

Combined effect of nano-silica and silica fume to improve concrete workability and compressive strength: a case study

CIVIL ENGINEERING

Efecto combinado de nano-sílice y humo de sílice para mejorar la trabajabilidad del concreto y la resistencia a la compresión: un estudio de caso

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Abstract

Several types of amorphous silica generate pozzolanic reactions at micro- and nano-scale improving concrete macro-structural characteristic. However, their addition can raise the viscosity of the mixture, thus decreasing its workability. In the present work is proposed an experimental setup to determine concrete compression and manageability performance adding Silica Fume (SF), 0.0, 6.5, 8.5, 10.0% w/b, colloidal silica (nSi), 0.0, 0.5, 1.0, 1.5, 2.0, 2.5% w/b and SF-nSi combined, 0.0-0.0, 0.2-8.5, 0.2-10% w/b (by weight), and an extensive overview of the literature for concrete mixtures with SF, nSi and SF-nSi combined. The results show that the addition of SF can increase compressive strength by 12%, nevertheless, can also lead to decreases in manageability of the sample (e.g., a decrease by 67% for a dosage of 10.0% w/b). On the other hand, nSi can increase compressive strength by up to 25% with a dosage of 2.5% and can increase the manageability by 78% for dosages greater than 0.5% w/b. When



combining the two materials, an increase in compressive strength was observed, along with a loss of workability. A statistical analysis of the data found in literature is proposed using MATLAB, highlighting interesting trends of behavior between SF and nSi with workability and compressive strength, as well as a lack of research for slump outcomes and for the effects of combined additives in concrete. Finally, a short cost-benefit analysis of the mixtures is proposed.

Keywords: Mechanical improvement of concrete; Cost-benefit analysis; nSi and macro-silica; workability and compressive strength.

Resumen

Varios tipos de sílice amorfa generan reacciones puzolánicas a escala micro y nano, que pueden mejorar las características macroestructurales del concreto. Sin embargo, su adición puede aumentar la viscosidad de la mezcla, disminuyendo así su trabajabilidad. En el presente trabajo se propone un montaje experimental para determinar el comportamiento de compresión y manejabilidad del concreto agregando Humo de Sílice (SF), 0.0, 6.5, 8.5, 10.0% p/b, sílice coloidal (nSi), 0.0, 0.5, 1.0, 1.5, 2.0, 2.5 % p/b y SF-nSi combinados, 0.0-0.0, 0.2-8.5, 0.2-10 % p/b (por peso), y una amplia descripción de la literatura para mezclas de concreto con SF, nSi y SF-nSi combinados. Los resultados muestran que la adición de SF puede aumentar la resistencia a la compresión en un 12%, sin embargo, también puede conducir a una disminución en la manejabilidad de la muestra (por ejemplo, una disminución del 67% para una dosis de 10,0% p/b). Por otro lado, el nSi puede aumentar la resistencia a la compresión hasta en un 25% con una dosificación del 2,5% y puede aumentar la manejabilidad en un 78% para dosificaciones superiores al 0,5% p/p. Al combinar los dos materiales, se observó un aumento en la resistencia a la compresión, junto con una pérdida de trabajabilidad. Se propone un análisis estadístico de los datos encontrados en la literatura usando MATLAB, destacando tendencias interesantes de comportamiento entre el SF y el nSi con la trabajabilidad y la resistencia a la compresión, así como la falta de investigación sobre los resultados del asentamiento y los efectos de los aditivos combinados en el concreto. Finalmente, se propone un breve análisis costo-beneficio de las mezclas.

Palabras clave: *Mejoramiento mecánico del concreto; Análisis coste-beneficio; nSi y macro-silica; trabajabilidad y resistencia a la compresión.*

1. Introduction

Silica fume (SF) is a type of amorphous silica with a higher specific surface than cement (SF,

19,800 m²/kg; cement, 314 m²/kg) (1). This led to its wide use in concrete for mechanical behavior improvement (2–5), despite its high cost (6). SF is composed of SiO₂ and is

characterized by high-purity spherical particles with a very fine size and high pozzolanic activity, which contribute to the improvement of cement-based compounds (7,8). SF also improves resistance and can be used advantageously when good quality aggregates are not available (9).

In recent years, interest in SiO_2 , ZnO_2 , Al_2O_3 , CuO , CaCO_3 , Fe_2O_3 , and TiO_2 nanotechnological products, processes, and applications has raised for their advantages in many aspects of material science, among which improvement of the antimycotic behavior of the materials and air-purifying purposes (10,11). Furthermore, they provide greater durability of concrete by modifying the micro-structural, mechanical, and durability properties of concrete and mortar, (12,13). Nano- SiO_2 (nSi) has been one of the most widely used materials, compared to other nanomaterials (14–16), as it is a very effective concrete additive, improving its strength, flexibility, and durability (17). Furthermore, it can be used as an additive to improve the workability and strength of high-performance and self-compacting concrete (18). Research has shown that the use of 1 kg of SF, in particular, can save 4 kg of cement (19). Additionally, 1% of nSi is equivalent to 10% of SF (20).

SF, or micro-silica, is a byproduct of the industrial production of silicon (21), having particle sizes within the submicrometric range. In concrete, amorphous silica presents a pozzolanic reaction, wherein SF reacts with

portlandite ($\text{Ca}(\text{OH})_2$, CH) as a result of alite hydration (22). Subsequently, amorphous silica fills the gaps between the cement particles and other constituent materials (which is called the filler effect), thus densifying the concrete (23). Another principle of the filler effect is that the nucleation of calcium silicate hydrate (C–S–H) phases (formed from alite hydration) occurs at the surface of the fillers. This effect accelerates cement hydration and is known as the sowing effect (24,25). Due to its extreme fineness and high content of amorphous silicon dioxide, silica fume is a highly reactive pozzolanic material. The mechanism of silica fume in concrete is basically due to three functions: Pore refinement and matrix densification, reaction with free limestone (from cement hydration), and interfacial refinement of paste-aggregate, (26).

Colloidal silica is a liquid nano-silica (nSi) of uniform shape and particle size between 1–1000 nm (19). It is produced from a sodium silicate solution. The solution is subject to an ion exchange to partially remove sodium ions, thus forming a silica suspension. Subsequently, the pH is adjusted to control the particle size. The appearance of nSi is a suspension of fine silica particles in an aqueous medium. Its main applications are as a binder in investment casting, paper processing and coating, and as a binder in refractories and construction materials (27). Regarding hydration, nSi is superior to SF, as it accelerates the process and leads to a compaction of the concrete microstructure at early stages of curing, as well as increasing the

hydration heat produced, due to the fast formation of CH crystals during the settlement of the cement paste. nSi particles react with CH and result in an additional amount of NSIH gel in the matrix. Therefore, there is an enhancement in the mechanical properties of the mixtures prepared with nSi, as related to the dosage (28). However, the dispersion of nanoparticles in the concrete is vital for the enhancement of the macro-, micro-, and nano-cementing properties. Additionally, there is a lack of data on the reaction mechanism of nSi (19). Combination of the two additives shows a great potential to improve concrete workability and compressive strength, because they are a complement, nSi works at nanoscale, while Sf works at macroscale. Furthermore, as mentioned by (29), nSi and SF supplied the voids in the micron size of cement particle and formed a denser concrete which leads to improving the concrete mechanical properties compared to conventional concretes.

