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Essential Improvements in Gypsum Mortar Characteristics

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ABSTRACT

Gypsum mortar is a common building material that can be used especially for plastering the walls. This mortar has three important weaknesses which can limit the gypsum mortar for building and statue construction. First; it has low compressive strength. Second; it has high water absorption, and third; it has low setting time. In the current study, cement, Nano silica, and a superplasticizer with polycarboxylate ether were used for solving the problems. The results showed that using cement with providing C-S-H can improve the mortar strength trend line. The results showed significant growth of 28th day compressive strength (from 9 MPa to 45 MPa). Using Nano silica increases the compressive strength by making C-S-H dense and decreases the water absorption to 1/3 of the control sample. Consuming polycarboxylate ether causes the uniform dispersion of Nanoparticles through mortar. This even diffusion blocks the pores and reduces their mean dimensions. The ANOVA test was used to find the main effective parameters on the 28th day compressive strength, water absorption, and setting time. In this regard, Nano silica (49.82% contribution), cement content (56.68% contribution), and superplasticizer (73.10% contribution) have the main roles in compressive strength, water absorption, and setting time, respectively.

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NOMENCLATURE

Subscripts

C	Abbreviation of cement	*MS*	Mean squares
P	Plasticizer	*DF*	Degree of freedom
N	Nano silica	*PCS*	Polycarboxylate ether superplasticizer
W	Water	*SEM*	Scanning electron microscopy
SS	Sum of squares	*TEM*	Transmission electron microscopy

1. INTRODUCTION

Mortars are the main building materials [1, 2] consisting of cement, water, gypsum, additives, etc. [3, 4]. Gypsum mortar with a special function in the building industry has its users [4]. Working with this material has its intricacy and delicate. So, it needs special skills and tricks. It is used to cover different surfaces and make them decorative. Researchers have investigated many methods or additives to provide a more stable mortar. For example, Morsy et al. [5] investigated the properties of a cementless mortar. They used gypsum with different percentages. Their results indicated that gypsum's addition in a certain amount had a remarkable effect on flexural and compressive strengths. In another study, removing cement was conducted using Flyash, Nano silica, and glass powder. This work enhances the mechanical properties and can help prevent the CO_2 from releases to the atmosphere [6]. In another case, Alexandra et al. [7] studied interactions between the hydration of alite and gypsum in cement compounds. They concluded that the interaction of gypsum with

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Aluminium accelerates the reaction of alite. In fact, gypsum has a positive effect on the composition of cement.

Naik et al. [8] replaced Fly Ash with conventional bricks. These bricks used cement and gypsum in their composition. This new brick can be used for constructing low-cost houses in the vicinity of the thermal power plant. Taha et al. [9] produced a new natural building material consisting of soil, cement, gypsum, and straw fibers. Their product can decrease the thermal conductivity; increasing fiber, cement, and gypsum are the reason for this phenomenon. They also demonstrated that barley straw fiber-reinforced bricks could have the highest values of thermal insulation. Frank et al. [10] evaluated gypsum's effects on the hydration of calcium sulfoaluminate cement. In this research, the amount of anhydrite was replaced by gypsum. Eventually, they found that the replacement of a part of the anhydrite by gypsum can improve hydration kinetics and compressive strength of mortar. Jeong et al. [11] examined the effectiveness of different gypsum and water quantity on the strength of the cement paste consisting of calcium sulfoaluminate belite. The results showed that the amount of gypsum controls the hydration of ye' elimite and belite in the cement composition. In another study conducted by Magdalena et al. [12], the rheology of gypsum compounds at high temperatures was investigated. In this study, the results demonstrated that pure gypsum has significant resistance at high temperatures. During a research Fernandez et al. [13] added nano-silica (NSI) and polycarboxylate ether superplasticizer (PCS) to aerial lime mortars. The addition of PCS could enhance the flowability of lime mortar. Besides, the setting time accelerated. The existence of either NSI or PCS in lime mortar can improve mechanical strengths. As a result of combining two materials, the microstructural of lime mortar modified and leads to the optimized mix design. Suleyman et al. [14] implemented the production of the mix design with polycarboxylate ether. It was proved that polycarboxylate ether with main chain length has specific actions on compressive strength, ultrasonic pulse velocity, and water absorption capacity. The results also indicated that the increase in main chain length can improve the mixtures' time-dependent flow performance. Changes in length also have significant effects on adsorption behavior. In another research conducted by Shengnan et al. [15], the chemical structure of polycarboxylate superplasticizer (PCS) in cement-based materials was investigated. The main chain length of PCS provides procrastination for the hydration of cement. Gypsum mortars have many defects that limited their functions. Low strength and setting time besides high water absorption are their main weaknesses. The authors' searches showed that enough efforts to improve the gypsum mortar characteristics have not been made. In this regard, with the combination of white cement, polycarboxylate ether, and nano silica, some successful attempts were made. "The current study is organized into three main sections. Section (2) is designated the experimental program described as materials, mix design, and test procedures. Section (3) is the result and discussion which discussed the achievements and mechanical and statistical results."

