

# Effect of particle size of colloidal nano-silica on the properties of the SCM based concrete

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**Abstract.** In the current study, effect of particle size of colloidal nano-silica on the properties of the SCM based concrete is studied. The nano-silica particle sizes adopted for the study are 30nm, 60nm and 90 nm. The M20 grade concrete system is made up of cement (C), fly ash (FA), micro-silica (mS) and nano-silica (nS). Compressive strength test at 60 days and pore structure analysis are carried out for C-FA-mS-nS system. The hydration of Portland cement is significantly influenced by the colloidal nanosilica (CNS) particle size. High doses of nS content boost the pozzolanic reaction and the creation of CSH and CASH gels, but they also have a negative impact on the strength development of the material by increasing microcracking due to the self-desiccation effect. The addition of nanosilica to concrete can significantly enhance its qualities after hardening because it ensures the pozzolanic reaction, the seeding effect, which both increase the degree of hydration, and the filling effect, which can fill the internal porosity defects. The cement-mS system's impermeability was greatly improved by the nS because it improved the microstructure, increased the complexity of the pore structure, and refined the pore structure.

**Keywords.** Colloidal nanosilica, micro silica, silica fume, flocculation, agglomeration

## 1 Introduction

In order to create a new generation of customised, multifunctional cementitious composites with improved mechanical performance and durability, nanotechnology entails alteration of the structure at the nanoscale scale [1]. Those particles known as nanoparticles have at least one dimension that is less than 100 nanometers (nm) [2]. Due to their strong reactivity,

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nanoparticles can serve as heterogeneous nuclei for cement pastes, accelerating cement hydration further, and as nano-filler, densifying the microstructure and reducing porosity [3-5]. Numerous nanoparticles, including nano-clay, nano-fly ash, nano-limestone, nano-TiO<sub>2</sub>, nano-Fe<sub>3</sub>O<sub>4</sub>, nano-Al<sub>2</sub>O<sub>3</sub>, and nano-SiO<sub>2</sub>, have been used to enhance the properties of cementitious composites as of late [6]. While certain nanomaterials are colloidal, in which the nanoparticles are equally disseminated, many nanomaterials are solid powders.

## **2 Macroscopic and microscopic silica**

The application of nanomaterials to cement science offers a novel notion for the resolution of the issue of how to improve the performance of cement-based materials on a micro level. Silicon oxides are also known as silica, with SiO<sub>2</sub> being the most common kind. It is the most prevalent element of the earth's crust and can be found in nature in crystalline form (as quartz sand). Contrarily, amorphous silica is produced industrially in a few different forms, such as silica gels, precipitated silica, fumed silica, and colloidal silica. Nano-sized (100 nm) SiO<sub>2</sub> has gained attention as a very promising mineral admixture due to the spike in interest in nanomaterials and their potential applications in creating high performance, sustainable, and durable concrete. The material's aggressive pozzolanic characteristics and small particle size are what have attracted this attention. Because of its higher surface area than its precursor, micro silica (mS), also known as silica fume, pozzolanic reactivity is significant.[7]

## **3 Types of nano-silica**

Both colloidal amorphous nano-silica and solid crystalline powder are readily available. Nanoparticles are uniformly scattered in colloidal form. A colloid form is a stable dispersion of particles with sizes ranging from 1 to 100 nm, which prevents gravity from causing them to settle. Other forms of silica are distinct from colloidal silica in a number of important respects. The fact that it is liquid rather than powder is the most obvious distinction. SiO<sub>2</sub> is present in dense, amorphous particles that make up colloidal silica. The [SiO<sub>4</sub>]<sup>4-</sup> tetrahedra that make up these particles are dispersed at random. Amorphous silica differs from crystalline silica in that it is randomly distributed at the molecular level. The main differences between the grades of colloidal silica based on particle size or specific surface area are pH and particle size. Another way to define particle size is in terms of specific surface area, where larger specific surface areas correspond to smaller average particle sizes. Titration can be used to figure out the colloidal material's particle surface area [8-11].

## **4 Objective of the paper**

The present study is carried out to evaluate the effects of different particle sizes colloidal nanosilica 30, 60, and 140 nm on the properties of the M20 concrete. The M20 grade concrete system is made up of cement (C), fly ash (FA), micro-silica (mS) and nano-silica (nS). Compressive strength test at 60 days and pore structure analysis are carried out for C-FA-mS-nS system

## **5 Mix proportions**

The following table presents the quantities of the materials used for the development of M20 grade concrete.

