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# Effect of Nano Silica De-agglomeration, and Methods of Adding Super-plasticizer on the Compressive Strength, and Workability of Nano Silica Concrete

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## Abstract

Nowadays, nano silica appears to be one of the attractive cement substitution alternatives for researchers. Several authors have studied the physical effects of its incorporation on cement, despite of significant inconsistencies in reported results, specially workability and compressive strength of resulting concrete. This paper presents a thorough experimental investigation testing more than 16 concrete mixtures, which covers some of the main reasons of these inconsistencies. One of these problems is the method of applying the nano particles, as they are highly agglomerated, and if applied directly in a bulk composite, they often lose their high-surface area due to grain growth or unavailability of the high surface area where it matters. The presented research investigated how agglomeration can affect the compressive strength, and workability of normal strength concrete. Different methods of de-agglomeration were tested, as sonication, homogenization, and stirring. Besides, optimization of the super plasticizer's addition timing to concrete incorporating nano silica (NS) is elaborately investigated by adding the superplasticizer in different timings of addition; starting by adding NS to superplasticizer and water then applying de-agglomeration method, ending with de-agglomeration of NS alone with portion of water, then adding SP to the other portion of water before application them to the dry components. Results showed that sonication proved to be the most significant de-agglomeration method as it enhanced the gain in compressive strength of concrete by 23% by using only 1% nano silica as cement substitution. Sonication of NS also helped increasing the concrete workability significantly as a result of better dispersion of NS. As for the superplasticizer addition time, sonication of NS alone with portion of water, then adding SP to the other portion of water before application them to the concrete dry components showed a significant performance as compared to the other timings, as the compressive strength reached an increase of 26% compared to the control specimens.

**Keywords:** Nano silica, concrete, plasticizers, agglomeration, workability, strength.

## 1. Introduction

Concrete is by far the most widely used construction material worldwide. Lately, various efforts were exerted to improve the environmental friendliness of concrete to make it suitable as a Green Building material. Foremost and

most successful in this regard is the use of suitable substitutes for Portland cement. Nano silica is one of the candidate materials to be used as a cement substitution.

Contradicting conclusions in previous research on the effect of adding nano silica (NS) on the compressive strength gain, as many researchers as (M.Sari et al. 1999), (Ji T. 2005), and (Qing Y et al. 2008) were able to increase the compressive strength with increasing replacement percentage, researchers as (M. Collepardi et al. 2000) reported that up to 28 days the compressive strength of normal concrete (NC) was greater than found for concrete blended with NS.

Contradiction in optimum percentage of replacement is clear compared to other publications. Although Some authors defend that the appropriate percentage of nano silica has to be small (1–5 wt %) due to agglomeration caused by difficulties to disperse the particles during mix as (Qing Y et al. 2007), (Li H et al.2006), (Li G. 2004), and (Shih Jeng-Ywan et al. 2006), while others indicate that the improvement of the properties can also be achieved with higher dosages, of about 10 wt% (Jo BW. 2007), others indicated that adding 10% SiO<sub>2</sub> nano particles with dispersing agents has been observed to increase the compressive strength of cementitious composites at 28 days by as much as 26%, compared to only a 10% increase with adding 15% silica fume (Qing Y et al. 2008). Others concluded that the addition of small amounts of SiO<sub>2</sub> nano particles has been observed to increase the strength results in improving the 28 day compressive strength by 10% and flexural strength by 25% (Ji T. 2005), and (A. Nazari et al. 2011).

The third inconsistency is related to the effect of adding NS on concrete workability; although authors like (Sobolev K et al. 2005, 2009), and (Li H et al. 2004) concluded that nano silica was found to improve concrete workability and strength, others like (Neville AM. 1996), and (Peter C. 1998) concluded that when NS (wt%) is incorporated into the mortar in the fresh state it has a direct influence on the water amount required in cement mixtures, and cause the need for higher amounts of water or chemical admixtures in order to keep the workability of the mixture (F Sanchez et al. 2010).

When fine particles are added to cement, materials have a strong tendency to form settlements or agglomerates when it contacts water. This phenomenon affects the rheological behavior of the paste (Park CK 2005), and the ultimate hardened properties (Qing Y et al. 2007). Thus, there is a need to increase the repulsive forces between adjacent colloidal particles, by adding proper chemical admixtures or by adding extra water to disperse the solid particles in aqueous solution (Luciano Senff et al. 2009).

So we can conclude that the major problem is the utilization of nano particles, as they are highly agglomerated and if used directly in a bulk composite, they often lose their high-surface area due to grain growth or unavailability of the high surface area where it matters.

Currently, another issue of great importance is the extensive use of chemical admixtures mainly to control/modify the fresh and hardened properties of concrete. A particular challenge of interest to the authors is to optimize the use of dispersing agents such as super plasticizers in high performance concretes containing high volumes of supplementary cementing materials (SCMs). Dispersing agents such as super plasticizers are commonly used in these concretes. There are, however, practical problems such as loss of workability with time that are controlled by interactions with cement components. Controlling the timing of the availability of an admixture in cement systems is essential for its optimal performance (Laila Raki et al. 2010).

This paper studies clarifying the conflict reported about the compressive strength, and workability results of nano silica concretes by studying the effect of different de-agglomerating, and dispersing techniques of nano silica on the gain in compressive strength, and workability of concrete, as well as optimizing the super plasticizer's most effective timing of addition to concrete mixtures to achieve the highest workability, and compressive strength results. The results will be evaluated through the compressive strength results; 7, and 28 days values, coefficient of variation of results, and modulus of elasticity, as well as scanning electron microscope plates.

