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Compressive strength and Scanning electron microscopy studies on Nanosilica admixtured cement mortar

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Abstract : Nanotechnology is a promising research fields that may significantly improve the mixture design, performance and production of cement based materials. The use of large quantities of cement produces increasing CO_2 emissions, and as consequence the green house effect. A method to reduce the cement content in concrete mixes is the use of silica fines. One of the silica fines with high potential as cement replacement and as concrete additive is nano silica. The aim of this study is to investigate the influence of adding nano silica particles, on the properties of fresh and hardened cement mortar through measurements of setting time, compressive strength, water absorption, porosity, in addition grain structures were examined through Scanning electron microscopy. Nano silica particles with size of 20nm have been used as a cement addition by 5, 7 and 10% by weight of cement content. The results of this experimental study reveal that the incorporation of nano silica enhances the early strength of cement mortar along with enhancement of strength at 28 days. Microstructure results indicate that nano scale silica behaves not only as a filler to improve microstructure, but also as an activator to promote pozzolanic reaction.

Key words cement, nanosilica,, strength, porosity, hydric parameters

1.Introduction

Nanosilica (NS) is a highly active and effective pozzolanic material for producing high performance cement materials. It enhances the cementitious properties like setting time, strength, durability, workability and one way to reduce the CO₂ emissions and global warming¹. The use of NS works to the root of efflorescence problem by eliminating the calcium hydroxide from the hydrating cement system. The setting behavior plays significant role in scheduling the various stages of construction process such as transporting, placing, compacting, finishing and also decide whether cement is set-accelerating or set-retarding nature. The mechanisms of cement hydration and the pozzolanic reactivity of NS were explained by number of authors' using DTA, XRD, SEM, FTIR and Electrical conductivity measurements ²⁻⁶. Further Porosity and hydric test measurements were arguably the most important component of the microstructure of cement paste. The water behavior plays a significant role on admixtured cement samples, particularly how they absorb water and how they dry were evaluated by hydric test ⁷. This hydric property affects compressive strength, permeability and durability of the cement mortar and concrete.

According to the effects of utilizing nano-silica in concrete like improvement of micro-structure, decrease of influence and porosities and also increase of compressive strength, it can be concluded that utilization of nano-silica in concrete will decrease cl ions influence in it. This is approved by the researches of ⁸

Cement nomenclature: C = CaO; $S = SiO_2$; $H = H_2O$; $C_3S = 3CaO.SiO_2$; $C_2S = 2CaO.SiO_2$; $C_3A = 3CaO.Al_2O_3$; $CH = Ca(OH)_2$

Qing, Y.et al researched the affect of nano-silica on hard cement paste in comparison to micro-silica and considered their reactions in cement paste⁹. They showed that the reactivity of nano-silica particles is higher than silica soot.

As far as we are aware, no hydric studies have been reported so far on NS admixtured cement mortar with ground water. The purpose of this paper is to present a detailed study on setting time, compressive strength, porosity and some hydric parameters of NS admixtured cement mortar.

2. Experimental

2.1. Materials

In the present study, a commercial Ordinary Portland cement and NS were used.

2.2.1. Mixing procedure

Nano silica particles with size of 20nm have been used as a cement addition by 5, 7 and 10% by weight of cement content. The water to cement ratio of 0.4 is common for all admixtured cement mortar.

2.2.2. Determination of setting time

Setting time of Ordinary Portland cement and NS admixture samples were measured using a Vicat's apparatus as per the standard procedure ¹⁰ and reported in Table 1.

2.2.3. Determination of Compressive strength

The compressive strength of the mortar cubes at 1 day, 1 week and 4 weeks of Portland cement and NS admixtured cement mortar were determined as per the standard procedure ¹⁰. The compressive strength results are the average of the three samples and reported in Table 1.

NS%	Setting time (h. min)		Compressive strength (MPa) 1 day 1 week 4 weeks		
	Initial	Final	-		
0	4.45	6.35	10.6	18.7	44.3
5	4.15	6.15	21.2	31.5	46.3
7	3.15	5.30	24.5	37.5	54.5
10	2.15	4.30	32.4	40.4	58.4

Table 1. Setting time and Compressive strength of NS admixtured cement mortar

2.2.4. Determination of Hydric parameters

Porosity of the mortar cubes made with Portland cement and NS admixtured cement mortar were determined at 1 day, 1 week and 4 weeks by oven drying method ¹¹ using the relation $((M_s-M_o)/(M_s-M_h)) \times 100$. The following hydric properties were obtained using the formula: Water absorption $(100 \times (M_t-M_o)/(M_s-M_h))$, Water absorption coefficient $((M_t-M_o)/t)$, Apparent density $(M_o/(M_s-M_h))$ and True density $(M_o/(M_s-M_h))$. Where M_t is the mass of the wet sample when in contact with water for a period of time t, M_s is the

mass of the saturated test sample, M_o is the mass of the dried test sample, M_h is the hydrostatic weight of the saturated test sample⁷. The computed results are shown in Fig. 1-3.

3. Results and discussion

3.1. Setting time measurements

The initial and final setting time of the Portland cement and NS admixtured cement paste with different waters are shown in Table 1. The setting time of the 5% NS admixtured cement paste are shorter than Portland cement paste. Because NS reacts rapidly with sulphate and produce high amount of calcium sulphoaluminate (ettringite, AF_t) in the early hydration period and hence shortens the setting time ¹². When the NS adding level is increased to 7% and 10%, there is a marginal reduction in setting time are observed compared with that 0% and 5% NS admixtured cement paste. This is due to greater water demand at higher NS percentage which produces a denser binder phase and hence speed up the setting time

3.2. Compressive strength measurements

The experimental results from compressive strength tests are summarized in Table 1. It is seen that for all age and all percentages of NS admixtured cement mortar shows higher compressive strength than that of the plain Portland cement ¹³. showed that the hydration of tricalcium silicate (C_3S) can be accelerated by addition of nanoscaled silica or C–S–H-particles. The increased rate of strength development is believed to be a result of the formation of additional reaction products, which produced the increased pore refinement. The additional reaction product may be C-S-H gel, AF_t and monosulphate.

