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Strength, Durability & Permeability Studies on Cement Concrete with & without Nano-Silica

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Abstract

The application of nanomaterials in cement/concrete has resulted in the development of smart materials which are durable, sustainable and high performance oriented apart from being eco-friendly. Several types of nanomaterials are being used by researchers all over the world to improve the properties of cementitious system, amongst them silica nanoparticles (SNPs) have been credited with high pozzolanic reactivity and pore filling effects. It is also very well known that permeability is the major factor for deteriorations of concrete structures in the long term thus affecting its durability. A thorough study was performed on cement mortar composites and standard grade concrete with the additions of colloidal silica nanoparticles(SNPs) in various dosages starting from 0.5%,0.75%,1.0%,1.25% and 1.5% by weight of cement(b.w.c.) to understand the impact from micro-structure improvement to macro-level property enhancement. The results show that in presence of colloidal SNPs (Indian branded) having 35-40% active nano content with 5-40nm particle size the strength increase at 28 days is 32% increasing to 59% at 90 days. An influence on durability is also observed with strength increasing from 4% to 8% with the addition of more SNPs from 0.75% to 1.0% b.w.c. The microstructural investigations reveal that silica nanoparticles refines the pore structure due to accelerated hydrated mechanisms leading to the creation of more hydration products and thus to a more denser microstructure. A M-40 grade standard concrete was prepared as per IS:10262 with the addition of optimized SNPs as found for cement mortar composites strength results at 28 days maximum strength keeping the water-cement(w/c) ratio fixed at 0.4. Tests were conducted on concrete to find out the compressive strength and permeability of concrete. The compressive strength of concrete containing nano materials were found to be 24% more than that of normal cement concrete (NCC) of M-40 grade. The permeability values were found to be decreased by 57% with the addition of nano silica particles to concrete.

Keywords: Cement, Concrete, Durability, Nanomaterials, Permeability, Strength, Structure

1. Introduction

The word 'silica' has a very broad meaning; it includes silicon dioxide, the main component of earth's crust, in all its crystalline, amorphous, soluble or chemically combined forms where the silicon atom is surrounded by 4 to 6 atoms. From macroscopic scale to microscopic scale, silica has shown to have a profound influence on cement hydration which occurs at nanoscopic scale. But Nanosilica is different from the standard pozzolanic materials like flyash or silica fumes in both particle size & method of application as it follows the nanoscale (1-100nm) as described by ACI-241R-17. Though concrete has remained the most popular and used construction material in all ages, nanotechnology or nanomaterials in concrete has become a new novel field showing the potential to revolutionize the cement & concrete industry. A common method to make concrete stronger & durable is by using pozzolanic materials like flyash & silica fumes which are the suppliers of free silica in the matrix. While nanosilica particles have been used in a wide range of concrete applications, they have not been studied to the extent as that of previous generation of pozzolanic materials that measure along the micron scale or more. The reactivity of nanosilica is due to surface Si-OH groups as shown in Fig.1, which cover the nano-particle and constitute the nucleation sites for cement hydration [1].It is hypothesized that the presence of soluble salts of Na^{+}, Ca^{+2} etc. helps in keeping the cement hydration process active[14].The primary advantage of nanosilica in increasing early strength development of the cementitious matrix comes from its greater amount of surface area of free silica as compared with other mineral admixtures or pozzolans. Similar to flyash, nanosilica is an ultrafine pozzolan: it combines with calcium hydroxide in the hydrating

the backbone or foundation of concrete strength.Due to the smaller size of nano silica particles, there is a greater total surface area of free silica available for immediate pozzolanic reaction and densification, thus increasing the early strength and durability of concrete [2].Other possible means by which nanosilica enhances the density of hydrated cement matrix are through pozzolanic reaction, condensation of C-S-H and accelerated breaking of cement

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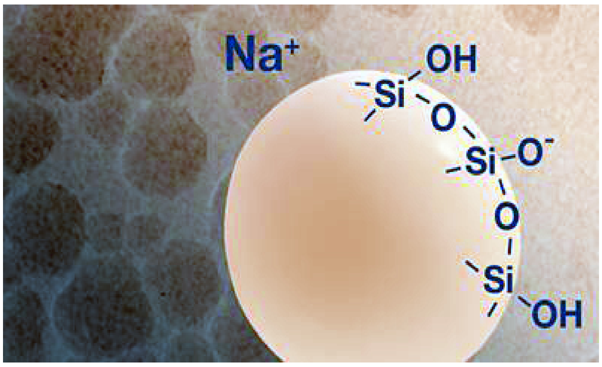