In this study, an experimental procedure was carried out to evaluate the effect of the individual and combined additions of SF and nSi (in the form of a colloidal suspension) on the

concrete workability and compressive strength. The most and relevant studies in the last decade are discussed and compared to see the international trends in the matter. Finally, an overview of the economic aspects of those mixture is proposed emphasizing the context of a developing country like Colombia.

2. Methodology

In this research, for all tests, a high early strength cement (HESC) was used, according to American Society for Testing and Materials (ASTM) (2020a). HESC means that the required compressive strength of the concrete can be reached significantly before the 28 standard days. This was used in all mixtures of the present work, in order to streamline laboratory times. This will not affect the final results of the work, as has been shown in the literature (31,32). Additionally, it was used a superplasticizer type F additive based on polycarboxylates, a fractured boulder-type coarse aggregate from old river channels of the Colombian Caribbean area with nominal maximum size of 9.53 mm, and natural siliceous sand. The characteristics of the aggregates are given in Table 1 y Table 2.

Table 1. Features of the aggregates used in the preparation of the mixture.

Parameter	Sand	Coarse aggregate
Wear (M. Angels) maximum (%)	-	12.48
Clay lumps maximum (%)	-	0.71
Fractured faces minimum (%)	-	75.70
Absorption (%)	2.35	1.00
Dry density (Kg/m ³)	2512	2462.66
Saturated density (Kg/m ³)	-	2487.29
Apparent density (Kg/m ³)	-	2525.00
Humidity (%)	3.08	0.20
Fineness module (%)	3.28	-

The silica fume utilized (SiO₂) had a percentage >95% by weight, specific surface area of 30,000

m²/kg, humidity of 3–5%, and colloidal silica density of 1.14 kg/l.

Table 2. Grading of aggregates with ASTM-C33 limits

Sieve No. (mm)	% Passing
25.4	100.00
19.0	100.00
12.5	100.00
9.5	99.18
4.8	26.95
2.4	0.95

2.1. Mixture proportions

For the compression tests, binary mixtures of three concrete specimens were prepared: 1) for three SF dosages: 6.5, 8.5, and 10.0% w/b; 2) binary mixtures (from three test tubes) for each NSI dosage: 0.5, 1.0, 1.5, 2.0, and 2.5% w/b; 3);

and, finally, ternary mixtures were prepared with combinations of the two materials and cement, with a single dose of colloidal silica (0.2% w/b) and two dosages of silica fume (8.5 and 10.0% w/b). Additionally, three standard cylinders were prepared, which did not contain SF or NSI. The mix design is detailed in Table 3.

Table 3. The mix design of the present study

The above-mentioned percentages and experimental work taking as starting point the combinations were set after an extensive ranges found in literature (Table 4).

Table 3. The mix design of the present study

Sample	Cement (kg/m³)	Water (kg/m³)	Sand (kg/m³)	Coarse aggregate (kg/m³)	SP (% w/b)	Silica fume (SF) (kg/m³)	Colloidal Silica (% w/b)
SF0-NSI0	448.25	210.0	568.24	1021.76	0.6	0	0
SF6.5-NSI0	448.25	224.7	568.24	1021.76	0.6	31.38	0
SF8.5-NSI0	448.25	228.9	568.24	1021.76	0.6	40.34	0
SF10-NSI0	448.25	233.1	568.24	1021.76	0.6	49.31	0
SF0-NSI0.5	448.25	210.0	568.24	1021.76	0.6	0	0.5
SF0-NSI1	448.25	210.0	568.24	1021.76	0.6	0	1
SF0-NSI1.5	448.25	210.0	568.24	1021.76	0.6	0	1.5
SF0-NSI2	448.25	210.0	568.24	1021.76	0.6	0	2
SF0-NSI2.5	448.25	210.0	568.24	1021.76	0.6	0	2.5
SF 8.5 NSI0.2	448.25	228.9	568.24	1021.76	0.6	40.34	0.2
SF 10-NSI0.2	448.25	233.1	568.24	1021.76	0.6	49.31	0.2

Colloidal silica is a liquid nano-silica (nSi)

2.2. Preparation and testing of mixtures

The mixtures were made in a 35-rpm piece of rotating equipment. Aggregates, cement, and

silica fume were mixed for 2 minutes, followed by the addition of water containing pre-mixed additives (mixing time of 10 min). As for the SC

procedures, a mixture with water was previously made.

For each mixture, three 10 x 20 cm cylinders were prepared, in order to carry out the compressive strength tests. The mixtures were compacted inside molds in three layers, applying 25 strokes. All specimens were settled for 24 h at room temperature and were subjected to curing under water up to the 7th day.

After the mix was completed, the workability level was assessed using the Abrahams cone test, according to the ASTM C143/C143M - 20 Standard Test Method for Slump of Hydraulic-Cement Concrete, (2020). The procedure to determine the f_c was performed in accordance with the ASTM C39 / C39M - 20 Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens, (2020), using a hydraulic press of *Rosemberg-Laboratory Equipment*, Model 3100-PE-D of 2019 with 0.1 kN sensitivity.

Furthermore, an overview of the literature is proposed, to oversee the research trend at an international level regarding the addition of SF and nSi to improve concrete workability (Slump) and compressive strength (f_c) and compare with the case study.

Finally, a short discussion regarding the cost–benefit analysis is presented below.

3. Results and Discussion

3.1. Workability

Figure 1 shows a loss of workability resulted when incorporating SF, considering the

difference in the slump values of the mixes. When SF was used, there was a 67% reduction in manageability, from 9 to 3 cm. The surface area of SF increases the attraction forces between its siliceous particles, which results in the clustering of particles and, thus, decreases the mixture's slump and workability. An increase in slump occurred only with the addition of nSi (Figure 1 b) to the mixtures, with a 78% increase from 9 to 16 cm, while the combination of nSi and SF led to a total reduction in workability, carrying the slump back to zero (Figure 1c). The loss of workability in the latter sample may have been due to the small particle size capturing a significant amount of water from the original mixture, as well as the excess of pozzolanic materials preventing the complete chemical reaction from occurring.

Table 4 and Figure 2 show the slump results of some of the most relevant studies around the world in recent years. The relationship between the slump of the mixture with SF, nSi and SF+nSi, respectively, with the slump of the base mixture without additives (Slump base). The ratio “Slump/Slump base” allows to normalize the data found in the literature and compare different studies. In the literature can be observed that slump for SF addition in concrete is less studies than nSi, while the SF+nSi addition is the least studied. Due to multiple variables in play in each mixture, such as different types of aggregate, water/binder ratio, superplasticizer, among others, it is difficult to find a proper mathematical model to describe

slump variation in function of SF, nSi and SF+nSi concentrations valid worldwide. Nevertheless, it is observed a trend in the data found, suggesting that for higher SF percentages

the mixtures shown a loss of slump ratio (Figure 2), with values lower than 1, according to the present study experimental part.

