Construction and Building Materials 31 (2012) 135–143

Contents lists available at SciVerse ScienceDirect

Construction and Building Materials

journal homepage: www.elsevier.com/locate/conbuildmat

Aerial lime-based mortars blended with a pozzolanic additive and different admixtures: A mineralogical, textural and physical-mechanical study

A. Arizzi *, G. Cultrone

Universidad de Granada, Departamento de Mineralogía y Petrología, Campus Fuentenueva s/n, 18002 Granada, Spain

ARTICLE INFO

Article history:
Received 13 June 2011
Received in revised form 12 December 2011
Accepted 23 December 2011
Available online 25 January 2012

Keywords:
Metakaolin
Perlite
Water-retaining agent
Plasticiser
Pore system
Mechanical strength

ABSTRACT

This work deals with the effects of a lightweight aggregate, plus water-retaining and a water-reducing agents on the hardened properties of mortars in which the aerial lime is replaced by a 10% and 20% metakaolin content. The influence of different binder-to-sand ratios (1:3, 1:4, 1:6, 1:9 by weight) is also investigated here. A tight relationship between metakaolin content and mortar physical-mechanical properties (compressive strength and pore system) has been found. This study is especially helpful for the establishment of the adequate proportions of additives and admixtures to be used in aerial lime mortars designed for restoration works.

1. Introduction

The addition of inorganic or organic substances to artificial materials such as mortars and bricks is an ancient practice in the field of construction. There are evidences that many organic materials, especially proteins-based (i.e. blood, hair, straw, milk, eggs) [1] were used in the elaboration of historic mortars, in order to improve their workability, resistance and hardening.

Nowadays, although practices and materials have changed and new performances are required in modern constructions, additions are essential components of masonry materials, especially concrete. These additions are divided in two groups: *additives* (i.e. pozzolans, mineral fillers, ceramic powder) that are used in lime based mortars with the aim to improve certain properties or obtain special performances mainly related to the increase of mortar strength, and *admixtures*, added in low amounts (i.e. not higher than a 5% of the total mass) in order to produce a permanent modification in the fresh or hardened mortar, such as density decrease, workability improvement or waterproofing.

The use of lime as binder in mortars involves well-known inconveniences (i.e. slow setting and carbonation times, high drying shrinkage, low mechanical strength) [2] that, in the last 50 years, have been overcome with the use of Portland cement. On the other hand, the ill-omened effects of the use of Portland cement in the Architectural Heritage [3,4] have forced workmen, restorers and scientists to find out alternative materials apt to improve the performances of lime-based mortars. In this sense, it is opportune to use specific admixtures, such as air-entraining and water-retaining agents and pozzolans, which improve workability in the fresh state, and mechanical strength, water permeability and frost resistance in the hardened state of lime-based mortars.

The effects that some admixtures (i.e. water-retaining agents, air-entraining agents and water repellents) have on fresh and hardened performances of air lime-based mortars were almost unknown until the last decades, because the research on these substances was limited to concrete and cement mortars [5]. Only recently, researchers have demonstrated interest in highlighting the advantages and disadvantages of the use of admixtures on aerial lime pastes [6] and aerial lime mortars [7–10]. Nevertheless, none of these studies deals with the effect that those admixtures have on mortars in which a pozzolan is blended to the binder (aerial lime). There exist many evidences of the use of pozzolanic materials, such as brick pebbles or dust and calcined clays [11,12], in ancient mortars. Pozzolans were used in combination with lime to improve the resistance to moisture of rendering mortars, the compactness of floor bedding mortars and the mechanical strength of structural mortars [13,14]. Nowadays, the addition of pozzolanic additives (i.e. fly ashes, silica fumes and calcined clays) to aerial lime mortars is recommended because they confer good properties in the early age, high values of mechanical strength, low water permeability, good cohesion between binders and aggregates and durability [15–20].

* Corresponding author. Tel.: +34 58246614; fax: +34 958243368.
E-mail address: arizzina@ugr.es (A. Arizzi).

