Research Article

A STUDY OF MICROSTRUCTUR AND MECHANICAL PROPERTIES OF CEMENT MORTAR BY ADDING NANOPARTICLES

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Abstract: In this study, research has been done on the compressive and tensile strength of cement mortar containing 1, 3 and 5 percent by weight of cements Fe_2O_3nano-particles. The results show that the mechanical properties of samples containing 1 and 3 percent Fe_2O_3nano-particles is desirable then the ordinary cement mortar. SEM study about the micro structure of cement mortar containing nano particles and ordinary cement mortar showed that Fe_2O_3 nano-particles fills the pores completely and reduces the mixture of $CaOH_2$ with hydrates and increases the final density of the prepared samples. The mechanical properties results showed that by increasing iron oxide nano-particles the mechanical properties enhances to 5 percent.

Keywords: Mechanical properties; Fe_2O_3 nanoparticles; cement mortar; SEM, microstructure

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1. Introduction

In recent years, much attention is in the application of nano-particles in civil engineering, because nano-particles 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 nano-particles to improve their mechanical properties. Many researchers have studied the mechanical properties of cement based materials containing nano-particles.

Researches done shows that adding nano-particles to cementing materials improves the mechanical properties. Hui Li and co-workers in 2005 have examined the abrasion resistance of concrete containing TiO_2 and SiO_2nano-particles. The experiment results shows that the abrasion resistance of concrete containing nano-particles 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_2nano-particles 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 and co-workers in 2010 proved that addition of TiO_2 and ZrO_2nano-particles 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 nano-particles. [4, 5] Mohammad Reza Arefi and co-workers have studied the effect of adding SiO_2 particles with diameters and in different amount to the cement mortar. The research results showed that nano-particles due to higher specific surface area improve the mechanical properties and water permeability of cement mortar than the micro-particles. And because of the possibility of increase agglomeration of particles the nano-particles are with smaller diameter. [6]. Studies done on the effect of adding nano-particles to cement mortar shows that the nano-particles reduces the amount and size of calcium hydroxide 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, 8, 9] MeralOltulu and co-workers have investigated separately and combined the effect of adding nano-particles of Fe_2O_3, Al_2O_3 and SiO_2 to cement mortars containing silica fume. The research results show that adding these nano-particles separately causes the increase of compressive strength and improved capillary permeability. But, the interaction of nano-particles 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_3nano-particles to cementing materials shows that the cement mortar containing nano-Fe_2O_3 can sense its compressive stresses in elastic and non-elastic 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_3nano-particles. Lee and co-workers [7], showed that by adding nano-particles to cement mortar , samples containing 3 percent of nano-particles has the highest mechanical properties but, the research results of Ali Nazeri and co-workers [12, 13] has shown that the best compressive strength is related to the sample containing 1 percent of Fe_2O_3nano-particles.

The aim of this study is to find the optimized percentage of adding Fe_2O_3nano-particles 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 super plasticizer was a commercial sulphonated melamine formaldehyde polymer manufactured by Vandchemie 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	40nm	60m^2/g	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

Sample name	Water	Cement	Sand	Nano particles	Fe_2O_3	*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	336.5	1800	23.5		4.9

*CO: Control.

*SP: superplasticizer

2-2- Sample preparation

The high homogenous dispersion of nanoparticles 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 $250-300\,rpm$. 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 and water permeability 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 typical 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 percent of 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 to 3 percent, microstructure has improved completely and achieved better density. As shown in figure 1d, in samples containing 5 percent nanoparticles because of the agglomeration of nanoparticles voids are formed. These microstructure with the reduction of mechanical properties in these samples are appropriate.

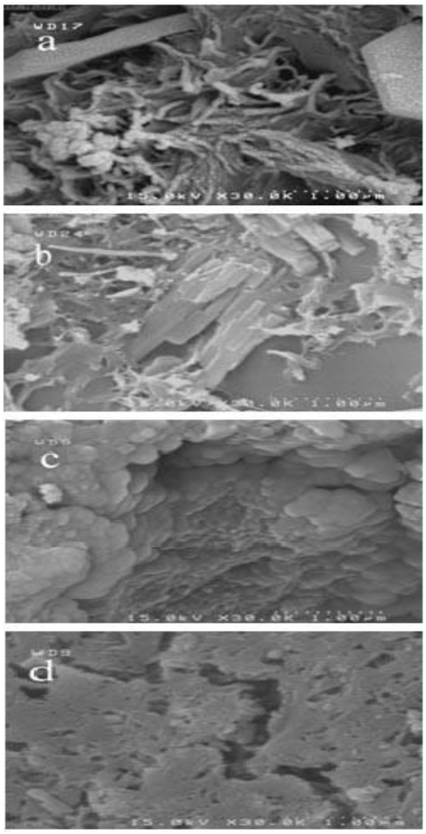

Figure 1. Microstructure of the samples, a) Sample of CO. b) Sample of 1NF. c) Sample of 3NF. d) Sample of 5NF

3.2. Mechanical property

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 percent Fe_2O_3 nanoparticles, the mechanical properties has improved than the ordinary cement mortar. Most of the strenght for the samples containing 3 percent of nanoparticles is Fe_2O_3. As indicated in figure 1 in a sample containing 1 percent of 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 to 3 percent 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 amount 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. But, the increased content of nanoparticles is reduced to 5 percent of the resistance. 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	-15.72	1.25	-17.22