## **2. Experimental work**

### *2.1. Materials*

Ordinary Portland Cement (OPC) conforming to ASTM C150 standard was used as received. Chemical and physical

properties of used cement are given in Table 1. SiO<sub>2</sub> nano particles with average particle size of 30 nm and 45 m<sup>2</sup>/g Blaine fineness produced from WINLAB laboratory chemicals, UK was used as received. The properties of SiO<sub>2</sub> nano particles are shown in Table 2. Transmission electron micrographs (TEM) and powder X-ray diffraction (XRD) diagrams of SiO<sub>2</sub> nano particles are shown in Figs. 1 and 2. Crushed limestone aggregates, as well as sand free of alkali-reactive materials were used to insure producing durable Concretes; the aggregates were mixed by percentages of 65% for coarse aggregate, and 35% for fines by volume. A polycarboxylate with a polyethylene condensate de-foamed based admixture (Glenium C315 SCC) was used. Table 3 shows some of the physical and chemical properties of polycarboxylate admixture used in this study.

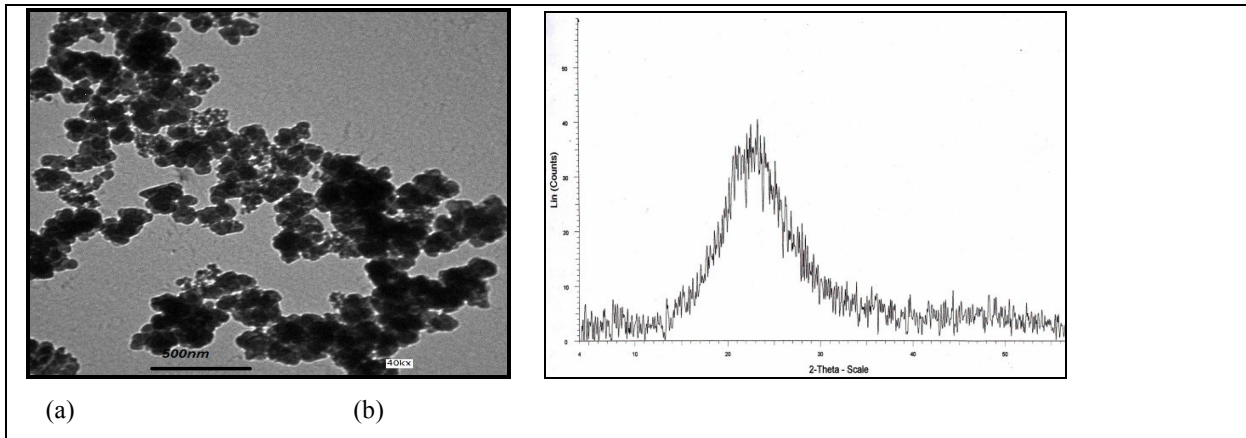


Figure 1: (a) TEM micrograph of SiO<sub>2</sub> Nano particles, (b) XRD analysis of SiO<sub>2</sub> Nano particles.

Table1. Properties of Portland cement (wt%).

Element	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	SO <sub>3</sub>	Na <sub>2</sub> O	K <sub>2</sub> O	L.O.I
Cement	20.13	5.32	3.61	61.63	2.39	2.87	0.37	0.13	1.96

Table2. Chemical composition of Nano SiO<sub>2</sub> (wt %).

Element	SiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>	MgO	CaO	Na <sub>2</sub> O	P <sub>2</sub> O <sub>5</sub>
NS	99.17	0.06	0.13	0.11	0.14	0.40	0.01

Table3. Physical and chemical characteristics of the polycarboxylate admixture.

Appearance	Off white opaque liquid
Specific gravity @ 20°C	1.095 ± 0.02 g/cm <sup>3</sup>
PH-value	6.5 ± 1
Alkali content (%)	Less than or equal to 2.00
Chloride content (%)	Less than or equal to 0.10

## 2.2. Experimental program

- The experimental program was divided into three main routes; the first investigates the effect of adding different percentages of –as received nano silica on compressive strength of concrete to reach optimum percent, the second compares three proposed methods for de-agglomeration of NS using sonication, homogenizer, and stirring, and the last is studying the effect of time and method of applying the super plasticizers on different properties of NS concrete.

- A total of 16 mixtures were prepared as shown in table 4. Sets of 6 cubes (15\*15\*15 cm<sup>3</sup>) were cast to perform compression strength tests after 7, and 28 days of water curing. Cubes were consolidated in accordance to ASTM C 192 in three layers on a vibrating table, where each layer was vibrated for 10 seconds, and then the

specimens were de-molded after 24 hours and cured in normal free water at room temperature until the day of testing.

#### 2.2.1 Effect of adding different percentages of nano silica (NS) as received on the gain in compressive strength of concrete.

Mixes prepared for this step with constant binder/aggregate weight ratio (B/A) of 1:3.57, and water/binder (W/B) ratio of 0.4. Mixtures COD, NSD1, NSD1.5, and NSD2 were produced with 0%, 1.0%, 1.5%, and 2.0% nano silica, used as received, by weight replacing cement. The amount of Superplasticizer (SP) was set to 0.42 wt% of the binder (cement plus nano silica). The mixtures compositions are shown in Table 4. Preparation of mixtures was performed in the following sequence:

(a) Weighing components, (b) mixing the solid components inside a turn tilt mixer for 1 min, (c) adding nano silica and superplasticizer into water for helping dispersing the nano silica, (d) adding the solution to the mixer and (e) finally mechanical mixing for 3 min.

#### 2.2.2. Effect of de-agglomeration of nano silica on the compressive strength of the resulting concrete.

Used de-agglomeration methods prior to application to concrete mix are: (a) Sonication for 5 min at 59 KHz frequency and 135 Watt absorbed power using a 4.5 liter capacity bath sonicator, since ultrasound treatment has been successfully used to break and disperse the silica fume agglomerates in order to obtain a more reactive material with a particle size distribution lower than 1  $\mu$ m (D. Martinez et al. 2008, and 2011). the use of sonication was thought to be more effective with the nano silica (Erich D. Rodriguez et al. 2012). (b) Homogenizing for 5 min using a basic rotor-stator homogenizer at speed of 24000 rpm, since the homogenizer's main role is to reduce particle size and disperse throughout a fluid and (c) finally stirring for 5 min using a low speed stirrer at speed of 120 rpm. The three methods were used for mixing 1% of nano silica, with a portion of the used water. The produced solution was then added to the rest of water and SP before being mixed in the mixer with the other dry components.

