HIGH STRENGTH MICRO/NANO FINE CEMENT

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Abstract

Ordinary Portland Cement (OPC) is the material most widely used in the construction industry all over the world, and by volume, concrete is its largest field of application. Previous works have shown that a grain size distribution of d95 <=24 µm and d50 <= 7 µm of superfine powder in a HPC can decrease the porosity, upgrade the microstructure and the macro-properties of the corresponding concrete incrementing the compression strength 100% of that of OPC. A new type of binder material for high and ultrahigh concretes based in new admixtures and energy modified cement is presented.
High Energy Milling (HEM) was applied to reduce the particle size of OPC, which has a particle size distribution such that 90 % of total particles correspond to 50 µm and might attain 320 kg/cm^2 of compressive strength after 28 days of curing. Milling results show a great particle size diminution of the clinker with 50 % of the powder ≤ 1 µm and 100 % ≤ 6 µm. A fine nanocement-binder mixture was obtained with the admixture of fine refined clinker and refined silica. Results using this Nano/Micro cement show an increment up to 2.5 times compression strength at 24 hours. After 28 days of curing and adding effective water reducer additives, values between 900 and 1100 kg/cm^2 are obtained which are almost four times higher than OPC's values, with a 0.32 – 0.34 water/cement ratio. The setting time and the compression strength can be modified according to different mixtures and different particle size distributions of this nanocement-binder

Key words: **Micro/nanocement, HEM, strength, particle size distribution.**

1. INTRODUCTION

The demand for high performance concrete (HPC) is rising increasingly along with the development of modern construction particularly for high-rise buildings, large-span, underground and marine structure. At present the general understanding of HPC in the world is that the concrete could possess super strength, high mobility and high durability [1].

Several research work has been done over the years, aimed at obtaining high mechanical performance cementitious materials. More recently two technologies have been adopted. The first concerns compact granular matrix concrete (such as DSP), also incorporating ultrahard aggregate (calcinated bauxite or granite). The other relates to macrodefect-free (MDF) polymer pastes. These pastes have very high tensile strength (above 150 MPa), particularly when mixed with aluminous cement [2].

In spite of these technologies, in this work, nanotechnology was been applied since micro/nanoparticles show unique physical and chemical properties different from those of the conventional materials. Because of their unique properties, micro/nanoparticles have been gaining increasing attention and been applied in many fields to fabricate new materials with novelty functions [3]. Considering this, we decided to apply High Energy Milling (HEM) as an innovative processing technique for the commercial production of high strength micro/nanofine cement. As a consequence, then in the long-term range, this technique should also be applied for the manufacturing of OPC at lower energy consumption [4].

2. EXPERIMENTAL

2.1. Raw materials

OPC clinker passing 20 mesh (850 μm), fabricated by Grupo Cementos de Chihuahua (GCC) Mexico and ASTM standard sand were the starting materials used for the refinements tests by HEM.

2.2. Experimental methods

2.2.1. Refinements

A commercial Simoloyer milling device (CM 20) [5] operated by computer was used in order to carry out the millings. HEM operations were dry-conducted in air atmosphere whereas the temperature of vial container was kept at room temperature with a refrigeration system. Operation conditions were as follows; cycles of 4.5 min. each, rotational speed: 500 rpm (first 4 min. of the cycle) and 200 rpm (next 30 seconds), ball to powder (B/P) ratio: 1/46.

2.2.2. Characterization

The starting and milled material was characterized by X-Ray Diffraction (XRD, Siemens D500), SEM-EDS (JEOL, JSM 5800-LV equipped with a system of x-rays energy dispersion DX prime by EDAX). The particle size distribution (PSD) was measured with a Cilas 1180 analyzer.

2.2.3. Sample tests

For each test, six cubic specimens were made from mortar mixture according to C 109 ASTM standard [6].

3. RESULTS AND DISCUSSION

A PSD comparison between OPC and refined clinker and standard sand are given in Figure 1. Special names were been given to the different materials according to their particle size.

According to Figure 1 a great particle size diminution was been observed particularly for Ultrafine Clinker (UFC) that has 100 % of powder under 5 μm. This particle size diminution can be observed in the SEM micrographs shown in Figure 2.

