Silica nanoparticle size effect on mechanical properties and microstructure of cement mortar

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Abstract: In this study, the effects of micro and nano silica particle size and their quantity on mechanical properties and microstructure of fabricated cement mortar was investigated. The measurements carried out at the 7th day after the production of the cement mortar. The results showed that the cement mortar containing silica nanoparticles had better mechanical properties compared with the pure cement mortar. The production method used here caused a homogenous dispersion of silica nanoparticles in the cement mortar; therefore, its mechanical properties increased even with addition of only 1% silica nanoparticles. In the othr words the compressive strength increased by more than 100% by addition of critical amount of SiO_2 nanopowder. The SEM, DTA, TGA, water permeability tests revealed that nano and microparticles not only work as fillers for the cement mortar's porosity and improves its microstructure, but also reduce the amounts of $Ca(OH)_2$ and other calcium hydrates.
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1. Introduction

Nowadays the application of nanostructured materials in civil engineering is widely studied and contrary to the ordinary bulk materials, nanoparticles exhibit unique physical/chemical properties which is are mainly caused by their higher specific surface area. There are few reports in the use of nanoparticles in concrete. Recently, Qing et al. investigated the addition effects of silica nanoparticles and silica fume on mechanical properties of the hardened cement paste (Qing et al, 2007). It has been found that through increasing the amount of silica nanoparticles in cement its compressive strength increased, which is particularly significant in the early ages; however, an increase in the amount of silica fume brings a decrease and increase in the compressive strength of the cement in early ages and later ages, respectively (Qing et al, 2007). Investigations have shown that the bonding strength of paste-aggregate interface is more than both reference specimen and the specimens containing silica fume (Qing et al, 2007). Through studying the abrasion resistance of the concrete with TiO_2 and SiO_2 nanoparticles, Li et al. have recently found that the nanoparticles addition improved the abrasion resistance of the concrete significantly, and the abrasion strength improvement by TiO_2 nanoparticles was greater in comparison with the improved concrete by SiO_2 nanoparticles (Li et al, 2006). Moreover, the flexural fatigue performance of the concrete containing nanoparticles of SiO_2 and TiO_2 has also been studied by Li et al. (Li et al, 2007) who found out that the concrete with 1 wt% of TiO_2 nanoparticles has the best fatigue performance in comparison with the concrete containing polypropylene (PP) fibres. (Li et al, 2007). It has also been shown that the addition of silica nanoparticles to the concrete can improve the freezing resistance of the high performance concrete (Baomin et al, 2008). Nazari et al. have demonstrated that the compressive resistance of the concrete will be improved by adding ZrO_2 and TiO_2 nanoparticles up to 1% of the cement weight (Nazari et al, 2010 , Nazari et al, 2010). In another study Hui Li et al investigate the nanoparticles effect in microstructure of cement mortar. They showed that by adding the SiO_2 and Fe_2O_3 nanoparticles, the needle shape hydrates and large crystals of $Ca(OH)_2$ share decreased and the formed structure was more dense and compact(Li et al, 2004).

The recent studies have considered the effects of different weight percents of some nanoparticles such as SiO_2, TiO_2, ash, etc on concrete and cement mortar, while there are only few reports focused on the effects of nanoparticles size on mechanical properties of the cement mortar. The size effect of ash particles on cement mortar have been investigated by Lin et al.; they found that a sludge ash of greater fineness can improve the compressive strength of the mortar (Lin et al, 2008). However, the size effect of silica particles on mechanical properties of cement mortar has not been investigated yet.

This study aimed to investigate the optimum size and amount of silica particles added to cement mortar. In order to study the effects of the particle size, especially nanoparticle size on the mechanical properties (tensile, compressive, and flexural strengths) and the microstructure of the cement mortar with silica nano and micro particles, we applied different diameters of silica particles (12nm, 60 nm, and 1micrometer) with different weight percentage of 1, 3, and 5% to our basic cement mortar.

