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Microstructure of lime and lime-pozzolana pastes with nanosilica

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ABSTRACT

Nanosilica particles (nS) were added to lime (L) and lime-pozzolana (LP) pastes to study the effect of nS as a pozzolanic admixture in L and to synergistically improve the pozzolanic reactivity of LP. Relationships between microstructure and mechanical properties of the pastes were examined. The macroporosity of both pastes decreased, and the compressive strength increased. SEM and X-ray μ-CT analysis accounted for explaining the inconsistent results between the porosity obtained by MIP and density by He-pycnometry. The strong pozzolanic reaction in LPnS explained the high consumption of mixing water, increment of density, and pores assigned to CSH. The SEM analysis also showed that BET and BJH can give erroneous results regarding the adsorption/desorption isotherms, thus affecting the values of the specific surface area and nanoporosity.
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1. Introduction

While nanotechnology in cement and concrete is maturing [1–3], to date, limited attention has been paid to lime-based systems containing reactive nanoparticles that can significantly improve the performance of lime mortars to be used in the repair of the built heritage [4–6]. Much of the work to date with nanoparticles to improve the properties of cementitious composites has been with nanosilica (nS) [7–10]. Nanosilica particles have much higher pozzolanic reactivity than that of silica fume products that are commonly used as ultrafine pozzolanas for producing cementitious materials with advanced properties [11]. By using colloidal silica, it is assumed that the mono-dispersed nanoparticles can act as fillers and seeds much more effectively than the agglomerates of silica particles generated from powders or slurries [12]. Particles of nS can act as nuclei for hydraulic phases due to their high chemical reactivity, which is partly assigned to their high surface-area-to-volume ratio [11]. The accelerating effect of nanosilica on cement hydration has been assigned to a rapid depletion of calcium ions by nS, which can keep the paste at low undersaturation of calcium ions, thus enabling a higher dissolution rate of calcium ions from clinker particles, and hence helping to shorten the induction period [13].

Investigation on the use of nS on the performance of cementitious composites has shown several positive outcomes, e.g., reduction of capillary pores leading to lower water absorption and sorptivity, improvement of the interfacial transition zone between the aggregates and binder [10], porosity reduction and increment of the early mechanical strength [10,14], improvement of the salt [15,16], and frost resistance [17]. The main drawbacks pointed out about the effect of nS particles on the properties of cementitious composites are the reduction of consistency and workability [10,18], lower hydration degree at later ages [19], and agglomeration of the nS particles leading to an increase of the void space [13,20].

Few research groups have recently started to study the effect of nS on the properties of lime-based systems; the main results are summarised as follows:

- The water/binder ratio increases with increasing amount of nS, whereas the workability and setting time decreases with the incorporation of nS [21,22]. Superplasticisers have been added successfully to tackle this problem [22,23].
- The mechanical strength increases with increasing percentage of nS at early ages (from 3 up to 28 days) in lime-pozzolana pastes [21] and until later ages in lime mortars (with 180 days [6] and from 7 up to 365 days [4]).
- The total porosity increases with increasing amount of nS in lime-pozzolana pastes [21] but decreases in lime mortars [4]. Stefanidou [21] assigned the porosity increment to the higher water/binder ratio of pastes with nS addition.
- Lime-pozzolana pastes with nS showed higher crystal size and sharp needle-like crystals [21] whereas lime mortars showed a denser matrix and honeycomb-shaped CSH structures [4].
- The progressive increase of nS shifts the mean pore size diameter towards lower diameters regardless of the curing time (from 7 up to 182 days) [4].
- The durability of lime mortars assessed by climatic chamber (cycles of temperature, relative humidity, rain and UV light), freeze-thaw cycles

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Table 1
Chemical composition of the natural pozzolana used in the preparation of LP and LPnS specimens.

	Na_2O	K_2O	CaO	MgO	Fe_2O_3	Al_2O_3	SiO_2	L.I.[a]	Cl^-	NO_3^-	SO_4^{2-}
Amount (wt. %)	2.78	4.05	15.25	8.22	1.89	9.79	49.57	8.45	0.34	0.0	0.23

[a] L.I. = loss on ignition

and magnesium sulphate attack was improved for the mortars with large amounts of nS (20 wt. %) [4] and the resistance to sodium sulphate was considerably higher for lime mortars with 1 wt.% [5] and 3 wt. % [6].

The results on the effect of nS particles on lime-based systems are promising and, given the limited published data, the present study aims at contributing for a better understanding of the effect of nanosilica on the micro-textural properties of lime-based pastes. The knowledge of the main physicochemical characteristics of the pastes, such as morphology and texture, are important parameters for the design, and for the prediction of the durability of mortar mixes. The objective of the present study is to determine the main micro-textural characteristics and micromechanical properties of lime and lime-pozzolana pastes with the addition of nS particles. A natural pozzolana of volcanic origin composed mainly of silica, calcium oxide, and alumina was used. Tests have been performed on pure aerial lime pastes and combination of lime and natural pozzolana pastes (1:1 wt.) with 1 year of age. The time parameter is important as these binders develop their properties slowly and the mechanism of carbonation and/or hydration, which is responsible for the strength increase, act competitively with deterioration actions such as cracking [24].

