



# Performance of Portland cement pastes containing nano-silica and different types of silica



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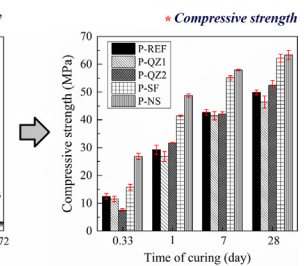
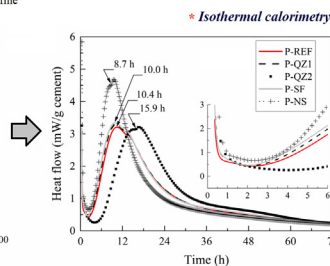
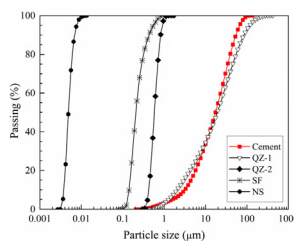
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## HIGHLIGHTS

- Four different silica-based materials were evaluated.
- Hydration, rheology, porous structure, and compression strength were studied.
- The use of nano-silica and silica fume was responsible for refinement of pores.
- Rheological properties were found to improve with addition of the different silicas.

## GRAPHICAL ABSTRACT

Four different silica-based materials: Quartz (QZ-1); Ultrafine quartz (QZ-2); Silica fume (SF); nano silica (NS)



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## ABSTRACT

This work compares the influence of nano-silica with three different silica-based materials (quartz, ultrafine quartz, and silica fume) on hydration, rheology, porous structure, and compression strength in cement pastes with 2.5% cement replacement. Results of calorimetry showed that the inclusion of the silica fume and of nano-silica resulted in an acceleration of hydration of the pastes. In regard to quartz, no change in comparison to the reference was observed until a median particle size of 0.6 µm was reached. The use of nano-silica and silica fume was responsible for refinement of the porous structure and for an increase in compressive strength of the paste in comparison to the reference, caused by their role on heterogeneous nucleation and pozzolanic reactivity. On the other hand, quartz and ultrafine quartz did not have a marked effect on these properties. The rheological properties were also found to improve with addition of the different silicas.

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## 1. Introduction

The use of both chemically active or inert mineral admixtures to cement-based materials has grown during the last few decades due to their technical advantages (increase in strength and durability) and environmental (associated to the reduction in cement

consumption). One example of this application is silica fume, one of the mineral additions that are most widely used in construction and building since the 1970s and that remains the focus of research due to its high reactivity [1–4].

In recent years, mineral additions that are either inert or that have low reactivity, besides very reduced sizes, have been capturing special attention due to a growing demand of new mineral additives, given the scarcity of traditional mineral admixtures, e.g. fly ash and blast furnace slag, in some countries. For instance, recent studies demonstrated the increase in pozzolanic activity of sugarcane bagasse ash with high levels of contamination of quartz

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due to reduction in size of its particles during grinding [5–6]. In another work, the incorporation of quartz particles (10–75%) in mortars promoted an increase in the degree of hydration in comparison to the reference mortar [7].

Besides these effects, the reduction in particle size by grinding can boost the effect of heterogeneous nucleation, due to the inclusion of new nucleation sites for deposition of the hydrated products [8–12]. Grinding can also be responsible for promoting partial amorphization and significant increase in specific surface area [5–6,13–18]. Indeed, mechanical size reduction has been widely used to render chemical reactivity to inert silica-based materials, especially in the application of high-energy milling, which breaks the particles and generates fine, ultrafine and nanometer-sized particles [17,19–23].

The colloidal nano-silica is composed of amorphous particles, with sizes that vary from 1 to 500 nm, which results in unique physical and chemical properties when compared to admixtures made up of coarser particles [24]. Byung-Wan et al. [25] observed that the use of colloidal silica improves the microstructure of both mortars and concrete. The influence of addition of nano-silica in cement pastes was also investigated by Singh et al. [26] and in pastes for oil drillings by Balza et al. [27], Santra et al. [28], and Pang et al. [29]. It was found that nano-silica has higher pozzolanic reactivity than silica fume [25,30,31]. However, Hou et al. [32] demonstrated that the paste containing silica fume presented higher degree of hydration of the cement in comparison to nano-silica, both replacing cement at 5% in weight. Similar results were found by Thomas et al. [33].

