



Effect of Nano-silica additions on Mechanical and Microstructure analysis of High Performance Concrete

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Abstract : Concrete is the material of choice where strength, performance, durability, impermeability, fire resistance and abrasion resistance are required. The hunger for the higher strength leads to other materials to achieve the desired results and thus emerged the contribution of cementitious material for the strength of concrete. Addition of pozzolonic admixture like the pulverized Fly ash (PFA) contributes to the improvement of strength and also by adding the Nano materials, concrete composites with superior properties can be produced. Nano Technology applied to concrete includes the use of Nano materials like Nano-silica, Nano fibers etc. The micro-level does not provide enough insights into building materials. Therefore, all around the world, increasing amounts of research funding are being diverted into the Nano level, which is claimed to have tremendous potential for the future. Nano-silica improves the microstructure by making it denser. The objective of this project is to study the mechanical and microstructural properties of M60 high performance concrete with Nano-silica as admixture partially replacing cement in 1%, 2%, 3% and 4%. Specimens namely cubes, cylinders and prism are cured for 14 days in standard environment, after this curing period test to analyze the mechanical properties are carried out. Compressive test, Split tensile test, Flexural test and test for Modulus of elasticity are carried out to study the mechanical properties. The mechanical properties start showing increasing trend with increase in the quantity of Nano-silica. TEM, EDS and SEM techniques are used to study the microstructure of the concrete. The Nano-silica addition reduces the pore amount and makes the concrete denser in microstructure level which in turn increases the mechanical properties.

Keywords: Nano – SiO₂, SEM, EDS.

1. Introduction

Nowadays the micro-level does not provide enough insights into building materials. Therefore all around the world, increasing amounts of research funding are being diverted into the Nano-level, which is claimed to have tremendous potential for the future. Nanotechnology is one of the most active research areas which have wide applications in almost all the fields. The fundamental processes that govern the properties of concrete are affected by the performance of the material on a Nano scale. As concrete is most usable material in construction industry it's been required to improve its quality. Concrete is a highly heterogeneous material produced by mixture of finely powdered cement, aggregates of various sizes and water with inherent physical, chemical and mechanical properties¹³⁻²⁶. Cement can be partially replaced by a number of mineral admixtures such as fly ash, silica fume, metakaoline etc., which have certain properties related to that of cement. By adding the Nano materials, concrete composites with superior properties can be produced. Nano Technology applied to concrete includes the use of Nano materials like Nano-silica, Nano-silica improves the microstructure and reduces the water permeability of concrete thus making it more dense and durable. Use of Nano-silica in HPC

improves the cohesiveness between the particles of concrete and reduces segregation and bleeding. Certain problems like longer setting time, lower compressive strength at higher percentages can be overcome by adding Nano-silica. The addition of supplementary cementitious materials in the concrete will not only improve the mechanical properties of concrete, but also its workability, alteration in setting times and durability. Siva³, performed experimental to study the mechanical properties of high-strength concrete of grades M60 and M70, at 28 days characteristic strength with different (2% and 4%) replacement levels of cement with Nano-Silica. Standard cubes (150mm x 150mm x 150mm), standard cylinders (150mm dia x 300mm height) and standard prisms (100mm x 100mm x 500mm) were considered in the investigation. The mechanical properties viz., compressive strength, flexural strength and splitting tensile strength, and stress-strain characteristics of high strength concrete with various replacement of Nano-silica viz., 2%, and 4%, has been considered. According to Sololev⁴, role of Nano particles of silica act as fillers in the voids or empty spaces. The well dispersed NS act as a nucleation or crystallization centers of the hydrated products, thereby increasing the hydration rate, that is, NS assisted towards the formation of smaller size CH crystals and homogeneous clusters of C-S-H composition. Moreover, they found that NS improved the structure of the transition zone between aggregates and paste.

2. Materials

2.1 Cement

Ordinary Portland cement of 53 grade conforming to IS 8112:1989⁹ of locally available RAMCO cement which comprises good quality. The chemical configuration of cement was found using X-ray fluorescence analysis and has the following properties are given in table 1.

