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Fine grain settling protocols for luminescence dating of museum objects sampled using the minimum extraction technique

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Abstract

This paper communicates a suitable laboratory protocol for settling the 4 – 11 µm fine grain fraction for OSL dating of archaeological samples from museum artefacts sampled using the minimum extraction technique. It provides a step-by-step methodology to facilitate adapting this protocol for use more broadly and demonstrates its ability to successfully isolate the fine grain fraction from tiny samples.

Keywords: fine grain settling, Stokes' Law, minimum extraction technique, ceramics, museum artefacts, optically stimulated luminescence

1. Introduction

The use of polymineral fine grains in luminescence dating has long been established and routinely used where a larger grain size fraction cannot be obtained. With fine grain dating, using typically 4 – 11 µm grains, the alpha component of the dose rate, with a range of 20 µm, irradiates the entire grain, so there is no need to remove an alpha attenuation layer (Wintle 1997; p. 770). Of course, including the alpha component to the dose rate requires estimation of the alpha efficiency, which will vary depending on the composition of the sample.

The fine grain method was first described by Zimmerman (Zimmerman, 1967, 1971), although subsequent alterations have been made to the Zimmerman method (e.g. Frechen et al. 1996; Fleming 1975; Fleming 1979, p. 58-59). While the method has both its pros and cons (a full discussion of this topic is beyond the scope of this paper), it is still very useful in a wide range of applications in luminescence dating today, particularly in archaeological settings (e.g. Anderson & Feathers 2019; Feathers 2009; Janz et al. 2015; Zink & Porto 2005).

The minimum extraction technique (MET) and extended MET sampling protocols are used to optimize OSL dating of archaeological artefacts housed in museum collections by extracting minute sample sizes (Hood & Schwenninger 2015; Hood 2022). MET protocols generally yield a sufficient quantity of fine grains for dating; however, as is to be expected, the process for isolating the 4 – 11 µm grain size fraction from the MET sample necessitates an adapted protocol compared to more standardised fine grain protocols, owing to the tiny sample sizes being worked with. Below, this adapted methodology, using the principle of Stokes' Law, is presented.[1]

2. Methodology: Fine grain sample preparation

Samples extracted using the (extended) MET protocol are in the form of drilled ceramic powder. A four-step process, as set out below, allows accurate isolation of the 4 – 11 µm fine grain fraction from the bulk MET sample.

Step 1: Dry sieving. When the MET sample is initially sieved (using small mesh or electro-formed hand held sieves[2]) to extract the coarse grain fraction, the < 90 µm grain size fraction is retained for the fine grain technique.[3]

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[1] It is also possible to sieve out the polymineral fine grain fraction of a MET sample using size specific electro-formed sieves, however the cost of such equipment and the labour-intensive cleaning process can be a deterrence.

[2] An electro-formed sieve is created by electro-deposition of a metal that allows precision construction of very fine sieves.

[3] It should be noted that other fine grain methodologies frequently incorporate an H_2O_2 and/or HCl wash into their fine grain preparation. However this has, to date, been avoided for MET samples owing to the extremely

Step 2: First settling. To isolate the 4 – 11 µm fraction from the < 90 µm fraction, it is necessary to settle the grains in a water column, using Stokes' law to determine the correct settling times for procuring this size fraction. The first settling step requires the removal of the ~ 12-90 µm grain size fraction using Stokes' Law (see, for example, Batchelor 2010, p.230-235). A calculation was made to determine the time it would take grains > 11 µm to settle in a solution of deionised water, based on the density and viscosity of the water, the temperature of the water, and the height of the graduated cylinder in which they were being settled.[4]

After adding the water to the cylinder, the MET sample is placed on a sheet of weighing paper, then rolled into a funnel so as to rapidly shoot the sample directly into the centre of the water column to avoid it adhering to the cylinder walls. A rapid movement of the sample into the water column is necessary to help break the surface tension of the water which can hold the MET sample owing to its extremely low mass. In the event of the sample staying on the surface of the water column, the surface tension can be broken by gently agitating it with a pin (or similar).

