

Review Article

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A review on partial substitution of nanosilica in concrete

<https://doi.org/10.1515/rams-2023-0157>

received March 07, 2023; accepted November 23, 2023

Abstract: One of the most popular and widely used cementitious nanoparticle materials is nanosilica (NNS). Although several researchers discuss how NNS affects the characteristics of concrete, knowledge is dispersed, making it difficult for the reader to assess the precise advantages of NNS. Therefore, a detailed review is required for the substitution of NNS in concrete. The present reviews collect the recently updated information on NNS as concrete ingredients. First, a summary of the manufacturing, physical, and chemical characteristics of NNS is provided. Second, the characteristics of fresh concrete are examined, including its effect on setting time, flowability, air content, and fresh density. Third, strength properties such as compressive, tensile, and flexure capacity are discussed. Finally, microstructure analyses such as scanning electronic microscopy and X-ray diffraction are discussed. The results show that NNS enhanced the mechanical and durability of concrete due to the pozzolanic reaction and microfilling voids but decreased the slump flow. The optimum dose is important for maximum performance. The typical optimum dose of NNS varies from 1 to 3% by weight of cement. This article also suggests future research directions to improve the performance of NNS-based concrete.

Keywords: nanosilica, compressive strength, scanning electronic microscopy

1 Introduction

The cement industry is the main source of concrete, which is recognized as the most common man-made material. Over the last several years, the total annual global output of concrete has increased by a million tons. Cement factories are regarded as the most energy-consuming and produce significant amounts of carbon dioxide (CO₂), which is one of the drawbacks of the construction industry [1]. It contributes to around 5% of the annual worldwide man-made CO₂ releases, of which fuel combustion accounts for 40% and chemical manufacturing processes for 50% of emissions [2]. According to research, cement is manufactured and consumed in amounts totaling 76.2 billion tons, which results in the release of 38.2 billion tons of CO₂ gas into the environment [3]. A study [4] also reported that the construction industry consumes large quantities of energy and releases harmful gases into the atmosphere. It is anticipated that with time, both the demand for concrete and the output of cement will increase. Due to its impact on climate change and the loss of natural resources, new materials and composite constructions are continually being developed to meet the demands of sustainability [5] and decrease the scarcity of traditional materials [6].

Many studies have been performed to lower the impact of the cement industry's and improved sustainability [7,8], either by increasing the effectiveness of the cement manufacturing process [9] or by using supplementary cementitious materials (SCMs), which substitute cement to some extent [10]. Fly ash, blast furnace slag, natural pozzolans, bagasse ash, blast furnace slag, silica fume, marble slurry, and kiln dust are a few of the SCMs that have been studied. According to Damtoft *et al.* [11], the use of new technologies may result in commercial advances in the production of SCMs. One of the most promising areas of study that may greatly advance cement-based materials' performance, manufacture, and mixture design is nanotechnology. Senff *et al.* [12] found that silica fume's pozzolanic reaction is quick and may more effectively promote the growth of concrete's strength because of its high finesse. Current advances in nanotechnology ensure that several types of nanosized amorphous

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silica, such as nanosilica (NNS), may be manufactured. According to Lin *et al.*, these materials have larger specific surface areas and activity than traditional silica fumes [13]. Synthetic water emulsions of ultrafine amorphous colloidal silica with diameters varying from 1 to 50 nm are used to create NNS [14].

Nanoparticles are very small, and, as a result, they exhibit special physical and chemical characteristics that vary from those of traditional materials [15]. Due to their distinctive features, nanoparticles have drawn more attention and have been used in a range of sectors to create novel materials [16]. Since the late twentieth century, the concept of employing nanostructures has spread to several areas, including the cement and concrete industry [17]. Industrially, nanomaterials are produced on a large scale [18]. The usage of nanoparticles in concrete has expanded recently due to the development of nanotechnology since they may improve the characteristics of concrete [19]. NNS is an interesting nanoparticle added to concrete to increase its mechanical and chemical qualities because of its appropriate and distinctive chemical and physical properties [20].

Drexler *et al.* [21] described nanotechnology as the control of matter's structure based on molecule-by-molecule management of products and byproducts. The greatest cutting-edge development in science and technology may be categorized as nanotechnology. The past few decades have seen a substantial increase in the demand for study and improvement in the area of nanotechnology and its applications due to its enormous commercial potential and economic effect. The goal of this study is to better understand how the materials behave at the nanoscale and to figure out how to make cementitious materials function better. Due to NNS's small particles sizes and a white powder that is collected by high-purity amorphous silica powder, it has several benefits, including a large specific surface area, strong surface adsorption, large surface energy, high chemical purity, and good dispersion. In addition, cement hydration is greatly facilitated by the addition of NNS to concrete and mortar, which also increases the efficiency of cement hydration [22].

Figure 1 depicts the NNS preparation process. Sulfuric acid and ethanol were combined to form a combination (S/E), and then 5 g of sodium orthosilicate was dissolved in 60 ml of distilled water and increased dropwise to the S/E blend. It was centrifuged, cleaned with water, and dried at 60°C for 10 h after 4 h of aging. The resultant powder was then calcined for 2 h at 500°C. For the safety data sheet (SDS) procedure-based approach, sodium orthosilicate was used to dissolve the SDS in distilled water.

Numerous concrete technology applications have used NNS [23]. Hou *et al.* [24] and Kawashima *et al.* [25] discovered that adding NNS to paste or concrete may dramatically enhance the materials' mechanical characteristics, even at low doses. Researchers discovered that the NNS concentration, water-to-cement ratio, and curing time were important for increasing the compressive strength (CS) when adding NNS to regular cement paste [26]. According to Dolado *et al.* [27], the compressive capacity gradually improves as NNS concentration increases from 0.2 to 12% by mass of cement. A study reported that the optimum percentage of NNS is 6% by weight of cement [28]. According to Shih *et al.* [29], the greatest strength improvement for concrete containing 0.6% NNS was 43%. However, when the NNS concentration was increased from 0.6 to 0.8%, the strength gain decreased to 19%.

A lot of studies conclude that NNS improved the strength properties of concrete. However, a comprehensive review is required, which collects all the relevant information, such as the optimum dose of NNS and the positive and negative impact on concrete properties. Therefore, this review aims to collect the recently updated information from already carried out research on NNS as cementitious materials to evaluate the past and current progress. The review will provide a guideline for the suitability of NNS as cementitious materials in concrete. The reader can judge the benefits of NNS in concrete without conducting tests, which saves time as well as costs.

The chemical and physical properties of NNS, fresh properties of concrete (setting time, flowability, air

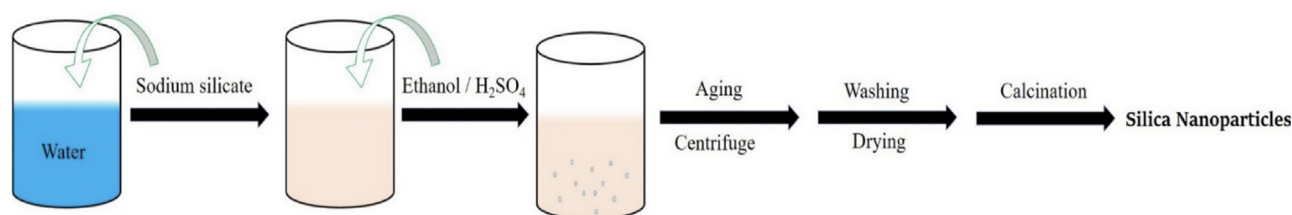


Figure 1: NNS preparation [16].

content, and fresh density), mechanical strength (compressive, tensile, and flexural strength [FS]), and microstructure analysis (scanning electronic microscopy [SEM], FTIR, and X-ray diffraction [XRD]) are the main aspects of this review. The review also indicates future research guidelines for the upcoming generation of NNS-based concrete to enhance its performance.

2 Physical and chemical properties of NNS

NNS is generally white in color, as shown in Figure 2. The physical characteristics of NNS as a cementitious material used in concrete according to past studies are shown in Table 1. NNS has a specific gravity between 1.2 and 2.20, which is lower than cement's specific gravity (3.15). Most researchers stated that NNS has a pH value higher than 7, indicating that it is often basic. Similar to this, many studies showed varying particle sizes between 15 and 35 nm. The size of the particles has an impact on the pozzolanic action of NNS. According to Ahmad *et al.*, when pozzolanic materials' particle sizes were reduced, the rate of their reactions accelerated [36]. The specific physical properties of NNS used in concrete can vary depending on the manufacturing process and the surface functionalization of the nanoparticles. However, in general, the use of NNS in concrete can lead to stronger, more durable, and more sustainable concrete structures. It should be noted that many researchers reported differences in the physical properties of NNS. It can be due to different manufacturing processes.