3.3. Hydric test

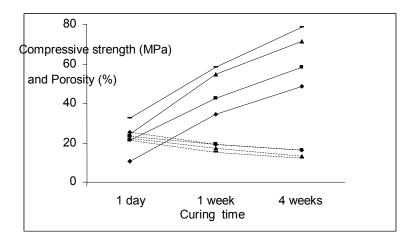


Fig. 1. Plots of compressive strength (---) and porosity (---) against curing time for NS admixtured cement mortar

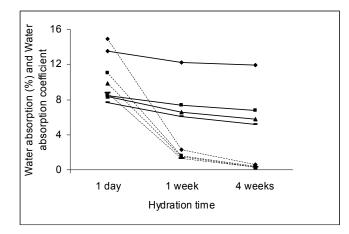


Fig. 2. Plots of water absorption (----) and water absorption coefficient (----) against hydration time for NS admixtured cement mortar

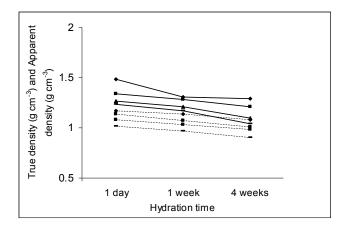


Fig. 3. Plots of true density (----) and apparent density (----) against hydration time for NS admixtured cement mortar

The results of the compressive strength and porosity *vs.* curing times for all mortar samples are shown in Figs. 1-3. Compressive strength [Fig. 1] increases with increase in NS percentage and consequently decrease in percentage of porosity are observed. This is due to high reactivity, additional C-S-H gel formed as a result of the pozzolanic reaction will have the effect of filling pores, thus reducing total porosity and increasing pore refinement. This due to high reactivity, fineness, pozzolanic reaction of NS will result the formation of reaction products within the capillary pore spaces and it increases the binding capacity of the paste.

NS level is increase the WA and WAC decreases with increasing curing time. This phenomenon is mainly dependent on capillary pores in cement paste ¹⁴. The replacement of NS, decreases/or refine the pores and hence decrease the absorption by capillary rise. At 1 day, the samples reached the saturation and there is a large difference in the WAC for different percentage of NS. At 4 weeks the difference is greatly reduced due to porous system.

3.4. Scanning Electron Microscopy

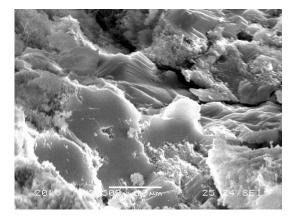
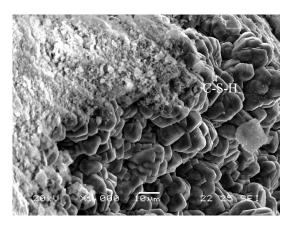


Fig. 4. SEM micrograph of OPC mortar at 4 weeks

Figs.4. shows, SEM pictures obtained for the specimens containing OPC admixtured cement mortar at 4 weeks. In OPC hydration products such as Ca(OH)₂ (plate shape), C-S-H gel (fibrous shape) are well observed ^{15, 16}. CH phase appears according to a well-crystallized habit, stacked as parallel layers are seen.

With the addition of NS to OPC mix, there also takes place the reaction of the calcium hydroxide produced by the hydration of the calcium silicate in cement with the silica and alumina reactivities in the pozzolanic materials. This pozzolanic reaction produces additional C-S-H gel, which grow into the capillary spaces and significant reductions in porosity ¹⁶.

With increase the percentage of NS resulting the voids are Filled with hydration products and hence the apparent density and true density are increases [Fig 5-7] The best results for hydric tests, porosity measurements, compressive strength and microstructures are used as a quality index for construction materials, especially to the NS admixtured cement mortar.



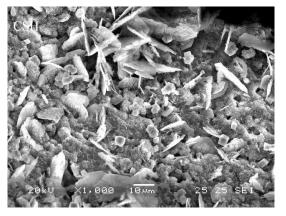


Fig. 5. SEM micrograph of OPC+ 5% NS admixtured cement mortar at 4 weeks

Fig.6. SEM micrograph of OPC+ 7% NS admixtured cement mortar at 4 weeks

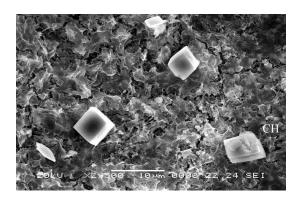


Fig.7. SEM micrograph of OPC+ 10% NS admixtured cement mortar at 4 weeks

4. Conclusions

The conclusions obtained from the results can be summarized as follows

- 1. As NS% level increases, it enhances the compressive strength and reduces the setting time and porosity compared to OPC.
- 2. Nano-silica consumes calcium hydroxide crystals, reduces the size of the crystals at the interface zone and transmute the calcium hydroxide feeble crystals to the C-S-H crystals, and improves the interface zone and cement paste structures.
- 3. Cement mortar containing nano-silica have more homogeneity binder, less pores, more adhesion at interfacial zone which is clarified in SEM analysis.
- 4. The water absorption of was found to be increased with increasing curing time in OPC and reduced with the addition of NS. A strong correlation
- 5. Compressive strength, Porosity and Hydric studies are confirmation of the well known accelerating effect of NS in the hydration of Portland cement. SEM

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