Table 1 shown in properties of cements

Description	Composition
Physical Properties	
Color	Grey
Specific gravity	3.15
Specific surface area (cm ² /g)	3540
Chemical Composition	
CaO (%)	62.8
SiO ₂ (%)	20.3
Al ₂ O ₃ (%)	5.4
Fe ₂ O ₃ (%)	3.9
MgO (%)	2.7
Na ₂ O (%)	0.14
K ₂ O (%)	62.8

2.2 Fine Aggregate

River sand was used as fine aggregate and the fineness modulus of the fine aggregate is 3.32 and it belongs to coarse sand category which can be used for concrete mixing. The specific gravity of the fine aggregate was noted as 2.64. table 2 shown in modulus of fine aggregate.

Table 2. Fineness modulus of Fine aggregate.

IS Sieve (Mm)	Weight retained (Kg)	Percentage Retained (%)	Cumulative percentage (%)	Cumulative percentage finer (%)
4.75	4.75	8	0.53	0.53
2.36	2.36	48	3.2	3.73
1.18	1.18	4	0.26	3.99
0.600	0.6	454	30.26	34.26
0.420	0.43	136	9.06	43.32
0.300	0.3	260	17.33	60.65
0.150	0.15	480	32	92.65
0.075	0.075	12	0.8	93.45
Pan	-	96	6.4	99.85

2.3 Coarse Aggregate

Aggregate passing through 16 mm sieve and retained on 12.5 mm sieve was used as coarse aggregate in the concrete mixture. The specific gravity of the coarse aggregate was noted as 2.65. The fineness modulus of the coarse aggregate is obtained as 6.8.

Table 3 Properties of Coarse aggregate

Properties	Observed values
Fineness modulus	6.80
Specific gravity	2.75
Bulk density	1530 kg/m ³
Loose density	1445 kg/m ³
Aggregate crushing value	29%
Aggregate impact value	32%
Maximum sie of aggregate	12.5 mm
Water absorption	0.15%

2.4 Nano SiO₂

Pure Nano-silica was brought and its properties are tested in manufacture company give by table 4 as shown and SEM figure in figure 4 and 5.

Table 4 Properties of Nano-silica

Test Item	Standard Requirment	Test Result
Specific surface area(M2/G)	200-210	202
pH value	3.7-4.5	4.12
Loss on drying @105 ⁰ c (5)	<1.5	0.47
Loss of ignition @1000 ⁰ c (%)	<2.0	0.66
Sieve Residue (5)	<0.04	0.02
SiO ₂ content	>99.8	99.88
Carbon content	<0.15	0.06

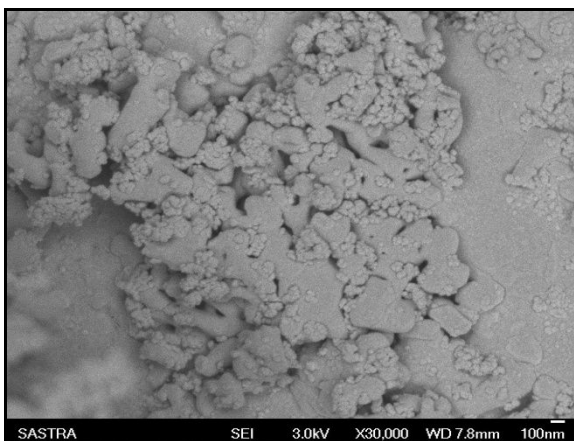


Figure 1 SEM of Nano SiO₂

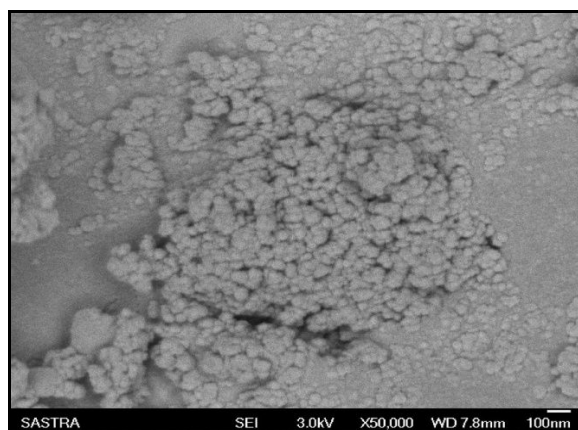


Figure 2 SEM of Nano-silica in 100nm

2.5 Super plasticizer

Complast SP430 (G) is used where a high degree of workability and its retention are required, here delays in transportation or placing are likely or when high ambient temperatures cause rapid slump loss. It facilitates production of high quality concrete.