Dynamic light scattering (DLS) was used to assess the particle size distribution and cumulative distribution, as

Table 1: Physical properties of NNS

Ref.	[31]	[32]	[33]	[34]	[35]
Specific gravity	2.20	—	1.2	—	—
Solid density ($\text{g}\cdot\text{m}^{-3}$)	—	2.39	—	—	—
Specific surface area ($\text{m}^2\cdot\text{g}^{-1}$)	650	—	120	100 ± 25	600–800
pH	—	10.5	—	6.5–7.5	8–9
Average particle size (nm)	15	10	35	10–25	15

shown in Figure 3, which revealed that the most likely particle size is 18 nm, which accounts for 25% of the suspension volume and 51% of the volume of the suspension accounting for particle size smaller than 18 nm. Additionally, only 5% of the measured suspension's volume portion comprised particles larger than 100 nm. This resulted in a well-dispersed NSS suspension in water with little agglomeration, but it did not ensure uniform NSS dispersion after blending with particles within concrete. Overall, NNS particles are small, ranging from 1 to 100 nm, which allows them to fill in the gaps between the cement particles in concrete.

Figure 4 depicts the NNS particle nature by SEM. It is obvious that NNS particles are spherical and have particle sizes less than 150 nm. Due to the actions of ball bearings, the spherical form of NNS particles has a favorable impact on the flowability of concrete.

Figure 5 displays the XRD of NNS, and Table 2 displays the chemical makeup of NNS employed in the concrete according to previous literature. According to ASTM C618-15, the cumulative concentration of oxides of silica, alumina, and iron for cementitious materials should be more than 70% [45]. The sum of mentioned chemical oxides (iron, alumina, and silica) is higher than 70%.

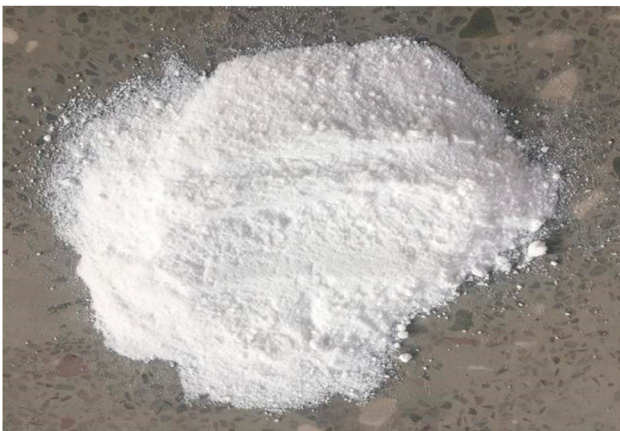


Figure 2: NNS powder [30].

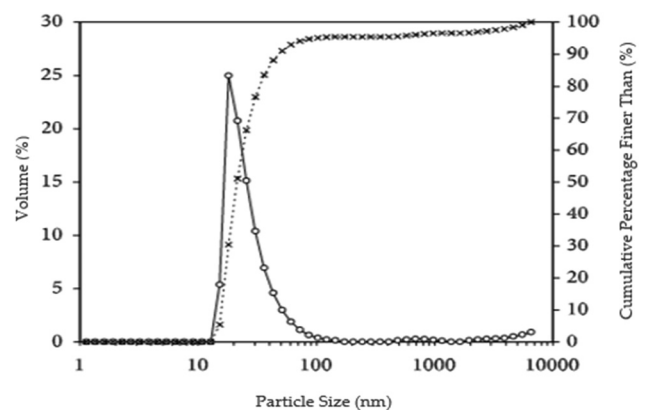


Figure 3: Gradation of NNS [37].

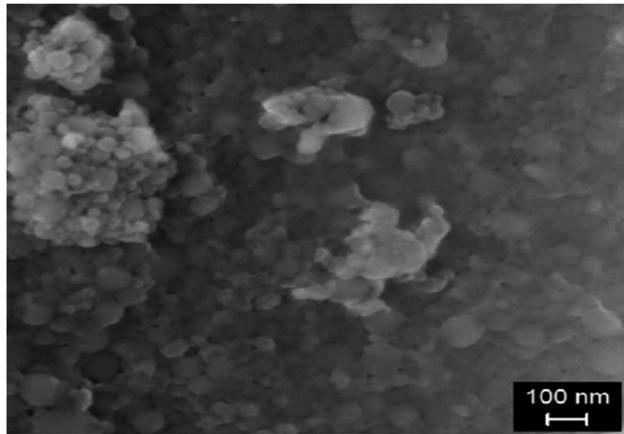


Figure 4: SEM of NNS particles [38].

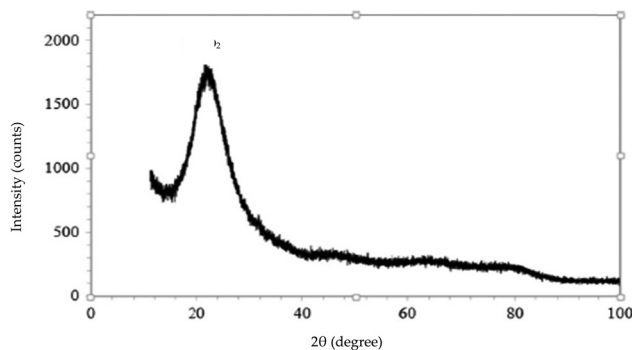


Figure 5: XRD of NNS [39].

Table 2: Chemical composition of NNS

Reference	[40]	[41]	[42]	[43]	[44]
SiO ₂	53.26	53.15	54	56.10	53.2
Al ₂ O ₃	43.93	38.44	43	40.23	43.9
Fe ₂ O ₃	0.3	2.65	1.3	0.85	0.38
MgO	0.49	0.47	0.8	0.16	0.05
CaO	0.36	0.17	0.8	0.19	0.02
Na ₂ O	—	0.08	0.7	—	0.17
K ₂ O	—	3.43	0.7	—	0.10

Therefore, NNS fulfills the criteria for use as cementitious materials in concrete production.

According to Zhang and Islam [46], NNS is a highly reactive pozzolanic material that interacts with cement hydration calcium hydroxide (CH) to produce calcium silicate hydrate (CSH). The pozzolanic reaction of NNS may have begun earlier than 24 h since the NNS had a specific surface area that was around ten times greater than that of silica fume. The shorter setting periods and increased early strength may have also been influenced by the NNS's pozzolanic reaction at an early age.

3 Fresh concrete

3.1 Setting time

According to previous research, Figure 6 displays the initial setting time (IST) and final setting time (FST). Liu *et al.* [47] used NNS 0–3.0% as a binding material. The IST and FST of the reference sample were 350 and 480 min, respectively, according to Liu *et al.* [47], as shown in Figure 6. With the substitution of 3.5% NNS, the IST and FST declined to 235 and 335 min, respectively. Similarly, Senff *et al.* [48] also stated that IST and FST declined with the substitution of NNS from 0 to 2.5% with an increase of 0.5%, as demonstrated in Figure 6. The IST and FST of concrete steadily shorten with increasing NNS dose under the same water/binder ratio, which is consistent with several earlier investigations [49]. This is a result of NNS's effective pozzolanic action. The hydration product of cement, CH(Ca(OH)₂), reacts with NNS at the beginning of the process to promote hydration, increase the exothermic rate of cement hydration, shorten the time needed for the hydration induction period, advance the acceleration period, and shorten the deceleration period. Therefore, with the inclusion of NNS, pastes' IST and FST were reduced [48]. Additionally, when the amount of NNS increased, the difference between the IST and FST was reduced [50]. In contrast, a study found that when compared to silica fume, NNS increased the mortar setting time [22].

3.2 Air content and fresh density

The fresh apparent density of mortar is reduced by the increasing NNS addition, as illustrated in Figure 7. This decrease may be ascribed to the combined effects of air entraining and the substitution of lighter NNS particles (2.21 g·cm⁻³) with denser cement particles (3.1 g·cm⁻³). When compared to mortars without NNS, as indicated in Figure 7, there is an increase in the air-entrained content of 79% with 2.5% of NNS. Therefore, it is important to take into account how entrained air affects the fresh apparent density. Additionally, Zhang and Islam found that adding NNS to concrete increases flowability without lowering the fresh density [46]. Hakamy *et al.* concluded that the agglomeration action caused porosity when additional nanoparticles were introduced [51]. The increase in porosity can lead to lower density.

