Cement and Concrete Research 64 (2014) 17–29

Contents lists available at ScienceDirect

Cement and Concrete Research

journal homepage: http://ees.elsevier.com/CEMCON/default.asp

Comprehensive study on mechanical properties of lime-based pastes with additions of metakaolin and brick dust

V. Nežerka [a,*], Z. Slížková [b], P. Tesárek [a], T. Plachý [a], D. Frankeová [b], V. Petráňová [b]

[a] Faculty of Civil Engineering, Czech Technical University in Prague, Thákurova 7, 166 29 Praha 6, Czech Republic
[b] Institute of Theoretical and Applied Mechanics AS CR, Prosecká 76, 190 00 Praha 9, Czech Republic

ARTICLE INFO

Article history:
Received 21 March 2013
Accepted 12 June 2014
Available online 4 July 2014

Keywords:
Microstructure (B)
Mechanical properties (C)
CaO (D)
Metakaolin (D)
Brick dust

ABSTRACT

In order to understand the behavior of lime-based mortars, and to identify the influence of pozzolans, it is important to investigate the microstructure and properties of lime-based pastes without any aggregates. In our work, nine different sets of pastes with additions of metakaolin and brick dust were studied. The chemical composition and microstructure were investigated by means of TGA, SEM–BSE microscopy and EDX analysis. The mechanical strength and fracture properties were determined from destructive tests, while the evolution of the Young's modulus was monitored using the resonance method. The study revealed that metakaolin exhibits much stronger pozzolanic activity than brick dust, and that the mechanical properties of pastes are not necessarily enhanced by the addition of pozzolans. However, the shrinkage reduction should result in the elimination of cracking around aggregates in mortars. These information contribute to better understanding of lime-based mortars and are essential for their proper modeling and design.

1. Introduction

The inconveniences involved in the use of lime as a binder (e.g. slow setting and carbonation, high shrinkage and low strength [1]) have been overcome in the last decades by the use of Portland cement. However, the negative effects of the Portland cement in the restoration of architectural heritage, such as low plasticity, excessive brittleness and early stiffness gain [2–5], together with the high content of soluble salts leaching over time, high thermal conductivity and open porosity (as demonstrated on grouting mortars [6]), have forced the restorers to exploit the lime-based mortars again. Moreover, many authorities have criticized the cement-based mortars for their esthetic incompatibility with the old masonry or old archeological surfaces [7].

Today's commercial limes are very pure, even though the regulating standard EN 459-1 is not very strict — the mass of CaO and MgO in the commonly used CL-90 lime hydrate should be higher than 90%. The presence of impurities in historic lime mortars was not usually harmful [8], the content of silica (SiO_2) and alumina (Al_2O_3) was mainly responsible for their hydraulic character. The reaction between lime and SiO_2 and Al_2O_3 leads to the formation of calcium silicates and aluminates [9]. The contemporary high-purity limes without any additives lack the required durability, strength and suffer from an enormous shrinkage. Therefore, the minerals have to be added to lime in the form of pozzolans — in our study we used metakaolin and finely crushed bricks.

Pozzolans, e.g. volcanic ash or crushed bricks, have been used since ancient times in combination with lime to improve the moisture resistance of mortars, resulting in freeze–thaw resistance [10], and also to increase their mechanical strength [11,12] and durability [1,5]. Mortars with the addition of pozzolans should be able to harden in a high relative humidity (they are called *hydraulic*) or when the access of CO_2 is limited as in the case of mortars supporting glazed tiles [5]. Crushed ceramic material from tiles, bricks and pottery was added to lime-based mortars during the Byzantine period [4], and crushed bricks in the joints of load-bearing masonry were extensively used in the Roman Empire as the pozzolanic material where no volcanic material was available [13]. Such mortars were also preferred from the early Hellenistic up to the Ottoman period in water-retaining structures to protect the walls from moisture, typically in baths, canals and aqueducts [14,15]. While fine brick particles were mainly used for rendering, larger crushed brick particles appeared mainly in masonry walls, arches and foundations [16]. The mortars containing crushed bricks were known as *Cocciopesto* in Roman times [19], *Horasan* in Turkey [17], *Surkhi* in India, and *Homra* in Arabic countries [18].

In recent years, the understanding of cement-based materials has been significantly promoted by their micromechanical modeling. Analytical and numerical homogenization have been used for stiffness prediction of alkali-activated pastes [20], upscaling the compressive

* Corresponding author.
E-mail addresses: vaclav.nezerka@fsv.cvut.cz (V. Nežerka), slizkova@itam.cas.cz (Z. Slížková).

http://dx.doi.org/10.1016/j.cemconres.2014.06.006
0008-8846/© 2014 Elsevier Ltd. All rights reserved.

strength of Portland cement mortars [21], or multi-scale simulations of three-point bending tests of concrete specimens [22]. Based on their work, a similar approach for the lime-based mortars was proposed by Nežerka et al. [23,24]. The last studies revealed that a realistic model of lime-based composites needs to incorporate the properties of pure paste and processes taking place in the matrix, such as shrinkage.

Despite a considerable amount of literature devoted to the lime-based mortars, the authors are not aware of any work dealing with pure lime pastes — the available studies usually focus on specific properties of mortars containing aggregates. In particular, they deal with their use and performance in ancient structures [16,19,25], their chemical composition [9,26] and influence of pozzolans [1,5,4,13], morphology of the individual phases [1,27–29], porosity [30–32], or mechanical properties [5,8,9,33]. The aim of this paper is to fill the gap by performing a systematic study on lime-based pastes, with the emphasis on their microstructure, porosity and mechanical properties. Information about the individual components used for the preparation of pastes can be found in Section 2. Microscopy images in Section 3.5, complemented by SEM–EDX elemental analysis, illustrate the morphology and reactivity of individual components. The development of stiffness, expressed by means of the dynamic Young's modulus, is presented in Section 4.1. Data on the compressive strength of individual mixes are contained in Section 4.2, followed by the results of tensile testing (Section 4.3) and information about the fracture properties in Section 4.4.