To compare the three methods, two series of mixtures were prepared; the first of them consists of three sets that were prepared with the same constituents mentioned in step one but using sonication, homogenizing, and stirring, the mixes are SD105, HD105, and TD105 respectively. For the sake of comparison a fourth mix PD1 was prepared by adding the nano silica as powder to the dry mix components, the second series consists also of three sets with the same constituents except they were prepared without adding super plasticizer, the mixtures were named SCD105, HCD105, and TCD105. For comparing the gain in strength of the mentioned sets a control mixture COCD was prepared without adding nano silica and super plasticizer as shown in table 4.

#### 2.2.3. Optimization of the super plasticizer's addition timing to the nano silica concrete mixtures.

Two different timings were chosen for the homogenized and sonicated NS in addition to the previously conducted SD105, and HD105 as shown in (Fig.4). First, powdered nano silica, water, and super plasticizer were added together before being sonicated (GSD105), where in the second mix SDG105 the sonicated nano silica was added to a portion of mixing water, and all poured to mixer for 1.5 min before 1 liter of water with the super plasticizer were added to the mixer, and then finally mixing the whole mixture for 2.5 min in the mixer. See table 4, and figure 4. Typically mixtures GHD105, and HDG105 were prepared using homogenization instead of sonication.

Table 4: Mixtures composition per 1 m<sup>3</sup>:

STEP	MIX	CEMENT (kg)	AGGREGATE		WATER (liter)	S.P. (liter)	N.S. (kg)	MIXING NOTES
			COARSE (kg)	FINE (kg)				
STEP 1	COD	480	1109	597	192	2	0	CONTROL
	NSD1	475.2	1109	597	192	2	4.8	NORMAL
	NSD1.5	472.8	1109	597	192	2	7.2	NORMAL
	NSD2	470.4	1109	597	192	2	9.6	NORMAL
STEP 2	SD105	475.2	1109	597	192	2	4.8	SONICATION
	HD105	475.2	1109	597	192	2	4.8	HOMOGENIZER
	TD105	475.2	1109	597	192	2	4.8	STIRRER
	PD1	475.2	1109	597	192	2	4.8	POWDER
	COCD	475.2	1109	597	193	0	4.8	CONTROL
	SCD105	475.2	1109	597	193	0	4.8	SONICATION
	HCD105	475.2	1109	597	193	0	4.8	HOMOGENIZER
	TCD105	475.2	1109	597	193	0	4.8	STIRRER
STEP 3	GSD105	475.2	1109	597	192	2	4.8	SONICATION
	SDG105	475.2	1109	597	192	2	4.8	SONICATION
	GHD105	475.2	1109	597	192	2	4.8	HOMOGENIZER
	HDG105	475.2	1109	597	192	2	4.8	HOMOGENIZER

Where: S.P: "super plasticizer", N.S: "nano silica".

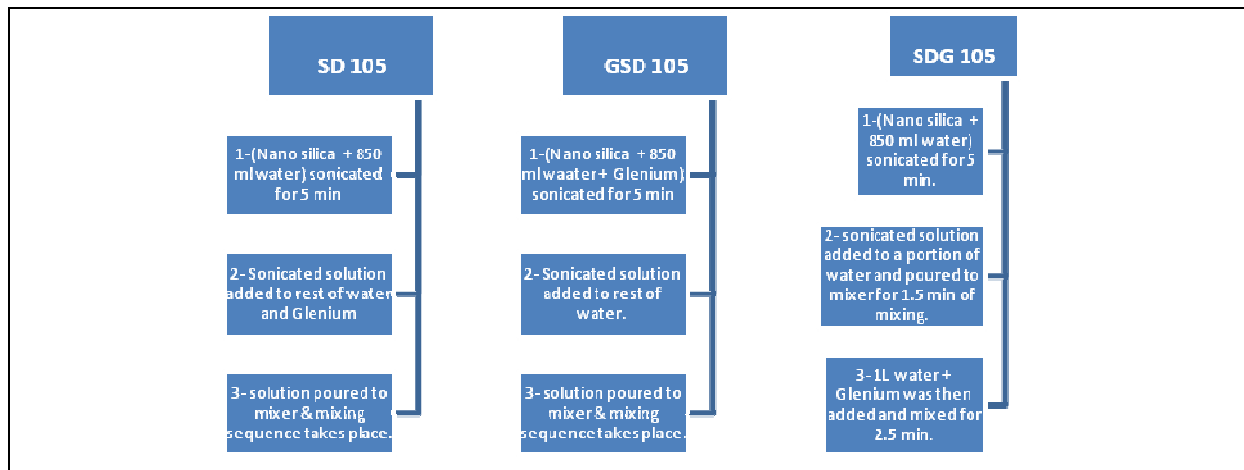


Figure 4: schematic diagram showing differences between mixing sequences.