Fig. 1. Nanosilica with surface Si-OH groups [11]

particles [3]. The excess formation of C-S-H from the pozzolanic and nucleation reactions densifies the cement matrix thus reducing the permeability of concrete [4, 5].Reducing permeability means reducing the saturation of concrete with water, stopping ingress of moisture and air & inhibiting corrosion of steel with cracking & spalling of concrete cover thus retaining the structure's water-tightness & thermal insulation properties of the inherent concrete. All these factors jointly or in isolation control the probability of deterioration mechanism of concrete [22].

Mineral pozzolanic admixtures are used to increase the engineering properties and increase the capacity for chemical resistance of concrete mixtures [6].Most mineral additives used in the concrete industry today are by-products or waste from coal combustion(flyash) or the production of ferro silicon alloys(silica fume)[7,8].The benefits that are gained from using these old generation mineral admixtures are through pozzolanic reactions and from the packing densification supported by the inherent chemistry, geometry & particle size diameter of the additives [8,9]. But through the advent of emerging technologies advanced materials like Nanosilica are produced which can manipulate molecular chemistry, structure & engineering properties of cement composites and concrete [10] at a much lower dosages than the other mineral admixtures. Also these emerging advanced materials like nanosilica have excellent compatibility with previous generation pozzolans like flyash [23]. The four mechanisms by which nanosilica enhances the molecular or nanostructures of hydrated cement matrix are proposed as follows:

(i) Accelerated breaking of cement [12, 13]: It has been forwarded in many literatures that when nanosilica has been mixed with OPC and water, increased breaking of alite (C_3S) and belite (C_2S) results in the generation of more C-S-Hs [12] which was due to the large and highly reactive surface area of nanosilica.Bjournstorm et al. also found that nanosilica accelerated both breaking and hydration of C_2S[1].By increasing breaking of these mineral silicates, ample compounds were formed which provided for the formation of additional C-S-Hs.So by using nanosilica the degree of cement hydration(the last cement particle that has reacted)is increased, thus reducing the amount of residual water and decreasing the amount of pores which retained them. This accelerated breaking of cement compounds is directly related to the use of nanosilica as a means to increase the strength & durability of cement composites and concrete [2, 4].

(ii) Enhanced pozzolanic reaction: chemically increasing the production of cementitious C-S-H through accelerated breaking of mineral silicates and via polymerization of excess dissolved calcium hydroxide and nanosilica [14].As nanosilica contributes to the densification of hydrated cement matrix through classic pozzolanic reaction, calcium hydroxide dissolves in the presence of water, creating pores in the C-S-H microstructure with the remaining solution being super-saturated with calcium oxide(CaO)-(Ca) and silica(SiO_2)-(Si).The Ca/Si ratio climbs down from a higher 3.0 to a lower 0.7 ,with the addition of nanosilica which combines with Ca from the calcium hydroxide solution with the concentration of Ca/Si increasing until a certain saturation and after which point the C-S-H gel starts forming in the fluid pore solution and in the free space of existing(hardened) C-S-H gel pore structure[18].This excess formation of C-S-H within the existing C-S-H densifies the cement matrix thus reducing permeability.

(iii) Increasing in packing density of particle-to-particle spacing (nanogranular material)[15,16]:The packing density of C-S-H microstructure is densely populated with pores ranging from macroscopic scale down to nanoscopic scale[15].C-S-H networks with a large volume of pores results in a lower density with lower strength. The void pore spaces whether filled with bulk water solution or solutes provides minimal or no-support in loading applications[19].Thus,in pores where nanosilica is not present, mechanical loading may cause the C-S-H structure to deform but when nanosilica is added they absorb some of the forces and increases the load needed for ultimate failure thus increasing the packing density of hydrated cement matrix.

