To study the effect of adding Fe_2O_3 nanoparticles on the morphology properties and microstructure of cement mortar

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Abstract: In this study, research has been done on the compressive and tensile strength of cement mortar containing Fe_2O_3 nanoparticles in the amounts of 1, 3 and 5% by weight of cement. The results show that the mechanical properties of samples containing 1% and 3% Fe_2O_3 nanoparticles are desirable than the ordinary cement mortar. SEM study about the micro structure of cement mortar containing nanoparticles and ordinary cement mortar showed that Fe_2O_3 nanoparticles fills the pores completely and reduces the large crystals of $Ca(OH)_2$ and the hydrate products are denser and compact. The mechanical properties results showed that by increasing Fe_2O_3 nanoparticles up to 5% reduces the mechanical properties.

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1. Introduction

In recent years, much attention is in the application of nanoparticles in civil engineering, because nanoparticles due to its small size possess unique properties such as high specific surface area and high activity. If nano materials are combined with traditional building materials, this may lead to production of building materials with unique properties and be useful for construction industry. On the other hand, building materials based on Portland cement (concrete, cement mortar, hardened cement paste), is one of the most used and important components used in the construction industry. Much research has been done on the partial replacement of cement with supplementary cementing materials such as Pozolon and nanoparticles to improve their mechanical properties. Many researchers have studied the mechanical properties of cement based materials containing nanoparticles.

Researches done shows that adding nano-particles to cement-based materials improves the mechanical properties. Hui Li and et al in 2005 have examined the abrasion resistance of concrete containing TiO_2 and SiO_2 nanoparticles. The experiment results shows that the abrasion resistance of concrete containing nanoparticles has improved and abrasion resistance of concrete containing nano TiO_2 is more than the abrasion resistance of concrete containing nano SiO_2 [2]. Other research results show that adding SiO_2 nanoparticles can improve the microstructure of the cement and result in the increase of freezing resistance with high performance concrete [3]. This is an important property for concrete exposed to frost. Ali Nazeri et al in 2010 proved that addition of TiO_2 and ZnO_2 nanoparticles increases the concrete compressive strength. These particles also reduce the workability of fresh concrete where in this case it is essential in the use of plasticizer in high percentage of nanoparticles [4, 5]. Mohammad Reza Arefi and et al have studied the effect of adding SiO_2 particles with diameters and in different amount to the cement mortar. The research results showed that nanoparticles due to higher specific surface area improve the mechanical properties and water permeability of cement mortar more than the micro-particles [6]. Studies done on the effect of adding nanoparticles to cement mortar shows that the nanoparticles reduces the amount and size of $Ca(OH)_2$ crystals and needle shaped hydrates and takes position as a nucleus in the cement paste and progresses the hydration of cement having high activity. Cause to create a homogenous and denser cement matrix and ultimately leads to the improved mechanical properties of cement mortar [7-9]. Meral Oltulu and et al have investigated separately and combined the effect of adding nanoparticles of Fe_2O_3, Al_2O_3 and SiO_2 to cement mortars containing silica fume. The research results show that adding these nanoparticles separately causes the increase of compressive strength and improved capillary permeability. But, the interaction of nanoparticles as binary and ternary combinations has a negative effect on the physical and mechanical properties of cement mortar [10].

The research done on the addition of Fe_2O_3 nanoparticles to cement-based materials shows that the cement mortar containing nano-Fe_2O_3 can sense its compressive stresses in elastic and inelastic system. Because nano-Fe_2O_3 can change the electric resistance of cement mortar with the loading applied. This property is useful for the monitoring structural health [11]. The flexural, tensile and compressive strength of concrete is increased and reduces the setting time of concrete [12, 13]. Conflicting results has been shown about the optimized percentage of adding Fe_2O_3 nanoparticles. Lee and et al [7] showed that by adding nanoparticles to cement mortar, samples containing 3% nanoparticles has the highest mechanical properties but, the research results of Ali Nazeri and et al [12, 13] has shown that the best compressive strength is related to the sample containing 1% Fe_2O_3 nanoparticles.

