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## Effects of Pozzolans and Nano Bentonite on Properties of Grout

H. Kouhsari, M. Madhkhan\*

Department of Civil Engineering, Isfahan University of Technology, Isfahan, Iran.

ABSTRACT: masonry, soil stabilization, etc. but for some applications, they need some modification in properties. Laboratory studies on the properties of cement grouts and the modification of these properties can pave the way for the greater use of these materials in applications such as the sealing of leachates of dangerous wastes buried at low depths, filling cracks in concrete bodies, etc. This study investigated the effect of silica fume, nano silica, nano bentonite, and superplasticizer on the physical and chemical properties of cement grout. For this purpose, 36 mixed designs in two separate groups for silica fume and nano silica were created. Then, the properties of fresh and hardened grout including flowability, bleeding, and 7, 28, and 90-day compressive strength were measured. The results showed that while nano bentonite and nano silica are effective in reducing bleeding, silica fume has a greater effect on this phenomenon. The greatest strength improvement was observed in the design containing silica fume and nano bentonite (CSB). While nano bentonite alone does not significantly improve the compressive strength of the grout, its combination with silica fume and nano silica has a notable positive impact on the compressive strength and bleeding of this mixture.

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#### **1-Introduction**

The cement-based grout consists of water and cement and also sand and common additives if necessary. This grout is commonly used to fill voids and cracks in concrete and masonry structures. In this respect, grout has the same use as mortar [1,2]. In geotechnical engineering, grout is used to repair and fill cracks in the body of rocks and tunnels and to control the infiltration of groundwater [3,4,5]. In recent years, grout has also found environmental applications, such as preventing the contamination of radioactive waste buried at shallow depths, soil stabilization, and landfilling [6,7,8].

The grout that is made with cement alone has many weaknesses. Depending on the application, the properties of grout can be improved to some extent by adding pozzolans and other additives. Additives can significantly alter the properties of grout, especially compressive strength, bleeding, and flowability. While grouts and their properties have been the subject of many studies, the impact of many additives on grouts is still unclear. This study aimed to examine the effect of some of these additives on the mechanical and physical properties of the grout.

Different additives have different effects on the properties of grout and other cement-based products. For example, the addition of silica fume and nano silica to concrete and mortar

mixtures is known to improve their durability, compressive strength, sulfate resistance, and physical and chemical properties in general [9,10]. Bentonite provides certain properties such as low permeability, adequate strength, and high ductility, which make it a good choice for applications such as preventing leachate leakage in landfills [11,12]. Adding kaolinite to a grout will decrease its viscosity, but increasing the cement and sodium silicate content of grout will increase its viscosity [13].

In a study on the use of high-strength cement grout in the repairing of structures, it was reported that a good combination of silica fume and natural pozzolans can give this grout a good balance between flowability, strength, and durability [14].

A study on the sealing properties of bentonite showed that the exchangeable sodium percentage (ESP) affects the sealing, swelling, viscosity, and gelation properties of this material. Also, the minimum clay content for creating normal sealing with 20% ESP was reported to be 65-75% [15].

In another study on the properties of cement grout with fly ash, polypropylene fibers, and superplasticizer, it was found adding polypropylene fibers may have an adverse effect on the properties of fresh grout but improves its resistance to cracking, shrinkage/expansion caused by wetting-drying

\*Corresponding author's email: madhkhan@iut.ac.ir



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SiO <sub>2</sub>	70.4
Al <sub>2</sub> O <sub>3</sub>	13.86
Fe <sub>2</sub> O <sub>3</sub>	2.91
CaO	1.77
MgO	2.87
Na <sub>2</sub> O	2.33
Percentage Passing Through the No. 200 Sieve	100

Table 1. Specifications of the nano bentonite used in this study (in percent).

cycles, and sulfate attack. It was also reported that adding the right amount of superplasticizer will improve the flowability, strength, and water impermeability of the grout [16]. In another study, it was shown that adding bentonite to a grout can effectively reduce bleeding, but makes the grout porous. It was also reported that silica fume has a better effect on the bleeding of the grout [17].

