NANOSILICA'S INFLUENCE ON CONCRETE HYDRATION, MICROSTRUCTURE, AND DURABILITY: A REVIEW

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ABSTRACT:

The usage of nanomaterials in the construction industry is becoming more and more popular. Among the several types of nanomaterials, nano silica (NS) has drawn interest due to its high specific surface area and strong pozzolanic reactivity. By enhancing concrete's performance, NS allows for the achievement of greater compressive strength and durability with less cement which lowers the carbon footprint associated with the cement production. Because they are substantially smaller than conventional supplementary cementitious materials (SCMs), NS particles are better able to fill the micropores in the cement matrix. This work provides a comprehensive analysis of earlier studies on the influence of NS addition on concrete properties. It investigates the impact of NS on the workability and setting time of fresh concrete. The study also explores how NS affects concrete mechanical characteristics, such as its modulus of elasticity, flexural strength, and compressive strength, with an emphasis on the underlying mechanisms driving these improvements. It is shown that NS apart from its filling ability, accelerates hydration process by acting as nucleation sites for hydration products to form through seeding effect. The microstructure becomes dense and pore size refinement occur. However, an ineffective NS dispersion serve as a barrier to add a larger percentage of NS to the cement paste. Overall, this research study offers insightful information about the potential of NS as an additive to improve concrete's sustainability and performance.

1. INTRODUCTION

The building sector mostly depends on cement as a vital component of concrete. Notwithstanding, the environmental ramifications of the cement sector, specifically with regards to its carbon dioxide emissions, pose noteworthy obstacles and prospects for enhancement. Any decrease in the cement content can have a major effect on lowering global CO_2 emissions because the cement industry as a whole contributes about 8% of the world's CO_2 emissions (Kemer et al. 2021; Mesboua et al. 2021). On average, 842 kg CO_2/tonne of clinker are produced throughout the OPC production process(Cement Sustainability Initiative 2016). This has led to prospects of suitable mineral additions in concrete to enhance properties. Apart from conventional SCMs, the growing application of nanotechnology in cementitious materials has attracted much attention in recent times as notable improvement in mechanical properties has been observed(Antonovič et al. 2010; Olafusi et al. 2019; Sobolev and Ferrada Gutiérrez 2005; Sobolev and Gutiérrez 2005).Improvements to the rheology, strength, and durability of concrete are a few examples of how nanotechnology is being used in sustainable building. Any alteration to concrete at the nanoscopic level and its constituents affects the behaviour of the material, including its durability and strength properties. Of all the different types of nanoparticles that have been studied, nano-silica (NS) have drawn a lot of interest as they enhance morphology of the concrete system on account of filler effect and pozzolanic reaction(Nazari et al. 2010; Sanchez and Sobolev 2010; Huseien, Khalid, and Mirza 2022; Sobolev 2016; Raki et al. 2010; Utsev et al. 2022; Olafusi et al. 2019; Sobolev and Gutiérrez 2005). In the past few years, a large number of research studies have documented dosages of NS in cementitious materials, such as paste, mortar, and concrete, ranging from 0.5 to 10%. The partial substitution of NS, reduces the cement content in the mix and hence leads to the reduction in the carbon footprint(Quercia and BROUWERS 2010). The size of NS is responsible for its notably high pozzolanic action(Agarwal 2006; Ahmad et al. 2022). Due to the NS minuscule size, they pack into all spaces and gaps, improving the interfacial transition zone (ITZ), which is thought to be a weak phase in cementitious materials(Sharkawi, Abd-Elaty, and Khalifa 2018; Du, Du, and Liu 2014; M. I. Khan and Siddique 2011).The results of nanoindentation unequivocally demonstrate that NS addition enhance the packing density of C-S-H particles in addition to speeding up the rate of hydration(Singh et al. 2017; Mondal et al. 2010). By analysing cement pastes containing NS using diffuse reflectance fourier transform infrared (DR-FTIR) spectroscopy, it was found that NS speed up the cement's dissolution rate (Björnström et al. 2004). As per electrochemical impedance spectroscopy test conducted on samples, it was found that when NS is added to cement mortar, the ionic transport resistance of the mortar is greatly enhanced, and the electric capacitance is lowered(He and Shi 2008).

This review attempts to contribute to a deeper understanding of the function of NS in improving the performance of concrete by synthesizing the results of existing research and identifying knowledge gaps and topics for further inquiry. The creation of more resilient, affordable, and sustainable concrete materials for the

construction sector can ultimately be influenced by this understanding.

2. PRODUCTION OF NANO SILICA

Because of their metal impurities, silica particles derived from natural resources are not suitable for use in advanced experimental and commercial uses. Thus, artificial silica is the emphasis and are produced in amorphous form. On the contrary, naturally Silica occurs in crystalline forms such as in quartz, tridymite, cristobalite. Two distinct methodologies, known as "bottom-up" and "top-down" approaches, are used in the synthesis of nanostructured materials. Figure 1 is illustratively explaining both the approaches, taken from (Rawat 2015). Characteristic of top-down design is that the initial size is reduced in dimension. In bottom up approach we are gradually moving from atoms or molecules to its cluster. "Bottom up" methods start with the design and synthesis of specially designed molecules with the capacity to self-assemble or self-organize into higher order mesoscale. The task is to create molecules that, when a particular chemical or physical trigger is regulated, will spontaneously assemble themselves(Silva 2004; Raki et al. 2010; Sobolev 2016; Rawat 2015). Different techniques are used to manufacture NS, and each product has specific uses in the industry. Few kinds that are used in cement composites research might be divided into categories as nanosilica gels (Scherer et al. 1995; Kim et al. 2013), nanosilica sols (Gaitero, Campillo, and Guerrero 2008; Björnström et al. 2004; Madani et al. 2014; Madani, Bagheri, and Parhizkar 2012), pyrogenic nanosilicas (Korpa et al. 2008; Bagheri et al. 2013; Korpa, Kowald, and Trettin 2008), precipitated nanosilicas (Bastien, Dugat, and Prat 1997; Agarwal 2006), hollow nanosilicas (Lim et al. 2020; Xuejun et al. 2022; Ghozi-Bouvrande, Pellet-Rostaing, and Dourdain 2021; Yang et al. 2020). The key factor causing the variations in the NS is their morphology. For the manufacture of nanoscale materials like NS, one of the most popular "bottom-up" techniques in chemical technology is sol-gel synthesis, which has been discussed in this paper. Apart from sol-gel, few other methods are “reverse microemulsion” and “flame synthesis”.