2. Material and Methods

2-1- Materials

The used Portland type II cement was chosen according to ASTM C150 (ASTM, 2005) standard. The silica nanoparticles used in this paper were bought from Skyspring Nanomaterials Inc. The characteristics of the silica nanoparticle were shown in Table 1. The super plasticizer was a commercial sulphonated melamine formaldehyde polymer manufactured by Vand chemie in Iran with relative density of 1.15. The content was adjusted for each mixture to ensure that no segregation would occur. Also, the distilled water was used for preparing all mixtures. Our utilized aggregate was the crushed silica sand with apparent density of 3.33g/cm^3 and the fineness modulus of 2.6. The sand was graded according to ASTM C33 (ASTM, 2007) standard. The largest diameter of these aggregate particles was 4.75mm.

Table 1. Characteristics of nano-SiO_2 particle

Item	Diameter	Specific surface area(m^2/g)	Density (g/cm^3)	Purity(%)
SiO_2	12nm	650	<0.14	99.9%
SiO_2	60nm	460-550	<0.14	99.9%
SiO_2	1µm	10	0.3-0.7	99%

2-2- Method

2-2-1- Mixing pattern

The mixing ratios of the ordinary cement mortar and the cement mortar containing nano and micro silica particles were shown in Table 2. The ratio of the water to cement compound (the cement and silica particles) was chosen 0.42. In this study the size of the used silica particles was 12-60 nm, and 1 micrometer by mixing portion of 1, 3, and 5 percentages.

2-2-2- Sample preparation

The high homogenous dispersion of silica particles strongly depends on stable suspension preparation. Hence silica nano powder was mixed with the distilled water and stirred for 6-10 hours by rotational speed of 250-300rpm. At first, the suspension of the silica particles and the superplasticizer were mixed in the mixer for 30 second, where the cement was added to this mixture simultaneously. Thereafter, the sand, from finest to coarsest, was added gradually to the mixture, and the mixing continued until the complete homogenization of the mixture. Then, the mortar was poured into the standard mold. In order to prepare the specimens for the flexural test, the mortar was poured into the molds with dimensions of 40×40×160 mm in two layers. Each layer was compacted by 15 impacts of a steel rod. For tensile test, the briquette specimens with 75×25×25 mm dimension were utilized.

Table 2. Mix proportions of specimens (kg/m^3)

Mixture type	Water	Cement	Sand	Nano -SiO_2	SP
CO	150	360	1800	—	—
1NS12	150	356.4	1800	3.6	4.8
3NS12	150	349.2	1800	10.8	6.08
5NS12	150	336.5	1800	23.5	7.36
1NS60	150	356.4	1800	3.6	4.48
3NS60	150	349.2	1800	10.8	5.76
5NS60	150	336.5	1800	23.5	6.72
1MS1	150	356.4	1800	3.6	3.84
3MS1	150	349.2	1800	10.8	4.48
5MS1	150	336.5	1800	23.5	6.08

*CO: Control.
*NS: cement mortar containing nano-SiO_2 (1%, 3%, 5%)
*MS: cement mortar containing micro-SiO_2 (1%, 3%, 5%)
*SP: superplasticizer

The mortar was poured in two layers, both of them compressed by 4 impacts of a steel rod. in order to prepare the specimens of the compressive and water permeability tests, the mortar poured into 50×50×50 mm cubic molds in three layers alternatively, which all layers compressed by 10 impacts of a steel rod. The molded specimens were covered with a plastic layer for 24 hours and then were cured in water at the room temperature up to end of the seventh day. Three specimens were prepared for each test and the average result was reported. The apparatus made by ELE Company, England was used for performing the mechanical tests. The microstructure of the specimens was studied by the scanning electron microscopy (SEM) Hitachi S-4160. The Thermo gravimetric Analysis (TGA) and Differential Thermal Analysis DTA thermal analyses were carried out by METTLER TOLEDO TGA/SDTA 851 by heating in the temperature range of 100-700 ˚C at the rate of 10˚C/min in the ambient atmosphere. Flexural tests were carried out according to ASTM C348 (ASTM, 2008). Compressive tests were carried out according to the ASTM C109 (ASTM, 2008) and tensile tests were carried out according to the ASTM C190 (ASTM, 1985).