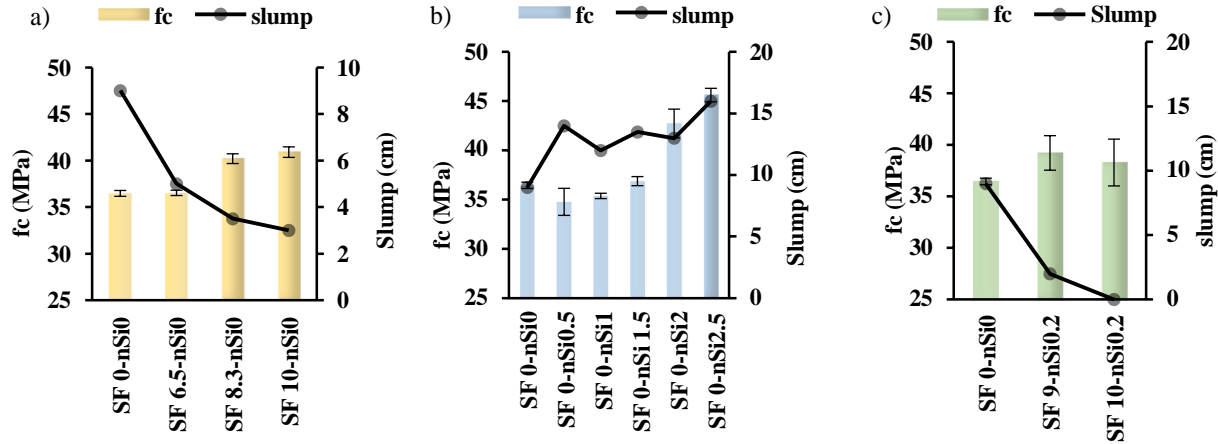


Figure 1. Behavior of settlement flow and compressive strength of mixtures when using: a) SF; b) nSi; and c) SF+nSi. Standard deviation for f_c results is reported. Slump data is based on one measurement.

Table 4. Literature studies using silica fume (SF) addition in concrete.

w/b	SF (%)	f_c (MPa)	f_c (Base) (MPa)	f_c/f_c base (MPa)	Slump (mm)	Slump (Base) (mm)	Slump / Slump (Base)	Reference
0.50	7.00	36.50	36.47	1.00	35.00	90.00	0.39	PS
0.51	8.50	40.10	36.47	1.10	35.00	90.00	0.39	PS
0.52	10.00	40.90	36.47	1.12	30.00	90.00	0.33	PS
0.28	5.00	79.27	76.52	1.04	740.00	790.00	0.94	(35)
0.28	10.00	88.25	76.52	1.15	690.00	790.00	0.87	(35)
-	3.00	-	-	-	129.00	137.00	0.94	(36)
-	5.00	-	-	-	110.00	137.00	0.80	(36)
-	8.00	-	-	-	105.00	137.00	0.77	(36)
-	10.00	-	-	-	100.00	137.00	0.73	(36)
0.40	8.00	71.70	59.25	1.21	30.00	40.00	0.75	(37)
0.35	8.00	77.90	64.95	1.20	30.00	35.00	0.85	(37)
0.30	5.00	113.20	105.70	1.07	140.00	140.00	1.00	(38)
0.30	5.00	106.20	97.30	1.09	128.00	157.00	0.82	(38)
0.30	5.00	80.50	68.10	1.18	181.00	181.00	1.00	(38)
0.30	5.00	62.80	44.70	1.40	115.00	125.00	0.92	(38)
0.36	3.00	33.00	37.20	0.88	135.00	135.00	1.00	(39)
0.36	5.00	39.50	37.20	1.06	140.00	135.00	1.04	(39)
0.36	3.00	35.00	37.20	0.94	140.00	135.00	1.04	(39)
0.36	5.00	42.00	37.20	1.12	135.00	135.00	1.00	(39)
0.79	5.00	30.00	27.50	1.10	-	-	-	(40)
0.79	10.00	32.00	27.50	1.16	-	-	-	(40)
0.79	15.00	33.00	27.50	1.20	-	-	-	(40)
0.79	20.00	33.50	27.50	1.22	-	-	-	(40)
0.65	5.00	41.50	37.00	1.12	-	-	-	(40)
0.65	10.00	45.00	37.00	1.22	-	-	-	(40)
0.65	15.00	47.00	37.00	1.27	-	-	-	(40)
0.65	20.00	48.00	37.00	1.30	-	-	-	(40)
0.60	5.00	46.50	41.00	1.13	-	-	-	(40)
0.60	10.00	50.50	41.00	1.23	-	-	-	(40)
0.60	15.00	52.50	41.00	1.28	-	-	-	(40)
0.60	20.00	54.00	41.00	1.33	-	-	-	(40)
0.52	5.00	57.00	50.00	1.14	-	-	-	(40)

0.52	10.00	62.00	50.00	1.24	-	-	-	(40)
0.52	15.00	64.00	50.00	1.28	-	-	-	(40)
0.52	20.00	66.00	50.00	1.32	-	-	-	(40)
0.45	5.00	68.00	59.00	1.15	-	-	-	(40)
0.45	10.00	74.50	59.00	1.26	-	-	-	(40)
0.45	15.00	77.00	59.00	1.31	-	-	-	(40)
0.45	20.00	79.00	59.00	1.34	-	-	-	(40)
0.36	2.50	63.79	60.33	1.06	787.00	645.00	1.21	(41)
0.36	5.00	65.01	60.33	1.08	820.00	645.00	1.26	(41)
0.36	7.50	68.86	60.33	1.14	795.00	645.00	1.22	(41)
0.35	11.00	43.70	39.35	1.11	700.00	720.00	0.97	(42)
0.45	11.00	31.50	28.80	1.09	755.00	785.00	0.96	(42)
0.31	8.00	57.27	43.93	1.30	150.00	170.00	0.88	(43)
0.31	10.00	70.32	43.93	1.60	110.00	170.00	0.65	(43)
0.31	12.00	70.53	43.93	1.61	70.00	170.00	0.41	(43)
0.38	10.00	56.50	51.80	1.09	650.00	750.00	0.87	(44)
0.38	10.00	63.40	52.50	1.21	760.00	840.00	0.90	(44)
0.50	5.00	46.20	46.70	0.98	180.00	180.00	1.00	(45)
0.50	10.00	47.50	46.70	1.01	180.00	180.00	1.00	(45)
0.38	10.00	56.50	51.80	1.09	650.00	750.00	0.87	(46)
0.38	10.00	58.30	52.00	1.12	700.00	800.00	0.88	(46)
0.38	10.00	63.40	52.50	1.21	760.00	840.00	0.90	(46)

w/b water/binder ratio; f_c = Compressive strength of the mixture with SF; f_c (Base)= Compressive strength of the mixture without SF; Slump= Slump of the mixture with SF; Slump (Base)= Slump of the mixture without SF. In light blue are highlighted the mixtures where slump flow was measured instead of standard slump. PS= Present Study

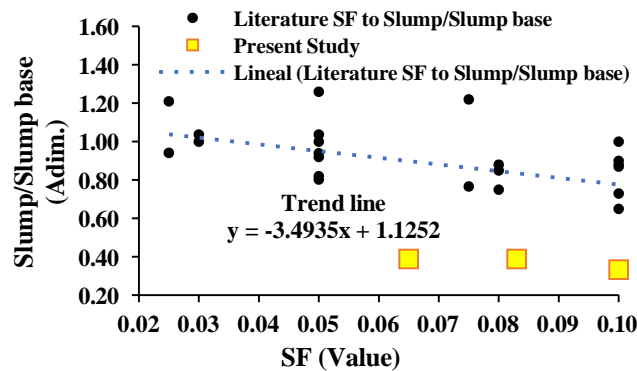


Figure 2. Slump behavior in the literature for SF

On the contrary, for nSi, a slightly increasing trend in the slump ratio was observed in the literature as an overall behavior (Figure 3). Notwithstanding, most of the cases found in literature show a slump ratio lower than 1, indicating a general decrease in slump when using this additive.