Materials in the range of nano and microparticles with this composition have been found to have different effects on the structure and on the performance of cement-based materials [34]. In this context, the present work investigates the influence of silicas of different natures and sizes on the hydration and on the physical and mechanical response of cement pastes on the basis of rheology, isothermal calorimetry, intrusion mercury porosimetry and compressive strength. A recognizably reactive nano-silica was used as a model reactive material.

## 2. Materials and methods

### 2.1. Paste constituents

Three different commercially available silica samples were initially used: quartz (Mineração Jundu, Brazil) with  $D_{50}$  of 19.3  $\mu\text{m}$  and BET specific surface area (SSA) of 1636  $\text{m}^2/\text{kg}$ ; silica fume (Silmix, Brazil) with  $D_{50}$  of 0.2  $\mu\text{m}$  and SSA of 14,366  $\text{m}^2/\text{kg}$ ; and colloidal nano-silica (Cembinder W50, Brazil) with  $D_{50}$  of 4 nm and SSA of 188,000  $\text{m}^2/\text{kg}$ . These silica samples were designated as QZ1 (quartz), SF (silica fume), and NS (nano-silica). From the QZ1, another quartz sample (designated as QZ2) was obtained by high-energy grinding. This procedure was performed by using the Szvegari attrition mill, model 1-S (Union Process, Inc., U.S.A.) with 50% filling and 2% sodium hexametaphosphate (Proquimios, Brazil) solution as dispersant (46.4 kg/t solid-solution mass ratio). The total grinding time was 10 h, being 2 h with silicon nitride balls (5 mm diameter) as grinding media, and Zirconox, a ceria-stabilized zirconia (0.5–1.0 mm diameter), during the remainder of the time. QZ1 and SF were used in solid state and QZ2 and NS as slurry with solids content in weight of 15 and 22%, respectively.

Fig. 1 shows the particle size distribution of different silica samples from laser diffraction analyses (Malvern Mastersizer 2000, U.K.). Deionized water was used as a dispersant for QZ1 and NS. Sodium hexametaphosphate (0.5%) and sodium polyacrylate (0.5%) solutions were used for dispersing QZ2 and SF, respectively, in the particle size analyzes. X-ray diffraction patterns (Bruker D4 Endeavour, Germany) of three original silica samples are displayed in Fig. 2. Obviously, quartz was identified in QZ1, and SF and NS were completely amorphous. Table 1 shows the oxide composition and main physical characteristics of silica samples, where it is possible to note that all samples presented  $\text{SiO}_2$  content higher than 93%. Moreover, the QZ2 also contained minor quantities of impurities, e.g.  $\text{P}_2\text{O}_5$ ,  $\text{Na}_2\text{O}$ , and  $\text{ZrO}_2$ , indicating the presence of the grinding dispersant and  $\text{ZrO}_2$  worn from the grinding media. A 2.5% total carbon content of silica fume was quantified by using a Leco SC 632 (U.S. A.) analyzer and is also given in Table 2. The density of dry nanosilica was 2524  $\text{kg}/\text{m}^3$ , while the suspension (15% in weight of  $\text{SiO}_2$ ) presented a density of 1100  $\text{kg}/\text{m}^3$  (provided by the manufacturer).

