An experimental investigation on nano-Al_2O_3 based self-compacting mortar

Bahareh Mehdizadeh Miyandehi[1], Babak Behforouz[2], Elham Mehrinejad Khotbehsara[3], Hamed Azar Balgouri[1], Shadi Fathi[4] and Morteza Mehrinejad Khotbehsara[5]

[1.] Department of Civil Engineering, University of Guilan, Rasht, Iran
[2.] Department of Civil Engineering, Najaf Abad Branch, Islamic Azad University, Najaf Abad, Iran
[3.] Department of Civil and Architecture Engineering, Zanjan Branch, Islamic Azad University, Zanjan, Iran
[4.] Department of Civil Engineering, University of Kurdistan, Sanandaj, Iran
[5.] Department of Civil Engineering, Bandar-Anzali Branch, Islamic Azad University, Anzali, Iran
BaharehMehdizadehMiayandehi@gmail.com

Abstract: This study investigates the effect of using Al_2O_3 nanoparticles on rheological, mechanical, and durability properties of self-compacting mortars (SCM) incorporating fly ash. Al_2O_3 nanoparticles (NA), with maximum size of 15nm, with three different quantities of 1%, 3%, and 5% of the binder by weight were partially replaced with cement with a constant fly ash amount of 25% of the weight of cement for all mixtures. Compressive and flexural strength tests were done at the ages of 3- and 7-day as early age, 28-day as standard age, and 90-day as late age to obtain the mechanical properties of samples. Also, water absorption, electrical resistivity, and rapid chloride permeability tests (RCPT) were considered observing the durability properties of SCM specimens containing fly ash. The results showed that 1% and 3% of the binder are the best percentages of the compressive strength and flexural strength of nanoparticles NA. For all combinations, the strength increased and reached their peak on the 90th day. This important effect can be seen on water absorption as well. In fact, specimens with 1% nano-Al_2O_3 had the lowest and greatest water absorption and resistivity respectively among all samples. In terms of chloride permeability, in general, specimens with different contents of nanoparticle had relatively similar resistance to chloride permeability. However, they were all more resistant than control sample.
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Keywords: Self-compacting mortar; Nano-Al_2O_3; Mechanical properties; Water absorption; Electrical resistivity; Rapid chloride permeability test (RCPT).

Introduction:

Nowadays, self-compacting concrete (SCC) has gained significant importance due to the benefits such as being able to be placed and compacted under its own weight without the need for vibration and without segregation or bleeding it offers. It is utilized to facilitate and ensure proper filling and good structural performance of restricted areas and heavily reinforced structural members [1-5]. But, there are some important reasons which indicate the advantages of using self-compacting mortar in comparison with self-compacting concrete. Basically, doing experimental tests on concrete is harder than mortar and also the mortar phase has the similar properties of concrete so the investigation of the properties of mortar instead of concrete would be reasonable [6]. Furthermore, except coarse aggregate, mortar contains all materials as well as the influence of the test variables will be as the same as those in the concrete. Many researchers have already carried out some research on the application of nano-sized materials and/or pozzolanic materials in self-compacting mortar and self-compacting concrete to improve their mechanical and physical properties [7]. However, there are a few studies on incorporating nano-Fe_2O_3 and nano-Al_2O_3 [8]. Nanotechnology and nano-science can be defined as the exploration, innovation, and application of nano-materials, which are characterized by at least one dimension in the nanometer (nm) range. The size range that engages greatly attention is below 100 nm [9]. Jalal et al. [4] investigated the effects of micro SiO_2 and nanoparticles on mechanical, rheological, durability and microstructural properties of high performance self-compacting concrete. As the results showed, using micro and nanosilica had significant effect on improving the properties of SCC. Oltulu et al. [3] studied the influence of nano-SiO_2, nano-Al_2O_3 and nano-Fe_2O_3 powders on compressive strengths and capillary water absorption of cement mortar containing fly ash. According to the obtained results, nano-powders by rising the pozzolanic activity could improve the mechanical and physical properties of mortars. Barbhuiya [6] used dolomite powder as an alternative material instead of limestone powder in SCC. The results expressed the possibility of manufacturing SCC utilizing fly ash and dolomite powder.

