Enhancing the Properties of Sulfate-Resisting Cement

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ABSTRACT

Sulfate-resisting cement is used in construction works when a sulfate attack is probable. This type of cement withstands sulfates due to its low C_3A content. On the other hand, the decrease in the quantity of C_3A leads to a reduction in the rate of early strength development. To overcome this problem, a hardening accelerator was added to the cement mix. To estimate the effect of the accelerator on some properties of hardened cement, compressive and flexural strength, and drying shrinkage tests were carried out. Four series of cement mortar mixes were made. The hardening accelerator was not added to the control series, while it was added to the others with three different percentages (of cement mass) of 0.5, 1, and 1.5%. The results revealed that the hardening accelerator enhances the compressive strength of all mortar ages while it slightly promotes flexural strength only at early ages. It was also observed that the hardening accelerator strongly inhibits the drying shrinkage strain.

Keywords-sulfate-resisting cement; hardening accelerator; flexural strength; compressive strength; draying shrinkage

I. INTRODUCTION

When a cement matrix is in contact with soil contaminated with sulfate, harmful reactions occur in the presence of water. These reactions, which are called the sulfate attack, finally lead to the deterioration of the construction member. In hardened cement paste, tricalcium aluminate hydrate (the result of reacting C_3A with water) reacts with sulfate to produce calcium sulfoaluminate (ettringite - $3CaO.Al_2O_3.3CaSO_4.32H_2O$), which is formed within the framework of the hydrated cement paste and has a larger volume than the reacted solid material. At early ages, the ettringite is helpful as it fills the capillary pores and thus makes the cement matrix denser [1]. However, if this reaction continues, even when the cement takes its strength, it leads to damages of the cement matrix due to the stresses generated by the expanded ettringite [1-3]. To overcome this problem and enhance the resistance of mortar or concrete of a structural member to the sulfate attack, Sulfate-Resisting (SR)-cement is employed. This type of cement resists the sulfate due to its low C_3A content [4]. On the other hand, C_3A is one of the cement phases that contribute to the early strength of cement paste due to its fast hydration reaction. Since SR-cement has low content of C_3A, it has a low early strength. There are cases that require high early strength of cement matrix, e.g. when the need arise to remove formworks in a short time for reuse in casting another member, whereas the concrete should acquire adequate strength before removing the formwork [5-7]. In addition, speeding up the early strength development is one of the most important requirements for repair works [8]. It gets worse when the construction work is carried out in cold weather, as cold slows down the reaction of the cement compounds.

Generally, in construction works, different types of accelerators [9] are used to reach the required strength quickly enough. The accelerators act as the reaction catalyst for one or more cement phases, specifically C_3S, C_3A, and C_4AF [10-15]. According to BS EN 934-2 [16], the accelerators are subdivided into two main categories, setting accelerators which are used to reduce setting time and hardening accelerators for increasing the early strength of cement paste. In the implementation of various construction works and researches, different types of accelerators have been used with Ordinary Portland Cement (OPC) [17-19].

The studies conducted on the effect of accelerators on the performance of SR-cement are rare, with [20] being one of the few. The current study aims to increase the early strength of this type of cement by adding a hardening accelerator. Hence, compressive and flexural strength tests were investigated. In addition, drying shrinkage tests were performed to check the cracking tendency as it affects the durability and the sustainability which is the focus of many studies nowadays [22-24].

II. EXPERIMENTAL PROGRAM

A. *Mortar Mix Properties and Proportions*

The SR-cement used in this research is classified under the type CEM I-SR 3 [24]. Its fineness is $4875cm^2/g$, while its initial and final setting times are 2:45h and 4:45h, respectively. The oxide and the compound compositions of this cement are shown in Table I.

TABLE I. COMPOSITIONS OF SR-CEMENT

Oxide compositions (%)		Compound compositions (%)	
CaO	60.71	C_3S	48.48
SiO_2	20.02	C_2S	21.06
Al_2O_3	4.23	C_3A	2.30
Fe_2O_3	5.27	C_4AF	16.02
MgO	1.81		
SO_3	2.38		
LOI	3.69		

The SR-cement has lower strength than OPC at early ages. To enhance the strength of SR-cement at early ages, a chloride-free hardening accelerator type SikaRapid-1 (Table II) was added to the cement mix. Its performance meets the requirements of [16].