(iv) Nucleation/condensation: increasing the heterogeneous nucleation of C-S-Hs and increasing the density of hydrated cementitious matrix through filling up of pores [17].As stated before heterogeneous nucleation due to surface Si-OH groups (Figure 1) leads to the growth of C-S-H.The mechanism by which nanosilica will allow the growth of C-S-H will be determined by the size of the electrical double layer and associated potential barrier at the surface of the particle. Due to their reduced particle size diameter nanosilica has a reduced surface potential when compared to larger particles of concrete, thus facilitating more C-S-Hs [20, 21].

So, it is seen that nanosilica particles which have a size of approximately 1/1000th the size of flyash particles, requires a very little dosage when compared to the other mineral additives to bring about more degree of hydration in the cementitious matrix system. It is proposed that some of the issues that have come up in the past using nanosilica in commercial applications (loss of workability and compressive strength) can be attributed to the lack of understanding the effect of particle sizes along with dosages w.r.to the age and strength of the cement matrix. This paper investigates the issue of nanosilica additions in various dosages in Portland cement composites and their effect of the strength & durability of it and also adding the optimised nanosilica dosage as found for cement composites, in M-40 grade concrete to find its effect on the permeability and densification of the standard concrete.

2. Material Used and Test Methods

The materials used were cement - OPC (43 Grade) conforming to IS: 8112-1989, Fine Aggregate (FA) – Natural River sand conforming to Zone II of IS: 383 – 1970, Coarse Aggregate (CA) comprising of 10mm maximum size(CA1) and 20mm maximum size(CA2), Potable water, Chemical Admixture- Polycarboxylate Ether and Mineral Admixture-colloidal Nanosilica (Ns) supplied by M/s Bee Chems.

2.1 Test on Cement Sand Composites as per IS:4031

Mortar Cubes of 70.7mm size were casted with 1 part of cement + 3 parts of sand with water added as per the normal consistency formula of Indian standards,IS:4031, i.e., according to the standard formula P'= (P/4 +3) (1 part Cement+3parts Sand). Here, P'=Quantity of water & P=Consistency of Cement used. i.e. amount of water used to make 300gms cement paste to support a penetration of 5-7mm in a standard Vicat mould with a Vicat needle. Nano Silica were added in various proportions ranging from 0%, 0.5%, 0.75%, 1.0%, 1.25%, 1.5% as per literature review w.r.to cement wt. keeping the w/c ratio fixed at 0.4.The cubes were then ordinary cured under water at a constant temperature of (27± 2) °C and tested for compressive strength and tested at 7 days, 28 days, 180 days & 365 days.

2.2 Test on Concrete as per IS: 516

For Compressive strength testing, concrete cubes of 150mmx150mmx150mm size were casted with cement, FA, CA & water in proportions as per the mix design followed by IS:10262-2009 for M-40 Grade concrete for 100 mm slump keeping the w/c=0.4.The mix proportions obtained were cement=400kg/m^3, CA=1293.04 kg/m^3 [CA1(90%)=1163.74 kg/m^3; CA2(10%)=129.3 kg/m^3], FA=687.54 kg/m^3, water=157 kg/m^3.The % fractions of CA1 & CA2 were obtained as per sieve analysis performed. Colloidal Nano Silica was added in optimized proportions as obtained in result (section 2.1). The cubes before being ordinary cured under water in a curing tank, at a constant temperature of 27± 2°C were tested tested for fresh properties like workability by slump test.After curing for 28 days compressive strength test on a 2000kN compression testing machine with loading applied at the rate of 140 kg/cm^2/minute was conducted.

Table-1. Material properties of nanosilica used

Item	Description
	Colloidal Nanosilica
Active Nano-content	35-40%
Particle Size	5-40nm
pH(at 20°C)	9-10
Sp.Gravity	1.08-1.32

2.3 Water Permeability Test on Concrete as per IS:3085

As there are no recognized standard test method to measure the permeability of concrete by ASTM and BS13, Permeability test procedures were carried out as per the standard IS: 3085-1965. To conduct the permeability test cubes of size 150mm were casted and cured for 28 days. After 28 days of curing, specimens were placed properly in the permeability apparatus. A Rubber sheet of 8mm thick and 150 x 150mm size with a central hole of 100 x 100mm was taken. This rubber sheet was then placed on the top and bottom surface of the cube in the permeability cell. Cover plate was then tightened properly. The rubber sheet acts as a washer and prevents the leakage of water through the annular space between specimen and cell. Suitable arrangements were made for supplying compressed air at 10kg/cm^2 to the cell by a compressor with an adequate supply of cleaned deaired water to be available for the purpose of water may be easily de-aired and constant supply of pressurized water. Collecting jar was placed under the concrete specimen to collect the discharged water from the concrete. The test was conducted continuously for 72hrs.as shown in Fig.2.

