Leadership in Sustainable Infrastructure
Leadership en Infrastructures Durables

Vancouver, Canada
May 31 – June 3, 2017/ *Mai 31 – Juin 3, 2017*

THE EFFECT OF NANO SILICA ON CEMENTITIOUS MATERIALS

Ghaly, Athnasious[1,2], Attia, Aya[1], Mostafa, Habiba[1], Sammour, Karim[1], Ghobrial, Mina[1], Fathy, Amr[1] and Abou-Zeid, Mohamed[1]
[1] Department of Construction Engineering, The American University in Cairo, Egypt
[2] athnasious93@aucegypt.edu

Abstract: Nano silica is a relatively new product that has come to markets in limited parts of the world. Its production and thus characteristics vary significantly in the absence of clear specification and guides for its use. This study aims at achieving better understanding of the performance of cementitious mortar prepared using imported Nano silica when compared with mixtures made with silica fume. The testing program included physical properties, chemical analysis as well as the compressive and flexural Strength. Another set of tests included water permeability, rapid chloride permeability, resistance of the mortar to sulphates, sulphuric acid. Results reveal that the Nano silica used enhances some of the cementitious mortar properties while substantial enhancement was not witnessed when compared to silica fume mixtures. Recommendations are provided to better utilize this innovative material and projects that are likely to make best use of its application.

1 Introduction

Nano-technology is concerned with the production of new materials designed from molecular scale with dimensions measured in nanometers. It evolved in the 1980s as a result of experimental advances such as the invention of the scanning tunneling microscope which allowed nano-metric particles to be both observed and tested. There are two approaches through which Nano structures are produced; Top Down and Bottom Up. Top Down refers to the cutting of material bulk to reach Nano scale, while Bottom Up refers to the Build-Up of material from scratch starting at the atomic level. Produced Nano structures have much more enhanced characteristics in comparison to their alternatives of larger scale. This results from the continuous modification of characteristics of such particles with diminishing size. Their composition, as a result of their diminished size, allows them to exhibit improved physical, chemical, mechanical, and biological properties. Due to the fact that they are so small in size allows them possess a great surface area that aids in the well distribution of particles and help particles possess a filling effect that extends to cement grain voids. This results in better homogeneously compacted microstructures. The impact of Nano-technology extends to many fields such as biology, chemistry, and many others. It has lately emerged into the science of materials and engineering. Nano Silica is one application of Nano-technology used in the science of materials. Studies conducted support Nano Silica's property enhancement in cementations materials.

2 Literature Review

1) Synthesis of Nano-silica from silica fume using an acid-base precipitation technique and PVA as a non-ionic surfactant:

In this journal entry, Nano technology's Top-Down process is applied to the synthesis of Nano-Silica from Silica Fume under a controlled environment using Poly Vinyl Alcohol as a dispersing agent. The whole synthesis process is explicitly presented and SEM images were taken to confirm the nano metric scale at which Silica Fume is broken down to. Nano-Silica purity was further investigated using the FTIR, XRD, and XRF tests which proved the sample to consist of pure Silica particles (Jafari, 2014).

2) Preparation of Nanoparticle Dispersions from Powdered Material Using Ultrasonic Disruption

In this paper, guidelines are provided for the use of Ultrasonic waves in the preparation of fully dispersed Nano-Silica powder in a liquid medium. Nano Silica is known to be hydrophobic, thus sonication is required for it to get fully dispersed in water. Full water dispersion result in uniform well-distribution of NS particles in mixes. Poor distribution would force particles to get concentrated in some parts more than the others which will give an adverse effect (Hackley, and Taurozzi, 2012).

3) Characteristics of blended cements containing nano-silica

The main objective of this work is to evaluate the effect of Nano Silica on different cement based mixes. A comparison is presented among different mixes made with various percentages of Nano Silica, Ordinary Portland Cement, and Granulated Blast Furnace Slag (GBFS). Free Lime estimation as well as combined water content are presented at different curing stages. Compressive strength, setting time, and water for consistency were also determined for all mixes. It was concluded that due to the addition of 1% of super plasticizer in Nano Silica mixes, water for consistency as well as setting time increased. Moreover, as the amount of Nano-Silica increased free lime as well as PH values decreased. Optimum improvement of mechanical properties was visualized in the mix containing 3-4% of Nano Silica and 45% GBFS. Results were further supported by the XRD, DTA, SEM, and IR testing (Morsi, Abd El Aleem, and Heikal, 2013).

