

## INVESTIGATION OF NANO STRUCTURED MESOPOROUS SILICA EFFECT ON COMPRESSIVE STRENGTH PROPERTIES OF NORMAL CONCRETE

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**Resumen:** En esta última década los investigadores invierten en nanotecnología con el fin de hacer una nueva generación de materiales de hormigón para obtener estructuras de hormigón estables. El funcionamiento de los materiales básicos de cemento depende intensamente de los rasgos de las partículas sólidas en dimensión de nano tamaño como partículas de porosidades de silicato, calcio, hidrato o nanómetro en el espacio transitivo interior entre partículas de cemento y piedra. En este laboratorio de investigación, estos cuatro experimentos incluyen una mezcla de dos series consisten en nanosilica y dos series de hormigón normal que se utilizan totalmente dieciséis muestras cúbicas. El efecto de la nanosilica con MCM-41 mesoporoso se examina sobre la resistencia a la compresión del hormigón normal. Las nanopartículas de sílice mesoporosa tienen la medida de superficie más de  $600 \frac{m^2}{gr}$ . Con base en la definición de IUPAC,

las partículas mesoporosas tienen el tamaño de cavidades en el rango de 2 a 50 nanómetros. La producción de estas composiciones es posible mediante un método de plantilla en un proceso químico. Para estudiar la microestructura de las partículas de sílice mesoporosa se utilizan las imágenes SEM y TEM del microscopio electrónico. Los resultados de la investigación son representativos del aumento de la resistencia a la compresión en una tasa definida que disminuye debido a la utilización de un nivel específico de nanomateriales.

**Abstract:** In this last decade the researchers invest in nanotechnology in order to make a new generation of concrete materials for getting stable concrete structures. The operation of basic cement materials is intensely depends on solid particles' traits in nanosize dimension like particles of silicate, calcium, hydrate, or nanometer porosities in the interior transitive space between cement and stone particles. In this laboratory research, these four experiments include a mixture of two series consist of nanosilica and two series of normal concrete that are totally used sixteen cubic samples. The effect of nanosilica with mesoporous MCM-41 is surveyed on normal concrete' compressive strength. Mesoporous silica' nanoparticles have the measurement of surface more than  $600 \frac{m^2}{gr}$ . Based on the

definition of IUPAC, the mesoporous's particles have the size of cavities in the range of 2 to 50 nanometer. The production of these compositions are possible by templating method in a chemical process. To study the microstructure of mesoporous silica particles are used the electron microscope's pictures SEM and TEM. The results of research are representative of increasing compressive strength in a definite rate that decreases because of using a specific level of nanomaterials.

**Keywords:** nanosilica, mesoporous silica, compressive strength, porosity, concrete.

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Undoubted concrete technology is a kind of science which is changed fast and intensely during the last few decades. Concrete is known as the most widely used material after water and the most common building materials in the world. This material has two basic parameters: resistance and durability {1}. More recently, nanotechnology which is originated from new and useful potentials in nanoscale significantly has attracted the interest of researchers. With the advancement of nanotechnology, nanomaterials have been developed that they can be added to concrete for the study of physical, chemical, and mechanical properties. We can mention different materials of produced nano like: Nano-silica, Nano-alumina, Nano Titania, Nano-zirconia, Nano-iron and etc. adding nano and production of hydrated silica CH cause increasing possibility of reaction with calcium hydroxide CH for increasing more resistance in cement structure and producing calcium silicate hydrate CSH. In addition, it fills the pores that are made by using nano-silica in concrete (Maheswaran, 2013). In normal concrete, a part of produced calcium hydroxide that originated from hydration of cement is washed gradually, and it comes out of concrete in the form of sediment that its effect remains in the form of microstructure pores. On the other hand in the samples containing nano-silica, this calcium hydroxide has chemical reaction with nano-silica, and it produces calcium silicate hydrate gel that causes to reduce amount of sediment and also it is washed out of concrete. (Beigi, Barnjian, Ahmadvand, Payatfi & Imran, 2012)

In this research, tried to use nanomaterials with porous structure by area more than twenty times than its non-porous counterpart.

The porosity is defined as pores containing hole, channel, and gap that the measure of depth is more than width. The way you can access the porosity can be in the form of closed pores (a, b, c, d, e, and f), open pores, f and b in the form of closed, or e in the form of all over open (extended). Also a porous material can have porosity I the form of a) cylindrical open porous b) closed cylinder c) in the form of ink container d) funnel and e) in the form of Downs and Ups (of porosity) that you can observe in the figure 1.