3.3 Slump flow

Figure 8 illustrates the slump flow with the substitution of NNS. Generally, slump flow decreased with NNS. Due to the

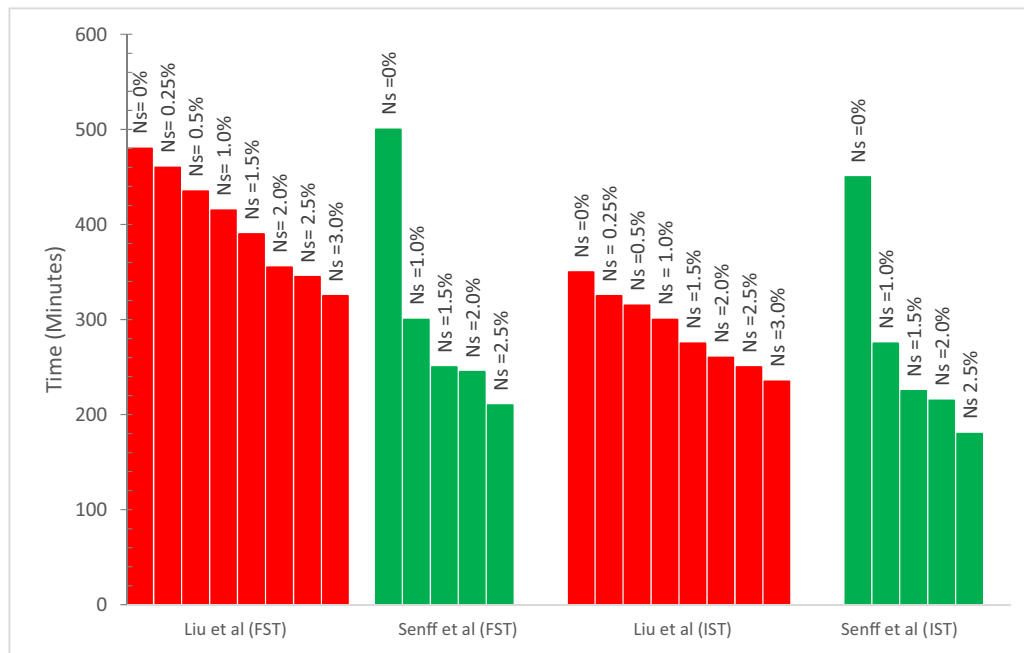


Figure 6: Setting time (Liu *et al.* [47] and Senff *et al.* [48]) (Ns indicates nano-silica percentages).

aggregation of nanoparticles, the addition of 5% of NNS seems to have decreased the workability of the hydrating solution [56]. A study also reported that pozzolanic materials (fly ash) decreased concrete flowability [57]. However, an increase in flowability was observed with silica fume [57]. The addition of 5% NNS seems to have made the hydration system less functional because of the agglomeration of nanoparticles [58]. According to research, the slump

value of concrete modified with NNS is between 80 and 100 mm, which is 40–60% less than that of unmodified concrete [23]. Due to its small particle size, NNS has a greater specific surface area, which also adversely affects the flowability of concrete. The porous nature of NNS particles also promotes the microsilica to absorb more water molecules during the mixing of the concrete, thereby reducing the slump of the concrete [59]. Yang *et al.*

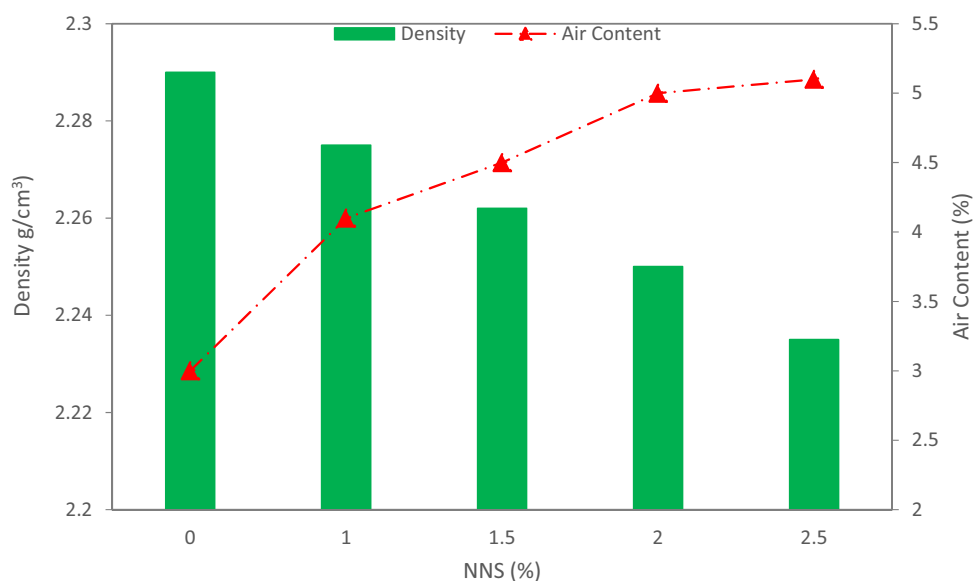


Figure 7: Density and air content (data source: Senff *et al.* [48]).

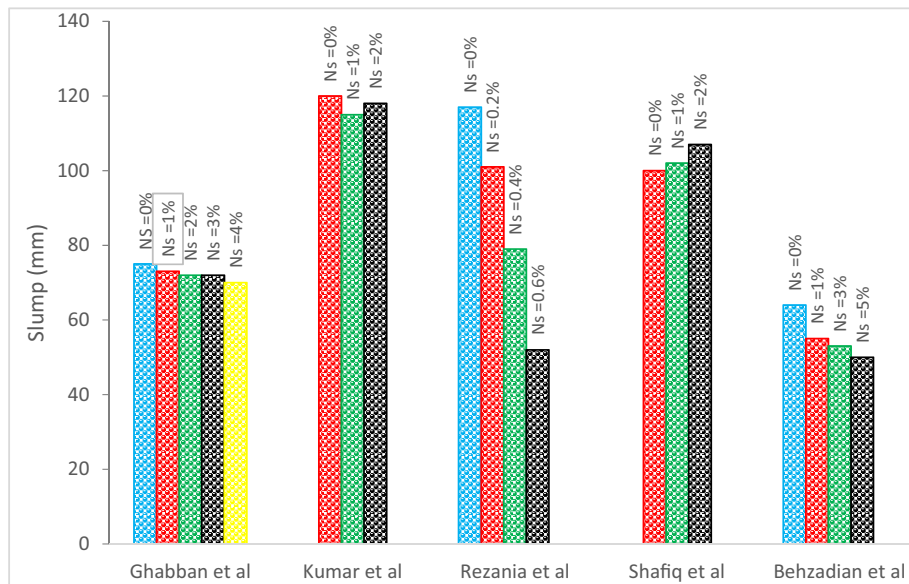


Figure 8: Slump flow [34,52–55] (Ns indicates nanosilica percentages).

recommend superplasticizers to improve the concrete slump flow [60].

The specific surface of the nanoparticles is more than the surface of the replacement cement, which increases the absorption of the free water accessible in the mixing of the required concrete. This causes the concrete slump lower than the blank blend. Additionally, it can be shown that the amount of slump reduction will be higher as a result of using two nanoparticles simultaneously. Nanoparticles increased the friction due to the differences in their sizes. Because of this, the slump reduction of hybrid concrete is greater than when just one of the nanoparticles is changed [52].

The amount of water required in the blend directly depends on how much NNS is added to the mortar while it is still in the fresh stage. This trend demonstrates that adding large surface area mineral particles to cement

mixes results in the requirement for more water or chemical admixtures to maintain mixture's flowability [61]. The significant water absorption in the aggregated NNS structure in the cement pore solution is the primary factor in decreasing flow ability. Nanoparticles are destabilized in the pore solution and develop a gel-like structure in the cement matrix. The schematic representation of NNS coagulation in concrete pore solution is shown in Figure 9. The cohesion and fluidity of the cement matrix are significantly improved by this arrangement [62].

Due to NNS's larger surface area to volume ratio, the particles have a greater water demand to cover them. Therefore, there is less blending water accessible for the other components of concrete, necessitating the use of larger plasticizer doses in the mix. However, it was also noted that the quantity of stabilizers has to be decreased

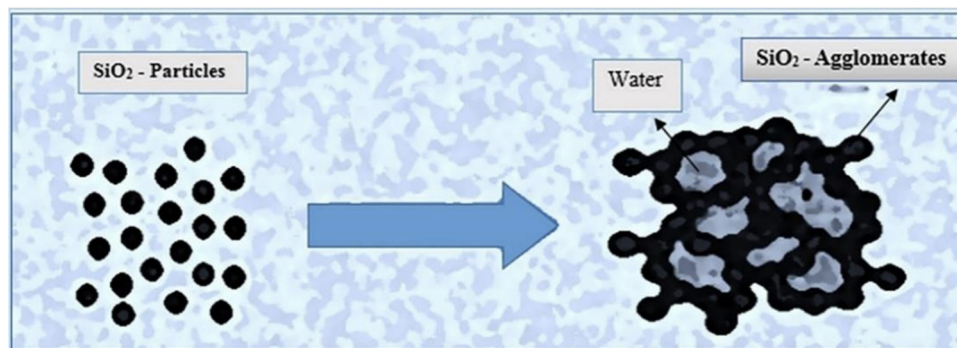


Figure 9: NNS coagulation [62].

simultaneously. The effects of NNS, which increases mixture viscosity, improve particle cohesiveness, and reduce concrete segregation, are responsible for this phenomenon. To produce a stable self-compacting combination, the superplasticizer and stabilizer doses must be optimized together. Based on these findings, it can be mentioned that NNS significantly improves mixture cohesion, which prevents segregation [38].

