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INVESTIGATING THE EFFECT OF NANO SILICA ON THE MECHANICAL PROPERTIES OF HARDENED CEMENT PASTE

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This study investigated the compressive strength of hardened cement paste and the formation of calcium silicate hydrate (C-S-H) with the addition of nano silica (SiO_2). Through this search, the development of the concretes strength was determined to better understand the process of cement hydration. Compressive strength testing was performed using MTS and Forney testing machines to determine stress-strain curves and elastic modulus of materials. The hydration process and formation of C-S-H and calcium hydroxide (CH) was examined using Scanning Electron Microscopy (SEM) and Fourier Transform Infrared Spectroscopy (FTIR). This study also incorporates the use of vacuum curing, in comparison to that of the traditional water curing method. Results indicate an increase in compressive strength using 1%, 3% and 5% of nano silica to cement replacement by volume in comparison to the control mix (without nano silica). The optimum cement replacement to yield maximum strength was of the 1% nano silica content. The formation of C-S-H increases significantly during the early testing days which correspond with the drastic increase in compressive strength. The hydration process continues to increase throughout the 56 day trails at a moderate rate. The traditional water curing method proves to be more efficient and beneficial than of the vacuum curing method. However, vacuum cured results showed only about a 5% reduction in strength after 56 day tests in comparison to the water curing method.

Keywords: Nano-silica, SiO_2, Mechanical properties, FTIR monitoring, SEM monitoring, CSH growth, Curing methods.

1 INTRODUCTION

In recent years, environmental aspects of material conversion have induced research to modify Portland cement to support the increasing demands for sustainability in the construction division. Such change can be obtained by using additives to change the chemical composition of the cement (Ylmén *et al*. 2009). Nano silica is an emerging field of interest in civil engineering application. Nano silica has been proven to be more efficient in enhancing concrete strength than that of silica fume (Bi *et al*. 2012). Experimental results of Givi and his co-researchers showed that nano-particles blended with concrete increased the compressive, flexural, and tensile strength at all curing ages (Givi *et al*. 2010). Due to pozzolanic reaction and a reduction of calcium hydroxide, nano silica has been proven to improve performance of cement-based materials through the increase production of C- S-H gel. It has the capability to react with the free lime during the cement hydration process and forms additional C-S-H gel, giving strength, impermeability, and durability to concrete. It has also been found that the rate of pozzolanic reaction caused by nano silica is proportional to the amount of surface area that is available for reaction (Jo *et al*. 2007). The main objective of this study is to investigate the effect of nano silica in cement on the hydration process and on the mechanical properties of cement paste over the course of 56 days. The study investigated the hydration process of Portland cement with increments of nano silica addition by cement replacement, monitored by Fourier Transform Infrared Spectroscopy (FTIR) and by Scanning Electron Microscope (SEM). The results of FTIR spectroscopic signatures were compared to the represented samples examined under SEM. Correlation of the spectroscopic features to the development of concrete strength was also determined. Strength testing was carried out by using Forney Universal Testing and MTS machines for compressive strength and modulus of elasticity. The investigation also studied the effects of water curing in comparison to vacuum curing. Concrete cured in airless environments or vacuum curing, is a new area of interest and was examined in comparison to the universally used water curing approach.

2 EXPERIMENTAL PROGRAM

Approximately 211 specimens were prepared to conduct several tests using 4 in. by 8 in. cylinders and 2 in. cubes. The cementitious materials used for this study were Portland cement Type 1 and nano silica. Polycarboxilate polymer based superplasticizer was added in conjunction with the nano silica. Nano silica is silicon oxide nanoparticles ($SiO2$) synthetic product of porous and nearly spherical particles. With the combination of superplasticizer, nano silica has been proven to perform as a viscosity modifying agent to produce high performing concrete. The particle size of nano silica ranges from 15-20 nm. For the experimental setup, four (4) mix designs were created. The first mix, M1, was the control mix with cement and water. The next three mixes, M2, M3, and M4, consisted of a 1, 3, and 5 percent of cement replacement by volume of nano silica respectively. The water to cement (w/c) ratio was 0.27 using superplasticizer. For the control mix, M1, eight (8) cylinders and six (6) cubes specimens were prepared for each curing condition. Five (5) cylinders and six (6) cubes were manufactured for M2 and M3. For mixes M2 and M3, four of the cylinders were placed under water curing and one cylinder was placed under vacuum curing. For M4, two (2) cylinders were manufactured, each under separate curing, and six cubes. All of the cubes manufactured were separated in half for each curing method, three cubes under water and three under vacuum. The cylinders were comprised of plastic molds, 4 inches in diameter and 8 inches in depth, in compliance with ASTM C-470. The cube modes were comprised of brass, exactly 2 inches in size, and able to form three cubic specimens within each mold.

