# Pozzolanic Activity and Compressive Strength of Concrete Incorporated nano/micro Silica

Mohammed S. Nasr<sup>(D)</sup> Building and Construction Department, University of Technology/ Baghdad. Email:msn\_alamar@yahoo.com Dr. Shakir A. Salih<sup>(D)</sup> Building and Construction Department, University of Technology/ Baghdad. Dr. Maan S. Hassan<sup>(D)</sup> Building and Construction Department, University of Technology/ Baghdad.

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# ABSTRACT

This paper aims to give a recommendation about the suitable nano silica proportion and curing method for testing its strength pozzolonic activity, in addition to suggest a mixing or batching procedure of this material with other concrete ingredients. The influence of nano and micro silica on concrete compressive strength is also studied. Three proportions of nano silica (0.5, 5, &10%), two curing methods (accelerated in oven and normal in water) and two tested ages are used in pozzolonic activity test of nano silica. For compressive strength test, eleven mixes are considered: without-adding (two mixes), three micro silica (M) mixes with replacement ratios of 5, 10 & 15%, four nano silica (N) mixes with replacement ratios of 0.5, 1.5, 3 & 5%, and three micro plus nano silica mixes (0.5N+9.5M, 1.5N+8.5M and 3N+7M%) are adopted. Results of pozzolanic activity test denoted that nano silica has remarkable pozzolanic activity for all tested replacements. However, it is suggest using 5% nano silica for performing this test by using either accelerated or water curing. Additionally, it was found that nano silica had more impact on compressive strength of concrete than micro silica for all tested ages. Where, maximum enhancement ratios of compressive strength were about 33% and 27% for 3% nano silica mix at 7 and 90 days age respectively, and 22% for 5% nano silica mix at 28 days age. For micro silica mixes, higher improvement ratios were found in 15% micro silica mix: 2%,5% &7% at 7,28 &90 days age respectively.

Keywords: nano silica; micro silica; batching method; pozzolanic activity; compressive strength.

# الفعالية البوزولانية و مقاومة انضغاط الخرسانة الحاوية على نانو/مايكرو سليكا

الخلاصة

يهدف هذا البحث لإعطاء توصية حول نسبة النانو سليكا وطريقة الانصاج المناسبة لفحص قوة فعاليتها البوز ولانية، بالإضافة الى اقتراح طريقة لخلط او مزج هذه المادة مع مكونات الخرسانة الاخرى. كما تم دراسة تاثير تضمين النانو والمايكرو سليكا على مقاومة انضغاط الخرسانة. لفحص الفعالية البوزو لانية للنانو سليكا، تم اعتماد ثلاث نسب تعويض (٥.٠، ٥، ١٠%)، طريقتي انضاج ( معجل في الفرن واعتيادي في الماء)و عمري فحص. تم اعتماد احد عشر نوعا من الخلطات الخرسانية في فحص مقاومة الانضغاط، خلطتان بدون أي اضافة و ثلاث خلطات للمايكرو سليكا (M) بنسب تعويضية ٥، ١٠، ٥٠% و اربع خلطات للنانو سليكا (N) بنسب تعويضية م النوين م ١٠، ٥%، ٥%، وثلاث خلطات

2412-0758/University of Technology-Iraq, Baghdad, Iraq

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للنانو مع المايكرو سليكا ( 0.5N+9.5M، 0.5N+8.5M و 3N+7K %). اظهرت نتائج فحص الفعالية البوزو لانية بان النانو سليكا تمتلك فعالية بوزو لانية معتد بها لجميع النسب المفحوصة، مع ذلك، تم التوصية باعتماد نسبة ٥% نانو سليكا لتنفيذ هذا الفحص باستخدام اما انضاج معجل او بالماء. اضافة لذلك، لقد وجد بان النانو سليكا ذات تأثير كبير على مقاومة انضغاط الخرسانة مقارنة بالمايكرو سليكا ولجميع الاعمار المفحوصة. حيث ان اعلى نسب تحسين لمقاومة الانضغاط كانت تقريبا ٣٣% و ٢٧% لخلطة ٣% نانو سليكا في الاعمار ٧ و ٩٠ يوم على التوالي، و ٢٢% للخلطة ٥% نانو سليكا في عمر ٢٨ يوم. بالنسبة لخلطات المايكرو سليكا، فان اعلى نسبة تحسين وجدت في خلطة ٥٠% مايكرو سليكا: ٢%، ٥% و ٧% في الاعمار ٧، ٢٨ و ٩٠ يوم على التوالي.