The increase in workability with nSi is probably due to the material's suspension features. Nevertheless, in some cases in the literature is

observed a loss of the material's workability with the incorporation of nano-silica (47,48), while other authors have observed increases in workability with dosages greater than 0.5% w/b (49). Therefore, to confirm these results, the same dosages were tested with another type of cement and aggregate, a plasticizer dose of 0.3% w/b, and nSi in different proportions (named nSi2). Figure 1 shows that an increase in workability was maintained by incorporating

nSi2, similar to that seen in Figure 1 b). This confirms the trend towards an increase in slump ratio by incorporating nSi with the same w/b ratio.

Furthermore, more than 60 mixtures with the addition of nSi were found in the recent literature specifically studying workability. Most

of the studies focused on a range between 0.5% and 3% of nSi. Workability is found proportional to nSi percentages in this range, nevertheless, for percentages higher than 3% fewer studies were found and with not a clear trend (Table 5, Figure 3).

Table 5. Literature studies using nano silica (nSi) addition in concrete.

w/b	nSi (%)	fc (MPa)	fc (MPa) (Base)	fc/ fc Base(MPa)	Slump (mm)	Slump (Base) (mm)	Slump / Slump (Base)	Reference
0.47	0.00	36.47	36.47	1.00	90.00	90.00	1.00	PS
0.47	0.50	34.80	36.47	0.95	140.00	90.00	1.56	PS
0.47	1.00	35.60	36.47	0.98	120.00	90.00	1.33	PS
0.47	1.50	36.90	36.47	1.01	135.00	90.00	1.50	PS
0.47	2.00	42.80	36.47	1.17	130.00	90.00	1.44	PS
0.47	2.50	45.60	36.47	1.25	160.00	90.00	1.78	PS
0.51	3.00	43.12	38.11	1.13	92.00	109.00	0.84	(39)
0.47	6.00	44.70	38.11	1.17	73.00	109.00	0.67	(39)
0.47	8.00	40.52	38.11	1.06	81.00	109.00	0.74	(39)
0.40	0.50	45.10	42.70	1.06	80.00	85.00	0.94	(50)
0.40	1.00	47.40	44.70	1.06	71.00	85.00	0.84	(50)
0.41	1.50	50.80	42.90	1.18	66.00	85.00	0.78	(50)
0.41	2.00	52.60	42.50	1.24	61.00	85.00	0.72	(50)
0.31	0.50	60.10	56.50	1.06	39.00	43.00	0.91	(47)
0.31	1.00	63.60	56.50	1.13	32.00	43.00	0.74	(47)
0.31	1.50	65.70	56.50	1.16	38.00	43.00	0.88	(47)
0.31	2.00	68.30	56.50	1.21	42.00	43.00	0.98	(47)
0.31	2.50	66.60	56.50	1.18	45.00	43.00	1.05	(47)
0.31	3.00	64.70	56.50	1.15	47.00	43.00	1.09	(47)
0.31	1.01	62.80	58.80	1.07	170.00	170.00	1.00	(43)
0.31	2.04	67.10	58.80	1.14	160.00	170.00	0.94	(43)
0.31	3.09	63.90	58.80	1.09	140.00	170.00	0.82	(43)
0.48	0.50	57.50	56.30	1.02	100.00	110.00	0.91	(25)
0.48	1.00	60.00	56.30	1.07	80.00	110.00	0.73	(25)
0.48	3.00	63.00	56.30	1.12	60.00	110.00	0.55	(25)
0.48	5.00	52.00	56.30	0.92	20.00	110.00	0.18	(25)
0.36	2.50	65.73	59.06	1.10	720.00	650.00	1.11	(41)
0.36	5.00	66.12	59.06	1.12	635.00	650.00	0.98	(41)
0.36	7.50	71.65	59.06	1.21	565.00	650.00	0.87	(41)
0.38	10.00	71.30	51.80	1.38	740.00	750.00	0.99	(44)
0.38	10.00	82.10	52.50	1.56	820.00	840.00	0.98	(44)
0.38	2.04	56.50	51.80	1.09	740.00	750.00	0.99	(46)
0.38	2.04	58.30	52.00	1.12	790.00	800.00	0.99	(46)
0.38	2.04	63.40	52.50	1.21	820.00	840.00	0.98	(46)
0.35	1.01	43.80	39.35	1.11	750.00	720.00	1.04	(42)
0.35	3.09	46.90	39.35	1.19	690.00	720.00	0.96	(42)
0.35	5.26	46.00	39.35	1.17	625.00	720.00	0.87	(42)
0.45	1.01	32.40	39.35	0.82	750.00	785.00	0.96	(42)
0.45	3.09	34.85	39.35	0.89	770.00	785.00	0.98	(42)
0.45	5.26	33.80	39.35	0.86	650.00	785.00	0.83	(42)
0.30	2.40	110.90	105.70	1.05	158.00	140.00	1.13	(38)
0.30	4.78	113.00	105.70	1.07	130.00	140.00	0.93	(38)
0.35	2.23	102.40	97.30	1.05	160.00	157.00	1.02	(38)

0.35	4.42	105.40	97.30	1.08	150.00	157.00	0.96	(38)
0.40	2.07	72.00	68.10	1.06	175.00	181.00	0.97	(38)
0.40	4.12	75.90	68.10	1.11	169.00	181.00	0.93	(38)
0.45	1.94	50.90	44.70	1.14	120.00	125.00	0.96	(38)
0.45	3.85	59.60	44.70	1.33	110.00	125.00	0.88	(38)
0.37	0.50	44.13	34.32	1.29	46.00	32.00	1.44	(51)
0.37	1.00	46.48	34.32	1.35	76.00	32.00	2.38	(51)
0.37	1.50	49.13	34.32	1.43	97.00	32.00	3.03	(51)
0.37	2.00	52.46	34.32	1.53	106.00	32.00	3.31	(51)
0.60	2.50	29.28	29.28	1.00	108.00	108.00	1.00	(52)
0.60	5.00	30.33	29.28	1.03	185.00	108.00	1.71	(52)
0.60	7.00	29.80	29.28	1.02	235.00	108.00	2.17	(52)
0.55	2.50	35.80	35.80	1.00	102.00	102.00	1.00	(52)
0.55	5.00	37.07	35.80	1.03	210.00	102.00	2.05	(52)
0.55	7.00	38.34	35.80	1.07	241.00	102.00	2.36	(52)
0.50	2.50	39.65	39.65	1.00	121.00	121.00	1.00	(52)
0.50	5.00	44.26	39.65	1.11	210.00	121.00	1.73	(52)
0.50	7.00	47.63	39.65	1.20	235.00	121.00	1.94	(52)
0.38	1.00	39.29	34.32	1.14	215.90	440.00	0.49	(53)
0.38	1.50	41.17	34.32	1.20	234.90	600.00	0.39	(53)
0.38	2.00	44.45	34.32	1.30	234.90	680.00	0.35	(53)
0.38	2.50	40.95	34.32	1.19	241.30	720.00	0.34	(53)
0.38	3.00	36.15	34.32	1.05	241.30	740.00	0.33	(53)
0.28	0.50	87.24	76.52	1.14	720.00	790.00	0.91	(35)
0.28	1.50	91.87	76.52	1.20	685.00	790.00	0.87	(35)