The question whether Al_2O_3 nanoparticles provide disadvantages apart from their advantages in self-compacting mortar or not have been answered by this study.

2. Materials and methods

2.1 Materials and mixtures

A total of 4 specimens with disparate proportions at 700 kg/m^3 of binder were prepared. The amount of fly ash was considered 25% of the weight of the cement as well as constant water to binder (containing cement, fly ash, and nanoparticles) ratio of 0.4 was investigated. In this study, ordinary Portland cement (ASTM C150) [10] type II and fly ash class F were used. The physical and chemical properties of cement and fly ash have been illustrated in Table 1. Natural river sand was utilized as the fine aggregate. A dosage of super plasticizer with density of 1.03 g/cm^3 based on polycarboxylate acid with the brand of Vand super plast, PCE conforming to ASTM C494 [11] TYPE F was utilized to achieve a better compacting. The content of nanoparticles which has been replaced partially with cement in mixtures was obtained through doing some preliminary experimental tests involved nanoparticles and theses ratios of 1wt%, 3wt%, and 5wt% of the cement replacement were considered and were generally constant in all mixtures. Maximum size of nano-Al_2O_3 (NA) utilized in the study was 15 nm, while its Blaine value was 200 m2/g and impurity was 99.8%.

Table 1. Chemical composition and physical properties of ordinary Portland cement and fly ash

Chemical analysis (%)	Cement	Fly ash
SiO_2	21.56	55.8
Al_2O_3	6.67	20.75
Fe_2O_3	6.17	6.66
CaO	49.88	4.12
MgO	4.51	1.9
SO_3	2.75	0.44
Specific gravity (g/cm^3)	3.15	2.2
Specific surface area (cm^2/g)	3250	285

2.3. Mixture proportioning

In this study, the processing of preparing specimens after conducting some preliminary experiments is obtained as following:

- Cement and sand were mixed for approximately 1 minute with the speed of 80 rpm.
- Fly ash, 30% of water, and nanoparticles were added and mixed speedily for about 1 minute.
- Then the mixture was rested around 1.5 minutes.
- Finally, the rest of water (70%) with super plasticizer were added and combined completely for 2 minutes.

The molding process of the specimens was performed by pouring mortars in the 50x50x50mm molds. The specimens were kept in the laboratory for 24 hours under constant ambient temperature to be cured. Then all specimens were stored in the water tank at a constant 20±1°C, until the day of the test.

3. Result and discussion

3.1 Properties of fresh self-compacting mortar

Standard slump tests conforming to EFNARC [12] were obtained to determine the workability of the concrete.

The slump flow diameter of the specimens ranges between 245 and 250mm while the V-funnel flow time (s) ranges from 10 to 11 seconds. As Table 2 shows, NA with ratio of 5% had the highest slump flow diameter as 250 mm and the lowest V-funnel flow time as 10 s respectively. Results indicate that the amount of V-funnel flow time slightly reduced with increasing the content of nanoparticles. In fact, the highest V-funnel flow time was observed in the specimens with the lowest percentage of nanoparticle. However, this phenomenon is different for slump flow diameter as the highest diameter was obtained at 5% for each type of mixture.

As indicated in Table 2, the rheological properties of specimens with addition of different nanoparticles increased slightly which is not considerable.

Table 2. Fresh properties of mortars

Label	Slump flow diameter (mm)		V-funnel flow time (s)
Control	245		11
1NA	248		10.6
3NA	248		10
5NA	250		10

3.2 Mechanical and durability properties of self-compacting mortars.

Compressive strength and flexural tests have been done to obtain the mechanical properties of the mixtures. Moreover, water absorption, Electrical resistivity and rapid chloride permeability test has been carried out.