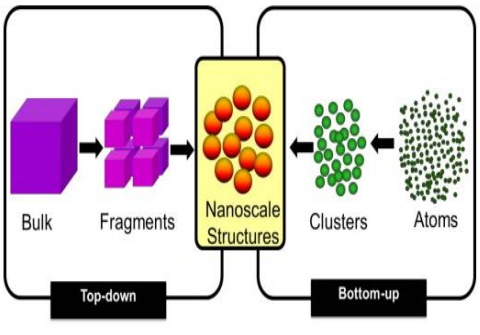


Figure 1. Figure illustrating top down and bottom-up approach for synthesizing nanoparticles, taken from (Rawat, 2015)

The flowchart depicting the sol-gel method is shown in Figure 2, taken from (Rahman and Padavettan, 2012). In this method, first step involves creation of a homogenous solution either by dissolving metal organic precursors in a solvent of organic nature, that is miscible with water, or by dissolving inorganic salts in water. Precursor solution is thereafter taken and subjected to hydrolysis. The mix is then stirred for some time until colloidal particles start forming, which is basically sol, sol that is dispersion of solids in an aqueous medium.

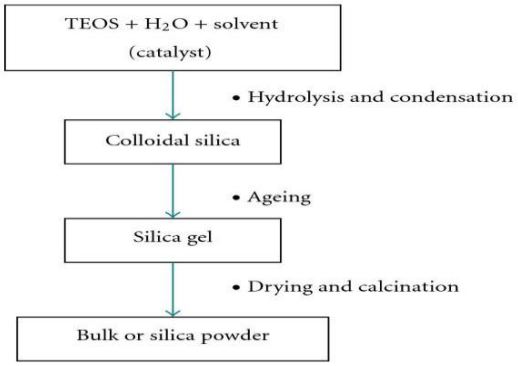


Figure 2. Flow chart of a typical sol-gel process, taken from (Rahman and Padavettan, 2012)

During stirring, two reactions are basically observed, one is Hydrolysis and other one being Condensation. Because the gel produced by the hydrolysis or alcoholysis process is wet or damp, the drying has to be done, in accordance the intended uses and characteristics of the gel(Bokov et al. 2021). There are 2 ways for drying. One is evaporative drying which leads to the pores shrinkage and formation of Xerogel. Supercritical drying is another method of drying, which is relatively more controlled one and leads to the formation of aerogels(Esposito 2019; Livage, Henry, and Sanchez 1988; Bokov et al. 2021). Figure 3 is illustrating variations which could be done within the sol gel process and consequently the end products will vary(“Aqueous and Nonaqueous Sol-Gel Chemistry” 2009).

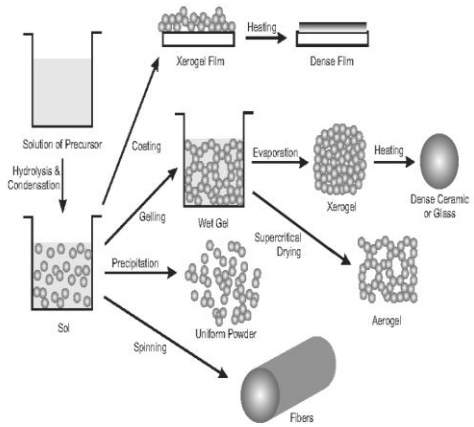


Figure 3. Different phases in the sol-gel process to regulate the product's ultimate shape (“Aqueous and Nonaqueous Sol-Gel Chemistry”, 2009)

3. DISPERSION OF NANO SILICA

The presence of Calcium ions in pore solution promotes the flocculation of NS by adsorption of ions on NS surface (Lavergne et al., 2019). In order to cater this, PCE based

superplasticizer has proven to be quite effective. This is primarily due to the long polymeric chain steric hindrance which stabilizes the once dispersed molecules and avoid their coalescing (Qian and De Schutter, 2018). The dispersion effect has also been illustrated in figure 4, as taken from (Feng et al. 2020). PCE based superplasticizer on account of its ability to adjust their molecular structure in the HPC formulation mix, brings a significant impact on flowability of the mix (Khan and Kumar, 2024).The effect of PCE could be realized from the reduction in mean diameter of NS from 7994.6 nm to 176.5nm when PCE was used as shown in figure 5 (Liu et al., 2020). Thus, to avoid NS from losing its nano properties its re-agglomeration is avoided by using PCE based superplasticizer. NS possesses high surface energy which drives them to agglomerate to reduce its surface energy and surface area. This agglomeration poses major challenge in terms of its substitution in cement to obtain HPC.

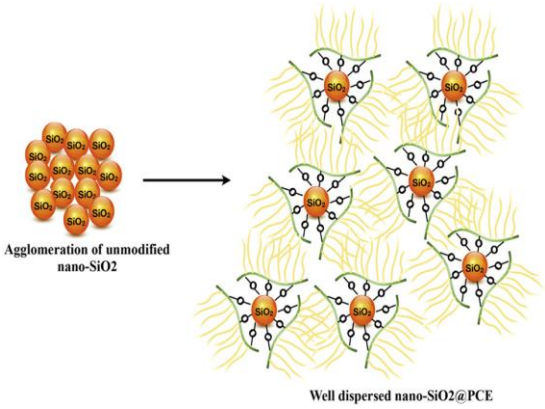


Figure 4. Dispersion of nano-silica after using polycarboxylate superplasticizer (Feng et al., 2020)

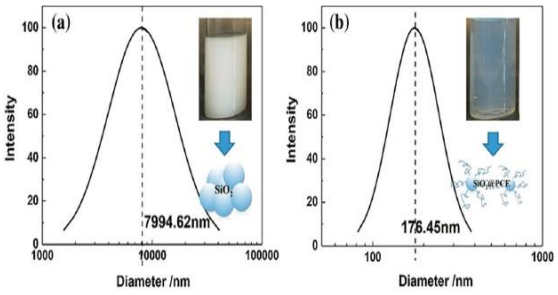


Figure 5. Showing particle size distribution of nS (a) before adding PCE (b)after adding PCE to nano-silica as mentioned in (X. Liu et al., 2020)