4) Application of Silica Fume and Nano-silica in Cement and Concrete – A Review

In this paper, a study is conducted on the development of silica fume application and for the concrete industry sustainable development where waste materials are reduced and energy is saved. This research investigates micro and nano structured silica and their effect on mechanical properties and durability is reported. It argue that no extensive studies have been conducted to investigate the effect of NS addition on cementitious materials. Different mixes are prepared; some containing Nano Silica, other Silica Fume. Multiple tests are carried out such as compressive strength, XRD, and SEM. Test results support that Nano Silica addition in Concrete would result in long lasting results (Gupta, 2013).

5) Utilization of Nano Silica as Cement Paste in Mortar and Porous Concrete Pavement

In this research the application of nano silica is investigated to be applied in concrete pavements. Nano silica was found to improve the mechanical properties of cementitious materials when incorporated with the right percentage. Nano silica enhances porous materials as it both improves cement paste as well as the interface between aggregates and paste. This is due to their Nano size which allows them to possess a filling effect that extends to cement grain voids. This results in better homogeneously compacted microstructures (Yusak, 2015).

6) Effect of Nano Silica on Mechanical Properties and Durability of Normal Strength Concrete

In this article, researchers reached the conclusion that Nano-Silica could be used as external coating for concrete as Nano-coated concrete have proven to be highly durable as it is less

permeable to chloride. Nano Silica also enhances concrete's mechanical properties (Gopinath, Mouli, Murthy, Lyer, and Maheswaran, 2012).

7) Effect of Nano Silica on Mechanical Properties of Conventional Concrete: A Case Study

This case study further investigates the effect of Nano Silica addition in conventional concrete and several tests are conducted to evaluate concrete's properties. A total of 8 conventional concrete mixes were prepared with different Nano Silica percentages (0%, 1%, 1.5%, and 2%). The incorporation of 1.5% of Nano Silica proved to be the most optimum for the improvement of concrete mechanical properties (Reddy, Mohan, and Reddy, 2014).

3 Objective & Scope

In this research, the main objective was to study this new type of materials that is recently introduced to the construction industry. It was very important to understand the physical and chemical properties of this material. The objective was studying the effect of the nano silica on the concrete (specifically Mortar) properties such as Compressive strength, Flexure Strength, Water Permeability, Chloride Permeability, and Chemical resistance, also compare it to the conventional mortar and mortar with silica fume

4 Experimental Work

4.1 Preliminary mixes:

After several preliminary mixes and trials prepared with NS, the following outcomes were found

- NS required careful mixing for the distribution of particles
- Sand absorption was assumed to be 0.5%, however, by conducting the sand absorption test it was found that it requires 1.4% instead.
- Self-compacting super plasticizer such as Viscocrete is needed as mixes suffered highly poor workability
- W/C ratio of 0.45 needed to reach a fairly workable mix

So as a result for those outcomes, mix design was set with a W/C ratio of 0.45 and the use of Sika-Viscocrete 3425 Super plasticizer. Different NS percentages were used resembling medium and high dosages which were recommended by the supplier (1.5% & 2%). The same applies for S.F (10%&15%). Another mix was prepared by combining SF with 10% and NS with 2% and last a control mix with Ordinary Portland Cement was also prepared to measure the efficiency of NS against.

4.2 Mixes:

Several preliminary mixes poured with different mixing techniques in the beginning of the research to reach the optimum mix design, and techniques that would be built on. It was concluded and later used in the relied upon mixes the following

Table 1: (Mix Designs)

Mix Design	W/C	Water (Kg/m3)	Cement (Kg/m3)	Sand (Kg/m3)	Silica Fumes (Kg/m3)	Nano Silica (Kg/m3)	Viscocrete (Kg/m3)
A (Conv.)	0.45	272	550	1749	0	0	11
B (N.S. 1.5%)	0.45	272	541.75	1749	0	8.25	11
C (N.S. 2%)	0.45	272	539	1749	0	11	11
D (S.F. 10%)	0.45	272	495	1749	55	0	11
E (S.F. 15%)	0.45	272	467.5	1749	82.5	0	11
F (N.S 2% +S.F 10%)	0.45	272	484	1749	55	11	11

4.3 Tests:

4.3.1 Tests on Aggregate:

- *Sieve analysis:* This test was conducted to check for the gradation of used the fine aggregate

4.3.2 Tests on the Nano Silica Powder:

- *Energy-dispersive X-ray spectroscopy (EDX):* This test is done on any material to analyze its chemical composition and provide the elements and its percentages in this material.
- *Scanning Electron Microscopy (SEM):* this is type of test that scan sample of the material and give a magnified picture of particles, and it has very high magnification power so that is can display image of the nano-particles and measure size of these particles.