3.2.1 Compressive strength

ASTM C109-93 was used to assess the compressive strength behaviour of specimens [13]. Figure 1 expresses the compressive strength results of mortar mixtures containing Al_2O_3. According to the results, compressive strength of specimens decreased with increasing the content of nano-Al_2O_3, to the extent that the strength reached the lowest amount in specimens containing 5% NA with 12MPa even less than 12.13MPa which was obtained by control specimens. So it indicates that enhancing the content of nanoparticles will not always increase the strength of samples. This result is conformed to the obtained one by Agarkar in 2012; in fact, the results revealed that adding up to 1% Al2O3 increases the compressive strength, whereas then it decreases [14]. Arefi et al. (2011) concluded that the compressive strength of mixtures decreases after replacing 3%wt Al2O3 nanoparticles with cement [15]. The mainly compose of microstructure of hydrated Portland cement paste is calcium silicate hydrate gel (xCaO_ $ySiO_2$_ mH_2O), which is abbreviated as C–S–H [9]. C-S-H is associated with the reaction of calcium silicates in cement and water and provides important strength of the hydrated cement paste compared to these four chemical compounds [16]. The appropriate amount of nanoparticles and the distance between them contribute to control the crystallization and so restricting the growth of $Ca(OH)_2$ crystal; so this phenomena makes the cement matrix more homogenous and compact [8]. Consequently, the strength of specimens is improved. However, with increasing the ratio of nanoparticles the distance between nano-particles reduces, the $Ca(OH)_2$ cannot develop satisfactorily, which leads to the small ratio of crystal to C–S–H gel and the weak microstructure of cement matrix. As a result, the strength of mortars decreases [17].

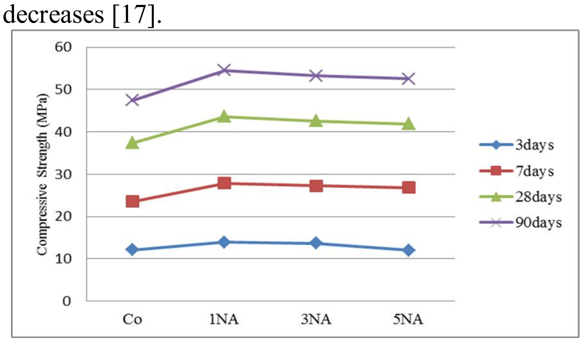

Fig 1. Compressive strength of nano-Al_2O_3 particle blended mortar specimens

3.2.2 Flexural strength

Flexural test were done based on the ASTM C293 [18] standard. The flexural strength results of mixtures containing NA are shown in Figure 2. By comparison the flexural strength of the specimens cured for 3, 7, 28 and 90 days, it could be mentioned that, apart from 3 days which the maximum amount of flexural strength can be seen in mixtures containing 1% NA, the strength increases with nano-Al_2O_3 particles up to 3.0% replacement and then it decreases, although the results of 1.0% replacement is even lower than those of the plain cement concrete. The maximum increase can be observed in mixtures with 3% NA at the age of 90 days which increased by around 13.8% in comparison with control sample. This can be seen in the determined results by Arefi in 2011. The flexural strength decreased by replacing more than 3% nano-Al2O3 [15]. Moreover, based on the results, replacing 5% of NA with cement did not affect the flexural strength of specimens significantly in contrast with 3% NA and even in most cases this ratio of Al_2O_3 decreased the strength slightly. In general, the flexural strength of specimens has increased through enhancing the content of Al_2O_3 nanoparticles. This might be due to the fact that NA arrests crack and interlocking influences between the slip planes, which improve the flexural strength of cement-based materials.