2.6 Water-cement ratio

The water cement ratio was kept at 0.33, as the percentage of silica fume increased; the requirement of water required also increased.

3. Experimental Program

3.1 TEM test on Nano-Silica

Transmission electron microscopy (TEM) is a microscopy technique in which a beam of electrons is transmitted through an ultra-thin specimen, interacting with the specimen as it passes through it. An image is formed from the interaction of the electrons transmitted through the specimen; the image is magnified and focused onto an imaging device, such as a fluorescent screen, on a layer of photographic film, or to be detected by a sensor such as a CCD camera. TEMs are capable of imaging at a significantly higher resolution than light microscopes. the instrument's user to examine fine detail—even as small as a single column of atoms, which is thousands of times smaller than the smallest resolvable object in a light microscope. TEM forms a major analysis method in a range of scientific fields, in physical, chemical and biological sciences. The sample TEM result showed in figure 3.

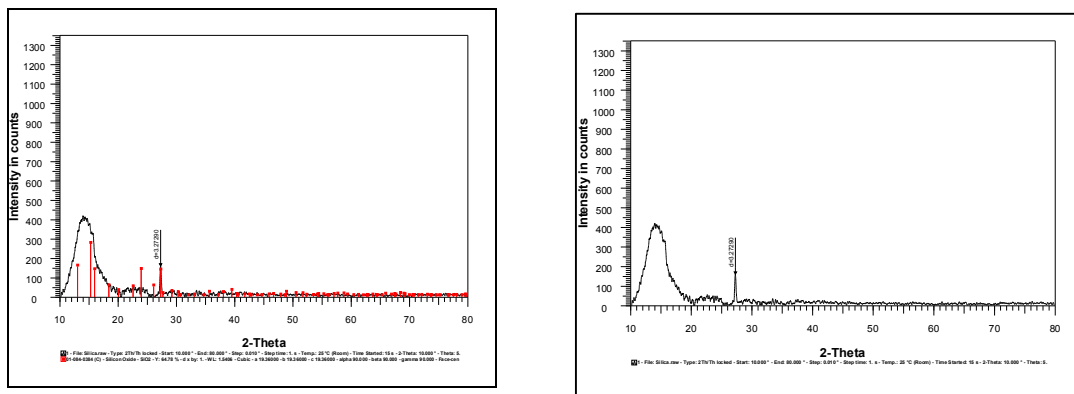


Figure 3 Tem Analysis

3.2 Concrete Mix design

The mix design can be defined as the process of selecting the suitable ingredients of concrete and determining their relative proportion with the object of producing concrete of certain minimum strength and durability as economically as possible. The purpose of designing the mix is to achieve the stipulated minimum strength and to make the concrete in the most economical manner.

3.3 Casting Details

Concrete cubes of size 100 mm x 100 mm x 100 mm were cast to study the compressive strengths of cubes replaced with Nano-silica after subjecting to curing in normal environment. Strength tests of cubes were performed in automatic compression testing machine with a capacity 3000 kN.

3.4. Cylinders

Cylinders of size 300 mm x 100 mm were casted study the effect of Split tension and Modulus of elasticity in Compression testing machine.

3.5 Prisms

Prism of standard size 500x 100x 100 mm is casted to study the flexural behavior in universal testing machine for two points loading.

3.6 Curing Environment

All the specimens were subjected to a normal curing for 14 days in water, and then the specimens are taken out and dried before testing.

4 Test Procedures

4.1 Compressive strength test

The compressive strength test is the most common test conducted because most of the desirable characteristic properties of concrete and the structural design purpose are qualitatively related to compressive strength. The test was conducted in compression testing machine of 3000kN capacity for different ages of concrete viz. 7, 14 and 28 days as per the specifications given in IS 5816: 1999 under normal room temperature and figure 4 shown in experimental setup of compression, spilt tension, flexural strength.