TABLE II. TECHNICAL DATA OF THE HARDENING ACCELERATOR

Property	Quantities and values
pH	8.3
Chloride ion	Nil
Conventional dry material content	35%
Density	1.17kg/l at +23°C
Viscosity	10mPa.s at +23°C

The water/cement ratio is designed to be 0.45 as per ACI 225R recommendation [25], which states that this ratio should be between 0.45 and 0.50 where there is a potential for sulfate attack. To evaluate the effect of the hardening accelerator, 4 series of SR-cement mortar mixes were prepared. The hardening accelerator was not added to the mix of the control series (S0.0). For the other series, i.e. S0.5, S1.0 and S1.5, the accelerator was added with percentages (of cement mass) equal to 0.5, 1, and 1.5%, respectively. It is worth mentioning that these percentages were chosen to be within the range mentioned in the data sheet of SikaRapid-1. The water/cement ratio varied from one series to another to achieve the same consistency as the control series. Primarily, this study was conducted to improve the performance of SR-cement at early ages by using a hardening accelerator. To avoid the effect of other factors and to achieve reliable results, standard sand conforming to BS EN 196-1 [26] was used to the mortar of all the series. This sand is natural, siliceous, and consists of rounded particles whose size distribution is presented in Table III. The cement: sand ratio was set at 1:3 for all the mixes.

TABLE III. PARTICLE SIZE DISTRIBUTION OF THE STANDARD SAND

Square mesh size (mm)	2	1.6	1	0.5	0.16	0.08
Cumulative sieve residue (%)	0	7	33	67	87	100

When an admixture is to be added, it is recommended that the prepared mix should have the same aggregate:cement ratio with the control mix, while the water percentage should be changed to maintain the same consistency [27]. Therefore, the water/cement ratio of the control mix (0.45) was manipulated by relying on the flow table test, which was performed according to BS EN 1015-3 [28]. Firstly, the test was carried out for the control series where the mean of 4 measured diameters of mortar was 128mm. Subsequently, for the other series, water was adjusted to achieve the same diameter of control mix (Figure 1).

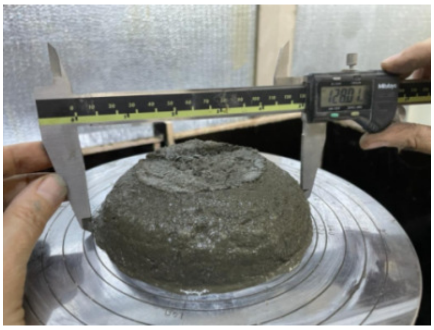

Fig. 1. Flow table test.

The constituents of mortar of all the mixes are shown in Table IV. The method proposed by [27] was adopted in mixing the materials of the 4 series.

TABLE IV. PROPERTIES AND QUANTITIES OF MORTAR COMPOSITIONS

Mortar compositions and properties	Quantity (kg/m^3)			
Cement: CEM I-SR 3	586			
Standard sand 0.8/2	1758			
Series symbol *	S0.0	S0.5	S1.0	S1.5
Water	263.70	261.75	256.54	251.33
Hardening accelerator	0.0	2.93	5.86	8.79

* For each symbol, the letter (S) refers to the word (series) while the number next to it indicates the percentage of the added hardening accelerator (%).

B. Specimen Preparation and Curing Conditions

The hardening accelerator is defined as an admixture that increases the rate of early strength development [16]. Therefore, flexural and compressive strength tests were performed to evaluate the accelerator effects. To perform the flexural and the compressive strength tests, according to [26], 3 specimens with dimensions of 40×40×160mm were prepared from each batch of mortar. The fresh mortar was cast into the prismatic molds in 2 layers and each layer was compacted by using the jolting apparatus (60 jolts per layer), (Figure 2).

(a) (b)

Fig. 2. A prisms-mold with 3 holes (of 40×40×160mm) installed on the jolting apparatus, before cooling the mortar and at the end of compacting (2 layers each 60 jolts). (a) Top view, (b) side view.

To assess the durability of the hardened cement mortar in terms of restricted members and to verify the possibility of cracking that may occur, the drying shrinkage test was carried out. The test was conducted according to ASTM C 596 [29]. Four prisms were prepared from each batch of mortar. The molds that are used for this test have internal dimensions of 25×25×285mm, where 2 stainless steel studs (gauge studs) are inserted at their ends to be embedded into the casted mortar. The distance between the inner ends of the gauge studs which is called gauge length, is 250mm [30] (Figure 3). The mortar was placed in two layers and each layer was compacted with a tamper [31].

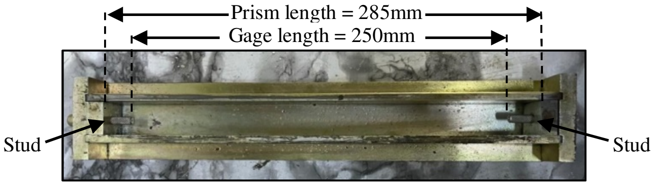

Fig. 3. Shrinkage mold of 25×25×285mm with steel studs inserted at the ends.