4.3.3 Tests on the Concrete:

- Compressive strength: Test was performed to find out the compressive strength of mortar mixes. This test was performed on cubes (5cm*5cm*5cm) at 3 days, 7 days, 28 days
- Flexure strength: This test is measuring the flexure strength of the mortar we are studying. Three-point test was performed only at 28 days on (4cm*4cm*16cm) beams
- Rapid Chloride Permeability: In this test, we were trying to study the performance of concrete in resisting the flow of chloride ions through it. This can help us in determining whether this material can be used in applications that require less permeability of chloride ions. The test was done on specimen after 28 days
- Water Permeability: This test measures the permeability of mortar or concrete to water by measuring how deep the water penetrates the specimen through three days under pressure. The test is done on specimen after 28 days.
- Chemical resistance:
    - Sulphuric acid (H2SO4): cubes were submerged in diluted sulphuric acid with concentration 5% after 7 days, and left for 7days.
    - Sulphate salts (Mg SO4): cubes were submerged in solution of salt with concentration (0.5 Kg MgSO4 / 3 liters' water) after 7days and left for 15 days

4.4 Results and Discussion:

4.4.1 On fine aggregate (sand):

First, water absorption test showed that the percentage of absorption was high (1.4%) for the type of sand used so this was taken into consideration while assigning the mix design. In addition to water absorption,

sieve analysis Table (2), Chart (1) Showed that the aggregates was well graded and having acceptable percentage of fines 1.1%.

Table 2: (Sieve Analysis)

Sieve #	Size (mm)	Percentage passing %
4	4.76	97.7
8	2.38	95.0
16	1.19	90.0
30	0.595	62.3
50	0.297	23.9
100	0.15	3.6
>200	0.075	1.1

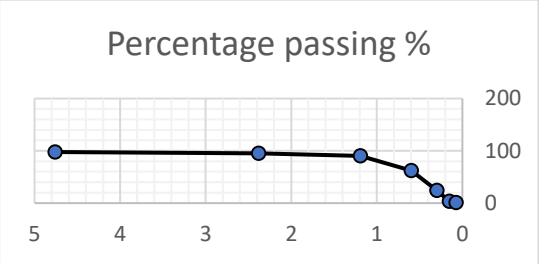


Figure 1: (Gradation curve)

4.4.2 Compressive Strength:

The following table (3) and chart summarize the Compressive test results for 3, 7, and 28 days. NS, when compared with the control mix, was observed to enhance the compressive strength of the mortar it's added to. However, when is compared to SF, its compressive strength yielded lower results. It is also observed that increasing the percentage of NS more than 1.5 results in compressive strength reduction.

Table 3: (Compressive Strength)

Mix Design	Compressive Strength (MPa)		
	3 days	7 days	28 days
Conv.	22.8	33.7	34.5
NS 1.5%	24.0	28.8	37.9
NS 2%	22.6	27.1	32.5
S.F. 10%	19.9	29.4	29.2
S.F.15%	16.8	25.8	42.6
SF 10% & NS 2%	18.8	26.1	33.5

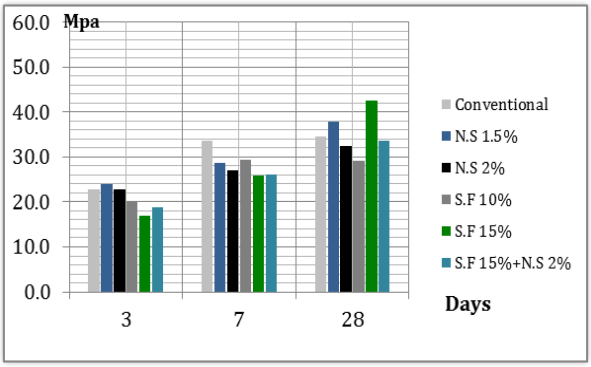


Figure 2: (Compressive Strength)

4.4.3 Flexure Strength:

In Table (4), the average flexure strength of two beams tested on 28 days shows that both silica fume and Nano silica has improved the flexure strength of mortar, however the nano silica had higher improvement in strength. Also it was observed that the effect of nano silica on flexure strength decreased when the percentage used is higher than 1.5% as shown in the 2% mix. Finally, it was found that when both nano silica and silica fume is used together, better results can be achieved than each independently.