Mahmoud et al. examined the thermal properties of the grout. They investigated that the cement-based grout has low thermal conductivity, which improves the thermal properties by adding sand and graphite [18]. In another study sun et al. by evaluating the strength of the coal-grout materials showed that the chemical grout composites have more has higher strength in the short term while, it is decreased in the long term [19].

Tan et al. used the Taguchi method to optimize the bleeding of cement grout. They showed that silica fume and bentonite are both effective in reducing bleeding, but fly ash has not much effect on this phenomenon [20]. In another study, Tan et al. showed that the effect of bentonite on 7, 14, and 28-day uniaxial compressive strength is very small, and silica fume has a better effect on this parameter [21].

A study on the effect of bentonite and fly ash on the bleeding of the grout showed that adding bentonite reduces the bleeding but fly ash doesn't have much impact on this form of bleeding [22].

In another study on the effect of the quality of bentonite on the performance of cement slurry, it was reported that using bentonite as a filler or one of the components of grout can provide good engineering properties at a reasonably low cost, provided that bentonite used in the process is of good quality [23].

Benyounes and Benmounah studied the effect of bentonite on the rheological behavior of cement grout in the presence of a superplasticizer. This study found that the cement grouts with higher concentrations of bentonite exhibited more non-Newtonian viscosity behaviors. It also reported that increasing the bentonite content of a mixture with a constant amount of superplasticizer will lead to a sharp increase in apparent viscosity and yield stress [24].

Fernandez et al. studied the calcium aluminate silicate

hydrates (C-A-S-H) produced by the reaction between bentonite and cement or concrete [25]. This study found that the wide distribution of Al and Si due to the clay matrix act as a support factor for the formation of the C-A-S-H phase. In other words, the chemical compounds that are typically formed by the C-A-S gel resulting from the hydration of the Portland cement can be transferred by the additional Calcium produced by environmental carbonation. They also reported higher amounts of Aluminum and Silicon in the C-A-S-H phase at higher temperatures.

Another factor that can influence the outcome of using additives in cement-based mixtures is how they are mixed. In general, there are four ways to produce grout with bentonite and cement: 1- Hydrating cement and then adding bentonite; 2- Hydrating bentonite and then adding cement; 3- Dry mixing cement and bentonite; and 4- the Smith method, which involves pre-hydrating bentonite before mixing it with Portland cement [26]. Another has shown that using dry mixing or wet mixing method has an impact on the permeability coefficient of the product [27].

Because of the previous studies carried out in this area, the present study investigated the effect of the addition of silica fume and nano silica in combination with nano bentonite and superplasticizer on the compressive strength, flowability, and bleeding of the grout and examined the physical and chemical properties of the resulting product.

#### 2- Materials and Methods

#### 2-1-Materials

The materials used in this study were type-1 Portland cement produced by Isfahan Cement Factory with a specific surface area of 2900 cm2/gr, superplasticizer with the commercial name SPN215 produced by Capco Co. (Iran), silica fume produced by Iran Ferroalloys Industries Co. (Iran), nano silica produced in the form of solid powder by Evonik (Germany), and nano bentonite (100% passing through the No. 200 sieve) produced by a mine in Sarayan, South Khorasan, Iran. The specifications of the nano bentonite according to the XRF test performed in the Department of Chemistry of the Isfahan University of Technology are given in Table 1.

Name	w/c	B (%)	S (%)	Name	w/c	B (%)	S (%)	Name	w/c	B (%)	S (%)
С	0.6	0	0	С	0.8	0	0	С	1	0	0
CSB1		1	15	CSB1		1	15	CSB1		1	15
CB2		2	0	CB2		2	0	CB2		2	0
CS		0	15	CS		0	15	CS		0	15
CB1		1	0	CB1		1	0	CB1		1	0
CSB2		2	15	CSB2		2	15	CSB2		2	15

Table 2. Mix designs with silica fume and nano bentonite.

