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Review

An Overview of the Use of Absolute Dating Techniques in Ancient Construction Materials

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Abstract: The reconstruction of the chronology of historical buildings is a tricky issue, as usually there are not historical documents that allow the assessment of construction phases, and some materials are hardly reliable for the use of dating techniques (e.g., stone). However, in the last two decades, important advances on the use of absolute dating methods on building materials have increased the possibilities of reconstructing building chronologies, although some advances are still scarcely known among archaeologists and architects. Recent studies performed on several kinds of mortars, fired bricks, mud-bricks, and even stone surfaces have shown that it is possible to date them. Both radiocarbon and luminescence dating have been the most frequently used techniques but others such as archaeomagnetism can also be used in some cases. This paper intends to give an overview of the recent achievements on the use of absolute dating techniques for building materials.

Keywords: absolute dating; mortar; brick; stone; building chronology

1. Introduction

Mud, wood, or rocks among others are typically naturally occurring materials used in ancient and historical buildings. Archaeological studies and physico-chemical analyses are usually performed on them to acquire information on construction materials and phases. One of the main goals in the archaeology of architecture is the reconstruction of the chronology of buildings. This is not difficult if written historical documents exist, but this is uncommon.

Researchers have used several dating techniques to acquire chronological information about buildings. Beyond the use of relative dating methods (e.g., building stratigraphy), absolute dating techniques are commonly used to obtain the chronology of construction phases through the analysis of some given building materials. Some instrumental techniques provide more or less accurate and precise numerical age ranges, but both precision and accuracy depends on the dating method used, the instrumental precision, certain properties of the analyzed material, and even the case study. Classically, organic materials and bricks have been dated by radiocarbon and luminescence, respectively, although some problems can be derived from the use of such materials and methods. However, recent developments in radiocarbon, luminescence, and other techniques (e.g., archaeomagnetism) have allowed the use of mortars and even stone for dating. The purpose of this work is to provide an updated overview of the state of the art on the use of absolute dating techniques on the different historical construction materials with special attention to the problems that can arise from each material and method.

2. Dating Wooden Materials

Wooden materials used in buildings can be dated with two methods: dendrochronology and radiocarbon. The first one provides more precise results (it is the most precise method), as it uses the growth rings of trees for dating the exact year of formation of the wood, but it requires timber of certain thicknesses and conditions. Dendrochronology is usually used to date timber, and researchers have built unbroken reference chronologies of rings to be used for dating that dates back to prehistoric times in Europe [1]. However, it can sometimes overestimate the age of a building structure due to two possible reasons: (i) The studied sequence of rings of a timber provides the age of a trunk core with old wood; and (ii) the timber has been reused, as usually occurs in wood used for roofing, and refurbished due to deterioration [2]. Radiocarbon is the alternative with advantages and disadvantages: It allows the dating of very scarce material (e.g., wood chips) but the obtained age is less precise [3]. The technique can also overestimate the age of a structure because of the same two reasons given above. In any case, the combined use of both techniques in wooden materials helps to recognize reused material and to identify construction phases [3].

3. Dating Bricks

Ceramic materials used in buildings are usually fired bricks, tiles, and less frequently pavements and pipes. Bricks have been usually dated by thermoluminescence (TL). This technique provides the time elapsed since the last firing of the material (at a temperature above ~300–400 °C) that usually corresponds to the moment of manufacture. The luminescence signal increases with time at a constant rate due to the naturally occurring radioactivity received by some minerals, typically quartz and feldspar inclusions in the ceramic matrix [4]. Heating the minerals (by TL) releases the trapped signal, allowing an estimate of the accumulated charge and the time elapsed since the brick was fired to be placed as part of a structure. The energy released per unit of time (year) from ionizing radiation on the dated material and the surrounding environment must also be estimated, and it is called the dose rate [4]. In ceramics and stony materials, there is a ubiquitous content in radioisotopes of the ^{238}U and ^{232}Th decay series and ^{40}K. In the last 20 years, there has been an important development of the so-called Optically Stimulated Luminescence (OSL) that has led to a preferential use of OSL for dating. OSL has been tested in bricks with success [5], providing some advantages over TL, as it requires few sample amounts and it provides ages with lower deviation. However, some problems can arise from the use of bricks for luminescence dating. The most frequent problems are three: (i) The exposure of bricks to high temperatures due to accidental fires occurred in the past can cause the "zeroing" of the luminescence signal and thus the underestimation of the real age; (ii) architectural modifications can distort the dose rate and subsequent age due to changes in the surrounding radioactivity received by the brick; and (iii) reused bricks will provide overestimated ages as the firing occurred some time before the reuse [5–8]. Luminescence dating can also be used in other ceramic materials such as tiles [9,10].