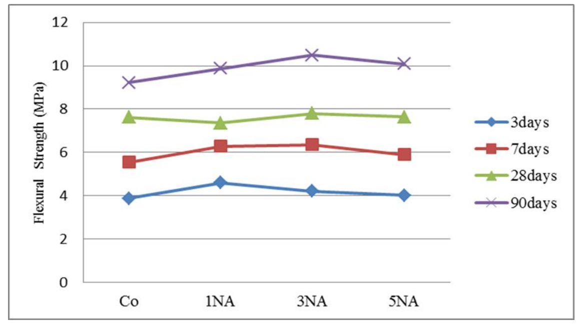

Fig 2. Flexural strength of nano-Al_2O_3 particle blended mortar specimens

3.2.3 Water absorption

The water absorption test was conducted according to ASTM C642 [19] on pouring mortars in the 50x50x50mm molds in 28 ages. 2 specimens were considered from each mixture and then the water absorption prosperity was evaluated based on the average obtained rate for each sample. Specimens were dried after 28 days immersing in the water by being placed in a ventilated oven at a temperature of 105±5 during 24 hours. Then the weight of samples was assessed by a digital scale. This procedure continued until the difference in mass was less than 5% of the dried one. The obtained result was considered as dried mass (M_S). To determine the saturated mass (M_D), specimens were put in the water for 72 hours and then have been weighed again, this happened for several times, until the difference in mass was less than5% of the heaviest sample. So the water absorption (W) was expressed as follow:

$$W = \frac{M_S - M_D}{M_D} \quad (1)$$

The water absorption of self-compacting mortars containing NA at various ratios is expressed in Figure 3. As the Figure shows, increasing the amount of nanoparticles led to a constant trend in samples containing NA. This point can be illustrated that water absorption in mixtures with nano-Al_2O_3 has increased significantly by replacing nanoparticles. In fact, the water absorption has increased continuously in samples containing NA additives. Oltulu and Sahin determined that replacing Al_2O_3 nanoparticles enhances the water absorption of specimens [3]. The addition of nano-Al_2O_3 into mixtures at a proportion of 1% yielded results dramatically differing from the control specimen, absorption decreased by about 4.2%. After increasing the rate of admixture to 3%, the absorption value was increased by 1.6% compared with NA samples, which is not appreciable. On the other hand, enhancing the ratio to 5% increased the water absorption again the highest amount of absorption can be seen in this proportion, see Figure 3. However, this rate is still lower than control ones. But, it was generally observed that using nanoparticles has reduced water absorption in comparison with plain mixture. In fact, nanoparticles are expected to fill voids of specimens and so reduce water absorption. Since water absorption test mostly indicates the voids volume in cement-based materials, it can be concluded that using nanoparticles of Al_2O_3 leads to reduce this volume in mixtures, so samples will be deterred from some damaging factors such as corrosion.

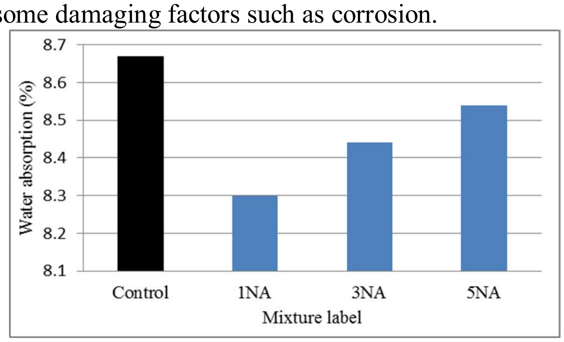

Fig 3. Percentage of water absorption of nano-Al_2O_3 particle blended concrete specimens.

3.2.4 Electrical resistivity

Cubic specimens with dimensions of 50*50*50mm were prepared and measured on the 90th day. Resistivity test includes an electrical resistance measurement device and two electrodes to be attached on both sides of samples and the values were calculated through utilizing Eq.2.

$$\rho = \frac{RA}{L} \qquad (2)$$

As can be seen from Figure 8, which shows the electrical resistivity of samples, in samples containing NA admixtures the electrical resistivity has decreased by increasing the ratio of nanoparticles. In fact, 1NA sample had electrical resistivity of 18kΩ.cm while it reached 14kΩ.cm by addition of 5% nanoparticles, 22% decreased, which is still more than control specimens. However, specimens containing different percentages of Al_2O_3 are placed in a category with low to moderate corrosion rate, while in control one the corrosion rate is high, see Table3. The influential role of nanoparticles in increasing the electrical resistivity can be due to the importance of them into compacting microstructure of mixtures. In fact, the better compaction, the more electrical resistivity. Meanwhile, in this test, 1% NA can be illustrated as the best proportion of nano-Al_2O_3.

