

AUT Journal of Civil Engineering

AUT J. Civil Eng., 4(4) (2020) 487-504 DOI: 10.22060/ajce.2020.16811.5603

Investigation on the Effect of Addition of Nano Alumina, Nano Silica, Nano Titania, and MWCNTs on Flexural and Compressive Strengths of Cement Mortar

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ABSTRACT: Being at the threshold of a revolution in nanotechnology, new advanced materials with higher knowledge contents, new functionalities, and improved performances are increasingly critical for industrial competitiveness and sustainable development. The current experimental research would focus on developing a new cement mortar material by partial replacement of cement with nanoparticles. Special concentration on controlling and presenting cement mortar flow rate according to ASTM C 1437, and also a comparison of mechanical performances of three Nanopowder and the Nanotube materials, could be considered as a distinctive and innovative part of this research. Such nanomaterials are the most useful ones with the most integrating effects. In this investigation, 60 prism specimens in four series each consisting of four specimens with nanomaterials and one benchmark, were prepared and molded-in triple-gang molds. Bending and compressive tests were conducted on the specimens at the age of 7, 28, and 90 days according to ASTM C348 and 349 standards. The results depicted that the best performances of investigated nanoparticles in increasing flexural and compressive strengths would occur in the early 7 days. Also, the diagrams indicated that specimens with 4% nano-silica provided up to 61% growth in 7 days compressive strength and 34% growth in 7 days flexural strength in comparison to average strengths of benchmark specimens. Thus, it can be recommended as the optimum mixing percentage of nanoparticles. Also, multi-walled carbon nanotubes, MWCNTs, showed acceptable performance in increasing the strength. Nano titania and nano alumina exhibited approximately neutral or negative effects on flexural and compressive strengths. The most important challenge in this study would be a dramatic decrease in the activity of nanoparticles in ages between 7 and 90 days.

1-Introduction

Nanotechnology is of global interest because of its high potentiality. It has been attracted more public funding than any other area of technology. It is also one of the areas of research that is truly multidisciplinary. Nanotechnology involves the manipulation of matter of nanometer length scales to produce nanomaterials, structures, and devices [1].

The related studies due to the contribution of nanotechnology to new products and processes have led to the application of nanoparticles in concrete, cement mortars, and cement pastes. Such materials are the most useful ones in construction [2-15].

Jo, et al. [16] compared the effects of nano-silica and silica fume in cement mortars. The first series of specimens contained 3, 6, 10, and 12% of nano-silica, and the second series of specimens contained 5, 10, and 15% of silica fume. The results showed that nano-silica had a better effect than micro silica and the best performance was related to the specimen containing 12% nano-silica with increasing compressive strength by 177% and 168% for ages of 7 and 28 days.

Review History:

Received: Jul. 27, 2019 Revised: May. 09, 2020 Accepted: Jun. 24, 2020 Available Online: Jul. 12, 2020

Keywords:

Nano alumina Nano-silica Nano-titania Multi-Walled Carbon Nanotubes Cement Mortar Mechanical Properties

Nazari, et al. [17] investigated the effect of the addition of nano alumina with an average diameter of 15 nm on concrete as a partial replacement of cement. They conducted a compressive strength test on cubic specimens with a dimension of 10 cm with 0.5, 1, 1.5, 2% contents of nano alumina. The results indicated that the best compressive strength performance was related to the specimen containing 1% nano alumina that led to enhancement of compressive strength up to 17% and 9% respectively for ages of 7 and 90 days.

Sadrmomtazi and Fasihi [18] studied cement mortar specimens that had nano-silica as a portion of cement. The result of the compressive test of cubic specimens with a dimension of 5 cm, showed that the best compressive performance was related to the specimen containing 9% nano-silica that caused a compressive strength increase of 76% for the age of 7 days and 35% for 90 days.

Ltifi, et al. [19] investigated cement mortar specimens containing 3 and 10% nano-silica with 9nm diameter as a portion of cement. They were molded in 4cm cubic specimens and cured for 28 days. It was observed that

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specimens containing 10% NS had a better effect than benchmark specimens. The increasing strength was 56% and 10% respectively for ages of 7 days and 28 days.

Shekari and Razzaghi [20] evaluated the influence of NA, NF, NS, and NT with a diameter of 10 to 25 nm on high performance concrete. They prepared 15cm cubic specimens whose cement materials contained 1.5% nanoparticles and 15% Metakaolin. They reported that nano alumina had a better effect than other nanoparticles with increasing compressive strength up to 55% for the age of 28 days.

