STUDY OF DURABILITY OF PORTLAND CEMENT MORTARS BLENDED WITH SILICA NANOPARTICLES

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ABSTRACT

In this paper the effects of nanosilica (NS) on porosity, capillary suction (UNE 8398:2008), compressive strength (ASTM C 349), and sulfate resistance (ASTM C 1012) were evaluated for mortars made with Portland cement (control) and partially replaced with a commercial NS suspension, in percentages by weight of 0, 1, 3, 5, and 10 %. Mortars with a water/binder (w/b) ratio of 0.55 and addition of superplasticizer, for flow correction, were prepared. NS showed that, it has an important role in pore refining, decreasing the total volume of pores and their diameters. Samples containing NS showed an important positive effect on the capillary suction and sulfate resistance. In the case of expansion due to sulfate attack, mortars with 5 and 10 % of NS decreased expansion by 90 and 95 % respectively after two years of immersion.

KEYWORDS: Nanosilica, pozzolan, durability, sulfate resistance, blended cement, Portland cement

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1. INTRODUCTION

Nanosilica (NS) has been demonstrated to have a high pozzolanic reactivity, with important implications for the behavior of cement-based composites in fresh and hardened states. In the fresh state, it has been found that NS reduces setting times, increases the release of hydration heat, and modifies the rheological behavior of cement pastes and mortars [1-11]. In the hardened state, researchers have reported that NS increases the compressive strength [1-4, 6, 12, 13], decreases the porosity and improves some aspects of durability [4, 5, 8, 11, 14-18]. Some works have found that NS produces important mineralogical changes, mainly on C-S-H and portlandite (CH). For C-S-H, NS accelerates its formation, the samples blended with NS have a higher content of C-S-H, and it allows formation of longer C-S-H chains in comparison to the control samples [16-20].

In the evaluation of the incidence of NS on durability, most researchers have argued that, because this material has such good pozzolanic activity, it is possible to expect that NS will have a major impact on the durability of the mixtures made with. There are few studies published to date where researchers make a real assessment of the performance of cement blended with NS and that, the mortars are exposed to aggressive agents. Among the papers found those by Gaitero et al. [11] and Deyu Kong et al. [22] stand out, they evaluate the reduction of the calcium-leaching rate of cement paste by addition of silica nanoparticles. Other interesting investigations include those by Min-Hong et al. [1], Deyu Kong et al. [22], Said et al. [8], and Mostafa et al. [23], which analyze the resistance to chloride-ion penetration made on concrete of Portland cement blended with NS, and the work of Berra et al. [24], which studied the use of NS for preventing expansive alkali-silica reaction in concrete. Quercia et al [25], which found that some durability indicators like conductivity, chlorine migration and diffusion coefficients, and freeze-thaw resistance were significantly improved in self-compacting concrete (SCC) with addition of NS. Ibrahim Rahel et al. [26 have researched the effect of nanosilica on fire resistance of cement mortars.

This work aims to discuss the results of the experimental evaluation of the effects of NS on porosity, water absorption, compressive strength, and sulfate resistance of Portland cement mortars. Regarding sulfate resistance, we show the evaluation of long term (more than two years) volumetric stability of mortars containing different percentages of NS immersed in a solution of magnesium sulfate (5 % by weight). Magnesium sulfate ($MgSO_4$) is considered the most aggressive agent for the Portland cement [27]. The attack of the $MgSO_4$ on C-S-H produces its decomposition to gypsum, brucite, and silica gel; this process is not directly related to ettringite formation [28]. There is a loss of strength and an adhesion into the cement paste, due to decalcification of C-S-H [28]. Hekal et al. [29] say that the decalcification of C-S-H, by the attack of magnesium sulfate, produces non-cohesive magnesium sulfate hydrate (M-S-H), as well as the expansion caused by the formation of expansive salts.

2. MATERIALS AND METHODS

In this work mortars of Colombian Ordinary Portland Cement (OPC), produced by Cementos Argos S. A., were prepared, with dry weight percentages of 0, 1, 3, 5, and 10 % of commercial NS produced by BASF Chemicals Company. The nanosilica was presented as an aqueous suspension, with a suspended solids concentration of 40 % on average and a pH of 10±1 at 20 ºC.