w/b water/binder ratio; fc= Compressive strength of the mixture with SF; fc (Base)= Compressive strength of the mixture without SF; Slump= Slump of the mixture with SF; Slump (Base)= Slump of the mixture without SF. In light blue are highlighted the mixtures where slump flow was measured instead of standard slump. PS= Present Study

Regarding the mixtures SF8.5-nSi0.2 and SF10.0-nSi0.2 of the present study, the manageability condition was critical, blocking good compaction of the sample and allowing an increase of the air content, therefore, leading to a reduction of the final resistance. Although the

pozzolanic material can react in smaller spaces to densify on a smaller scale, if the mixture has larger internal cavities due to the lack of workability of the mixture, the generated matrix does not have enough density to reflect the effect of pozzolanic additions.

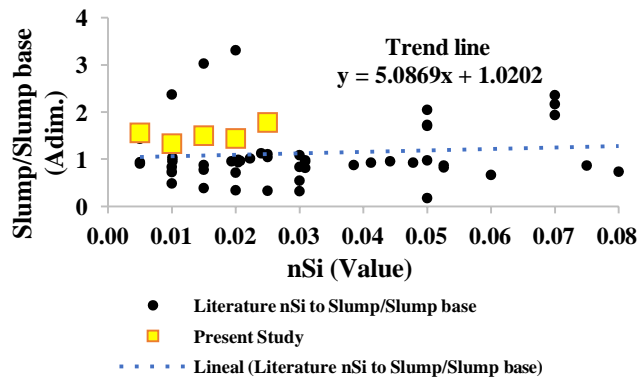


Figure 3. Slump behavior in the literature for nSi

Table 6. Literature studies using SF+nSi addition in concrete.

Mixture	w/b	% of SF	% of nSi	fc (MPa)	fc with only SF (MPa)	fc with only nSi (MPa)	fc (Base) (MPa)	Slump (mm)	Slump with only SF	Slump with only nSi	Slump (Base) (mm)	Reference
SF8.5-nSi0.2	0.50	8.50%	0.20%	39.23	40.10	42.80	36.47	20.00	35.00	130.00	90.00	PS
SF10-nSi0.2	0.50	10.00%	0.20%	35.44	40.90	42.80	36.47	0.00	35.00	130.00	90.00	PS
SF5-nSi0.5	0.28	5.00%	0.50%	95.25	79.27	87.24	76.52	690.00	740.00	720.00	790.00	(35)
SF5-nSi0.5	0.30	5.00%	0.50%	118.10	113.20	110.90	105.70	122.00	140.00	158.00	140.00	(38)
SF5-nSi1	0.30	5.00%	1.00%	121.40	113.20	113.00	105.70	110.00	140.00	130.00	140.00	(38)
SF5-nSi0.5	0.35	5.00%	0.50%	108.50	106.20	102.40	93.70	145.00	128.00	160.00	157.00	(38)
SF5-nSi1	0.35	5.00%	1.00%	111.60	106.20	105.40	93.70	129.00	128.00	150.00	157.00	(38)
SF5-nSi0.5	0.40	5.00%	0.50%	84.90	80.50	72.00	68.10	188.00	181.00	175.00	181.00	(38)
SF5-nSi1	0.40	5.00%	1.00%	90.60	80.50	75.90	68.10	187.00	181.00	169.00	181.00	(38)
SF5-nSi0.2.5	0.45	5.00%	0.50%	65.40	62.80	28.70	44.70	169.00	115.00	120.00	125.00	(38)
SF5-nSi1	0.45	5.00%	1.00%	73.30	62.80	39.70	44.70	166.00	115.00	110.00	125.00	(38)
SF2.5-nSi5	0.36	2.50%	5.00%	69.26	63.79	68.73	60.33	670.00	765.00	605.00	645.00	(41)
SF2.5-nSi2.5	0.36	2.50%	2.50%	82.17	63.79	65.73	60.33	685.00	765.00	695.00	654.00	(41)
SF5-nSi2.5	0.36	5.00%	2.50%	71.15	65.01	65.73	60.33	705.00	820.00	695.00	645.00	(41)
S10-nSi3	0.35	10.00%	3.00%	51.70	43.70	46.90	39.35	605.00	615.00	610.00	720.00	(42)
S10-nSi3	0.35	10.00%	3.00%	37.10	31.50	34.85	28.80	650.00	635.00	640.00	785.00	(42)
SF10-nSi5	0.35	10.00%	5.00%	33.90	31.50	33.80	28.80	625.00	635.00	580.00	785.00	(42)
SF10-nSi5	0.45	10.00%	5.00%	47.15	43.70	46.00	39.35	560.00	615.00	560.00	720.00	(42)
SF10-nSi1	0.45	10.00%	1.00%	34.70	31.50	32.40	28.80	715.00	635.00	645.00	785.00	(42)
SF10-nSi1	0.45	10.00%	1.00%	48.50	43.70	43.80	39.35	710.00	615.00	600.00	720.00	(42)
SF10-nSi2	0.38	10.00%	2.00%	87.90	63.40	82.10	52.50	740.00	760.00	820.00	750.00	(44)
SF10-nSi2	0.38	10.00%	2.00%	78.80	56.50	71.30	51.80	640.00	650.00	740.00	750.00	(44)
SF10-nSi2	0.38	10.00%	2.00%	78.80	56.50	71.30	51.80	640.00	650.00	740.00	750.00	(46)
SF10-nSi2	0.38	10.00%	2.00%	83.50	58.30	80.40	52.00	700.00	700.00	790.00	800.00	(46)
SF10-nSi2	0.38	10.00%	2.00%	87.90	63.40	82.10	52.50	740.00	760.00	820.00	840.00	(46)

w/b water/binder ratio; f_c = Compressive strength of the mixture PS= Present Study

It is worth mentioning that all samples had the same content of plasticizer material. Table 6 and Figure 4 show an interesting comparison between 25 observed that other authors found similar trends mixtures of SF+nSi found in literature. It is (35,38).

