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# Physical and mineralogical properties of experimentally heated chaff-tempered mud bricks: Implications for reconstruction of environmental factors influencing the appearance of mud bricks in archaeological conflagration events

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ABSTRACT

Sun-dried mud bricks are used around the world and have been found in the Levant and Mesopotamia since the Neolithic period. Their form and composition lend important information pertaining to social and technological meaning in human cultures. Fired mud bricks are well known in the southern Levant, often identified in Bronze and Iron Age strata and used as a marker for destruction by fire (conflagration events). Only a few studies have attempted to reconstruct conflagration conditions from fired mud bricks because many variables impact the formation of the final fired brick. These include brick composition, heat intensity (i.e., maximum temperature), heat duration and firing atmosphere (i.e., oxidizing vs. reducing). The myriad combinations of these factors may result in different appearance of fired bricks. Infrared spectroscopy is one method that has been exploited quite extensively in relation to fired clay-based materials: studies were conducted on powdered sediment samples for a fixed duration and in oxidizing conditions, producing calibration curves that were then utilized for reconstruction of past maximal heat. Here we report on an experimental study of the thermal behavior of mud bricks under differing composition, heat intensity, heat duration and firing atmosphere. We carried out experiments in a furnace oven using micro-thermocouples which allowed us to simultaneously measure heat across bricks, from edge to core. The resulting mud bricks were analyzed using Fourier Transform Infrared (FTIR) spectroscopy. We identify a previously unknown thermal effect that occurs in bricks tempered with organic material while they are fired; namely a correlation between the amount of organic temper and elevation of temperatures up to 100 °C above the oven chamber temperature. We record the color patterns obtained at different temperatures and duration of heating, as well as the colors obtained from heating in different atmospheres. We report that the FTIR spectrum of bricks heated in oxidizing conditions differs from that of bricks heated in reducing conditions at the same temperature. We note that the position of the main clay absorbance band cannot be used alone to infer firing temperature as its shift is not systematic. We show that combining this parameter with the width of the same band in the FTIR spectrum makes it possible to achieve better temperature reconstructions from fired bricks. Lastly, we report a small scale case study in which we tested the applicability of the experimental results to the remains of a mud brick wall unearthed within the largest known destruction event in the ancient city of Megiddo, i.e., Tel Megiddo Stratum VIA. We show that this wall was burnt as one unit, having a reduced core and oxidized outer part, where the core experienced temperatures in the range of 500–600 °C and the edge 600–700 °C. The detailed analysis of brick compositions carried out in this study further allows us to reconstruct the ancient bricks' preparation recipe. The results of this study bear important implications for future studies of archaeological conflagration events, and the destruction phenomenon in general.


## 1. Introduction

Mud bricks are common building materials used since the Neolithic in Eurasia. These artificially shaped and manipulated sediment blocks hold a wealth of information that can be retrieved through archaeological research. First and foremost, identification of intact mud brick walls

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makes it possible to reconstruct architectural units and thus site structure (or city/town plan; e.g., at Çatalhöyük, Mellart, 1975). Having a clear plan of urban space may further be used to infer social, political, economic and gender-related issues. More information can be retrieved from the bricks themselves, mostly through the study of their mineralogical and elemental compositions. By so doing researchers have been able to identify the sources for brick material across the landscape in relation to the studied sites, which further informs about human use of the landscape, soil/sediment procurement and its influence on human–environment interactions (e.g., Goldberg, 1979; Nodarou et al., 2008). Identifying the source materials also enables discussion of organization of labor and construction methods (e.g., Homsher, 2012; Emery & Morgenstein, 2007). Compositional similarities and differences among bricks have also been used to argue that mud bricks are useful for the identification of production choices, which in turn reflect cultural and ideological choices, and even act as means for symbolic communication (Love, 2012, 2013). Recently it was also demonstrated that the study of the degradation products of mud bricks provides important methodological insight which pertains to the formation of stratigraphy and microstratigraphy, the identification of roofs and floors, and reconstruction of activity areas (Friesem et al., 2011; 2014a; 2014b).

Many of the studies related to mud bricks deal mostly with sun-dried bricks. Excavators over the years also noted the presence of fired mud bricks, yet relatively few studies explored these in detail. In most cases the presence of fired bricks has been used to infer conflagration events. Differences in color of fired mud bricks have been noted by many, however only a few researchers designed experiments aiming to understand the meaning of such color differences (e.g., Stevanović, 1997). Experimental results indicated that the effect of fire on mud brick color depends on many variables, including differences in brick composition, heat intensity, heat duration, and availability of oxygen (e.g., Maritan et al., 2006; Twiss et al., 2008; Love, 2012). It appears that this experimental complexity hampered continued research into burnt mud bricks. However, extracting information from burnt mud bricks on heat intensity, duration and atmosphere is expected to open up a window into very important social, cultural and historical questions. It may be useful for reconstruction of heat regime during conflagration events which may allow for differentiation between intentional and accidental fire. It may be helpful in distinguishing between types of destruction events, which in turn may affect historical interpretation. It is therefore important to study the effect of heat on mud bricks.

