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Influence of clay minerals addition on mechanical properties of air lime–metakaolin mortars

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HIGHLIGHTS

- Two sets of lime mortars with addition of clay minerals and metakaolin were analysed.
- Clay minerals and metakaolins cause microstructural changes of mortars.
- Lime with higher bulk density is more suitable to be substituted by additives.
- Clay minerals substituted air lime in 1st set clearly improved mechanical strengths.

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ABSTRACT

Two sets of mortars differing in type of lime and metakaolin, with air lime:sand volumetric ratio 1:3 were prepared with the aim to be used for restoration of historic masonries. The first set involved air lime and 20 wt.% of metakaolin with more impurities (calcite and kaolinite, respectively) and with higher bulk densities compared to materials from the second set. Clay minerals (sepiolite, zeolite A, palygorskite and vermiculite) characterised by high specific surface areas and thus able to keep water in the structure and promote pozzolanic activity of metakaolins were used as additives in air lime and air lime–metakaolin mortars and their impact was evaluated in a view of mechanical strength at 28, 90 and 180 days. Substitution of air lime from the first set by clay mineral and/or metakaolin caused improvement of mechanical strengths predominantly at latter ages, while lime mortars from second set suffer by lack of binder when other additives are supplemented and just palygorskite incorporation improves flexural strength, while vermiculite and metakaolin create mixture with improved compressive strength than lime mortar alone.

1. Introduction

For the adequate restoration of cultural heritage, the compatibility between old mortars and the rehabilitation materials is obligatory. The survey for this compatibility is also supported by the fact that old materials to be renovated, already had evidenced to have suitable mechanical properties and acceptable performances throughout the centuries [1,2]. Recent restoration interventions are based on utilisation of analogous chemical composition of binders, aggregates and mineral additions, as they derive from the study of historic mortars [e.g. 3,4]. The main component of old renders is usually lime, occasionally supplemented by the presence of pozzolanic or other additives. For this reason, air lime mortars and/or mixed with pozzolanic additions (natural or artificial) have been studied widely with the intention to be used as mortars for the construction of historic buildings [1,5–13]. Addition of high reactive pozzolans to lime creates mortars similar to historic ones that display an advanced durability and high values of mechanical strength. Nowadays many researchers are paying attention mainly to metakaolin which is an artificial pozzolanic additive mainly due to its capacity to react with calcium hydroxide creating typical pozzolanic products [14–23]. In addition, positive effect of natural clay minerals such as sepiolite [24–28], vermiculite [29] or synthetic zeolite A [30] on lime mortar's characteristics has also been the object of recent research. In some rehabilitation cases, it is inevitable to provide restoration interventions in places of lack of humidity, or unfavourable conditions such as difficult access to CO_2 or desiccation conditions (wind, heat). This is more so in Portugal due to its climatic

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conditions, extensive coastline and building traditions. For this reason the main scope of the present study is to develop blended mortars based on lime with additions of metakaolin and clay minerals characterised by high specific surface areas, able to adsorb water molecules and provide more humid conditions promoting pozzolanic activity of metakaolin. From natural clay minerals have been chosen sepiolite and palygorskite typical also for their fibrous structure and from commercial ones zeolite type A and expanded vermiculite. Lime/aggregate volumetric ratio selected for this study was 1:3.

2. Materials, mortar composition, conditioning

Taking into account the principal mortar components (air lime and metakaolin), two sets of mortars were prepared:

1. With powdered commercial air lime CL 90 (AL) (Calcidrata, S.A., Portugal) and siliceous river sand and formulated with air lime:sand volumetric ratio of 1:3. Lime binder was replaced by:
   (a) 5 wt.% of fine (FS) and coarse (CS) commercial sepiolite (Sepiolita 15/30, Minas de Paracuellos del Jarama, Madrid, Spain) and 5 wt.% fine (FZ) and coarse (CZ) commercial synthetic zeolite A pellets Phonosorb 551 (Grace Davison, USA);
   (b) 20 wt.% of commercial metakaolin (MK) (EcoPozz, Portugal);
   (c) by both, (a) and (b).

Individual specimens are in the text marked as follows (Table 1).

Water added to mortars was calculated to provide an appropriate workability, accomplished by the flow table test with values around 130–140 mm according to the Standard EN 1015-2 [31]. 19% of water, considering total mortar mass, was added to air lime (AL) and air lime + metakaolin (AL20MK) mortar. Forasmuch sepiolite and zeolite pellets have high specific surface areas and consequently higher water demand, 23% and 21% of water was necessary to add to fine/coarse sepiolite and zeolite mortars, respectively.

2. Second set of mortars was prepared with powdered commercial air lime (AL) (Lusical H100, Portugal) with classification CL90 and siliceous river sand and formulated with air lime:sand volumetric ratio of 1:3. Lime binder was replaced by:
   (a) 5 wt.% of palygorskite (P) (Minas the Torrejon, Spain) and 5 wt.% of expanded vermiculite (V) (Aguiar & Mello, artificial product of natural vermiculite calcination between temperatures of 700 °C and 1000 °C);
   (b) 20 wt.% of commercial metakaolin (MK) (AGS Mineraux, France);
   (c) by both, (a), (b) and with 5 wt.% of fine (FS) commercial sepiolite (Sepiolita 15/30, Minas de Paracuellos del Jarama, Madrid, Spain) and 20 wt.% of commercial metakaolin (MK) (AGS Mineraux, France).

