To study the effect of adding Al_2O_3 nanoparticles on the mechanical properties and microstructure of cement mortar

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Abstract: In this work, research has been done on the compressive, tensile and flexural strength of cement mortar containing Al_2O_3 nanoparticles in the amounts of 1, 3 and 5 percent by weight of cement. The results show that the mechanical properties of samples containing 1 and 3 percent Al_2O_3 nanoparticles are desirable then the ordinary cement mortar. But by increasing Al_2O_3 nanoparticles to 5 percent, the mechanical properties reduce severely. SEM study about the microstructure of cement mortar containing nanoparticles and ordinary cement mortar showed that Al_2O_3 nanoparticles reduces the $CaOH_2$ crystals and fills the pores and increases the density of cement mortar.
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1. Introduction

By using extensively mineral addition for the improve performance of cement-based materials. Partial replacement of cement with mineral additions improves the performance cement-based materials in fresh and hardened states. In the recent years, using nanoparticles has developed due to its small size possess unique properties such as high specific surface area and high activity. Several reports are present about adding nanoparticles to cement materials, that most of them have focus on the silica nanoparticles.

Ye Qing et al, have shown that adding SiO_2 nanoparticles to hardened cement paste increases the compressive strength and bond strength of paste-aggregate interface more than silica fume addition which means because of SiO_2 nanoparticles due to high specific surface and amount of atoms in the surface has higher chemical reaction area [1].

Wang Baomin and et al showed that adding SiO_2 nanoparticles can improve the microstructure of the cement and result in the increase of freezing resistance with high performance concrete [2].

Mohammad Reza Arefi and et al, have studied the effect of adding SiO_2 particles with different diameters and different amount to the cement mortar. The research results showed that nanoparticles due to higher specific surface area improve the resistance properties and water permeability of cement mortar than the micro-particles. Then, samples consisting nanoparticles, samples with nano-silica large diameters have better effect in improving the mechanical properties. And because of the possibility of increase agglomeration of nanoparticles is more with smaller diameter [3].

Study has been conducted with focus on comparison effect of adding different nanoparticles. For example, study which has compared the addition of TiO_2 nanoparticles with SiO_2, the result shows that the abrasion resistance and flexural fatigue performance of concrete containing TiO_2 nanoparticles is more than the abrasion resistance of concrete containing the same amount of SiO_2 nanoparticles [4, 5]. Also, improving of resistance to chloride penetration for the concrete containing TiO_2 is more than the concrete containing the same amount of SiO_2 [6].

There exists report about the addition of Al_2O_3 nanoparticles to cement-based materials. Ali Nazeri and et al have researched the effect of addition Al_2O_3 nanoparticles with mechanical properties and percentage of water absorbtion of concrete which is cured in water and saturated limewater. Their research results showed that by addition of nanoparticles till two percent improves the mechanical properties and concrete penetration that the amount of this improvement for the sample is more which have cured in limewater [7, 10].

The aim of this study is to research the effect of adding Al_2O_3 nanoparticles to cement mortar and to find optimized percentage of adding nanoparticles and also finding mechanism to improve the mechanical properties of cement mortar.

2. Material and Methods

2.1. Materials and mixture proportions

ASTM C 150 [11] Type II portland cement was used. The Al_2O_3 nanoparticles with average particles size of 20 nm which were purchased from Skyspring Nanomaterials Inc were used. The characteristics of the Al_2O_3 nanoparticles were shown in Table 1. The superplasticizer (a commercial sulphonated melamine formaldehyde polymer) with relative density of 1.15 was employed to achieve good workability. The content was adjusted for each mixture to ensure that no segregation would occur. Also, the distilled water was used for preparing all mixtures. The fine aggregate was crushed silica sand with a fineness modulus of 2.4, the apparent density of 3.33 gr/cm^3. The sand was graded according to ASTM C33 [12] standard. The largest diameter of these aggregate particles was 4.75mm.

Table 1. Characteristics of nano- Al_2O_3particles

Average particle size	Specific surface area (m^2/g)	Density (g/cm^2)	Purity (%)
20nm	200	0.9	99.9%

The proportions of the mixtures were presented in table 2. The ratio of the water to binder (the cement and Al_2O_3 nanoparticles) was chosen 0.42. In this study the mixtures were prepared with the cement replacement of 1%, 3% and 5% by weight of binder.