# **INTRODUCTION**

In concrete, nano science concerns with describing and measuring of the micro and nano scale structure of cementitious materials to assess their influence on concrete macro-structure properties using special techniques. The process of including of nano-sized tubes or particles in concrete and studying its behavior and properties, or transplantation upon cement particles, nano-additives, aggregate and cement phase to supply surface implementation, can be called nano-engineering of concrete [1]. One of the widely used nano materials in concrete is nano silica [7, r,  $\epsilon$ ,  $\bullet$ ]. Nano silica is classified by many researches as pozzolanic material. Based on results obtained by previous study [1], nano silica had an effective pozzolanic activity more than that in silica fume. Also it was found in the same study, that, at early age, calcium hydroxide crystals at interface between aggregate and cement paste were absorbed significantly in high performance concrete after including of nano silica. Lazaro et al [V] investigated the pozzolanic activity of synthetic-olivine nano silica with surface area of 100 – 400 m<sup>2</sup>/g. A 7% replacement of cement with olivine nano silica was investigated according to CEN EN 196-1 standard. Results showed that nano silica had pozzolanic activity of 101% at 7 days age compared with control specimens.

**Jalal et al [8]** examined the effect of nano and micro silica in high performance selfconsolidating concrete (HPSCC). The studied properties were rheological, mechanical, durability and microstructure. Three cement contents(400, 450 and 500 kg/m<sup>3</sup>), 10% micro silica, 2% nano silica and (10%+2%) micro and nano silica were used. Water/binder ratio was fixed at 0.38. Results revealed that nano and micro silica blended mixtures increased the resistivity and reduced chloride ions percentage of HPSCC. Water and capillary absorption were also reduced in blended micro and nano materials especially at high binder content (500 kg/m<sup>3</sup>). Improving of consistence and reduction the probability of bleeding and segregation were showed by rheological test. Compressive and tensile strength were improved. In case of microstructure, more packing and refinement were noticed.

**Abyaneh et al [3]** studied the synergic effect of nano and micro silica on concrete water absorption, electrical resistivity and compressive strength. The used proportions were 2% for the former and 10% for the latter as partial replacement of cement weight. Plain and 10% micro silica concrete mixes were also made for comparison. It was found that mix including (2%+10%) nano and micro silica, respectively, had higher electrical resistance and lower water absorption when compared with other mixes. Similar results were noticed for compressive strength test at the researched ages: 7, 14 and 28 days.

Because of there is no standardized procedure till now for testing pozzolanic strength activity of nano silica, and due to its growing using in concrete building materials, this study is performed to impart suggestions about mixing or batching procedure of powdered nano silica with other concrete ingredients, in addition to give a suitable proportion and curing method for testing the strength pozzolznic activity of this material. The research is aimed also to make a comparison between nano and silica addition on compressive strength property of concrete.

# Experimental work Materials

# • Cement

Commercially available ordinary Portland cement (Type I), named Karasta, is used in this study. Chemical and physical properties of cement are shown in Tables 1 and 2. They are conformed to Iraqi specifications (I.Q.S) No. 5/1984 [9].

Table (1) Chemical composition of cement.						
Oxides Composition	Content, %	Iraqi specifications (No.5/1984), %				
CaO	63.5	-				
$SiO_2$	21.9	-				
$Al_2O_3$	4.3	-				
Fe <sub>2</sub> O <sub>3</sub>	4.5	-				
MgO	3.8	<5				
$SO_3$	2.1	<2.5				
Free lime	0.8	-				
L.O.I.	3.4	<4				
L.S.F.	0.88	0.66-1.02				
Insoluble residue	1.2	<1.5				

Table (1):- Chemical composition of cement.

## • Fine aggregate

Natural sand brought from Al-Amara city is used in this work. The fine aggregate is conformed to Iraqi specifications No. 45/1984, zone two [10], as shown in Table 3.

