



The influences of quartz content and water-to-binder ratio on the microstructure and hardness of autoclaved Portland cement pastes

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ABSTRACT

The present paper evaluates the influence of the amount of quartz and water/binder ratio on microstructure development in cement pastes. Samples without quartz and with 10%, 20%, 30%, 40% and 50% cement replacement by quartz and with water/binder ratio of 0.1, 0.2, 0.3, 0.4, 0.5, 0.7 and 1.0 were autoclaved at 220 °C for 8 h under a 2.1 MPa pressure. The microstructure was characterized by elemental distribution X-ray maps, XRD, SEM and FTIR and the hardness and elastic modulus of the cement matrix were determined by dynamic hardness testing. The results showed that a significant fraction of quartz flour reacted under hydrothermal conditions, tobermorite and xonotlite were detected in samples with high quartz content and traces of α -C₂SH were present in samples with low quartz content. Samples with a w/b of 0.2 and 30–40% of cement replacement by quartz exhibited the higher values of hardness and elastic modulus.

1. Introduction

The hardening of concretes and the development of mechanical strength is associated with its degree of hydration [1]. Heat treatment is widely used in the precast concrete industry in order to accelerate concrete curing. This process often facilitates cement hydration, especially for pozzolanic reactions, and it modifies the microstructure of cement pastes and consequently expedites early-age concrete strength development [2]. However, elevated curing temperatures could be harmful since the paste may exhibit coarser C-S-H morphology and more open pores structure [3,4]. Curing at temperatures higher than 110 °C–120 °C prompts crystallization of C-S-H, and, in absence of siliceous materials, calcium silicate hydrate (C-S-H) tends to convert in a crystalline phase α -C₂SH [Ca₂SiO₃(OH)₂], rich in Ca. The formation of this phase is considered detrimental because the α -C₂SH is responsible for the reduction in solid volume resulting in high permeability and, as consequence, decreasing the mechanical strength and the useful life of the concretes [5,6].

It is known that the C-S-H is the main product of hydration of Portland cement and when the hydration process happens at room temperature, the C-S-H formed is poorly crystalline and with variable Ca/Si ratio (0.6–2.3) [7,8]. Under hydrothermal conditions, C-S-H gel can convert to crystalline phases and in this case, it is preferred to add pozzolanic materials because they can react with calcium hydroxide produced by cement hydration, form additional C-S-H with lower Ca/Si

ratio and prevent the α -C₂SH formation [9,10]. The transformation of C-S-H amorphous in crystals can be advantageous because these phases are associated with the improvement of mechanical properties, permeability, and durability of Portland cement pastes [11–13]. The relevant forms of crystallized calcium silicate hydrate are tobermorite [Ca₅(OH)₂Si₆O₁₆·4H₂O] and xonotlite [Ca₆(Si₆O₁₇)(OH)₂]. The tobermorite is formed at a lower temperature than xonotlite [14,15] and the transition temperature is approximately at 140 °C [16].

According to Luke [6], in theory, the addition of any pozzolanic material could be used to reduce the Ca/Si ratio and to form crystalline C-S-H. However, higher compressive strength was obtained using quartz flour at 35%–40% by weight of cement. Moreover, xonotlite was formed only when quartz flour was used in Portland cement pastes [17,18]. Although the quartz flour is considered as an inert material at ordinary temperatures [19], under hydrothermal conditions and with temperatures around 170 °C–220 °C the quartz flour becomes chemically reactive. In this case, the quartz can react with calcium hydroxide forming additional C-S-H [20].

