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The usage of ultra-fine cement as an admixture to increase the compressive strength of Portland cement mortars

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HIGHLIGHTS

- ▶ UFC used as a substitution of the binder material leads to optimize the cement PSD.
- ▶ The largest compressive strength was shown by the mixture of 30 wt%UFC and 70 wt%CPC30R.
- ▶ The material becomes more brittle when UFC amount increases and *W*/*C* ratio is reduced.
- ▶ Only a 30–40 wt% of UFC is required to improve mechanical properties.
- ▶ The use of costly additions may be disregarded, getting as a result a less expensive product.

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ABSTRACT

The High-Strength Cement (HSC) technology is based on modification of Normal Use Portland Cement (NUPC) by complex admixtures, optimization of mixture design and grinding process. The present work shows the results of preparing and using Ultra-Fine Cement (UFC) as a binder material addition with no extra admixtures of different chemical composition to increase the compressive strength of mortars. The aim of this work is to demonstrate how a NUPC can be converted into a sort of HSC; it was observed that the 70–60 wt%NUPC + 30–40 wt%UFC blend satisfies the goal of increasing mortar's compressive strength.

1. Introduction

It is known that the mechanical properties of certain types of concrete, mortars or grouts based on Portland cement can be improved through the use of the so-called Ultra-Fine Cements (UFCs) [1–3]. In this paper, UFC is used to refer to cements of particle size lower than 15 μm. Practice has demonstrated that the finer the cement particles size the larger the water demand and so the compressive strength of the materials prepared with UFC is reduced. Geymayer et al. [4] reported some particular characteristics observed during the UFC hydration process, namely: (i) very short setting time and (ii) compressive strength retrogression taking place few days after cement paste hardening. Sarkar and Wheeler [5,6] were able to minimize these effects using different admixtures during the mortar mixing.

Some of the Portland cement mortars with improved mechanical properties include repairs and reinforcement of tall buildings, mine subsidence, dam joints, and masonry structures restoration

Abbreviations: μm, micro-meter; cm^2/g, squirt centimeters per gram; g, gram; min, minute; °C, degrees celsius; μW, micro-Watt; s, second; h, hour; rpm, milling Speed; wt%, weight percentage; ASTM, American Society for Testing and Materials; B/P, ball to powder ratio; BSSA, blaine specific surface area; CPC, composite Portland cement; CPC30R, Mexican composed Portland cement; HEBM, high-energy ball mill; HSC, high-strength cement; NUPC, normal use Portland cement; PSD, particle size distribution; SP, superplasticizer; UFC, ultra-fine cement; W/C, water/cement ratio.

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[7,8]. In 1992 both Krizek et al. [9] and Liao et al. [10], performed experimental research using a grout with a mixture of ultra-fine cement and sodium silicate; the compressive strength increased with the cured grout age.

High-strength cements (HSCs) contain twice amount of silicon dioxide SiO_2, aluminum oxide Al_2O_3 and magnesium oxide MgO than Normal Use Portland Cement (NUPC) [11]; commonly provided by the addition of Silica Fume or micro-nano Silica (SiO_2), and Blast Furnace Slag (Al_2O_3 and MgO) [12,13], among other sources. Moreover, cement particles fineness of HSC is much larger than its counterpart. That is, the Blaine Specific Surface Area (BSSA) is of about 3120 cm^2/g and 5790 cm^2/g for NUPC and HPC, respectively. Sobolev [14] has reported outstanding mechanical properties for HSC mortars with compressive strength up to 148 MPa. HSC based mortars possess low permeability, high-resistance to chemical attack, thermal resistance, and excellent freezing and thawing resistance.

In this work, originality comes from the fact that in spite of using a common NUPC the reported compressive strength is driven closer to that of HSC not by varying its original chemical composition neither by producing a complex additives admixture but by reducing the NPC particles to a submicron size (11 μm, D_{90}) while mechanically activating the cement particles and generating a better particle size distribution. In this context, the aim of this work is to determine the effect of using UFC as an addition for the preparation of Portland cement mortars and to report on their particular physical and compressive strength features. The UFC used was prepared from a commercially available Composite Portland Cement (CPC) [15] which was processed by the high-energy ball milling (HEBM) technique at laboratory scale. The Particle Size Distribution (PSD) indicated that 90% of the particles of the precursor UFC powder were smaller than 11 μm, while the BSSA was over 9000 cm^2/g.

2. Experimental study

CPC, as specified by the Mexican Standard NMX-C-414-0NNCCE-2004 [15], is a combination of Portland Cement Clinker, Calcium Sulfate, and a mix of *pozzolanic materials* (natural substances of siliceous or silico-aluminous composition or a combination thereof, they do not harden in themselves when mixed with water but, when finely ground and in the presence of water, they react at normal ambient temperature with dissolved calcium hydroxide $Ca(OH)_2$ to form strength-developing calcium silicate and calcium aluminate compounds. These compounds are similar to those which are formed in the hardening of hydraulic materials. Pozzolanas consist essentially of reactive silicon dioxide SiO_2 and aluminum oxide Al_2O_3), blast furnace slag and/or limestone. This kind of cement is comparable with European Portland–composite cement CEM II/B-M according with EN 197-1 standard [16].