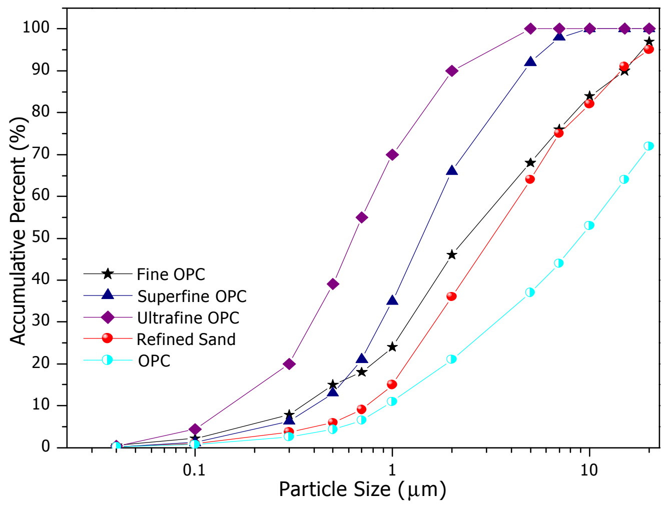


Figure 1. PSD comparison between OPC and refined materials.

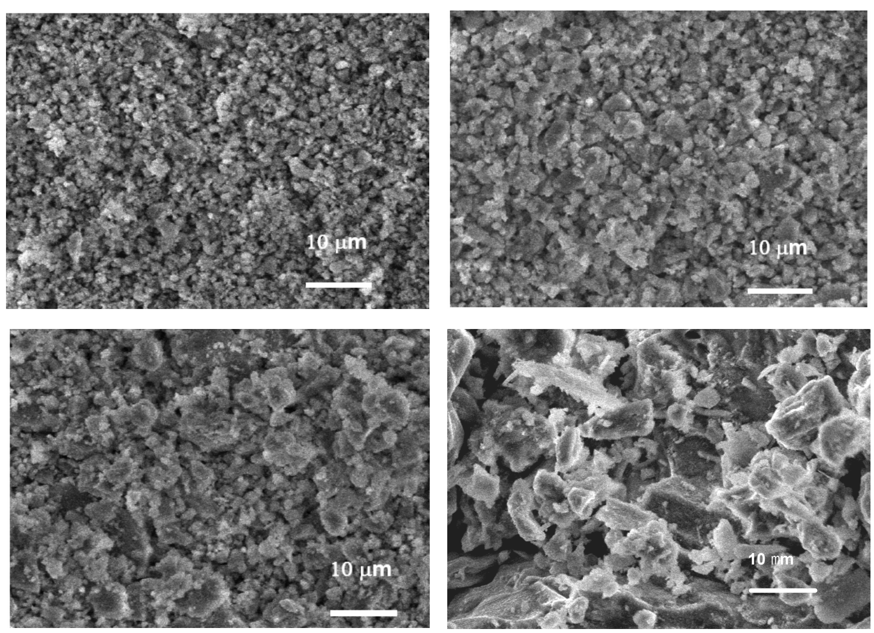


Figure 2. SEM micrographs of: a) Ultrafine, b) Superfine, c) Fine and d) Starting material OPC.

The three types of milled material shows a much more homogeneous microstructure as well as a much smaller grain size to the presented one by the not-refined clinker (> 20 μm). A very fine material can be observed in Figure 2a with a grain size ≤ 2 μm, for the superfine clinker and fine clinker (Figure 2b and c, respectively) a grain size between 5 and 10 μm can be observed.

X ray diffraction comparison patterns are presented in Figure 3. It can be observed that the grinding process doesn't affect the material structure.