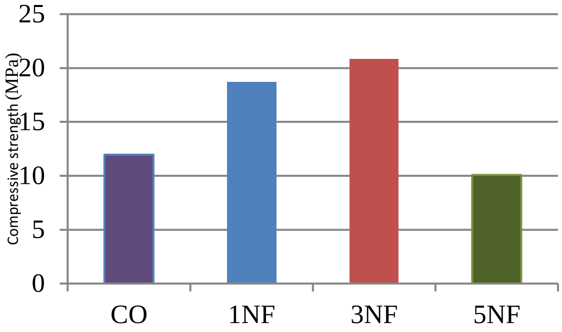

Figure 2. Compressive strength of samples

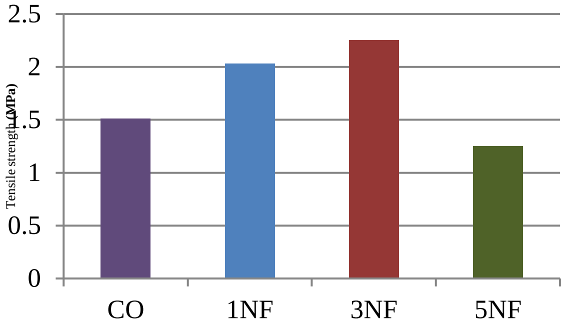

Figure 3. Tensile strength of samples

4. Conclusion

With respect to the experimental results of tensile and compressive strength it is expected that adding of Fe_2O nanoparticles to 3 percent 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 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 5NF is lower than the ordinary mortar sample.

References

1. Zhang MH, Li H. Pore structure and chloride permeability of concrete containing nano-particles for pavement. Constr Build Mater 2011; 25: 608–616.
2. Li H, Zhang MH, Ou JP. Abrasion resistance of concrete containing nano-particles for pavement. Wear 2006; 260: 1262–1266.
3. Baomin W, Lijiu W, Lai FC. Freezing Resistance of HPC with Nano-SiO2. J Wuhan UnivTechnol-Mater(Sci. Ed) 2008;23:85-8.
4. Ali Nazari, ShadiRiahi, ShirinRiahi, SeyedehFatemehShamekhi and A. Khademno. Assessment of the effects of the cement paste composite in presence TiO2 nanoparticles. Journal of American Science.
5. Riahi S, Nazari A. Physical, mechanical and thermal properties of concrete in different curing media containing ZnO2 nanoparticles. Energy and Buildings 2011; 43: 1977–1984.
6. Arefi MR, Javaheri MR, Mollaahmadi E, Zare H, AbdollahiNejand B, Eskandari M. Silica nanoparticle size effect on mechanical properties and microstructure of cement mortar. Journal of American Science, 2011; 7(10).

7. Li H, Xiao H.G, Yuan J, Ou J. Microstructure of cement mortar with nano-particles. Compos: Part B: Eng 2004; 35: 185–9.
8. Byung-Wan Jo, Chang-Hyun Kim, Ghi-ho Tae, Jong-Bin Park. Characteristics of cement mortar with nano-SiO2 particles. Construction and Building Materials 21 (2007) 1351–1355.
9. Qing Y, Zenan Z, Deyu K, Rongshen C. Influence of nano-SiO2 addition on properties of hardened cement paste as compared with silica fume. Constr Build Mater 2007;21:539–45.
10. Oltulu M, Sahin R. Single and combined effects of nano-SiO2, nano-Al2O3 and nanoFe2O3 powders on compressive strength and capillary permeability of cement mortar containing silica fume materials. Science and Engineering A 528 (2011) 7012–7019.
11. Li H, Xiao HG, Ou JP. Study on mechanical and pressure-sensitive properties of cement mortar with nanophase materials. Cement and Concrete Research 34 (2004) 435–438.
12. Ali Nazari, ShadiRiahi, ShirinRiahi, SeyedehFatemehShamekhi and A. Khademno. Benefits of Fe2O3 nanoparticles in concrete mixing matrix. Journal of American Science. 2010;6(4) 102-106.
13. Ali Nazari, ShadiRiahi, ShirinRiahi, SeyedehFatemehShamekhi and A. Khademno. The effects of incorporation Fe2O3 nanoparticles on tensile and flexural strength of concrete. Journal of American Science. 2010;6(4) 90-93.
14. ASTM C150. Standard Specification for Portland Cement. American Society for Testing and Materials;2005.
15. ASTM C33. Standard Specification for Concrete Aggregates. American Society for Testing and Materials;2007.
16. ASTM C109 / C109M. Standard Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or [50-mm] Cube Specimens). American Society for Testing and Materials;2008.
17. ASTM C190. Method of Test for Tensile Strength of Hydraulic Cement Mortars. American Society for Testing and Materials;1985.
18. Arefi MR, Mollaahmadi E, AbdollahiNejand B, Fattah M, High performance self -cleaning cement mortar composite and coats prepared by TiO2nanoparticles. Submited in Journal of Materials Science.
19. Li H, Zhang MH, Ou JP. Flexural fatigue performance of concrete containing nano-particles for pavement. Int J Fatigue 2007;29: 1292–1301.