# • Coarse aggregate

Crushed gravel with size range (5-19 mm), see Table 4, and compatible with Iraqi specifications No. 45/1984 **[10]** is used as coarse aggregate.

rubic (2). Thysical properties of comente							
Physical properties	Results	Iraqi specifications (No.5/1984)					
Setting time (Vicat apparatus), min							
Initial setting	105	≥45					
Final setting	150	≤600					
Compressive strength, MPa							
3 days	22.1	≥15					
7 days	30.8	≥23					

Table (2):- Physical properties of cement.

Sieve opening (mm)	Accumulative passing,	Accumulative passing according to				
Sieve opening (inin)	%	Iraqi specifications (No.45/1984), %				
10	100	100				
4.75	98.4	90 - 100				
2.36	87.95	75 - 100				
1.18	73.6	55 - 90				
0.60	39.8	35 - 59				
0.3	10.71	8-30				
0.15	0.7	0-10				
0.075	5	≤ 5				
Sulfate content						

Table (3):- Sieve analysis and sulfate content of sand.

Property	Result	Iraqi specifications (No.45/1984)			
SO <sub>3</sub> , %	0.37	$\leq 0.5$			
Table (	4):- Sieve analysis and s	ulfate content of gravel.			
Siava ananing (mm)	Accumulative	Accumulative passing according to			
Sieve opening (mm)	passing, %	Iraqi specifications (No.45/1984), %			
37.5	100	100			
20	95	95-100			
10	36	30-60			
5	3	0-10			
0.075	0.09	$\leq 3$			
Sulfate content					
Property	Result	Iraqi specifications (No.45/1984)			
SO <sub>3</sub> , %	0.069	≤ 0.1			

#### Micro silica (M) ٠

Micro silica purchased from BASF Company is used as pozzolanic admixture. Physical and chemical specifications of micro silica are shown in Table 5. These properties are provided by the manufacture, except of activity index property, which was determined experimentally in Babylon Technical Institute Labs.

Tuble (c). Specifications of milero sinca					
Property	Value or description				
Material structure	Densified micro silica				
Color	Gray				
Specific gravity	$2300 \text{ kg/m}^3$				
Fineness (blain)	$> 15000 \text{ m}^2/\text{kg}$				
SiO <sub>2</sub>	> 85 %				
CaO	< 1 %				
Chloride content	< 0.1 %				
SO <sub>3</sub>	< 2 %				
Activity index	> 105 %				

# Table (5):- Specifications of micro silica

### Nano silica (N)

Nano silica bought from Evonik Company; Turkey, is used as concrete admixture in this research, see Figure 1. It's called commercially as Aerosil 150. The number 150 refers to specific area of the product, as shown Table 6 which includes as-received properties of nano silica.

Table (6):- Specifications of nano silica				
Property	Value or description			
Specific surface area (BET)	$150 \pm 5 \text{ m}^2/\text{g}$			
Color	white			
Primary particle size	14 nm			
SiO <sub>2</sub> content	99.8 %			
Loss on ignition	$\leq 1 \%$			
Moisture (after 2 hour at 150°C)	$\leq 1.5 \%$			
pH (in 4% dispersion)	3.7-4.7			

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Bulk density (approximate value)	30 g/l
Tapped density (approximate value)	50 g/l



Figure (1):- Nano silica used

#### • Chemical admixture (superplasticizer)

Glenium 54 (G54) high range water reducing admixture, purchased form BASF Company, is used as workability adjusting material for concrete mixtures.

#### • Water

Tap water is used for mixing and curing of all concrete mixes and specimens.

#### **Batching of nano silica:**

Due to the high surface area  $(150 \text{ m}^2/\text{g})$  of powder nano silica, which involves special treatment, it is important to look for appropriate way to batch or mix it with other concrete constituents. Many batching (or mixing) ways are noticed in the literature for powder type nano silica, one of these methods [8] included direct adding of nano silica with other concrete materials, while the other, [11, 11] embraced mixing it with a part of mixing water (and/or superplasticizer) for a limited period then the blend is added to the remaining materials (binder, sand and gravel); and another method [11] comprised mixing of nano silica with mixing water then dispersing and deagglomeration of nano silica particles in water by ultrasonic technique. To obtain a suitable mixing or batching procedure for the available nano silica with else concrete ingredients, taking into account the information provided by the literature, trials are made and as follows:

Trial one : in which, it is planned to mix nano silica with water in a ratio of 15% (nano silica to the total suspension) by weight, then dispersing the nano silica in water by ultrasonic method. However, after mixing with water (before sonication), the blend is formed big conglomeration (about 5 cm size) as shown in Figure 2-left. In other word, the mixture did not behave as a liquid. That exhibition may be contributed to the large water demand needed for the high surface area of nano silica to be wet. It is important to note that the agglomeration may depend on particle surface properties, for example: whether it absorbs water or not. Where, it is thought that this effect is reduced for impermeable surfaces. At last, the mixture, and subsequently the mixing procedure, is discarded before placing it in ultrasonic device.