After 72hrs cubes were then taken out from the cell. During the test it was found that there was no any permeate through the concrete. As there was no any permeation of water the steady flow method was discarded. Hence to find the co-efficient of permeability Depth of Penetration method was employed by which the cubes were splitted and depth of penetration was measured in the specimen at different locations and average depth of penetration was obtained. The method was developed by Valenta, equivalent to that used in Darcy's Law.

The Coefficient of Permeability (K) in (m/s) is given by,
$= e^2H/2ht$
Where,
e = Fraction of volume of Concrete occupied by Pores=0.04;
H = Depth of Penetration in Concrete in meters;
h = Hydraulic Head in meters=50m;
t = Time under Pressure in seconds= 72hrs=259200secs.

2.4 X-Ray Diffraction (XRD) Test

Fig. 2. Water Permeability Test Apparatus in IIEST

The X-Ray Diffraction (XRD) technique is used to measure atomic spacing between lattice layers in a crystal. This can be calculated from the X-Ray Diffraction test results using the Braggs Law equation, which is as follows: $n\lambda = 2d \sin\Theta$, where, λ = wavelength of the incidental X-Ray beam; d = the atomic spacing between the layers; Θ = angle of incidents; n is taken as unity.XRD testing is used to find out the qualitative mineral analysis i.e. whether the sample is crystalline or amorphous. Using this test quantitative determination of glass and crystallization components can be done for any samples. The measurement of X-ray diffraction line intensities is the basis for quantitative phase analysis. X-ray diffraction techniques are widely used for determining the identity and structure of crystalline materials. Graph is drawn between 2θ and counts. Thus the quantitative and qualitative measures of the sample can be done by using this test. Here our nanosilica sample was tested for XRD results.

2.5 Scanning Electron Microscopic (SEM) Test

SEM analyser is an instrument used for the examination and analysis of the micro structural characteristics of the solid sample. Microstructural or morphological characteristic of a sample is found out by using the instrument Hitachi S-3000H in Dr.M.N.Dastur School of Material Sciences laboratory in IIEST, Shibpur. Appearance of the specimen's microstructural image is gained by this test and it gives the direct result of the large depth of field. At greater depth of field much more information about the specimen can also be found out. This instrument can give the microstructural structure of the specimen for various magnifications also. Here cement mortar with & without optimized nanosilica additions were tested for SEM results at 28 days.

3. Test Results and Discussions

3.1 Test Results for Cement Composites

The following Table 2 shows the Strength of the cement composites along with the dosage type & age of the specimen. The test results reveal that with fewer dosages of nanosilica the strength is reduced due to the agglomeration

Table-2.Strength of cement composites with nanosilica addition at various dosages & ages

% Nano additions b.w.c. in ordinary Portland Cement	7 day	28 day	180 day	365 day
	Comp. Str. (N/mm^2) (% incr.)	Comp. Str. (N/mm^2) (% incr.)	Comp. Str. (N/mm^2) (% incr.)	Comp. Str. (N/mm^2) (% incr.)
0% Ns	21.08	31.89	30.01	30.01
0.5% Ns	23.85 (13.14%)	35.51 (11.35%)	27.47 (-9.2%)	26.76 (-4.29%)
0.75% Ns (optimized)	27.73 (31.54%)	42.27 (32.55%)	32.52 (8.4%)	31.5 (4.96%)
1.0 % Ns	25.07 (18.93%)	37.36 (17.15%)	33.68 (12.2%)	32.41 (8.0%)
1.25% Ns	23.17 (9.91%)	30.85 (3.26%)	35.24 (17.4%)	31.3 (4.29%)
1.50% Ns	23.81 (12.95%)	37.79 (18.5%)	31.23 (4.07%)	29.12 (-2.96%)

