of approximately 2.3 Gy/min, and calibration was carried out with Risø calibration quartz (Hansen et al., 2015). Equivalent dose (D_e) determination was done using the *Analyst* software package, V4.12 (Duller, 1999), and rejection criteria determined for use with MET sampling were used (see Table 1 caption; Hood 2017). Internal dose rate (\dot{D}_{int}) measurements were obtained by ICP-MS analysis.

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4. Results

Table 1 presents the final D_e measurement results and the internal dose rate for each vessel obtained using ICP-MS.

Of the 10 vessels examined, all three wine jars produced acceptable OSL signals, but three of the seven wavy-handled vessels did not (X5483, X5485, X5487), which is a relatively high degree of failure. Of these three vessels, two are made of marl clay and one from Nile silt, both materials for which successful OSL measurements were performed here and in other studies. It is unfortunate that X5483, X5485 and X5487 did not yield results, as all three were particularly diagnostic.

5. Discussion

Figure 3 presents the relative ages of the seven vessels for which OSL results were obtained. There are two extreme values, X5482 and X4112, whose relative ages of 1.15 and 0.7 would imply that they are roughly 700 years older and 1500 years younger than the rest of the group respectively, which is not consistent with known historical sequences (further discussion below).

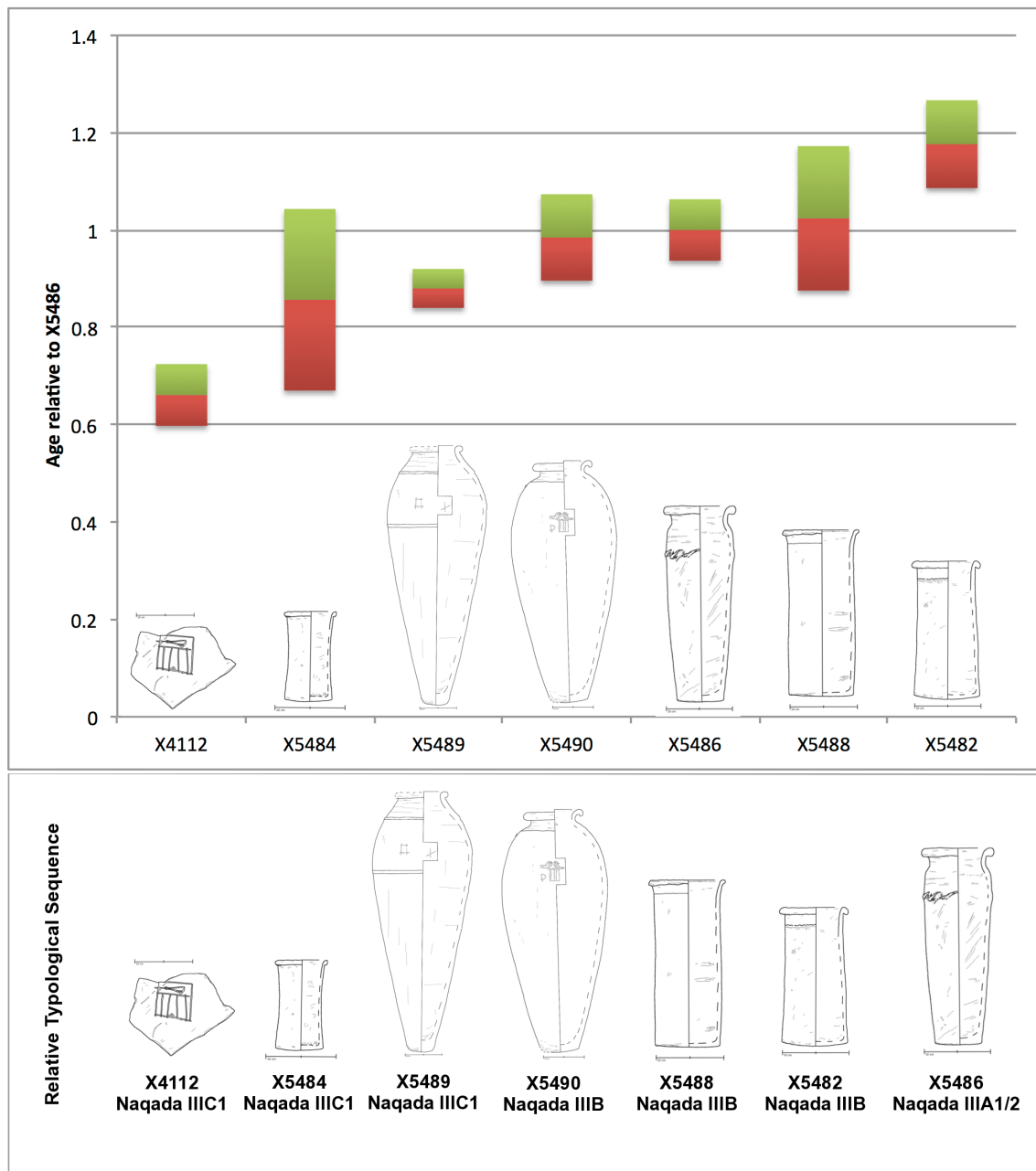


Figure 3. Upper: this graph shows the relative sequence of each vessel that produced a D_e measurement in the Turah/Hierakonpolis assemblage, based upon X5486 which acts as an anchoring reference point for the sequence (see text). The relative numerical age and error are to be found in the final column of Table 1. In turn, this graph therefore indicates the chronological progression of each vessel relative to X5486, with X4112 being the youngest in the assemblage. The red and green bars denote the upper and lower errors with the boundary between them being the central age value. Lower: in contrast with the upper figure, this figure depicts the relative typological sequence for this material based upon archaeological evidence, and thus visually demonstrates the issues encountered with some of the OSL D_e measurements.