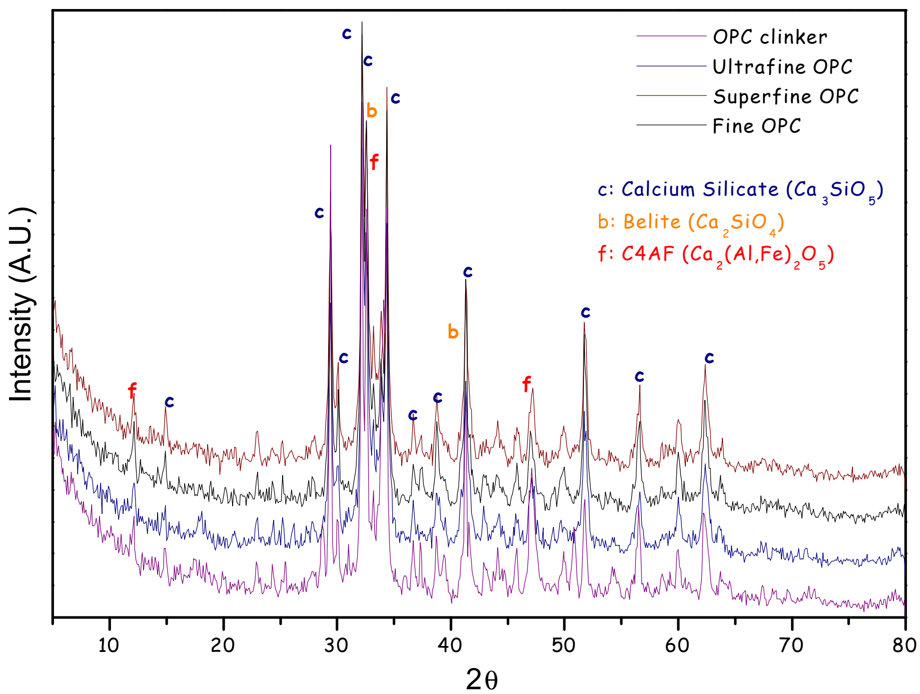


Figure 3. XRD patterns of: Ultrafine, Superfine, Fine and Starting material OPC clinker.

In order to compare the compressive strength of the refined material (UFC, SFC and FC) against OPC cubic specimens were tested using a Universal testing machine 20063 Cernusco s/N (Capacity Force max.= 1300 kN, by Controls) after 1, 3, 7 and 28 days of curing.

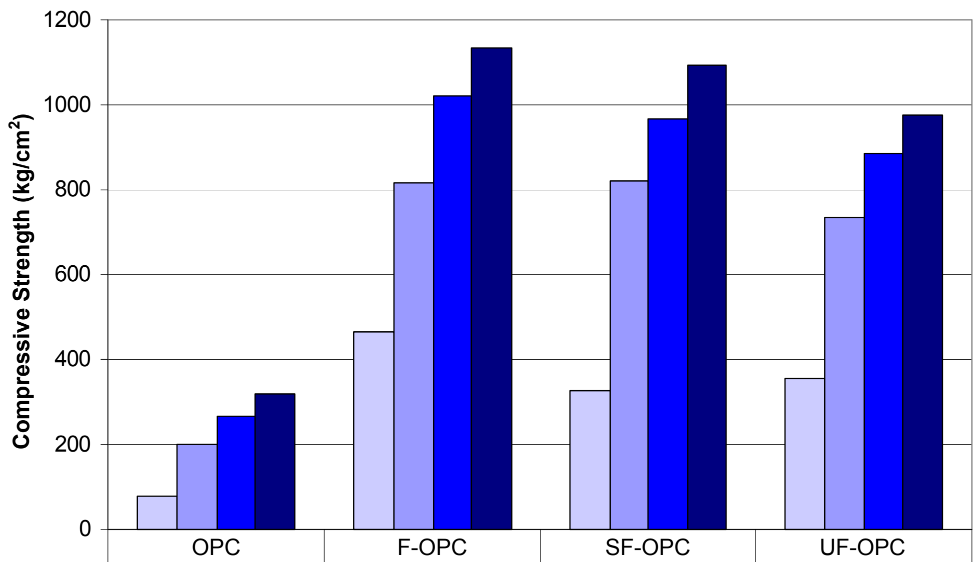
Different tests was been done varying the substitution amount of refined material added to the mixture (mortar). Best results were been obtained as follows: substitution of 30 % of OPC to the corresponding refined material and 20 % of the standard sand to refined sand as well as Table 1 shows.

Table 1. Mortar design using Refined Clinker and Sand.