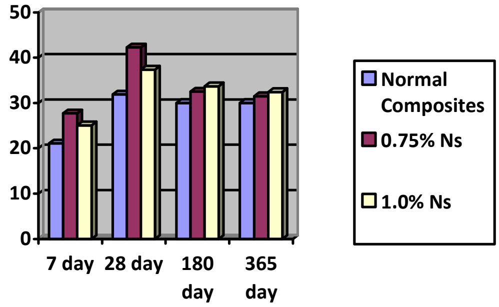

Fig.3: Chart showing the early & later compressive strengths (in N/mm^2) of some typical nanosilica dosages in cement composites.

of the fewer nanosilica particles in the hydrated cement matrix while more additions of nanosilica particles tend to increase the rate of C-S-H production ultimately reaching a optimized value at 0.75% additon.This threshold optimized value is compromised in the long run with more nanosilica additions dissoluting the remaining calcium hydroxide of the hydrated cement matrix as discussed in the previous section, for more generation of C-S-Hs till the strength gain is maximized at 365 days with 1% nanosilica addition as shown in Fig.3..However, with more nanosilica dosages the oversaturated cement matrix fails to contribute to any further strength gains as seen from Table 2.

3.2 Test Results for Workability & Compressive Strength in Concrete

The following Table 3 shows the affected properties (both fresh & hardened) of M-40 concrete with the addition of nanosilica.

The Table 3 results show that the workability of concrete has shown a slight increase over the normal concrete which may be explained due to the fact that nanosilica additions have maintained on the fresh property of concrete without any need of tempering with water or chemical admixtures. The compressive strength of the nanosilica added concrete has increased due to the decreased size and huge surface area of the nanoparticles, which resulted in more hydrate development and cement matrix densification thus enabling the matrix structure to bear more load through support from the other adjoining hydrated cement matrix members.

Table-3.Fresh & Hardened Properties of concrete with nanosilica addition at 28 days

Property of M-40 Grade Concrete Property	Normal Cement Concrete (M-40)	M-40 Nanosilica added Concrete (With optimized 0.75% nS Addition b.w.c.)
Fresh Property [Slump(mm)]	100	105 (5%)
Hardened Property @ 28 days Compressive Strength (N/mm^2)	40.12	49.75 (24.1%)

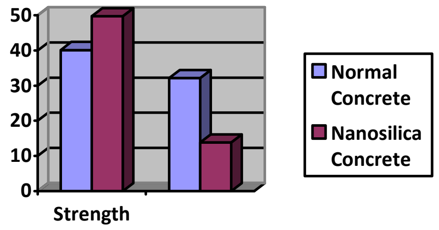

Fig.4: Chart showing the compressive strengths (in N/mm^2) & water permeability (in depth of water penetration) of concrete with & without nanosilica.

3.3 Test Results for Water Permeability in Concrete

The following Table 4 shows the water permeability of the concrete specimen with & without the addition of nanosilica.

As shown in Fig.4, the permeability test results indicate that with nanosilica additions there is a propensity in the reduction of pore size of the C-S-Hs which in turn makes the hydrated cementitious matrix more densified and less permeable to water flow.

3.4 XRD Test Results for Nanosilica

The XRD results in Fig.5 show that there is no well defined peak or peaks as found in the crystalline materials.So, the nanosilica used here is amorphous in nature, which possesses no liability towards health & environment as possessed by its crystalline (micro-silica for example) counterparts.

3.5 Microstructural Test Results

As shown in Fig.6 & Fig.7, the microstructure at 500X of normal cement mortar composites investigated in the scanning electron microscopy, communicate lesser amount of C-S-H hydrated products while that of nanosilica added cement mortar composites indicate the presence of a comparatively larger C-S-H hydration products thus

Table-4.Water Permeability of Concrete at 28 days

Type of Concrete	Physical Dimensions Avg.Density (Kg/m^3) = Weight(Kg)/ Volume(m^3) (%incr.)	Depth of Penetration in Concrete in meters (H).	Fraction of volume of Concrete occupied by Pores(e)	Avg. Coeff. of Permeability K (in m/s) (% incr.)
Normal Cement Concrete	2536.18	0.01383	(0.02+0.06)/2=0.04	$1.985x10^{-12}$
		0.015		
		0.06767		
Nanosilica added Concrete (0.75% nS b.w.c.)	2622.31 (3.4%)	0.01434	(0.02+0.06)/2=0.04	$0.854x10^{-12}$ (-56.98%)
		0.00834		
		0.01884		