Table 4: (Flexure Strength)

Mix Design	Flexure (Mpa) 28 days
Conv.	2.20
N.S 1.5%	3.50
N.S 2%	2.83
S.F. 10%	2.73
S.F.15%	3.17
SF 10% NS 2%	3.30

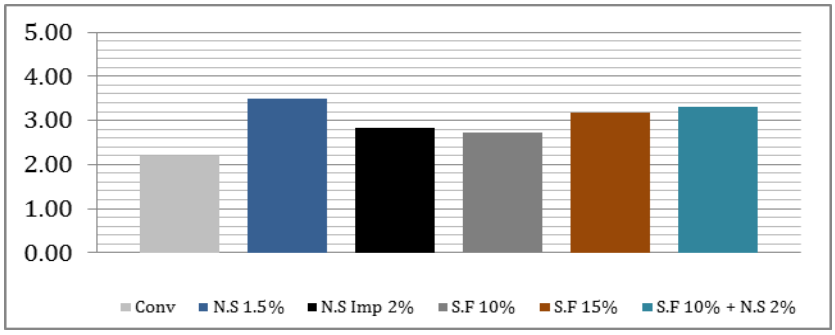


Figure 3: (Flexure Strength)

4.4.4 Sulphuric Acid Resistance:

After measuring the weight loss in the specimens that were exposed to acidic solution, the following results was found as in Table (3). It was found that the nano silica had improved significally the ability of mortar to resist the acidic chemical attack. The effect of Silica fume was minor. It was observed that use of silica fume with nano silica in mortar, gave negative effect on acidic resistance.

Table 5: (Sulphuric acid resistance)

Mix	Conv.	N.S 1.5%	N.S 2%	S.F 10%	S.F 15%	SF 10% N.S 2%
Δ Weight (%)	-15	-12.8	-10.3	-14	-14.2	-27

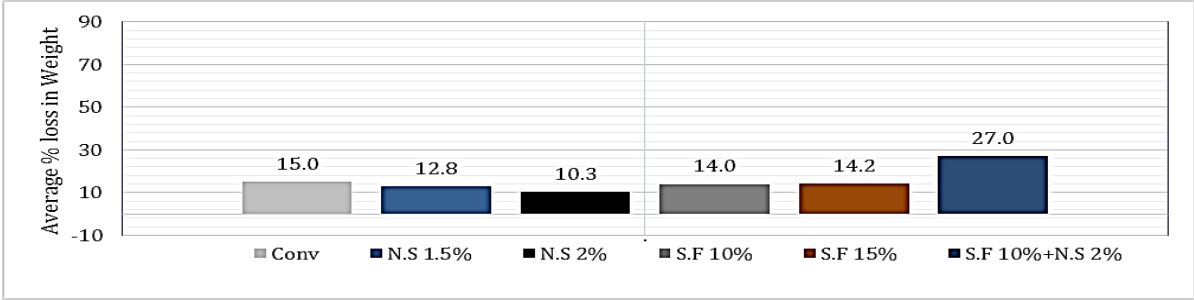


Figure 4: (Sulphuric acid Resistance)

4.4.5 Sulphate salts Resistance:

After measuring the weight loss in the specimens that were exposed to acidic solution, the following results was found as in Table (4). It was found that the nano silica had improved significally the ability of mortar to resist chemical attach of sulfates. Also the Silica fume has improved the ability of mortar to resist chemical attach of sulphates.