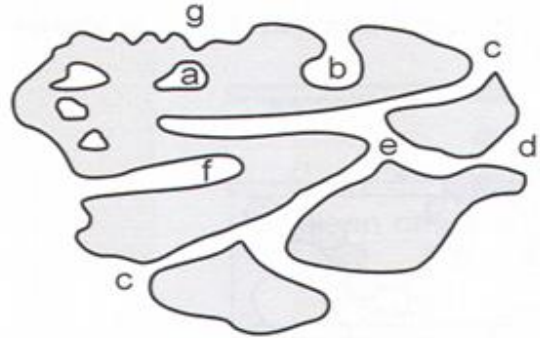


Figure 1. Porosity Types (McCusker, Liebau & Engelhardt, 2003)

According to the IUPAC definition, porous materials are divided into three categories. Based on this definition, any porous material can be easily categorized (McCusker, Liebau & Engelhardt, 2003). In this category, the materials which have pore diameter less than two nanometers are known as Micropore, and the ones with pores 2 to 50 nanometer named Mesoporous materials, and also the ones with pore diameter more than 50 nanometer are known as Macropore materials.

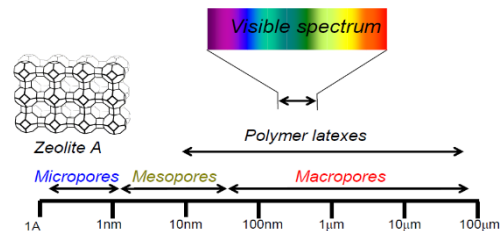


Figure 2. Categories of porous material by IUPAC (McCusker, et. al, 2003)

Mesoporous materials' pore size is in the range of 2 to 50 nanometers. Ordered Mesoporous materials have ordered and arranged pores in a crystalline system that the pore distribution is close to each other. Providing these combinations are possible by snap method.

The successful synthesis of Mesoporous materials is reported for the first time by Beck and his colleagues as M41S family in 1992. This family contains their types of structures that each one has highly ordered channels and close pore size distribution (Tang, Li & Chen, 2012). The figure 3 shows pores morphology of these combinations. Figure 3 shows (a) MCM-41 that has ordered hexagonal channels. The figure (b) 3 shows MCM-48 that has complex three-dimensional channels. The figure (c) 3 is about MCM-50 that has layer structure. Providing these materials leans on

gathering self-assembled surfactants as determining factor of structure that is known as liquid crystal templating and this liquid crystal can have hexagonal structure, cubic structure, or lamellar structure.

Compared with the microsilica, nano-silica is more pure than silicon dioxide  $\text{SiO}_2$  and also has higher specific surface area. These two main properties create major differences between these two materials. (Haydari, Ali, Taheri, Head of Toning, n. d.).

Ramezaniapour and his colleagues (2009) demonstrated that water permeability in concrete containing nano-silica is reduced noticeably compared to normal concrete and concrete containing Silica Fittings (Ramezaniapour, Jahromi & Moody, 2009).

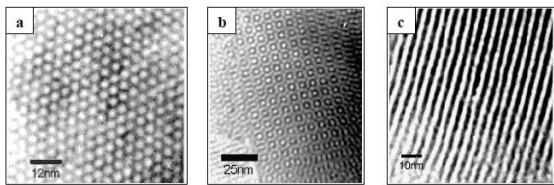


Figure 1. picture TEM different structures of Mesoporous silica (McCusker, et. al, 2003)

Ching and his colleagues (2005) observed that nano-silica has higher pozzolanic properties than micro silica, and it increases higher resistance. One of the reasons for improvement of compressive strength is originated from pozzolanic activity of nano-silica. According to this, silica nanoparticles containing percent purity more than 99 % and high specific surface area react with crystals of calcium hydroxide resulting from hydration and produce calcium silicate hydrate gel. This reaction probably improves the connection of paste aggregate.

Li and his colleagues (2004) believe that one of the reasons for increasing resistance of concrete is the effect of filling particles in the pores of cement paste. They demonstrated the placement of nanoparticles prevents from increasing crystals of calcium hydroxide and thereupon the amount of crystal is reduced versus increasing volume of hydrated calcium gel in cement matrix (Li, Hui-Gang & Jin-Ping, 2004).