Sometimes radiocarbon can be used for dating ceramic materials although this still has not been tested on brick samples. Three cases allow the use radiocarbon for dating ceramics. Firstly, carbon derived from organic matter present or added to the original clay matrix can survive the firing temperatures if this temperature is not too high or very high temperatures are not reached in the brick core. This has been observed in ancient pottery [11,12]. Secondly, carbonates (e.g., calcite and shells) can be added to the raw clay paste. Shells can be used for dating [13], while calcite can be converted to carbon oxide during firing and combined with carbon dioxide to form calcium carbonate during cooling [14]; some old calcium carbonate have been found in this last case. Thirdly, soot and smoke can be adsorbed to the brick surface during firing under reducing conditions, being suitable for dating [15].

Based on the principle that the slow rehydroxylation process of clay fired in the past, Wilson *et al.* [16] proposed a new dating method for ceramics. This is based on the fact that ceramic materials can be returned to their initial as-fired condition by heating to a suitably high temperature. Wilson *et al.* [17] and Savage *et al.* [18] proposed that, by carrying out experiments of heating at 500 °C lasting up to a few weeks, both mass gain and expansion increase linearly with time ($t^{1/4}$). This effect will occur due to chemical recombination with environmental moisture. Wilson *et al.* [16,19] proposed that the measurement of mass gain kinetics, together with the assumed total mass gain since manufacture (obtained by reheating), provides an accurate self-calibrating method for ceramic dating. They also showed experimental accurate results on a series of specimens of a known age up to 2000 years. This method has been called rehydroxylation or mass-gain dating. However, later studies have found several problems. Shoval and Paz [20] tested the method in both calcareous and non-calcareous pottery samples of several archaeological periods and found that different factors influence the rehydroxylation rate leading to deviations in the mass-loss values in relation with the age. These factors are the clays used [21], the amount of calcite, the amount of coarse particles (temper), the firing temperature, and the burial or environmental conditions in the case of bricks. Moreover, Burakov and Nachasova [22] compared archaeomagnetic and mass-gain ages of old ceramics (including bricks) and found some factors causing deviations. Prior to the first proposal of this method, it was suggested that rehydroxylation is unlikely to be structurally identical to the original unheated mineral [23,24]. In fact, Cultrone *et al.* [25] remarked early some additional open questions regarding rehydroxylation such as the firing temperature ranges, the duration of firing (soaking time), and the effect of amorphous remnants and meta-clays in the rehydroxylation. Thus, more research is needed to ensure the use of this low-cost dating technique as an absolute dating method suitable for getting accurate and reproducible results.

Mud-bricks (non-fired bricks) have also been used in some parts of the world, even earlier than fired bricks. They have been used for dating buildings via radiocarbon from some organic components present in the mud matrix, such as charcoal, small wood fragments, or reeds. The problem of using small wood fragments and charcoal is similar in that these are derived from the use of radiocarbon for timber to date buildings [26], as has probably occurred in studies dating the Pyramids of Egypt [27]. The use of reeds seems to be the best option and has provided good results [26,28], as they are unlikely to be reused, but they are more hardly preserved. Recently, OSL dating has been tested on mud-bricks. Daylight bleaching can occur during the production of bricks (extraction, transport, and mixture of minerals) before molding. The OSL age of mud-bricks of Syrian sites were recently compared with radiocarbon and luminescence ages of the same sites with matching results [29–31]. In some cases, mud-bricks have also been dated by archaeomagnetism, as non-heated materials may also acquire an archaeologically relevant magnetization. Mud-bricks are wet clays placed into molds and dried by baking in the sun, acquiring a remanence at the time of molding known as shear or Shock Remanent Magnetization (SRM) [32], which is proportional to the ambient field. Thus, archaeomagnetic dating records the production of the mud-brick [33]. There are examples of successful [34] and unsuccessful uses of this method on mud-bricks [35].

4. Dating Mortars

Mortars (joint mortars, plasters, and renders) are probably the most studied historical building material by archaeometric techniques. They are an interesting source of data from past cultures and technologies: They provide data on the provenance of raw materials and the technology (crushing, sieving, mixtures, firing) of manufacture [36–38]. Different kinds of traditional mortars exist, mud, gypsum, and lime mortars being the most frequent. Lime mortars have been the most historically used worldwide and attempts to date them have been performed in the last 50 years by radiocarbon and later by luminescence.