Note that the workability of fresh mortar was unaffected by mortar containing small dosages of NNS, regardless of diameter. There is an apparent loss of consistency with an increase in NNS concentration (between 3 and 5% by weight). The difference between mortars, including NNS with diameters of 100 and 250 nm is very attractive. Nanomaterials may significantly reduce liquidity and make it harder to achieve an ideal balance of flowability due to NNS's high surface area. The consistency of mortar is decreased by 40% in a sample, including 5% by weight of 250 nm NNS, whereas it is reduced by 18% in a sample, including 5% by weight of 100 nm NNS. This effect is most likely related to NNS's hydrophobic characteristics. Structures with larger diameters need more water because the hydrophobic properties of silica decrease as its diameter increases. As a result, there is not enough free water available to provide cement mortar with enough workability [63]. NNS particles are pozzolanic substances, in contrast to the previously employed nanostructures described above. Thus, silica nanoparticles have a dual function in cement paste: improving the

filler's workability and, via the pozzolanic reaction, improving the paste's mechanical characteristics [64].

4 Mechanical strength

4.1 Compressive strength

Figure 10 and Table 3 illustrate the compressive strength (CS) of concrete with the substitution of NNS according to previous studies. Generally, the CS improved with the replacement of NNS as cement. According to Dolado *et al.*, the CS gradually improves as NNS content increases from 0.2 to 12% by mass of cement [27]. It was discovered that strength improvements were up to 65% greater than the pastes made without NNS. Shih *et al.* [29] discovered that the greatest strength improvement for mortar containing 0.6% NNS by weight of binder was 43%, but the strength gain decreased to 19% as the NNS concentration was increased to 0.8% by mass of cement. According to Jo *et al.*, silicon dioxide nanoparticles are more effective than silica fumes in boosting mortar's CS [69]. According to Tadayon *et al.*, adding microsilica to concrete improves its CS, particularly at early ages [70].

The study demonstrated that adding the correct amounts of NNS, typically 0–6% by weight of cement, increased the concrete CS and durability [71]. According to Horszczaruk *et al.*, the pozzolanic capabilities of NNS enhance the CS

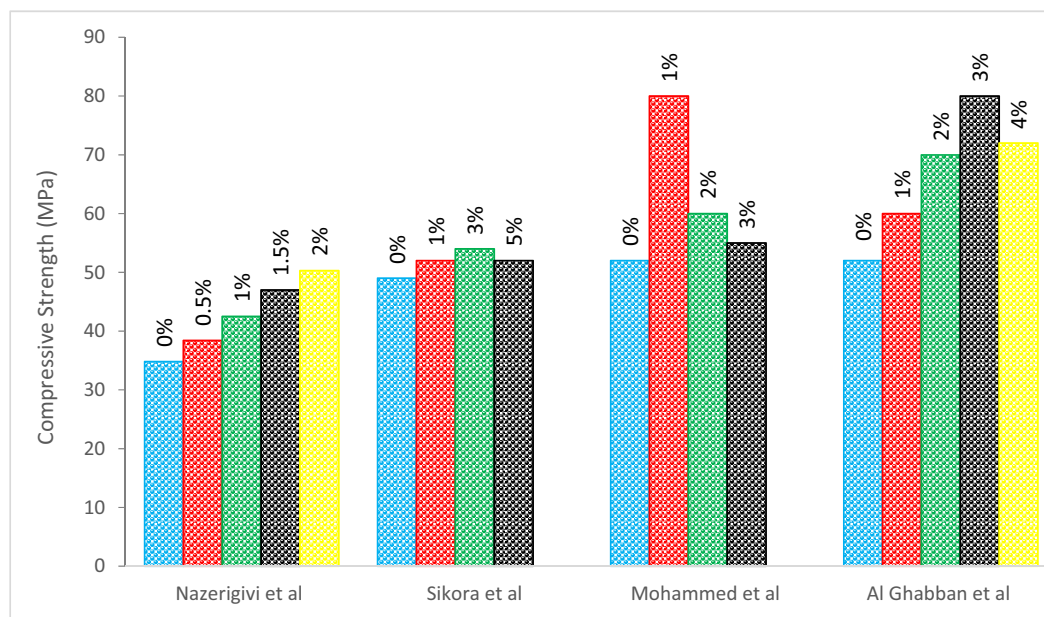
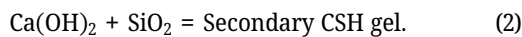


Figure 10: CS (data labels show NNS%) (Nazerigivi *et al.* [65], Sikora *et al.* [63], Mohammed *et al.* [39], and Al Ghabban *et al.* [53]).

and decrease the overall permeability of cured concrete, producing finer hydrated phases (C–S–H gel) and a densified microstructure [72]. Despite having attractive features and a wide range of possible applications, adding NNS to cementitious composites degrades their consistency due to the high-water requirements of the nanoparticles. According to Czarnecki and Łukowski [73], the addition of polymers, even in small quantities, improves the rheological characteristics of cementitious composites and lowers the detrimental effects of NNS on consistency.

It has been revealed that raising the percentage of NNS increases the CS of concrete specimens. The increased CS is primarily a result of NNS's pozzolanic activity. In the presence of water, the pozzolanic interaction between the NNS and the Ca(OH)_2 released during cement hydration results in the production of additional CSH gel [74]. Eqs. (1) and (2), respectively, depict the hydration and pozzolanic processes:



A conceptual diagram is shown in Figure 11 for the proposed reinforcing mechanism of the hybrid impacts of NNS and graphene oxide on the strength characteristics and hydration products. By forming covalent bonds between the

Si–OH of NNS and the active groups of graphene oxide, SiO_2 particles were effectively distributed over the graphene oxide layer when NNS particles were added to the graphene oxide dispersion [75] because of its high surface energy, preventing NNS particles from aggregating. The active hybrid nanomaterials offered growth points for the creation of the hydration product throughout the cement hydration process. Early on, the hydration reaction process was increased by the nucleation impact of functional groups on graphene oxide nanosheets.

The NNS particles had a pozzolanic impact throughout the process, reacting with the hydration product Ca(OH)_2 to create secondary CSH, which adds to the strength of the cured cement paste. In addition, CSH gels expand around the hybrid structure because of nanomaterial electrostatic interactions. In the end, CSH and secondary CSH combined to build a network structure based on nanosheets. CSH gel is a chemical that provides binding properties and contributes significantly to strength. Additionally, the extra CSH enhances the microstructure in the paste and transition zone, reducing the penetrability of the concrete by filling up the capillary holes and resulting in increased CS of concrete. Due to an improvement in the bond between the aggregates and the hydrated cement as well as the filling action of NNS, which results in less porosity of the concrete structure, specimens in sulfuric conditions

Table 3: Summary of concrete performance with NNS

Ref.	NNS substitution rate	Particle size (nm)	Water ($\text{kg}\cdot\text{m}^{-3}$)	Slump (mm)	Optimum dose (%)	% Increased compression strength	% Increased flexural strength	% Increased tensile strength
[55]	0, 1, 3 and 5%	20–30	216.1	Decreased	3.0	29	9.2	25
[32]	0, 1, 2, 3, 4 and 5%	10	—	—	—	3	—	—
[2]	0, 3 and 6%	35	156	—	—	18.36	—	40
[38]	0, 1, 2 and 4%	150	200	—	—	28	33	—
[66]	0, 0.5, 1.0, 1.5 and 2.0%	0.9–20	—	—	—	4	10.36	—
[33]	0, 1, 2 and 3%	35	157	—	—	25	36	—
[34]	0, 1 and 2%	10–25	175	Decreased	—	7	2	11.5
[52]	0, 0.2, 0.4 and 0.6%	15–20	493	Decreased	—	No effect	—	—
[67]	0, 1, 2, 3 and 4%	—	—	—	3	22	9	18
[54]	0, 1 and 2%	—	175	Decreased	1	5	12.5	3.7
[39]	0, 1, 2 and 3%	10–25	98.24	—	1	53	60	25
[68]	0, 1 and 2%	15	292	—	—	17.7	10.8	24.4
[53]	0, 1, 2, 3 and 4%	—	164	Decreased	3	53.8	63	68
[63]	0, 1, 3 and 5%	—	172.3	—	3	10.2	No effect	—
[16]	0, 12, 3, 4, 5, 10 and 15%	—	225	—	4	12.6	22.2	—
[65]	0, 0.5, 1.0, 1.5 and 2.0%	15–80	180	—	—	44	—	—