2.1 Mechanical Properties

The Forney machine was used for cylinder compression and the MTS used for cube compression. MTS 810 Landmark Servohydraulic Testing system was used. These systems include MTS software, FlexTest controls, MTS servohydraulic technology, and a complete selection of accessories. The load rate for the MTS machine was 0.01 in/s. The Forney Universal Testing Machine was used to carry out all cylinders testing, including compression and modulus of elasticity. The Forney machine has a 400 kip load capacity and a load rate of 12,000 lbs/min.

2.2 Scanning Electron Microscope (SEM)

SEM EVO LS made by ZEISS, was used for microstructure characterization and analysis of the hardened cement paste specimens. EVO LS has full environmental capabilities to capture nano scale interactions of samples under various pressures, temperatures, and humidities. During experimentation, the SEM was set into the environmental mode (EP) which allows atmosphere in the chamber since the sample is based on hydration. The scanning speed was set to four, which automatically changed the scanning cycle to 731 ms. The EP target set to 50 Pa, the KV value was set to 10 KV and the beam current to 46.0 µA.

2.3 Fourier Transfer Infrared Spectroscopy (FTIR)

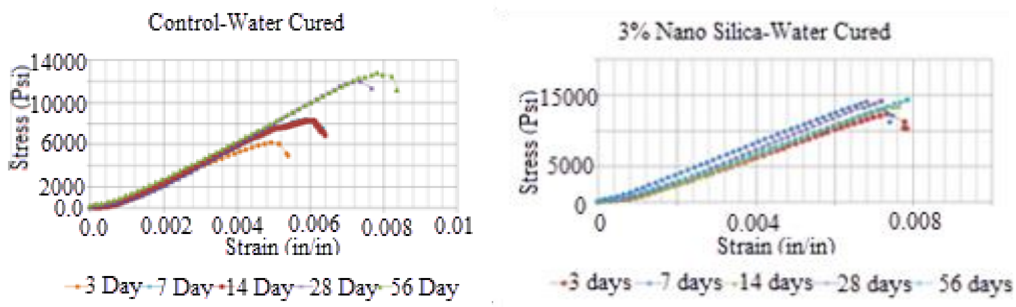
FTIR examines the chemical properties of the material and monitors the changes in mechanical properties as the reactions take place. In this study, Thermo Scientific Nicolet iS10 FT-IR Spectrometer and OMNIC Specta software were used to perform FTIR monitoring. Several steps were followed to carry out FTIR testing. This includes: sample preparation, background check to subtract traces of dissolved gases and solvent molecules, placing the specimen onto the FTIR plate and collect at least 5 spectrums for each sample, the average of all the spectrums collected were recorded and used for results and discussion. After each sample spectrum has been obtained, data analysis was carried out and observed using the OMNIC software. For each sample, the absorption frequency bands assigned to each sample spectrum was observed for normal modes of vibrations within the molecules. These spectrums were later analyzed and compared within the discussion.

3 EXPERIMENTAL RESULTS

In this study, the hydration process of Portland cement with additives of nano silica has been monitored. With the use of SEM and FTIR, signatures of C-S-H which produce most of the concretes' strength, has been determined and examined from 3 to 56 days. Also, using Forney and MTS testing equipment, the overall strength of the harden cement pastes at all testing ages were determined and recorded for analysis.