Mortars can be dated by radiocarbon if organic components are present, although the obtained age does not always correspond to the mortar age. The possibility of dating the lime binder of mortars by radiocarbon has been extensively studied since 1964 [39,40]. The lime binder ($CaCO_3$) is formed due to the slaking with water of the quicklime (CaO obtained by firing crushed limestone to 900 °C) and the further reaction of atmospheric CO_2 as the mortar hardens [41]. However, some problems have arisen after intensive empirical work. One of them is the mortar content in old limestone that appears as lumps within the binder [37]. Lumps are incompletely burned limestone fragments, being composed of old carbon that leads to age overestimation [41,42]. Moreover, crushed limestone is sometimes used as aggregate, adding more fossil carbon that is difficult to remove from the binder [43]. Additionally, the mortar hardening occurs at rates that range from years to decades [44,45] and low hardening rates will yield radiocarbon ages younger than the mortar age. Moreover, several decay processes usually affect the lime as a result of water present in the built environment (e.g., rainwater and rising dump) causing dissolution and re-crystallization of $CaCO_3$ [46], being the younger carbon incorporated into the mortar lime and resulting in age underestimation [41].

Several approaches have been tested to separate the old lime from the binder for radiocarbon dating. Previous analyses are necessary to separate them (e.g., microscopic observations), and several procedures have been proposed for separation—most of them by mechanical processes. Nawrocka *et al.* [47,48] carefully crushed and repeatedly froze and thawed mortar samples to separate the lime binder, based on the fact that limestone fragments in the aggregate are stronger than the more porous lime binder. They found reliable ages in some cases. Marzaioli *et al.* [49] found good results by the procedure described by Nawrocka *et al.* [47] for laboratory produced mortars with quicklime obtained at different firing temperatures. More recently, Ortega *et al.* [50] combined mechanical and physical procedures to extract lime particles of less than 1 μm in size. They obtained consistent ages, although this procedure still should be tested in other cases. Lindroos *et al.* [51] proposed previous cathodoluminescence (CL) and Mass Spectrometry (MS) studies to identify and characterize carbonates. They dissolved the lime in phosphoric acid, collecting successive CO_2 increments to acquire a CO_2 evolution pressure curve and a ^{14}C age, and stable isotope profiles of successive dissolution increments to interpret radiocarbon profiles, identify contaminated carbonates and show possible dating errors due to contamination. This method, combined with mechanical separation and age control, provides an important percentage of reliable ages (around 80%) in a further study of Heinemeier *et al.* [52], but the method is time-consuming and expensive.

Luminescence dating of some mortars has been tested, providing good results in the last 15 years. Luminescence analyses are usually carried out on the mortar quartz of the aggregate sand as the grains are exposed to daylight during the mortar manufacture (extraction, transport, and mixture of sand with the binder), zeroing the geological luminescence signal. In the earliest studies, Botter-Jensen *et al.* [53] used OSL in joint mortar samples of a modern building and found that at least some quartz grains were well bleached during mortar manufacture, with dating being possible. Later, Zacharias *et al.* [54] provided the first mortar OSL age, introducing a new way to date buildings. The most important problems of OSL dating of mortars are the assessment of the burial equivalent dose (accumulated charge), the luminescence sensitivity of quartz grains, and any residual dose remaining due to incomplete zeroing (due to a poor daylight exposure). The latter causes age overestimation, and it has been regarded as particularly problematic in acquiring reliable age estimates. Goedicke [55] studied the partial bleaching of old mortars and concluded that methods of sand extraction, transport from gravel pits, and mortar preparation remained virtually unchanged for centuries due to the use of small tools, although, due to the bulk amounts of transported sand, not all grains could have been sufficiently exposed to daylight to bleach the geological signal. Thus, he recommended the use of statistical tools to choose the most suitable method to assess the degree of zeroing. Jain *et al.* [56] investigated the OSL of different mortar samples, including render, whitewash, and an inner plaster. They found an accurate dose-depth curve in the walls of the building, but also poorly bleached and weakly sensitive quartz, resulting in the poor precision of ages [57]. They also suggested the use of coarser quartz, as this is better when bleached, although later works found reliable results with fine grains [58]. More recently, Goedicke [59] predicted the reliability of dating mortars and assessed the level of bleaching of quartz for mortar samples, while more recent attempts have found very good results for Roman and medieval mortars [58,60].