Table 6: (Sulphate Salts Resistance)

Mix	Conv.	N.S 1.5%	N.S 2%	SF 10%	SF 15%	SF 10% NS 2%
Δ Weight (%)	0.31	0.09	0.06	0.2	0.21	0.08

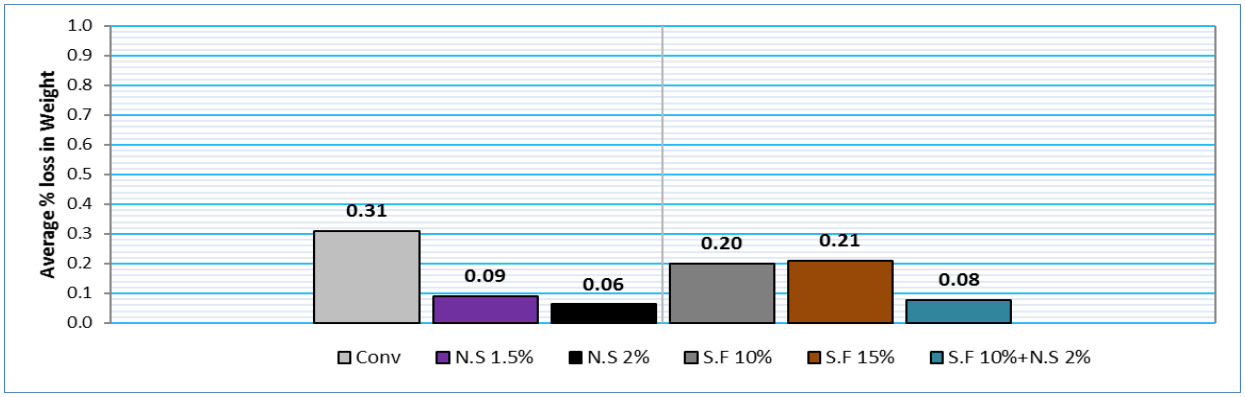


Figure 5: (Sulphate Salts Resistance)

4.4.6 Water Permeability:

After three days of applying the water under pressure to mortar cubes, the results as shown in Table (3) shows that both the Silica fume and nano silica has improved the impermeability of mortar against water, however the silica fume was more effective in limiting the permeability of mortar.

Figure 6: Cubes internal section after test

Table 7: (Water Penetration test)

Mixes	Penetration (cm)
Conv.	1.4
NS 1.5%	1.3
NS 2%	1.3
S.F 10%	1.0
S.F15%	0.5
S.F 10%+NS 2%	0.5

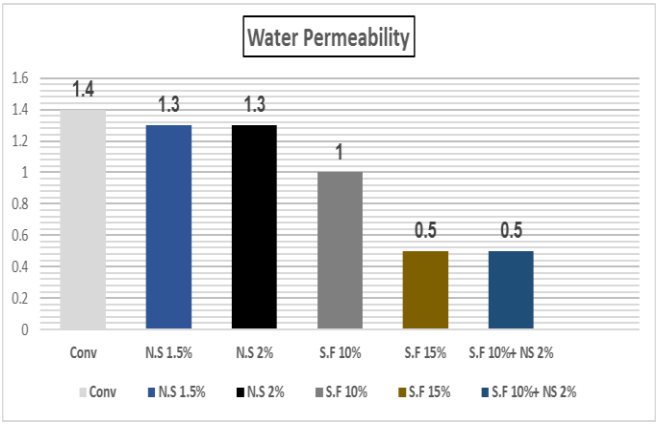


Figure 7: (Water Penetration results)

4.4.7 Rapid Chloride Permeability:

After exposing the mortar specimens to the chloride ion test, it was observed from the results shown in Table (4) that the nano silica has improved the ability of mortar to resist the flow of chloride ions in it. However, the mortar with silica fume was more resistive to chloride ions than the nano silica.

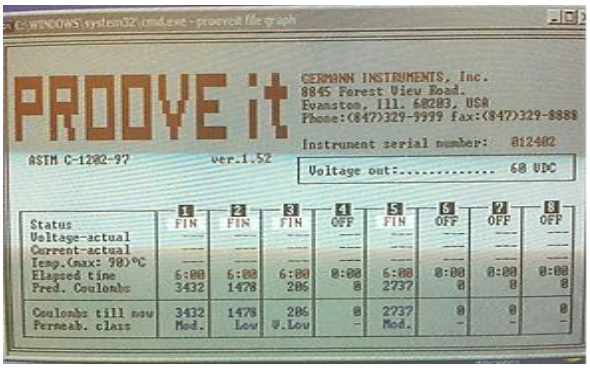


Figure 8: RCP Results screen sample

Table 8: (RCP test Results)