Collins, et al. [21] studied the use of 0.5% MWCNTs in cement pastes. The results of compressive tests on 5*10 cm cylindrical specimens specified that the addition of MWCNTs had a positive effect on specimens that contained superplasticizers for better dispersion. Also, they reported that compressive strength was increased by 26% for the age of 28 days.

Stefanidou and Papayianni [22] researched cement paste that contained nano-silica with an average diameter of 14 nm. Prism specimens including 0.5, 1, 1.5, and 2% nano-silica as a portion of cement were molded in 2.5*2.5*10 cm molds. They found that specimens with 0.5% nano-silica showed the best compressive performance by enhancing compressive strength approximately 36% and 100% for the age of 7 and 28 days. Also, the specimen containing 2% nano-silica exhibited the best flexural performance by increasing the strength up to nearly 12% and 21% for the ages of 7 and 28 days.

Liu, et al. [23] studied cement mortar specimens containing 0.02, 0.08, 0.1, and 0.2% of CNTs. The results from compressive and flexural tests on 4*4*16cm prism specimens specified that specimen containing 0.08% CNT had a better effect in comparison to the benchmark and resulted in an 18% and 19% increase in compressive and flexural strengths for the age of 90 days.

Haruehansapong, et al. [24] investigated the effect of particle size of nano-silica including 12nm, 20nm, and 40nm, on compressive strength of cement mortar specimens. They measured compressive strength in specimens containing 3, 6, 9, and 12% of nano-silica. The outcome after 28 days curing indicated that specimens containing nano-silica with the dimension of 40nm had better results and compressive strength increased by 74% and 54% for 7 and 28 days respectively.

Salemi, et al. [25] explored the influence of the addition of nano alumina and nano titania with an average diameter of 8 and 15 nm for concrete. They coordinated tests on 10 cm cubic samples containing 2% of nanoparticles. The results indicated that the best compressive performance was related to the specimen containing 2% of nano titania which led to improvement of compressive strength up to 12% and 27% respectively for ages of 7 and 120 days.

Naji Givi, et al. [26] researched the effect of the lime solution on 10 cm cubic concrete specimens containing 0.5, 1, 1.5, and 2 % nano-silica with an average diameter of 15nm as partial replacement of cement in two series. The first series of specimens were cured in water and the second one in a lime solution. The 7 and 90 days compressive results showed

that in the first series, specimens containing 1% nano-silica demonstrated better results including 21 and 13% growth in compressive strength. In the second series, specimens containing 2% of nano-silica demonstrated better results including 56 and 42% growth in compressive strength.

Cao, et al. [27] investigated the influence of cellulose nanocrystal (CNCs) additions on the performance of cement paste. Mechanical tests showed an increase in flexural strength of about 30% with only 0.2% of CNCs. Isothermal calorimetry (IC) and thermogravimetric analysis (TGA) show that the degree of hydration (DOH) of the cement paste is increased when CNCs are used.

Wang, et al. [28] examined the effects and mechanisms of nanofillers on the bond strength and interfacial microstructures between aggregates and cement mortars. The experimental results indicated that all types of investigated nanofillers (NT, NS, and NZ) can enhance the bond strength between aggregates and cement mortar.

By reviewing previous researches in the field of application of nanoparticles in cementitious mixtures including concrete, cement mortar, and cement paste, it can be concluded that all researchers by scattering results have made effort to complete this complex puzzle. Therefore, the current research aims to investigate the effect of the addition of nanoparticles on cementitious mortar mixtures considering different aspects.

Special observation has been carried out on controlling and presenting cementitious mortar flow rate according to ASTM C 1437. Also, a simultaneous comparison between three nanopowder and the nanotube materials has been performed considering the mechanical characteristics. The current experimental examinations could be considered as a distinctive and innovative part of this research compared to the previous related ones.

The abbreviations for nanoparticles that were used in this paper would be presented as follows:

Nano Alumina-Al $_2O_3$ (NA) - Nano-silica-SiO $_2$ (NS) Nano Titania-TiO $_2$ (NT) – Multi Wall Carbon Nano Tubes (MWCNTs or CNT).