2. Materials

To make our study useful for practical applications, we used only materials commonly available in the Czech Republic, and similar to the materials that can be found within European market. Their chemical composition was determined by means of EDXRF analysis (Table 1) using Oxford Instruments X-Supreme 8000. The particle size distribution (Fig. 1) was measured using a Laser analyzer CILAS 920, by which the materials containing particles of a diameter ranging from 0.3 to 400 μm can be analyzed. Measurement of the specific surface area was done by the gas adsorption, BET method, using the device Micromeritics ASAP 2020. Moreover, metakaolin and crushed bricks were characterized in terms of their pozzolanic activity, which indicates the ability of pozzolans to react with lime (see Section 3.1).

2.1. Lime

In our study, white air-slaked lime (CL90) Čertovy schody, Czech Republic, of a great purity (98.98% of CaO + MgO) was used. The chemical composition is summarized in Table 1 and the grading curve is plotted in Fig. 1. The most frequent particle diameter found in lime was 15 μm, diameter at 50% was equal to 13 μm and diameter at 90% to 38 μm. The specific surface area of lime used in the present study was 16.5 m^2/g.

Table 1
Chemical composition of used materials.

Component	Amount [% by weight] (±0.5%)		
	Lime	Metakaolin	Bricks
CaO	97.8	0.17	3.22
MgO	1.16	0.75	2.69
SiO_2	0.65	52.1	54.1
Al_2O_3	0.00	43.4	25.3
K_2O	0.12	0.66	4.11
Fe_2O_3	0.10	0.85	8.03
TiO_2	0.00	1.87	1.45
SO_3	0.08	0.00	0.00
P_2O_5	0.01	0.00	0.00
Mn_2O_3	0.01	0.00	0.00
SrO	0.01	0.00	0.00

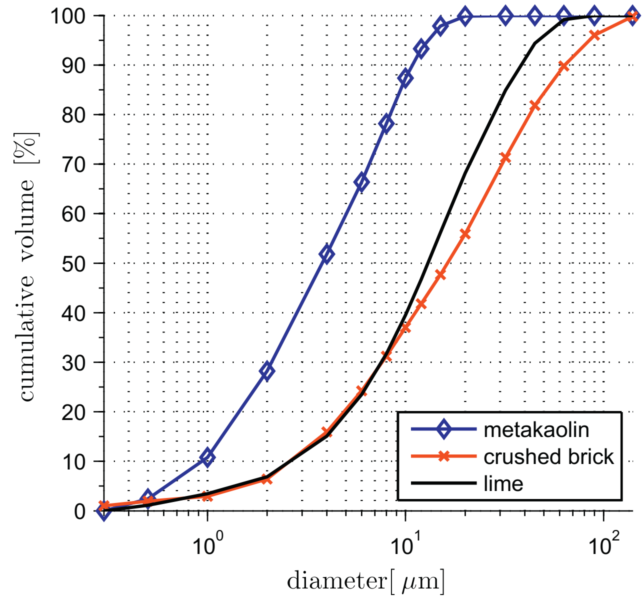

Fig. 1. Grading curves of individual materials.

2.1.1. Metakaolin

Metakaolin is produced by calcination of kaolinitic clays. From the chemical point of view, metakaolin consists of minerals that are necessary for hydraulic reactions — siliceous content is always high (around 60%) as well as Al_2O_3 content (around 30%) [5]. The addition of metakaolin into the mix should result in an enhanced strength of lime pastes and increased durability, while the vapor transport properties should be superior compared to the case when Portland cement is used [6].

Metakaolin used in our study was a finely ground burnt claystone (commercial name Mefisto L05, České lupkové závody Inc., Nové Strašecí, Czech Republic), with relatively high amount of alumina and quite poor in silica. This pozzolanic material has the particle diameter at 50% equal to 4 μm, at 90% to 11 μm, and the specific surface area of 12.7 m^2/g. Its main chemical components are summarized in Table 1 and its grading curve appears in Fig. 1.

2.1.2. Crushed bricks

According to the study by Silva et al. [34], the amorphous components of crushed brick aggregates are mainly represented by aluminosilicates that are able to react with lime by making the interfacial surface alkaline. This chemical reaction produces calcium–silicate–hydrate (CSH) and calcium–aluminum–silicate–hydrate (CASH) at the brick–lime interface, giving the mortars a hydraulic character. The smaller the particles, the bigger the surface per volume and consequently also the hydraulic reactivity. However, when interpreting the results of Silva et al., it should be taken into account that they dealt only with a single type of crushed brick aggregate, while it is known that the reactivity is dependent on the composition of clay used for the brick production, as well as on the firing temperature. In order to gain the required pozzolanic activity, the ceramic products should be fired at the temperatures between 600 and 900 °C [35].

In our study, the brick dust was produced in the laboratory by firing clay bricks from the factory in Štěrboholy, Czech Republic, at the temperature of 750 °C and subsequent grinding and sieving. The fraction of 0.063–0.120 mm obtained by sieving was used for the study, but the exact size of the particles was determined later by the Laser analyzer. The latter method revealed slightly different characteristics of the grain size distribution: diameter at 50% was equal to 16 μm and diameter at 90% to 64 μm, see Fig. 1. This was due to the fact that for the Laser analysis, the burnt brick grains were dispersed in water, causing a