Table 3. Mix designs with nano silica and nano bentonite.

Name	w/c	B (%)	N (%)	Name	w/c	B (%)	N (%)	Name	w/c	B (%)	N (%)
С		0	0	С	0.8	0	0	С	1	0	0
CNB1		1	1	CNB1		1	1	CNB1		1	1
CB2	0.6	2	0	CB2		2	0	CB2		2	0
CN	0.6	0	1	CN		0	1	CN		0	1
CB1		1	0	CB1		1	0	CB1		1	0
CNB2		2	1	CNB2		2	1	CNB2		2	1

#### 2-2- Experimental design, specimen preparation, and curing

The experiments of this study were performed on two types of cement-based grout mix designs: (1) a mix design containing silica fume, nano bentonite, cement, and superplasticizer; and (2) a mix design containing nano silica, nano bentonite, cement, and superplasticizer. Given the ratios considered for the components of the grout and the additives, a total of 36 mix designs (18 with silica fume and 18 with nano silica) were created. The names and details of these mixed designs are given in Tables 2 and 3. Using the method instructed in BS1170 [28], the superplasticizer content was calculated to be 1.2% (Fig. 1). For this purpose, first, the test was performed for grout with a specific flowability, and then the superplasticizer was added until the grout subsided to the desired ring.

To make the grout, the bentonite was pre-hydrated with a bentonite/water weight ratio of 9:1 and cured for 24 hours. Then, the total water content of the mix design minus the amount used in the pre-hydration process was poured into the mixer. The additives (silica fume or nano silica) were then slowly added and left to mix for 3 minutes. The pre-hydrated bentonite was added one minute before mixing ended [6]. The method proposed by Li et al. [29] was used to make sure of the proper mixing of nano silica-containing mixtures. For this purpose, the nano silica powder was added to some of the design water and mixed for 5 minutes before being added to the grout mixture. For measuring 7, 28, and 90-day compressive strength, the grout was poured into  $70 \times 70 \times 70$  mm cubic molds and placed in a humid room for 24 hours. Then, the specimens were taken out of the mold and placed in water for 7, 28, or 90 days.

The names of mix designs containing silica fume are given in Table 2. In this table, C denotes the designs that only contain cement, B denotes the designs that contain nano bentonite and the number shows the amount of bentonite used in the mix (in percentage), and S denotes the design that contains silica fume. The numbers in front of C show the w/c ratio of the design, which could be 0.6, 0.8, or 1. The mix designs containing nano silica are also named in the same way, except that nano silica is denoted by N instead of S. The percentage of nano silica in mixed designs was considered 0 and 1 percent. The mix designs containing nano silica are shown in Table 3.

#### **3- Results and Discussion**

#### 3-1-Flowability

Flowability is an important property of the grout which completely depends on its mixed design. A good flowability is essential for the grout to be injectable and reach the depth of cracks and small discontinuities. The flowability of the grout was measured using the flow cone method in accordance with ASTM C939 [30]. An image of the flow cone used in this method is shown in Fig. 2.



Fig. 1. The test performed to determine the optimum amount of superplasticizer.



Fig. 2. Measurement of flowability using the flow cone method.

In this method, 1.725 liters of freshly made mixture is poured into the flow cone and the time of efflux, which is the time it takes for the mixture to pass through the outlet once the valve is opened, is measured. It should be noted that the time of efflux of 1.725 liters of water is  $8\pm0.2$  seconds. Also, the flowability of those materials whose efflux time is longer than 35 seconds should be measured by the flow table. The results of this test for the grouts made in this study are presented in Figs. 3 and 4.