2. EXPERIMENTAL PROGRAM

Four main tests such as compressive strength, water absorption, setting time, and scanning electron microscope (SEM), were done for evaluating the mechanical properties of the Gypsum mortar. Also, for finding the main significant parameter, the ANOVA test was done by XLSTAT V. 2016. The details of the experimental program are as follows.

2. 1. Materials Introduction For producing the mortar, gypsum, white cement, superplasticizer, water, and nano silica were used. The gypsum was from the Semnan gypsum factory by the Aeineh trademark. After transferring gypsum stone to the factory, it crushed and entered the cooking kiln for about one hour. Then, it has been grinded as special particle sizes. Cement was manufactured by the Semnan cement factory. The superplasticizer was Polycarboxylate Ether from LG (White Damavand) factory. It has a light brown color, less than 0.01 chloride ion, pH of 7, and 1.08 density. It is necessary to use a kind of water that has no harmful material for mortar making. In this regard, the potable water of Shahrekord was used. Silicon oxide nanoparticles were Aerosil®200 with white color, 200 m^2/g specific surface area, purity of +99%, and 11-13nm particle dimension. Figure 1 shows the TEM of utilized nano SiO_2.

2. 2. Mix Design First gypsum and cement were mixed by 50:50 ratios for 3 minutes in a 5L drum. After making a solution with superplasticizer (1% and 1.5% of the total weight of mixture), water, and nano silica (0.5, 0.75, and 1% of cement weight) was added. The stirring of solution continued until complete dispersion of

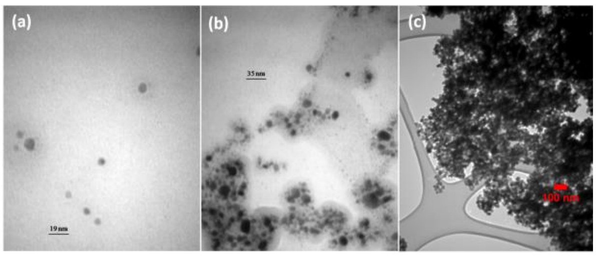

Figure 1. TEM of Nano silica particles with different scales; a) 19 nm, b) 35nm, c)100nm

particles in it. The solution poured into the drum. The mixing procedure was sustained for 5 minutes with average speed. It should be noted that the three end minutes were done by the higher speed of the drum. Table 1 shows the mix design of the current study, and the methodology is as Figure 2. It is important to save a sequence. First, the desirable flowability should be seen. After gaining a sample with enough setting time, the other steps, such as compressive strength and water absorption, can be done. Finally, samples with the highest compressive strength will be the gypsum mortar, problem solvers. Table 1 summarized, C the abbreviation of cement, N is used as nano silica, and P is the plasticizer. For compressive strength, water absorption 5 cm cubic molds were poured by mortar. The molds were opened after 1st, 7th, and 28th days after water curing at 25°C (ASTM C 109). The setting time test was done on the first day of mortar production. Section 2.3 explains the experimental test procedures.

TABLE 1. Mix design

Mix label	Nano SiO_2* (%)	Superplasticizer* (%)	W/C
C5P1	0	1	0.4
C5P1.5	0	1.5	0.4
C5P1N0.5	0.5	1	0.5
C5P1N0.75	0.75	1	0.5
C5P1N1.0	1	1	0.5
C5P1.5N0.5	0.5	1.5	0.5
C5P1.5N0.75	0.75	1.5	0.5
C5P1.5N1.0	1	1.5	0.5

*weight ratio of cement

2. 3. Test Procedures

2. 3. 1. Compressive Strength After curing, samples were broken by using a 2000 KN underload hydraulic jack. Since the samples had relatively small dimensions, they were stand in the special flexible encasement. With increasing the load, the case's ceil is coming down and distributing the uniform pressure in each direction. The samples were broken after 1st, 7th, and 28th days.