Table 2: Mix proportion of samples

Mixture type	Water	Cement	Sand	Nano-Al_2O_3	*SP
*CO	150	360	1800	-	-
1NA	150	356.4	1800	3.6	3.68
3NA	150	349.2	1800	10.8	4.29
5NA	150	342	1800	18	4.9

*CO: Control.
*SP: superplasticizer

2-2- Sample preparation

The high homogenous dispersion of nanoparticles strongly depends on stable suspension preparation. Hence Al_2O_3 nano powder was mixed with the distilled water and stirred for 6-10 hours by rotational speed of 250-300 rpm. At first, the suspension of the Al_2O_3 nanoparticles 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. For tensile test, the briquette specimens with 75×25×25 mm dimension were utilized. 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 tests, the mortar was poured into molds to form cubes of size 50×50×50 mm in three layers alternatively, which all layers compressed by 10 impacts of a steel rod. 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. 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. Six specimens were prepared for each test and the average result was reported.

2-3 Test methods

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. Compressive tests were carried out according to the ASTM C109 [13] and tensile tests were carried out according to the ASTM C190 [14]. Flexural tests were carried out according to ASTM C348 [15].

3. Results and Discussion

3.1. Mechanical properties

Results of compressive strength, tensile strength after curing for seven days is shown in table 3 and figure 1. It can be understood from the table that by adding Al_2O_3 nanoparticles till 3 percent compressive, tensile and flexural strength increases and then by increasing the quantity of nanoparticles to 5 percent, strength reduces less than the ordinary cement mortar. This may be due to the fact that the quantity of nanoparticles present in the mix is higher than the amount required to combine with the liberated lime during the process of hydration, thus leading to excess silica leaching out and causing a deficiency in strength as its replaces part of the cement material and does not contribute to its strength [16].

This issue is because nanoparticles due to their high surface energy have the tendency towards agglomeration. When nanoparticles are over added to the mortar it is not uniformly distributed in cement mortar and due to agglomeration weak areas appear in the cement mortar. Whereas, few amounts of nanoparticles even if not distributed uniformly it increase the strength, this is because that small quantity of nanoparticles agglomeration does not create weak zone [17].

Table 3: Mechanical properties of samples

Mixture no.	Compressive strength at the 7th day		Tensile strength at the 7th day		Flexural strength at the 7th day	
	Target (MPa)	Enhanced extent (%)	Target (MPa)	Enhanced extent (%)	Target (MPa)	Enhanced extent (%)
CO	11.96	-	1.51	-	2.2	-
1NA	17.25	44.23	2.52	66.89	3.25	47.73
3NA	19.54	63.38	2.74	81.46	3.74	70
5NA	10.9	-8.86	1.4	-7.3	2.04	-7.27

The mechanisms of Al_2O_3 nanoparticles that causes the increases of strength of cement mortar it can defined as the Al_2O_3 nanoparticles because of high specific surface area causes the consumption of crystalline $Ca(OH)_2$ which quickly formed during the hydration of cement process and fills the void structure of C-S-H gel and finally the hydrated products are made denser and compact.

In other words, when nanoparticles are uniformly distributed in the cement mortar, each particle is contained in a cube pattern and the distance between nanoparticles can be adjusted. After the beginning of cement hydration process, nanoparticles due to their high activity develops and accelerate the cement hydration and hydrate products of nanoparticles are surrounded as kernel. If the quantity and distance between these nanoparticles is suitable, nanoparticles prevents the growth of $Ca(OH)_2$ crystals [5]. The past research of these researchers show that with excessive increase of nanoparticles quantity, the nanoparticles distance decreases and $Ca(OH)_2$ crystals due to limited space cannot grow enough and finally the crystal quantity is reduced [18]. This factor along with the agglomerated nanoparticles causes the mechanical properties of the sample 5NA is lower than the ordinary mortar sample.

The results show that the addition of Al_2O_3 nanoparticles, increasing amount of tensile and flexural strength is more than compressive strength, which agrees with the research results by Ali Nazeri and et al [7, 8].