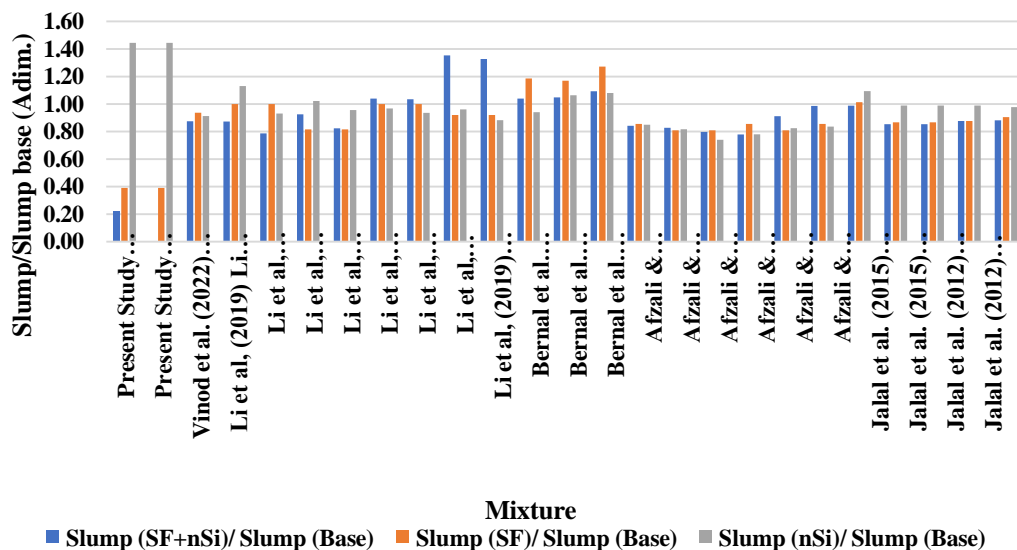


Figure 4. Slump behavior in the literature for a) SF, b) nSi, c) SF+nSi mixtures

3.2. Compressive strength

In the first stage, the compressive strength results (in Figure 1 a) show an increase with the addition of SF (from 36.47 MPa to 40.90 MPa). However, this was tangible only with dosages above 6.5%, reaching an increase in resistance

up to 12.2%, compared to the standard mixture (see Figure 5). The increase in f_c was due to the formation of C–S–H bonds, during the hydration of the mixture, between the particles of cement and the silicon of SF, according to the literature (18,50).

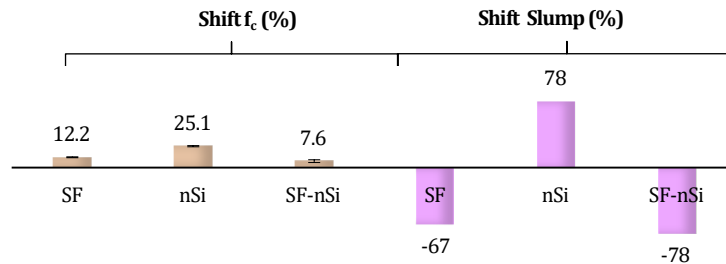


Figure 5. Percent variation of the standard sample containing no pozzolanic addition in a) Compressive strength; and b) Slump for higher values of compressive strength, compared to plain mix. Data includes standard deviation where applicable.

On the other hand, as can be seen in Figure 1 b) in the case of nSi, an increase in compressive strength was observed with dosages from 2%, reaching an improvement in f_c up to 25% with the addition of nSi (Figure 5), in accordance with Gesoğlu et al., (2012), (54). The initial increase in compressive strength with the addition of nSi was attributed to the increase in the dimensions and rigidity of CSH chains during hydration of the mixture's components, which can be produced by the presence of active nucleation sites from the pozzolanic reaction. This contributed to the densification of the microscale concrete structure by diminishing the concrete's porosity and improving the properties of the interfacial transition zone (17,22). In advanced stages, nSi modifies the internal structure of the C–S–H gel, increasing the

average chain length, which leads to a denser structure. Hence, it is advisable to study this behavior at latter stages. The amount of CH drops, due to the pozzolanic reaction, while the large pores are partially or completely filled with hydration products (especially secondary C–S–H), (55).

As a decline in resistance was not achieved with the dosages utilized to confirm the results. In the literature (56), the same dosages were tested with another type of cement and aggregate, a plasticizer dose of 0.3% w/b, and nSi (named nSi 2). Figure 6 shows a drop in f_c , (56). This may have been because, for high SC dosages, the content of chemically combined water increases through the pozzolanic reaction of the nSi and, therefore, there is no free water to provide complete settlement. On the other hand, the

excessive presence of nSi particles fills the pores and prevents $\text{Ca}(\text{OH})_2$ crystals from growing

and increasing the resistance, as has been shown previously, (57).

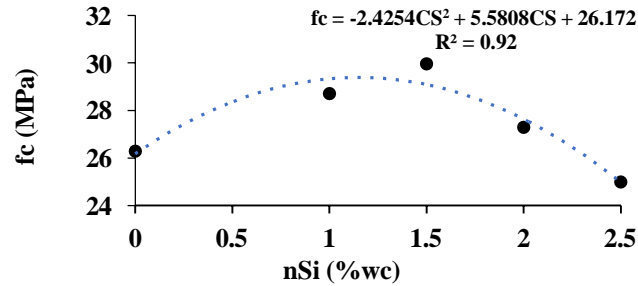


Figure 6. f_c results from second mix with nSi for the present study (PS). Adapted from (56).

Figure 7 shows the results found in the literature, compared with the present study. It is noted that for percentages lower than 3% of nSi there are exponential increases in compressive strength,

according to the literature. However, for higher percentages no more significant increases are observed.

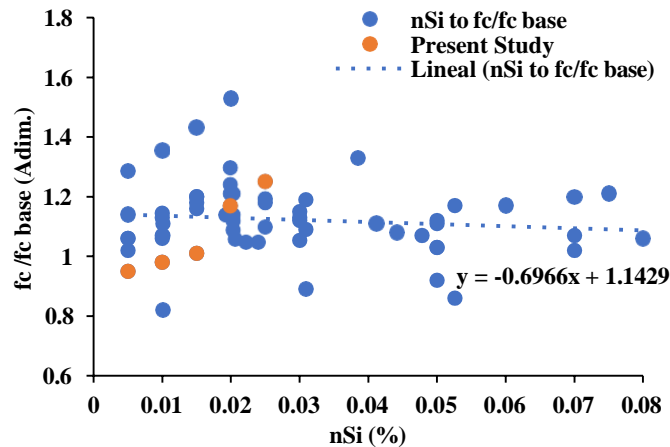


Figure 7. f_c (MPa) Improved/ f_c (MPa) results of other studies in the literature with nSi addition compared with the present study.

With the results using nSi 2, the materials affected the optimum dosage point, propitiating a decline in resistance for high dosages of SC. Previous research on the microstructure of concrete has revealed that an increase in the dose

of nano-silica from 3% to 6% did not increase CH consumption, suggesting that the overall improvement in performance associated with the increased addition of high nano-silica may be

mainly due to the effect of the physical filler in the cement-based matrix (58).