As expected, using higher W/C ratios increases the flowability of the grout. In applications where high flowability is essential, it is reasonable to increase W/C, but it should be remembered that this may adversely affect the properties of the grout in both fresh (e.g., bleeding and efflux time) and hardened (e.g., compressive strength) states. As shown in Figs. 3 and 4, adding additives to the grout reduces

its flowability. As bentonite content increases from 1% to 2%, efflux time increases and flowability decreases. Because of the high specific surface area of silica fume and nano silica, the grouts containing these pozzolans require more water to react with all pozzolanic grains. As a result, in these groups, excess water remaining from cement hydration is consumed in the reaction with silica fume and nano silica, leaving little water to provide flowability. The bentonite-containing grouts also show decreased flowability, which can be due to two reasons: 1- the consumption of water in the decomposition of montmorillonite of bentonite; 2- the use of a portion of design water for pre-hydration of bentonite (in 9:1 ratio), which is deducted from the water available for reaction with cement. Therefore, adding bentonite to the mix design also leads to reduced flowability. As the bentonite content increases from 1% to 2%, the amount of water needed to pre-hydrate



Fig. 3. Time of efflux of the grouts containing silica fume and nano bentonite.



Fig. 4. Time of efflux of the grouts containing nano silica and nano bentonite.

bentonite also increases, which leads to a further decrease in flowability. As the results show, designs containing silica fume have longer efflux times than designs containing nano silica. Given the higher specific surface area of nano silica, it is expected to cause a greater reduction in flowability. Therefore, the longer efflux time of grouts containing silica fume can be due to the larger amount of silica fume consumed in the grouts used with this additive.

#### 3-2-Bleeding

Bleeding refers to the appearance of water on the surface of concrete or grout before hardening; an adverse phenomenon that is caused by the inability of the solid grains to hold all the water of the mixture. This parameter was measured by performing the standard test for the expansion and bleeding of freshly mixed grouts in accordance with ASTM C940 [31]. In this method,  $800\pm10$ , milliliters of grout are placed in a graduated cylinder and the volume and time of the start of the test are recorded. Then, changes in the volume of the grout are measured every 15 minutes. This continues until there is no change in the volume of the grout in two consecutive readings. After these measurements, bleeding is quantified

$$Bleeding = \frac{V_W}{V_1} \times 100 \tag{1}$$

Figs. 5 and 6 show the bleeding of mixed designs containing silica fume and nano silica. As these figures show, increasing the W/C ratio increases the bleeding dramatically. As a result, mix design C1 has the highest rate of bleeding among the designs (7.5%). When there is more water in the cement paste, more water can pass through the capillary pores in the cement and move toward the free surface, which results in the accumulation of more water on the surface of the specimen. The water rising through the capillary pores can dissolve the cement compounds and bring them along to the surface, thereby reducing the adhesion and increasing the porosity of the matrix, which results in a permeable product with reduced durability. Adding bentonite, silica fume, or nano silica can effectively reduce the bleeding phenomenon. Among these additives, silica fume has the greatest effect on bleeding. At W/C=0.6, the grouts containing pozzolans



Fig. 5. Bleeding of the grouts containing silica fume.



Fig. 6. Bleeding of the grouts containing nano silica.

(C6N and C6S) showed zero bleeding. At higher W/C ratios, however, there was a slight increase in the bleeding. Nano bentonite also reduced bleeding but was not as effective as silica fume or nano silica. The cause of reduced bleeding in grouts containing nano bentonite is the consumption of water in the pre-hydration phase and in the decomposition of montmorillonite, which leaves less water in the cement paste for bleeding.

#### 3-3-Compressive strength

The compressive strength of grout depends on the structure of its cement paste and can serve as a good measure of its quality. In this study, compressive strength was measured according to ASTM C109 [32] (Fig. 7). This test was performed on cubic specimens of size  $7 \times 7 \times 7$  cm after 7, 28, and 90 days of curing. Figs. 8 to 13 show the measured strength of grouts containing silica fume and nano silica specimens.

As can be seen, silica fume (15%) and nano silica (1%) both increase the compressive strength of the grout. As the W/C ratio increases, the time it takes for the grouts containing silica fume and nano silica to reach an acceptable level of

compressive strength decreases. For example, the grouts containing silica fume and nano silica made with W/C=0.6 have a higher compressive strength than the control grout. However, the higher the W/C ratio is, the lower the ultimate improvement in the compressive strength of mix designs containing silica fume and nano silica.