In another research, Beigi and his colleagues (2013) showed that using nano-silica compound will increase mechanical properties to the optimum

percentage of one and a half percent nano-silica and three percent micro silica and then slowly reduced.

This research also shows that the influence of elastic modulus compared to changes in percentage of nano-silica is more noticeable than changes of polypropylene fibers.

Ji (2005) believes that the most important reason for development of compressive strength of concrete with nano-silica is the filling role of nanoparticles in the pores of cement paste, and he thinks the reason is because silica nanoparticles have high microlithic and surface adhesion of the particles, they are placed between calcium silicate hydrate gel and fill get particles and increasing durability of concrete.

In research, Qing and his colleagues worked on the effect of increment of silica nonparticles on the properties of hardened cement paste compared to micro silica, and they surveyed the way these materials react with cement paste. They demonstrated silica nanoparticles have higher reactivity compared with micro silica (Qing, Zenon, Kong & Chen, 2005).

## 2. LABORATORY PROGRAM

To survey the influence of nano-silica compound along with super lubricant with the base of carboxylate and the brand SRJ750 and also choosing the best percentage for adding these materials were used 1.5 percent and 4 percent of cement weight. For getting these results, the compressive strength were extracted at the ages of 7 and 28 days. For compressive strength of cube samples  $10*10*10$  cm and for both of ages were provided sample. In addition, the results were determined at any age.

## 3. USED MATERIALS

Cement: in this research, the used cement belonged to Fars factory's cement type 2.

Stone aggregates: the used sand belonged to Sad Saze mine.

Water: Shiraz drinkable water was applied for all samples.

Nano materials: used compound include: nano-silica (1.5 % and 4.5 %) with super lubricants (0.6 %). The tables 1 and 2 present chemical and physical properties of stone aggregates and cement in concrete samples.

Chemistry test	ASTMC150	Foundry
Sio	20	20.38
Al <sub>2</sub> O <sub>3</sub>	6max	5.21
Fe <sub>2</sub> O <sub>3</sub>	6max	5.49
CaO	-	60.64
MgO <sub>2</sub>	5max	2.62
Na <sub>2</sub> O	-	0.34
K <sub>2</sub> O	-	0.86
SO <sub>3</sub>	3.5	2
PQL	-	0.84
IR	0.75max	0.6
LOI	3max	0.63

Table 1. chemical properties of Fars factory cement type 2.

Specifications	Coarse grain	Fine grains
Density	2.85	2.60
Water absorption	0.2	0.5
Volume unit weight	1630	1550
Fm	-	2.63
D max	25	5

Table 1. physical properties of stone aggregates in concrete samples.

#### 4. MIXING RATIOS

The concrete mix plan based on the nominal maximum particle size is 25 mm. according to this regulation AC1211-09, unit weight of fresh concrete is 2300 kilogram per cubic meter and also the volume of coarse grain sand with fineness modulus more than 3 is determined 28 percent per unit volume of concrete. Of course, amendment of the plan AC1211-09 is done based on available building materials I the area. Due to the use of silica nanomaterials with porous structure for comparison and how porous materials react with cement hydration, and also how junction area of cement paste adheres to aggregates of normal concrete are used two kinds of plans: the first one with fine-grain sand and the other one with coarse sand grading. Coarse gravel and fine-grained materials became grading based on standard ASTM C136. The tables 3 and 4 present sand grading chart and the concrete mix plan.

#### 5. CREATING, PREPARATION AND STORING SAMPLES

To make mix, at first, used materials are very carefully weighed. Then weighed sand is poured into the retractable awning electric mixer and

afterwards some weighed water is added – as much as the grains need to turn to SSD (saturated surface dry) and it should mix for 3 seconds. Then nano-silica compared with cement is added to mix and the mixing operation is done for 2 minutes. In the final step, remained water in which super lubricant is sufficiently poured is added, and mixing need to continue for two more minutes. Immediately after finishing mixing, Slump tests are performed on the mixture. Afterward the samples are molded and after compressing, it should remain within mold for 24 hours. Later, after the molds are open, they immediately experience curing in the water and lime-saturated environment. Meanwhile, the average temperatures of the lab must be about 22 to 25 degrees Celsius. Water-cement ratio should be used in terms of using super lubricant  $w.c = 0.4$ .