0950-0618/$ - see front matter © 2011 Elsevier Ltd. All rights reserved.
doi:10.1016/j.conbuildmat.2011.12.069

Our aim is to study the changes in mineralogical, textural and physical-mechanical characteristics caused by the addition of different admixtures (both organic and inorganic) in mortars composed by a calcareous aggregate and an aerial lime blended with metakaolin. Three types of admixtures have been considered and blended together or individually in mortars: a lightweight aggregate (perlite), a water-retaining agent (cellulose derivative) and a plasticiser (polycarboxylate). The study of the fresh properties of mortars is not considered here, since the effectiveness of these admixtures in exerting their function is already known within the scientific community [5,8,10,21,22]. On the other hand, there is a lack in the petrophysical characterisation of partially carbonated mortars with such mixtures. For this reason, the characteristics of mortars will be investigated after 60 days since their elaboration.

Perlite is an obsidian derivative that is transformed into a very light, porous and fire resistant material, after a rapid heating at temperatures between 900 and 1100 °C. It has a low density, due to the formation of bubbles inside it that cause an expansion up to 15–20 times its original volume. Perlite is used as additive in mortars, concrete and bricks because it provides thermal insulation, reduces fissures and improves long-term mechanical performances [21] and durability [22].

The cellulose derivative increases the adhesion power of mortar and controls the water retention capacity in the fresh state [23], thus lowering the shrinkage during drying and the water film formation. It confers an initial high consistence to the mortar, although this is not maintained during the mortar application phase (consistence changes from very high to moderate).

On the other hand, the polycarboxylate is a synthetic polymer that, by dispersing the lime particles, supplies a high maintenance of the mortar workability and reduces the voids content [24].

This study deals with the morphological, textural, mineralogical and physical-mechanical modifications that the addition of these admixtures produces in mortars, when added alone or blended together. One part of the study refers to mortars in which these admixtures are blended in different combinations whilst the binder-to-aggregate ratio is maintained fix. Another part focuses otherwise on the modifications that occur in mortars prepared with fixed amount of the same admixtures but different binder-to-aggregate ratios.

2. Materials and methods

All mortars were prepared with a calcitic dry hydrated lime (CL90-S, [25]) produced by ANCASA (Seville, Spain) and a calcareous aggregate (CA) with a continuous grading from 0.063 to 1.5 mm. The pozzolan used is a metakaolin (MK) (CLASS N POZZOLAN, [26]), produced by Burgess Pigment Company (USA).

The chemical composition (major and minor elements) of these components (CL, CA and MK) was studied by means of a Bruker S4 Pioneer X-ray fluorescence spectrometer (XRF) (with wavelength dispersion equipped with a goniometer that analyses crystals (LIF200/PET/OVO-55) and Rh X-ray tube (60 kV, 150 mA)), as it is shown in Table 1. The mineralogy of metakaolin was also characterised by means of X-ray diffraction, using a Philips PW-1710 (disoriented powder method, analysis conditions: radiation Cu Kα (λ = 1.5405 Å), 3–60° 2θ explored area, 40 kV voltage, 40 mA current intensity and goniometer speed of 0.1° 2θ/s), and its X-ray pattern is shown in Fig. 1.

Table 1
Chemical composition of the calcitic lime (CL), calcareous aggregate (CA) and metakaolin (MK).

Oxides (%)	CL	CA	MK
SiO_2	0.35	0.16	50.84
CaO	78.01	59.59	0.22
SO_3	1.39	0.03	0.09
MgO	0.70	0.87	0.66
Fe_2O_3	0.10	0.04	0.44
Al_2O_3	0.18	0.06	45.26
K_2O	0.05	0.01	0.24
P_2O_5	0.04	0.01	0.17

Eight mixtures were prepared with different binder/sand (B/S) ratios, metakaolin-to-binder proportions and admixtures amounts, as shown in Table 2.