The results also show that the addition of nano bentonite does not have much impact on the strength, only improving it in some designs. The addition of bentonite has slightly decreased the compressive strength at younger ages, especially in the designs with high W/C ratios; an effect that can be attributed to increased porosity in the cement paste. With the increase in nano bentonite content from 1% to 2%, the compressive strength has increased more rapidly at an early age and occasionally exceeded the strength of the control design (without additives). It seems that a combination of fine-grained silica fume and nano silica with course-grained bentonite has been able to form a compact mixture with a slightly improved strength. The mix designs containing silica fume or nano silica in combination with nano bentonite have significantly higher compressive strength than the control design. For example, the 7-day compressive strength of these designs (with W/C=0.6, 0.8, and 1) is roughly 1.5 times



Fig. 7. Fracture of the specimen containing silica fume and nano bentonite in the compressive strength test.



Fig. 8. 7-day strength of grout containing silica fume.



Fig. 9. 28-day strength of grout containing silica fume.



Fig. 10. 90-day strength of grout containing silica fume.







Fig. 12. 28-day strength of grout containing nano silica.



Fig. 13. 90-day strength of grout containing nano silica.



Fig. 14. 7-day compressive strength of the mix designs containing silica fume and nano silica.

that of the control design. The CSB1 designs have a higher compressive strength than the CSB2 designs. However, this difference decreases with age and the increase in W/C. This difference can be attributed to two reasons: first, the reaction of silicon and aluminium in silica fume and bentonite with the by-product of hydration (calcium hydroxide) leads to the formation of C-S-H gel [33], which results in greater strength. Second, the combination of fine-grained material such as Silica Fume with a coarser material such as bentonite creates a mixture with good compaction and therefore higher strength than the designs with silica fume or nano silica alone.

Among the tested designs, those with silica fume and nano silica, and 1% nano bentonite have the highest strength improvement because of their compact structure. Among these designs, those with lower W/C ratios are stronger.

# 3- 4- Compressive strength of mix design containing silica fume and nano silica

To better illustrate the effect of additives on the compressive strength of the grout, their strength diagrams

are plotted in Figs. 14 to 16. At the age of 7 days, the Mix designs containing silica fume have a higher compressive strength than nano silica ones, but at older ages, the opposite is true. This could be because of the high specific surface area of nano silica compared to silica fume, and the fact that it has higher SiO2 available for reacting with calcium hydroxide, which is a by-product of cement-water reaction in the formation of C-S-H gel [34].

#### 4- Conclusion

This study investigated the effect of three additives, namely silica fume, nano silica, and nano bentonite on the physical and mechanical properties of cement-based grout. The results of this investigation are summarized below.

In general, adding silica fume, nano silica, and nano bentonite to the grout increases its efflux time, which means reduced flowability.

Silica fume and nano silica have the greatest impact on the bleeding of the grout. Although bentonite also has a positive effect on bleeding. At low W/C ratios, grouts containing



Fig. 15. 28-day compressive strength of the mix designs containing silica fume and nano silica.



Fig. 16. 90-day compressive strength of the mix designs containing silica fume and nano silica.

silica fume and nano silica have almost zero bleeding. In the designs with W/C=0.6, the presence of 1% and 2% bentonite reduced the bleeding by 61 and 74%, respectively.

The addition of silica fume and nano silica to the mix design has a positive effect on the compressive strength of the grout at all ages. The addition of nano bentonite reduced the 7-day compressive strength, especially at lower bentonite ratios. As grouts containing nano bentonite aged, their compressive strength became almost constant. In some of the specimens with 2% nano bentonite, there was a slight increase in compressive strength.

At the age of 7 days, the designs containing silica fume have higher compressive strength than the nano silica ones, but at the age of 28 and 90 days, the designs containing nano silica exhibited higher compressive strength than those containing silica fume.

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