With the addition of both siliceous compounds, a 7.6% increase in f_c was observed (Figure 5); the combined effect of siliceous pozzolanic additions increased the amount of SiO_2 particles, which are very fine and have a high surface area-to-volume ratio. Therefore, when the amount of SiO_2 is greater than a critical value, the clustering of particles occurs and, so, they cannot be dispersed well in the mix, as has been reported previously (25). On the other hand, the amount of SiO_2 may have exceeded the amount Table 5 and Table 6. It is feasible that, with higher doses of SF, there may still be an increase in f_c as found in the literature.

necessary to deplete the $\text{Ca}(\text{OH})_2$ required during the hydration reaction to form the gel that gives rise to the C–S–H bonds, (24). Consequently, it is impossible to conform the homogeneous and dense microstructure of the materials, as the de-clustering of the nanoparticles is essential to achieve an improvement in concrete properties (59).

Although SF and nSi pozzolanic materials were added in this study, the results of the optimal ranges found for SF (10.0%) and NSI (1.5–2.0%) were consistent with those found in the

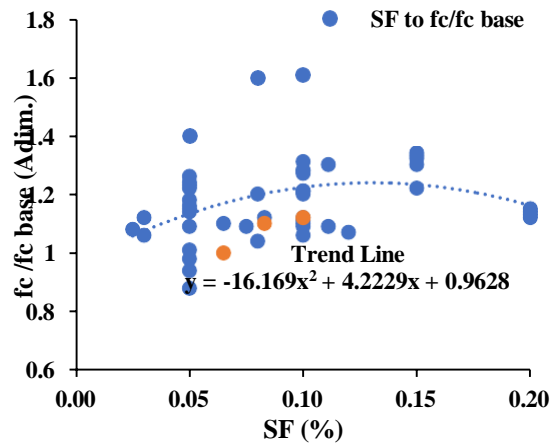


Figure 8. f_c (MPa)/ f_c base (MPa) results of other studies in the literature with SF addition compared with the present study.

Table 5 and Table 6). The main purpose of this analysis was to fit each f_c data set to a probability distribution model, considering SF, nSi and SF+nSi. This analysis was performed

using MATLAB R2013a (The MathWorks Inc., Natick, Massachusetts, US), in which all statistical inferences assumed a confidence level of 95% ($\alpha = 0.05$). The goodness of normal fitted

distributions for each addition type (SF, nSi and SF+nSi) was assessed using a procedure based on the log likelihood ratio test, which results showed values between 5.55 and 39.75, meaning that the normal distribution is a good estimator of the data. Figure 9 a) and b) shows the normal and the cumulative distributions of the data.

However, the workability of the mixture must be improved. By combining the optimal doses

without adding more superplasticizer, the resistance dropped dramatically, leading to results opposite to those observed in Table 4. This means that, in ternary mixtures, dosing of the additive at an important amount is vital to promoting the capacity to generate a compact matrix, thus improving the resistance.

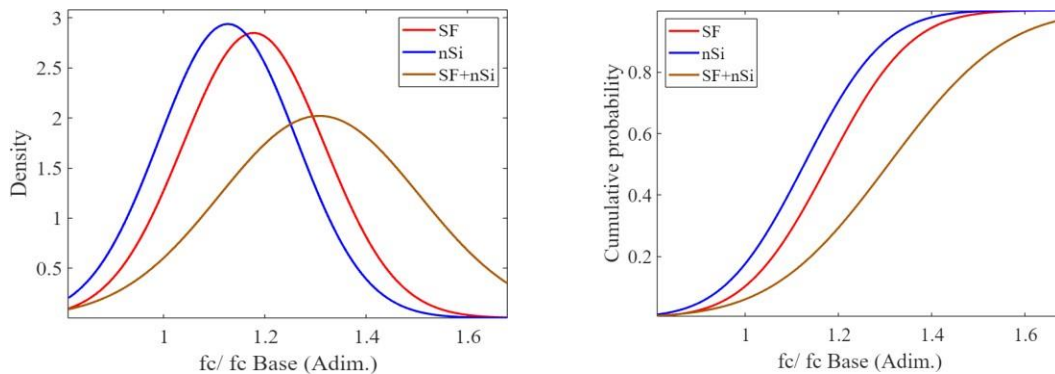


Figure 9. a) f_c/f_c base Normal Distribution; b) f_c/f_c base Cumulative distribution, for SF, nSi and SF+nSi.

When investigating the properties of high-performance self-compacting concrete with SF, they significantly improve for ternary samples whose agglomerate is cement, SF, and nSi. Increasing the SF content greatly increases the compressive strength, especially at latter stages. In comparison with control samples, when replacing up to 10% of SF in binary mixtures (with cement binder and SF), compressive strength was increased by 21.0% after 28 days. In contrast, 2% replacement of nSi in binary mixtures increased the compressive strength by 56% after 28 days, while 10% replacement of SF and 2% of nSi in ternary mixtures increased the compressive strength by 67% after 28 days. In

binary mixtures, the enhancement in compressive strength was greater in mixtures containing 2–3.5% nSi and the highest occurred in ternary mixtures. When cement fractions are substituted by amorphous silicas at the microscopic scale, a more refined and packaged pore structure of the concrete can be observed, which can lead to an increase in the concrete strength and durability properties (44,46,60). Aside from the compressive strength (f_c), the rheological, thermal, and transport properties have been shown to improve in mixtures containing nSi and SF. However, when replacing high quantities of SF (beyond 15%) or nSi (beyond 3.5%), the yield decreases (60).

The rheological properties of the mixtures containing 2% of nSi have shown workability levels close to those of the mixtures without additives (44). The maximum contraction without restriction increased by about 80% for mortars with nSi at 7 days and by 54% at 28 days, compared to mortars with SF within the same periods (60).

Table 5 and Table 6 show the results of the optimum compressive strength tests at 28 days in which a percentage of cement with SF, nSi (in colloidal or powder form), and/or a combination of both was used. Furthermore, Table 3-4 and 5 show the mixtures where the individual and joint behavior of SF and nSi in concrete were assessed simultaneously. In that group, when a comparison was made with the mixtures in which the SF was replaced by nSi, there was a 30% increase in f_c , and a 41% increase when adding both materials as a replacement for cement. The average values taken as optimal in the studies were 10.3% of SF and 2.3% of nSi.

In studies where high amounts of nSi were used, the amount of additive added is generally above 3.5 % w/b (42,60), even as high as 5% w/b (20). In investigations, such as those of Behfarnia and Rostami, (2017) or Bernal et al., (2018), where the percentage of nSi was high, the concretes were made with special procedures, through the addition of other materials that require alkaline activation with Na (OH) or the implementation of curing with limestone water. Alkaline

The different contents of nSi, with values approaching 2% nSi, also improved the modulus of elasticity, flexure resistance, load and displacement behavior, and fracture energy after 90 days. Ternary samples of cement, nSi, and SF seem to yield better results than concrete samples that only contain nSi (20).

activation occurs with the reaction between solid aluminosilicate powders, such as fly ash, blast furnace slag, or metakaolin, and an alkaline solution. Pozzolanic materials are combined with hydrated calcium hydroxide (CH) to form hydrated calcium silicate (C-S-H), which is responsible for the strength of hydrated cement pastes (61). In the same line, it is worth mentioning that Jalal et al., (2012) have achieved the highest values of f_c in concrete, using limestone aggregate and siliceous sand with partial replacement of limestone filler. These materials provide a higher content of calcium carbonate, which allows for greater CH formation and helps to complete the pozzolanic reaction. Furthermore, lime filler has been shown to accelerate the hydration of C_3S , thus increasing early compression resistance (62). On the other hand, Jalal et al., (2012) ensured good manageability by using a high dose of superplasticizer (3.12% w/b), a viscosity modifying agent, a maximum size of low coarse aggregate (12.5 mm), and established values close to the optimum ones seen in

Table 5 and Figure 3 (i.e., 0% SF and 2% nSi) for their mixture.