Table3. Relationship between electrical resistivity and corrosion rate [18]

Electrical resistivity ρ (kΩ cm)			Corrosion rate
>20			Low
10–20			Low to moderate
5–10			High
<5			Very high

3.2.5 Rapid Chloride Permeability Test (RCPT)

RCPT can be utilized to monitor the quality of the hardened mortar or concrete. Cylindrical samples with diameter and thickness of 10cm were cut to be changed to a cylinder with diameter and thickness of 10cm and 5 cm in accordance with ASTM C-1202 [21]. Then the transmission charge was recorded with a PC for approximately 6 hours. This is noteworthy that the obtained results indicate the strength of specimens against chloride permeability and does not determine the permeability of samples. Figure 4 shows the obtained RCPT results. As can be inferred from the results, using nanoparticles has positively influenced chloride permeability of mixtures. In fact, mixtures with 3wt% NA had the lowest charge passed amongst the other combinations as well as can be placed in a category with low chloride permeability. Although mixture with NA at 5wt% had the worst results in contrast with the other samples it still belongs to a category with moderate chloride permeability and has more resistance to chloride permeability compared to control one (Table 4). It shows that chloride permeability of the samples is mostly decreased when nanoparticles used. The resistance to chloride penetration of concrete may attribute to refining the pore structures. Consequently, as the results expressed, nanoparticles fulfill a momentous function in reducing the amount of chloride permeability with disparate ratios and so in increasing the durability of mixtures. Thus, it can be concluded that all samples, with different contents of nanoparticles, have been effectively affected through adding nanoparticles, compared with control specimens.

Table 4. Chloride Permeability Based on Charge Passed [19]

Charge Passed (Coulombs)	Chloride Permeability
>4000	High
2000–4000	Moderate
1000–2000	Low
100–1000	Very low
<100	Negligible

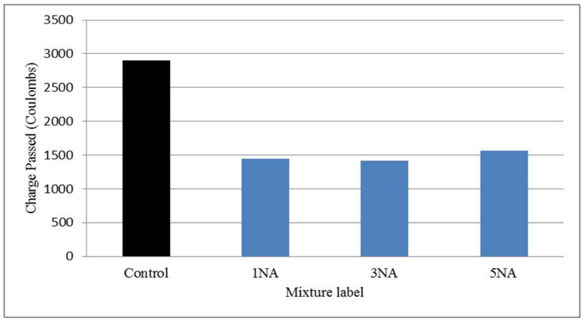

Fig 4. Rapid Chloride Penetration Test (RCPT) results

Conclusion:

The results show that the mixtures containing Al_2O_3 nanoparticles have significantly higher compressive and flexural strength in contrast with that of specimens without nano-Al_2O_3 particles at every age of curing. Moreover, it is found that the optimum level of nano-Al_2O_3 which could be advantageously replaced with cement was achieved 3.0% for the flexural test, except 1%NA. This amount would be mentioned as 1% for compressive test. Although partial replacement of cement by nano-Al_2O_3 particles reduced percentage of water absorption of concrete specimens, this ratio increased with the enhancement of nanoparticles this rate is opposite in resistivity test. In fact, the electrical resistivity of specimens decreased through enhancing the content nanoparticles and reached the lowest amount in mixtures containing 5% NA.

In terms of RCPT, using nano-Al_2O_3 improved the resistance of specimens to chloride permeability dramatically, but this ratio was not affected by increasing the content of nanoparticles.

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