Many researchers have experimented with various techniques to incorporate nanoparticles into concrete. Before adding NS, Ji (Ji, 2005) first proceeded with the addition of superplasticizer in water and then agitated the mixture quickly for two minutes. Following a 30-second rotation in a rotary mixer containing cement, fly ash, fine and coarse aggregates, the ready-mixed liquid containing water, NS, and superplasticizer (SP) was added. The concrete mixture was lastly stirred for an additional one and a half minutes. Li et al. (H. Li, Zhang, and Ou, 2006) firstly added cement and aggregates into the concrete mixer rotating the mixture at slow speed for about 2 minutes. Then separately in a mortar mixer, NS were added to the SP and water mixture, and the mixture was agitated at a high speed for five minutes. Defoamer was also added while stirring was done. This mixture from mortar mixer was then added to the concrete mixer and was given a final rotation for 2 minutes at small speed to achieve the desired dispersion effect. Thus, it can be observed that in order to utilize the intended beneficial effects of NS, the agglomeration has to be counteracted by the appropriate use of superplasticizer.

4. EFFECT OF NANO SILICA ON FRESH STATE PROPERTIES OF CONCRETE

4.1 Setting Time

As the proportion of NS increases, cement paste's initial and final setting times decrease (Snehal, Das, and Akanksha 2020; Kooshafar and Madani 2020). Utilizing 7% NS shortened the initial and final setting time by 73 and 121 minutes, respectively (Kooshafar and Madani, 2020). The primary reason is that NS at the nanoscale can function as crystal nuclei to quicken cement hydration, so cutting down on the slow induction phase of hydration and considerably lowering the first setting time. Additionally, the hydration process can be further aided by the pozzolanic interaction between NS and Ca $(OH)_2$, which shortens the ultimate setting time (Isfahani et al., 2017). There was notable decrement observed in both initial and final setting time values, when NS was added in concrete with either high volume FA or high-volume slag (Min Hong Zhang and Islam, 2012a). The addition of 2% NS shortened the fly ash concrete's initial and final setting periods by 90 and 100 minutes, respectively. The reduction values in case of slag concrete was observed to be 95 minutes for initial setting time and 105 minutes for final setting time (Min Hong Zhang and Islam, 2012a).

4.2 Workability

It is commonly acknowledged that when more NS is added to substitute cement, the flowability of cement mortar declines. Cement paste flow diameter was investigated by Berra et al. (2012). It was found that adding 0.8 weight percent NS caused the flow diameter to drop from 227 cm^2 to 56.8 cm^2. After using 3.8 weight percent NS, the flow diameter was further decreased to 13.9 cm^2. The slump value dropped as the concrete's NS content rose (Mukharjee and Barai ,2014). Water is drawn to the surface of NS due to their large surface area and unsaturated bond content in terms of formation of Silanol Groups (Si–OH). As a result, the amount of water required to keep the concrete mixture fluid becomes inadequate as some water is utilized in Silanol bond formation (Mukharjee and Barai, 2020). According to Ghafari et al.(Ghafari et al. 2014), the maximum amount of NS that might be added for an acceptable range of slump flow is 3% by weight. In case of recycled aggregate concrete (RAC), the NS substitution decreased the slump values more than the normal concrete because of the adhered mortar in case of RAC, which is quite pervious and absorbs water (Mukharjee and Barai, 2014). As per Elkady et al. (2013) concrete workability significantly increased by approximately 35% when nS was well-dispersed and de-agglomerated because the ultra-fine particles entrapped free makes it easier for the particles to roll over one another. It suggests that improving the dispersion of nanoparticles enhances the workability of concrete. As per

Elrahman et al. (Elrahman et al., 2019) reported that the introduction of NS in cement system results in drop in consistency and an increase in the fresh mixture's viscosity. When NS is substituted for cement in self-compacting light weight aggregate concrete, the consistency of the mixture is improved and the fresh density is decreased (Gesoglu et al., 2016). When the characteristics of NS-containing self-compacting concrete was examined, they discovered that NS increases the cohesiveness of the mixture (Collepardi et al., 2002). It lessens segregation and bleeding water, which ultimately enhances the rheological behaviour parameters of concrete in fresh state.

5. EFFECT OF NANO SILICA ON HYDRATION CHARACTERISTICS

From the calorimetry curve shown in figure 6, it is evident that the "rate of reaction" is increasing as NS content is increasing, the difference being more pronounced at early ages (Rupasinghe et al. 2017). Furthermore, compared to clean cement paste, the dormant period of cement with 2 weight percent NS is shortened by 4 hours, according to the results of a calorimetric test (Palla et al. 2017). It was demonstrated by Asgari et al. (2016) that the cement hydration rate in NS solution is significantly higher than that in water solution. Apart from the technique of isothermal calorimetry, solid-state nuclear magnetic resonance (NMR) spectroscopy can also be employed to investigate the impact of NS on cement hydration (Dimitrijevic, Henglein, and Meisel, 1999; Minet et al., 2006; Ek et al., 2001). Singh et al.(Singh et al. 2017) using NMR studies found that adding 10% NS can raise the hydration rates of C_3S by roughly 51% and 80% at 24 and 36 hours of hydration, respectively. In addition, Shih et al. (2006a) found that at 56 days, the hardened cement composite with 0.6 weight percent NS had a 2.8% higher hydration degree. NS incorporated specimens have a greater C-S-H content than the control and SF-incorporated sample, according to TGA quantification of the hydration products of FA-concrete (Singh et al. ,2019). The research indicates that the NS particles enhance the CSH packing density and quickens the early hydration rate (Singh, Ali, and Sharma, 2016). Due to their large surface area, NS act as a location for the precipitation of calcium silicate hydrate (CSH).