2- Materials and experimental programs

2.1. Materials

Ordinary Portland cement (OPC) complying with requirements of ASTM C150 standard was used in this research as binder material. The Chemical analysis is summarized in Table 1.

Sand with greater grading was selected for controlling workability and diminishing superplasticizer consumption. Therefore natural silica sand complying with the DIN EN-196 standard was selected instead of the ASTM C778 standard. The grading of DIN EN-196 Sand is presented in Table 2.

Polycarboxylate-based superplasticizer was employed for controlling the flow rate and makes the better dispersion of nanoparticles in the cement matrix.

The physical and chemical properties of nanoparticles are summarized in Table 3. TEM and SEM images provided by the manufacturer are presented in Fig.1.

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Chemical analysis	Cement (Wt. %)
SiO ₂	22.44
Al_2O_3	4.95
Fe ₂ O3	4.13
CaO	63.76
MgO	1.16
SO_3	2
K_2O	0.68
Na ₂ O	0.36
LOI	2.34

Table 1. Chemical analysis of OPC.

Table 2. DIN EN-196 Sand grading.

Square Mesh Size (mm)	Cumulative Retained (%)
0.08	99
0.16	87
0.50	67
1.00	33
1.6	7
2.00	0

Table 3. Physical and chemical properties of nanoparticles.

Nano Type	APS (nm)	SSA (m²/gr)	Density (g/cm ³)	Purity (%)
SiO ₂	11-13	200	2.2 <0.1 B	99+%
Al ₂ O ₃ (Gamma)	20	138	3.89	99+%
TiO ₂ (80%Anatase)	20	10-45	3.94 0.46 B	99+%
MWCNT	10-30	270	_	>95+%
B: Bulk Density	*MWCNTs Length: 10µm			



Fig. 1. SEM and TEM Images of nanopowders (a) NT (b) NA (c) NS.

Mixture Code	Sand (g)	Water (g)	Cement (g)	NA (g)	SP (%)
Bench-NA	1350	245.5	490.9	0	0.04
NA1	1350	245.5	486.0	4.91	0.19
NA2	1350	245.5	481.1	9.82	0.33
NA3	1350	245.5	476.2	14.73	0.44
NA4	1350	245.5	471.3	19.64	0.49

Table 4. Mix proportion of NA contained specimens

Table 5. Mix proportion of NS contained specimens

Mixture Code	Sand (g)	Water (g)	Cement (g)	NS (g)	SP (%)
Bench-NS	1350	245.5	490.9	0	0.06
NS1	1350	245.5	486.0	4.91	0.41
NS2	1350	245.5	481.1	9.82	0.73
NS3	1350	245.5	476.2	14.73	1.04
NS4	1350	245.5	471.3	19.64	1.77

Table 6. Mix proportion of NT contained specimens.

Mixture Code	Sand (g)	Water (g)	Cement (g)	NT (g)	SP (%)
Bench-NT	1350	245.5	490.9	0	0.03
NT1	1350	245.5	486.0	4.91	0.07
NT2	1350	245.5	481.1	9.82	0.09
NT3	1350	245.5	476.2	14.73	0.13
NT4	1350	245.5	471.3	19.64	0.18

Table 7. Mix proportion of MWCNTs contained specimens.

Mixture Code	Sand (g)	Water (g)	Cement (g)	CNT (g)	SP (%)
Bench-CNT	1350	245.5	490.9	0	0.05
CNT1	1350	245.5	490.4	0.49	0.09
CNT2	1350	245.5	489.9	0.98	0.14
CNT3	1350	245.5	489.4	1.47	0.19
CNT4	1350	245.5	488.9	1.96	0.23

2.2. Specimen preparation

The water to binder (The sum of cement and nanoparticles) ratio of all mixtures was considered as 0.5. Tables 4-7 illustrate the mixture proportion of mortar specimens containing different nanoparticles and also the percentage of superplasticizer usage by the weight of the binder. Weights of the raw materials are according to the below mixture proportions:

In the first step, nanoparticles were stirred with a portion of water in the Hamilton Beach instrument at a high speed during 5-6 minutes as shown in Fig.2. Then the remained water was added to Hamilton Beach and they were started to mix at a slow speed to prevent splashing from the bowl for 1-2 minutes. It shall be noted that the temperature of the water was controlled in the standard range $(23^{\circ}C\pm3)$ during this research.



Fig. 2. Mixing nanoparticles and water.