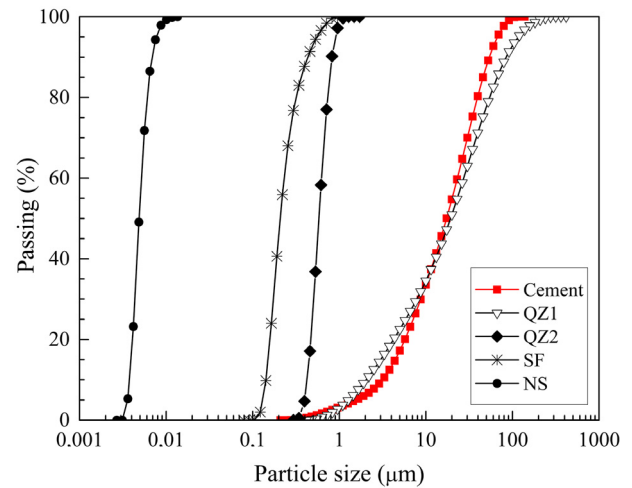


Fig. 1. Particle size distributions of silica samples and Portland cement.

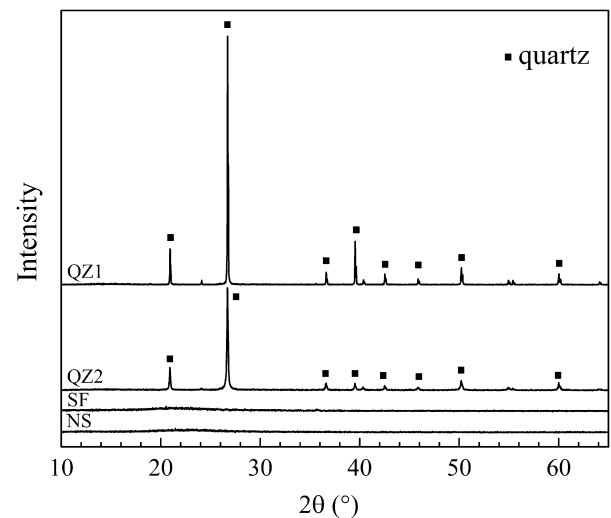


Fig. 2. X-ray diffraction patterns of QZ1, QZ2, SF, and NS (Cu-K $\alpha$  radiation; 35 kV e 40 mA; 0.02° step increment; 1 s counting time).

Brazilian Class G Portland cement [35] (Holcim), which is an ordinary Portland cement (without mineral addition) used in oil well cementing operations, polycarboxylate-based superplasticizer with 31.6% solid content (BASF Glenium 51, Brazil), and deionized water were used during this study. Particle size distribution and oxide composition of cement are displayed in Fig. 1 and Table 1, respectively. Table 1 also shows some important characteristics of the Class G cement.

### 2.2. Preparation and characterization of cement-based pastes

A reference paste (P-REF) with no mineral addition, and other four pastes with each silica replacing 2.5% of the cement mass (named P-QZ1, P-QZ2, P-SF, and P-NS) were designed with a water-solid ratio of 0.4. The superplasticizer was used at dosages of 0.021% (P-REF), 0.018% (P-QZ1), 0.005% (P-QZ2), 0.027% (P-SF), and 0.573% (P-NS) considering a fixed spread diameter (ranging from 100 to 120 mm) from Kantro minislump tests [36] – truncated cone with 60 mm height, 20 mm diameter at the top, and 40 mm diameter at the bottom. The similar superplasticizer contents of P-SF and P-QZ1 in relation to that used in P-REF were due to the low cement replacement level (2.5%), which is typical for pastes containing nano-silica [37–39]. The lowest superplasticizer content of P-QZ2 could be justified by the use of sodium hexametaphosphate solution as dispersant during grinding.

The rate of hydration heat was recorded for a period of up to 7 days using a TAM Air (TA Instruments, U.S.A.) isothermal calorimeter at 23 °C. Approximately 6 g of each paste was introduced in glass ampoules and measurements of heat flow were recorded every 1 min. For this test, the pastes were mixed by adding solid materials to water and superplasticizer in a 100 ml beaker and stirring with a spatula by hand for 1.5 min. For other tests, the pastes were mixed in a high-shear blender type mixer (Chandler 30360, U.S.A.) with bottom driven blades for 10 min at

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