Figure 4. Test setup for Compressive, Spilt tension, Flexural strength

4.2 Split tensile strength test

This is an indirect test to determine the tensile strength of cylindrical specimens. Splitting tensile strength tests were carried out at the age of 28 days for the concrete cylinder specimens of size 150 mm diameter and 300 mm length, using compression testing machine of 3000kN capacity as per IS:5816-1999. The load was applied gradually till the specimen splits and readings were noted.

4.3 Flexural strength test

Flexure test was done on beam with universal testing machine (UTM) at a loading rate of 1.8 KN/min. The test was done at the age of 28 days for the casted beams. The flexural strength test was determined using

the relationship $F_b = \frac{PL}{BD^2}$

4.4 SEM and EDX Results

A scanning electron microscope (SEM) is a type of electron microscope that produces images of a sample by scanning it with a focused beam of electrons. The electrons interact with atoms in the sample, producing various signals that contain information about the sample's surface topography and composition. The electron beam is generally scanned in a raster scan pattern, and the beam's position is combined with the detected signal to produce an image. SEM can achieve resolution better than 1 nanometer. Specimens can be observed in high vacuum, in low vacuum, in wet conditions (in environmental SEM), and at a wide range of cryogenic or elevated temperatures. SEM test is done on 2% and 4% Nano-silica partially replacing cement. EDS relies on an interaction of some source of X-ray excitation and a sample. Its characterization capabilities are due in large part to the fundamental principle that each element has a unique atomic structure allowing unique set of peaks on its X-ray emission spectrum.

5. Results and Discussions

5.1 Compressive strength test

Three specimens of size 100 mm x 100 mm x 100 mm were used for compression testing for each batch of mix. Clean and surface dried specimens were placed in the testing machine. The platen was lowered and touched the top surface of the specimen the load was applied gradually and maximum load was recorded. Figure 4 to 5 as shown in mechanical properties of nano silica.

Table 5 shown in Mechanical Properties of Nano SiO₂

Sample Name	% of Nano-Silica	Compression Strength (MPa)	Split tensile strength (MPa)	Flexure strength (MPa)	Modulus of Elasticity (10 ⁴ Mpa)
MCA	0	53.03	5.05	5.09	4.17
MCB	1	56.12	5.06	5.11	5.13
MCC	2	57.31	5.09	5.31	5.82
MCD	3	44.60	3.84	3.62	4.41
MCE	4	39.81	3.19	2.11	3.74

5.2 Split Tensile strength Test

Split tensile test was conducted on cylinders of size 150mm diameter and 300mm height. Clean and surface dried specimens were placed in the testing machine. The platen was lowered and was allowed to touch the top surface of the specimen. The force was applied and maximum load at which the specimen failed was recorded. Table 5 shown in mechanical properties of nano silica.