Mix Design	Passing Coulombs	Permeability class
Conv.	2737	Mod.
N.S 1.5%	1370	Low
N.S 2%	1478	Low
S.F 10%	206	Very low
S.F 15%	223	Very Low
S.F 10% +N.S 2%	320	Very Low

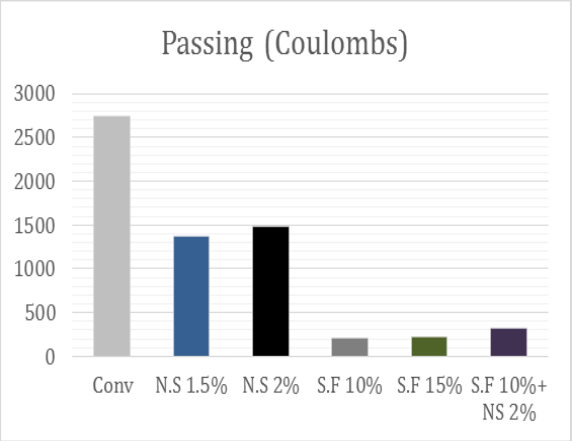


Figure 9: RCP coulombs passing

4.4.8 SEM:

Scanning Electron Microscopy is used very effective in microanalysis of solid inorganic materials. Scanning electron microscopy is performed at high magnifications, generates high-resolution images and precisely measures very small features and objects. It gives information about the sample's topography and composition. SEM is used in this research to get a magnified picture of the nanoparticles to check the nano-metric size and monodispersity of particles

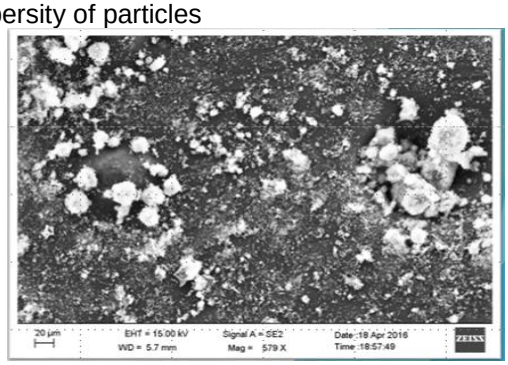
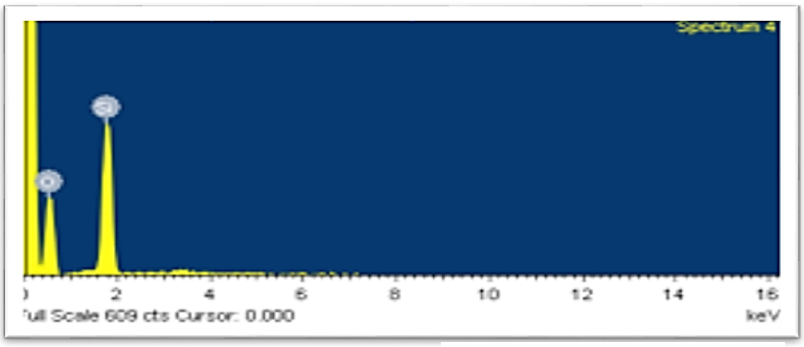


Figure 8: SEM Result

4.4.9 EDX:

EDX is a non-destructive testing tool that helps determine the morphology and chemical composition of a material, the test was done on Nano-Silica. The data collected composes spectra that show peaks of the elements composing the material tested. NS was found to be approximately 100% pure



Element	Weight%	Atomic%
O K	57.52	70.39
Si K	42.48	29.61
Totals	100.00	

Figure 9: EDX Results

5 Conclusion:

The outcome of this research can be concluded in the following:

- There is no clear evidence of a paramount enhancement for Nano silica over silica fume
- Mixture made with fine materials such as silica fume and Nano silica seem to be sensitive to the selection of appropriate super plasticizer in the mix
- All dosages Nano silica used didn't contribute to a large increase in compressive strength of the cubes tested
- The imported Nano silica in particular contributed to an enhancement of the flexural strength more than the compressive strength
- Most of the Nano silica mixtures exhibited lower permeability and higher resistance to Sulphuric acids and Sulphate salts.
- Nano silica needs careful handling and mixing to minimize high variability in results
- All mixes with Nano silica suffered from drop in workability