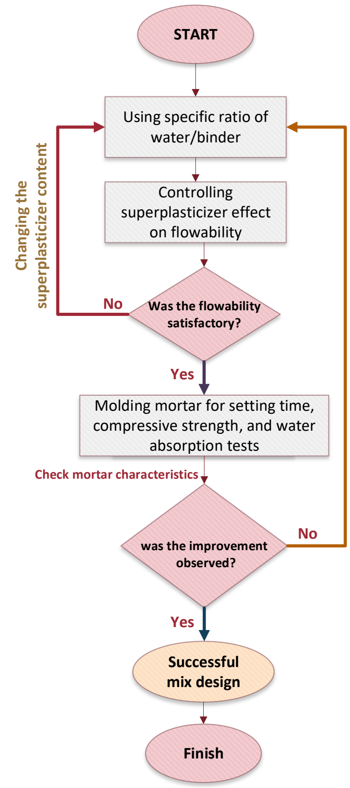

Figure 2. Methodology flowchart

2. 3. 2. Water Absorption The water absorption test was implemented after 28 days of curing. Based on ASTM C 642, the saturated surface dry (SSD) samples were weighed. Then samples were kept in an oven for 24 h at 110 ± 5ºC. Finally, samples were weighed again.

2. 3. 3. Setting Time The test was done based on ASTM C 191 by Vicat needle experiment. In this regard, a needle with 1.13 mm diameter and 300g weight was released on the mortar surface during some special periods (Figure 3, shows the Vicat instrument). The ruler instrument measured penetration. The initial and final time of the setting was measured to determine the hardening time of the mortar.

2. 3. 4. Scanning Electron Microscope (SEM) SEM was conducted by AIS 2100 from Seron

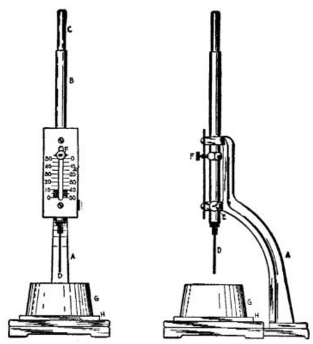

Figure 3. Vicat instrument (ASTM C 191); A) frame, B) movable rod, C) plunger end, D) removable steel needle, E) setscrew, F) adjustable indicator, G) cone frustum as mortar holder, H) glass slide

technology. For preparing samples first, a section was taken out from the 28th day crushed specimen after breaking by a hydraulic jack. It was sieved through a 125 µm mesh and prepared for gold coating. Pictures were detected with 1 and 10 µm scales.

3. RESULTS AND DISCUSSION

3. 1. Compressive Strength Figure 4, illustrates the compressive strength test results. After adding cement with 50% of the mortar's total weight, the compressive strength shows the improvement trend, especially after 28 days. Subsequently adding the Nano silica particles to the mortar, the compressive strength improves significantly.

For example, C5P1.5N1 (with 42MPa) compared to the control sample (with 7.2 MPa) proves the new mix design's good performance. Using cement provides an opportunity for strength progresses. So as time passes and cement hydration completion, the mortar microstructure becomes denser [16]. The development of C-S-H is the main reason for strength enhancement. Nanomaterials have various behavior. In almost all of them, they can fill the nanopores of mortar or cement, decrease the porosity, and develop the strength. But nano silica with a silicate base can improve the C-S-H purity and make it denser [17]. It is interesting to pay attention to the C5P1N0.75 and C5P1N1 strength differences with C5P1.5N0.75 and C5P1.5N1. The superplasticizer with polycarboxylate Ether base provides a particular property. It is clear that by rises the Nanoparticles, the water demand increases [18, 19]. Providing higher amounts of water can prevent gaining strength by causes the pores due to the higher W/C ratio. So superplasticizer maintains the workability and reduces the need for water [20]. The longer chain of polycarboxylate can increase the hydration level. Moreover, it disperses nanoparticles uniformly through mortar. This uniform diffusion blocks the pores and reduces their mean dimensions [13]. Doleželová et al. [21] improved the strength of the mortar by using slag. Slag can increase strength, but it raises the weight significantly. Nano silica has a superior effect. It enhances the strength and does not change the weight of samples. It is worth attention that micro-silica (silica fume) can not provide acceptable strength results compared to nano silica [22].