Multiple characterization techniques are available to investigate the microstructure of Portland cement pastes. In the case of Portland cement pastes cured under hydrothermal conditions, XRD allows detecting crystallized C-S-H, such as tobermorite and xonotlite. At the same time, through SEM imaging by secondary electrons (SEM-SE) it is possible to identify the hexagonal crystalline plates of calcium hydroxide [21], amorphous C-S-H and platy-like and needle-like structures

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characteristics of tobermorite and xonotlite, respectively [22]. The FTIR spectroscopy is a powerful technique to analyze the hydration of cement and has been used to study the degree of silicate polymerization [23]. The characteristic band of silicates appears in the range of 970 cm^{-1} due to Si-O asymmetric stretching vibration in Q_2 units [24]. At the same time, the band which appears around 1200 cm^{-1} is due to Si-O stretching vibration in Q_3 units. Silicate chains with Q_2 units are formed in C-S-H with a low degree of polymerization whereas the occurrence of Q_3 units is due to the formation of C-S-H with a high degree of polymerization. Thus, the occurrence of Q_3 units suggests the modification from amorphous C-S-H to crystalline calcium silicate hydrate. Consequently, tobermorite and xonotlite have a characteristic band at around 1200 cm^{-1} , and this is more intense in the xonotlite (1202 cm^{-1}) than in tobermorite (1212 cm^{-1}). The sharp peak at around 3611 cm^{-1} is characteristic to the stretching vibrations generated by the CaO-H in xonotlite [25].

The micromechanical properties of C-S-H can be determined through depth-sensing indentation [26]. Nanoindentation provides the opportunity to determine the elastic modulus (E) and the hardness (H) of each phase present in Portland cement pastes [27]. This is only possible since the coupled indentation/SEM technique allows correlating the micromechanical properties with a specific phase [28,29]. In this case the utilization of backscattered electrons in SEM (SEM-BSE) is needed, which make it possible to differentiate the phases due to backscattering coefficient [30,31].

Portland cement pastes with the addition of Si-rich materials and cured in autoclave may exhibit better mechanical properties than their counterparts cured at ordinary temperature. Furthermore, as previously described, the higher strength in autoclave curing was obtained when quartz flour was used as cement replacement instead of pozzolanic materials. Although it is known that quartz flour becomes reactive under hydrothermal conditions, the modifications in the microstructure and the reason for the better mechanical properties are not fully understood. Therefore, the aim of this present paper is to investigate the influence of the amount of quartz flour and water/binder (w/b) in the microstructure and the micromechanical properties of Portland cement pastes cured in autoclave.

2. Material and methods

Ordinary Portland cement (OPC) containing up to 5% limestone filler and ground quartz flour (QF), as the cement replacement, were used in the present work. The particle size distributions were determined using laser granulometry and the curves are shown in Fig. 1. The chemical composition of the OPC, determined by energy dispersive

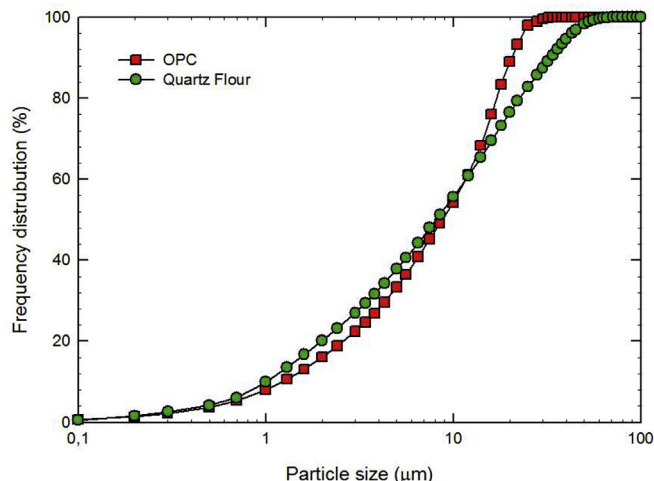


Fig. 1. Particle size for the OPC and quartz flour.

Table 1

Chemical composition (wt. %) of the OPC.