The durability (sulfate resistance and capillary suction) on mortars of cement with different percentages of substitutions of cement by NS was evaluated. Sulfate resistance was determined by measuring the longitudinal change in mortar bars with 0, 1, 3, 5, and 10 % NS; mortar bars were immersed in a solution of $MgSO_4$ with a concentration of 5 %, for 154 weeks, according to ASTM C1012 [30]. Capillary suction was evaluated on mortars, after 3 days of normal curing, with replacement of 0, 5, and 10 % of cement by NS, according to UNE 8398:2008 [31]. Compressive strength was evaluated on mortars with 0, 5, and 10 wt. % of substitution of cement by NS after 1, 3, 7, and 28 normal curing days, according to ASTM C349-02 [32].

To evaluate durability and compressive strength mortars were prepared in accordance with the procedure in ASTM C305 [33]. Binder-to-Ottawa-sand (b/s) ratio used was 1/2.75, being binder (b) the sum of cement plus nanosilica. A constant water-to-binder (w/b) ratio of 0.55 was used. The w/b ratio was corrected for the amount of water incorporated by the suspension. To achieve a flow between 105 – 115 % corresponding amount of superplasticizer (SP) required was incorporated (Table 1), according to ASTM C109 [34]. The superplasticizer (460 Pozzolith BASF Chemicals) was homogenized with the mixing water in order to achieve the optimum dispersion of the NS-particles in the mixes.

Table 1. Mixture proportions of mortars.

Sample	**Cement**	**NS**	**b/s ratio**	**w/b ratio**	**superplasticizer**		**Flow**
	(g)	**(g)**			**(g)**	**(%)**	**(%)**
Control	500	0	1:2.75	0.55	1	0.2	114.8
5%NS	475	25	1:2.75	0.55	3	0.6	106.0
10%NS	450	50	1:2.75	0.55	13	2.6	110.4

In order to evaluate the effect of NS on porosity, pastes with 0, 5, and 10 wt. % of substitutions of cement by NS were prepared and evaluated by a Micromeritics Autopore IV Mercury Porosimeter. Samples were mixed manually with a w/b ratio of 0.4 and they were cured for 28 days at 20 ºC and 98 % relative humidity (RH). Seeking to improve the dispersion, the NS-particles suspension was pre-mixed with the mixing water.

2.1 CHARACTERIZATION OF MATERIALS

The chemical composition of the materials used, cement and NS, are presented in Table 2. These analyses were carried out in an X-ray Fluorescence ARL 8680s Total Cement Analyzer using the wave dispersion method under standard ASTM C114-03 [35]. These results allowed conclude that, NS is high purity silica (Table 2).

Table 2. Chemical composition of materials

Parameters (%)	Cement	Nanosilica (NS)
SiO_2	20.13	93.56
Al_2O_3	4.37	0.00
Fe_2O_3	3.71	0.39
CaO	64.30	0.22
MgO	2,27	0.13
Na_2O	2.27	0.62
K_2O	0.31	0.02
SO_3	1.99	0.30
Loss on ignition (LOI)	2.44	4.46
Free lime (FL)	0.33	

The NS has mean particle size of 98.65 nm, measurements were carried out in a Zetasizer of Malvern Instruments Ltd. The specific surface area (SSA) was determined in a Micromeritics Gemini 2380 by N_2-physic-adsorption using BET method. Values of SSA found were 1.14 m^2/g and 51.4 m^2/g for the cement and the NS respectively.

The X-ray diffraction (XRD) of the mineral addition (Figure 1) was performed in a PANalytical X'Pert PRO MPD, using a 2θ range of 2° to 70° with a step of 0.02° and an accumulation time of 30

s. It can be established that, NS has very low crystallinity and high purity. A broad peak was centered at $2\theta = 21.60º$ and appeared as baseline deviation in the 2θ ranges 20.06º and 26.50º.