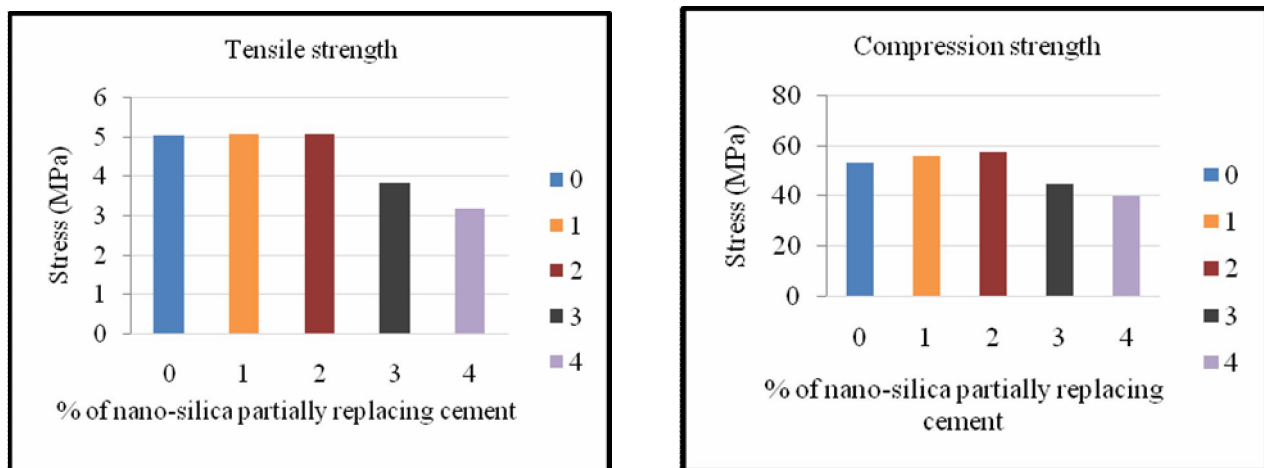


Figure 5 Tensile and Compressive strength of Nano-silica

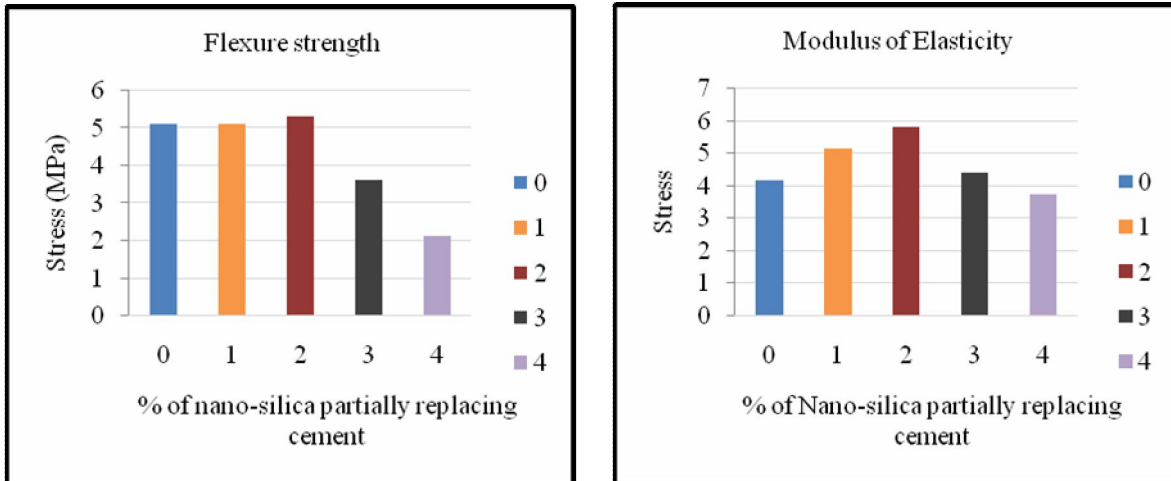


Figure 6 Flexural strength and Modulus of Elasticity of Nano- Silica

5.3 SEM and EDS Results

A scanning electron microscope (SEM) is a type of electron microscope that produces images of a sample by scanning it with a focused beam of electrons. The electrons interact with atoms in the sample, producing various signals that contain information about the sample's surface topography and composition. The electron beam is generally scanned in a raster scan pattern, and the beam's position is combined with the detected signal to produce an image. SEM can achieve resolution better than 1 nanometer. Specimens can be observed in high vacuum, in low vacuum, in wet conditions (in environmental SEM), and at a wide range of cryogenic or elevated temperatures. SEM test is done on 2% and 4% Nano-silica partially replacing cement. EDS relies on an interaction of some source of X-ray excitation and a sample. Its characterization capabilities are due in large part to the fundamental principle that each element has a unique atomic structure allowing unique set of peaks on its X-ray emission spectrum.