Individual specimens are in the text marked as follows (Table 2).

Table 1
Composition of mortars (1st set).

Reference	Materials
AL	Air lime + sand
FSAL	Fine sepiolite + air lime + sand
CSAL	Coarse sepiolite + air lime + sand
FZAL	Fine zeolite + air lime + sand
CZAL	Coarse zeolite + air lime + sand
AL20MK	Metakaolin + air lime + sand
FSAL20MK	Fine sepiolite + air lime + metakaolin + sand
CSAL20MK	Coarse sepiolite + air lime + metakaolin + sand
FZAL20MK	Fine zeolite + air lime + metakaolin + sand
CZAL20MK	Coarse zeolite + air lime + metakaolin + sand

Table 2
Composition of mortars (2nd set).

Reference	Materials
AL_2	Air lime + sand
PAL_2	Palygorskite + air lime + sand
VAL_2	Vermiculite + air lime + sand
AL20MK_2	Metakaolin + air lime + sand
PAL20MK_2	Palygorskite + air lime + metakaolin + sand
VAL20MK_2	Vermiculite + air lime + metakaolin + sand
FSAL20MK_2	Fine sepiolite + air lime + metakaolin + sand

21% of water was added to AL_2, PAL, AL20MK_2 and PAL20MK; 23% to FSAL20MK_2 and 19% to VAL and VAL20MK mortars.

2.1. Conditioning

Mortar prisms 40 × 40 × 160 mm of all the mortars were prepared. Air lime mortars (AL, AL_2) were cured during all tested ages 28, 90 and 180 days in a chamber with a relative humidity of 65 ± 5% and temperature 20 ± 2 °C. Specimens containing metakaolin and/or FS, CS, FZ, CZ, P, V were stored in moulds for the first 2 days in a chamber at 20 ± 2 °C with a relative humidity of 95 ± 5% and then remoulded and kept for next 5 days at the same conditions. Then the specimens were maintained at a relative humidity of 65 ± 5% and temperature 20 ± 2 °C; and cured up to ages of 28, 90 and 180 days according to the Standard EN 1015-11 [32].

3. Methods

The fine size materials (sepiolite and zeolite) were obtained by dry grinding of the coarse (original) materials in a Ceramic Instruments mill (S2-1000-M) with porcelain jars and alumina balls, during 15 min.

Particle size distribution of fine materials was performed with X-ray grain size analyser Sedigraph 5100 from Micromeritics, following the BS 3406-2 [33].

Bulk densities of materials were determined according to Certification CSTB Cahier [34].

The mineralogical composition of the specimens was determined using a Philips X'Pert diffractometer equipped with Cu Kα radiation.

The microstructural and chemical homogeneity was analysed by scanning electronic microscopy, SEM/EDS (Hitachi SU 70 coupled with EDAX Bruker AXS detector).

Flexural and compressive strength tests were carried out on 3 probes of individual mortar following Standard EN 1015-11 [32] on (SHIMADZU: AG-IC 100 kN) equipment, with loads of 10 and 50 N/s for flexural and compressive strength, respectively.

The dynamic modulus of elasticity was determined based on the fundamental longitudinal resonant frequency following the BS 1881-209 [35].

4. Results and discussion

4.1. Characterisation of materials used for mortars preparation

4.1.1. Particle size distribution and bulk density of materials

Values of particle size distribution and bulk density of all materials used for mortar preparation are reported in Table 3. The lowest D50 is that of palygorskite (0.1 μm) with bulk density 733 kg m^{-3}. Medium values of D50 are attributed to metakaolin (France), fine sepiolite and air lime (Calcidrata) 1.3, 2.4 and 3.2, respectively. Top D50 values has air lime (Lusical) (7.0 μm), metakaolin (Portugal) (10.0 μm) and fine zeolite (16.0 μm). The highest bulk density (1140 kg m^{-3}) of fine materials is provided by fine zeolite. Related to particle size distribution of coarse materials, expanded vermiculite is composed of the largest particles (<4 mm) and in the same way has the lowest bulk density (121 kg m^{-3}) of all the materials (Table 3). Distribution of sand particles ranging between 0.125 and 0.5 mm is followed by coarse sepiolite 0.3–1.2 mm and finally by coarse zeolite between 1.2 and 2.5 mm. Bulk density of sand (1560 kg m^{-3}) is the highest from all materials.

Table 3
Particle size distribution and bulk density of materials.

Material	D50 (μm)	Particle size distribution (mm)	Bulk density (kg m^{-3})
Air lime (Calcidrata)	3.2		460
Air lime (Lusical)	7.0		380
Metakaolin (Portugal)	10.0		673
Metakaolin (France)	1.3		296
Fine sepiolite	2.4		526
Fine zeolite	16.0		1140
Palygorskite	0.1		733
Vermiculite		<4	121
Coarse sepiolite		0.3–1.2	680
Coarse zeolite		1.2–2.5	1290
Sand		0.125–0.5	1560