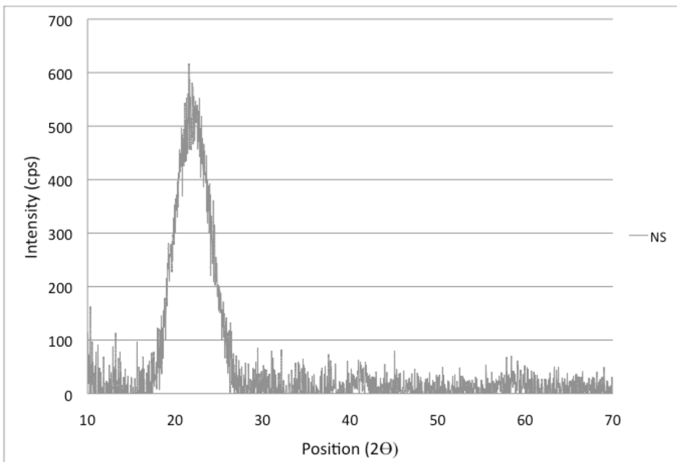

Figure 1. X-ray diffractogram of NS

Zeta potential was measured to different values of pH in order to evaluate the behavior of the NS dispersion when mixed with Portland cement (Figure 2); the suspension pH was modified using "ammonium hydroxide". It can be seen that, NS suspension for pH between 7 and 14 is in the stable zone; potential values are lower than -30 mV [36-37], whereby, it is possible to conclude that, the NS will not agglomerate into the paste of Portland cement.

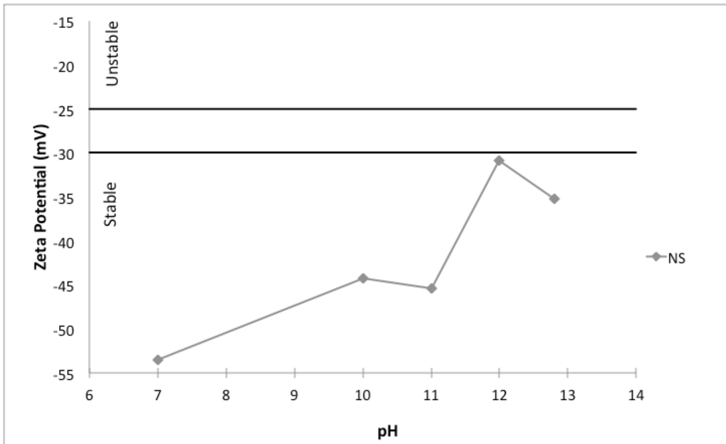

Figure 2. Zeta potential of the NS suspension for different values of pH

3. RESULTS AND DISCUSSION

3.1 CAPILLARY SUCTION

This property controls the transport of fluid in the unsaturated concrete and depends on the concrete pore structure and type of fluid used [38]. Results of the tests of capillary suction (absorption of water with time) of the mortars prepared with cement (control) and blended cements (5 and 10 wt. % of NS), after three curing days are presented in Figure 3.

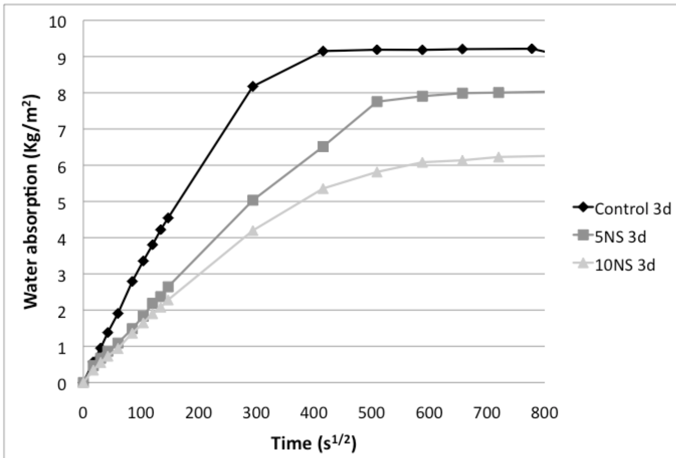

Figure 3. Capillary suction of different mortars after three curing days: control mortar (control 3d), 5 wt. % of NS replaced (5NS 3d) and 10 wt. % of NS replaced (10NS 3d)

There is a significant decrease in absorption rate and noteworthy differences in the quantity of absorbed water at the end of the test with the increase of substitution of the cement by NS (Figure 3). This fact is evidenced because the slope the first part of the curves of capillary suctions (between 0 and 300 $s^{1/2}$), which is associated to the water absorption rate, decreases with the increase of NS percentage in mixes (control = 0.026 $Kg/m^2s^{1/2}$, 5NS = 0.018 $Kg/m^2s^{1/2}$, and 10NS = 0.012 $Kg/m^2s^{1/2}$), showing that, the mortar with 10 wt. % of NS has less than half the water absorption rate in comparison with the control sample. Additionally, total water absorption also decreases with the content of NS as shown by the values after 500 $s^{1/2}$, 5NS shows a reduction of 11.1 % and 10NS of 33.3 % in comparison with the control sample.

