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New mortars fabricated by electrostatic dry deposition of nano and microsilica additions: Enhanced properties

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HIGHLIGHTS

- New mortars prepared by electrostatic dry deposition of nanosilica and microsilica.
- Optimal hydration degree and C-S-H gels percentages for mortars with 10% of addition.
- Enhanced compressive strength of the cement mortars with additions.
- Lower chloride migration coefficients and higher electrical resistivity values.
- Microstructurally, the role of ettringite in durable properties is evaluated.

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ABSTRACT

This work reports on the preparation and characterization of cement-based materials with additions of nano and micrometric silica. Their microstructure, mechanical properties and resistance to penetration of aggressive agents were studied. Mixtures of cement pastes and mortars with different percentages of nano and microsilica additions were prepared, with such additions being electrostatically incorporated into all cases in the anhydrous cement particles before the mixing process. The samples were characterized by the following experimental techniques: thermogravimetric analysis/differential thermal analysis (TGA/DTA), compressive strength tests, determination of the chloride migration coefficient, scanning electron microscopy (SEM), electrical resistivity measurement and mercury intrusion porosimetry (MIP). A relevant improvement in the mechanical and durable properties of the additivated samples when compared with the reference material was obtained.

1. Introduction

Given that cement is the most commonly used binder material in civil construction, it is a current main topic of research in materials science. In recent decades, many researchers have used different types of additions in Portland cement when seeking to modify porosity, morphology, composition and nanostructure of C-S-H gels in order to improve the durable and mechanical properties of the starting cement paste [1].

The importance of porous microstructure in the durable properties, especially concerning electrical resistivity and chloride penetration is evident. Thus, when the microstructure becomes denser, higher electrical and chloride penetration resistances are obtained for cement specimens. In such a sense, pozzolanic materials provide a denser and more discontinuous and tortuous microstructure via pozzolanic reactions. Among the most interesting of pozzolanic admixtures, silica additions in cement-based materials have shown a sound performance [2].

Furthermore, use of nano additions has attracted significant attention in recent years. Nanosilica, together with several oxides, is among the most used additions [3–5]. The main objectives sought in obtaining new materials depend on the type of nanoparticle chosen. Thus, nanosilica and nanoalumina addition mainly aim to improve the characteristic properties of cement-paste materials: enhancement of compressive strength, densification of the aggregates/paste interface, decrease of the permeability and, therefore, optimization of durability, among others [6,7].

Besides this, other additions of metallic oxides are concentrated in eventual functional abilities of the material. In such a sense, it is worth citing titanium oxide, which has had a role connected to

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cement self-cleaning that has been widely tested with numerous examples, including specific applications, being reported [8]. Iron oxide constitutes another promising additive, one which concentrates the attention of interesting research concerning magnetic behavior and possible applications [9].

In particular, nano and microsilica additions have been used in recent decades for preparing cementitious materials with highly enhanced performance. Such additions are typically incorporated into the mixing process, leading to interesting improvements in mechanical and durable properties, such as increases in the compressive strength by 43.8% with 0.6% of addition of colloidal nano-SiO_2 [10] and up to 70% when the addition percentage reaches 4% [11]. Furthermore, significant improvements in the durable properties have also been obtained, such as electrical resistivity values of concretes with 20% of nano or microsilica additions that were increased by 300% [12]. Lastly, chloride migration coefficient values of mortars with 5% of nanosilica were found to decrease by around 78% [13].

In all cases, one of the main difficulties in their application is due to operational problems associated with difficulty in workability. In order to optimize addition efficiency, a good dispersion inside the material is required that implies ensuring a good dispersion of particles in the mixing water and subsequent successful compaction of the material.

The authors have previously studied the hydration process of silica additivated cement pastes obtained by two different addition methodologies [14], finding that the best results were shown by the samples prepared by the electrostatic addition method. In particular, workability was improved when the addition of nano and microsilica was made in the anhydrous cement particles instead of incorporating them during the mixing process. Furthermore, promising results in compressive strength and resistance to the penetration of aggressive agents in mortars prepared in such a way were obtained. This method, therefore, has been used for obtaining new cement pastes and mortars.