	CaO	SiO ₂	Al ₂ O ₃	SO ₃	MgO	CO ₂	LOI
OPC	56.47	24.59	7.19	2.6	2.43	2.74	3.5

X-ray fluorescence, is shown in Table 1. A total of 12 samples of cement pastes were cast. The samples were divided into two groups. Thus, samples with w/b ratio of 0.2 and 0%, 10%, 20%, 30%, 40% and 50% cement replacement by quartz were prepared in order to evaluate the effect of the amount of quartz in the final product. Also, samples with 30% cement replacement by quartz and w/b ratio of 0.1, 0.2, 0.3, 0.4, 0.5, 0.7 and 1.0 were prepared to evaluate the effect of w/b ratio. In order to prepare the samples the dry products were mixed together for about 3 min and then half volume of water was added and re-mixed for 3 min. Finally, the remaining water was added and re-mixed for 5 min. In order to obtain a better distribution of water in the samples with low w/b ratio (w/b of 0.1 and 0.2) a confining pressure was applied to the fresh mix. It has been shown that the application of confining pressure to the fresh mix of concrete can remove entrapped air and excess water [32]. Visual inspection revealed good homogeneity of the mix after pressing. Pastes were cast using prismatic steel molds ($25 \times 25 \times 100\text{ mm}$) and stored at room temperature of about $25\text{ }^{\circ}\text{C}$ for 24 h. The samples were then autoclaved under a 2.1 MPa pressure for 8 h at $220\text{ }^{\circ}\text{C}$.

In order to identify the hydration products from the XRD patterns and FTIR, the samples were finely ground to a size of approximately $63\text{ }\mu\text{m}$. XRD scans were carried out on a PANalytical Empyrean equipment. The diffractograms were recorded using Cu K α radiation ($k = 1,5406\text{ \AA}$) in a 2θ range from 3.06° to 50° , step size of 0.06 and step time of 3 s. The different phases were identified using the ICDD powder diffraction file database. The Fourier Transform Infrared (FTIR) spectrum was measured by a spectrometer Shimadzu IRAffinity-1 in the range $600\text{--}4000\text{ cm}^{-1}$ with a spectral resolution of 4 cm^{-1} .

The samples for ultra-microhardness testing and SEM-BSE analysis were embedded in acrylic resin without impregnation so as to avoid the effect of the resin on the results of the micromechanical testing. After hardening of the resin at room temperature, the samples' surface was polished with silicon carbide papers (#400, #600 and #1200). Final polishing was carried out using $9.0\text{ }\mu\text{m}$, $3.0\text{ }\mu\text{m}$, and $1.0\text{ }\mu\text{m}$ diamond pastes. Alcohol and mineral oil were used as the lubricant instead of water to avoid dissolving calcium hydroxide. The samples were cleaned in alcohol under ultrasonic vibration between grinding and polishing steps.

Dynamic hardness tests were carried out in a Shimadzu DUH-211 ultra-microhardness tester using a Berkovich indenter. Experiments were performed using load-unload cycle at a constant loading rate of 5.0 mN s^{-1} and dwell time of 10 s. A grid of 50 indentations was applied within a marked region on the sample surface and the area was observed in SEM. After each indent was located and visually classified, load-depth curves were analyzed and the results stemmed from the abnormal load-depth curves were not used. The focus of this work was to determine the hardness in the C-S-H phase and at least 20 out of 50 indents were located in this phase in every sample.

The microstructure of the samples was investigated by using Quanta FEG 3D FEI field emission gun scanning electron microscope (FEG-SEM). The SEM study was carried out using 15 kV accelerating voltage, 7.0 spot size and 10.1 mm working distance. Micrographs were recorded using BSE detector and secondary electron (SE) detector. The BSE imaging was used to differentiate phases within hydrated cement paste, and the morphology of all samples was observed on fractures surfaces using with SE imaging. For elemental mapping characterization, SEM coupled with Energy-dispersive X-ray spectroscopy (EDS) was used. All samples were coated with carbon in order to improve conductivity.

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