This behavior of the NS shows as this mineral addition has a significant effect on refinement of pores, i.e., NS decreased mortars permeability, due to the rupture of the pore interconnections. Furthermore, NS seems to produce a decrease of total volume of pores. These effects of NS are due to its high specific surface area, their small particle size and good particle dispersion within cement pastes. Thus facilitating chemical reactions necessary to produce a high bulk density cementitious matrix with more C-S-H and less calcium hydroxide (CH), as was suggested by Ozyildirim and Zegetosky [39]. This must be added to a physical effect of filler as was claimed by Gaitero et al. [21]. In order to assess the evolution of their porosity, the samples were assayed in a Mercury Intrusion Porosimeter (MIP).

3.2 STUDY OF MERCURY INTRUSION POROSIMETRY (MIP)

The mercury intrusion porosimetry technique (MIP) was carried out to evaluate the total volume of pores in percentage in cement pastes with and without NS (Table 3). In addition, from the MIP measurements the porosity, the pore-size distribution and the pore network can be analyzed. In the case of pastes, mortars or concretes the pore structure affects the mechanical behavior, permeability and durability of them [40].

Table 3. Total porosity of pastes (w/b=0.4, 28 days curing time)

Samples	Total porosity (%)
Control	19.18
5NS	19.74
10NS	16.84

Values of total porosity for the control sample and sample with 5 wt. % of NS are almost equal. The sample with 10 wt. % of NS substitution achieves 12 % reduction in the total porosity with respect to the control sample.

Gaitero et al. [41] proposed that, the porosity in pastes with NS decreases because the nanoparticles work as fillers in the space between the grains, and although they are very small to improve the efficiency of packing by themselves, NS-particles serve as nucleation sites for the growth of the hydration products and these products are responsible for reducing porosity. The fact is that, due to the pozzolanic activity of NS the amount of C-S-H gel increases because of the reaction of NS with portlandite, as was reported previously by Tobón et al. [19]. This fact helps one to understand that, the porosity decreases in pastes because portlandite with a laminar structure tends to pack more freely than the C-S-H gel. Besides, the quantity of water required for calcium ions in the C-S-H gel is greater than that in the portlandite, which reduces the free water, which is the main cause of the formation of pores [21].

MIP allows comparisons of the distribution of pore size. Figure 4 shows the differential volume of mercury intrusion versus pore diameters obtained for the different samples, which is a useful representation to determine pore size groupings and quantity of pores.

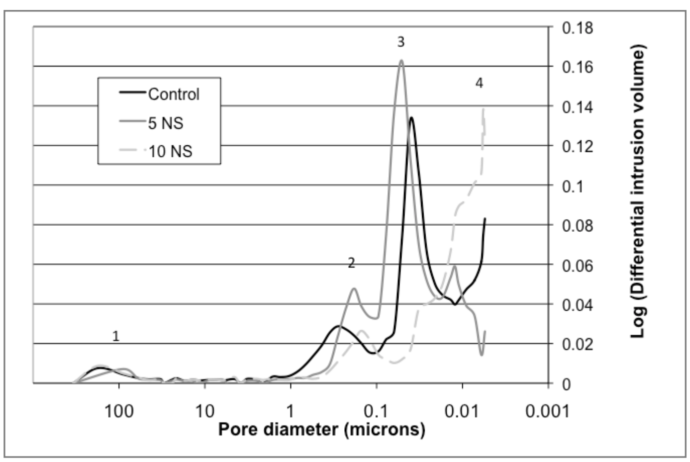

Figure 4. Pore-size distribution of the studied samples

In the samples, four families of pore size can be identified, represented in the graph peaks, where the differential intrusion volume is greater than zero: family 1, around 150 μm diameter; family 2, about 0.2 μm diameter; family 3, around 0.04 μm diameter; and the family 4, about 0.005 μm diameter.

Some authors like Pedeferri and Bertolini [42], Mindess et al. [43], and Cabeza et al. [44] have proposed the following classification for pores according to their size: gel pores under 10 nm (family 4 in this case), capillary pores with diameters between 10 nm and 1.0 μm (families 2 and 3) and pores greater than 1.0 μm (family 1). They emphasized the importance of this classification from the viewpoint of the durability of concrete, since most of the mass transfer through the concrete take place through the capillary pores, whereas the gel pores practically do not contribute to transport. In this respect, other researchers as Hou et al. [18] have established a linear relationship between the volume of capillary pores and transport properties in cement pastes. Pores larger than 1.0 μm are associated with air voids and consequently they are considered coarse pores.

