

## Microscopy studies in support of dating of mortars of historical buildings

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There are several problems regarding the dating of the works in the historical buildings (see, for a review, Sanjurjo-Sánchez [1]). Some old constructions could be older than the written records of a region. Even for younger works there could be problems in the keeping of the records of the interventions, or there might be lack of records for whole lesser important buildings. Mortars are a potential material for dating of the built environment works, since they are ubiquitous and not reusable (contrary to what happen with other materials such as brick or stone). They are also heterogeneous materials composed of different components that are potentially separable. Among absolute dating methods, optical stimulated luminescence (OSL) dating is the only one that can be applied to mortars with and without lime and considering the age interval of mortars up to a few decades [1].

For OSL dating of mortars, the aggregate sand is the most suitable component but several issues needs to be considered, such as the bleaching of the signal in the studied phases (quartz or feldspar grains) before application on the considered building. The age estimated by luminescence is calculated as the ratio between the measured total absorbed dose (estimated as equivalent dose by luminescence) and the dose-rate of the ionizing radiation in the environment surrounding the dated material (minerals) [2]. The calculated age of the dated material is the time elapsed since the last exposure of the mineral grains to sunlight before being used in the mortar (afterwards the grains are protected from light and accumulate the absorbed dose due to surrounding radiation). It is necessary to measure the signals in several aliquots (composed of either several grains or a single grain) of the mortars minerals in each sample in order to assess the possible effect of inherited dose (referred as incomplete or partial bleaching due to insufficient exposure to daylight). The separation of grains from the mortar binder is a very time-consuming process. Presently are being developed efforts for the measurement of the signal in individual grains in planar samples without separation from the binder using confocal microscopy [3].

The mineralogical and textural characterization of mortar samples using optical microscopy and scanning electron microscopy could be useful for sample preparation and separation of minerals used for dating. These studies will also help in the characterization of the 3D heterogeneity of the dose rate surrounding the grains that are used for dating which is due to the heterogeneous distribution of radioactive elements (K, U, Th) in the mortar. Microscopy studies will help to identify mineral phases having these radioactive elements that contribute for the dose rate (feldspars, biotite, zircon, etc.) and might be used for elaborating element distribution maps. Microscopy studies will also help to assess the proportions in the mortar of the grains that will be used in dating as well as the presence of grainsize fractions, contributing to assess the suitability of the samples, the processing required and the possibility of performing analyses of several grainsize fractions. In general, it is advisable to use quartz

grains as its OSL geological signal (the inherited signal before use in the mortars) is easily effaced and it has a timestable signal. However, in some samples, as in the case of samples from historical works, the available amount of mortar sample (generally very restricted) combined with the lack of quartz in the samples, might imply that there few quartz grains available for dating. Alternatively, one can also use feldspar grains to achieve a higher number of measured aliquots, but feldspars show an unstable signal (referred as anomalous fading) and the geological, inherited, signal is not so easily effaced. The existence of different grainsize fractions should be considered in dating by OSL since, in the same conditions of surrounding radiation, smaller grains have a higher dose. Additionally, there could be differences in partial bleaching in different grainsize fractions. Stereomicroscopy observations can be used to control the number of grains in each aliquot in order to evaluate the possible effect of partial bleaching since a great number of grains will mask this effect.

In the case of lime mortars, historical mortar samples can also be dated by  $^{14}\text{C}$  analyses [4]. Petrographical studies will be crucial to select the samples and for the interpretation of results given the problems mostly related to the presence of inherited (unburned) limestone lumps (that will give older ages than those of the preparation of the mortar) and the recrystallisation of calcite in the buildings related to weathering processes (providing younger ages).

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