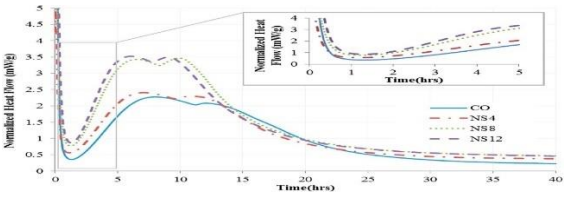


Figure 6. Showing isothermal calorimetric curves for different compositions with CO, NS4, NS8 and NS12 representing nano-silica substitution of 0%,4%,8% and 12% by weight in cement paste, respectively. First five hours of hydration heat is zoomed in and shown in top right corner. Taken from (Rupasinghe et al., 2017)

As a result, CSH forms on the surface of NS particles before C_3S and C_3A, which are the reactive phases. As maximum concentration of [OH−] ions reach earlier at the end of the pre-induction phase rather than at the end of the induction period, a fast precipitation and consumption of Ca^{2+} and OH^- from silica species will result on a shortening of the induction period (Korpa, Kowald, and Trettin, 2008). In order to examine impact of NS on crystal size of portlandite, portions of the diffractogram showing the development of portlandite crystals during the first 24 hours of hydration were examined as shown in figure 7 (Korpa, Kowald, and Trettin, 2008). It was found that the paste containing NS has the least amount of crystalline portlandite. The smaller size of the portlandite crystals formed in case of cement paste containing NS was explained by the wider portlandite peak formed in diffraction pattern as shown in part (c) of figure 7. When roughly estimated for crystal size by Korpa et al. (Korpa, Kowald, and Trettin 2008), the average crystal size of the paste containing SF was measured at 155 nm, while the paste containing NS had an average size of 53 nm.

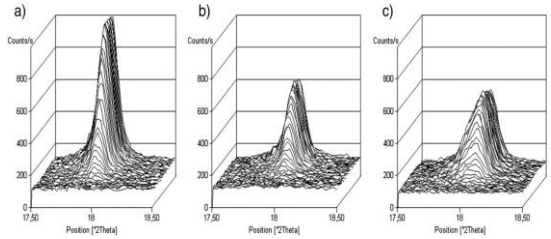


Figure 7. Showing portions of diffraction patterns showing the development of Portlandite crystals over the first twenty-four hours of hydration reactions for following three cases :(a) Cement paste without any pozzolana (b) Cement paste containing SF (c) Cement paste containing NS. Taken from (Korpa, Kowald, and Trettin 2008).

6. NUCLEATION EFFECT CAUSED BY NANO SILICA ADDITION

In addition to serving as a filler, nano-silica particles also speed up the cement hydration process by increasing the number of nucleation sites available for the cement particles. As per studies conducted by Singh et al. (2016), the highest nucleation effect of NS was observed at 8 hours of hydration, when the rate of product formation exceeded the control mix .66% greater CSH and 61% more CH with 10% NS addition was observed from the findings of XRD and TGA analyses (Singh, Ali, and Sharma, 2016). An early pozzolanic reaction will occur on the silica surface when nano-silica is introduced into the cement's hydration system, forming C-S-H seeds (Madani, Bagheri, and Parhizkar 2012; Land and Stephan 2012). The mechanism for the seeding effect schematically as explained by Yu et al. (2014) is shown in figure 8(a), which states that the nano silica addition in the cement system generates additional sites, which provides surface for the hydration products to form. Thus, in a way accelerating the hydration by acting as potential CSH seeds, as now apart from hydration products to form on cement surfaces, additional surfaces have been generated. In the second part of figure 8, that is 8(b), the effect of amount of NS addition in the cement mix is explained by Yu et al. (2014) . It is shown that in the first case, as the amount of NS in the mix is low (below 2 percent), the

additional surfaces for hydration products is limited, consequently porosity is large. In the second case, when the amount of NS is too large (more than 5 percent), again the observed porosity is large because although the potential CSH seeds increases in this case and hence hydration product become denser, the increased viscosity of mix on account of excessive NS addition causes large amount of air to be entrapped, thus posing problem. Therefore, it is recommended to add optimum amount of NS to balance out negative effect of entrapped air with positive seeding effect.

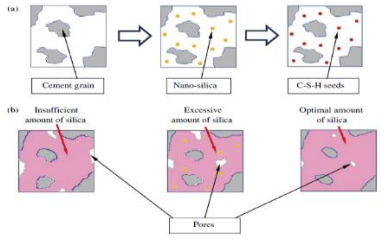


Figure 8. Showing (a) Seeding effect due to NS addition (b) impact of the NS content on UHPC porosity and microstructure development. Taken from (Yu, Spiesz, and Brouwers, 2014)

7. EFFECT OF NANO SILICA ON MECHANICAL STRENGTH OF CONCRETE

7.1 Compressive strength

The studies have shown that even a slight percentage incorporation of NS in concrete brings about significant increment in values of early age compressive strength (Shaikh, Supit, and Sarker, 2014; Sun et al., 2019; VYAS et al., 2020; Ghafari et al., 2014). Optimised dosage has been found within the range of 2 to 3% (Praveen Kumar and Dey, 2023; Jalal et al., 2015; Kumar, Kumar, and Kujur, 2019; Shaikh, Supit, and Sarker, 2014; Sun et al., 2019; Jaishankar, T. Poovizhi, and Saravana Raja Mohan, 2018; Du, Du, and Liu, 2014; Ghafari et al. 2014; Min Hong Zhang and Islam, 2012b). Naji Givi et al. (Naji Givi et al. 2010) after conducting studies on two different sizes 15nm and 80nm concluded that 15nm sized NS was having more compressive strength during early ages of curing while 80nm sized NS was exhibiting more compressive strength at 90 days. More heterogeneous nucleation sites are present in nanoparticles with smaller sizes, which results in more early age strength according to following equation 1(Porter, Easterling, and Sherif 2021; Müller 2007).

$$I = A.N_T.\exp\left(\frac{-\Delta G}{KT}\right) \quad (1)$$

Where, I denote nucleation rate, ΔG stands for critical free energy required for nucleation denotes absolute temperature, K stands for Bultzman's constant, A is a constant and N_T indicates the number of nucleation sites. Thus, for a given temperature, increasing the number of nucleation sites would enhance the nucleation rate of C–S–H gel formation. Although 80nm sized NS was having fewer nucleation sites compared to 15nm NS, but there was a significantly greater chance of achieving the crucial nucleation volume, resulting in better C–S–H gel formation at curing period of 90 days (Naji Givi et al., 2010). Naji Givi et al. (2013) also observed that in binary blended system using NS, the positive increment in values of compressive strength was seen up to 1.5% replacement before seeing a downward trend at 2%, still the strength at 2% replacement was more than the control mix (Naji Givi et al., 2010). As per Li et al. (2006) homogeneous dispersion of NS for size ranging between (5nm to 15 nm), in case of large dosages becomes challenging to achieve and as a result causes weak pockets formation in concrete, resulting in loss of compressive strength. As per Singh et al.(Singh et al., 2019) employing NS proved to be beneficial in improvement of compressive strength at early ages in nano engineered FA concrete. In another study by Yu et al. (2014), compressive strength improvement was found to be more pronounced at 3 days curing as compared to curing for 7 days and 28 days. This is suggesting that at an early age, NS particles significantly accelerate the cement's hydration process.