### 3. Results and Discussion

#### 3.1. Effect of NS hardened concrete

##### 3.1.2. Optimization of the as received nano silica percentages on the compressive strength gain.

- The mechanical strength of tested mixes increased with increasing the amount of the silica nano particles up to 2%, and then the compressive strength leveled up at 7.3 % gain. This slight increase can be attributed to the clear agglomeration in the –as received – NS which is clear in TEM, and XRD photos shown previously (Fig1)

- For the contents higher than 1.5%, the nano particles cannot easily disperse within the cement matrix, and due to their high surface energy, they become agglomerated, hence a weak area of empty spaces such as voids appeared. Consequently, the structure formed in such conditions cannot be homogenous and compacted (M.R. Arefi et al. 2011).
- From the tested samples, the strength of early specimens (7 days) is dependent on the concentration of CSH, produced from C3S, C2S, and CH (pozzolanically transformed). The contributions of early strength for all three mixes can be attributed to the cementitious and pozzolanic reaction. When agglomeration exists, high surface interaction occurs, and consequently the available  $\text{SiO}_2$  free ions decreases, and so the early compressive strength decreases which can also be attributed to the water that was intended for hydration was hindered by the agglomerated particles.
- While in the early stages, larger and /or agglomerated particles in the mix that had not completely dissolved in solution will reduce porosity in the pores/capillaries of the CSH gel by packing into some of these voids, in doing so, the density of the CSH will slightly increase in the plastic form and convert into a denser compressive bearing structure after hydration completes by the 28<sup>th</sup> day (Jonathan S. Belkowitz1 and Dr Daniel Armentrout. 2010), that explains why increasing agglomerated nano silica particles decreases early strength, while slightly increasing late strength.

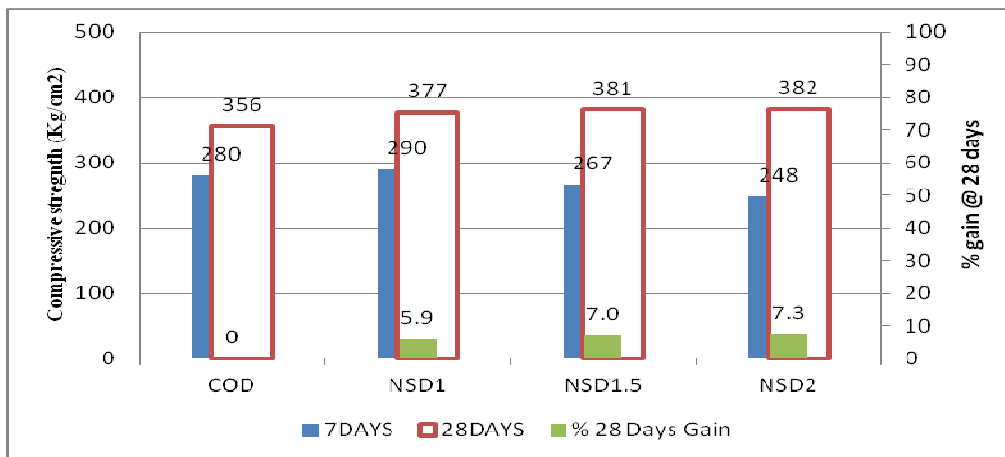


Figure 5: Compressive strength of - as received- NS Concrete mixes.

### 3.1.3. Effect of de-agglomeration methods on NS concrete:

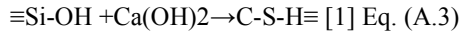
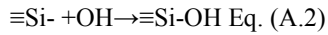
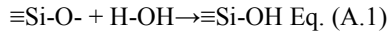
Proposed de-agglomeration methods were applied on NS concrete mixes, with and without adding superplasticizer to the mixes, to avoid interference effect on results.

#### 3.1.3.1. Results of super plasticized NS concrete mixtures:

##### 3.1.3.1.1. Effect of de-agglomeration methods on NS concrete compressive strength:

As it can be seen from figure 6, mix SD105 where nano silica was sonicated for five minutes before being mixed showed the most significant gain in strength in both early (15%), and late strengths (23%) this can be attributed to the previously mentioned effect of sonication in de-agglomerating, and dispersing of nano particles where higher number of surface atoms in the nano particles translates into a larger number of free and unsaturated atomic bonds on the surface of the nano particles, which makes them thermodynamically unstable and leads to an increase in the appropriate surface area for chemical reactions ( Zhang et al. 2002). The presence of higher number of unsaturated atomic bonds of  $\equiv\text{Si}-$  and  $\equiv\text{Si}-\text{O}-$  on the surface of the nano particles, resulting the silica nano particles react strongly

with Ca(OH)<sub>2</sub> in the following way:



- Also nano silica can produce more nucleation sites for the hydration products, and has higher pozzolanic activity that results in increasing the early strength as well as the late strength,
- As for mix HD105 the homogenizer helped increasing the late strength by 15%, but without any significant effect on the early strength, this can be attributed to that the homogenizer helped decreasing the agglomeration but at the same time it influenced the particle size distribution changing it to a narrower distribution. A well graded mixture of sizes can mix throughout covering more surface area of cement paste than a narrow distribution of sizes (Jonathan S. Belkowitz<sup>1</sup> and Dr Daniel Armentrout. 2010), the better distribution helped in a better packing density and so a higher late strength too, and this explains why mix SD105 had higher late strength than HD105.
- For mix PD1 a slight increase in late strength was reported without any increase in early strength this is due to agglomeration as it was discussed in step one results,
- while for mix TD105 the results showed a significant loss of about (-13%) in strength at 28 days, that can be attributed to the increase in agglomeration caused by the low stirring speed, the agglomeration of the nano silica might have resulted in decreased nucleation effect, and also in increasing the nano silica size, and as mentioned by (Jonathan S. Belkowitz<sup>1</sup> and Dr Daniel Armentrout. 2010), When the silica diameter increases, the rate of early pozzolanic reaction decreases, this strength loss was also mentioned by (Tobon et al. 2010).

### 3.1.3.1.2. Effect of de-agglomeration methods on elastic modulus:

The above discussion can be confirmed by the results of table 5, where the coefficient of variation of the sonicated mixture is the lowest of the mixes which indicates the better dispersion of the nano particle in the cement matrix, and its homogeneity and so was the results for the elastic modulus that showed an increase for the sonicated mix of about (7%) from the control mix, and about (11%) from the homogenized mix, such results indicates the better compaction, and denser cement matrix for the sonicated mix, the elastic modulus result will be confirmed later in the SEM results section of the discussion.