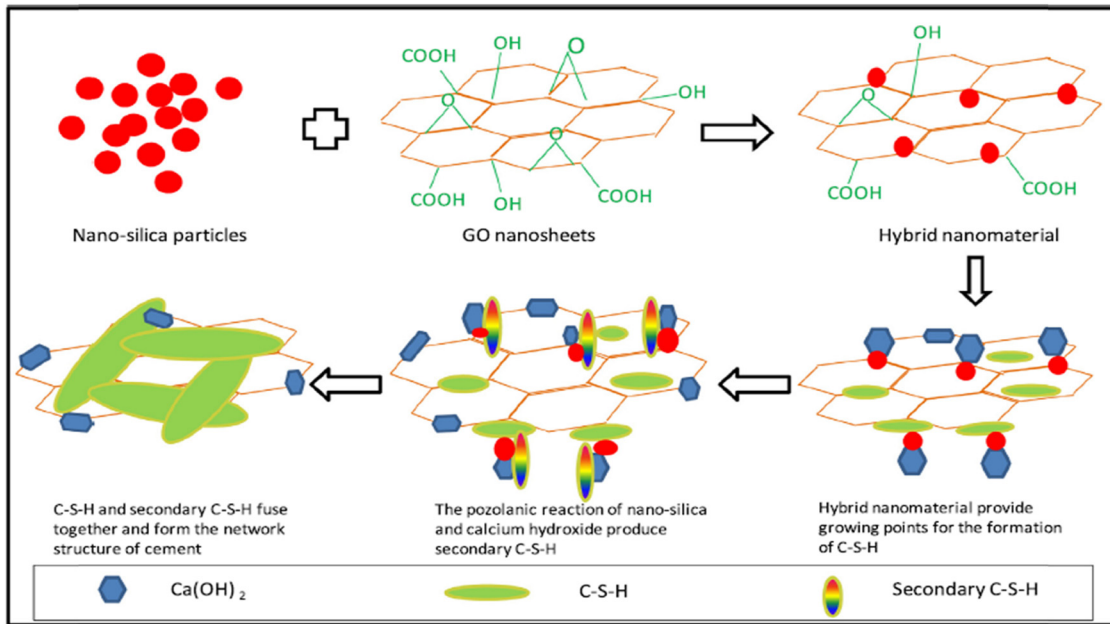


Figure 11: Nanomaterials effect on the hydration process [35] (used with permission from Elsevier).

containing NNS had CS that were higher than reference specimens across all ages. A study also claimed that copper tailing decreased porosity due to filling action [76]. Porosity and strength are inversely related to each other. The decrease in porosity leads to more compact mass that results in more strength. Additionally, the CS of concrete specimens is raised by raising the pH value of solutions, particularly at low pH values [74]. Contrary to the study results, other scholars found little to no impact of NNS on concrete CS [19]. It has been shown that adding simply NNS to the mixture has no discernible impact on the CS of the concrete. The CS measured across all ages increased by less than 5% with the addition of 1% NNS. Using 2% NNS, however, resulted in an uncertain (less than 3%) loss in CS at the ages of 28 and 90 days [34].

The compressive strength (CS) aging relation is described in Figure 12, where 28 days' CS was chosen as the control strength from which other NNS doses are compared at various curing days. When 2.0% of NNS is substituted for reference concrete, the CS of concrete after 3 days is 36% lower than the reference concrete's (control concrete's CS after 28 days), but after 7 days, the difference is only 11%. The concrete CS is 12% higher at the same dosage of NNS (2.0%) than reference concrete after 28 days of curing. NNS does not significantly increase CS up to 7 days (early age). At 28 days after curing, however, a significant improvement in CS was seen (12% improvement). The pozzolanic reaction of NNS is responsible for the significant increase in CS after 28 days since it develops more

secondary CSH gradually. Similar research revealed that the pozzolanic process moves more slowly than cement hydration [77].

4.2 Flexural strength (FS)

Figure 13 and Table 3 depict the flexural strength (FS) of concrete with substitution of NNS according to previous studies. Generally, the FS of concrete improved with the substitution of NNS in the same trends as observed in the CS of concrete. However, it should be noted that the increase in FS was higher than the increase in CS. This could be because NNS inclusion decreases pore volume without necessarily reducing the pore size. With the addition of NNS, the ratio of flexural to CS increases, perhaps suggesting increased fracture toughness [66]. The two main causes of the concrete's increased strength are the pozzolanic reaction and the nano-filling effect of NNS [78]. Particularly, because of their very high reactivity and enormous specific surface areas, NNS particles may interact with the water molecules in the concrete mixture to produce silanol groups (Si-OH). Then, a C-S-H gel is created when Si-OH combines with the Ca^{2+} in the $\text{CH}(\text{Ca}(\text{OH})_2)$ crystals [79]. Additionally, the unreacted NNS particles are scattered in smaller areas and fill the empty spaces, enhancing the concrete's compactness and pore structure. NNS particles, as was previously indicated, have large specific surface areas, and when more NNS is added than it is necessary, particle

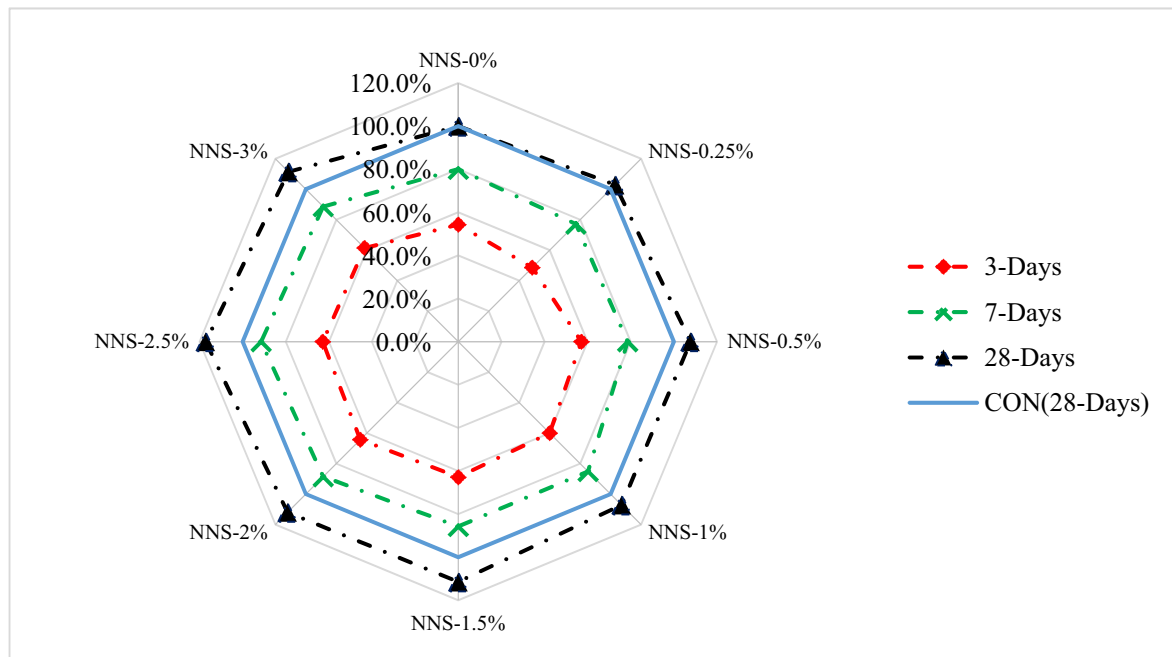


Figure 12: Relative CS (data source: Liu *et al.* [47]).

agglomeration occurs in the mixture [80]. Additionally, because of the nanoparticles' excellent water absorption properties, more water is necessary to hydrate the cement, which reduces the strength and causes inadequate cement hydration [81]. According to Tawfik *et al.* [67], the ideal proportion of NNS is 3%, beyond which the FS decreases even if the result of a 4.0% replacement is still better than the control

concrete. Furthermore, the optimum result is 6%, after which the FS declines even if the result of an 8.0% replacement is still better than the control concrete. Additionally, consumption of Ca(OH)_2 was shown to be the main factor contributing to an increase in FS at an early age in mixes containing nanoparticles [67]. The CS and FS of the NNS-based mortars were greater than the reference sample, according to Li *et al.* [82]. They also