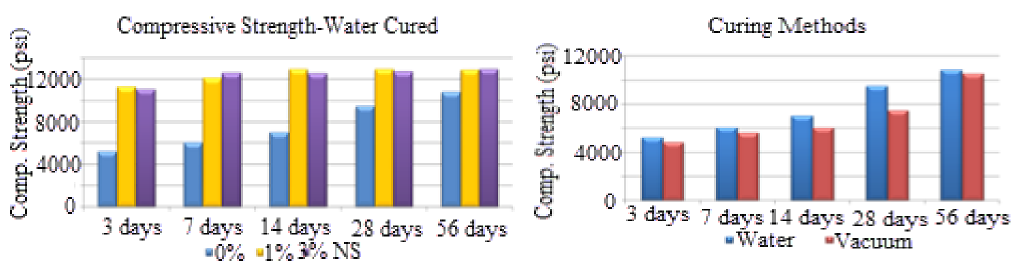
3.1 Compressive Strength

Stress-strain curves were developed from each cube tested with the MTS Landmark Servohydraulic testing system. As stated in the experimental procedure, each material was casted into three cubes; control 0%, 1%, 3% and 5% of nano silica to cement replacement for water and vacuum curing. The stress-strain curves created from the three cubes were averaged together to obtain a single averaged stress-strain curve for each material tested. Although more than twenty figures were produced, Figure 1 displays (a) the stress-strain curves for the control samples for all days and water cured, and (b) the 3% of nano silica to cement replacement for the water cured samples. The Figure shows an increase in strength from the 3 day tests, ascending to 56 day tests. Figure 1(b) shows that with the incorporation of nano silica, the stresses at all testing ages are much greater than that of the control samples. The stresses for the 3% nano silica samples range between 12,300 psi for the 3 day results and 14,346 psi for the 56 day results. The Forney Universal Testing Machine was use to conduct all cylinder compressive strength tests for each sample. Again, several graphs were produced but Figure 2(a) shows the average compressive strengths for all water cured testing cylinders at ages 3, 7, 14, 28 and 56 day tests. As shown in the figure, the 1% and 3% nano silica samples perform significantly higher than that of the controlled. Figure 2(b) displays the average cylinder strength of the 0% controlled samples that were water and vacuum cured at all testing ages.



(a) (b)

Figure 1. Stress-strain curves: (a) controlled-water cured and (b) 3% nano silica-water cured.

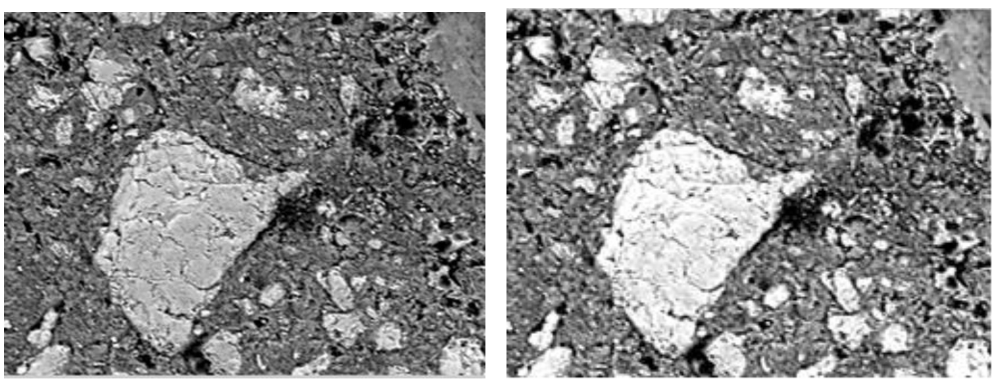


(a) (b)

Figure 2. Compressive strength results for: (a) all water cured samples and (b) curing methods.

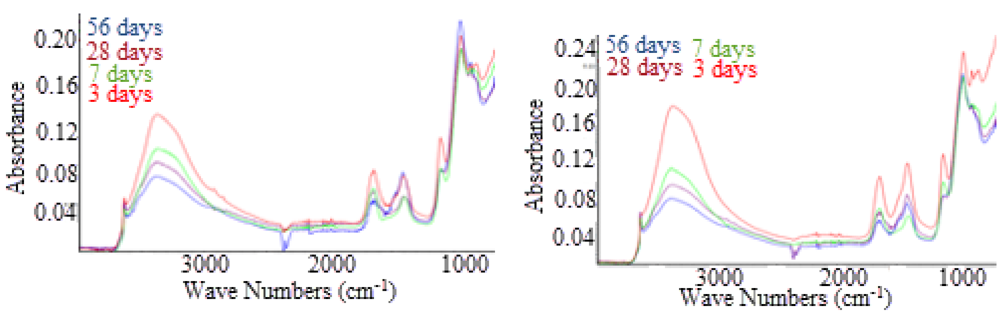
3.2 Scanning Electron Microscope (SEM)