OPC (%)	Refined OPC [UF-OPC,SF-OPC, FC-OPC] (%)	Std. Sand (%)	Refined Sand (%)	Additive (%)	w/c
70	30	80	20	2.25	0.32-0.34

Because the refined material posses a great surface area (low particle size) a superplasticizer was been used to increase the fluidity, since when the cement specific surface area is bigger, the normal consistency water required weight is increased and fluidity is lowed [7].

Last researches was been showed that the cement particles bigger than 60 μm had not big influence on the cement hydration and strength development. The particles less than 5 μm has most contribution to the early strength, the particles between 5 and 30 μm are the main which bears the strength development and the particles between 30 and 60 μm had most contribution to the later strength [7]. The compressive strength results are given in Figure 4.



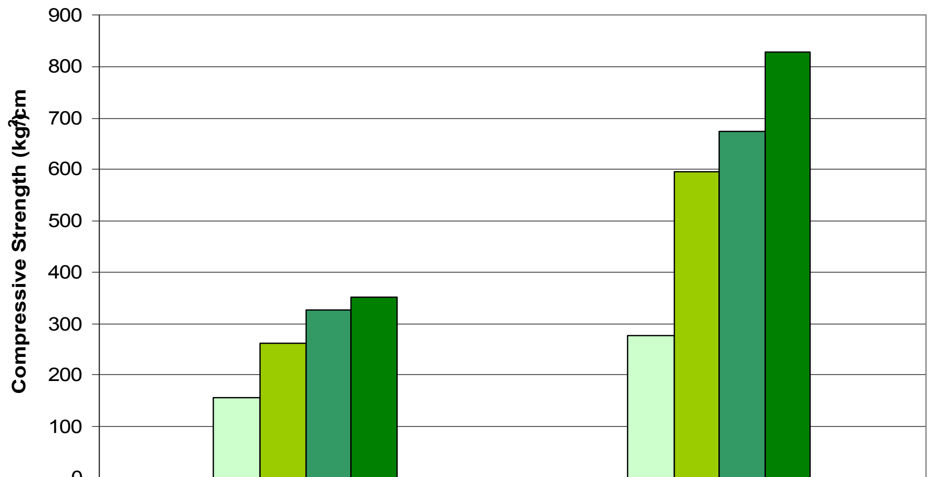
	OPC	F-OPC	SF-OPC	UF-OPC
24 hrs.	76	465	327	355
3 days	200	816	820	735
7 days	265	1022	969	884
28 days	318	1135	1094	974

Figure 4. Compressive Strength results of: Ultrafine, Superfine, Fine and OPC.

It can be observed that OPC compressive strength results agree with the expected according to C 109 ASTM standard. However, the compressive strength values were been improved for the FC, SFC and UFC.

These results are, by far, greater than OPC values; about 4 - 6 times higher than conventional material after 1 day of curing and after 28 days of curing reached values of, almost, 1150 kg/cm^2. That occurs because, under the same conditions, when the specific surface area of reaction media is bigger, the reaction speed is faster. Therefore when the fineness of cement is bigger, the hydration speed is faster, the hydration product increases more, then it causes the slurry from suspending state to the configuration of agglomeration crystal, the set time is shortened and the increase range of compressive strength is enhanced.

Several tests of concrete mixtures have been made following the same mortar design using the corresponding aggregates. Until now, the compressive strength results show a good mixture behavior, the results for one of the test are shown in the Figure 5.



	Standard OPC	Test 1
24 hrs.	157	278
3 days	263	596
7 days	326	675
28 days	352	826

Figure 5. Comparison of Compressive Strength results between Fine OPC and Standard OPC concrete mixtures.

These results show an excellent improvement on the compressive strength values shown by the Fine OPC concrete mixture that reach values about 2 times higher against Standard OPC shows. Optimization of concrete mixture designs are now in process.

4. CONCLUSIONS

OPC clinker and ASTM standard sand were been refined using HEM, obtaining a great particle size diminution respect to the one of the conventional cement (OPC).

Compressive strength results have shown that the refined material posses a much better strength than the one that OPC presents (3 to 6 times higher at different ages).

The preliminary results of concrete mixture tests show a great behavior reaching compressive strength values of 830 kg/cm^2 (2 times higher that standard OPC) after 28 days of curing.

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