Trial two (depended one): in which, separately dry patching of nano silica and cement (before mixing with other constituents) is considered. Hand-held mixer, as shown in Figure 2-right, is used

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for mixing. Many mixing speeds and periods are tried to reach a most suitable one. Finally, it is found that mixing speed of about 480 rpm and time of mixing of 10 minutes are appropriate parameters to get acceptable mixture of powder nano silica and cement using the above mixer.

For unifying purposes, the same mixing procedure (but with different mixing time) and mixer device are depended for micro silica, where, it is also mixed separately with cement. Due to the lower surface area of micro silica in comparison with nano silica, the former is mixed with cement for 2 minutes with the same mixing speed (480 rpm) used in the latter. In case of micro plus nano silica mixtures, the process included mixing of cement and micro silica firstly for 2 minutes, then the nano silica is added to the blend (cement + micro silica) and additional 10 minutes of mixing is performed for the whole mixture (cement + micro silica + nano silica). Thus, the total mixing time of 12 minutes is applied for this case.

In summary, the depended powdered mixing times of cement with micro and/or nano silica are:

- Cement plus micro silica: 2 minutes.
- Cement plus nano silica: 10 minutes.
- Cement plus (nano silica + micro silica): 12 minutes.

After that, the blended binder materials are mixed with the other concrete ingredients as the mixing steps that described in the next sections.





### **Concrete mixes**

Eleven types of concrete mixes (twelve mixes in total) are depended. Without adding (Nil)- type (two mixes) is used as control. Other mixes types are contained admixtures (micro and nano silica). Micro silica (M) admixture is used in three proportions (mixes): 5, 10, 15% as a partial replacement of cement weight, while, nano silica (N) is used in four proportions (mixes): 0.5, 1.5, 3, 5% as a partial replacement of cement weight. Another three mixes containing micro plus nano silica (0.5N+9.5M, 1.5N+8.5M, 3N+7M) as a partial replacement of cement weight are also made.

Several trial mixes included different amounts of superplasticizer (Glenium 54) are executed to make mixes slumps within the range of 14- 17 cm. Procedure provided by ASTM C143 [11] is adopted for test the slump. So, the fixed parameters for all mixes are: water/cement, slump, coarse and fine aggregate fractions, and binder (cement, cement and micro silica and/or nano silica) content. Table 7 shows mixes details and symbols.

#### **Concrete mixing**

Mechanical mixing, using  $0.1 \text{ m}^3$  electrical mixer is used for all concrete mixtures. The general procedure of mixing is adapted from ASTM C192 [19] with some variations. Mixing steps can be summarized as follow:

- Mixing water is divided to two parts, the major one (about 75%) is mixed separately with the whole amount of super plasticizer (G54) for nearly 1 minute;

- Before mixer operating, coarse aggregate with a part of mixing water (about 25%) is added and the mixer is operated for few revolutions ( $\leq 0.5$  minute) then it is stopped;

- After that, binder materials (cement, cement and micro silica and/or nano silica which mixed together previously); fine aggregate and the major part of mixing water plus G54 are fed to the mixer then it is operated for 3 minutes;

- Thereafter, the mixer is rested for 1 minute;

- Finally, concrete ingredients are mixed for additional 2 minutes.

So, the total mixing time is about 5.5 minutes, excluding rest time.