The aim of this study is to find the optimized percentage of adding Fe_2O_3 nanoparticles and to achieve high strength mortar and finding mechanism to improve the mechanical properties of cement mortar.

2. Material and Methods

2.1. Materials and mixture proportions

ASTM C 150 [14] Type II portland cement was used. The superplasticizer 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. Crushed silica sand was used with apparent density of 3.33 gr/cm^3 and the fineness modulus of 2.6. The sand was graded according to ASTM C33 [15] standard. The largest diameter of these aggregate particles was 4.75mm. The Fe_2O_3 nanoparticles were purchased from Skyspring Nanomaterials Inc. The characteristics of the Fe_2O_3 nanoparticles were shown in Table 1.

Table 1. The characteristics of nanoparticles

Nanoparticle type	Diameter	Specific surface area (m^2/g)	Purity(%)
Fe_2O_3	30 nm	60	99%

The mixture proportions of the ordinary cement mortar and the cement mortar containing Fe_2O_3 nanoparticles were shown in Table 2. The ratio of the water to binder (the cement and Fe_2O_3 nanoparticles) was chosen 0.42. In this study the mixtures were examined with the cement replacement of 1%, 3% and 5% by weight of cement.

Table 2: Mix proportion of samples (kg/m^3)

Sample name	Water	Cement	Sand	Nano Fe_2O_3 particles	*SP
*CO	150	360	1800	-	-
1NF	150	356.4	1800	3.6	3.68
3NF	150	349.2	1800	10.8	4.29
5NF	150	342	1800	18	4.9

*CO: Control- Ordinary cement mortar
*SP: superplasticizer

2-2- Sample preparation

The high homogenous dispersion of nanoparticles strongly depends on stable suspension preparation. Hence Fe_2O_3 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 Fe_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. 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 [16] and tensile tests were carried out according to the ASTM C190 [17].

3. Results and Discussion

3.1. Microstructure of samples

The microstructure of samples is shown in figure 1. As shown in the figure, it can be seen in the microstructure the ordinary mortar samples of large crystals of $Ca(OH)_2$. The microstructure of cement mortar is non dense and the voids can be observed. Microstructure of the sample containing 1% nanoparticles is similar to ordinary mortar. In both large crystal $Ca(OH)_2$ is observed with the difference that the voids are reduced and the mortar structure is more denser. With the increase of nanoparticles quantity up to 3%, microstructure has improved completely and achieved better density. As shown in figure 1d, in samples containing 5% nanoparticles because of the agglomeration of nanoparticles voids are formed. These microstructures with the reduction of mechanical properties in these samples are appropriate.

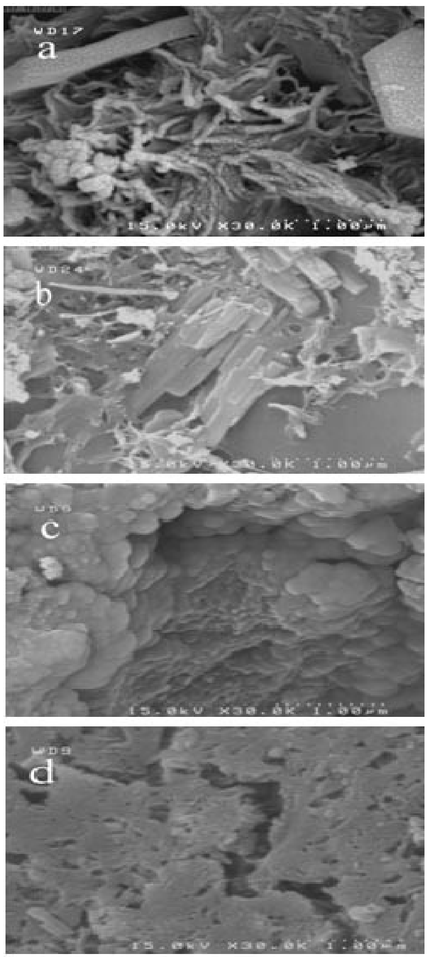

Figure 1. Microstructure of the samples,
a) Sample of CO. b) Sample of 1NF.
c) Sample of 3NF. d) Sample of 5NF