Other mortars have been shortly investigated. Portland cements have been used in buildings since the nineteenth century. Goksu *et al.* [61] used both coarse quartz and polymineral fine grains of three types of Portland cement for retrospective dosimetry and found problems achieving pure quartz and feldspar and insufficient zeroing. However, Thomsen *et al.* [62] obtained low equivalent doses on quartz grains of commercial concrete, proving that OSL can be used to date young cement samples that are a few decades old. Roman concrete (*Opus caementicium*) was also tested for OSL with partial success [59]. In addition, mud mortars were studied by OSL by Feathers *et al.* [63]. They dated mortars collected from an early Andean monumental in Peru and obtained reliable results for 14 samples.

An alternative method proposed for dating mortars is archaeomagnetism. As occurs with mud-bricks, theoretically, mortars used for murals and cements can also acquire a stable magnetization if they have ferromagnetic minerals. In such cases, they could be dated by archaeomagnetism [33]. In a wet mass, magnetic particles can align themselves with the ambient magnetic field and become fixed in this position when the paint or cement dries. In paints, this is known as pictorial remanent magnetization (PiRM) [64]. It is the same mechanism that occurs in underwater sediments, known as a post-depositional remanent magnetization (pDRM). In such cases, the method will date the application of the paint or mortar, although this possibility has only been used for studying the archaeomagnetic fields and intensities in ancient murals and cements; it still has not been used for dating [64].

5. Dating Stone Surfaces

In the last 20 years, some luminescence tests have attempted to date stones surfaces. The purpose of such tests has been dating the moment when stones were incorporated into building structures. Firstly, Liritzis [65] successfully dated the moment when a rock surface was exposed to daylight for the last time in carved megalithic stone buildings. The use of stones as building materials requires extraction from the quarry, cutting, transport, and setting the blocks in structures. During such processes, the daylight zeroes the luminescence signal up to a certain depth. During the construction of a building, most stone surfaces are placed in structures remaining shielded from daylight, with the exception of the outer surface in a wall. Thus, luminescence can be used for dating these events. The method has been tested on magmatic (e.g., granite and basalts), sedimentary (e.g., limestone and sandstone), and metamorphic (e.g., quartzite and marble) rocks [66–72].

Carefully sampling and processing have been regarded as very important for surface dating [69,70,73]. In addition, the dose rate must be carefully assessed, combining several methods if possible [69–71,73]. Both TL and OSL have been used for this approach on quartz, polymineral grains, and thin slices that cut out of the surface of the studied stones (with a special method called high resolution OSL, HR-OSL [74]). Recently, Sohbati *et al.* [72] and Chapot *et al.* [75] proposed and successfully tested a novel approach for dating stone surfaces. The approach enables the dating of stone surfaces not shielded from daylight. In a new surface exposed to daylight, the zeroing occurs at different rates depending on the depth from the surface (shallower grains bleach at higher rates). In this method, the luminescence signal is measured at several depths, up to a depth at which no zeroing occurred [72,75]. Thus, the remaining signal at any depth depends on the time of exposure of the studied rock surface. The error of surface dating usually ranges from 5% to 20% [76], being thus a promising technique for dating buildings in the future.

6. Conclusions

Studying the chronology of historical buildings is a difficult task, as written historical documents are usually scarce or imprecise. Some absolute dating methods have been used on building materials as an alternative to reconstruct building histories. They provide a large number of options to know the age of a building, although some care is needed in choosing the dating methods and the target materials. In the last 20 years, important developments have allowed the dating of materials previously considered unsuitable for this purpose. In some cases, radiocarbon can be used for dating fired or unfired bricks (mud-bricks), luminescence being the most used method for these materials. Recent developments on luminescence dating enable its use for dating almost all kinds of mortars (e.g., lime, mud, and concrete), while radiocarbon can be used in lime mortars or mortars containing organic matter. Luminescence dating procedures have also been developed to date the stone surfaces of buildings, which can be useful in the case of buildings in which other methods cannot be applied or have been unsuccessful. Other methods, such as archaeomagnetism have also been used for mud-bricks, and it could probably be applied to mortars in the future. Finally, the new rehydroxylation dating method has been tested, with reliable results in some cases, but still must be studied in depth to use it as a dating tool without independent age cross-check. With the combination of such methods and an appropriate collaborative work among specialist in dating techniques and archaeologists, it should be possible to date most, if not all, of the buildings and structures in the future.

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