Mix symbol	Cement, kg/m <sup>3</sup>	Sand, kg/m <sup>3</sup>	Gravel, kg/m <sup>3</sup>	w/b	Super- plasticizer, kg/m <sup>3</sup>	Micro silica, kg/m <sup>3</sup> (rep. %)	Nano silica, kg/m <sup>3</sup> (rep. %)
Nil	400	725	1050	0.45	1.859		
5M	380	725	1050	0.45	2.118	20 (5%)	
10M	360	725	1050	0.45	2.518	40 (10%)	
15M	340	725	1050	0.45	3.2	60 (15%)	
0.5N	398	725	1050	0.45	2.8		2 (0.5%)
1.5N	394	725	1050	0.45	3.718		6 (1.5%)
3N	388	725	1050	0.45	5.318		12 (3%)
5N	380	725	1050	0.45	9.718		20 (5%)
0.5N+9.5 M	360	725	1050	0.45	4.259	38 (9.5%)	2 (0.5%)
1.5N+8.5 M	360	725	1050	0.45	5.859	34 (8.5%)	6 (1.5%)
3N+7M	360	725	1050	0.45	7.059	28 (7%)	12 (3%)

Table (7):- Mixes symbols and proportions (according to ACI 211.1 [16]).

# Tests

#### **Compressive strength**

This test is carried out according to BS 1881-part 116 [17]. A number of 108 concrete cubes with 150 mm size are cast to observe the compressive strength development of concrete mixes with time. The studied ages are: 7, 28 and 90 days. Three cubes are made for each mix at the specified age. After de-molding, the specimens are immersed in tap water until testing age.

#### Pozzolanic activity index of nano silica

Because of there is no standardized procedure till now for testing pozzolanic strength activity of nano silica, and due to its growing using in concrete building materials, a small program is performed to test it as shown in Figure 3. Mortar cubes of 50 mm side length are used for this test. Three proportions: 0.5, 5 and 10 %, as replacement of cement weight, are depended for this test. The last ratio is as used in ASTM C 1240 [18] for testing strength activity index of silica fume. While, the first two proportions represent the minimum and maximum proportions that used in

concrete mixes of this work. It is believed that, due to its high surface area, small proportions may have the benefit in pozzolanic activity test of nano silica.

Two methods of curing are done: the first is normal curing of mortar cubes in tap water for 6 and 27 days after de-molding, and accelerated curing at 65 °C for 6 days as in the previously denoted standard for silica fume, is used in the second method. Grading of the used sand is conformed to BS 4550-part 6 [19] which also depended for cement strength test in Iraqi specifications. Mortar mixes details for nano silica pozzolanic activity investigation, for six cubes, are shown in Table 8. Mixing of mortar is made manually as the following procedure:

- The total amount of mixing water is mixed-up with superplasticizer (G54) independently;

- Cement, nano silica (previously batched as mentioned in the above sections) and sand are blended in dry state for 1 minute;

- Then, mixing water (and G54) is added to the mixture and mixed for 1 minute;
- Mixing process is stopped for 30 second; and
- The whole blend is finally mixed for 1 minute.

ASTM C 1437 [20] procedure is used for measuring mortars flow. Mortars flow is specified to the range (153- 161 mm) as shown in Table 8. Tamping of mortar cubes is made using tamping rod as described in ASTM C109 [21]. A total of 36 specimens of (50 mm) mortar cubes (3 for each case) are made to investigate about the pozzolanic activity index of nano silica admixture. After about 20 to 24 hour, these cubes are de-molded and aren't cured in tab water or in the oven until the ending of the 24 hour. Mortar cubes, for normal curing case, are put in tab water and tested at 7 and 28 days age. For accelerated curing state, mortar specimens are placed in tightly closed glass container and laid in the oven with temperature set of  $65^{\circ}$ C for six days then they are taken out and tested at 7 days age.

Strength activity index can be determined using the following equation [22]:

Strength activity index of pozzolanic material =  $A/B \times 100$  (1)

Where,

A: average compressive strength of mortar cubes of mixes incorporated admixture.

B: average compressive strength of mortar cubes of without adding (control) mixes.



Figure (3):- Graphical representation of pozzolanic activity test of nano silica.

Mix type	Cement, g	Sand, g	Nano silica, g (%)	w/b	Superplasticizer, % of cement wt.	Flow, mm
Control	500	1375		0.485		154
0.5NP	497.5	1375	2.5 (0.5)	0.485	0.15	153
5NP	475	1375	25 (5)	0.485	3	161
10NP	450	1375	50 (10)	0.485	6.5	154

Table (8):- Mortar mix proportions, for six cubes, of nano silica pozzolanic activity test.

# **Results and discussion**

#### **Compressive strength test**

Results of compressive strength test of concrete mixes for 7, 28, 90 ages are shown in Figure 4-a to c. Each result represents the average of three cubes. Due to that two mixes were cast as control (without adding), so, average of six cubes is considered for each result of control mix. If error is occurred during test, the cube result is discarded and the average is determined from the remaining cubes.