7.2 Flexural strength

The size effect of NS in determining the flexural behaviour of the concrete was evident from the study of Givi et al (2010), in which the higher flexural strength values was seen for 80nm sized NS as compared to 15nm NS. The comparison result remained same at all days of curing and at dosages up to 2 percent cement replacement. In another study, Givi et al. (2011) observed that in case of specimens cured under water, for up to 1 percent NS addition, flexural strength increased, but beyond that substitution, the flexural strength went down. However, in case of specimens cured under lime solution, these increasing values of flexural strength was observed up to 2 percent NS addition in the mix. After 28 days of curing, in case of natural coarse particles as aggregates, the changes in flexural strength values after 3% NS usage, was seen from 4.40 MPa to 4.96 MPa while the same amount of NS addition in case of concrete containing recycled aggregates caused the flexural strength values to shift from 3.98 MPa to 4.61 MPa (Mukharjee and Barai, 2014). In lightweight aggregate concrete, Elrahman et al. (2019) demonstrated a partial improvement in initial flexural strength, noted for a low concentration of NS. As per Yu et al. (Yu et al. ,2014)the flexural strength of HPC can be greatly increased by adding NS to the concrete together with hybrid fibres made of polypropylene and steel.

7.3 Modulus of elasticity

Soloma et al. (2013) measured an increased elastic modulus in case of concrete containing NS relative to the control concrete, suggesting that the NS addition leads to more stiffness properties. The elastic modulus of all concrete mixes with FA contents ranging from 25 to 75 percent, as well as concrete without FA, was improved by adding 2% NS (Sivasankaran, Raman, and Nallusamy, 2019). The most recent study found that adding NS to concrete up to 3% only slightly increased its elastic modulus (Mukharjee and Barai, 2020). As per a study by Belkowitz et al. (2015), utilizing the larger sized NS particles at higher dosages and the smaller sized NS at lower dosages produced the highest modulus of elasticity values. Thus, the use of NS boosts the concrete's modulus of elasticity in addition to its compressive strength.

8. EFFECT OF NANO SILICA ON DURABILITY ASPECTS

8.1 Porosity

The pore structure can be measured using a variety of techniques, including gas adsorption, helium flow, mercury intrusion porosimeter (MIP) and optical approach (Zhou, Ye, and van Breugel, 2010; X. Wang et al., 2019; Abell, Willis, and Lange, 1999). Because of its ease of use, speed, and pore diameter measuring range, the MIP approach is widely employed to characterize the pore structure in porous material (Zhou, Ye, and van Breugel, 2010; X. Wang et al., 2019; Abell, Willis, and Lange, 1999). Singh et al. (Singh, Ali, and Sharma, 2016) investigated the porosity of cement paste after 24 hours of hydration as shown in figure 9. They reported that when NS was added, the capillary porosity value of C-S-H decreased from 29% to 12% at 10% NS addition by weight. And same amount of NS addition raised the gel porosity from 14% to 22% in comparison to the control cement paste. This shift in porosity distribution was also studied by Wang et al. (Wang et al., 2016), which is shown in figure 10. It can be inferred from the figure 10 that pore size distribution has shifted towards the finer side, with reduction in large sized capillary pores, as NS are added. Also, overall reduction in porosity has been observed, for instance, using 3% NS caused the cement paste's overall porosity to drop by 5.4% after 28 days (Wang et al., 2016). As per Huang et al. (Huang et al., 2020), the finer NS significantly decreased the mix's overall porosity and pore connectivity compared to the course one. Kong et al. (Kong et al., 2019) investigated comparison between usage of two different forms of NS, one was colloidal silica sol consisting of mono-dispersed nano-silica, and second was nano-silica powder with agglomerates in µm size range. They found that the volume of the macropores decreased more when colloidal sol with mono dispersed nano silica were added than when NS powder with agglomerates were added. When colloidal sol with mono dispersed nano silica was added, the volume of micropores (capillary pores) in the 100 nm–5 µm range was lowered slightly less than when NS powder with agglomerates was used(Kong et al. 2019).

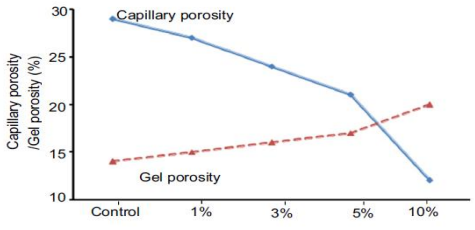


Figure 9. Showing capillary and gel-porosity in C_3S at 24 h of hydration with different NS dosages (Singh, Ali, and Sharma, 2016)

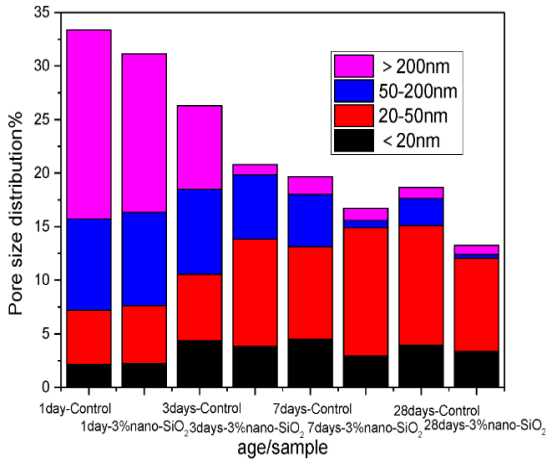


Figure 10. Showing porosity comparison between control mix and 3% NS addition at 1 day, 3 days, 7 days and 28 days respectively (Wang et al., 2016)