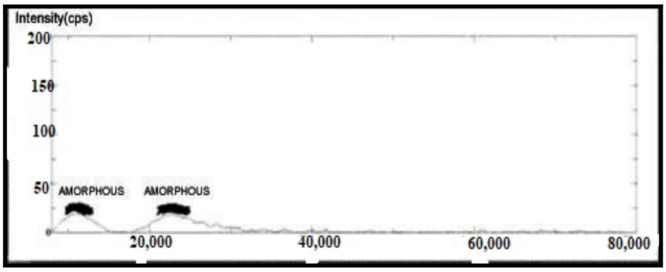

Fig.5: XRD pattern for Nanosilica

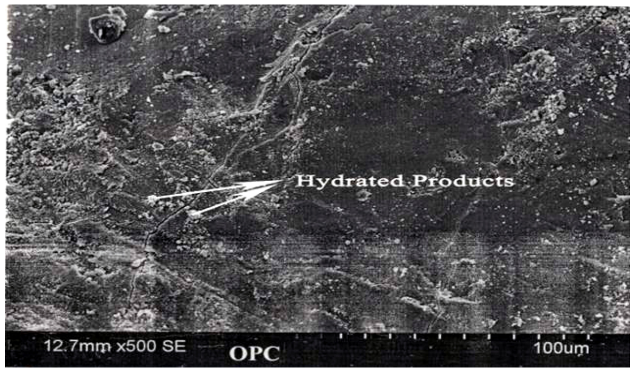

Fig.6: Microstructure of normal cement mortar composites conforming for the dense hydrated cement matrix and low permeability, as corroborated by our test results.

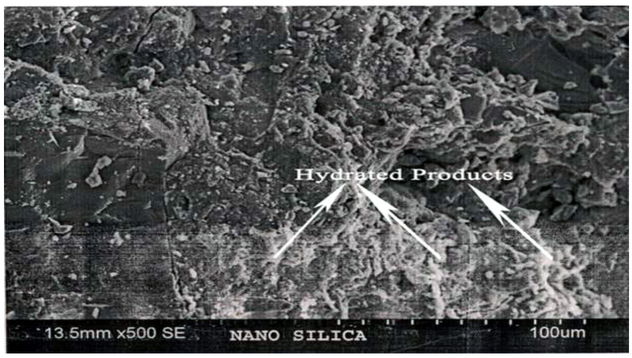

Fig.7: Microstructure of nanosilica added cement mortar composites

4. Conclusions

Results show that there is an optimization in the dosages of nanosilica addition in cement composites at 28 days, with maximum strength achieved at a particular dose of 0.75% nanosilica bw.c. But in the later stages more strength is obtained at higher dosage which means that further C-S-Hs are produced by breaking the mineral silicates of cement by nanosilica resulting in a more durable nansilica enabled cement mortar composite.However, reduction in strength is observed at a lower dose due to agglomeration of smaller number of nanosilica particles and also if the dosage is increased further due to supersaturation of nanosilica.When introducing the optimized 0.75% nanosilica dosage in standard concrete, there is a reduction in the water permeability due to reduction in pore sizes of C-S-H gel in the matrix resulting in a densified concrete.

In the recent times, the Indian codes like IS: 383, IS: 456 & IS: 10262 have revised their texts to highlight on the use of mineral pozzzolonaic additives such as flyash and silica fumes.But despite the National Building Code-2016 suggestion to use nanomaterials as alternative and sustainable materials in building construction is yet to pay off. It may be mentioned that silica fumes which has been in use for over 30 years and is widely available from most major concrete suppliers in UK with brands like 'Chronolia' (Lafarge), 'Rapidcrete' (Breedon), manufacturers 'Diamondcrete' (Aggregate Industries) and 'Easyflow' (Hanson), have more than 70% have particle size diameter within 100nm range [24], but they are not marketed as nanomaterials by the concrete industry.So, commercialization of nano-products still remains a challenge.

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