3.2. Mechanical properties

Results of compressive strength, tensile strength after curing for seven days is given in table 3. It can be understood from the table that the sample containing 1 and 3% Fe_2O_3 nanoparticles, the mechanical properties has improved than the ordinary cement mortar. As indicated in figure 1 in a sample containing 1% nanoparticles than ordinary samples the structure of cement mortar is compacted, but still $Ca(OH)_2$ large crystals is observed. With the increasing quantity of nanoparticles up to 3% the $Ca(OH)_2$ large crystals are removed and the microstructure of the mortar is completely compacted. The mechanism of Fe_2O_3 nanoparticles which increases the strength of cement mortar can be described as follows that the addition of Fe_2O_3 nanoparticles reduces the quantity and size of $Ca(OH)_2$ crystals and fills the voids of C-S-H gel structure and ultimately the structure of hydrated products are denser and compact [7]. But, the increased Fe_2O_3 nanoparticles up to 5%, the mechanical properties reduces. 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 zone appear in the cement mortar.

This phenomenon can be explained as when the nanoparticles are uniformly distributed in cement mortar each particle has a cubic pattern and distance between the nanoparticles is adjustable. After beginning the cement hydration process the hydrated product are distributed and surrounds the nanoparticles as the nucleus. If the amount and distance between the particles is appropriate, nanoparticles prevents the growth of $Ca(OH)_2$ crystals [2]. 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 5NF is lower than the ordinary mortar sample. Thus the effect of the nanoparticles agglomeration and non-desirable influence on the entire structure causes local cracks and ultimately reduces the mechanical properties.

The results show that the addition of Fe_2O_3 nanoparticles, increasing amount of compressive strength is more than tensile strength. The reason for this is the presence of micro cracks in the cement mortar. The impact of these micro cracks on tensile strength is greater than the compressive strength [2, 19].

Table 3: Mechanical properties of samples

Sample name	Compressive strength		Tensile strength	
	(MPa) amount	Percent increased (%)	(MPa) amount	Percent increased (%)
CO	11.96	-	1.51	-
1NF	18.71	56.44	2.03	34.43
3NF	20.81	74	2.25	49
5NF	10.08	-9.7	1.25	-17.22

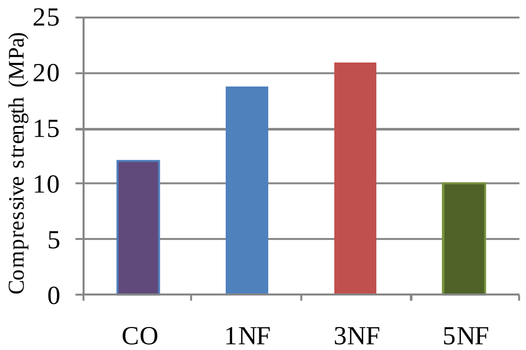

Figure 2. Compressive strength of samples

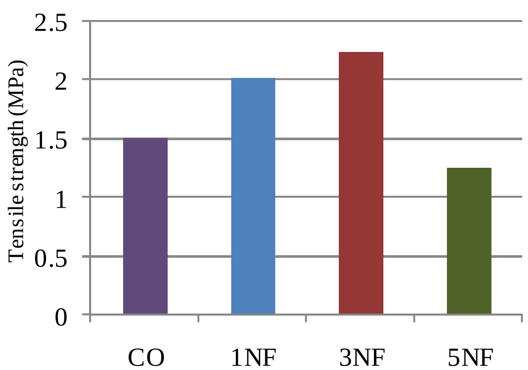

Figure 3. Tensile strength of samples

4. Conclusion

With respect to the experimental results of tensile and compressive strength it is expected that adding of Fe_2O_3 nanoparticles up to 3% by weight of cement can act as a filler for strengthening the micro structure of cement and also reduces the quantity and size of $Ca(OH)_2$ crystals and fill the voids of C-S-H gel structure and finally structure of hydrated product is compacted and denser. With the increase of nanoparticles quantity up to 5% 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 5NF is lower than the ordinary mortar sample.

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