3. Results

3-1- Mechanical properties

The mechanical tests (compressive, tensile, and flexural tests) carried out 7 days after the specimens were prepared, and the results are shown in Table 3. It can be inferred from the table that the addition of silica nano and micro particles has improved the mechanical properties of the cement mortar. By considering the mixing method and the homogenous dispersion of the particles in the cement paste, even by adding only 1% silica particles to the cement mortar, the mechanical properties of the cement mortar have considerably increased. The nano and micro silica particles added to the cement mortar act as chemical filler and improve the physical structure of the cement mortar. Also it is noticeable that these nanoparticles provide some nucleation sites for hydration products. Moreover, they react as a pozzolan with $Ca(OH)_2$ during the hydration of the cement which can probably strengthen the paste-aggregate bonding (Taylor et al, 1990, Zelic et al, 2000). As it is shown in Fig. 1, the cement mortar with silica nanoparticles exhibits superior mechanical properties than the cement mortar containing micro size silica particles. The specific surface area and the number of the surface atoms of the silica nanoparticles are more than the specific surface area and the number of the surface atoms which appear in micro silica particles. This higher number of the surface atoms in the nanoparticles translates into a larger number of free and unsaturated atomic bonds on the surface of the nanoparticles, which makes them thermodynamically unstable, as compared with the micro size silica particles. Furthermore, by reducing the size, the number of the surface atoms on a particle increases which leads to an increase in the appropriate surface area for chemical reactions (Zhang et al, 2002). Therefore, in comparison with the micro size SiO_2 particles, the nanoparticles have more surface energy and their surface atoms have strong reactivity, which makes the surface atoms easily react with the other external atoms in the surroundings. Also, in comparison to the micro size silica particles, the addition of the silica nanoparticles to the cement mortar can produce different effects on its mechanical properties. There is higher number of unsaturated atomic bonds of ≡Si- and ≡Si-O- on the surface of the nanoparticles, resulting the silica nanoparticles react strongly with $Ca(OH)_2$ in the following way:

≡Si-O- + H-OH→≡Si-OH Eq. (A.1)

≡Si- +OH→≡Si-OH Eq. (A.2)

≡Si-OH +$Ca(OH)_2$→C-S-H≡ [1] Eq. (A.3)

Table 3. Mechanical properties of the specimens

Mixture no	Compressive strength At the 7th day	
	Target (MPa)	Enhanced extent (%)
CO	11.42	0
1NS12	25.28	121.37
3NS12	28.613	150.55
5NS12	23.96	109.81
1NS60	24.27	112.49
3NS60	29.25	156.16
5NS60	23.19	103.09
1MS	20.64	80.73
3MS	16.55	44.89
5MS	22.82	99.82
Mixture no	**Tensile strength At the 7th day**	
	Target (MPa)	Enhanced extent (%)
CO	1.46	0
1NS12	2.25	54.54
3NS12	2.70	85.52
5NS12	1.50	2.971
1NS60	2.56	75.45
3NS60	3.02	107.11
5NS60	2.48	70.24
1MS	1.96	34.08
3MS	2.08	42.46
5MS	2.1	43.76
Mixture no	**Flexural strength At the 7th day**	
	Target (MPa)	Enhanced extent(%)
CO	1.81	0
1NS12	3.33	83.64
3NS12	4.08	125.19
5NS12	3.16	74.26
1NS60	3.59	98.16
3NS60	4.36	140.63
5NS60	3.47	91.18
1MS	3.47	91.18
3MS	3.13	72.43
5MS	3.8	109.38

Hence nano-silica can produce more nucleation sites for the hydration products, and has higher pozzolanic activity compared to the micro silica. Therefore, the addition of silica nanoparticles can enhance the mechanical properties and bond strength of the paste-aggregate interface, which improve the interface structure. According to investigation, the addition of a small amount of nano silica can enhance the stability and mechanical properties of cement mortar. As it is shown in Fig. 1, it was found that the specimens containing the silica nanoparticles with diameter of 60 nm exhibited higher strength than the specimens mixed by nanoparticles with 12 nm diameter. This can be described by the fact that the dispersion of the nanoparticles with higher specific surface area is more difficult than the bigger particles, and resulting them to proper agglomerate. These effects led to form the weak zone and forming the micro-cracks in high content, and smaller SiO_2 nanoparticles.