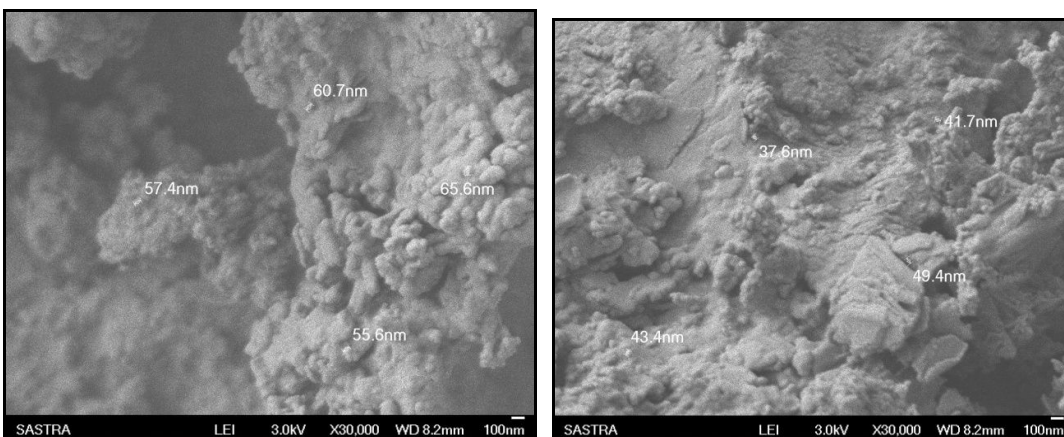


Figure 7 SED result of Concrete with 4% Nano-silica partially replacing cement

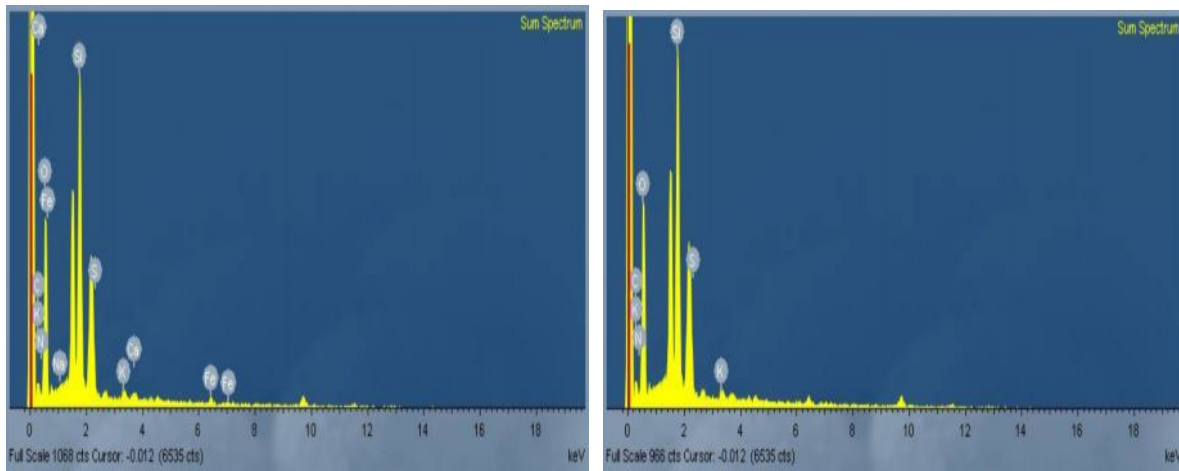


Figure 8 EDS graph for Concrete with 4% Nano-silica partially replacing cement

6. Conclusions

The following were the conclusions arrived from the experiments conducted on specimens in Standard environment.

- The above discussions described that the influence of NS along with cement, concretes, supplementary cementitious materials and other cementitious materials. Considerable improvement in the properties of permeability, pore filling effects, microstructure analysis and strength were reported. As a whole, the entire review showed the ultimatum in using the Nano technology in general and Nano silica in particular. However, there is a gap or room available for further research towards the fruitful application of Nano silica for construction with different Nano structure characterization tools, which will be enable to understand many mysteries of concrete.
- The compressive strength of concrete had shown an increasing trend with the increase in the quantity of Nano-silica but the increment was stopped when the Nano-silica was beyond 3%.
- The percentage increase in compressive strength and split tensile strength of concrete with the Nano-silica at 3% is 14% more compared to control concrete.
- The increase in flexural strength is only 2% at 3% Nano-silica partially replacing concrete compared to control concrete.
- The strength of concrete has drastically decreased by 50% when the Nano-silica is at 4%.
- It has also been observed that for combination of 4% Nano-silica the strength is decreased as the dosage might have been crossed the optimum level. Therefore the optimum amount of Nano-Silica partially replacing cement is 3%.
- Nano-silica in high performance concrete cause to reduce in pores size and the concrete structures will be more dense and durable.

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