8.2 Water absorption

One important intrinsic feature that has a negative impact on the longevity of reinforced concrete is the water permeability. Compared to control mix, the water absorption coefficient dropped from 1.35 to 0.95 (m^2/s), while the water absorption value reduced from 4.8% to 3.78% when 2 % NS was added (Givi et al., 2011; Naji Givi et al., 2011). The result could be explained by the "filler effect" and "pozzolanic reaction" provided by the NS which has made the microstructure denser. The results of experiment demonstrated that adding NS can reduce the water absorption rate of the control concrete during 28 days and 90 days curing periods (Supit and Shaikh, 2015). In comparison to the reference concrete, introducing NS to cement paste formulations reduces its sorptivity and water absorption (Ghafari et al., 2014). This indicates that on account of pozzolanic activity of NS, there are fewer capillary pores connections as additional hydration products are caused due to NS addition.

8.3 Chloride ion penetration

He and Shi (He and Shi, 2008) used various kinds of nanomaterials to investigate the microstructure and chloride permeability of Portland cement mortar. The value of D_{Cl^-} (Diffusion coefficient of chloride anion) fell by 61.7% from 6.82×10^{-11} m^2/s (control specimen) to 2.61×10^{-11} m^2/s when one percent NS (by weight of cement) was added and electromigration test was performed. Rapid chloride permeability test was performed by Zhang and Li (Zhang and Li, 2011) in order to evaluate chloride ion permeation in the concrete mix with the addition of NS. It was observed that when compared to the control, the cement containing 1 weight percent NS had a chloride diffusion coefficient that was 18.04% lower. But when the amount of NS content increases, the reduction in value drops. Cement containing 3 weight percent NS had a chloride diffusion coefficient that was 10.49% lower than the control. This might be the result of NS being agglomerated when a lot of content was used. As per Liu et al. (Liu et al., 2018), the reduction in water to cement ratio brings about more pronounced effect of NS addition on chloride ion permeation and diffusion coefficient. This could be due to enhancement of NS role

in improving hydration when the water to cement ratio are at lower values. Du et al. (Du, Du, and Liu, 2014) demonstrated that a relatively tiny dosage (0.3%) of NS significantly reduced the concrete's chloride ion permeability. The reduction in chloride migration and diffusion coefficients values were observed to be 28.7% and 31%, respectively, at 0.3% NS dose.

8.4 Freezing thawing resistance

The freezing of concrete can occur during construction in cold weather resulting in the weakening and surface cracking of concrete. Frost damage can be assessed by looking at how it affects the development of interior cracks and scaling on the concrete's surface (Şahin, Akkaya, and Taşdemir, 2021). Loss in dynamic modulus of elasticity is typically used to identify internal degradation and surface scaling can be calculated using the mass of particles that have been loosened from the surface. Introduction of NS to concrete increased the pavement's resistance of the bridge deck to frost, as per the experimental research conducted in China's cold climate regions (Cheng and Shi, 2019) . The mass loss rate of concrete with NS was found to be 0.87% lower than that of concrete without NS and dynamic elastic modulus was 18.2% greater than the concrete without NS after 300 cycles of freezing and thawing. Specifically, when the frequency of freeze-thaw cycles was rising, it became increasingly evident how NS enhances concrete's ability to withstand frost. Behfarnia and Salemi (2013) conducted an experiment in which after 50, 150, and 300 cycles of freezing and thawing, the specimens' compressive strength was assessed. It was found that the amount of strength loss in the NS containing concrete was significantly less than in the normal concrete. For instance, after 300 freezing and thawing cycles, concrete containing 3 percent by weight NS only lost 28% of its strength, compared to 100% loss for normal concrete. Similarly, compressive strength reduction in samples with 5 percent and 7 percent replacement of cement by NS, was observed to be 16.28% and 23.2%, respectively after 300 cycles. This could be explained by the reasoning that NS apart from imparting filling ability on account of its extreme fineness, also makes the microstructure dense by utilizing the lime crystals through pozzolanic action. As a result of having a denser microstructure than regular concrete, concrete containing NS absorbs less water and is more frost resistant.

8.5 Calcium leaching

Hydration products gradually dissolving as a result of calcium ions migrating to the pore solution can be called Calcium Leaching. Due to its stability at calcium concentrations greater than 20 mol/m^3, CH is a key component in the calcium leaching process(Berner 1988). Typically, basic solution (pH > 12.5) exists in the micropores in cement hydration products. When the pH falls below 12.5, calcium ions will begin to leach from the cement hydration products. The Ca/Si ratio falls and the precipitation of C-S-H gel is slowed down as the amount of calcium ions lowers, resulting in the deterioration of cement hydration products' mechanical strength. Gaitero et al.(Gaitero, Campillo, and Guerrero, 2008) observed that the reduction in cement paste samples' 28-day compressive strength after 9 days due to immersion in 6 M ammonium nitrate solution was 73% respectively. 6 wt.% NS addition caused this reduction percentage to drop to 9%. NS can produce a significant quantity of C-S-H gel by a pozzolanic reaction with both CH (calcium hydroxide) and the Ca^{2+} ions during the dissolution, thus causes a reduction in calcium leaching rate. The other factor contributing to the decrease in calcium leaching is the lower porosity, which makes it harder for calcium ions to enter lower pH environment and slows down the pace of degradation.

9. POZZOLANIC ACTIVITY OF NANO SILICA

The methods used to investigate the pozzolanic activity fall into four primary categories: X-ray diffraction, electrical conductivity (XRD), chemical extraction, and thermogravimetric analysis (TGA). The first to develop a technique for assessing pozzolanic activity by measuring electrical conductivity were Raask and Bhaskar (Raask and Bhaskar, 1975). The test was performed for determination of pozzolanic activity of pulverized fly ash (pfa) assessing its electrical conductivity in a solution of hydrofluoric acid. Luxan et al. (Luxán, Madruga, and Saavedra, 1989) proposed pozzolanic index, which was determined by comparing a calcium hydroxide pozzolanic suspension's initial and ultimate electrical conductivity. The suspension's electrical conductivity decreases as a result of the reaction between the pozzolanic substance and calcium hydroxide.