Dry materials were mixed by an electrically driven mechanical mixer for 30 s. Then the water and nanoparticle solution from the Hamilton beach bowl was added to the mixer bowl and mix with dry materials for 30 s at high speed. Afterward, the mortar was rested for 90 s. Next, a polycarboxylate-basedsuperplasticizerwasaddedtothemortar.

For determining the workability and controlling the flow of mixtures in the standard range as mentioned in ASTM C 348, the flow of mixtures was determined by using a flow table conforming to ASTM C230 as shown in Fig.3.

A layer of mortar of about 25mm in thickness was placed inside the mold and tamped 20 times then the mold was filled, and the second layer was tamped similar to the first layer. Then, the mortar was cut off to a plane surface and the mold lifted away from the mortar 1 min after completing the mixing operation. Immediately, the table was dropped 25 times in 15 s. Mortar flow was determined by measuring the diameter of the mortar along the four lines.

The flow rate of $110 \pm 5\%$ should be obtained, and if not the previous steps should be repeated. Thus, the

percentage of added superplasticizer would be changed.

In the next step, the specimens were constructed according to ASTM C 348 standard as shown in Fig.4. The curing of the specimens for 7, 28, and 90 days is shown in Fig.5.

2.3. TEST METHOD

2.3.1. Flexural test

After 7, 28, and 90 days of curing of specimens under ASTM C 348, immediately following to removal of specimens from storage water they were wiped to a surfacedry condition and were removed of any loose sand grains or incrustations from the faces that would be in contact with the bearing surfaces of the points of support and load application.

Hereafter, specimens were loaded with a flexural testing device with a load rate of 4.5 Kg/s as shown in Fig.6. The load was applied at a uniform rate to prevent shock and normal to the loaded surface of the specimen in such a manner as to avoid all eccentricity of loading.



Fig. 3. Determination of Flow rate



Fig. 4. Molding of prism specimens.

2.3.2. Compressive test

Parts of fractured prism specimens in bending with a length of not less than 65 mm, were tested under compression based on ASTM C 349 with a load rate of 140 Kg/s.

2.3.3. Scanning Electron Microscopy (SEM)

Selected samples after being sputter-coated with a layer of gold were tested by using a scanning electron microscope at Amirkabir University of Technology for investigating dispersion of nanoparticles as shown in Fig.7.



Fig. 5. Curing of the specimens.



Fig. 6. Breaking specimens.

3- Results

3.1. Flexural strength

Obtained results from flexural strength tests are presented in Tables 8-11 and Fig.8-11.

3.2. Compressive Strength

Obtained results from compressive strength tests are presented in Tables 12-15 and Fig.12-15.



Fig. 7. SEM Testing apparatus.

Mixture Code	Flow (%)	Flexural Strength (MPa)			
		7 D	28 D	90 D	
B-NA	111	6.5	6.5	6.6	
NA1	112	5.4	5.9	5.5	
NA2	114	5.0	5.1	5.7	
NA3	113	4.7	5.4	5.4	
NA4	108	5.0	5.4	5.1	



Fig. 8. Flexural strength of NA contained specimens.

Mixture Code	Flow (%)	Flexural Strength (MPa)			
		7 D	28 D	90 D	
B-NS	115	5.6	6.4	6.4	
NS1	109	5.5	5.5	5.7	
NS2	106	7.1	7.2	6.5	
NS3	115	6.5	5.7	6.2	
NS4	113	7.6	7.7	7.3	





Fig. 9. Flexural strength of NS contained specimens

Table 10. Flexural stre	ngth of NT	contained	specimens.
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Mixture Code	Flow (%)	Flexural Strength (MPa)			
		7 D	28 D	90 D	
B-NT	111	5.9	6.6	6.7	
NT1	111	6.0	7.2	7.5	
NT2	107	6.3	6.5	6.8	
NT3	106	5.8	6.9	6.9	
NT4	106	6.1	5.8	6.3	



Fig. 10. Flexural strength of NT contained specimens.

Mixture Code	Flow (%)	Flexural Strength (MPa)		
		7 D	28 D	90 D
B-CNT	113	4.9	6.3	6.3
CNT 0.1	108	6.7	7.4	7.2
CNT 0.2	107	6.7	7.2	6.6
CNT 0.3	110	6.8	7.5	7.4
CNT 0.4	113	7.0	6.8	7.4





Figure 11. Flexural strength of CNT contained specimens.