3. 2. Water Absorption Figure 5 shows that the control sample has the highest water absorption rate (approximately 27%). Based on what Krejsová et al. [23] showed, the high water absorption is due to large, primarily pores of gypsum (1µ-3µm). The gypsum, without any processing, can absorb water and moisture from the air. So for humid countries, the gypsum should not be used as plasters or other products. Since it absorbs water, expands, and collapses.

In some situations, it shows the yellow stain and becomes dirty. In this regard, a distinctive process should be done. Using cement reduces the water absorption to around 20%. It is not satisfactory. The water absorption index is high enough to cause the gypsum crash. Using silicate Nanoparticles as additive besides polycarboxylate ether can solve this problem. Nanoparticles fill the pores, help the generation of C-S-H [23], and increase the mortar density. Nevertheless, they have a high specific area that can grow water demand and negatively affect mortar water absorption. Using polycarboxylate can inhibit the water addition and make W/C constant [24]. Besides, it disperses nano SiO_2 through cement particles and makes an agglomeration by bonding bridges between them [13, 14, 25]. Figure 6 shows the polycarboxylate effects on dispersion and bonding of cement and Nanoparticles. A sample such as C5P1N1 has 10% water absorption, nearly 1/3 of gypsum mortar absorption (Control sample).

3. 3. Setting Time Figure 7 shows the results of the final setting time by the Vicat experiment. For gypsum mortar without any processing, the setting time happens immediately (5 min). This prevents amateur workers from appropriate working with gypsum. Adding

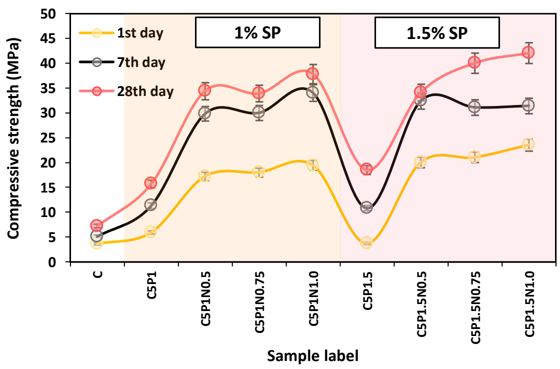

Figure 4. Compressive strength of samples

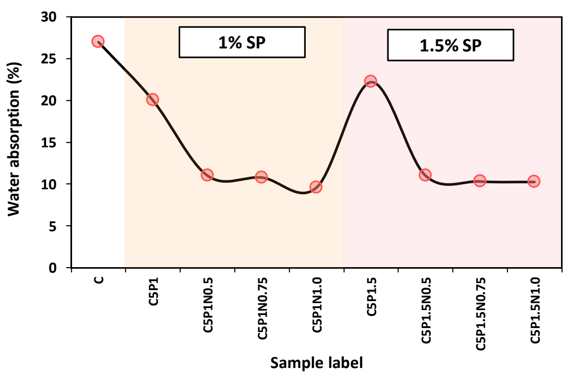

Figure 5. Water absorption of samples

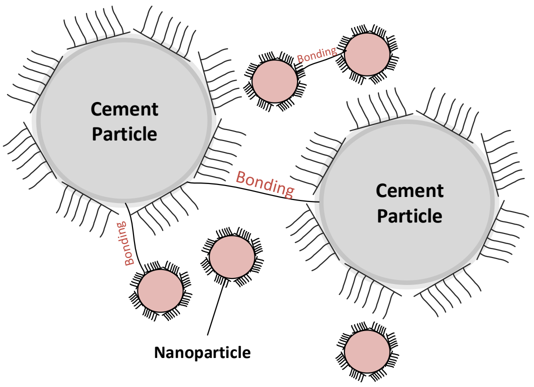

Figure 6. Long chains and bonding bridges in processed gypsum mortar with Nanoparticles, cement, and polycarboxylate ether