According to Qing et al. (2007) NS has a considerably higher pozzolanic activity than silica fume. NS with a large number of unsaturated surface bonds, "Si-O- and "Si-" as compared to SF results in faster reaction between SiO_2 and Ca $(OH)_2$ (Qing et al., 2007)Results from thermogravimetric investigation showed that NS consumes a lot more $Ca(OH)_2$ than silica fume does, particularly at early hydration age (Ghafari et al., 2014). The addition of NS resulted in an increase in pozzolanic activity and nucleation sites, which raised the hydration heat in HVFA concrete (Li, 2004).

10. MICROSTRUCTURE OF CEMENT PASTE CONTAINING NANO SILICA

Said et al. (Said et al., 2012) found by thermal and microstructural study that the NS content had a significant impact through filler and pozzolanic effect, which allows the nanoparticles to enhance the pore structure of the NS-containing concrete. The NS addition resulted in reduction of portlandite crystals, as they were utilized by NS particles, on account of pozzolanic reaction to form secondary CSH (Esmaeili and Andalibi, 2013). It was seen that these secondary CSH showed lower Ca/Si ratios and higher stiffness values(Quercia and Brouwers 2010). Even with a low dosage of NS, Du et al. (Du, Du, and Liu, 2014)found that the concrete's ITZ (interfacial transition zone) became more homogenous and less porous. The ITZ between aggregates and the cement matrix in SCC (self-compacting concrete) containing FA and colloidal NS was found to have disappeared (Durgun and Atahan, 2018). Coarser pyrogenic NS resulted in more compacted and denser microstructure as compared to finer NS because of the agglomerates formation tendency in finer pyrogenic NS (Khaloo, Mobini, and Hosseini, 2016).

Mondal.et.al (Mondal et al., 2010) showed that the average chain length of C-S-H gel is increased by nano silica, as demonstrated by the ^{29}Si MAS-NMR (Solid state Magic Angle Spinning-Nuclear Magnetic Resonance) spectra of the cement pastes containing nano silica. The amount of high stiffness CSH increased from 38% to 50% as NS content increased from 6% to 18%, thus illustrating that addition of NS alters the relative amounts of the two C-S-H phases, favouring the high-stiffness phase's development over the low-stiffness phase (Mondal et al., 2010).This further explains the reasoning behind reduction in calcium leaching rate with the addition of NS (Gaitero, Campillo, and Guerrero, 2008). The polymerization of silicate chains is accelerated by SNPs, according to FTIR data (Singh, Ali, and Sharma, 2016). The inclusion of NS was found to raise the mean chain length (MCL) values in numerous investigations (Moon et al., 2016; Monasterio et al., 2015). The MCL of the hydration products of cement rose by 10.6%, from 3.11 to 3.44 (Moon et al., 2016).In a study by Gaitero et al. (Gaitero, Campillo, and Guerrero, 2008), three types of colloidal dispersion (CS) form of NS named CS1, CS2 and CS3 and fourth dry powder (ADS) form were studied and compared for different attributes. Their properties are tabulated below in Table 1 as per (Gaitero, Campillo, and Guerrero, 2008).

Naming	Particle size	SiO_2 content (wt.%)	Form
CS1	30 nm	45%	Colloid
CS2	20 nm	20%	Colloid
CS3	120 nm	40%	Colloid
ADS	15 nm (individual particle size) 1400 nm (agglomerated size)	95%	Powder

Table 1. Showing different types of colloidal NS utilized in its study by Gaitero et al.(Gaitero, Campillo, and Guerrero 2008)

To characterize the degree of condensation of silicate units, Q^n terminology is employed and is shown in figure 11, taken from (Gaitero, Campillo, and Guerrero, 2008).The suffix number exponentially attached with Q, provides details regarding the connectivity of every silicon atom with the oxygen atom, for instance, Q^0, Q^1, Q^2 indicates those silicon atoms which are connected to zero, one and two oxygen atoms respectively. Q^2 (1Al) represents the bond between an atom of silicon and an atom of aluminum, apart from connection with two oxygen atoms. In the NMR spectra, each of these Q^0, Q^1, Q^2, Q^3, Q^4 or Q^2 (1Al) links gives rise to a distinct peak. The test results of ^{29}Si MAS-NMR as found by Gaitero et al. (2008), is shown in figure 12. It shows that when the material is cured for one day, unreacted NS was clearly visible (Q4 peak~110 ppm), but by the end of the curing procedure, such peak was missing. In a similar study by Shih et al. (2006b), the NMR curves of cement paste without NS, and with 0.6 percent NS was analysed, as shown in Figure 13(a) and 13(b) respectively. An index that can be used to determine the relative quality of Portland cement composite is Q^2/Q^1 ratio, with a greater value denoting better quality mechanical characteristic (Kjellsen, 2000). The Q^2/Q^1 ratio as calculated by Shih et al. (Shih, Chang, and Hsiao, 2006b) went up to 0.2927 from 0.0806, when NS was added. This outcome demonstrates that the addition of NS improved the cement composite's compressive strengths. Furthermore, the amorphous nature of the hardened cement paste's microstructure can be assessed using the change of FWHM (full width at half maximum) values at Q^n peaks (Cong and Kirkpatrick, 1996). The FWHM value of the Q^1 peak in cement composite significantly decreased by 29.23% following the addition of NS, thus suggesting that the O-Si-O net's organization order appears to become regular with a pattern of sharing an oxygen atom (Shih, Chang, and Hsiao, 2006b). Further as calculated from results shown in figure 10 by Shih et al. (Shih, Chang, and Hsiao, 2006b) by at 56 days, the hardened cement composite containing NS had a degree of hydration that is roughly 2.8% higher than that of pure hardened cement paste. Consequently, it is observed that the addition of NS improved the microstructures of the hydrated cement composite by looking at the improvements in degree of hydration and Q2/Q1 ratio values, as well as the decrease in FWHM value (Shih, Chang, and Hsiao, 2006b).

