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Comparing the pozzolanic activity of aerial lime mortars made with metakaolin and fluid catalytic cracking catalyst residue: A petrographic and physical-mechanical study

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HIGHLIGHTS

- FC3R shows lower pozzolanic activity than MK in aerial lime mortars.
- The big amount of particles >10 μm in FCC3R is responsible for its low reactivity.
- Metakaolin develops more aluminate phases and improves mortar strength.
- Ettringite is formed in FCC3R mortar samples.

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ABSTRACT

We investigated the viability of a fluid catalytic cracking catalyst residue (FC3R) as an alternative sustainable pozzolanic additive in aerial lime mortars. The pozzolanic activity of FC3R was compared to that of metakaolin (MK) by chemical-mineralogical, petrographic and physical investigations. The FC3R showed lower pozzolanic activity than MK when added to aerial lime mortars, owed to the size of FC3R particles that generate less hydrated phases and give place to lower mechanical resistances in mortars. We also demonstrated that FC3R is not a compatible material for use in repair mortars, due to the formation of the harmful soluble salt ettringite.

1. Introduction

Together with other waste materials such as blast furnace slag and fly ash, fluid catalytic cracking (FCC) catalyst residue is considered a good, alternative and sustainable source of aluminosilicates [1] intended for use in many building applications, such as mortars [2,3], cold asphalt concrete [4], roof tiles [1] and paving [5], among others. Despite the fact that FCC is a residue of the oil industry, its use in construction has been demonstrated to be environmental viable, since the concentration of leached heavy metals is under the limits established by environmental legislation [6].

The effectiveness of FCC as supplementary cementitious material in ordinary Portland cement (OPC) has been demonstrated by numerous studies, the majority of them showing the contribution of FCC (in binary or ternary systems) in the increase of OPC strength and chemical resistance [7,8]. According to those studies, the FCC shows higher pozzolanic activity with respect to other similar additives, such as fly ashes [9], although the resistance of concrete with FCC under sulphate attack is similar to that induced by metakaolin [10].

Previous studies have demonstrated that FCC shows similar [11] or even higher [12] pozzolanic activity compared to metakaolin, when added to cement mortars. However, its effectiveness as pozzolanic material in non-hydraulic (aerial) lime mortars has not been investigated yet.

Adding pozzolanic additives to aerial lime mortars is a common practise in construction, especially in the restoration sector, as this improves the properties of aerial lime mortars both in the fresh and hardened state (e.g. mechanical strength, water permeability and durability, [13–15]). In this context, metakaolin is one of the most exploited pozzolanic materials, showing a high level of pozzolanic activity, below that of silica fume but greater than fly ashes [16,17].

With the aim to investigate the effectiveness of FCC as pozzolanic additive in aerial lime mortars, its pozzolanic activity has

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been compared here to that of metakaolin, by studying the mineralogical, textural, mechanical and aesthetical properties of lime mortars after 28 days and 4 months since their preparation. Differences in the mortar properties were related to the intrinsic characteristics of the additives.

2. Materials and methods

2.1. Raw materials and mortar preparation

The raw materials used for the production of mortars were:

- a calcitic dry hydrated lime (named CL, CL90S [18]) produced by ANCASA (Seville, Spain).
- a siliceous sand (named SA) with continuous grading between 0.063 and 2 mm, supplied by the company ARGOS d.c. (Granada, Spain);
- metakaolin (named MK, CLASS N POZZOLAN [19]), produced by Burgess Pigment Company (USA);
- a fluid catalytic cracking catalyst residue (named FC3R), supplied by BP-Oil España S.A. refinery in Castellón (Spain), previously ground for 20 min [2] in the presence of DARAGRIND® 155, Grace Construction Products Ltd. (industrial additive added to reduce particle agglomeration).

The chemical and mineralogical composition of the two additives, MK and FC3R were studied by means of X-ray fluorescence (XRF) and X-ray diffraction (XRD). Instruments used were, respectively: a Bruker S4 Pioneer X-ray fluorescence spectrometer with wavelength dispersion, equipped with Rh X-ray tube (60 kV, 150 mA) and LIF200/PET/OVO-55 crystals; and a Panalytical X'Pert PRO MPD diffractometer, with automatic loader (45 kV voltage; 40 mA current; CuKα radiation ($\lambda \approx 1.5405$ Å); 3 to 70° 2θ explored area; 0.01° 2θ/s goniometer speed). Mineral phases were identified using the X-PowderX™ software package [20]. An indicative value of the amorphous versus crystalline phases (a/c) was provided. This ratio is based on the mean value of the intensities, standard deviation and area of the crystal reflection [20].

The particle size distribution of MK and FC3R was analysed by means of a Mastersizer 2000LF from Malvern Instruments (in a range of 0.02–2000 μm). Samples were dispersed in ethanol and sonicated for 20 s before the measurement.

