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Rheological properties of aerated cement pastes with fly ash, metakaolin and sepiolite additions

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HIGHLIGHTS

- Siliceous fly ash, metakaolin and sepiolite were used in aerated cement pastes.
- Yield stress of fresh pastes was calculated with a slump test.
- Pore network was studied by imbibition methods and X-ray CT.
- Relation between rheological properties and pore network was studied.
- Addition used act as catalyst of aluminum corrosion and expansion.

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ABSTRACT

Rheological properties on aerated cement pastes are studied in order to assess their influence in the pore network of the hardened aerated mortar. For this purpose, different cement types with metakaolin (MK) and sepiolite (SP) additions have been used. Volume and temperature increased during expansion; water retention and rheology of the fresh aerated cement pastes were studied to characterize the fresh mortar. Finally, pore network in the hardened aerated mortar was analyzed by imbibition methods and X-ray computed tomography. Conclusions drawn included that the incorporation of siliceous fly ashes (SFAs) in ordinary Portland cement together with additions of MK or/and SP provided the largest expansion speed increase and the lowest relative density of the foamed mortar.

1. Introduction

Vent chambers of buildings façades require thermal insulating materials to have low fire reaction (for instance, belonging to class A) to avoid fire spread. Current thermal insulating materials are usually based on polymer foams, which hinder fire protection since they produce toxic gases. Therefore, developing new low density fireproof insulating materials is essential. To this aim, lightweight cement mortars are good candidates since their inorganic nature makes them fireproof materials.

Aerated concrete is a lightweight concrete in which air is produced by an aerating agent and then entrapped in the mortar matrix. A possible use is spraying this aerated paste replacing polymeric foams in vent chambers.

Autoclaved aerated concrete is made with aluminum powder, based first on the corrosion of aluminum powder to produce gas, and then it is cured in an autoclave for activating the reactivity of the silica fraction of the aggregate.

Corrosion of aluminum in aqueous media follows these chemical reactions [1]:

$$Al + 3H_2O \rightarrow 3/2H_2 + Al(OH)_3$$

$$Al + 2H_2O \rightarrow 3/2H_2 + AlOOH$$

Aluminum is one of the most reactive metals, but the rate of corrosion is defined by the oxide film present on its surface, which represents a passive film in the pH range of about 5–9. The oxide film dissolution rate is high in strong acidic or alkaline solutions and with high temperature of water [1,2].

Previous investigations on hydrogen generation, from corrosion of aluminum powder, were focused in looking for additions of

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products for accelerating the dissolution of the aluminum oxide film.

These additions were incorporated to the composition of aerated cement pastes, for testing the effect of cement paste on the expansion reaction rate. This research offers advances in the hydrogen generation field to study the aerating process of cement pastes with aluminum powder [3,4].

The use of pozzolanic additions as MK, in lime-based mortars is a common practice in restoration works, where a controlled strength behavior is a basic requirement [5,6]. MK is the result of the dehydroxilization of kaolin in an endothermal process, which produces a very reactive amorphous state. Hydraulic phases are produced during pozzolanic reaction between lime and MK [7].

Sepiolite is a clay with thixotropic behavior, used as a thickener for cement slurry, paint and additives. In cement research, sepiolite, can also be used as a carrier agent of several products, as it has been done with biocide substances for restoration purposes, due to its zeolitic behavior [8].

Microstructure of aerated cement pastes with these additions has been studied earlier by the authors [9].

The purpose of this research work was to analyze the effect of cement selection and the addition of MK and SP in the expansion of aerated concrete. With this aim, this research focuses on the study of properties of fresh and hardened mortar.

2. Materials and methodology

2.1. Raw materials

2.1.1. Binders

Cements used in this research were Calcium Aluminate Cement (CAC) from Kerneos Aluminates (France), White Portland Cement (WPC) and Common Portland Cement with Fly Ash (PC), (designated as BL I 52.5 R and CEM II A/V 42.5 R respectively, according to the European Standard UNE-EN 197-1), both from Tudela Veguin (Spain).

Aerial binder used was high calcium lime (CH), (classified as CL-90-S according to the European Standard ENV 459-1) from Calcasa (Madrid) with a specific surface of 1.5 m^2/g. See Table 1 for chemical composition.