The flow of the fresh mortar pastes, determined according to the European Standard EN 1015-3 [27], is comprised between 120 and 150 mm. Mortars were conserved during 7 days in normalised steel moulds (4 × 4 × 16 cm) at T = 20 ± 5 °C and RH = 60 ± 5%, instead of being cured at a RH of 95%, following the modification of the standard EN 1015-11 [28] proposed by Cazalla [2]. Despite the presence of metakaolin, the mortars studied here are not hydraulic mortars but aerial ones, hence the preference of curing them at conditions that favour carbonation more than hydration.

After desmoulded, they were cured at the same conditions of T and RH for 60 days in total. Then, mineralogical, morphological and textural characteristics of mortars were determined.

The mineralogical phases of both internal and external zones of mortar samples were determined by means of two different techniques: thermogravimetry (TGA) and X-ray diffraction (XRD). In the first case, it was employed a Shimadzu TGA-50H thermogravimetric analyser, working in air in a temperature range of 25–950 °C, with a heating speed of 5 °C/min. For the XRD analysis, it was used a Panalytical X'Pert PRO MPD diffractometer, with automatic loader and X'Celerator detector, 4–70° 2θ explored area. The identification of the mineral phases was performed by using the X-Powder software package [29].

For the textural study, mortars fragments were metalized with a carbon layer and the microstructure analysed by using a Carl Zeiss Leo-Gemini 1530 field emission scanning electron microscope (FESEM).

Open porosity (P_o, %) and pore size distribution (PSD, in a range of 0.002 < r < 200 μm) were determined using a Micrometics Autopore III 9410 porosimeter (mercury injection porosimetry, MIP). Mortar fragments of ca. 1 cm^3 were oven-dried for 24 h at 60 °C before the analysis.

Flexural and compressive strength were measured by means of a hydraulic press INCOTECNIC-Matest. According to the EN 1015-11 [28] standard, flexural assays were carried out on three samples per mortar (of 4 × 4 × 16 cm). The six samples obtained after the flexural rupture were used for the compressive assays.

3. Results and discussion

3.1. Mineralogical phases of mortars

The XRD patterns of the core (IN) and the surface (EX) of mortars are shown in Fig. 2. The main phase formed in mortars is calcite (CC, Fig. 2) because of the high presence of calcium carbonate as aggregate and also because of the carbonation of lime. The portlandite amount found in mortars after only 28 days of carbonation is low compared to the quantity that is likely to be found in aerial lime-based mortars without admixtures. This is because a part of the portlandite dissolved in water transforms into calcite (i.e. carbonation) whilst another part is involved in the lime-pozzolan reactions (i.e. hydration) favoured by the alcaline environment. The fact that mortars prepared with a lower amount of metakaolin (CCMPCR3-9 with 10% of MK on the total binder) show slightly higher peaks of unreacted lime (i.e. portlandite) in their X-ray diffraction patterns (Fig. 2) confirms that a faster lime consumption does not indicate a quicker carbonation but only the development of hydrated phases.

Calcium alluminate and silicate hydrates of variable steichiometry are formed after activation of the alluminate and silicate phases of metakaolin, in presence of calcium hydroxide (i.e. portlandite) and water [30]. By means of XRD analysis, three general hydrated phases were detected: CSH, ($CaO–SiO_2–H_2O$) or calcium silicate hydrates; CASH, ($CaO–Al_2O_3–SiO_2–H_2O$) or calcium alumina silicate hydrate; and CA$\hat{C}$H, ($Ca_4Al_2(CO_3)(OH)_{12}·6H_2O$) or monocarboalluminate. The latter is one of the mono-phase calcium hydrates and derives from the reaction between the reactive alluminates of metakaolin and the CO_3^{2-} ions present in mortars [31]. In mortars with fixed amount of admixtures, lower metakaolin content and different B/S ratios (i.e. CCMPCR3-9), CA$\hat{C}$H phases have precipitated in very low amounts compared to the other mortars, and they have been detected only in the internal part of the mortars (Fig. 2). Calcium silicate hydrates (CSH) and calcium alumina silicate hydrate (CASH), which are among the main hydrated phases formed at room temperature after pozzolanic reaction of metakaolin [30,32,33], were detected in very low amounts by