

Exploring The Advancement Enabled by Nano-Silica in Cement-Based Materials: A Comprehensive Review

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ABSTRACT

At present, the focus of current research lies predominantly in unraveling the fundamental science of cementitious materials at the nano and atomic levels. Simultaneously, researchers are working diligently to enhance the durability and sustainability of concrete, leading to a significant improvement in the mechanical properties of cementitious materials through the incorporation of nano-silica. This review paper aims to provide a comprehensive overview of the influence of nano-silica addition on the mechanical, durability and microstructural characteristics of paste, mortar and concrete. It also offers insight into the current developments in the application of nano-silica in these materials. Lastly, the paper discusses the potential future trends and implications of using nano-silica in cement-based materials.

Keywords: Nanoparticles; Concrete; Durability; Mechanical Properties; Microstructure; Scanning electron microscope (SEM); X-Ray Diffraction (XRD).

1. Introduction

Cementitious materials hold immense importance in the construction industry, yet their applications should not obscure their inherent complexity. They are composite materials with intricate, multi-scale internal structures that have evolved over centuries. Specifically, the cement paste matrix is a porous substance comprising calcium hydroxide (portlandite), aluminates, and unhydrated cement (clinker) embedded in an amorphous, nanostructured hydration product known as the Calcium-Silicate-Hydrate (C-S-H) gel (Gaitero, Campillo, & Guerrero, 2008). This gel dominates the hydration product in cement paste, not only due to its prevalence (making up 50-70% by volume) but also due to its remarkably favourable mechanical properties.

Nanoparticles, with their high surface-area-to-volume ratio, such as those with a 4-nm diameter, possess over 50% of their atoms on the surface, rendering them highly reactive (Wiesner & Bottero, 2007). The behaviour of these materials is primarily influenced by chemical reactions at the interface and their tendency to form agglomerates. When trying to saturate a larger surface area, this process reduces the available free dispersant water in aqueous systems within the mixture. Consequently, the inclusion of

nanoparticles significantly alters the behaviour of mortars and concrete affecting their physical, mechanical and microstructural development not only in the fresh state but also in the hardened state (Senff, Labrincha, Ferreira, Hotza & Repette, 2009).

In recent years, the use of nanoparticles has garnered significant attention across various application fields, enabling the creation of materials with novel functionalities. When ultrafine particles are incorporated into Portland cement paste, mortar or concrete, materials with distinct characteristics from conventional ones are obtained (Lea, 1998; Li, Xiao, Yuan, & Ou, 2004; Neville, 1996). The performance of these cementitious-based materials is profoundly influenced by nano-sized solid particles, such as C-S-H or nano-sized porosity at the interfacial transition zone between cement and aggregate particles. Key properties affected by these nano-sized particles or voids include strength, durability, shrinkage, and steel-bond (Collepari *et al.*, 2005). For example, nano-silica nanoparticles can fill the spaces within the C-S-H gel, acting as nano-fillers. Additionally, through a pozzolanic reaction with calcium hydroxide, they increase the amount of C-S-H, leading to greater matrix densification, thereby enhancing the strength and durability of the material (Choolaei, Rashidi, Ardjmand, Yadegari, & Soltanian, 2012; Hou *et al.*, 2013; Zapata, Portela, Suárez, & Carrasquillo, 2013).

Previous research (Bjornström, Martinelli, Matic, Borjesson, & Panas, 2004; Lea, 1998; Qing, Zenan, Deyu & Rongshen, 2007) suggests that the inclusion of nanoparticles significantly modifies both the fresh and hardened state properties when compared to conventional mineral additives. Colloidal silica particles appear to substantially impact the C₃S hydration process (Bjornström *et al.*, 2004). Nano-silica reduces the setting time of mortar in comparison to silica fume (SF) (Qing *et al.*, 2007) and decreases bleeding water and segregation while improving the cohesiveness of mixtures in their fresh state (Collepari, Olagot, Skarp, & Troli, 2002). Nano-silica-added cement paste exhibits reduced setting time (Lin, Lin, Chang, Luo, & Cai, 2008; Singh, Agarwal, Bhattacharyya, Sharma, & Ahalawat, 2011; Singh, Bhattacharyya, & Singh, 2012), a shorter duration of dormant and induction periods of hydration, quicker attainment of the peak heat of hydration, and increased production of calcium hydroxide at early stages (Ltifi, Guefrech, Mounanga, & Khelidj, 2011; Senff *et al.*, 2009). When combined with ultrafine fly ash, it assures better performance than SF alone (Lea, 1998; Li, Xiao, & Ou, 2004; Li *et al.*, 2004).