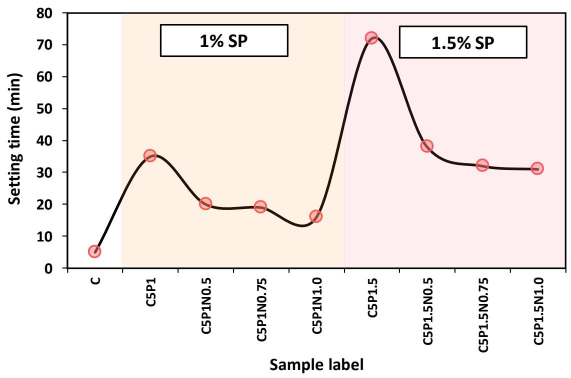

Figure 7. Results of setting time of gypsum mortar

superplasticizer causes a longer time of setting. Increasing the superplasticizer from 1 to 1.5% makes mortar retarder. For example, C5P1.5N0.5 need 70 minutes for the setting. It is ideal for the sculptors and plastering of walls.

It should be noted that using Nano silicate with a high specific area can cause lowering the setting time. So for controlling the hardening of mortar and providing flowability, adjusting the superplasticizer content is necessary.

3.4. SEM Analysis

As evident in Figure 8, the mortar has a homogenous and dense shape (a), and C-S-H can be obvious by flat hexagonal shape (b). The presence of pseudohexagonal, flat surfaces, and blade shapes are evidence of gypsum crystals (c). They were also detected at [26].

3.5. ANOVA

3. 5. 1. ANOVA for 28th Day Compressive Strength

For providing a better index of finding the most influential parameter on mortar strength, the ANOVA test was utilized. With a dataset of some samples with different cement and superplasticizer contents, the ANOVA was done. Table 2 shows the degree of freedom (DF), Sum of squares (SS), Mean squares (MS), and contribution of each parameter on compressive strength.

As expected, Nano silica, with an approximate 50% contribution, has the leading role on 28th day compressive strength. Although, the effects of cement content on mortar strength property can not be ignored. The superplasticizer, with a 1.2% contribution, has no significant effects on mortar strength characteristics.

3. 5. 2. ANOVA for Water Absorption

The ANOVA test for a setting time showed that cement content with about 57% contribution has the most crucial part in the mortar's water absorption behavior. Table 3 demonstrates the ANOVA results for water absorption effective parameters. Besides, superplasticizer with nearly under 1% contribution, has no active role. So for adjusting the gypsum mortar water absorption paying attention to cement content is crucial.

3. 5. 3. ANOVA for Final Setting Time

The ANOVA test also was used for setting time. With a dataset of some samples with different cement and superplasticizer contents, the ANOVA was done. Table 4 shows the contribution of each parameter in the final setting time.

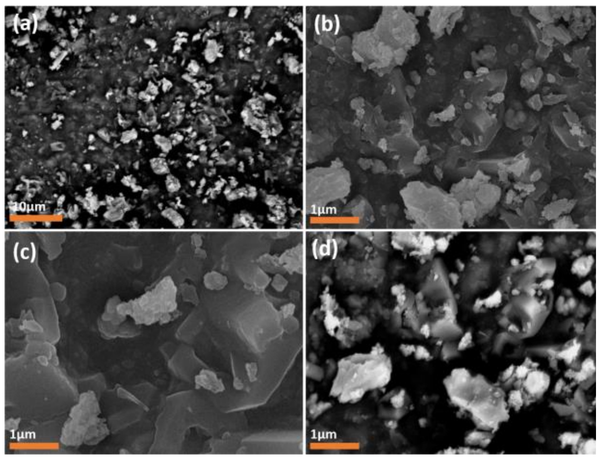

Figure 8. SEM analysis of gypsum mortar (C50S1.5N1 sample)

TABLE 2. ANOVA for 28th day compressive strength

Source	DF	SS	MS	contribution (%)
Cement	4	574.13	143.534	46.210
Nano silica	3	619.01	206.338	49.822
Superplasticizer	2	15.42	7.7130	1.241
Error	8	33.87	4.234	2.726
Total	17	1242.44	361.820	100

TABLE 3. ANOVA for water absorption

Source	DF	SS	MS	contribution (%)
Cement	1	342.60	342.60	56.68
Nano silica	1	228.49	228.49	37.80
Superplasticizer	1	0.81	0.81	0.13
Error	14	32.56	2.32	5.39
Total	17	604.47	574.23	100

TABLE 4. ANOVA for final setting time

Source	DF	SS	MS	contribution (%)
Cement	4	681.29	170.323	8.90
Nano silica	3	544.37	181.458	7.11
Superplasticizer	2	5597.0	2798.508	73.10
Error	8	833.81	104.227	10.89
Total	17	7656.5	3254.516	100

With an approximate 73% contribution, superplasticizer was the most influential parameter in setting time. This verifies the experimental results (section 3.3).