Results of micro silica mixes indicated, for 7 days age test, that 10M and 15M mixes have not significant improvement on compressive strength (about 1% and 2%, respectively) in comparison with control mixes. In contrary, 5M mix decreased the compressive strength by about 4%. Similar behavior can be noticed at 28 days age for 5M and 10M mixes. Where, compressive strength is decreased by about 6% for the 5M mix and increased by about 1% for the 10M mix. On the other hand, 15M mix gives-up 5% boosting related to control mixes. At later age (90 days), the three mixes impart raise in compressive strength with about 2, 6 and 7% for 5M, 10M and 15M mixes respectively as compared with control mixes. In a general description for micro silica mixes behavior, it is shown that the development in compressive strength is enhanced with time up to 90 days age though that lower adding of micro silica (5%) showed lower strength at lower ages (7 and 28 days), where, it gave increasing in strength at later ages (90 days). This may be attributed to the need of micro silica to calcium hydroxide that released by cement hydration to react as a pozzolanic reaction. This releasing is increased with curing age and hence increases the pozzolznic hydration.

For nano silica mixes, results showed that all mixes (0.5N, 1.5N, 3N and 5N) show growing in compressive strength for all tested ages compared with control mixes. These results are compatible to the literature [2, 8]. Also, it is found that, the enhancement is increased with increasing of nano silica proportion for 7 and 28 days ages. At 7 days age, the maximum boosting ratio (about 33%) is gotten by 3N mix. This action is due to the accelerating effect of hydration process provided by nano silica admixture. For 28 days age, a raise ratio of 22%, which represents the maximum increasing percent at this age, is obtained by 5N mix, while at 90 days age, a higher increasing (about 27%) is acquired by 3N mix. Additionally, results denoted that small replacement of cement with nano silica (0.5%) afforded remarkable enhancement in compressive strength especially at early age, but, this enhancement is diminished with curing age. Where, an increase of about 18, 7 and 6 % is gained by 0.5N mix at ages of 7, 28 and 90 days respectively. The following reasons may interpretative the promoting in concrete compressive strength caused by adding of nano silica:

- The higher pozzolanic action of nano silica (as demonstrated in pozzolanic activity test).
- The high surface area, which mean higher area provided for reaction.
- Good packing influence between cement particles.

- Due to the nano size of silica particles, the mechanical properties become higher in comparison with micro or macro size particles that mean by incorporating the concrete with good properties material, it leads to enhance its properties with a specific manner.

- Consuming of soluble calcium hydroxide particles and replaced it by solid C-S-H gel which leads to pore refinement of concrete microstructure.

- Densifying the interfacial transition zone (ITZ) between the matrix and coarse aggregate surfaces leading to increase the bond between them and strengthening this region.

- The nucleation effect of nano silica particles that make acceleration for cement hydration, and so, increase the releasing of calcium hydroxide required for pozzolznic reaction.

For nano plus micro silica mixes, results denote that all mixes, except of 0.5N+9.5M mix, impart higher compressive strength than reference mixes for the observed ages. 0.5N+9.5M mix reduced the compressive strength by about 7% at 28 days age, but it is increased at 7 and 90 days age by about 7% and 25%, respectively. For 1.5N+8.5M and 3N+7M mixes, the augmentation in compressive strength are comparable at 7 days age (about 17%) and 28 days age (about 9%) relative to control mixes. At 90 days age, the nano plus micro silica mixes appeared important enhancement in compressive strength compared with control mixtures. The higher development is about 35% that gained by 1.5N+8.5M mix. In addition to the above causes that led to compressive strength development for separate adding of micro silica and nano silica which may present together in nano plus micro silica mixes, specific cause it is believed to appear more obviously here, represented by packing action. Where, it is believed that micro silica can occupy in between cement particles voids, and nano silica particles are set within micro silica voids that lead, as a result, to good pore refinement inside concrete micro structure. This effect is supported by the pozzolanic reaction of the both materials. Based on this concept, 1.5N+8.5M mix can be considered as to have optimum packing due to its large enhancement in compressive strength at early and later ages. At final, it is obvious that nano silica has more impact on compressive strength than micro silica for all tested ages.