2. Experimental part

2.1. Materials and sample preparation

The analysis of the materials used for the production of the pastes was performed before the specimen preparation. Hydrated lime (type N according to ASTM C206) and natural pozzolana from the island of Nisyros (Greece) were used to prepare the specimens. The chemical composition of the pozzolana is given in Table 1. The nanosilica (nS) used was supplied by Sigma–Aldrich and, according to the provider, it is produced by pyrolysis and has a primary particle size of 14 nm.

The grain size of lime and pozzolana was determined with a particle analyser (Mastersizer 2000 Malvern). The pozzolanicity index of the natural pozzolana was determined according to the international standard ASTM C311-13. The properties of the raw materials are presented in Table 2. Additional tests with X-ray powder diffraction, scanning electron microscopy, and transition electron microscopy were performed in the nS grains and can be found in [14].

Nanosilica was added in 1.5 wt. % of the binder weight, following the results obtained by Stefanidou and Papayianni [14] who mentioned an optimum amount between 1 and 2% wt. (with the same type of nS) for both the microstructure and mechanical strength. The most significant issue for all nanoparticles is that of effective dispersion. Aggregation of the nanoparticles reduces the benefits of their small size by creating unreacted pockets leading to a potential for concentration of stresses in the material [1]. Therefore, 6 g of nS was firstly mixed with 200 ml of water and subsequently, the solution of nS was subjected to ultrasound treatment (using a HYDRO 2000MU Mastersizer 2000 system) for 60 min to avoid agglomeration and promote a good dispersion of nS in the binder matrix. Agglomerates of nS could be observed by naked eye before the ultrasound treatment; these disappeared afterwards. A pre-determined amount of additional water was added to each mixture to obtain a consistency of 6 ± 1 mm in all pastes with the Vicat method (EN196-3:1994). The water content of the colloidal nS was considered as a part of the mixing water.

The paste prisms were prepared in casts of 25 × 25 × 100 mm and cured in high humidity conditions: 90% RH (20 °C) for the first 28 days and afterwards at 60% (20 °C) until the testing date. The samples were tested at 2 months of age for mechanical strength and nanoindentation, and at 1 year of age for the other tests. Before the nanoindentation test, small slices (25 × 25 × 5 mm) were cut from the specimens with a diamond saw and subsequently they were polished with a series of grinding papers (grit sizes nos. 2000 and 4000). The specimens were then washed with alcohol in an ultrasonic bath to obtain the required flat and smooth surface to perform the analysis. Table 3 presents the composition of each specimen. The water/binder ratio (w/b) increased significantly for the pastes with nS incorporation, particularly for the paste with pozzolana (increment of 12%), which is in line with the results reported in the literature concerning the addition of nS to lime-pozzolana pastes [21], to Portland cement pastes [14,19], to lime mortars [4,23,25] and to cement mortars [12,26]. The reduction of workability of the fresh mixtures, reflected on the higher amount of water necessary to achieve the same consistency as the reference mixes, has been assigned to the high water adsorption by nS due to its large specific surface and high nanoscale porosity [13]. Thus, the free water available for lubricating the granular system is reduced.

2.2. Testing equipment

2.2.1. Microstructure

Cross-sections of the prismatic specimens (25 × 25 mm) were prepared by impregnating them with an epoxy resin followed by drying at 60 °C and then polishing. Afterwards, the specimens were sputter-coated with a thin layer of carbon and analysed with the scanning electron microscope (SEM) MIRA II LMU (Tescan, Czech Republic) equipped with energy dispersive X-ray detector (EDX) from Bruker Corporation (Germany).

Images of the fresh fracture of each paste were collected with SEM to study the influence of nS addition on the morphology. Before the analysis, the specimens were dried at 60 °C, then broken to have a freshly fractured surface, which was then coated with gold and observed under the SEM. The images were collected under high voltage (15 kV)

Table 2
Properties of the materials used for the preparation of the pastes.

Material	Code	Density (g/cm^3)	Mean particle size (μm)	Specific surface area (m^2/g)	Pozzolanicity index (MPa)	$Ca(OH)_2$ content (wt. %)
Lime	L	2.47	10.8	2.25	–	75
Pozzolana	P	2.40	11.6	1.82	10.5	–
Nanosilica	nS	2.20	0.014	200	–	–

Table 3
Paste identification code, composition by weight and water/binder ratio.

Paste code	Composition	nS[a] (wt. %)	w/b[b]
L	L	–	0.78
LnS	L + nS	1.5	0.82
LP	L : P (1 : 1)	–	0.66
LPnS	L : P (1 : 1) + nS	1.5	0.75

[a] Values are expressed in wt.% of nS with respect to the weight of binder.
[b] w/b: water/binder ratio, i.e., water/hydrated lime and/or water/hydrated lime + pozzolana ratio.

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