A 10 mL glass graduated cylinder is used for this first settling step owing to both its small size being the most appropriate for ease of dealing with MET-sized samples, and the glass facilitating the decanting of the sample.[5] Resulting settling times are presented in Table 1; the settling times presented in Table 1 and Table 2 are based on an average temperature of 19 °C; however, the ambient temperature of the deionised water was observed to fluctuate between 19 °C and 26 °C and thus the water used for each sample was measured prior to settling, and the exact temperature was used to calculate settling times).

Cylinder volume	Cylinder height	Drop height (average)	Settling time
10 mL	8.6 cm	4.3 cm	6 min 46 sec

Table 1: Settling times (at 19 °C) to remove the 11 – 90 µm grain size fraction.

At the end of the allotted settling time, the water column now holds in suspension grains ≤ 11 µm, and the base of the cylinder holds the > 11 µm grains. The water must now carefully be decanted into an identically sized graduated cylinder, leaving the settled material (i.e. the > 11 µm material remaining at the bottom on the cylinder) in approximately 1 mm of water, ensuring that none of the settled material is transferred during the decanting process. Upon being decanted into the next cylinder, the second step should begin immediately.[6]

Step 3: Second settling. Once the water in which the ≤ 11 µm fraction is suspended is decanted into a glass 10 ml cylinder, the next step to isolate the 4 – 11 µm grains from the ≤ 3 µm fraction begins immediately. As we are now isolating the 4 – 11 µm grain size fraction, the settling times are adjusted accordingly (see Table 2). Once the settling time is complete, the water column (which now contains grains 3 µm or smaller in suspension) can be poured off and discarded, or kept according to preference. The material now in the base of the cylinder is the desired 4 – 11 µm grain size fraction, and can now be collected in Step 4.

Cylinder volume	Cylinder height	Drop height (average)	Settling time
10 mL	8.6 cm	4.3 cm	51 min 8 sec

Table 2: Settling times (at 19 °C) to remove the 4 – 11 µm grain size fraction.

Step 4: Preparation of liquid suspension. The material left in the base of the cylinder after Step 3, that is, the 4 – 11 µm grain size fraction, is now carefully washed into a small glass vial using acetone[7] and is then stoppered and kept in liquid suspension until fine grain aliquot preparation is carried out.

Upon the completion of the four steps above the sample is then ready to be mounted as fine grain aliquots. To make an aliquot, as with the coarse grain technique, a small aluminium disc 10 mm in diameter and 0.5 mm thick is used. The disc is placed in a small glass vial (measuring 12 mm in diameter and c. 38 mm in height, to facilitate subsequent removal of the aliquot). However, it is essential that the disc is lying flat on the base of the vial so that the fine grains cannot become trapped under the disc, which could affect the preheating of the aliquots when being measured. This can be achieved by placing ~ 0.25 mL of acetone in the vial first and then placing the aluminium disc on top of the acetone, as this creates a suction force, ensuring the disc remains at the bottom of the vial. The glass vial containing the sample

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small sample mass and the likelihood of sample loss if another treatment step is introduced at this stage. However, ultimately, this decision should be made based on the individual nature of the material one is working with, i.e. a large carbonate and/or organic component to the sample may necessitate employing these additional steps, even at the expense of precious sample loss.

[4] A selection of online calculators are available to facilitate the calculation of settling times.

[5] If the sample is irreplaceable, i.e. there is no ability to obtain more, it may be preferable to use new glass cylinders only in MET fine grain preparation, in order to avoid potential sample contamination which could occur during cleaning between samples since fine grain material is invisible to the naked eye and MET samples are usual a 'once-off' without the possibility of obtaining more material in the event of sample contamination. However, the environmental and financial impact must also be considered for such a decision, and it is perhaps better practice to only use a new cylinder on particularly valuable/irreplacable sample.

[6] The ~ 12 – 90 µm fraction can be washed into a glass vial and stored in liquid suspension if required.