### Nano silica pozzolanic activity test

Results of mortar compressive strength cubes of nano silica mixes and their pozzolanic activity compared to control mixes are illustrated in Table 9. Every result refers to average of three cubes. It is obvious from Table 9 that all tested proportions of nano silica material show remarkable pozzolanic activity in comparison with control mix. If it's compared with strength index of 105% that limited by ASTM C 1240 **[18]** for silica fume, results demonstrated that the three proportions used are exceeded this specification. Results denote that 10NP oven cured mix impart maximum strength activity with about 215%, that may refer to the senior reaction intensity of this material with cement components. For 5NP mix, results point out to convergence activity percent between 7 days accelerated curing and 28 days normal curing mixes (about 168 and 165 %, respectively).



Figure 4:- Compressive strength results of micro and nano silica admixtures at (a) 7 days age; (b) 28 days age; and (c) 90 days age.

Low ratio of nano silica (0.5NP mix) also shows important activity for normal and accelerated cured mixes. This behavior gives the advantage to test the pozzolanic activity of nano silica material using small rather than large proportions. But it is necessary to say that the nano silica used has a surface area, which of considered as significant parameter for chemical reaction, of  $(150 \text{ m}^2/\text{g})$ , and nano silica products with different surface areas are commercially obtainable. Hence, using of such proportion (0.5%) in low surface area nano silica products (like; 100 m<sup>2</sup>/g) may give lower activity than the nano silica used. In other word, it is preferable to use a proportion considered suitable for different nano silica surface areas present in the markets. For this reason, based on the obtained results, a 5% of nano silica proportion is adequate for testing pozzolanic activity index for nano silica substance. It may be questioned why don't use 10% nano silica though this ratio gave more than 200% activity at 7 days in both normal and accelerated curing. The answer of this question is that, at 28 days age water curing, the activity of 10% and 5% of nano silica are comparable (about 165%), so, it is preferable to use low rather than large ratio.

In case of curing type, the two considered curing method (accelerated and normal) are adequate for testing the pozzolanic activity. Thus, it is up to the researcher for choosing the curing method and the testing age. The two methods impart acceptable results.

Mir armhal	<b>Compressive strength in Mpa (pozzolanic activity, %)</b>					
with symbol	7 days (Normal)	7days(accelerated)	28 days (Normal)			
NilP	18.8 ()	21.67 ()	29.31 ()			
0.5NP	22.46 (119.5)	24.27 (112)	32.66 (111.4)			
5NP	34.8 (185.1)	36.47 (168.3)	47.93 (164.7)			
10NP	38 (202.1)	46.73 (215.6)	48.33 (164.9)			

Table (9):- Compressive strength of mortar cubes and pozzolanic activity of nano silica

#### **CONCLUSIONS:**

Conclusions obtained by this study are summarized as follow:

1. Direct adding of high surface area nano silica material to the mixing water leads to conglomerate the blend and subsequently loss the high surface area provided by nano silica. Thus, dry batching of nano silica with cement (and thereafter with other constituents) is suitable procedure.

2. Nano silica has remarkable pozzolanic activity reaches to about 215% for 10NP accelerated curing mix compared with control mortar. Small proportion (0.5%) is also showed noticeable activity. However, it is suggested to use 5% nano silica in performing the activity index test. The convergence of activity results of 7 days accelerated and 28 days normal curing represent another cause to suggest using 5% ratio. For curing type, it is possible to use accelerated or water (normal) curing for this test. The two methods yield acceptable results.

3. The addition of 5% of micro silica decreased the compressive strength of concrete by about 4% and 6% at 7 and 28 days respectively, however, the compressive strength is increased by about 2% at 90 days age. For 10M and 15M mixes, compressive strength is increased slightly at 7 days age (1% and 2% respectively). But, significant increase is noticed at 90 days age (about 6% and 7% respectively).

4. The addition of nano silica leads to important increase in compressive strength for all tested proportions, where, max increasing ratios of 33% and 27% for 3N mix are obtained at 7 and 90 days age respectively, and 22% for 5N mix at 28 days age.

5. The incorporating of nano and micro silica at the same time in concrete mixes enhances packing effect between different size particles (cement, micro and nano silica). This improves the compressive strength of the composite. This behavior is noticed by the prominent increasing in compressive strength (about 35%) at 90 days age for 1.5N+8.5M mix.

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