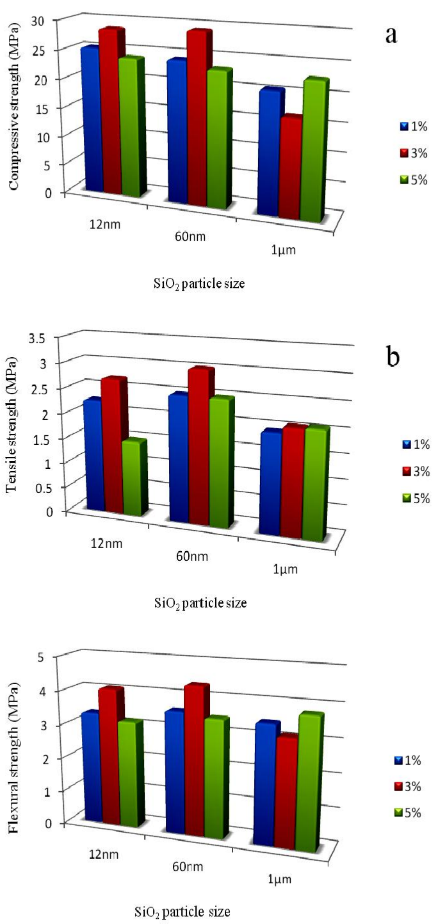

Figure. 1. Compressive strength (a), Tensile strength (b), and Flexural strength (c) of various M-NS particle size at different proportion of M-NS/C at the 7th day

It was evidenced that the mechanical strengths increased through increasing the amount of the silica nanoparticles up to 3%, and for higher values of the nanoparticles, the mechanical properties decreased. For the contents higher than 3%, the nanoparticles cannot easily disperse within the cement matrix, and due to their high surface energy, they become agglomerated, hence a weak area of empty spaces such as voids appeared. Consequently, the structure formed in such conditions cannot be homogenous and compacted. As it shows in Fig. 1a, in the sample with the SiO_2 particle size of 12nm and 60nm, the compressive strength at the portion of 3% nanoparticles is higher than other portions of additives, but the sample with the micro size of SiO_2 did not show this effect. This means that in the nano size particles of SiO_2 we need to find the optimum proportion of nanoparticle additive because of the surface interfering effect. In same way, the tensile strength of specimens improved by SiO_2 nanoparticles, highly depend on the optimum ratio of nanoparticles additives, but in the micro size of SiO_2 particles, by increasing the percentage, the tensile strength is less affected (Fig. 1b). The flexural strength of specimens shows same behavior as compressive strength because of same mechanism in particle bonding (Fig. 1c).

As it's shown in table 3, by adding the SiO_2 nanoparticles, the increasing ratio of compressive strength is higher than that of tensile and flexural strength. This fact is in compatibility with some similar works (Li et al, 2006). These results can be explained by appearing the fine micro-cracks in the cement mortar. As reported in some works (Li et al, 2006, Li et al, 2007), the effects of these micro-cracks on tensile and flexural strength are more considerable (Shuan et al, 2007). By increasing the SiO_2 nanoparticles up to 5 percent (series NS12 and NS60), the triple strength of specimens totally decreased, But in MS series, the triple strengths increase continually by increasing the SiO_2 micro particles ratio (table 3). This means that the increase of the SiO_2 particle size in the cement mortar shows less emphasis on the triple strength behavior which can be described by decreasing the total surface effect. According the findings at this work, the prepared cement mortar workability totally depends on SiO_2 particle diameter and applied amount. In other word, by increasing the SiO_2 nanoparticles more than the critical percentage and decreasing the SiO_2 particle size less than the critical percentage, the cement mortar workability decreased and the micro-cracks start to rise and weaken the mechanical properties of the cement mortar. Hui Li et all showed that by applying the TiO_2 nanoparticls in concrete, by increasing the amount of used nanoparticles, the appeared micro-cracks was increased and workability obviously fell down (Li et al, 2006, Li et al, 2007). As it shown in table 3, by increasing the SiO_2 nanoparticles in 5NS12 and 5NS60 series up to 5%, the agglomeration effect beside the increase of micro-cracks, weaken the triple strength specially the flexural and tensile strength which highly depend on micro-cracks in cement mortar. In the MS series, by increasing the SiO_2 particles up to 5%, the triple strengths were totally increased because of less surface effects on agglomeration probability and workability decrease.