Table 5: Coefficient of variation and modulus of elasticity results for step two mixtures with super plasticizer.

Mixture	COD	TD105	PD105	HD105	SD105
C.O.V.	4.5	12.5	8.5	7	4
E (Kg/cm <sup>2</sup> )	289200	190596	200896	277101	309091



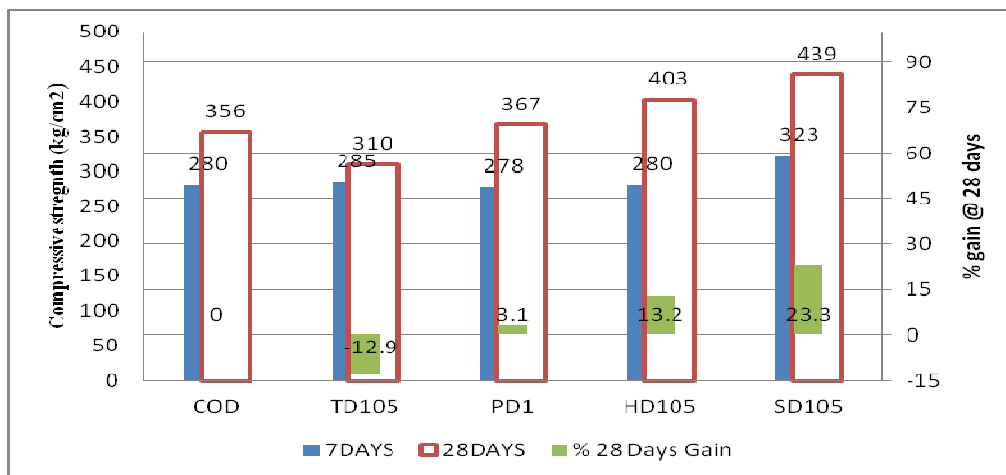


Figure 6: Effect of de-agglomeration methods on compressive strength of NS mixes with superplasticizer.

### 3.1.3.2. Results of NS concrete mixtures without super plasticizer:

#### 3.1.3.2.1 Effect of de-agglomeration method on compressive strength, and modulus of elasticity of concrete:

As it can be seen from figure 7, and table 6, the mixtures made without adding super plasticizer behaved in the same trend for the three examined means of de-agglomeration, the sonicated mix SCD105 showed the best results for both early (16%) and late (15%) strengths as well as having the lowest C.O.V. result and the highest elastic modulus with an increase of (5%) than the control mix, and (11%) than the homogenized mix. As for the homogenized mix HCD105 the results showed high late strength (11%) without any change on the early strength results, and finally for the stirred mix TCD105 a loss of about (-8%) in strength was observed. The results of those mixtures helped confirming the above mentioned results of the super plasticized mixtures.

Table 6: Coefficient of variation and modulus of elasticity results for step two mixtures without superplasticizer.

Mixture	COCD	HCD105	SCD105
C.O.V.	2	4	2
E (Kg/cm <sup>2</sup> )	204214	192753	214834



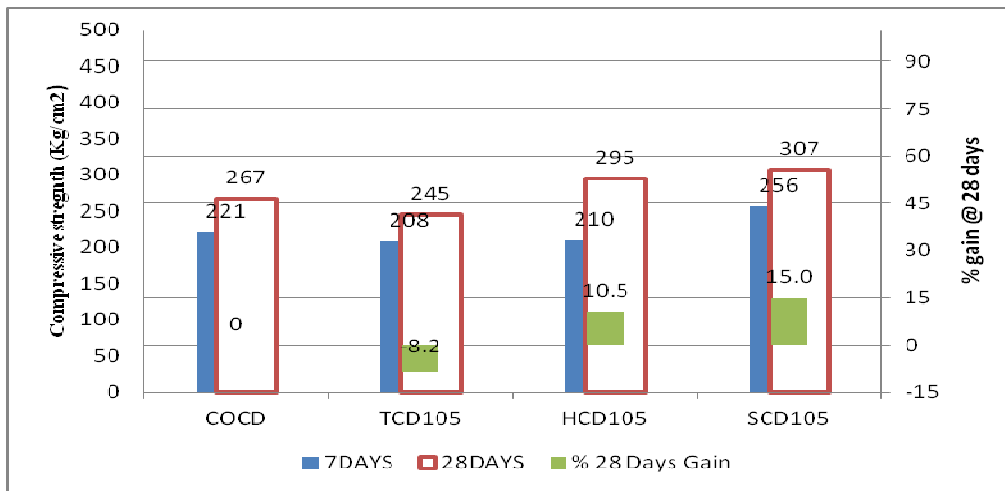


Figure 7: Effect of de-agglomeration methods on compressive strength of NS mixes without superplasticizer.

#### 3.1.4. Results of application of super plasticizer using different timing on the compressive strength gain:

The results shown in figure 8 and table 7 indicate that the timing of adding the super plasticizer influenced the compressive strength results as well as the modulus of elasticity significantly.

- For mix GSD105 where the Glenium was added to the nano silica before being sonicated the compressive strength showed the least gain in strength after 28 days of curing (15%), while no gain in strength was reported for the early strength results, this can be attributed to two reasons; the first is that the Glenium covered the agglomerated nano particles which in turn difficult the de-agglomeration of the nano silica at the proposed sonication time (5 min), and so not all of the aggregated silica got well dispersed, while the other is by covering the agglomerated nano silica particles the Glenium got attached to the silica particles losing its chance of being well dispersed and as a result it could not reach all of the cement particles. This scenario will be confirmed later by the slump results where a loss in slump will be indicated for such mix.

