Optimisation of preparation and measurement protocols for luminescence dating of small samples from a suite of porcelains and faiences

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As part of a wider study into the production and importation of porcelain and faience into Portugal, stimulated luminescence is being used to test typology-based chronologies for ceramics from various archaeological sites in Lisbon and Coimbra. To achieve this, procedures are being developed to facilitate luminescence testing of small samples (<100mg) from these different types of ceramic, following a standard method.

Conventional approaches to luminescence measurement, take small samples from weaker ceramic fabrics such as faience, and use powder obtained directly by drilling using a tungsten bit. Porcelain is compact and drilling or crushing can remove luminescence signals and alter a sample's behaviour, so it is conventionally measured in slices cut from a core drilled through the piece. Both approaches produce a polymineral sample and hence variations in luminescence behaviour depending on the sample's composition. A defined grain-size fraction may be separated from the powder by Stokes settling, but results from slices of porcelain could also be affected by differences in the doses absorbed by different grain sizes. Dating measurements are ideally conducted on specific mineral and grain-size fractions isolated from much larger samples (e.g. entire sherds ~20g).

In the present study cores have been drilled from the broken faces of sherds using hollow diamond tipped bits, and cleaned to remove any contamination. Initially, parts of some larger cores were coarsely crushed and unprocessed material was measured by TL using the Multiple Aliquot Additive Dose technique. Signal levels were used, but luminescence behaviour attributed to the presence of calcite was poor. Other crushed sub-samples were treated briefly in HCl and HF to remove carbonates and surfaces affected by crushing, and single aliquots were measured using a combined sequence designed to test the signal levels and behaviour of Predose and Regenerative TL, and Regenerative OSL signals. Material losses from this preparation were unacceptably high, but the Predose TL signal was identified as most commonly present at usable levels. Subsequent work focussed on the isolation of fine grains of quartz for Predose TL measurement. Cleaned cores were treated in 1M HCl for 4 days to weaken the ceramic by removing carbonates: this allowed both faience and porcelain to be disaggregated into powder. Predose TL signals from the residual silicates from porcelains were low but behaved well. Those from faiences were higher but subject to interference. After settling to isolate the >60 and <11 μm fractions, samples were treated in fluorosilicic acid. Sufficient quartz enriched material (~5 mg) has been obtained for absorbed dose determination on a number of aliquots for each sample, to allow absolute age estimation based on the average of the Predose TL results, combined with dose rates from INAA and a limited number of soil samples retained from the excavations.