Clay minerals are a basic component in mud bricks. As in firing of pottery, fired bricks also become consolidated and hardened, which makes fired bricks preserve better than unfired bricks. Clay minerals are known from previous studies to be effective recorders of firing temperature. For example, in heat-related mud-based industrial/domestic installations such as ovens, furnaces, and where metallurgical activities took place, researchers were able to reconstruct use temperatures through infrared spectroscopy (e.g., Eliyahu-Behar et al., 2012; Gur-Arieh et al., 2013). The same approach was also deployed in pottery studies (e.g., Shoval et al., 2011). These studies showed that infrared spectroscopy enables to distinguish between heated and unheated clay, and even determine rough estimates of temperature ranges (Berna et al., 2007; Eliyahu-Behar et al., 2012; Friesem et al., 2014a; Gur-Arieh et al., 2013). We note that thermoluminescence is also a technique suitable for reconstruction of temperature from heated clay-based materials, but relative to infrared spectroscopy it is more time consuming and expensive. It is reasonable to assume that infrared spectroscopy will be useful in determining firing temperature of mud bricks, yet previous studies utilizing infrared spectroscopy did not test the effects of heat intensity, duration and atmosphere on clay-based materials.

The overall aim of the current study is to build a methodological framework that will allow reconstruction of the parameters that result in what we identify in excavations as bricks of varying colors. We will test: (1) the thermal behavior of bricks as they are heated under controlled laboratory conditions, with varying compositions, heat intensity, heat duration and availability of oxygen, and (2) the effect of the above changing factors on brick color and its clay infrared spectrum.

### *1.1. Background to the study of mud bricks in conflagration events in the southern Levant*

Destructive conflagration events are documented in the archaeological record mainly since the Neolithic (Stevanović, 1997; Twiss et al., 2008). In the southern Levant, the number and magnitude of destructions by fire are pronounced in the Bronze and Iron Ages, ca. 3rd to 1st millennia BCE. Some of the massive destruction events have been used as chronological anchors across the region and serve for interpretations of the history of the southern Levant, mostly within the realm of biblical archaeology. Despite the abundance of reported destruction levels in many sites in the southern Levant, there is relatively little deliberation on methodological aspects of this phenomenon (Dever, 1992; Finkelstein, 2009).

Field identification of destruction events in Levantine Bronze and Iron Age sites relies primarily on macroscopic criteria such as presence of smashed vessels (rather than pottery sherds) on floors covered by ash, sometimes including charred materials, and sometimes overlain by accumulation of stone and/or mud brick collapse (Finkelstein, 2009). Differences in colors of mud bricks are often interpreted as reflecting exposure to different heat regimes, though exact temperatures cannot be determined based on color alone.

Utilization of microscopic, molecular and elemental methods to the study of heat intensity in Levantine Bronze and Iron Age destruction events was thus far conducted in two Iron Age contexts, employing Fourier Transform Infrared (FTIR) spectroscopy. The effect of heat on the FTIR spectrum of clay minerals has been extensively explored (Farmer, 1974; Shoval et al., 1991, 2011; Berna et al., 2007; Savage et al., 2008; Clegg et al., 2012). It has been shown that reversible and irreversible structural changes with heat occur in clay minerals, producing significant spectral changes that allow identification of heating temperature. These spectral changes are stable for long periods of time as shown by Shoval et al. (1991; 2011) who found that re-hydroxylation in ca. 3000 years old fired pottery is only partial (apparent by the presence of an OH band at 3620 $cm^{-1}$, while the OH band at 3690 $cm^{-1}$ is absent). Similar observations were made by Berna et al. (2007), Eliyahu-Behar et al. (2012), Gur-Arieh et al. (2013, 2014) and Friesem et al. (2014a) who worked in various locations in Israel, Uzbekistan and Greece, where different types of soils and sediments occur. They all point to the same spectral changes in heated clay minerals, and show that these spectral changes are preserved for thousands of years. It is therefore concluded that despite partial rehydroxylation, it is possible to use FTIR spectra of clay minerals in order to deduce past firing temperatures.

In a study of sediments associated with destruction, Berna et al. (2007) show a temperature gradient (ranging between 1000 and 500 °C) associated with a mass of collapsed burnt mud bricks in Tel Dor, dating to the late Iron Age I. They studied this destruction event two-dimensionally, on an excavation section. Their study relied heavily on the infrared analysis of clay minerals and included an experimental calibration of the effect of heat on the infrared spectrum of clay minerals. They found that the highest recorded temperatures within the brick pile – in excess of 1000 °C – corresponded to yellowish-white sediments in the center of the pile, and that lower temperatures – ranging between 500 and 1000 °C – corresponded to red, brown and gray colored sediments (Fig. 8 in Berna et al., 2007). Namdar et al. (2011) studied an Iron IIA destruction level at Tell es-Safi/Gath where they conducted detailed mapping of mineralogical and heat intensity changes across space, in three dimensions in one excavation square (5 × 5 m). They identified that the most intense heat was associated with the roof