[7] It is also possible, of course, to use acetone instead of distilled water in the aforementioned steps, and indeed there is an argument to be made for this facilitating drying and settling. However, owing to additional considerations (costs and additional chemical use), our preference has been to only use acetone in this final step. The minute amount of mixing between water and acetone in this step has, in our observation, not hindered aliquot preparation. Additionally, if acetone is being used instead of water in all steps, then the potential issue with surface tension noted above for distilled water is usually avoided.

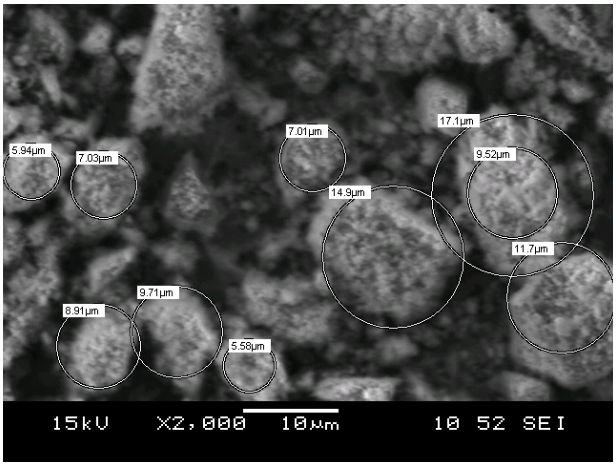

Figure 1: SEM image showing distribution of fine grain sizes for the brick test sample after settling to isolate the 4 – 11 µm fraction.

suspended in acetone prepared in Step 4 above is then gently agitated to ensure that all grains are once again in suspension (the vial should not be inverted so as not to trap fine grains around the lid) and, using a disposable pipette, 1 mL of liquid sample is added to the vial and is left to settle onto the disc.[8] This vial is then left open and placed in a fume cupboard so that the acetone can dry off. Once the acetone is evaporated, the disc can be removed carefully from the vial using tweezers. The aliquot will now have a thin monolayer of fine grains which have settled upon its surface during the evaporation process and is ready for measurement.[9]

3. Results

The fine grain preparation process was tested to ensure that it was accurately isolating the 4–11 µm grain size fraction. For a test sample, once aliquots were prepared a selection were examined under a scanning electron microscope (SEM) to quantify grain sizes.

SEM examinations showed that the described methodology is suitable for isolation of fine grains for OSL dating. The majority of grains observed were within the 4 – 11 µm grain size fraction, although with some slightly smaller (~ 3 µm) and some slightly larger (~ 12 – 17 µm) grains occurring too (Figure 1). Given that this slightly increased grain size distribution is inevitable due to the use of average drop heights and assumption of spherical grain geometry upon which Stokes' law is based, this variance is considered acceptable, and even the slightly larger grains are still smaller than the alpha range, i.e. 20 µm.

4. Conclusion

This paper has presented a step-by-step methodology to isolate fine grains for OSL measurement from samples obtained using the MET or extended MET protocol. It provides a means by which to maximise the quantity of measurable material from OSL dating when working with museum objects — for which only minute sample sizes can be obtained — by making use of the multi-mineral fine grain fraction as well as coarse grain mineral fractions. The methodology can be readily and easily implemented by other luminescence practitioners working with minute museum samples, or indeed wherever only small sample sizes are available for luminescence dating.

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[8] In standard fine grain aliquot preparation, it is necessary at this step to be very careful not to use too much liquid and thus allow too many fine grains to adhere to the disc. It is often also necessary, in standard methodology, to dilute the sample suspension. However, due to the very small amounts sampled during MET extraction, it has been observed that the quantity of fine grain material usually retained from a MET sample, when prepared in accordance with the steps outlined above, produces a good grain density for aliquot making (i.e. not too cloudy, that is, not too many grains present), which means it is not necessary to dilute the sample. However, as there can be a considerable amount of variation between different sample types, practitioners may still find dilution necessary for some samples.

[9] This monolayer should not be visible to the naked eye, and thus it is not necessary to attempt to add more grains via additional liquid suspension.

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Reviewer

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