3-2- The microstructure of the specimens

Fig. 2 shows the SEM images of used nano and micro silica particles to investigate the mechanisms predicted by the mechanical properties and water permeability. The addition of the silica particles exerted influence on the hydration, and introduced some changes in the microstructure of the cement mortar. The silica particles act not only as chemical filler to improve the microstructure of the cement mortar but also as promoter for the pozzolanic reaction (Jo et al, 2007). Therefore, the addition of the silica particles can be effective in enhancing the performance of the cement mortar. As it can be seen from SEM images shown in Fig. 2, the silica particles filled the vacancies between the cement aggregates, and thus, increased the compactness of the cement mortar. It also could be found out that the specimen with 60nm particle size of SiO_2 exhibited the best mechanical properties among the other specimens. It showed more homogenous, compacted and uniform structure (Fig. 2c). Moreover, Fig. 2.d indicates that the compactness of the microstructure decreased by increasing the particles size, due to a decrease in specific surface area and reduced pozzolanic activity.

3-3- XRD results

Fig. 3 shows the XRD results of manufactured specimens in constant portion of 3% micro and nano SiO_2 with different SiO_2 average particle size of 12nm, 60 nm, and 1 micrometer in cement mortar. As it is shown in Fig. 3 the intensity of the peaks corresponding to $Ca(OH)_2$ crystals drastically decrease when micro and nano SiO_2 particle are added to the aggregate of cement mortar. It also shows that $Ca(OH)_2$ intensity drop by decreasing the particle size of SiO_2 from 12 nm to 60 nm. This can be described by hydration of $Ca(OH)_2$ to C-S-H crystals. By increasing the particle size of SiO_2 to one micrometer, the corresponding peak to $Ca(OH)_2$ appeared again which can be interpreted that the additive could not participate in transition of $Ca(OH)_2$ to C-S-H crystals.

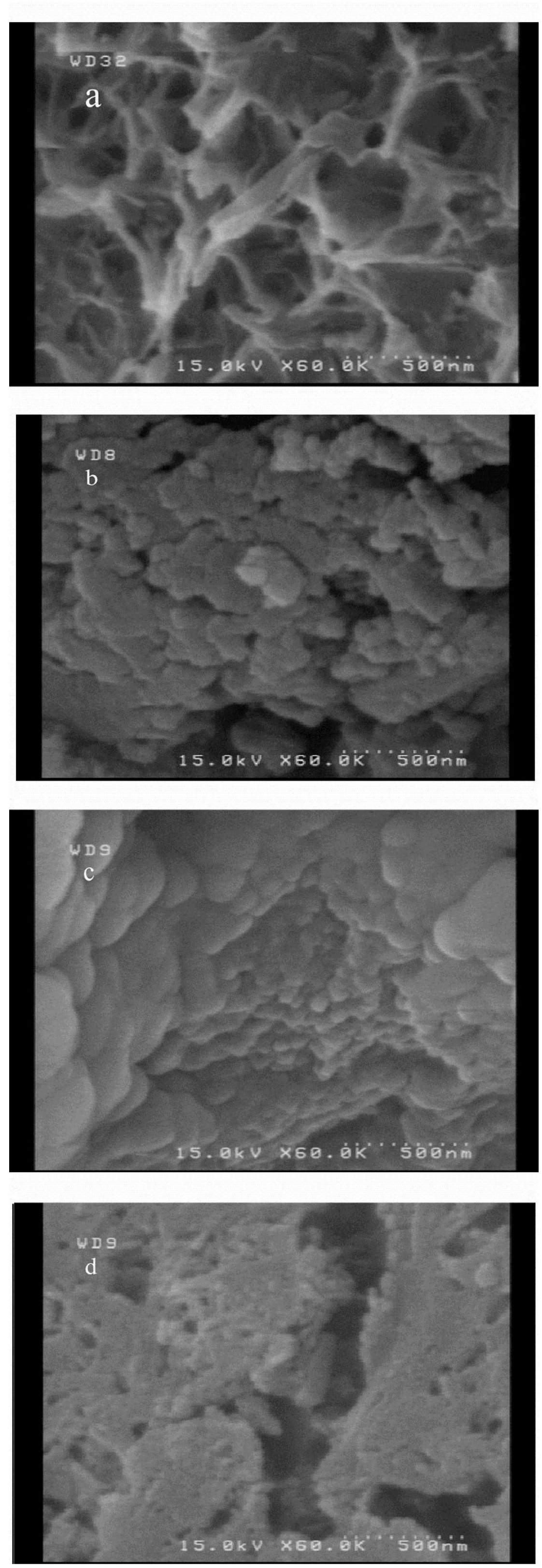

Figure. 2. The FE-SEM image of (a) CO specimen, (b) 3NS12 specimen, (c) 3NS60, (d) 3 MS