#### 6. COMPRESSIVE STRENGTH TEST

Compressive strength tests were performed on samples 10\*10\*10 cm at the age of 7 and 28 days like following figures 4 and 5 on the control plan and nano-silica. This test was done based on standard ASTM C39. Also table 5 shows compressive strength ratio of cube samples with non-standard dimension to compressive strength of standard samples.

sand gradation									
FM	Bt limit		Up limit		Percentage rejected (%)	Percent retained (%)	Weight retaining (gr)	Sieve size	
	USA	Iran	USA	Iran					
0	100	100	100	100	100	0	0	9.5	3.8
3.48	95	89	100	100	96.52	3.48	32	4.75	4
40.26	80	60	100	100	59.74	36.78	328	2.36	8
65.51	50	30	85	90	34.49	25.24	232	1.18	16
90.10	10	5	30	40	17.74	16.76	154	0.6	30
96.84	2	0	10	15	6.75	6.75	62	0.15	100
138.84					-37.84	4100	29	pan	
3.78	FM					137.84	919	total weight	

Table 3. Sand gradation



Figure 4. sand gradation

Design name	Gravel	Gravel	Sand	Cement	Water	Nano silica	Hrwrw/c	
C1	630	-	1200	350	140	-	2.10	0.4
C2	-	630	1200	350	140	-	2.10	0.4
Ns 1.5	630	-	1200	350	140	5.25	2.10	0.4
Ns 4.5	-	630	1200	350	140	5.25	2.10	0.4

Table 4. mix plan ratios of control samples and nano

Size model	100	150	200	250	300
Ratio strength	1.10	1.00	0.97	0.94	0.9

Table 5. Cube sample



Figure 2 Control and nano sampling of fresh concrete



Figure 3 Seven-day nano sample

## 7. PRESENTING THE RESULTS

In this part, the results of the compressive strength of nano-silica and cube samples are studied. The tables 6 and 7 show compressive strength of plan sample of coarse gravel and plan of fine gravel at the ages 7 and 28 days.

design	Model S	Model N
7days	328	313
28days	340	348

Table 6. Plan samples of coarse gravel and 1.5 % nano

design	Model S	Model N
7days	245	228
28days	298.7	236.9

Table 7. Fine gravel samples and 4.5 % nano

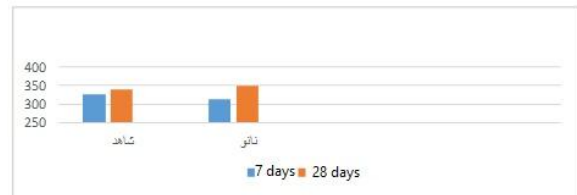


Figure 6. Compressive strength of coarse gravel plan



Figure 7. Compressive strength of fine gravel plan

## 8. ANALYSIS OF THE RESULTS

Three-percent increase is achieved based on compressive strength test with added silica nano materials (1.5 %) to cement with coarse gravel plan. Also when silica nano materials (4.5 %) are added to cement with fine gravel plan, there will be thirteen-percent reduction of resistance. Respectively, the figures 6 and 7 show the increment process of coarse gravel's nano sample and also reducing fine gravel sample. The resistance is increased because of strong pozzolanic reaction in nano silica and the resistance is reduced because of very high specific surface of nanoparticles that when they exceed a definite amount (optimum value), they can adhere to each other by a physical

reaction and produce unstable hunks, so in this experimental study we can know one and a half of nano silica as optimum percentage. Optimum value of micro silica with specific surface 20 square meters per gram is seven to eight percent of the weight of cement materials when the goal is durability of concrete, and in some cases that we want to increase resistance of concrete, about ten percent of material weight are cement. While the goal is the optimum value of nano silica with very high specific surface, we will have 1.5 to 3 percent of the weight of cement materials in this laboratory research, in some cases that we need the resistance of concrete. It is the representation of economic saving in consumption of cement and pozzolanic materials.

## 9. CONCLUSION

1. At the early ages of concrete, compressive strength of nano sample is reduced in both of coarse gravel and fine gravel plan compared with control sample.
2. Three-percent increase and thirteen-percent reduction of compressive strength are achieved in this research, respectively, nano's coarse gravel and fine gravel plans compared with control sample.
3. According to the results of compressive strength of two plans of fine gravel and coarse gravel, the coarse gravel is improved with a small percentage of nano silica. According to this, for economic saving, we can use less cement to make normal concrete.

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