4. CONCLUSION

Gypsum mortar is a common building material that can mostly be used for plastering the walls. This mortar has three important weaknesses, which can limit the gypsum mortar for building and statue construction. First, it has a low compressive strength. Second, it has high water absorption, and third, it has a short setting time. In the current study, the authors focus on solving the mentioned problems. In this regard, cement, Nano silica, and a superplasticizer with polycarboxylate ether base have been chosen. The results of mechanical tests are as follows:

- The compressive strength with using white cement shows the improvement trend. Cement generates tobermorite and changes the microstructure of mortar. Then, C-S-H by traping the gypsum crystals can increase the density. It is the main reason for reaching 45MPa (C50P1.5N1) from 9 MPa (Control sample).
- Adding Nano silica increases the compressive strength by making C-S-H dense. It reduces the capillary pores and the water absorption to 1/3 of the control sample. For C5P1.5N1, the water absorption showed about 10%, nearly 1/3 of the control sample water absorption.
- Using polycarboxylate ether causes the uniform dispersion of Nanoparticles through mortar. This even diffusion blocks the pores and reduces their mean dimensions.
- The results also proved that polycarboxylate ether increases the setting time without extra water to the mixture. So it can help the compressive strength and water absorption properties by keeping the W/C ratio constant.
- The samples with 1.5% superplasticizer have longer setting times. C5P1.5, with about 70 min, is the most retarder sample.
- Using Nanosilica with a high specific area can develop the C-S-H and reduce the setting time. So for Nano modified samples such as C5P1.5N1 (30 min), although the setting time reduced from 70 to 30 min, it is acceptable compared to the control sample with about 5 min.
- SEM pictures showed the C-S-H hexagonal microstructures, which are dispersed through mortar. Also, they showed pseudohexagonal, flat surfaces, and bladed shapes of gypsum crystals. In these pictures, the ettringite did not detect.
- The results of ANOVA for finding the main effective parameter on 28th day compressive strength showed that Nano silica has the leading role in compressive strength (49.82% contribution) and cement content (46.21% contribution) has the second important character on mortar strength property.
- The contribution of cement, Nano silica, and superplasticizer proved that cement content (56.68% contribution) significantly affected mortar water absorption. The superplasticizer with about under 1% contribution has not active participation in water absorption of mortar.
- ANOVA also proved that superplasticizer (73.10% contribution) is the most influential parameter on mortar setting time, and Nano silica can not play a major role in it (7.11% contribution).

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Persian Abstract

چکیده

ملات گچی دارای سه ضعف مهم است که می‌تواند آن را برای ساخت مجسمه و ساختمان‌ها محدود کند؛ دارای مقاومت فشاری پایین ، جذب آب زیاد و زمان گیرش کم است. در مطالعه حاضر، از سیمان، نانو سیلیس و یک ابر روان‌کننده با پایه‌ی پلی کربوکسیلات اتر برای حل مشکلات استفاده شد. نتایج نشان داد که استفاده از سیمان با ایجاد C-S-H می‌تواند روند رشد مقاومت را بهبود بخشد. نتایج مقاومت فشاری ۲۸ روزه نشان داد (از ۹ مگاپاسکال به ۴۵ مگاپاسکال) استفاده از نانو سیلیس نه تنها با ایجاد تراکم C-S-H باعث افزایش مقاومت فشاری می‌شود، بلکه باعث کاهش جذب آب به نسبت یک سوم نمونه شاهد می‌گردد. مصرف پلی کربوکسیلات اتر باعث پراکندگی یکنواخت ذرات نانو در ملات شده که این امر حتی حفرات را مسدود کرده و ابعاد متوسط آنها را کاهش می‌دهد. همچنین، از آزمون ANOVA برای یافتن پارامترهای اصلی موثر در مقاومت فشاری ۲۸ روزه، جذب آب و زمان گیرش استفاده شد. در این راستا، نانو سیلیس (۴۹/۸۲ درصد مشارکت)، سیمان (۵۶/۶۸ درصد) و فوق روان کننده (۷۳/۱۰ درصد) به ترتیب نقش‌های اصلی در مقاومت فشاری، جذب آب و زمان گیرش دارند.