Curves depicted in Figure 4 show that, for the sample with 10 wt. % of NS, the mineral addition has produced an important pore refinement: decreases the maximum pore diameter of the family 2 (it goes from 0.28 μm to 0.15 μm for the control sample and the sample with 10 wt. % of NS respectively), virtually disappear the pores of family 3 and favors the formation of much smaller pores (family 4 of 5 nm in diameter approximately) with respect to the control sample. This suggests the formation of new phases that fill the pores wholly or partially. From the viewpoint of durability this is very important because the capillary porosity virtually disappears in the sample and this kind of porosity moves toward gel pores. This reduction in the diameter of the pores means that, these pastes have a high capillary impermeability, hindering access of aggressive agents to its interior and increasing resistance to deterioration by chemical action.

In the sample with 5 wt. % of NS a pore refinement was also generated, but not so significantly as in the previous case: 5 wt. % of NS decreased the average diameter of the family 1 (diameter changes from 170 μm to 90 μm in comparison to the control sample) and family 2 (goes from 0.28 μm to 0.18 μm). Moreover, 5 wt. % of NS favors the formation of pores of 50 nm (family 3) and 12 nm (family 4).

This result on decreased volume of capillary pores agrees with the findings of authors like Ghafari et al. [45], which have obtained results in this direction for ultra-high performance concrete (UHPC) blended with NS.

Figure 5 shows the behavior of the pastes during the process of intrusion (continuous line) and extrusion (dashed line) of the mercury in the porosimetry test.

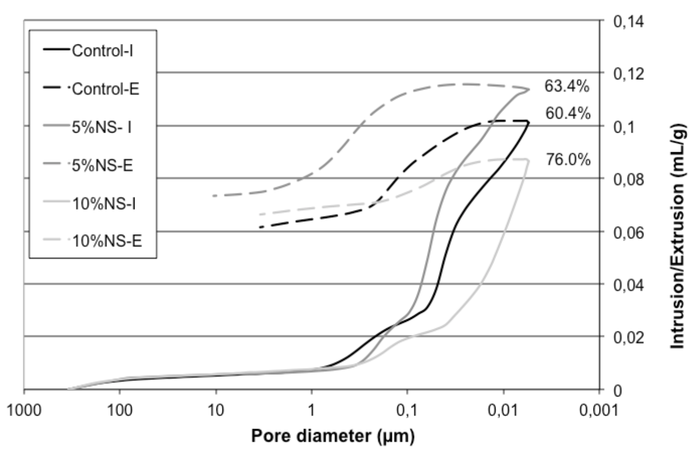

Figure 5. Intrusion (solid lines) and extrusion (dashed lines) curves in the MIP tests

In Figure 5 one can see that for pore diameters less than 0.3 μm, the intrusion curve for the sample with 5 wt. % of NS (5%NS-I), has a higher slope, showing that, in this range of diameters its pore volume is higher. These curves also confirm the decrease in the pore volume and their diameter in the sample with 10 wt. % of NS (10%NS-I), reflected in lower slopes and a rightward shift, i.e. towards smaller pore diameters.

When the combined effect of the mercury intrusion and extrusion in each sample is evaluated (Figure 5), it is seen that, much of the mercury remains into the sample when the pressure is decreased, in the case of this control sample the value was 60.4 %. The effect of this nanoparticle is primarily represented in the increase of the percentage of mercury retained in the extrusion process 63.4 % for sample with 5 wt. % of NS and 76.0 % for the sample with 10 wt. % of NS. The percentage of retained mercury increases with increasing percentage of cement replacement by NS. This behavior suggests a greater tortuosity as a result of the refined pore system. That is, the pores lost some of their connections and suffer a decrease in their diameter, due to the effect of the NS. This effect prevents the mercury, which came under pressure, to easily escape from the network of pores during the extrusion process.