Furthermore, mortar or concrete with SF demonstrates enhanced compressive strength when compared to formulations without additives (Jo, Kim, Tae, & Park, 2007; Li *et al.*, 2004; Li, Zhang & Ou, 2006). The addition of nano-silica augments the quantity of C-S-H and C-A-H in the paste (Tobon, Paya, Borrachero & Restrepo, 2012). Incorporating nano-silica into cement paste and mortar requires additional water to maintain workability (Quercia, Hüsken, & Brouwers, 2012). To avoid adverse effects on workability, Berra, Carassiti, Mangialardi, Paolini, and Sebastiani (2012) recommend a delayed addition of water, suggesting that instead of adding all the mixing water at once, a certain amount of water should be added later on. Nano-silica samples exhibit less strength loss after exposure to elevated temperatures (Lim, Mondal & Cohn, 2012). Mortar with a high volume of fly ash displays higher residual strength after exposure to 700°C, and dehydration of C-S-H generates calcium silicate, which acts as a new binding material, preserving residual strength (Rahel, Hamid, & Taha, 2012). The addition of nano-silica also modifies the porosity of cement paste and increases the average chain length of silicate chains (Gaitero *et al.*, 2008; Porro *et al.*, 2005).

Until now, only a limited number of studies have explored the utilization of nanoparticles, including nano-silica (nano-SiO₂), nano-titanium oxide (nano-TiO₂), nano-iron (nano-Fe₂O₃) and nano-alumina (nano-Al₂O₃) (Li, 2004; Shekari & Razzaghi, 2011). Furthermore, there is a relatively small body

of research dedicated to the production of nano-sized cement particles and the development of nano binders. Focusing the literature review on the use of nano-silica in cementitious compositions, research suggests that incorporating nanoparticles into cement matrices can enhance the durability and mechanical properties of cement-based materials. In particular, nano-silica (nS) has found extensive use in this field due to its high reactivity and substantial specific surface area, resulting in a high degree of pozzolanic activity. Additionally, nano-silica accelerates the dissolution of C₃S and the formation of C-S-H, with its activity inversely proportional to its size, while also providing nucleation sites for C-S-H (Jo, Kim, & Lim, 2007). Even minor additions (0.6 wt. % binder) of nano-silica have proven highly effective, offering improvements in the mechanical properties of cement-based materials comparable to much larger amounts of silica fume (SF), especially at early stages and for concretes with typical strength grades (Pourjavadi, Fakoorpoor, Khaloo, & Hosseini, 2012). Nano-indentation studies have indicated that the addition of nano-silica significantly alters the proportions of low stiffness and high stiffness C-S-H (Hou *et al.*, 2013; Mondal, Shah, Marks, & Gaitero, 2010).

2. Nanomaterials in Cement Composites

Extensive research is underway in the construction industry to enhance the performance of various building materials and the development of durable and sustainable concrete is a prominent focus, as evident in Table 1. Among the various nano-materials,

Table 1. Summary of use of Nano-silica in cementitious compositions

S. No.	Author	Concrete/ mortar	Materials used for comparison	Nano material used	Size of particle (nm)	Fineness (m ² /g)	Water cement ratio	Additive or replacement	Properties
1	Senff et al. (2009)	Paste	–	nS 1.5%, 2%, 2.5%	9	300	0.35	Replacement	Flow table, rheological behaviour, XRD
2	Qing et al. (2007)	Paste 1:0.22:0.025	SF 2%, 3%, 5%	nS 1%, 2%, 3%, 5%	15	160	0.22	Additive	Compressive strength, bond strength, Setting time
3	Ltifi et al. (2011)	Paste, mortar	–	nS 3%, 10%	9	300	0.5	Replacement	Compressive strength, flexure strength
4	Senff et al. (2010)	Mortar	SF 0–20%	nS 0–7%	9	300	0.35–0.59	Additive	Rheological, compressive strength, water absorption
5	Berra et al. (2012)	Paste	–	nS 0.8%, 3.8%	10	345	0.5	Additive	Physical mechanical stability test