Mortars were prepared with a fixed binder-to-sand proportion, equal to 1:3 by weight, and variable dosages of additives, equal to 10, 15 and 20 wt% on the total amount of binder. Samples were named MK10, 15, 20 and FC10, 15, 20 according to the type and amount of additive. The replacing percentages were chosen as the most recommended ones for both metakaolin and FC3R [3,21]. A control mortar (BLANK), only composed of lime and sand, was also prepared with 1:3 binder-to-sand dosage by weight (Table 1). The water dosage for every mixture was established determining its flow in a range between 130 and 160 mm (Table 1), according to the European Standard EN 1015-3 [22]. After mixing [23], mortars were casted in standardized moulds (40 × 40 × 160 mm) and cured for 7 days in the mould and the following days out of the mould [24], under controlled temperature (T = 20 ± 5 °C) and relative humidity (RH = 60 ± 5%). The chosen curing conditions favour carbonation more than hydration, so as the aerial character of the mortar mixture is predominant on the hydraulic character of the pozzolanic components, in a similar way as in Arizzi and Cultrone [21]. Mortars were cured under the same conditions for 4 months in total before their study.

2.2. Mortar characterization

Mortar samples were analysed after 28 days and 4 months of curing. These time intervals were chosen because they are considered the most representative of the evolution of lime mortar properties over time due to the carbonation process [25]. For the study of mineralogy, porosity and texture both the external (1 cm from the surface) and the internal (core) zones of samples were analysed.

Mineral phases were determined by means of X-ray diffraction, at the same working conditions as those described above, whilst the aluminate and silicate hydrated phases (such as calcium silicate hydrates ($CaO\text{-}SiO_2\text{-}H_2O$, *CSH*), calcium alumina silicate hydrates ($CaO\text{-}Al_2O_3\text{-}SiO_2\text{-}H_2O$, *CASH*) and calcium alumina hydrates ($CaO\text{-}Al_2O_3\text{-}H_2O$, *CAH*), which are mainly amorphous) were determined by means of thermal measurements, using a SHIMADZU TGA-50H analyser (N_2 atmosphere; 10 °C/min heating rate; 25–950 °C temperature range; 70 mg sample).

The morphology and size of mineral phases and the texture of mortars were studied by means of field emission scanning electron microscopy (FESEM), using

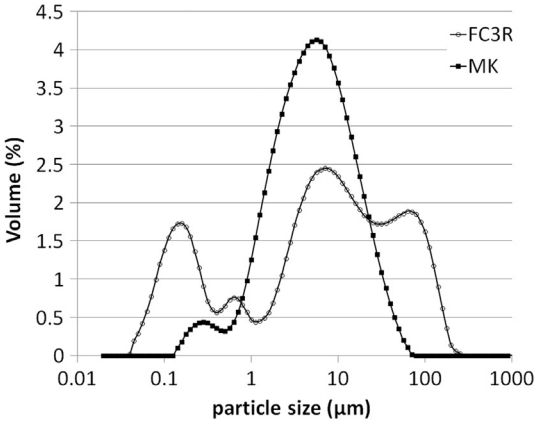

Fig. 1. Particle size distribution of metakaolin (MK) and fluid catalytic cracking catalyst residue (FC3R).

Table 1
Mortar names, components, dosages (by weight) and flow. CL, calcitic dry hydrated lime; MK, metakaolin; FC3R, fluid catalytic cracking catalyst residue; SA, siliceous sand; L:A:S, lime:additive:sand dosage; W:L, water:lime dosage.

Mortar name	CL (g)	MK (g)	FC3R (g)	SA (g)	L:A:S	additive (% on tot. binder)	Water (%)	W:L	Flow (mm)
BLANK	500	–	–	1500	1:0:3	0	23	0.92	133
MK10	450	50	–	1500	0.9:0.1:3	10	24	1.07	134
MK15	425	75	–	1500	0.85:0.15:3	15	24	1.13	135
MK20	400	100	–	1500	0.8:0.2:3	20	25	1.25	148
FC10	450	–	50	1500	0.9:0.1:3	10	23	1.02	147
FC15	425	–	75	1500	0.85:0.15:3	15	23	1.08	151
FC20	400	–	100	1500	0.8:0.2:3	20	23	1.15	158

Table 2
Chemical and mineralogical composition of metakaolin (MK) and the fluid catalytic cracking catalyst residue (FC3R), determined by means of X-ray fluorescence (XRF) and X-ray diffraction (XRD). Legend: Qtz, quartz; Mul, mullite; Ame, amesite; Alb, albite; Fau: faujasite; a/c: amorphous versus crystalline phases ratio; – = absent; * = 0–10%; ** = 10–20%; *** = 30–35%; **** = 35–45%. Values are given in wt%.

XRF

	SiO_2	Al_2O_3	CaO	Fe_2O_3	MgO	Na_2O	K_2O	S
MK	50.80	45.26	0.22	0.44	0.66	0.30	0.24	0.03
FC3R	46.35	42.57	0.34	0.48	0.65	1.91	0.11	0.14

XRD

	Qtz	Mul	Ame	Alb	Fau	a/c
MK	*	***	**	–	–	****
FC3R	**	–	–	***	**	***