- While authors as (Qing Y et al. 2008) reached a gain in strength of 26% using 10% nano silica addition, mixes SD105, and SDG105 showed with only 1% nano silica a significant gain in both early (approx. 15%) and late strengths (26%), as for the coefficient of variation results the SDG105 mix showed a very significant result that indicates how that method helped in a better use of the plasticizer in dispersing the nano silica particles, and so was the elastic modulus results which showed how such a method helped in making a great use of the plasticizing effect of the Glenium by reaching high strength without losing ductility.

The results in figure 9 and table 8 showed the same trend for both the compressive strength, and the elastic modulus results of homogenized mixes GHD105, HD105, and HDG105, while it showed no significance on the C.O.V. results.

Table 7: Coefficient of variation, modulus of elasticity, and slump results for the sonicated mixtures of step three.

Mixture	COD	GSD105	SD105	SDG105
C.O.V.	4.5	10.5	4	1
E (Kg/cm <sup>2</sup> )	289200	266983	309091	230459
Slump (cm)	14	9	17	19

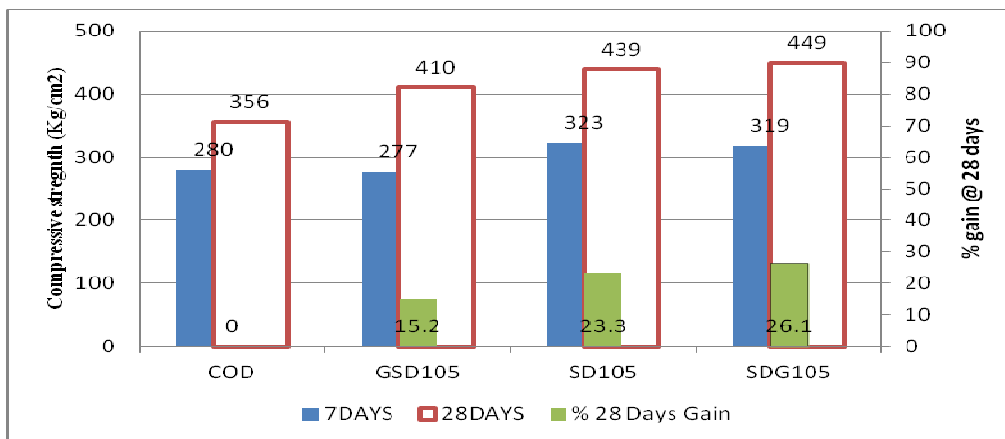


Figure 8: Compressive strength of sonicated NS concrete, using different timings of application of SP.

Table 8: Coefficient of variation, modulus of elasticity, and slump results for homogenized vs. sonicated mixture of step three.

Mixture	COD	SDG105	HDG105
C.O.V.	4.5	1	7
E (Kg/cm <sup>2</sup> )	289200	230459	208695
Slump (cm)	14	19	9

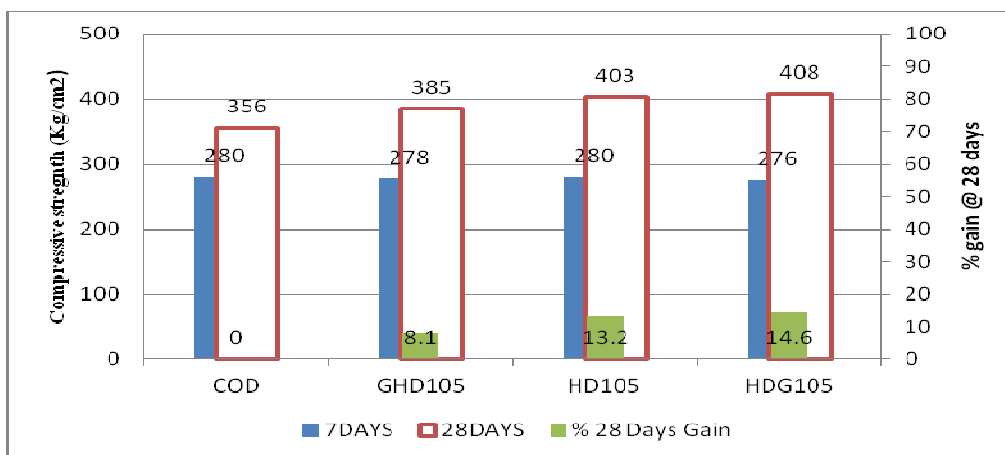


Figure 9: Compressive strength of homogenized NS concrete, using different timings of application of SP.

### 3.2. Effect on NS fresh concrete

#### 3.2.1. Effect of NS agglomeration on slump results:

In order to study the effect of agglomeration on the workability, a comparison was held between the three proposed de-agglomeration methods, at first for mixes without super plasticizer to avoid interfering effect of the Glenium, results in figure 10 showed that as the nano particles get well dispersed the workability increases, this can be attributed to the fact that ultra fine particles have a higher workability as a consequence of high free water among the particles where the rolling effect can be observed. However, the use of densified silica particles has a negative effect on the workability of fresh mixes, where this rolling effect disappears due to the presence of large irregular agglomerates. The earlier mentioned rolling effect is similar to the effect of fly ash (E.B. Nelson et al. 2006). It improves or facilitates the flowing of the cement paste, resulting in a lower water demand to obtain the same slump (G. Quercia et al. 2012), this conclusion was also mentioned by (Erich D. Rodriguez et al. 2012).

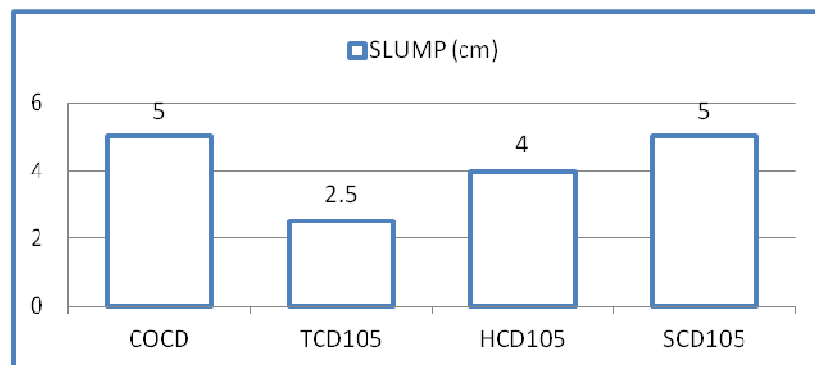


Figure 10: Effect of NS de-agglomeration methods on slump of nano silica concrete without superplasticizers.