6	Kawashima et al. (2012)	Mortar	Fly ash	nS 2%, 5%	20–10		0.43–0.45	Additive	Compressive strength, rate of heat of hydration
7	Hou et al. (2013)	Mortar	–	nS 5%	10 nm		0.4	Additive	Hydration heat, morphology, CH content
8	Stefanidou (2012)	Paste	–	nS 0.5%, 1%, 2%, 5%	14	200	0.34–0.51	Additive	Flexural and compressive strength, microstructure
9	Li et al. (2004)	Mortar	Nano-Fe ₂ O ₃ (3–10%) Silica fume (3–15%)	nS 3%, 5%, 10%	15 ± 5	15 ± 5	0.5	Replacement	Compressive and flexure strength, microstructure
10	Jo et al. (2007)	Mortar 1:2:45	SF 5%, 10%, 15%	nS 3%, 6%, 10%, 12%	40	60	0.5	Additive	Compressive strength, SEM, rate of heat of evolution
11	Zapata et al. (2013)	Mortar	SF	nS 1.5–6%	25	109	0.35, 0.4	Replacement	Compressive strength, SEM, XRD
12	Oltulu and Şahin (2013)	Mortar	Fly ash	nS 0.5%, 1.25%, 2.5%	12	200	0.4	Replacement	Compressive strength
13	Aly et al. (2012)	Mortar	Waste glass 20%, 40%	nS 3%	5	500	–	Replacement	Compressive, fracture, flexural and impact strength, DTA/TGA, SEM
14	Gaitero et al. (2008)	Paste	–	nS 20%, 40%, 45%, 90%	20, 30, 120, 1,400		0.4	Additive	Compressive strength, flexure strength
15	Zhang et al. (2012)	Concrete	SF	nS 0.5%, 1%, 2%	12 and 7	200.1 and 321.6	0.45	Replacement	Compressive strength, rate of heat of hydra-

									tion, porosity
16	Zhang and Islam (2012)	Concrete	Fly ash, slag and SF	nS 1%, 2%	12	200.1	0.45	Replacement	Compressive strength, rate of heat of hydration, Setting time
17	Pourja - vadi et al. (2012)	Concrete	SAP 0.1%, 0.3%	nS 0.5%, 1%	19	172	0.45	Replacement	Compressive strength, flexure strength
18	Li (2004)	Concrete	Fly Ash 50%	nS 4%	10 ± 5	640 ± 50	0.28	Replacement	Hydration heat, poresize
19	Naji Givi et al. (2010)	Concrete	–	nS 0.5%, 1%, 1.5%, 2%	15 and 80	160 ± 12 and 560 ± 32	0.4	Replacement	Compressive, flexure, and split tensile strength
20	Heidari and Tavakoli (2013)	Concrete	Ground ceramic powder 10–40%	nS 0.5%, 1%		200 ± 30	0.5	Replacement	XRD, compressive strength, water absorption

2.1. Improving Properties with Nano-Silica in Paste and Mortar

Numerous researchers have examined the influence of nano-silica on both pastes and mortars. The incorporation of nano-silica into cement pastes is undertaken to gain insights into the hydration process and the evolution of microstructures. This approach primarily serves as a means to delve into the fundamental science underlying cement hydration. On the other hand, investigations into mortars are carried out to explore their rheological properties and mechanical characteristics.

2.1.1. Optimizing Fresh Concrete Characteristics

The introduction of nano-silica into pastes resulted in a noticeable decrease in both the Initial Setting Time (IST) and Final Setting Time (FST), as documented by Senff *et al.* in 2009. Moreover, it was observed that as the content of nano-silica increased, the difference between IST and FST decreased, a finding supported by Ltifi *et al.* in 2011 and Qing *et al.* in 2007. When considering the impact of nano-silica on the rheological behaviour of cementitious mixtures, a consensus among most researchers exists regarding the substantial increase in water demand in comparison to control mixtures. In mortars, the loss of plasticity, accompanied by heightened torque and yield stress, became more evident in mixtures containing nano-silica due to its higher surface area, as reported by Senff, Hotza, Repette, Ferreira and Labrincha in 2010 and Senff *et al.* in 2009.