The errors associated with the remaining five vessels are broad compared to the difference in relative ages between them. This means that any concrete statement about whether one vessel is older or younger than another must be treated with caution. However, Figure 3 also clearly demonstrates a strong relationship between the relative OSL sequence and the typological ceramic sequence. While it is beyond the scope of this paper to enter a discussion of the full archaeological implications and analysis of the OSL results (inter-

ested readers are referred to [Hood, 2017](#)), it is appropriate to say that the relative OSL age sequence of vessels is in excellent keeping with established ceramic typology of the Naqada Culture. Indeed, with the exception of X5486, all vessels are in sequence in accordance with latest typological research ([Hendrickx, 1996, 2006](#); [Köhler, 2004](#); [Köhler & Smythe, 2004](#); [Van Den Brink, 1996, 2001](#)).

Although X5486 dates to the Naqada IIIA1/IIIA2 period, it is placed in the OSL relative sequence as being younger

than X5488 and X5482, which is not in keeping with the relative archaeological sequence, which places X5488 and X5482 within the later Naqada IIIB period. Within the OSL relative sequence it is almost impossible to distinguish between X5486 and X5488, due to the significant errors associated with X5488. Indeed, this is the situation between all three vessels (X5482, X5486 and X5488): their associated errors make it difficult to distinguish a true chronological sequence here, except for confidently being able to state that all three vessels are chronologically older than X5484. This is a resolution error and could be further refined should a larger data set be available. In the case of X5482, the discrepancy in relative age between it and X5486/X5488 is approximately 15% (see Figure 3), which in terms of numerical age gives an error of ~750 years. This is very unlikely and could be the result of a measurement error: a single aliquot within the OSL data produced a D_e measurement which was significantly higher than the other measurements and is an outlier. However, as this aliquot could not be discounted based upon the standard rejection criteria applied, it was considered best practice to include this measurement even though it is likely to produce an overestimation in the OSL measurement.