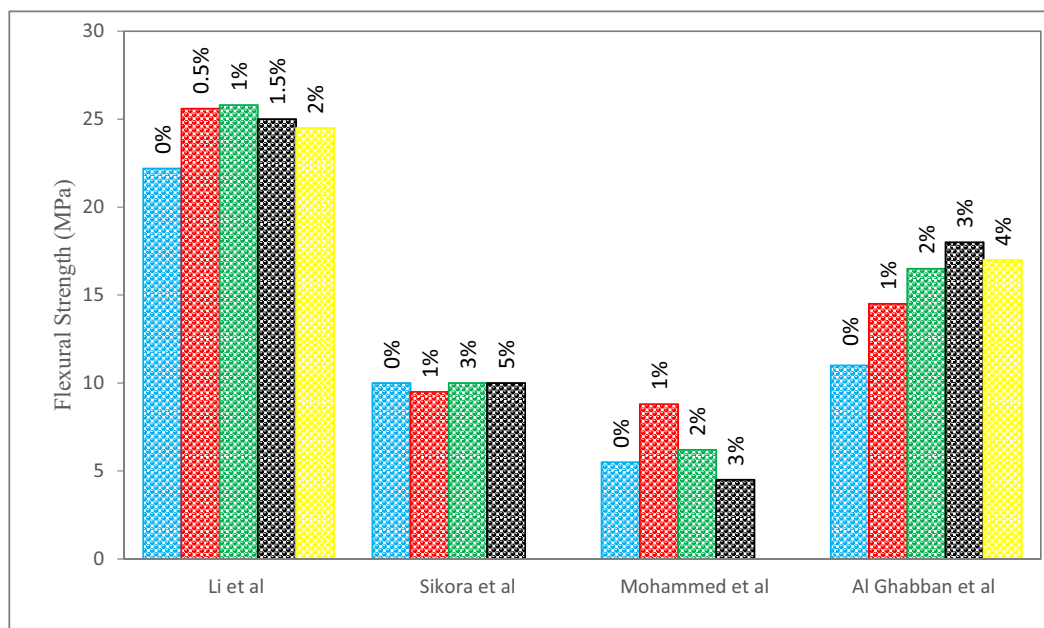


Figure 13: FS (data labels show NNS%) (Li *et al.* [66], Sikora *et al.* [63], Mohammed *et al.* [39], and Al Ghabban *et al.* [53]).

concluded that if the nanoparticles were homogeneously scattered throughout the mortar, the cement paste's microstructure could be improved [82]. Results revealed that the compressive and FS of concrete, including quartz and a mixture of quartz and barite aggregate, were raised by boosting the NNS up to 2% and lowered by increasing the NNS content between 2 and 5% [83]. It can be concluded that NNS has the potential to be used in conventional cement to enhance its hydration process and mechanical qualities. The inclusion of NNS can significantly speed up the pozzolanic process, react with the CH more often, and enhance C–S–H conversion [63].

4.3 Split tensile strength (STS)

Figure 14 and Table 3 depict the STS of concrete with substitution of NNS according to previous studies. Generally, the STS of concrete improved with the replacement of NNS in the same trends as the CS of the concrete. According to Khaloo *et al.*, 12 nm NNS had a larger improvement impact than 7 nm NNS on the STS of concrete. They found that NNS with a smaller specific surface area dispersed more readily in water [84]. The STS of NNS-modified concrete was increased by 16.10% when 3% NNS was used instead of cement. The strengthening impact of adding silica fume on the STS was more efficient when compared with adding NNS [81]. According to Singh *et al.* [85], 1.0% NNS concentration increased the STS by around 7% compared to the control mix. The strong bond between paste and

aggregates, which was made possible by the use of highly refined NNS particles, is the cause of increased concrete STS [85].

The amount of concrete-breaking STS was shown to diminish at a specific point when the quantity of nanomaterials was raised. As a result, it is believed that adding a certain quantity of nanoparticles is crucial to improving the STS of concrete since adding more nanoparticles than necessary weakens the microstructure. The appearance of weak zones because of insufficient nanoparticle dispersion might serve as evidence for decreased strength. The rapid consumption of Ca(OH)_2 that was produced during hydration, especially in the early stage related to the nanoparticles' greater reactivity, might be the cause of the greater STS in concrete that includes nanoparticles. Also, the presence of more nanoparticles in the mixture would be necessary to blend the free lime during hydration development. Because silica has been partially replaced by cementitious material but is not contributing to the strength, an excess quantity of silica leaches out and affects a weakness in the strength [67]

When compared to the STS at 28 days, it did not substantially improve after 56 days. The lack of appropriate dispersion of the NNS particles and the absence of CH production for the pozzolanic reaction of unreacted silica fume may be the cause of the lower strength improvement at later ages [68]. The increase is attributed to the cementitious matrix's particle packing because of the creation of more C–S–H. The strength of the control mix was equal to or greater after the addition of the NNS into the mix. In

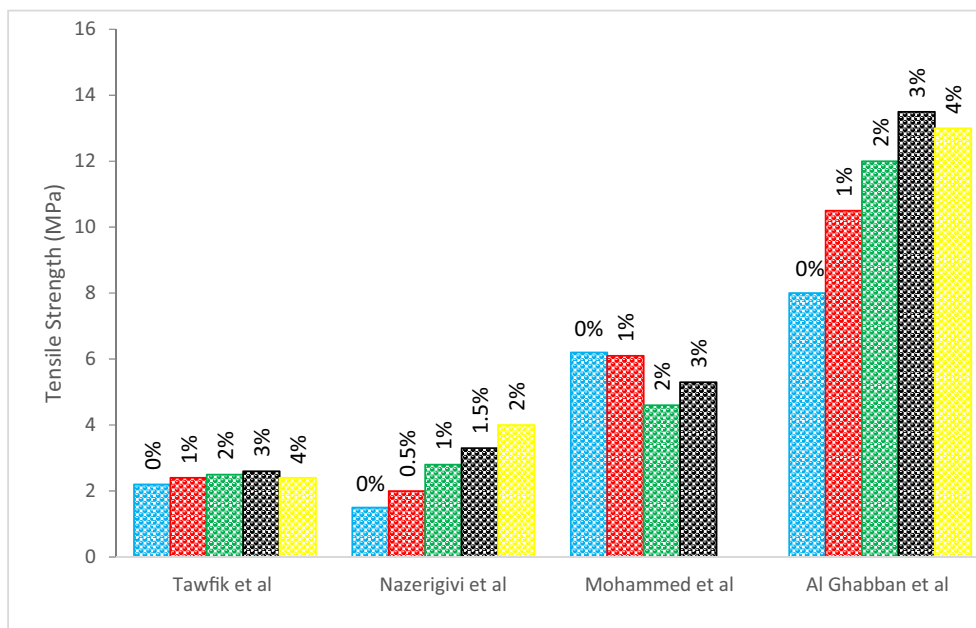


Figure 14: Tensile strength (Tawfik *et al.* [67], Nazerigivi *et al.* [65], Mohammed *et al.* [39], and Al Ghabban *et al.* [53]).

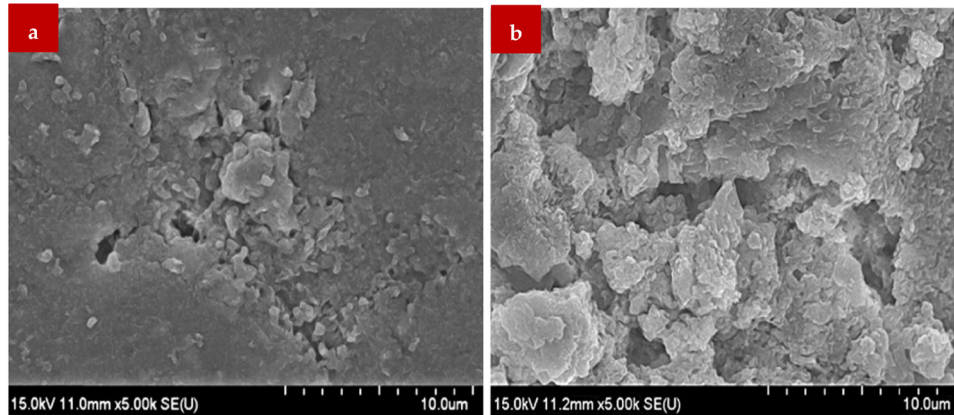


Figure 15: SEM results of the reference sample: (a) limewater and (b) NH_4NO_3 [87].

comparison to the control mix, the strength was raised by 5 and 0.5%, respectively, with the addition of 1 and 2% NNS [54]. In contrast, a greater amount of NNS in the composition causes the concrete strength to decline due to the greater surface area of the NNS. Therefore, inadequate water is available, which leads to the agglomeration of nanoparticles [86].