Furthermore, studies conducted by Berra *et al.* in 2012 and Kawashima, Hou, Corr and Shah in 2012 on the rheology of cement pastes with varying water-to-binder (w/b) ratios suggested the delayed addition of

water by reserving a certain amount to be added at a later stage. Additionally, the inclusion of nano-silica in cement paste and mortar resulted in an increased demand for water to maintain workability. The observation that the water content in mixtures with nano-silica in the fresh state directly influenced the required amount of water supports the notion that the addition of high surface area mineral particles to cement mixtures necessitates higher water quantities or chemical admixtures to preserve the mixture workability. In conditions where the water content remains constant, an increase in nano-SiO₂ content encourages particle packing, reducing the space between particles and limiting the presence of free water, ultimately leading to greater internal friction between solid particles, as explained by Ltifi *et al.* in 2011. The inclusion of nano-silica thickens the cement paste and accelerates the hydration process (Qing *et al.*, 2007). Fly ash's spherical morphology enhances the flow ability of cementitious materials, while nanoparticles increase stiffness due to their higher specific surface area, consequently reducing the fluidity of fly ash cement-blended nano-silica mortar, particularly as the nano-silica content increases (Kawashima *et al.*, 2012). Researchers investigated the pozzolanic activity of nano-silica and the adsorption of colloidal nano-silica (CNS) on calcium hydroxide (CH). They observed that the pozzolanic reaction of nano-silica was completed within 7 days of hydration (Hou *et al.*, 2013).

2.1.2. Enhancing Mechanical Performance in Concrete

In the context of nano-silica's impact on the mechanical strength development of cementitious materials, it was noted that the addition of nano-silica to Portland Cement (PC) pastes resulted in an enhanced compressive strength. The degree of improvement depended on various factors, including the nano-silica content, water-to-binder weight ratio (w/b), and curing time. Research encompassed multiple facets, including the examination of paste compressive strength (Berra *et al.*, 2012; Qing *et al.*, 2007; Stefanidou, 2012), bond strength (Qing *et al.*, 2007), and flexural strength (Stefanidou, 2012). Overall, a general trend was observed, with increased paste strength, especially in the early stages, associated with heightened pozzolanic activity. Significant enhancements were documented, with increases of approximately 17-41% at the 3rd and 28th days (Qing *et al.*, 2007), 7-11% at the 7th day (Berra *et al.*, 2012), and an average increase of around 25% upon the addition of 0.5-2% nano-silica (Stefanidou, 2012). The research indicated that the optimal nano-silica content for improved strength was approximately 5% (Qing *et al.*, 2007), 0.8% (Berra *et al.*, 2012), and 0.5% (Stefanidou, 2012). Furthermore, there were notable increases in bond strength, ranging from 16-43% at 7 days to 26-88% at 28 days (Qing *et al.*, 2007). Additionally, the flexural strength was observed to be at its peak at the 3-day mark with 1-2% nano-silica content (Stefanidou, 2012).

Li *et al.* (2004) conducted an investigation focusing on cement mortars incorporating nano-SiO₂ and nano-Fe₂O₃ to explore their potential for enhanced mechanical and smart properties. Their research revealed a significant improvement in compressive strength within the mortar mixes, ranging from 5.7 to 20.1% at 7 days and 13.8 to 26% at 28 days. Likewise, Jo *et al.* (2007) observed a substantial increase, with compressive strength in mortar mixes showing a range of 53.67 to 63.9% at 7 days and 52.5 to 62.7% at 28 days. They suggested that when using higher amounts of nano-silica, adjustments to water and superplasticizer dosages were necessary to prevent excessive self-desiccation and cracking in the specimens. These findings were corroborated by Ltifi *et al.* (2011) who noted a 6.9 to 16.9% increase in compressive strength at 90 days (Zapata *et al.*, 2013). In the context of fly-ash concretes to which nano-silica was added (Hou *et al.*, 2013; Kawashima *et al.*, 2012; Oltulu & Sahin, 2013) nearly identical results were observed, with early age strength gains reaching as high as 60%, which eventually aligned with the

strength of various mixtures (Kawashima *et al.*, 2012). Furthermore, there was a significant strength increase, averaging 63%, across different mixtures (Oltulu & Şahin, 2013). Moreover, flexural strength exhibited an increase of 28% at 7 days and 19.2 to 27% at 28 days (Li *et al.*, 2004), along with enhancements in fracture energy and impact strength at 28 days (Aly *et al.*, 2012).