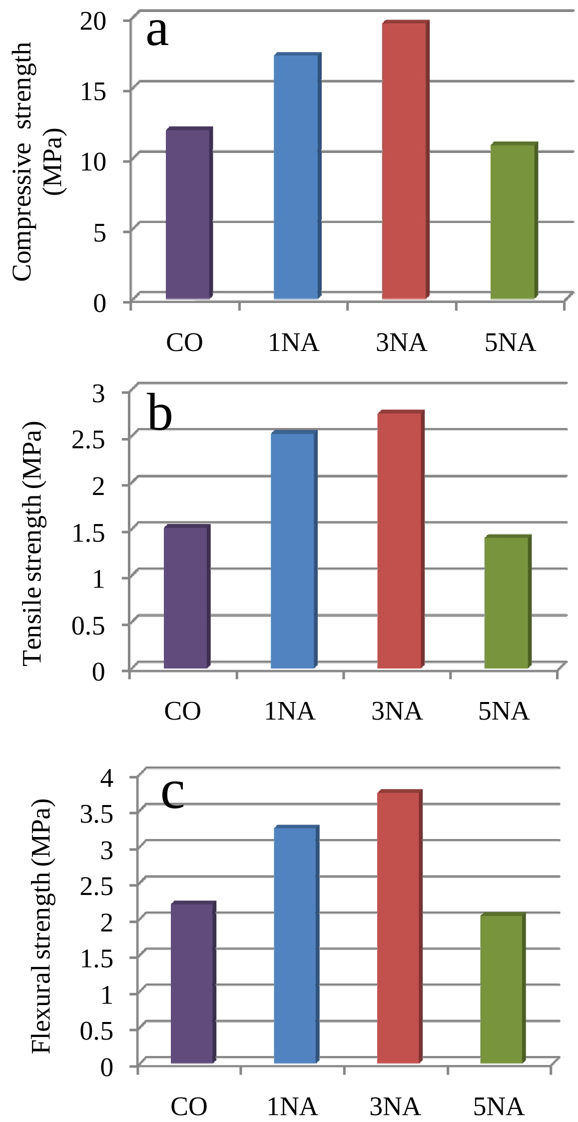

Figure 1. (a) Compressive strength, (b) Tensile strength, and (c) Flexural strength of samples

3.2. Microstructure of samples

To study the mechanism which improves the strength, SEM test has been conducted. The microstructure of samples is shown in figure 2. As shown in the figure, adding Al_2O_3 nanoparticles causes difference in the microstructure samples. In microstructure samples of ordinary cement mortar there exists large crystals of $Ca(OH)_2$. Microstructure of cement mortar is not dense and voids can be seen. As shown in figure 2b, in sample containing 1 percent nanoparticles relative to sample of ordinary cement mortar the structure of cement mortar has become denser and the voids decreased but still large crystals of $Ca(OH)_2$ are observed. But, with increasing quantity of nanoparticles to 3 percent, large crystals of $Ca(OH)_2$ are eliminated and microstructure of cement mortar is completely denser.

As shown in figure 2d, in samples containing 5 percent nanoparticles because of the agglomeration of nanoparticles voids are formed. These microstructures with the reduction of mechanical properties in these samples are appropriate.

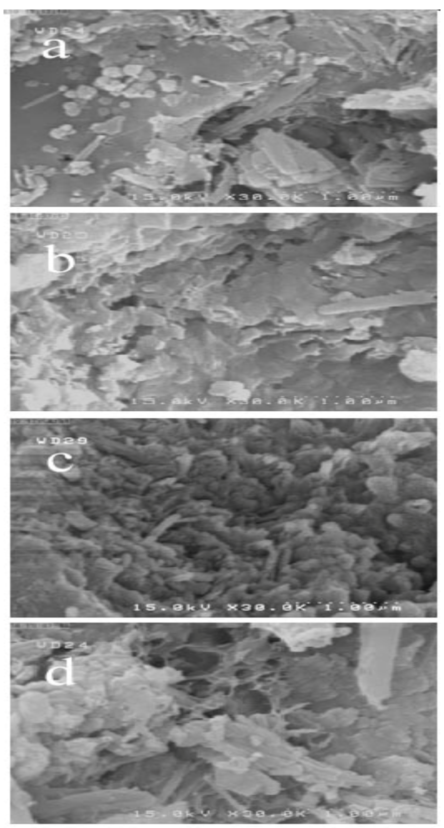

Figure 2. Microstructure of the samples,
a) Sample of CO. b) Sample of 1NA.
c) Sample of 3NA. d) Sample of 5NA

4. Conclusion

With respect to the experimental results of compressive, tensile and flexural strength it is expected that adding of Al_2O_3 nanoparticles to 3 percent weight of cement can act as a filler for strengthening the microstructure of cement and also reduces the quantity and size of $Ca(OH)_2$ crystals and finally structure of hydrated product is compacted and denser. But with the increase of quantity of Al_2O_3 nanoparticles to 5 percent there is decrease in nanoparticles distance and $Ca(OH)_2$ crystal due to limited space cannot grow to appropriate size. This factor along with the agglomerated nanoparticles causes the mechanical properties of the sample 5NA is lower than the ordinary mortar sample.

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