5 Microstructure analysis

5.1 Scanning electronic microscopy (SEM)

Scanning electronic microscopy (SEM) studies further showed that the nanoparticles were equally distributed and functioned not only as a filler but also as an activator to enhance hydration products and improve the microstructure of the cement paste [19]. Figures 15 and 16 reveal that reference

concrete and 2% NNS have denser microstructures. It is because of the pozzolanic and filler actions of NNS, as was previously indicated [88]. According to SEM images, exposure to the NH_4NO_3 solution caused the mortars to develop pores. The breakdown of portlandite and calcium-silicate-hydrate produced these holes [89]. A study also noted a porous microstructure [90]. Analysis of Figures 15b and 16b reveals that after being exposed to NH_4NO_3 solution, the control mortar became more porous than 2% NNS. This demonstrates that the mortars' resistance to NH_4NO_3 was successfully enhanced by NNS. This is in line with the analysis of toughened characteristics at different leaching phases, which revealed that the inclusion of NNS made mortars more resistant to decalcification.

The resistance of photocatalytic mortars to the decalcification attack was enhanced by the incorporation of NNS. The mortars' loss of mechanical and physical qualities caused by the leaching attack decreased as the quantity of NNS in the mortars increased. Similar to this, after being attacked by leaching, the microstructure of mortars

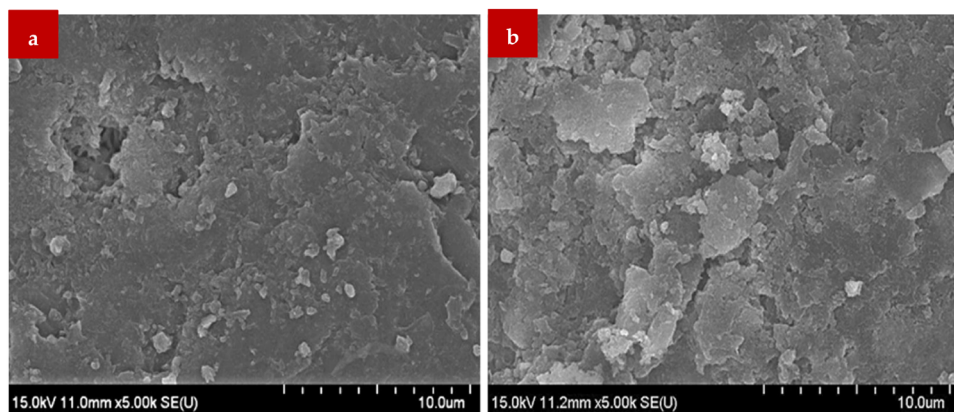


Figure 16: SEM results of 2% NNS: (a) limewater and (b) NH_4NO_3 [87].

containing NNS was more stable. The synergistic effects that were produced in the mortars because of the inclusion of NNS caused the improvement in leaching resistance. First, NNS particles are very tiny and function as the reaction's nucleus in cement hydration. Around these particles, cement hydration products are created, and pores are filled. This causes the pore structure to be improved [85]. Second, NNS may fill spaces and serve as a filler [14]. As part of the decalcification process, hydration products are dissolved and subsequently diffused into the aggressive environment outside [89]. The diffusion of Ca^{2+} ions and the penetration of aggressive ions are both delayed by the presence of NNS because it lowers pore size and permeability. Finally, NNS functions as a pozzolanic material. Portlandite is consumed, and calcium-silicate-hydrate gel is produced [85]. Portlandite

is the most susceptible hydration product during a leaching attack, according to earlier investigations [91]. Due to the pozzolanic impact of the NNS, the quantity of sensitive portlandite decreased with the addition of NNS, and leaching was further decreased. Ghafari *et al.* also discovered that the use of NNS improved the concrete's structural efficiency. The amount of capillary holes in the concrete matrix steadily reduced as the NNS concentration increased [92].

Similarly, the microstructure of concrete is noticeably improved by NNS, as seen in Figure 17. The hydration products are not tightly coupled with the aggregate, the ITZ is weak, and there are clear fractures, as illustrated in Figure 17(a), which also demonstrates the severe faults in the microstructure of the 0% NNS sample. Figure 17(b) shows that adding 0.5% NNS may increase cement hydration to

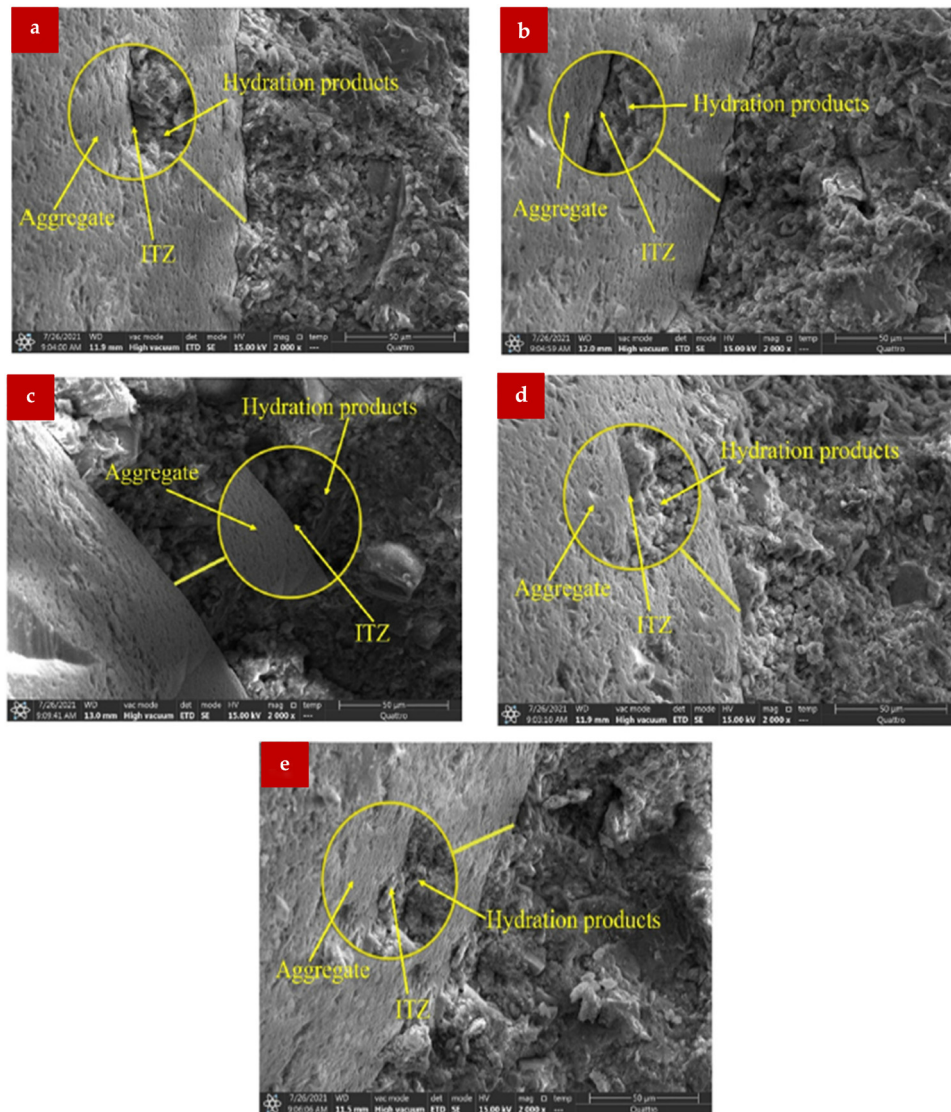


Figure 17: SEM results of (a) 0%, (b) 0.5%, (c) 2%, (d) 2.5%, and (e) 3% NNS [47].

some degree and produce denser and more uniform hydration products after the concrete has had its cement content reduced by 20%. In the interface transition zone (ITZ), there are, nevertheless, still gaps and unfinished packages. Because of the physical and chemical impacts of NNS, the NNS may minimize porosity in cement paste and ITZ between the cement paste and aggregate. It has also been claimed that NNS may lower CH crystal size in the ITZ more efficiently than silica fume [93].