6 Recommendations for Future Research:

- Silica Particles must be stored in a humid-free, temperature-controlled environment to prevent the clustering of particles which would give an adverse effect on mixes prepared
- Nano Silica particles (NS) are known to be hydrophobic. Special consideration should be given to NS mixing method. Suppliers should provide for the most convenient method that would yield a uniform distribution of particles inside the mix
- In case supplier recommends a wet mixing method for NS particles, in which particles are first dispersed into water before mixing with other components, exact amount of water should also be provided that would allow for full dispersion of particles in solution
- Cube size is highly correlated with the distribution of Silica particles. 5*5 cubes were used in compression strength testing. This resulted in large variations is cubes' strengths. Larger sized cubes should be considered that would result in more reliable results
- To further study the efficiency of NS, different mixes should be prepared with different percentages of NS particles. Different admixtures should also be added to NS particles as a replacement of cement
- Though a percentage of 1.5 was found to be optimum when it comes to compressive and flexure strengths, a higher percentage resulted in better chemical properties such as lower permeability

and higher chemical resistance, thus, it is recommended that percentages are increased in case the main objective is not higher strengths but better chemical properties
- Different cement types should be tested with NS to check if results would follow different patterns than the ones obtained.
- Efficiency of NS addition to concrete is recommended to investigate if results follow the same patterns acquired through testing on mortar

7 Recommendations for Future Applicators:

It is recommended that the NS mortar prepared is used for the following applications:

- Chemical resistance paste:
  Results show high chemical resistance properties of Nano Silica mortar, so it is preferred to be used as a material covering concrete parts that are most likely to be aggressively attacked by chemicals
- Injection for cracks:
  It is recommended that this mortar be used for crack injection for structures subjected to probable chemical attacks especially sulphate.

8 Acknowledgment:

- Dr. Mohamed Nagib Abou-Zeid, Thesis Advisor and Dean of the School of Science and Engineering
- Dr. Mohamed Abdelraouf, Thesis Advisor
- Dr. Tamer Shoeib, Chemistry Department Chair
- Dr. Wael Mamdouh, Chemistry Department Professor
- Eng. Amr Fathy, Thesis TA
- All AUC's Research Center (STRC) assistants

9 References:

- Yusak, Mohd Ibrahim. "Utilization of Nano Silica as Cement Paste in Mortar and Porous Concrete Pavement." *ResearchGate*. Trans Tech Publications, Switzerland, July 2015. Web. 12 Feb. 2016.
- S. GOPINATH, P.CH. MOULI, A.R. MURTHY, N.R. IYER, S. MAHESWARAN, P.Ch. Mouli, A.R. Murthy, N.R. Iyer3, and S. Maheswaran. "Effect of Nano Silica on Mechanical Properties and Durability of Normal Strength Concrete." AUC Libraries Article Finder. Archives of Civil Engineering, n.d. Web. 14 Feb. 2016
- Heikal, Mohamed, S. Abd El Aleem, and W.m. Morsi. "Characteristics of Blended Cements Containing Nano-silica." HBRC Journal 9.3 (2013): 243-55. Web.
- Gupta, Sakshi. "Application of Silica Fume and Nanosilica in Cement and Concrete – A Review." Journal on Today's Ideas - Tomorrow's Technologies JOTITT 1.2 (2013): 85-98. Web.
- Jafari, Vajihe, and Ali Allahverdi. "Synthesis of Nanosilica from Silica Fume Using an Acid-base Precipitation Technique and PVA as a Non-ionic Surfactant." Ultra Fine Grained and Nanostructured Materials 72 (2014): 105-12. Web.
- Taurozzi, J.s., V.a. Hackley, and M.r. Wiesner. "Preparation of Nanoparticle Dispersions from Powdered Material Using Ultrasonic Disruption - Version 1.1." (2012). Web.
- Reddy, T. Ashwini, Madan Mohan, and Sreena Reddy. "Effect of Nano Silica on Mechanical Properties of Conventional Concrete: A Case Study." International Journal of Applied Sciences, Engineering and Management ISSN 2320-3439 03.06 (2014): 36-39. Web.