The SEM was used to obtain a microscopic image of samples at different ages. From the images obtained, a further analysis using MatLab was conducted to filter the images using a Gaussian filter and to determine the percentages of chemical products for which that image portrays. From grey level imaging, four main phases were identified: porosity (P), calcium hydroxide (CH), calcium silicate hydrate (C-S-H), and unhydrated products (UP). Other products such as limestone and other hydrated products were not distinguished. Figure 3(a) shows the original SEM image of harden cement paste sample containing 1% nano silica after water curing and tested at 7 day. Figure 3(b) shows the same sample after filtered using the Gaussian filter. Features from this image are lighter and more distinguished. From the results gathered from the MatLab analysis of all testing samples, a table was generated to portray the results obtained. Results show an increase in C-S-H formation from 3 day to 56 day for the control and all nano silica mixtures. Results show a maximum amount of C-S-H formation in the 5% nano silica samples than that of the 1% and 3% nano silica samples. Also, at all testing ages, samples with nano silica out performed or contains more C-S-H content than the control. Also, results from vacuum cured specimens show similar results to the water cured ones. The amount of C-S-H found in the vacuum cured control samples and the 3% and 5% nano silica samples recorded nearly exactly the same percentage during the 56 day results, in comparison to the water cured samples at 56 days.



(a) Unfiltered SEM image (b) Filtered SEM image

Figure 3. SEM image of a7 days water cured 1% nano silica section.

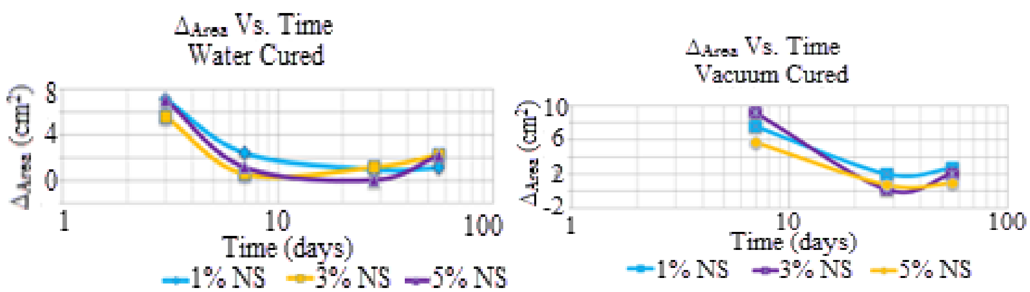


(a) (b)

Figure 4. Absorbance of control: (a) water cured and (b) vacuum cured samples, 3 to 56 day tests.

3.3 Fourier Transfer Infrared Spectroscopy (FTIR)

The hydration process was monitored for 3, 7, 28, and 56 days, by acquiring an FTIR spectrum for each sample under water and vacuum curing. The recorded spectra for the water and vacuum cured samples are displayed in Figures 4(a) and 4(b) respectively. The curing days increase from 3 to 56 days, from bottom to top of the spectra. From previous literature, signatures of hydration can be seen within the 900 to 1200 cm^{-1} region. Also notice the large peak in the area of 3400 cm^{-1}, which can be associated with hydrogen bond (O-H) or capillary water within that region. The remaining spectra for the 1%, 3% and 5% nano silica spectrums for both water and vacuum cured are collected but not shown. Once obtaining the possible C-S-H formation from each sample, further analysis was conducted. The amount of C-S-H found within each sample various amongst the different percentages of nano silica. Results show a drastic increase in C-S-H found from the 28 day to 56 day control samples. Figure 5(a) illustrates the change in area of C-S-H of 1%, 3% and 5% of nano silica replacement water cured control, while Figure 5(b) shows the change of C-S-H area in samples cured in vacuum. With the incorporation of nano silica, the hydration process tends to follow a similar pattern to that of the control mix. The data shows a significant decrease in hydration after 3 day and from there the hydration tends to increase at a slow pace leading up to the 56 day. The same occurrence takes places during the vacuum cured samples as well.



(a) (b)

Figure 5. Change in area of C-S-H for 1%, 3% and 5% of nano silica (a) water and (b) vacuum.

References

Bi, J., I. Pane, B. Hariandja and I. Imran, The Use of Nanosilica for Improving of Concrete Compressive Strength and Durability, App. Mechanics and Materials, pp 4059-4062, 2012.

Givi, N., S. Rashid, F. Aziz and M. Salleh, Experimental Investigation of the Size Effects of SiO2 Nano-particles on the Mechanical Properties of Binary Blended Concrete, Composites Part B: Engineering 41(8): 673-677, 2010.

Jo, B.-W., C.-H. Kim, G.-h. Tae and J.-B. Park, Characteristics of Cement Mortar with Nano-SiO2 Particles, Construction and Building Materials, 21(6): 1351-1355, 2007

Ylmén, R., U. Jäglid, B.-M. Steenari and I. Panas, Early Hydration and Setting of Portland Cement Monitored by IR, SEM and Vicat Techniques, Cement and Concrete Research 39(5): 433-439, 2009.