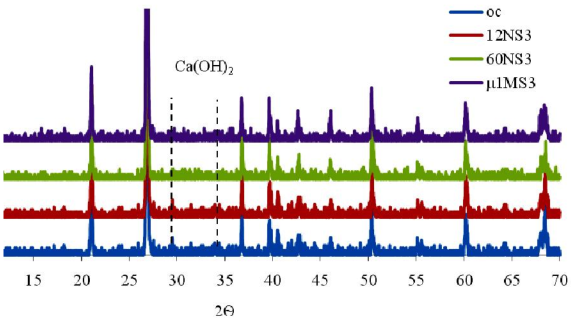

Figure. 3. XRD of CO specimen, 3NS12 specimen, 3NS60, 3 MS

As it mentioned before, this type of transition improve the mechanical properties of fabricated specimens at this work because of decreasing the $Ca(OH)_2$ needle shape structure to C-S-H crystals (Li et al, 2004) . As it is shown in XRD Figure, the addition of SiO_2 nanoparticle with 12 nm, decrease the $Ca(OH)_2$ needle shape intensity peak less than when SiO_2 nanoparticles with 60 nm are applied, which is because of nanoparticle agglomeration. This event does not allow the nanoparticles to disperse homogenously in the mortar and prevent the participation in hydration reaction.

3-4- Water permeability test

To investigate the permeability of prepared specimens, by demoulding the cement mortar, they cured in water for 5 days, and dried at the ambient atmosphere for 2 days. Then, the specimens were weighed (W_{dry}); and then submersed in water for other 24 hours, and then, weighed again (W_{wet}). The percent of the water absorption (permeability) was calculated by the following formula:

$$P\% = \frac{W_{wet} - W_{dry}}{W_{dry}} \times 100$$

The results were shown in Table 4. As it can be seen, the maximum and the minimum permeability are corresponding to the ordinary cement mortars without silica particles and 3NS60 specimen respectively. The SEM images of improved specimens also confirm these results (Fig. 4). Consequently by more compactness caused by the SiO_2 nanoparticles filling effect and C-S-H production from $Ca(OH)_2$, the water permeability obviously decreased. Two major parameters affect directly in water permeability; the porosity and surface energy. The porosity of manufactured specimens deceased by adding the SiO_2 nanoparticles and consequently did the water permeability. The surface energy introduces the surface hydrophilicity ability which corresponds to photocatalytic activity of nano SiO_2 particles by using the UV light to excite electrons from valence bond to conduction band. Considering the low UV irradiation to surface and existing the more harmful ions in mortar, the photocatalytic properties seem to play a less important role in hydrophilicity. Hence at this case, the porosity plays the main role in water permabeality of prepared mortar. The specimen containing 3% silica nanoparticles exhibited the highest resistance against permeability among the other specimens, as the well as superior mechanical properties obtained through tensile, compressive, and flexural tests. This can be described by agglomeration tendency of SiO_2 nanoparticles and consequently high viscosity of molded mortar in quantity up to 3%. In quantities less than 3% SiO_2 nanoparticles, the acquired amount to fill and participate in hydration reactions was not enough.

Table 4. Water permeability properties

Mixture no	Permeability (%)
CO	4.23
1NS12	2.26
3NS12	2.09
5NS12	2.64
1NS60	1.84
3NS60	1.62
5NS60	3.4
1MS	1.84
3MS	2.43
5MS	2.03

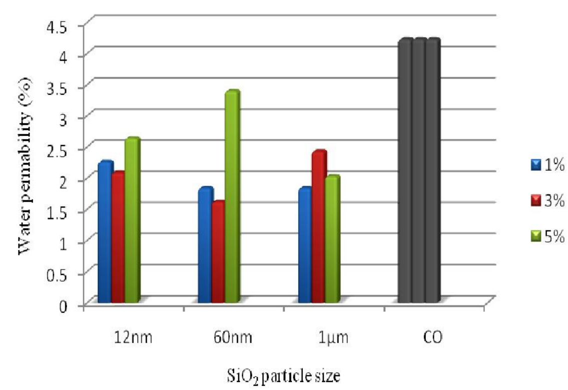

Figure. 4. Water permeability of specimens with various M-NS particle size and different percentage of M-NS particles.