We must also discuss sherd X4112. With regard to its relative OSL sequence, it should be noted that although fitting in with the relative sequence of the other two wine jars, the difference between the relative ages of X4112 and X5489/X5490 sits at approximately 20% (see Figure 3). This is far too large a discrepancy to fit with the relative typological sequence as it would place X4112 roughly 1000 years later, in absolute terms, than the rest of the assemblage.

6. Conclusion

This paper has demonstrated that the application of OSL dating as a relative numerical dating method has benefits for examining the relative typological sequence of ceramics. With the small data set and large uncertainty in the dates presented, it is currently only possible to demonstrate this technique as a proof-of-principle and to make broad statements when comparing the OSL vs. archaeological relative chronologies. However this in no way invalidates the usefulness of OSL dating as a relative numerical dating tool, but rather means that more data (i.e. more vessels) are required than in this pilot study. If several examples of each vessel type were sampled, in combination with good statistical modelling and, even better, a technique such as cladistics (i.e., Hood & Valentine, 2012), OSL as a relative dating technique would be a powerful tool. Even with the limited data available, we can make the following positive observations: that the five non-outlier ages are well clustered and could be considered consistent given errors with a spread in relative numerical ages of around 5% (that is, roughly 250 years), and are consistent with the known historical chronology.

Although the wavy-handled/cylindrical vessels have proved somewhat problematic owing to issues surrounding a small data set, the wine jar assemblage demonstrates that the relative sequence of ceramics achieved through OSL dat-

ing of this assemblage has been in full agreement with the pre-existing ceramic sequence described for the Naqada Period.

In the future, this technique could be used on suitable archaeological materials world-wide. It may be of particular value when working with museum contexts, where limited archaeological information is available, or where the internal chronology of an assemblage is little understood. This technique could also be used to identify forgeries in museum collections.

While in itself this paper has not yielded new information (the relative sequences of the wavy-handled vessels and wine jars are well documented and well understood in Egyptian archaeology), this paper has demonstrated that OSL dating can be of benefit to relative chronology as well as absolute, providing a framework for implementing OSL dating as a relative numerical dating method. While the usefulness of this technique will be heavily dependent upon individual assemblages and the quality of available relative dating methods, further potential for this technique is significant in the museum world and further advances in ceramic chronology could be made as a result.

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References

- Adams, B. *Ancient Nekhen: Garstang in the City of Hierakonpolis*. SIA Publishing, New Malden, 1995.
- Banerjee, D., Murray, A., Bøtter-Jensen, L., and Lang, A. *Equivalent Dose Estimation Using a Single Aliquot of Polymineral Fine Grains*. *Radiation Measurements*, 33(1): 73–94, 2001.
- Duller, G. *Luminescence Analyst Computer Programme*. Aberystwyth, v4.12 edition, 1999.
- Durcan, J., King, G., and Duller, G. *DRAC: Dose Rate and Age Calculator for trapped charge dating*. *Quaternary Geochronology*, 28: 54–61, 2015.
- Garstang, J. *Excavations at Hierakonpolis, at Esna and in Nubia*. *Annales du Service des Antiquités de l'Égypte*, 8: 132–148, 1907.