Mixture Code	Flow (%)	Compressive Strength (MPa)		
		7 D	28 D	90 D
B-NA	111	31.3	39.0	44.3
NA1	112	30.8	35.3	34.5
NA2	114	28.9	33.0	34.8
NA3	113	27.7	28.9	30.7
NA4	108	27.4	30.7	32.3

Table 12. Compressive strength of NA contained Specimens..



Fig. 12. Compressive strength of NA contained Specimens.

Mixture Code	Flow (%)	Compressive Strength (MPa)		
		7 D	28 D	90 D
B-NS	115	29.2	35.1	41.1
NS1	109	32.2	35.8	37.5
NS2	106	43.9	47.8	49.5
NS3	115	38.4	41.1	42.4
NS4	113	49.7	55.0	55.1





Fig. 13. Compressive strength of NS contained Specimens.

Table 14.	Compressive	strength of NT	contained Specimen	s.
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Mixtura Cada	Flow	Compressive Strength				
Mixture Coue	(%)	(MPa)				
		7 D	28 D	90 D		
B-NT	111	32.7	38.7	44.1		
NT1	111	33.7	39.7	43.1		
NT2	107	33.4	28.3	42.3		
NT3	106	33.2	39.8	41.7		
NT4	106	33.5	38.9	40.1		



Fig. 14. Compressive strength of NT contained Specimens.

Table 15. Compressive strength of CNT contained Specimens.

Mixture Code	Flow (%)	Compressive Strength (MPa)		
		7 D	28 D	90 D
B-CNT	113	27.8	34.1	39.0
CNT 0.1	108	38.1	43.8	46.6
CNT 0.2	107	38.4	44.3	48.2
CNT 0.3	110	39.5	45.5	48.6
CNT 0.4	113	40.4	44.2	49.1



Fig. 15. Compressive strength of CNT contained Specimens.

4-Discussion

It can be concluded that the addition of nanoparticles would increase water absorption and change the speed of hydration [27] which results in producing compacter microstructure and reducing porosity than benchmarks. However, the performances of nanoparticles are different from each other. Therefore, the discussion regarding such differences is presented as below:

4.1. Flexural Strength

To have a better comparison, linear diagrams according to Fig.16 and Fig.17 are presented which would enable the reader to observe at a glance the investigated effect of nanoparticles on flexural strength of cementitious mortar specimens for 7 to 90 Days.



Fig. 16. Variation of 90 days flexural strengths in comparison to the benchmark.



Fig. 17. Variation of 7 days flexural strengths in comparison to the benchmark.



Fig. 18. Variation of 7 days compressive strengths in comparison to the benchmark.



Fig. 19. Variation of 7 days compressive strengths in comparison to the benchmark.

It was concluded that nanoparticles had the maximum flexural strength performance in the first 7 Days. After that, flexural strength would increase with a slow slope. Figures show that specimens containing 4% and 2%

nano-silica have the best flexural performances in 7 days, for which the strength increased up to 34% and 24%, in comparison to benchmark specimens respectively.

The other point that could be concluded in these figures is the negative effect of nano alumina on flexural strength. Specimen containing 4%NA demonstrated the weakest flexural performances in this series by decreasing strength up to 24%. The breaking point observed in the nano-silica diagram in a specimen containing 3% nano-silica, refers to the importance of mortar flow. As an explanation, a higher flow rate (115%) in this specimen in comparison to the previous specimen containing 2% nano-silica, has led to such a break in the diagram.

4.2. Compressive Strength

For this comparison, linear diagrams are presented in Fig.18 and Fig.19 which would depict at a glance the effect of investigated nanoparticles on compressive strength of cementitious mortar specimens in 7 to 90 Days.



Fig. 20. SEM images taken from nano contained specimens (a) MWCNTs (b) NA (c) NS (b) NT.



Figure 21. Percentage of superplasticizer consumption in nanoparticle contained specimens.

Similar to the discussion for flexural strength improvement, it has been found that nanoparticles had the maximum compressive strength performance in the first 7 Days. After that, the strength would increase with a slow slope.

Figures show that specimens containing 4% and 2% nanosilica and 0.4% CNT, had the best compressive performances in 7 Days which increased strength up to 61%, 43%, and 31% in comparison to benchmark specimens respectively.

Identical to the discussion proposed before, nano alumina particles present a negative effect on the compressive strength.