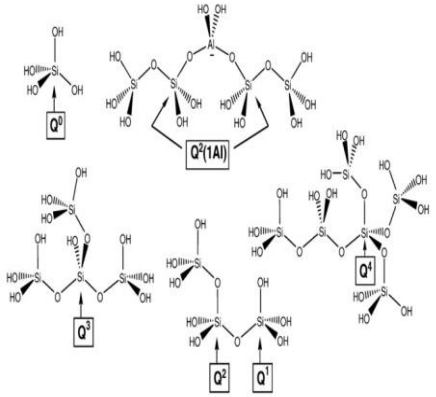


Figure 11. Schematic representations of the various silicate chain types found in the C-S-H gel, along with their corresponding NMR analytical terminology, taken from (Gaitero, Campillo, and Guerrero, 2008)

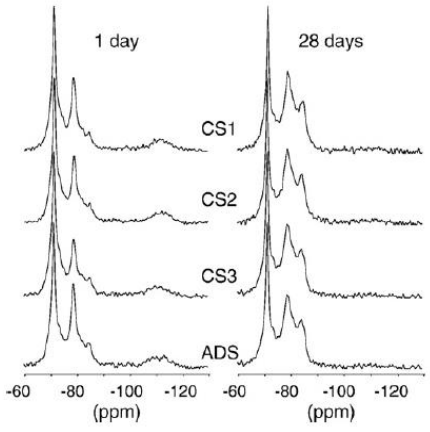


Figure 12. Showing ^{29}Si MAS-NMR spectra results of different types of silica nanoparticles as mentioned in Table 1, after 1 (left) and 28 (right) days of curing (Gaitero, Campillo, and Guerrero, 2008)

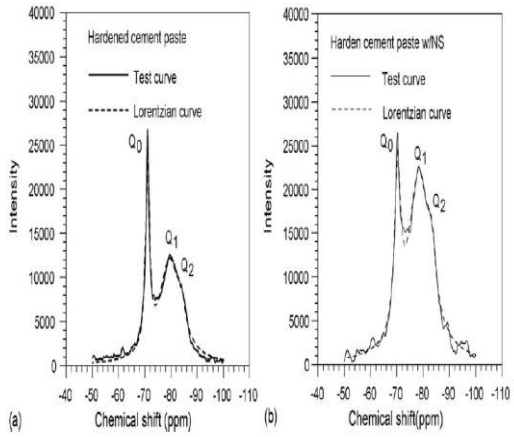


Figure 13. Showing NMR curves of hardened cement paste: (a) without NS and (b) with addition of NS (Shih, Chang, and Hsiao, 2006b)

11. APPLICATION OF NANO SILICA MODIFIED CEMENT COMPOSITES

NS can improve cementitious composites' longevity and raise the safety of buildings like dams and airport pavements made of concrete (Biricik & Sarier, 2014). NS modified concrete is appropriate for use in fire-resistant construction, such as tunnels and basement buildings, due to its improved thermal stability and less cracking at high temperatures. Cementitious composites' thermal conductivity was reduced by 4.5% as compared to control when 4.5% NS was added (Bao et al., 2019). By lowering the cement content, increasing durability, and utilizing industrial by-products, the use of NS in concrete promotes sustainability and is appropriate for green construction projects that seek LEED certification or other sustainability requirements (Council, 2022; Di Gaetano et al., 2023; Hanif et al., 2017; Liu et al., 2016; Mei et al., 2018). Vashistha et al. (Vashistha et al., 2019) investigated how the composition of C_2S clinker was affected by NS generated from paper mill waste. When NS was present, the production temperature of C_2S dropped from roughly 1200°C to roughly 900°C. The use of NS-modified concrete in maritime constructions such as piers, docks, and seawalls are advantageous due to its enhanced resilience against chemical attacks and penetration of chloride ions (Brent Rollins, 2016; He & Shi, 2008). NS has been applied to building materials to increase cementitious composites' ability to withstand skidding and sliding tendency of vehicle tyre in contact. Adamu et al. (Adamu et al., 2018) examined how a hybrid of NS and crumb rubber affected the skid resistance and durability of high-volume fly ash roller-compacted concrete pavement and found that NS usage increased skid resistance up to 20 percent. In cementitious composites, NS has a significant impact on the shielding of ray radiation. Studies were conducted to investigate the impact of NS on the shielding capabilities of Steel Slag Heavyweight Concrete (SSHWC) against X-ray and γ-ray radiation (Khalaf et al., 2021). Evidently, it was found that NS can improve SSHWC's capacity to isolate X-rays and γ-rays through shielding. This makes NS modified composite very useful in nuclear reactor material uses and where radioactive wastes has to be stored. Thus, NS concrete finds plethora of uses from high rise buildings, pavement materials, green buildings, precast concrete segments, increasing skid resistance of road, reducing carbon footprint to even shielding against radioactive rays.

12.CONCLUSION

The critical effects of NS addition on the mechanical, fresh state properties, and durability characteristics of cementitious composites have been summarized in this paper. The following are the primary findings that may be made based on the review:

- The addition of NS reduced the cement paste's workability. It takes more water molecules to moisten the surface of NS because of its large surface area.
- The cement paste setting times, both initial and final, decreased as the NS content increased.
- Adding up to 2-3% of NS could improve the mechanical properties and durability. This is due to NS's ability to quicken the hydration process and quickly absorb lime for pozzolanic reaction.
- Because of its pozzolanic reactivity and nucleation impact, adding NS to cement composites can accelerate the hydration reaction rate, particularly in the early stages of hydration.
- Because NS can participate in the pozzolanic reaction to consume CH content, including NS can considerably reduce the loss from calcium leaching. NS enhances calcium stabilisation by changing the internal structure of the C-S-H gel in the direction of longer silicate chains.
- The application of NS and SCM together improves concrete's mechanical strength, hydration and reduces pore connectivity. Furthermore, the cement matrix's nano-sized gaps and cracks are efficiently filled by NS particles, enhancing the material's overall structural integrity and durability.
- However, the mechanical and durability qualities of cement-based materials may suffer if significant volumes of NS particles (more than 5% replacement of cement) are applied. This is explained by the high specific surface areas of the NS particles, which raise the risk of nanoparticle agglomeration and, as a result, lead to poor dispersion of the particles in the matrix based on cement. To ensure optimal performance gains, it will be imperative to address these problems through the development of surface-modified NS and enhanced dispersion techniques.

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