2.1.3. Improving Durability in Concrete

Jo *et al.* (2007) found that nano-silica has a dual role: enhancing microstructure and promoting the pozzolanic reaction. Gaitero *et al.* (2008) observed reduced calcium leaching in cement paste with added nano-silica due to paste densification, portlandite transformation into C-S-H through pozzolanic reaction, and changes in C-S-H gel structure, leading to increased paste stability and stronger bonding. With increasing nano-silica content, mortars exhibited higher water absorption and apparent porosity, along with uncontrolled shrinkage and weight loss, most notably at 7% nano-silica by weight. In cement-based materials with fly ash, calcium hydroxide (CH) generated during cement hydration is essential for later-stage pozzolanic reactions. The addition of nano-silica significantly affects CH content in fly ash-cement paste, with more pronounced depletion when both nano-silica dosage and fly ash replacement ratio are high (Kawashima *et al.*, 2012). Hou *et al.* (2013) observed accelerated cement hydration and gel structure maturation in paste with colloidal nano-silica (CNS). This acceleration results from improved cement particle dissolution and the preferential precipitation of hydration products on the CNS particle surface. It's important to note that while CNS speeds up cement hydration in the early stages, it may hinder later phases.

2.2. Enhancing Concrete Performance with Nano-Silica

The incorporation of nano-silica into cement concrete represents a direct approach in the utilization of nanomaterials. Researchers have conducted studies on the mechanical and durability properties, as well as the microstructure analysis of concrete with nano-silica, as detailed below.

2.2.1. Characteristics of Fresh Cement-Based Materials

Several researchers have observed a reduction in setting times when nano-silica is introduced into concrete, mirroring the findings observed in pastes and mortar (Zhang & Islam, 2012; Zhang, Islam, & Peethamparan, 2012). Furthermore, the inclusion of nS in varying quantities resulted in decreased initial and final setting times, alongside reports of increased viscosity and yield stress, as documented by Pourjavadi *et al.* (2012).

2.2.2. Enhancing Mechanical Properties with Nano-Silica

Li *et al.* (2004) reported a substantial 81% increase in 3-day compressive strength, and a similar trend was observed in later stages with the inclusion of 4% nano-silica in high-volume fly ash concrete. Naji Givi, Abdul Rashid, Aziz, and Salleh (2010) also documented heightened compressive strength at all age intervals for concretes blended with nano-silica, up to a maximum limit of 2%, utilizing average particle sizes of 15 and 80 nm. These findings remained consistent for split tensile and flexural strength. Pourjavadi *et al.* (2012) reported a decrease in compressive strength due to the adverse influence of superabsorbent polymer when used in conjunction with nano-silica. However, this effect did not extend to flexural strength, which remained unaffected. An increase of approximately 23-38% at 7 days and 7-14% at 28 days in the compressive strength of nano-silica concrete was observed. In contrast, the increase in

flexural strength was comparatively modest, averaging 9.4%. Zhang and Islam (2012) and Zhang *et al.* (2012) introduced GGBFS, fly ash, and slag into their study, observing a 22% increase in compressive strength at 3 days and an 18% increase at 7 days for concretes incorporating GGBFS. In the case of fly ash and slag, the increase was even more pronounced at 30% at 3 days and 25% at 7 days. Heidari and Tavakoli (2013) incorporated nano-silica into ground ceramic concrete, resulting in an early-stage improvement in strength.