The main objective of this work is the preparation and study of the properties, at both the microscopic and macroscopic scale, of new cement-based materials – paste and mortar – with nano and microsilica electrostatically incorporated. The evolution of the relevant properties is analyzed depending on the granulometry of the addition, percentage of addition and curing age. In these materials the silica additions have been included by substituting the anhydrous cement at 4% and 10%. The specimens have been characterized from the use of techniques such as mercury intrusion porosimetry (MIP), scanning electron microscopy (SEM) and thermogravimetric analysis (TGA/DTA). The transport capacity of aggressive agents was studied from determination of the chloride migration coefficient and electrical resistivity, with the strength properties being evaluated from compressive strength testing.

2. Material and methods

2.1. Materials

Cement pastes and mortars were prepared with Ultraval Ordinary Type I 52.5R Portland cement (C52.5R). Commercial nano and microsilica were used as additions. Nanosilica (NS) was provided by Cab-O-sil and microsilica (MS) by Ferroatlántica SL. In some cases, in order to improve the fabrication of the probes, the superplasticizer SIKA Viscocrete 5720 (SP) was incorporated. The additions were in all cases electrostatically incorporated into the anhydrous cement particles before the mixing process. Both the NS and MS were included, partially replacing the anhydrous cement, and used a modified version of the method described elsewhere [15]. Normalized sand was used in the mortar preparation. The composition and other relevant characteristics of the employed materials are shown in Table 1.

Cement pastes were prepared at a water/binder ratio equal to 0.4 (w/b = 0.4). Both additions (nano and microsilica) were included as a substitute of anhydrous cement. In the case of mortars, the water/binder ratio was 0.5 and the cement/aggregates ratio was fixed at 1/3 according to normalized mortars.

In Tables 2 and 3, dosages and the nomenclature of paste and mortar samples are shown. The notation of the samples consists of a "P" or an "M" depending on whether the sample is of paste or mortar, a number that indicates the percentage of addition, and an "N" or an "F" depending on the type of addition, nanosilica and microsilica, respectively. The method used is a variation of that described in the UNE-EN 196-1 [16], in which the number of compaction jolts was changed.

Depending on the dimensions necessary for each test, different steel moulds were used. Thus, for electrical resistivity and chloride migration tests, cylindrical moulds with a length of 15 cm and diameter of 10 cm were used and the specimens compacted on a shaking table for 40 s. For the rest of the tests, prismatic moulds with dimensions $4 \times 4 \times 16$ cm were prepared for mortar specimens. Moulds with dimensions $1 \times 1 \times 6$ cm were used for the cement paste probes, using 180 jolts in a compacting unit in these cases. The samples were maintained for 24 h in a chamber at 20 °C and 100% of humidity. After that period, they were demoulded and inserted again into the chamber until the desired curing ages: seven and 28 days. After the required time, a representative portion of the probes was removed and soaked in isopropanol for 24 h and then placed, in order to stop hydration, in a laboratory oven at 40 °C for 24 h.

2.2. Methods

TGA/DTA profiles were registered by using Setaram Labsys Evo TGA-DTA apparatus. A quantity of 50–100 mg of sample was heated at 10 °C/min up to 1200 °C. Paste specimens were used as representative of the corresponding mortars for the TGA/DTA characterization in order to optimize the quantification of the results.

For the compressive strength tests, the standard UNE-EN 12390-3 was used [17]. They were conducted in an IBERTEST press, with a maximum capacity of 1500 kN. The ion transport in mortar specimens was evaluated though chloride migration tests according to the standard NT BUILD 492 [18], which permitted the non-steady-state migration coefficient (Dnssm) to be obtained. Lastly, the electrical resistivity measurements were carried out by following the procedure UNE 83988-1 [19], using GIATEC RCON equipment.

Table 1
Composition and characteristics of the materials.

Analyte (%)	C52.5R	MS	NS
SiO_2	19.20	95.37	99.9
Al_2O_3	6.07	0.34	0.05
Fe_2O_3	1.70	0.16	0.003
CaO	63.41	0.08	–
MgO	2.56	0.04	–
SO_3	3.38	0.15	–
K_2O	0.82	0.30	–
Na_2O	0.33	0.18	–
TiO_2	–	–	0.03
LOI*	2.09	2.70	1.00
Mean diameter (μm)	5.7	15.0	0.2–0.3
BET specific surface (m^2/g)	–	23	200
Blaine specific surface (cm^2/g)	4200	–	–

* Loss of ignition.