3-5- TGA and DTA tests

Figures 5 and 6 shows the DTA and TGA graphs of the ordinary cement mortar and 3NS60 specimen. Two peaks are shown at the temperatures of 430˚ C and 574˚ C in the DTA graph. The appeared peak at 430˚ C corresponds to the dehydration of $Ca(OH)_2$ (Vedalakshm et al, 2003). According to Fig. 5, the reaction enthalpy of the ordinary cement mortar has been reduced by adding the silica nanoparticles. The amount of $Ca(OH)_2$ was estimated by he measured weight reduction from the TGA graphs. The weight reduction of the ordinary cement mortar and the one containing silica nanoparticles were 1.14% and 0.42% respectively. As it is evident, the peak has been reduced by adding the nanoparticles, which means that the presence of the silica nanoparticles reduced the amount of $Ca(OH)_2$ during the formation of the cement paste. Therefore, the silica nanoparticles decrease the growth of $Ca(OH)_2$ crystallites, which consequently increases the mechanical properties of the sample. The second peak located at the temperature of 574˚ C corresponds to the decomposition of quartz, which is same in both graphs of the ordinary and specimens with nanoparticles.

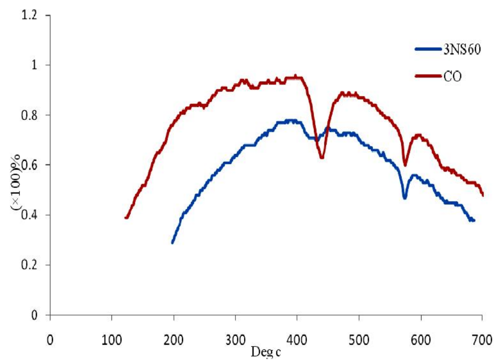

Figure. **5.** DTA of CO and 3NS60 specimens

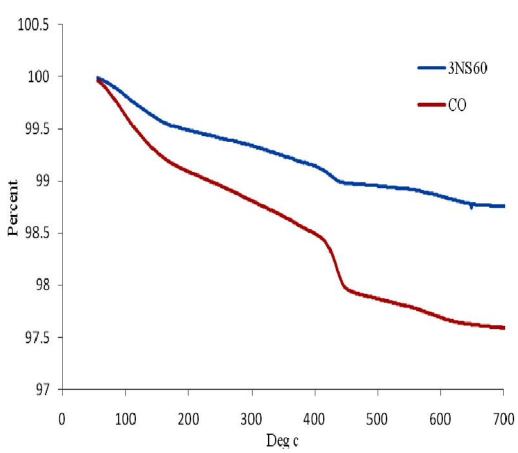

Figure. 6. TGA of CO and 3NS60 specimens

4- Conclusion

Through adding different amounts of silica nanoparticles with different particle sizes to cement mortar instate of cement, we experimentally investigated the mechanical properties, microstructure of the mortar, water permeability, and structure of appeared phases in mortar. The addition of the nanoparticles and microparticles of silica to the ordinary cement mortar increased it's the mechanical properties (tensile, compressive, and flexural strengths). Even with the addition of only 1% silica particles to the concrete, its mechanical properties enhanced considerably, which directly affected by mixing method and the homogenous dispersion of the particles within the cement paste preparation. Considering the higher specific surface area of the silica nanoparticles than the silica microparticles, the cement mortar with nanoparticles exhibited superior mechanical properties in comparison to the cement mortar containing the microparticles. The cement mortar containing silica nanoparticles of 60 nm diameter exhibited better mechanical properties, and more compacted and regular microstructure than the cement mortar containing the silica particles with 12 nm or 1 micrometer particle size. This is resulted from the fact that the homogenous dispersion of the smaller nanoparticles in the cement paste and the probability of their agglomeration are respectively more difficult than the larger nanoparticles, which is directly caused by higher specific surface area of the smaller nanoparticles. The silica particles reduce the amount of the $Ca(OH)_2$ crystallites. The quantity higher than 3% for silica nanoparticles result in less homogenous dispersion of the particles in the mortar. Regarding the investigated parameters, the SiO_2 nanoparticles control the hydration process and by interfering in the formed phases could affect the mechanical and chemical properties of prepared mortar.

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