3.3 COMPRESSIVE STRENGTH DEVELOPMENT

Control mortar achieved a compressive strength of 12.85, 24.16, 36.46, and 47.83 MPa at 1, 3, 7, and 28 days respectively. The compressive strength of the mortar containing NS (5 and 10 wt. % replacements) was compared according to the calculation of the improvement in compressive strength as follows (1):

$$I = \frac{Sbs*100}{Scs*C} - 100 \qquad (1)$$

Where:

I: improvement in compressive strength (%)

Sbs: compressive strength of blended sample (MPa)

Scs: compressive strength of control sample (MPa)

C: cement content in the blended sample

In Figure 6 the improvement of compressive strength of the blended samples with NS in comparison to the control sample is shown. For all curing times (1, 3, 7, and 28 days), the change was positive, i.e., NS produced a significant improvement of compressive strength for blended samples, at early and late ages. This behavior is a result of high pozzolanic activity of NS, which allows an increase the quantity of C-S-H. This increase of C-S-H plus the filler effect of NS, modifies the network of pores. The improvement of compressive strength is significantly higher in samples with 10 wt. % of NS, they achieve an increase of almost 120 % in comparison to the control sample after 1 curing day and at other curing ages the increment is close to 80 %. These results are consistent with those found in capillary suction and MIP, where 10 wt. % of NS produced higher pore refinement.

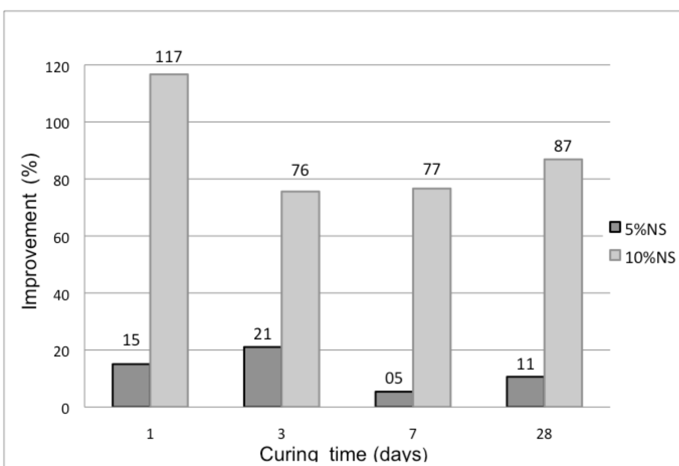

Figure 6. Improvement of the compressive strength for samples blended with NS

3.4 SULFATE RESISTANCE

Control mortar and mortars with NS (1, 3, 5, and 10 wt. % of replacements) were exposed to sulfate attack by immersion in 5 % $MgSO_4$ solution for 154 weeks. The expansion was measured each one or two weeks. Expansion values in mortars produced by sulfate attack are graphically shown in Figure 7. As can be seen, the expansion decreased as the percentage of cement replacement by NS increased. In the last week (154) with the replacement of only 1 wt. % of NS a 49 % reduction in expansion was achieved, with 3 wt. % of NS the expansion was 63 % lower, and with 5 wt. % of NS the expansion was 83 % lower in comparison to the control sample. For the samples with 5 and 10 wt. % of NS, the expansion after 100 weeks of immersion was not very important (0.29 and 0.15 % respectively) compared to 2.82 % that the control sample expanded, namely, mortars decreased expansion by 90 and 95 %, respectively. These expansion values for NS mortars were much lower than expected by the dilution effect. From this it can be stated that, with only 5 wt. % of cement replacement by NS the expansion can be controlled in mortars subjected to $MgSO_4$ attack.

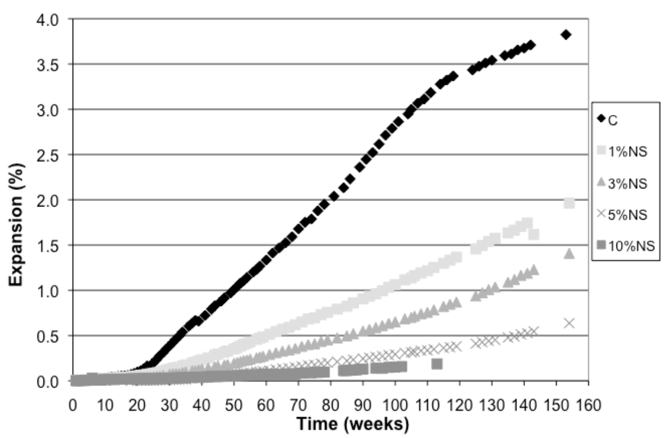

Figure 7. Expansion of mortars immersed in 5 % magnesium sulfate solution

The behavior of mortars with and without cement replacement by NS was equal in terms of expansion up to approximately 20 weeks (Figure 7), thereafter the expansions increased at different rates in accordance with the mixture. Expansion value of 0.1 % has been identified as important for possible initial degradation; this value is achieved for control sample at 20 weeks, for 5 wt. % of NS sample at 57 weeks and for 10 wt. % of NS sample at 77 weeks (Figure 7), this shows that, NS can make an Ordinary Portland Cement (OPC) resistant to sulfate attack (ACI C201-2R).