To refine the cement particle size, the HEBM process was carried out in dry conditions, using a commercial Simoloyer CM01-2L horizontal device (Zoz GmbH, Germany). To diminish powder contamination due to the erosion of the milling chamber, the internal walls of the vial were coated with Si_3N_4 small bricks. The milling parameters set during the HEBM were as follows: ball to powder ratio (B/P) = 20 (by weight), rotor milling speed = 900 rpm, milling time = 30 min, grinding media consisted of 2.0 kg of commercial stabilized ZrO_2 balls (5 mm diameter); therefore, 100 g of CPC30 were processed each time the milling process was conducted. Experimental details and characterization of UFC preparation are described elsewhere [17]. Table 1 shows the chemical composition of the CPC30R [15] (CPC = Composite Portland Cement, 30 = strength class of 30 MPa, *R* = high-early strength) used as raw material, as well as its final composition after being treated by the HEBM (UFC) [17]. It is worth mentioning that due to the milling process was conducted by different batches of 100 g (all the processed material was obtained from the same cement bag); therefore, it was not possible to ensure that the material used to determine the chemical composition before and after the HEBM was exactly the same one and due to that during the milling process any different material than Portland Cement (e.g. pozzolanic or high-silica material) was not mixed with CPC30R, the slightly variation of the different oxides amounts showed in Table 1 could be associated with normal spread of the distribution of the constituents of Portland cement [18,19]; therefore, the variations of different percentages were considered as normal and negligible as pointed out by Sarkar and Wheeler [5]; as a result, it was assumed that the HEBM process conducted with the parameter combinations reported herein did not conduct any significant chemical reaction in the solid state and only contributed to the particle size reduction of cement powder as previously reported [17].

Other materials used for the preparation of the mortars included: standard-graded sand 30–100 (in accordance with ASTM C778) [20], and superplasticizer (SP) polycarboxylate-based, specification *F* per ASTM C494 [21] provided by a Swiss vendor, which is a Water-reducing, high-range admixture that reduces the quantity of mixing water required to produce concrete of a given consistency by 12% or greater, it was selected in order to reduce water content required to achieve suitable workability.

Previous to the preparation of cement mortars, the UFC powders were analyzed to determine PSD, BSSA, as well as the heat released (reported herein as temperature variation) through its hydration process. The PSD of cements was determined using a light scattering device in liquid mode using kerosene as dispersant, while performing ultra-sound agitation for 1 min prior to measuring; each measurement was run at least three times. The Blaine Specific Surface Area (BSSA) was determined in accordance with ASTM C204 [22]; using a Blaine apparatus at 19 °C, the cement density was taken as 3.15 g/cm^3, whereas the porosity of the sample was $\varepsilon = 0.530$. A commercial TAM-Air calorimetry equipment TA Instruments (eight channels and sensitivity of ±4 μW) was used to monitor the rate of heat flow evolved from the cement paste specimens prepared exclusively for heat measurements as described later.

Additionally, X-ray diffraction patterns for as-received cement and UFC were recorded at room temperature in Bragg–Brentano geometry by means of a Bruker D8 Advance diffractometer with monochromatic Cu Kα radiation and a secondary graphite monochromator, from 10° to 70° 2θ, at 30 kV and 30 mA. The samples were dried mounted onto a backmounted sample holder to avoid preferred orientation of the crystallites. Analytical conditions were set as follows: step size of 0.025° and counting time per step of 8 s in order to obtain experimental diffraction profiles with the lowest statistical errors. Small aperture slits were used to avoid the contribution of the sample holder to the X-ray pattern.

The mortars were prepared using a sand:cement mass ratio of 1:2.75 according to ASTM C109 [23], the UFC replaced the CPC30R from 0% to 100%; Table 2 lists the various formulations investigated. Three *W*/*C* ratios were investigated: 0.485 (per ASTM C109 [23]), 0.385 and 0.285; the two smaller ratios were set up considering that the strength of Portland cement mortars increases by reducing such a ratio [24,25]. The mortars were mechanically mixed following ASTM C305 standard practice recommendations [26]. The amount of SP added to the cement mixture varied, depending on the UFC content and the *W*/*C* ratio, in order to obtain a paste flowability as close as possible to 110% ± 5%, stated in ASTM C109 [23]. The initial and final setting of the mortars was evaluated using a Vicat apparatus following ASTM C191 [27]. The mortars were cast into five-centimeter cube molds, left to set in a moist cabinet at 23 ± 2 °C and, after demolding, they were cured by immersing into a lime water storage stainless steel tank for further curing at the same temperature that into the moist cabinet (23 ± 2 °C), in accordance with the ASTM C109 [23] and ASTM C511 [28] standard procedures. The compressive strength of the mortars was measured after 1, 3, 7 and 28 days of curing, using a Universal Testing Machine of 30 ton capacity. Finally, the stress–strain (compressive) curves at 28 days for all cubes were recorded using two Linear Variable Displacement Transducers (LVDTs) attached each one to a lateral face of the cubes, curves are shown in Fig. 5.

3. Results and discussion

3.1. Particle size distribution (PSD)

The UFC has 90% of its volume with a particle size under 11 μm, as shown in Fig. 1 (see 100% UFC sample). For comparison purposes, Fig. 1 also shows the PSD of several CPC30R-UFC cement mixtures. It is worth noting that PSD of CPC30R cement after milling process (which is named 100% UFC sample) lead to a reduction of more than 50% of the original size.

Table 1
Chemical composition of studied CPC30R and UFC cements.

Chemical composition (wt%)											
Cement	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	TiO_2	P_2O_5	Mn_2O_3
CPC30R	21.10	5.69	2.24	64.08	2.00	2.65	0.22	0.61	0.28	0.14	0.09
UFC	20.85	5.52	2.60	63.75	1.84	2.73	0.18	0.78	0.19	0.25	0.15