**Table 1.** Quantities of the materials used for the development of M20 grade concrete.

Grade	Cement kg/m <sup>3</sup>	Fine Aggregate kg/m <sup>3</sup>	Coarse Aggregate		Water L
			20mm size kg/m <sup>3</sup>	10mm size kg/m <sup>3</sup>	
M20	324	747.29	470.13	680.55	167.83

## 6 Optimization of SCMs

The following table presents the optimized amount of fly ash to be used in concrete of M20 grade

**Table 2.** Optimization of fly ash content in M20 grade concrete

S.No.	Cement	Fly ash	Compressive Strength (MPa) at 60 days
1.	100%	0%	26.89
2.	90%	10%	26.91
3.	80%	20%	30.39
4.	70%	30%	32.36
5.	60%	40%	21.57
6.	50%	50%	18.42

The following table presents the optimized amount of micro-silica to be used in fly ash-based concrete of M20 grade

**Table 3.** Optimization of micro-silica content in M20 grade flyash concrete

S.No.	Cement (C)	Fly ash (FA)	Micro-silica (mS)	Compressive Strength (MPa) at 60 days
1.	65%	30%	5%	31.26
2.	64%	30%	6%	32.08
3.	63%	30%	7%	34.56
4.	62%	30%	8%	37.44
5.	61%	30%	9%	31.29
6.	60%	30%	10%	24.15

Small amounts of pozzolanic C-S-H would form as a result of mS's limited pozzolanic activity, indicating that at young ages, mS mostly serves as a filler rather than a "seed" for C-S-H nucleation.

The following table presents the optimized size of nano-silica to be used in SCM admixed concrete of M20 grade

**Table 4.** Optimization of micro-silica content in M20 grade fly ash concrete

S.No.	Cement (62%) + Fly ash (30%) + Micro-silica (8%)			Compressive Strength (MPa) at 60 days
	Nano-silica (nS) (30 nm)	Nano-silica (nS) (60 nm)	Nano-silica (nS) (90 nm)	
1.	1%	-	-	39.40
2.	2%	-	-	46.21
3.	3%	-	-	45.97
4.	-	1%	-	42.31
5.	-	2%	-	51.08
6.	-	3%	-	49.66
7.	-	-	1%	36.03
8.	-	-	2%	38.25
9.	-	-	3%	33.51

The strength of 28 days was raised by increasing nS dose. The compressive strength reaches a maximum of 51.08 MPa, as opposed to 32.36 MPa in the C-FA system, especially at 2% nS. In contrast to the lower strength at the later age, which was caused by the coating effect brought on by the pozzolanic reaction of nS on the surface of FA, the increased strength at the early age was connected to the refined pore structure and the hastened hydration process. The acceleratory impact of nS is brought on by the seeding effect, which is distinct from the filler effect. The huge specific surface area of the nS also has a seeding impact by adsorbing calcium ions and acting as C-S-H nucleation sites.

## 7 Porosity

The interface between FA particles and cement particles would be greatly improved by nS, which might increase compressive strength and impermeability. Brenauer-Emmett-Teller's (BET) nitrogen adsorption method yields the C-FA-mS-nS system's pore diameter, total pore surface area, total pore volume, and porosity from the Adsorption isotherms and BJH isotherms.

**Table 5.** Pore structure analysis of C-FA-mS-nS system

	C-FA system	C-FA-mS-nS system
Average Pore Diameter (nm)	17.321	3.265
Total Pore Surface Area (m <sup>2</sup> /g)	1.654	4.658
Total pore volume (cc/g)	0.0143	0.0068
Porosity (%)	1.82	0.64

It was discovered that nS and mS were able to greatly increase the impermeability in the C-FA system for the following reasons: firstly, nS with nano-size could exert the filling effect to block capillary pore. Due to its high reaction activity, it could effectively participate in the pozzolanic reaction and serve as a nucleation seed to cause the cement to hydrate. As a result, the formation of nanoscale C-S-H gel was facilitated, which could also significantly improve

the impermeability by filling or blocking capillary pores. Additionally, nS served as a nucleation seed and consumed CH to induce the hydration of cement, hence promoting the system's overall hydration level. This considerably increased the microstructure of the C-S-H gel and the interface between the gel and FA, increasing the impermeability. The microstructure and pore structure were enhanced, which significantly increased the impermeability.

## 8 Microstructural characterization

Using scanning electron microscopy-energy dispersive X-ray analysis (SEM-EDX), it is possible to quickly determine the samples' elemental structure.

**Table 6.** Ca/Si ratios

S.No.	Cement (62%) + Fly ash (30%) + Micro-silica (8%) system	Ca/Si ratio
	Nano-silica (nS) ( 60 nm)	
1.	1%	1.88
2.	2%	1.55
3.	3%	1.51