#### 3.2.2. Effect of super plasticizer addition timing on slump results:

Results presented in figure 11 showed that the separation between the addition of nano silica and the addition of the super plasticizer into the mixer enhanced not only the dispersion of the nano silica and consequently the strength, but also the C.O.V. results. And the workability of mix SDG105 Since better dispersion helps in a better particle size distribution (PSD) and so better compaction of mix and as it was concluded by (H.J.H.Brouwers and Radix. 2005) in a previous work that it could be expected that if one would optimize the PSD down in the nanometer range, the workability and stability could be maintained while further reducing the necessary super plasticizer content, on the other hand agglomerated particles has less PSD and so less compaction and more voids that need more water to fill (G. Quercia et al. 2012), that can be seen for mix GSD105 where the 5mins sonication was not enough for dispersing and de-agglomerating the nano silica particles, the slump was affected by the traces of agglomeration that still exists and more water was to be added if reaching the same slump result as mixes SD105, and SDG105 was targeted.

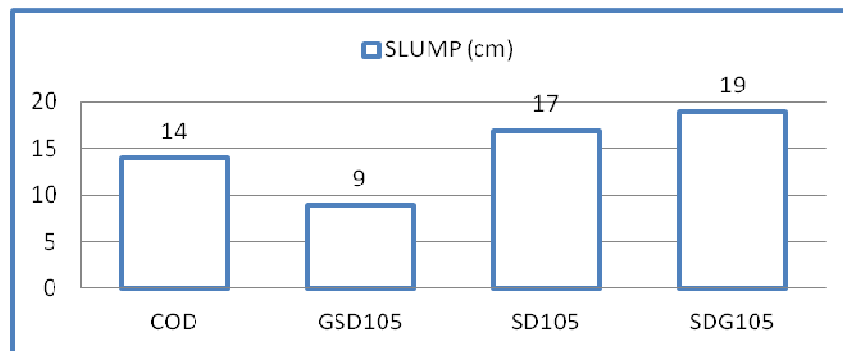


Figure 11: Effect of super plasticizer addition timing on slump results of NS fresh concrete.

### 3.3. Scanning electron microscope results:

As it can be concluded from figure 12, the control mix COD has a weaker structure with large number of voids and the C-S-H looks porous and is littered with CH crystals. Compared to the sonicated nano silica mix SDG105 that showed a denser structure of C-S-H that is well organized, while for the PD1 mix the plate showed the un-reacted nano silica particles due to agglomeration surrounded by the CH particles.

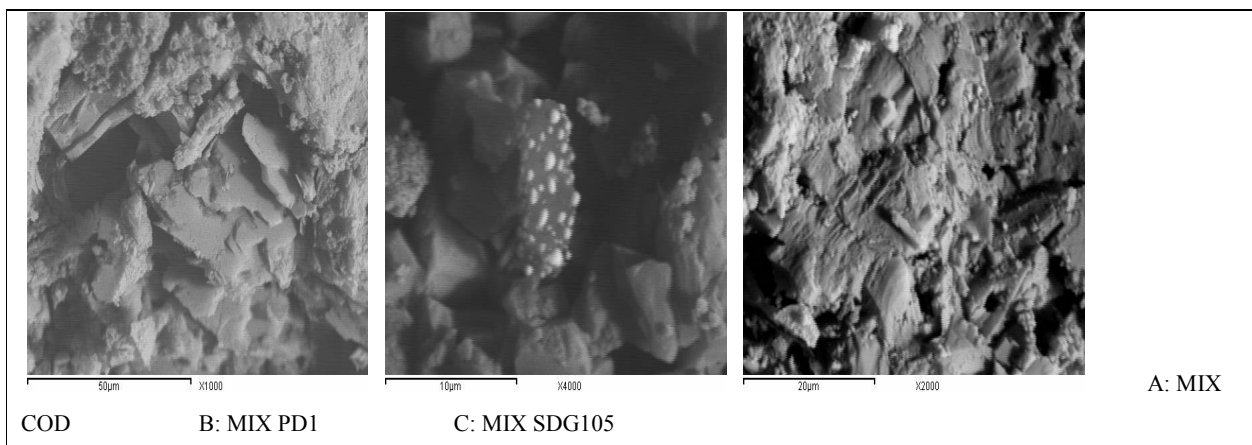


Figure 12: SEM plates for a: mix COD, b: mix PD1, and c: mix SDG105.

## 4. Conclusion

From the presented research the following can be concluded:

- The method of utilization of nano silica with or without super-plasticizers in concrete mixes needs special attention, as it can cause a wide span of variation in resulting fresh/ and hardened concrete characteristics.
- The use of nano silica particles as received from the manufacturer will result in unpredictable, and varying compressive strength values ranging from loss to gain in strength depending on its level of agglomeration.
- The use of 5 min sonication showed a significant effect on de-agglomerating nano silica particles when compared with other methods.
- 5 min sonication of 1% on nano silica particles before being added to concrete mix enhanced the compressive strength gain by 23% compared to control sample, this value was reached by much higher percent (reaching 10%) of NS in previous literature without applying sonication.

- The delayed addition of the super plasticizer to sonicated nano silica concrete increased the compressive strength to 26% higher than the control mix.
- The de-agglomerated nano silica addition showed a denser, well organized C-S-H structure.
- Finally, better dispersion, and de-agglomeration of nano silica particles increased concrete workability significantly (about 35%) as compared with other methods of de-agglomeration studied.