The aggregate of the material significantly defines the final resistance values. For example, the f_c values of Afzali Naniz & Mazloom, (2018) were much lower than the others, as their coarse aggregate was light. Mortar f_c values are generally higher, as the fineness of the material leaves less pores in the mixture; however, it is also necessary to emphasize the fact that mortar mixtures incorporate higher dosages of SF and a lower w/b ratio. In the research reported in the literature shown above, mixing water was kept constant, as part of the cement was replaced by pozzolan. Another way of combining amorphous silicas is as an addition; that is, maintaining a constant weight of the cement and changing the amount of water in the mixture to maintain the w/b ratio.

Additionally, the authors found in the literature numerous applications of nSi and SF also in mortars although it is not the object of this study. It was noted that with the addition of nSi up to 2% there is agreement in the literature on the benefits over f_c . However, as for concrete, when the percentages exceed 3% the benefits in terms of f_c are the object of discussion, having contrasting results in the literature. Furthermore, with the addition of SF, a minor increase in f_c is noted in the literature. Nevertheless, percentages greater than 6% - 8% show there may still be an increase in f_c as found for the concrete.

3.3. Cost–benefit analysis

The present study shows concrete mixtures with the addition of SF and nSi with an overview in the literature. However, in developing countries, there is a significant lack of research on this issue and an almost total absence of cost–benefit analyses.

Therefore, even though it was not the main objective of this study, a simplified cost–benefit analysis is presented in, considering only direct costs and applied to the Colombian context. The cost of concrete was calculated per m^3 , according to material local prices (in Cartagena) from quarries and cement makers, including labor costs. Therefore, the costs were calculated in Colombian pesos (COP) and converted to USD by applying the exchange of COP 4,392.00 for 1 USD (rate at September 2022).

Cost variation was found to range between +17.9% and +72.2%, while strength variation was between -4.6% and +25%. This means that a cost increase does not always correspond to a strength improvement. Furthermore, unit cost per MPa of strength showed that the lower rate corresponded to the control sample, 3.03 (USD/MPa), while the most convenient between the other mixtures proposed was SF0-NSI2.5, with 3.11 (USD/MPa). In fact, the latter, with the 2.5% nSi addition to the standard mixture, reached up to a 25% strength (f_c) increase.

Table 7. Cost–benefit analysis

SF (%)	nSi (%)	Cost (USD)/m ³	f_c (MPa)	Δf_c (MPa)	Δ Cost (%)	Δ Strength (%)	Cost (USD)/ f_c (MPa)	Cost (USD)/ f_c Improved (MPa)
0.0	0.0	87.7	36.47	-	-	-	2.40	-
6.5	-	134.2	-	0.03	53.1	0.1	-	3.68
8.5	-	142.4	-	3.63	62.4	10.0	-	3.55
10.0	-	150.1	-	4.43	71.2	12.1	-	3.67
-	0.5	103.3	-	-1.67	17.9	-4.6	-	2.97
-	1.0	105.6	-	-0.87	20.4	-2.4	-	2.96
-	1.5	107.8	-	0.43	23.0	1.2	-	2.92
-	2.0	110.0	-	6.33	25.5	17.4	-	2.57
-	2.5	112.3	-	9.13	28.1	25.0	-	2.46
8.5	0.2	144.1	-	2.76	64.4	7.6	-	3.68
10.0	0.2	150.9	-	-1.03	72.2	-2.8	-	4.25

Cost = cost of one m³ of concrete in (USD); USD = United States of America Dollar; Δ Cost = Cost variation between f_c Improved and f_c (per m³); Δ Strength = Strength variation between improved mixture and standard mixture; f_c Improved = $f_c + \Delta f_c$.

Further studies should progressively increase the nSi rate, which appears to be the most promising mixture, as well as carrying out sustainability assessments. In the literature, encouraging results can be found in this regard. Gunasekara et al., (2020) proposed a sustainability assessment of High-Volume Fly Ash (HVFA) concrete and Portland Concrete (PC) with and without nano-silica. They estimated the emissions factor considering the manufacturing processes, which corresponds to the energy consumption and emission activities during raw material extraction, transportation to production plant, and production processes. They finally found that the addition of nSi into the mix design caused a cost increment in HVFA concrete, corroborating what was found in the present work. However, cost reductions could be achieved by reducing transport costs and through efficient manufacturing techniques and procurement strategies.

The addition studied in this study can be considered cost-effective compared to other additives with similar effects found in literature such as nano iron (64), nano-metakaolin (NMK) (65) and nano-clay (66).

4. Conclusion

The main objective of this study was to evaluate the concrete compression behavior at various SF, nSi, and combined SF-nSi dosages, keeping constant the plasticizer dosages, w/b ratio, and the stone aggregates. Under the proportions studied and with the materials used, it can be concluded that SF dosages between 6.5% and 10.0% w/b can increase the compressive strength from 36.47 MPa to 40.90 MPa (12%), but significantly decrease the workability of the sample, generating a spike in viscosity that could hinder the compaction of the mixture in the formwork.

In contrast, nSi raised the compressive strength from 36.47 MPa to 45.63 MPa (25%) with doses above 1.5–2% w/b and increased the workability at dosages greater than 0.5% w/b, which may be due to the liquid suspension state of the nanomaterial. Improvements in strength can be attributed to the augmented production of C–S–H gels during the hydration and densification of the microstructure.

The results achieved suggest the need to optimize the SF and nSi dosage, when used simultaneously, in order to find the balance between workability and mechanical performance, given the fact that, within the proportions studied (nSi 0.2% and SF 10.0%), the loss of workability was up to 100%. This may be due to the sizes of the micro- and nanoparticles and their reactivity, which prevents the proper setup of the fresh mixture within the important molds, as well as their porosities, which limit the increase in mechanical resistance. A 2% nSi dosage can achieve the same effect as a 10% SF dosage; however, the optimum dosage points to reach a higher f_c may vary with the type of materials used.

The extensive literature review confirms that for higher SF percentages it is observed a loss of workability, while for nSi is observed a slightly positive trend for Slump/Slump base. However, most of the studies focus on nSi percentages lower than 3%. For future research is suggested an extensive experimental campaign considering SF percentages from 1% to 20% and nSi from 0.5% to 10%, whit intervals of 0.5% expanding

the physicochemical and mechanical parameters to be monitored.

Finally, a cost–benefit analysis was proposed, which has rarely been studied worldwide for such concrete additions. Authors did not wish to be exhaustive, in this regard. However, it should be noted that there is enormous potential in adding these types of fibers. Decreases in the unit cost of these materials could progressively lead to the entry of such mixtures into the construction market in the near future. For future research Life-Cycle Assessment (LCA) is suggested to clearly evaluate and identify environmental impact of the solution studied.

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