- Hansen, V., Murray, A., Buylaert, J.-P., Yeo, E.-Y., and Thomsen, K. *A New Irradiated Quartz for Beta Source Calibration*. *Radiation Measurements*, 81: 123–127, 2015.
- Hendrickx, S. *The Relative Chronology of the Naqada Culture. Problems and Possibilities*. In Spencer, J. (ed.), *Aspects of Early Egypt*, pp. 36–69. British Museum Press, London, 1996.
- Hendrickx, S. *Predynastic–Early Dynastic Chronology*. In Hornung, E., Krauss, R., and Warburton, D. (eds.), *Ancient Egyptian Chronology*, pp. 55–93. Brill, Leiden, 2006.
- Highcock, E., Hood, A., and Schwenninger, J.-L. *Luminescence as a Relative Dating Tool: Part A – Theory*. *Ancient TL*, 37(2): 9–13, 2019.
- Hood, A. *New Insights into Old Problems: The Application of a Multidisciplinary Approach to the Study of Early Egyptian Ceramic Chronology, with a Focus on Luminescence Dating*. PhD thesis, University of Oxford, 2017.
- Hood, A. and Schwenninger, J.-L. *The Minimum Extraction Technique: A New Sampling Methodology for Optically Stimulated Luminescence Dating of Museum Ceramics*. *Quaternary Geochronology*, 30: 381–385, 2015.
- Hood, A. and Valentine, J. *The Application of Cladistics to Early Dynastic Egyptian Ceramics: Applying a New Method*. In Knoblauch, C. and Gill, J. (eds.), *Egyptology in Australia and New Zealand 2009: Proceedings of the Conference Held in Melbourne, September 4th–6th*, pp. 47–59, Oxford, 2012. Archaeopress.
- Kaiser, W. *Zur Inneren Chronologie der Naqadakultur*. *Archaeologia Geographica*, 6: 69–77, 1957.
- Köhler, E. *On the Origins of Memphis: The New Excavations in the Early Dynastic Necropolis at Helwan*. In Hendrickx, S., Friedman, R., Ciałowicz, K., and Chłodnicki, M. (eds.), *Egypt at its Origins: Studies in Memory of Barbara Adams. Proceedings of the International Conference “Origin of the State. Predynastic and Early Dynastic Egypt”, Kraków, 28th August–1st September 2002*, volume 138 of *Orientalia Lovaniensia Analecta*, pp. 295–315, Leuven, 2004. Peeters.
- Köhler, E. *Early Dynastic Egyptian Chronologies*. In Shortland, A. and Bronk Ramsey, C. (eds.), *Radiocarbon and the Chronologies of Ancient Egypt*, pp. 224–234. Oxbow Books, Oxford, 2013.
- Köhler, E. and Smythe, J. *Early Dynastic Pottery from Helwan – Establishing a Ceramic Corpus of the Naqada III Period*. *Cahiers de la Céramique Égyptienne*, 7: 123–144, 2004.
- Köhler, E. and Thalmann, J.-P. *Synchronising Early Egyptian Chronologies and the Northern Levant*. In Höflmayer, F. and Eichmann, R. (eds.), *Egypt and the Southern Levant in the Early Bronze Age*, volume 31 of *Orient-Archäologie*, pp. 181–206. Rahden/Westf., Leidorf, 2014.
- Mauz, B. and Lang, A. *Removal of the Feldspar-derived Luminescence Component from Polymineral Fine Silt Samples for Optical Dating Applications: Evaluation of Chemical Treatment Protocols and Quality Control Procedures*. *Ancient TL*, 22(1): 1–8, 2004.
- Murray, A. and Wintle, A. *Luminescence Dating of Quartz Using an Improved Single-Aliquot Regenerative-Dose Protocol*. *Radiation Measurements*, 32(1): 57–73, 2000.
- Petrie, W. *Sequences in Prehistoric Remains*. *Journal of the Royal Anthropological Institute of Great Britain and Ireland*, 29: 295–301, 1899.
- Petrie, W. *Diospolis Parva*. Egypt Exploration Fund, London, 1901.
- Riso, T. *Guide to “The Riso Single grain laser OSL system”*. Technical report, Riso National Laboratory, 2007.
- Van Den Brink, E. *The Incised Serekh-Signs of Dynasties 0-1, Part I: Complete Vessels*. In Spencer, J. (ed.), *Aspects of Early Egypt*, pp. 140–58. British Museum Press, London, 1996.
- Van Den Brink, E. *The Pottery-Incised Serekh-Signs of Dynasties 0-1 Part II: Fragments and Additional Complete Vessels*. *Archéo-Nil*, 11: 23–100, 2001.

Reviewer

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