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### References

- A. Nazari, S. Riahi, (2011), The effects of SiO<sub>2</sub> nano particles on physical and mechanical properties of high strength compacting concrete. *Composites, Part B*, 42, 570–578.
- D. Martinez, J. Paya, J.M. Monzo, M.V. Borrachero, (2008) Granulometric activation of densified silica fume (CSF) by sonication, *Adv. Cem. Res.* 20 (3) 129–135.
- D. Martinez, J. Paya, J.M. Monzo, M.V. Borrachero, (2011), Effect of sonication on the reactivity of silica fume in portland cement mortars, *Adv. Cem. Res.* 23 (1) 23–31.
- Erich D. Rodriguez, Lourdes Soriano, Jordi Paya, Marya Victoria Borrachero, José M. Monzo, (2012), Increase of the reactivity of densified silica fume by sonication treatment, *Ultrasonics Sonochemistry*, 19, 1099–1107.
- E.B. Nelson, D. Guillot, (2006), Cement additives and mechanism of action, Chapter 3, *Well Cementing*, second editions, Schlumberger ltd., Sugar Land, Texas, U.S.A. pp. 71–80.
- F. Sanchez, K. Sobolev, (2010), Nanotechnology in concrete – A review, *Construction and Building Materials*, 24, 2060–2071.
- G. Quercia, G. Hüsken b, H.J.H. Brouwers, (2012), Water demand of amorphous nano silica and its impact on the workability of cement paste, *Cement and Concrete Research* 42 344–357.
- H.J.H. Brouwers, H.J. Radix, (2005) Self-compacting concrete: theoretical and experimental study, *Cem. Concr. Res.* 35 2116–2136.
- Ji T. (2005), Preliminary study on the water permeability and microstructure of concrete incorporating nano-SiO<sub>2</sub>. *Cem Concr Res*, 35(10), 1943–7.
- Jonathan S. Belkowitz1 and Dr Daniel Armentrout, (2010), an investigation of nano silica in the cement hydration process, Concrete Sustainability Conference, *National Ready Mixed Concrete Association*, Denver.
- Jo BW, Kim CH, Tae G, Park JB. (2007) Characteristics of cement mortar with nano- SiO<sub>2</sub> particles. *Constr Build Mater*, 21(6):1351–5.
- Li H, Xiao H-g, Yuan J, Ou J. (2004), Microstructure of cement mortar with nano particles. *Compos B Eng*, 35(2):185–9.
- Luciano Senff, Joao A. Labrincha, Victor M. Ferreira, Dachamir Hotza, Wellington L. Repette, (2009), Effect of nano-silica on rheology and fresh properties of cement pastes and mortars, *Construction and Building Materials*, 23, 2487–2491.
- Li H, Zhang M, Ou J. (2006) Abrasion resistance of concrete containing nano-particles for pavement. *Wear*, 260(11–12):1262–6.
- Li G, (2004) Properties of high-volume fly ash concrete incorporating nano-SiO<sub>2</sub>. *Cement Concr Res*, 34(6):1043–9.
- Laila Raki, James Beaudoin, Rouhollah Alizadeh, Jon Makar and Taijiro Sato, (2010) Cement and Concrete Nanoscience and Nanotechnology, *Materials*, 3, 918-942; doi:10.3390/ma3020918.
- M. Sari, E. Prat and J. Labastire. (1999), High strength self-compacting concrete original solutions associating organic and inorganic admixtures, *Cement and Concrete*, 29, 813-818.
- M.R. Arefi, M.R. Javaheri, E. Mollaahmadi, H. Zare, B. Abdollahi Nejand, M. Eskandari, (2011), Silica nanoparticle size effect on mechanical properties and microstructure of cement mortar, *Journal of American Science*, 7(10), 231-238.

- Neville AM. Properties of concrete. 4th ed. England: ELBS with Addison Wesley Longman; 1996.
- Peter C. Older I. Lea's chemistry of cement and concrete. 4th ed. London: Arnold; 1998.
- Park CK, Nohb MH, Park TH. (2005), Rheological properties of cementitious materials containing mineral admixtures. *Cement Concr Res*, 35(5):842–9.
- Qing Y, Zenan Z, Li S, Rongshen C. (2008), A comparative study on the pozzolanic activity between nano-SiO<sub>2</sub> and silica fume. *J Wuhan Univ Technol – Mater Sci*, Ed;21(3), 153–7.
- Qing Y, Zenan Z, Deyu K, Rongshen C. (2007) Influence of nano-SiO<sub>2</sub> addition on properties of hardened cement paste as compared with silica fume. *Constr. Build Mater*,21(3):539–45.
- Sobolev K, Flores I, Torres-Martinez LM, Valdez PL, Zarazua E, Cuellar EL. (2009) Engineering of SiO<sub>2</sub> nano particles for optimal performance in nano cement based materials. In: Bittnar Z, Bartos PJM, Nemecek J, Smilauer V, Zeman J, editors. *Nanotechnology in construction: proceedings of the NICOM3 (3<sup>rd</sup> international symposium on nanotechnology in construction)*. Prague, Czech Republic;. p. 139–48.
- Sobolev K, Ferrada-Gutiérrez M. (2005) How nanotechnology can change the concrete world: Part 1. *Am Ceram Soc Bull*, 84(10):14–7.
- Ji T. (2005) Preliminary study on the water permeability and microstructure of concrete incorporating nano-SiO<sub>2</sub>. *Cem Concr Res*,35(10):1943–7.
- Tobon, J. I; Restrepo, O. J; Paya, J. (2010), Comparative analysis of performance of Portland cement blended with nanosilica and silica fume, *DYNA*, 163, 37-46.
- Zhang YL, Li CD. (2002), Nano-structured technology and nano-structured plastics. Beijing, China: *China Light Industry Press*; [in Chinese]; pp 8–15, 386–9.

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