



## Evaluation of heat treated clay for potential use in intervention mortars

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### ABSTRACT

In this study, raw material compositions, basic physical, mineralogical, microstructural and hydraulic properties of lime mortars used in two selected historic buildings were determined by XRD, SEM-EDS and TGA analyses. The results showed that the mortars were hydraulic due to the use of pozzolanic aggregates. Taking into account the hydraulic characteristics of mortars due to the use of pozzolanic aggregates, the possibility of obtaining hydraulic mortars by using pozzolanic aggregates produced from heated commercial clays was investigated. For this purpose, four clay samples used in the ceramic industry in Turkey were heated at varying temperatures of 400, 450, 500, 550, 600, 800, and 1200 °C with a heating rate of 10 °C/min. Pozzolanic properties of heated clay samples were determined. The results showed that commercial clays studied are well suited for use as pozzolanic aggregates when they are heated between 500 and 700 °C. This is also confirmed by testing the compressive strengths of the three month aged laboratory-produced mortars that contained thermally treated clay (at 600 °C) as pozzolanic aggregates. Compressive strength of this mortar was around 5 MPa which is satisfactorily high.

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### 1. Introduction

Mortars, produced by mixing binder and aggregates, have been used for bedding, jointing and rendering brickwork and stonework. The oldest mortar used for building was mud, and is still used in many countries throughout the world. In ancient Egypt, mud mortar was used with sun-dried bricks and later gypsum was used as binder in the brick vaults of monumental buildings (Davey, 1961). Lime mortars were commonly used in buildings from Greek times through the beginning of the 19th century (Vendrell-Saz et al., 1996). They are primarily composed of lime as binder and aggregate as filler material.

Lime is produced by calcination of limestone to convert carbonate into oxide (quicklime). This oxide when mixed with water transforms into the hydroxide of calcium. Aggregates generally comprise a large part of the volume of lime mortar and the characteristics of the aggregates chosen are critical for the performance of the mortar. Aggregates can be classified as inert aggregates and pozzolanic aggregates. Inert aggregates do not react with lime. However, the pozzolanic aggregates are active and react with lime in the presence of water (Davey, 1961). Pozzolanic aggregates may be divided into two separate groups; those consisting of natural pozzolans and those of artificial pozzolans (Lea, 1940; Cowper, 2000; Moropoulou et al., 2004).

Natural pozzolans include such materials as some diatomaceous earths, opaline, cherts, shales, tuffs and volcanic ashes. The artificial pozzolans are mainly products obtained by the treatment of natural materials such as clays, shales and fly-ash (Lea, 1940). The most common artificial pozzolan is obtained by the heat treatment of clay. The characteristics of clay are therefore important to have an idea about its pozzolanic character. The loss of combined water in the structure of clay leads to destruction of the crystal structure after thermal treatment. The silica and alumina transform into an amorphous state (Baronia and Binda, 1997; Charola and Henriques, 1999). When they are mixed with lime and water, they can produce pozzolanic reactions. Therefore, the determination of the temperature range where the clay turns into an unstable amorphous state is very important.

Determination of lime mortar characteristics for conservation works of historic buildings became an important task in the second half of the 20th century due to the extensive damage of cement mortars used in historic buildings (Rodriguez-Navarro et al., 1998). Historic buildings should be conserved by original materials and intervention materials used in the restoration must be compatible with original ones. Hence, the characterization of building materials plays an important role in restoration works of historic buildings.

The aim of this work is the characterization of aggregates that were used in historic lime mortars collected from Çukur Hamam and Hacemescidi, and according to the results, to determine the characteristic of clay samples which will be used as pozzolanic aggregate for the production of intervention mortars that are compatible with the original ones.

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## 2. Experimental procedure

### 2.1. Characterization of lime mortars and their aggregates

In this study, lime mortars were collected from two buildings which were both constructed in 14th century. These buildings are known as Çukur Hamam (Ç) and Hacet Mescidi (H) and are located in the city of Manisa in western Turkey (Fig. 1). Table 1 shows codes and locations of all samples. Basic physical properties, microstructural features, mineralogical and chemical compositions of the mortars were determined by XRD and SEM-EDS. TGA analysis was done in a separate study by one of the authors (Budak, 2005).

Bulk density and porosity of the mortars were determined by measuring the dry, water saturated under vacuum, and hydrostatic weights of samples (RILEM, 1980). Lime and aggregate ratios of mortars were determined by dissolving the carbonated lime in mortars with dilute hydrochloric (HCl) acid (Jedrzejewska, 1981; Middendorf and Knöfel, 1990).

Pozzolanic activities of the aggregates (less than 53 µm size) were determined by measuring the differences in electrical conductivities (mS/cm) before and after addition of the samples into saturated calcium hydroxide solution (Luxan et al., 1989). The mineralogical compositions, microstructures and chemical compositions of aggregates were determined by XRD and SEM-EDS analyses. Philips X-Pert Pro X-Ray Diffractometer (CuK $\alpha$  radiation) in the range of 2–70° and Philips XL 30S FEG Scanning Electron

**Table 1**

Identification codes of collected samples and their description.

Identification codes	Description
Ç-M-BB1	Brick masonry mortar (dome starting point)
Ç-M-BB2	Brick masonry mortar (dome)
Ç-M-SS	Stone masonry mortar (entrance door)
Ç-M-BB3	Brick masonry mortar (dome)
H-M-BB	Brick masonry mortar (dome)
H-M-SS	Stone masonry mortar (wall)

Ç: Çukur Hamam (bath), H: Hacet Mescidi (Mosque), BB: mortar samples from two layers of brick, SS: mortar sample from two layers of stones and M: mortar.

**Table 2**

The commercial codes of clay samples.

Codes	K-31	K-103	K-244	K-261
Source	Tamsa Seramik A.Ş	Yüksel Seramik A.Ş	Kalemaden A.Ş	Kalemaden A.Ş

Microscope (SEM) coupled with X-Ray Energy Dispersive System (EDS) were used in the analyses (Table 2).

### 2.2. Heat treatment of commercial clays

In this study, the possibilities of producing pozzolanic aggregates by calcination of some commercial clays were investigated. For this



Fig. 1. Map of Turkey showing the location of Manisa.

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