4.3. Microstructure

Analysis and interpreting of SEM images of cement mortars are very complex. However, such images could be used for observing the dispersion of nanoparticles in the cement matrix. In this research, samples were sputter-coated with a gold layer and were examined using a scanning electron microscope at the Amirkabir University of Technology.

Figure 20 shows SEM images taken from specimens

containing 0.4% CNT, 4% NA, and 4% NS, 4% NT which had the most percentage of replacement with cement in this research. The observation indicates that mechanical stirring and addition of polycarboxylate SP could result in better dispersion of nanoparticles. However, agglomerations in some parts of cement mortars are not avoidable. This agglomeration cannot be related to mechanical stirring, since previously other methods such as ultra-sonication had the same outcome [21]. Also, highly attractive Van der Waals forces between the nanoparticles would cause the creation of coherent agglomerates.

4.4. Superplasticizer consumption

As previously mentioned, for controlling cement mortar flow rate in the standard range, constant W/C ratio was considered as 0.5 and tried to control flow rate by addition of polycarboxylate based Superplasticizer. The rate of superplasticizer consumption for different nanoparticles is presented in Figure 21. It is noted in Figure 21 that nano-silica consumed the most superplasticizer in comparison to other investigated nanoparticles in this research. The approximately linear ascend in consumption of SP by increasing nanoparticle addition is observed.

Also, it should be considered that for MWCNTs despite their low percentage of usage in cement mortars, considerable SP consumption would be observed.

5- Conclusions

This paper reports the results of investigations of the addition of three types of nanopowders (NA-NS-NT) and one type of nanotube (MWCNTs) on the mechanical properties of cement mortars. Therefore based on these results below conclusions can be drawn:

- 1)It is found that the most activity of nanoparticles occurred in the first 7 days of curing and afterward dramatic decrease in the activity of nanoparticles between the ages 7 to 90 days could be observed. Such a phenomenon could be resulted from the slower growth in strength of 28 days and 90 days in comparison to the benchmark. Also, it can be concluded that nanoparticles with the increasing speed of hydration cause earlier strengthening.
- 2)The best mechanical performances in flexural and compressive strength can be presented as below respectively:
- a).Nano-silica: This nanoparticle had the best mechanical performance among investigated nanoparticles. The best performance was related to the specimen containing 4% NS which improved flexural and compressive strength up to 10% and 34% in 90 days respectively.
- b).MWCNTs: This nanoparticle despite a lower percentage of usage was compared with other investigated nanoparticles in the current research. It exhibited acceptable mechanical performance. The best performance in this series of specimens was related to the specimen containing 0.4% CNT by increasing flexural and compressive strength up to 10% and 16% at the age of 90 days.
- c)Nano titania: This nanoparticle presented slight enhancement in 7 days flexural and compressive strengths. however, it can be observed that a dramatic decrease in 90 days strength was seen, especially for the specimen containing 4% NT for which the decrease of compressive strength up to 30% in comparison to benchmark was recorded. It shall be noted that specimens containing 1% NT exhibited the best performance in this series, by increasing flexural and compressive strength up to 14% at the age of 90 days.
- d). Nano alumina: This nanoparticle showed the weakest mechanical performance among the investigated nanoparticles. All specimens containing NA had a decrease in flexural and compressive strengths. The most reduction in strength occurred for specimens containing 3% and 4% NA.
- 3) As can be observed, for the specimen containing 3% nanosilica, flow rate plays a key role in the final mechanical properties of cement mortars in a way that leading to a dramatic change in flexural and compressive strengths.

- 4) SEM images show that specimens containing nanoparticles have compacter microstructure. Although, high-speed mechanical stirring and usage of superplasticizer would result in better dispersion of nanoparticles in the cement matrix. It cannot prevent agglomeration in some parts of cement mortars.
- The most superplasticizer consumption was related to specimens containing nano-silica. Also, superplasticizer consumption of CNT was considerable. Also, the lowest SP consumption was demonstrated by NT contained specimens.

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HOW TO CITE THIS ARTICLE

B. Akhoundi, A. Hojatkashani, Investigation on the Effect of Addition of Nano Alumina, Nano-silica, Nano Titania, and Mwcnts on Flexural and Compressive Strengths of Cement Mortar, AUT J. Civil Eng., 4(4) (2020) 487-504



DOI: 10.22060/ajce.2020.16811.5603

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