Figure 17(c)–(e) shows that the microstructure of concrete greatly improves with a progressive increase in the NNS dose. The hydration products are bundled up with the aggregate at the thick interface transition zone of the 2%

NNS specimen. The slurry and aggregate interface bonding are greatly improved. The ITZ of the 2% of NNS specimens was closer to that of the 1.5% of NNS specimens. The aggregate was entirely encased in the hydration products, which underwent a morphological transition from scattered gel to regular, ordered crystals. To create a consistent and dense structure, the crystals were entangled. The hydration products are encircled by the aggregate in the thick interface transition zone of the specimen made of 2% NNS. Significantly improved interface bonding occurs between the slurry and the aggregate. The ITZ of the 2% of the NNS specimen was more consistent with the 0.5% specimen. The hydration products fully encased the aggregate, and its

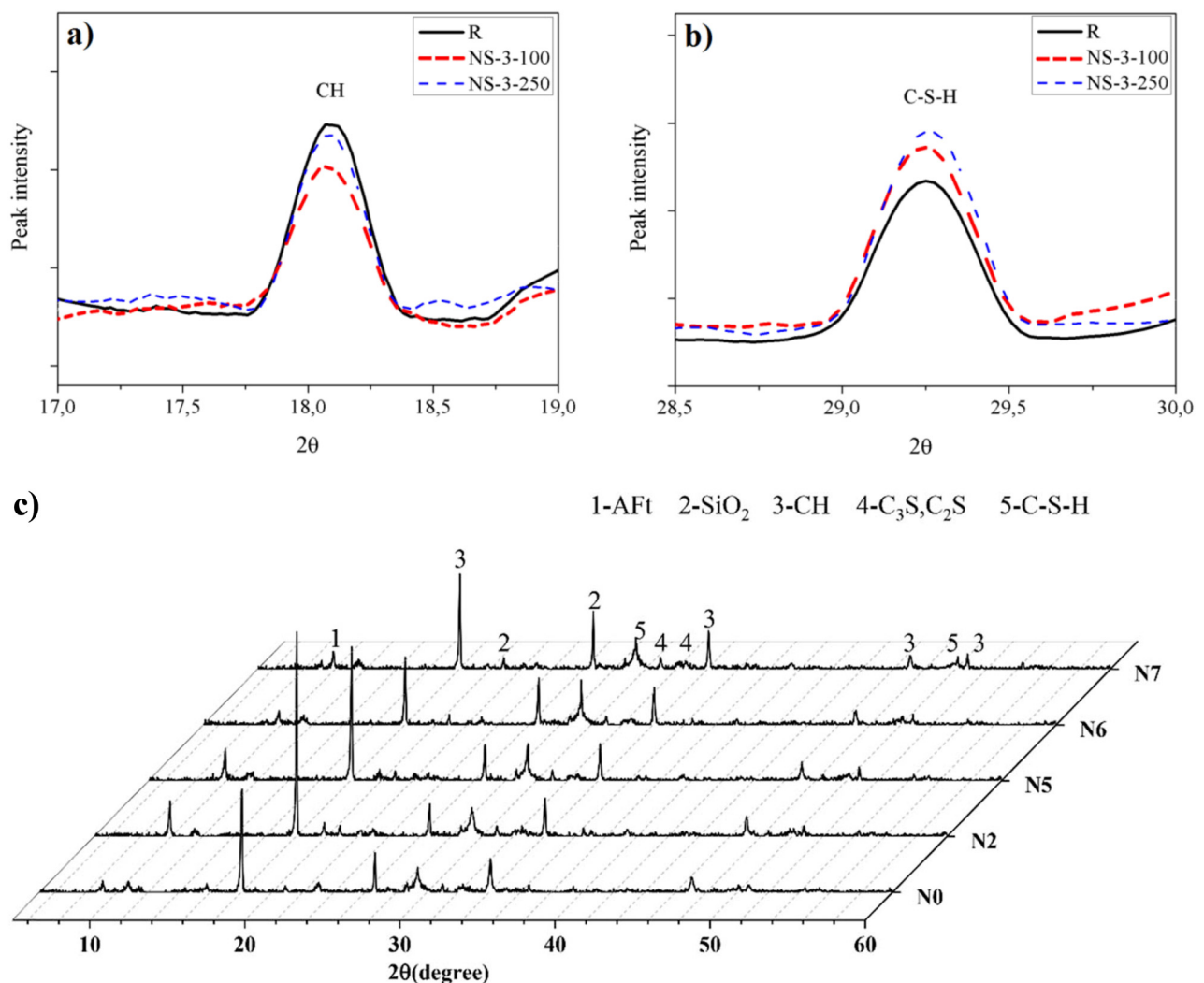


Figure 18: XRD results: (a) CH, (b) CSH, and (c) different peaks of hydration [47,63].

scattered gel shape was altered to that of regular, ordered crystals. A homogeneous and thick structure was created by the crystals' interlocking [94]. The 3% NNS specimen demonstrated that the interfacial concrete's hydration products and excessive zone's ability to be regulated by NNS started to deteriorate. This occurred as a result of the NNS dose being excessive and susceptible to aggregation in concrete materials. Additionally, the hydration of CSH produced by the action of ash with the presence of NNS aggregates did not have cementitious qualities, creating a weak ITZ, affecting the binder properties and ultimately lower in the concrete strength.

5.2 XRD

The XRD test is used to detect the crystalline precipitates of the amorphous structure during geopolymerization [95]. For XRD examination, samples with a 3% NNS of 100 and 250 nm particle size showed the greatest increase in CS, according to Sikora *et al.* [63]. For comparison, the peaks of CH and C–S–H have been chosen at 18° and 29°, respectively, as proposed by Aly *et al.* [96] and Kim *et al.* [97]. As seen in Figure 18(a), the hydration of cement results in the formation of a strong CH peak. When NNS is added, the strength of the CH peak decreases, which illustrates how the pozzolanic reaction consumes the CH. In parallel, specimens containing NNS showed an increase in the C–S–H peak intensity, as shown in Figure 18(b). The data shown below indicate that NNS (regardless of diameter) demonstrates some pozzolanic activity and has an accelerated hydration rate.

Similar to Figure 18(c), numerous distinctive peaks may be seen in the XRD pattern that represents various crystal structures in concrete materials. Silica (SiO_2), tricalcium silicate (C_3S), dicalcium silicate (C_2S), CSH (C–S–H), ettringite (AFt), and CH are the primary constituents. The distinctive peaks of C_3S and C_2S in each set of samples were found to be at a low level by comparing XRD patterns. The distinctive peak of CH declined first and then raised with an increase in NNS dose, while the characteristic peak of C–S–H increased first and then reduced. The peak value of CH is the lowest and the peak value of C–S–H is the highest when the dose of NNS is 2.5%. This is because NNS exhibits strong pozzolanic action and nucleation effects, which aid in consuming free CH, lowering the amount of hydration products that cannot provide strength, and producing C–S–H to increase the density of the internal space of concrete and improve its mechanical properties.

6 Conclusions

The fundamental science of cementitious materials at the nano/atomic level is now the major focus of study. Researchers are also working to increase concrete durability and have found that adding NNS substantially increases the strength characteristics of cementitious materials. The review describes the effect of NNS addition on the fresh properties, strength, and microstructural properties. The analysis may lead to the following conclusions.

- The physical properties of NNS show that it can be used as a concrete ingredient. The chemical structure of NNS depicts that it has the creditability to be used as a binder.
- Setting time declined with the replacement of NNS as the pozzolanic action continued gradually as associated with cement hydration. NNS decreased the fresh density due to the substitution of denser cement particles with lighter NNS particles.
- The concrete slump flow declined with the substitution of NNS due to its fineness, which required extra paste to cover them.
- The concrete strength properties improved with NNS substations due to combined pozzolanic reaction and micro-filling voids. However, the optimum amount is critical as the larger amount negatively affects the concrete strength due to a lack of flowability.
- Different research studies recommend a distinct ideal dose of NNS due to a difference in the source or different manufacturing process. However, the average range of the ideal dosage of NNS is 1 to 3% by weight of cement.
- SEM results show that the interfacial transition zone (IZT) was considerably decreased with the substitution of NNS due to microfilling cavities and pozzolanic action. XRD analysis demonstrates that CH declined with the addition of NNS, which indicates the pozzolanic activity of NNS.

7 Recommendations

A comprehensive review of NNS substitution in concrete depicts that NNS can be utilized in concrete due to pozzolanic action and micro-packing voids, which improve concrete strength and durability properties. However, future research needs to address the following issues to get more benefits of nanotechnology.

- Although NNS enhanced the concrete's strength properties, concrete still has less tensile capacity which causes abrupt (brittle) failure with no deformation. Brittle

failure is unacceptable for a structural member from a safety point of view. Therefore, this study recommends a detailed investigation of NNS-based concrete to improve tensile capacity.

- A detailed investigation of the environmental and cost-benefit analysis of NNS-based concrete is required.

Acknowledgments: The authors are thankful to the Swedish College and Nanjing University of Science and Technology for their support in the development of this review article.

Funding information: The authors state no funding involved.

Author contributions: All authors have accepted responsibility for the entire content of this manuscript and approved its submission.

Conflict of interest: The authors state no conflict of interest.

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