Percentages of expansion according to the NS replacement ratios in mortars for weeks 25, 50, 75, and 100 of immersion in 5 % $MgSO_4$ solution are shown in Figure 8.

Samples with NS exhibited a decreasing trend of expansion with increasing substitution percentage, which leads to a very low expansion, for the sample with 10 wt. % of NS. Even after 100 weeks of immersion, NS produces an expansion decrease above 60 % just with a 1 wt. % substitution and an expansion decrease close to 95 % with a 10 wt. % substitution (Figure 8).

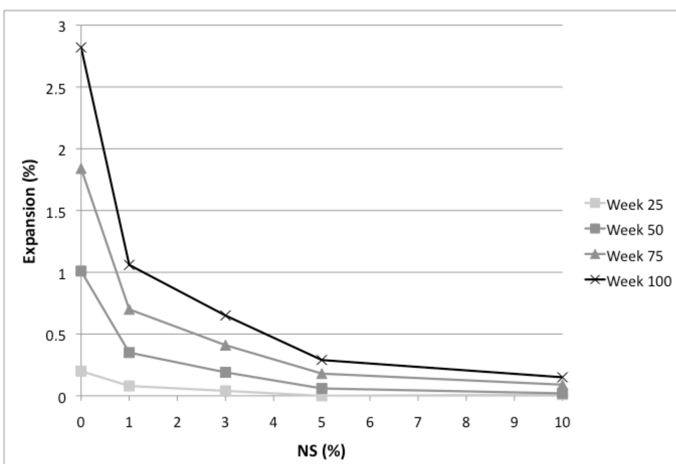

Figure 8. Correlation between the percentage of replacement and expansion after 25, 50, 75, and 100 weeks of immersion in 5 % $MgSO_4$ solution

4. FINAL DISCUSSION

Results showed that, from 5 wt. % substitution of cement by NS significantly reduced the total porosity in pastes and mortars of Portland cement, but more importantly the NS produced a pore refining of the cementitious matrices, mainly reducing the percentage of capillary pores and increasing gel pores.

This effect on the pore structure is very important for the performance of mortars, because the size of the pores that most affect the durability and the compressive strength of mortars are the capillary pores, and as it was demonstrated in this research the NS practically eliminated this kind of pores.

Regarding durability, the fact that the gel pore size was increased mean that most aggressive agents cannot penetrate the cementitious matrix, due to that the pores have very small diameter. This happens to $MgSO_4$, which is unable to penetrate the cement matrix and cannot break the C-S-H to generate expansive salts, for this reason the mortars with NS have lower expansion after the aggressive attack of these sulfates in comparison with mortars of OPC without NS. Furthermore, thanks to pore refining, NS reduces the absorption rate, which means that, any damage that might occur in mortar will be slower over time.

5. CONCLUSIONS

Nanosilica particles produced a significant refinement of pores, defined as the breakdown of interconnections among them and decreased the total pore volume, which resulted in a lower absorption rate and a smaller total absorption of fluids by mortars.

It is noteworthy that the pore sizes that are most diminished due to the effect of NS are those known as capillary pores, NS helps the formation of pores with diameters below 10 nm, called gel pores, which have a very positive effect on the compressive strength and durability (evaluated by sulfate resistance). With 10 wt. % of NS the capillary porosity practically disappeared.

The improvement of compressive strength is significantly higher in samples with 10 wt. % of NS, after 1 curing day they achieve an increase near to 120 % in comparison to the control sample and at other curing ages the increment is close to 80 %.

In the assessment of sulfate resistance, it was found that, mortars with 5 and 10 wt. % NS decreased expansion by 90 and 95 % respectively after two years of immersion. For this reason, it is possible to say that, this replacement fraction, using this type of NS, is sufficient to control the expansion by $MgSO_4$ attack and to make an ordinary Portland cement a sulfate resistant cement (ACI C201-2R).

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Código de campo cambiado

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