2.1.2. Additions

Pozzolanic material used was Metakaolin (MK) from Grace, S.A. with a specific surface of 9–30 m^2/g and a fineness of D_{50} = 4–12 μm. As viscosity modifier and microfibers reinforcement, dry micronized Sepiolite (SP) Pansil from Tolsa, S. L. (Madrid) with a specific surface of 320 m^2/g was also used. See Table 1 for chemical composition.

2.2. Experimental techniques

2.2.1. Pastes preparation

Two reference pastes were designed for testing, the first one with WPC/CAC/CH with 5:1:4 ratio, and the second one with PC/CH with 4:1 ratio. All mixes have 0.8% of aluminum powder content. Water/reactive powders ratios, by weight 0.8 or 0.6. Mixing proportions of this research are given in Table 2. Pastes were mixed with helical-ribbons impeller rotated by a turbine mixer providing a speed of 3000 RPM.

2.2.2. Gas produced and temperature during expansion

To measure the gas produced and temperature increase during the expansion, an adiabatic box built for the experiment with expanded polystyrene walls was used. 300 cm^3 of paste were poured in 10 × 10 × 10 cm polyethylene molds placed inside the adiabatic box. Temperature was measured with an immersion digital thermometer and gas produced was determined by both, density of fresh cement paste and volume increase. The latter was measured with a vertical scale marked in the mold, until temperature increase stopped.

2.2.3. Yield stress of the paste

Yield stress of the paste was calculated measuring the flow diameter with a slump test. To that end, aluminum powder was removed from the mixture to avoid the expansion effect, the generation or air bubbles modify the workability of the aerated mortar. Pastes were poured into a truncated cone mold (D_1 = 100 mm, D_2 = 70 mm, h = 60 mm). The fresh paste was tamped ten times, and then the mold was carefully lifted vertically upwards, so as not to disturb the cement paste. Finally, when paste is spread, two perpendicular diameters were recorded. Yield stress was measured every 5 min with the same method.

2.2.4. pH

Right after mixing, a digital hand-held pH meter from Hanna was used to measure the pH (acidity or alkalinity) of the pastes without aluminum powder.

2.2.5. Density and porosity

Bulk density, relative density and porosity were determined according to EN 1936:2006. The experiment was done on samples of 50 × 50 × 50 mm^3.

Pore network was studied with an X-ray computed tomography using an YXLON's unipolar constant potential X-ray systems with 225 kV/30 mA, to produce tomographic imaging of the specimens. A 3D model was created using a software of materialize to study the pore size and form.

3. Results and discussion

3.1. Rheological properties

Behavior of fresh cement-based materials can be studied with the Bingham model, as commonly accepted. This model determines that material flows when shear stress τ is higher than yield stress τ_0. Shear rate $\dot{\gamma}$ depends on the former stresses and on plastic viscosity μ according to following equation

$$\tau = \tau_0 + \mu\dot{\gamma} \quad (1)$$

Then, concrete behavior can be measured with yield stress τ_0 (Pa) and viscosity μ (Pa s) values.

3.1.1. Yield stress

Yield stress is calculated using a slump method. According to Murata [10], a slump test is the measurement of the amount of final deformation of concrete due to its own weight. Yield stress (τ_0) is:

$$\tau_0 = \frac{W_x}{2 \cdot \pi \cdot R^2} \quad (2)$$

where W_x is the dead weight of cement paste in the cone and R is the radius of spread paste.

Table 1
Chemical composition of the materials used for this research.

Oxides (%)	CEM II A-V 42.5 R (PC)	BL I 52.5 R (WPC)	Metakaolin (MK)	Sepiolite (SP)	CAC	Air lime (CH)
SiO_2	26.0–28.0	18.0–23.0	52.00	63.18	6.00	–
Al_2O_3	7.50–9.50	3.50–4.80	44.00	3.95	37.00	–
Fe_2O_3	4.00–5.00	0.12–0.17	1.40	0.54	18.50	–
CaO	52.0–56.0	58.0–62.0	0.10	0.63	39.80	93.00
SO_3	2.30–2.80	–	0.10	–	0.40	–
Na_2O	0.10–0.20	–	0.05	0.67	0.20	–
K_2O	1.20–1.40	–	0.40	1.38	0.20	–
MgO	1.30–1.70	0.23–0.41	0.10	20.23	1.50	0.50
TiO_2	–	–	3.00	0.13	4.00	–
L.O.I. 1000 °C	–	–	1.07	–	–	–