The average Ca/Si ratio in the C-FA-mS (reference) system was 1.88; 2.0% NS decreased it to 1.55, and an increase in dosage to 4.0% further decreased it to 1.46. In reality, the CH content in C-S-H was reflected in the Ca/Si ratio of hydrates, and in general, a larger Ca/Si ratio indicated a bigger amount of CH. Based on this, it is possible to explain why the Ca/Si ratio was reduced by adding nS by the fact that nS with a nano-size penetrated these regions close to the aggregates and reacted with CH to lower the Ca/Si ratio. Significant improvements were made to the entire microstructure, including the ITZ microstructure, which clearly increased the strength. The relative concentration of CH in hydrates may be reflected in the peak intensity of CH in the XRD pattern, which would explain the cementitious system's hydration process. In the C-FA-mS-nS system, the pozzolanic reaction of F, mS, and nS consumes CH produced by cement hydration to lower the amount of CH in hydrates. The C-S-H gel would be created by the reaction of the nS and CH, and the consumption of CH would cause the cement to hydrate and produce more CH. The nanoparticle-containing nS may also serve as a nucleation seed to cause cement to hydrate and form hydrates. According to reports in the literature, a coating on the surface of FA may be created by the pozzolanic reaction of nS, mS, and FA; if the coating was better developed, it might even slow down the pozzolanic reaction of FA. This was likely one among the factors that caused nS to weaken C-FA-mS as people aged. The interface between FA particles and cement particles would be greatly improved by nS, which might increase compressive strength and impermeability. Based on the explanation above, it was determined that nS sped up the hydration of cement and, most likely, sped up FA's pozzolanic reaction only a little bit. Due to the consumption of FA and nS in the pozzolanic reaction, the amount of CH in hydrates was decreased; however, the amount of C-S-H gel was increased due to the rapid hydration of cement and the pozzolanic reaction of nS and mS. The effects of nS on hydration can be determined by measuring the cement's hydration heat within a specific time frame. The amount of CH generated by the seeding effect is more than that consumed by the pozzolanic activity on the first day of curing, and this behaviour gets worse as the particle size gets smaller. The CH concentration significantly drops at 3 days as pozzolanic activity increases, and this effect becomes more prominent as particle size decreases. As cement hydration is accelerated by nS, the amount of C3S and C2S decreases, and pozzolanic activity causes the CH content to decrease, increasing the amount of C-S-H gel. The hydration

process is dominated alternately by the pozzolanic activity of nanosilica and the seeding impact, but both contribute to the increased cement hydration level.

## 9 Conclusions

1. Nano-SiO<sub>2</sub>, which is a highly reactive substance, speeds up the formation of C-S-H (calcium silicate hydrate) and the dissolution of C<sub>3</sub>S (tricalcium silicate).
2. The hydration of Portland cement is significantly influenced by the colloidal nanosilica (nS) particle size.
3. As the nS content rises, the pozzolanic reaction and the creation of CSH and CASH gels both increase, but it also causes more microcracking due to the self-desiccation effect, which has a detrimental influence on the strength development of the material.
4. Using Blaine's air permeability method, it was discovered that the surface areas of nano and micro-silica were 230000 m<sup>2</sup>/kg and 22000 m<sup>2</sup>/kg, respectively.
5. Calcium silicate hydrate (CSH) gel is produced when nanosilica particles mix with the free lime (calcium hydroxide) produced during cement hydration. This produces a cohesive gel structure that raises the stability and viscosity of the plastic.
6. The new CSH binder improves cement paste density, strengthening the bond between cement grains, and offers a significant increase in early compressive strength, reducing cement setting time. By interacting with the free lime and transforming it into calcium silicate hydrates, it also contributes to an increase in final strength.
7. Nanosilica ensures the pozzolanic reaction, the seeding effect (which both increase the degree of hydration) and the filling effect (which can fill the internal porosity defects); as a result, its addition to concrete can effectively increase its mechanical property and durability after hardening.
8. The findings showed that as the colloidal nanosilica particle size was decreased, the paste's flowability declined. The fundamental justification was that, in an ionic environment, the sol may produce flocculation that restricts some free water, a behaviour that got worse as particle size shrank.
9. The compressive strength of the 60 nm nS material was higher than that of the other two, and hydration heat analysis further demonstrated the activities of hydration under various conditions. This conclusion was mostly caused by flocculation's restriction of the initial chemical activity, such as pozzolanic and seeding effects of nanomaterials.
10. When silica fume content is higher than 8%, agglomerates form that are concentrated in a narrow area and prevent the production of both CSH and geopolymer phases.
11. The pozzolanic reaction of mS was likely slightly sped up by the addition of nS because nS not only consumed calcium hydroxide in the pozzolanic reaction to cause the cement to hydrate but also served as a nucleation seed to promote the formation of C-S-H gel. The cement-mS system's impermeability was greatly improved by the nS because it improved the microstructure, increased the complexity of the pore structure, and refined the pore structure.
12. It was determined, based on the discussion above, that nS accelerated cement hydration and, most likely, it somewhat sped up FA's pozzolanic reaction. Due to the consumption of FA and nS in the pozzolanic reaction, the amount of CH in hydrates was decreased; however, the amount of C-S-H gel was increased due to the rapid hydration of cement and the pozzolanic reaction of nS and mS. The hydration

process is dominated alternately by the pozzolanic activity of nanosilica and the seeding impact, but both contribute to the increased cement hydration level.

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