2.2.3. Improving Durability with Nano-Silica

Numerous researchers (Li, 2004; Zhang & Li, 2011; Zhang *et al.*, 2012) have reported significant reductions in concrete's water absorption, capillary absorption, water absorption rate, and water permeability. Introducing 4% nano-silica into the mix refined the distribution of pore sizes and reduced porosity, even during short-term curing (Li, 2004; Zhang & Li, 2011). Furthermore, an increase in the dosage of nano-silica led to a decrease in capillary porosity (Zhang *et al.*, 2012). Incorporating nano-silica also resulted in a decreased water absorption capacity in concrete (Jalal, Pouladkhan, Norouzi, & Choubdar, 2012; Zhang *et al.*, 2012). Researchers noted an improved resistance to chloride penetration in concrete with the addition of nano-silica (Jalal *et al.*, 2012; Zhang & Li, 2011). Zhang and Islam (2012) investigated the behaviour of high-volume fly ash and slag concretes with nano-silica, revealing that nano-silica not only reduced the duration of the dormant period during hydration but also accelerated the hydration process. Additionally, the incorporation of nano-silica in fly ash and slag concrete led to a reduction in chloride ion penetration.

3. Microstructure Analysis in Nano-Silica-Cement Composites

Multiple studies (Aly *et al.*, 2012; Hou *et al.*, 2013; Ji, 2005; Jo *et al.*, 2007; Kong *et al.*, 2012; Li *et al.*, 2004; Pourjavadi *et al.*, 2012; Qing *et al.*, 2007; Senff *et al.*, 2009; Stefanidou, 2012) have extensively employed XRD and SEM analyses to provide insights into their research. Li *et al.* (2004), utilizing SEM imagery, made the noteworthy observation that nanoparticles serve not only as fillers but also as activators, promoting the hydration process and enhancing microstructure when uniformly dispersed in cement paste. Similarly, Ji (2005) demonstrated through ESEM testing that the microstructure of concrete incorporating nano-silica exhibited greater uniformity and compactness in comparison to conventional concrete. Qing *et al.* (2007) scrutinized XRD powder patterns of NS and SF, revealing that both exhibited strong broad peaks at around 23° and 22° (2θ), characteristic of amorphous SiO_2 . SEM investigations conducted by Jo *et al.* (2007) validated the impact of nano-silica particles on hydration behaviour and the microstructure of the solidified paste.

The mixture containing nano- SiO_2 displayed a dense, compact formation of hydration products and a reduced presence of $\text{Ca}(\text{OH})_2$ crystals. Furthermore, it was established that NS more effectively diminished the size of CH crystals at the interface compared to SF (Qing *et al.*, 2007). Senff *et al.* (2009) demonstrated that the addition of nano-silica led to increased CH production in the early stages, surpassing that in samples without nano-silica. Aly *et al.* (2012) employed SEM micrographs to reveal that the most densely structured mortar was observed in specimens featuring a hybrid combination of Waste Glass Powder (WG) and Colloidal Nano-Silica (CS). Stefanidou (2012) reported denser structures in nano-modified samples. Kong *et al.* (2012) observed pronounced improvements in the microstructure of hardened cement paste (HCP) and the Interfacial Transition Zone (ITZ) in mortar due to the introduction of nano-silica, irrespective of agglomerate size. The gels arising from the pozzolanic reaction of the

agglomerates did not serve as binders, as the gels formed during cement hydration could not infiltrate them. Pourjavadi *et al.* (2012) disclosed that the incorporation of 1% nano-silica led to a reduction in the porosity of hardened cement paste, primarily owing to its exceptional pozzolanic performance and the heightened production of C-S-H gel. Furthermore, the microstructure experienced substantial enhancement due to micro and nano-filling effects. The size and quantity of portlandite crystals diminished as a consequence of the pozzolanic reaction and the control exerted by nano-silica on crystal growth.

4. Conclusions

The literature review has provided several significant findings:

- Incorporating nano-silica into cement-based materials enhances mechanical properties and durability.
- Nano-silica accelerates the hydration process and influences the microstructure of cement paste.
- The addition of nano-silica reduces water absorption, capillary absorption, and water permeability in concrete.
- Compressive strength in nano-silica-blended concrete is notably improved, particularly at early stages.
- Nano-silica activates the pozzolanic reaction in fly ash-based cements, enhancing their performance.
- Nano-SiO₂ particles densify the interfacial transition zone, contributing to long-term strength and durability.
- Nano-SiO₂ acts as both a filler and a promoter of the pozzolanic reaction in mortar cement microstructure. These findings collectively emphasize the potential and benefits of incorporating nano-silica in cement-based materials."

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