For all the mentioned tests, after casting the mortars, each mold was covered with a plate of glass to prevent water evaporation. Then, the molds were placed in a moist air cabinet where the temperature was 23°C and the Relative Humidity (RH) was 95%. After 24h, the specimens of the flexural strength test were taken out of the molds and then placed in a water tank until the age of testing. It should be noted that BS EN 196-1 [26] recommends performing the compressive test on the halves of the prisms broken by the flexural test. For shrinkage molds, ASTM C 596 [29] states that when the strength of the mortar is inadequate to allow proper removal from the molds at 24h, the moist curing in the molds is continued for another 24h. Since sulfate-resisting cement has low strength at an early age, the specimens were kept in molds (in a humid air cabinet) for up to 48h. After they were removed from the molds, the specimens were stored in water for another 24h [29]. At the age of 72h, the specimens were transferred to be stored under laboratory conditions at 23°C and 52% RH, until the end of the test.

C. *Experimental Procedure of Flexural Strength Test*

This test is used to evaluate indirectly the tensile strength. The flexural strength test was performed using the 3-point loading method (Figure 4) according to [26]. The load was applied at a rate of 50N/s until the fracture of the hardened mortar prism. As stated in [26], the two halves of a broken prism were covered with a damp cloth and were kept for the compression strength test. To evaluate the effect of the hardening accelerator on the flexural strength at early ages and to ensure that it does not have detrimental effects at late ages, the test was carried out at different ages of SR-cement mortar (1, 3, 7, 14, and 28 days). For each of the 4 series, 5 mixes were prepared (1 per age) where 3 prisms were cast from each, i.e. 15 prisms were cast for each series with a total of 60 prisms being used in flexural strength tests. The flexural strength R_f was calculated according to:

$$R_f = \frac{1.5 \times F_f \times l}{b^3} \quad (1)$$

where F_f is the maximum applied load to the middle of a prism, b is the square section side of a prism, and l is the distance between the supports.

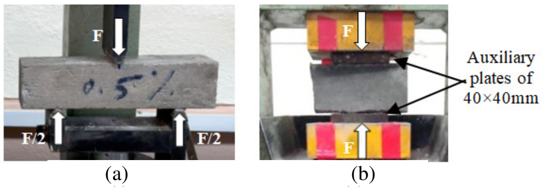

Fig. 4. Mechanical strength test: (a) Flexural strength test, (b) compressive strength test executed on the halves of a broken prism from the flexural test.

D. *Experimental Procedure of the Compressive Strength Test*

The compressive strength test was conducted according to [26]. Depending on this standard, the test was carried out on the two halves of a prism broken in the flexural test. Thus, as the flexural test, the compressive test was done at the mortar ages of 1, 3, 7, 14, and 28 days, for each of the 4 series. Two auxiliary plates of 40×40mm were placed over and under a prism half during the loading time to make the applying load be projected on a length of 40mm (and an area of 40×40mm). The prism half was laterally centered to the platens and longitudinally in such a way that the end face of the prism overhanged the auxiliary plates. The load was applied with a rate of 2500N/s (Figure 4). For each series, since the flexural strength test was performed on 3 prisms per age, the compressive strength test was performed on 6 prism halves at each of the 5 testing ages (1, 3, 7, 14, and 28 days). Therefore, for the 4 series, the compressive strength test was carried out on 120 prism halves.

E. *Experimental Procedure of Drying Shrinkage Test*

Drying shrinkage is a term used to describe the contraction in the length of a test specimen. The contraction is caused by any factor except externally applied forces under stated conditions of temperature and RH [29]. The drying shrinkage test was performed on 4 prisms (of 25×25×285mm) for each of the 4 series (S0.0, S0.5, S1.0, and S1.5) [29]. Thus, in total, 16 prisms were prepared for the test. The accuracy of the dial gauge used in this test was 0.001mm. It was reset to zero by a reference bar each time just before taking the reading of a test specimen (Figure 5). According to [29], at the age of 3 days (2 days in the moist air cabinet, plus 1 day in water), the specimens were removed from the water, swept with a wet cloth, and then the initial reading was taken and recorded. Subsequently, the specimens were placed in the air under laboratory conditions (temperature of 23°C and 55% RH) until the end of the test. Readings for each prism were taken after 4, 11, 18, and 25 days of air storage, according to [29]. For greater accuracy and certitude, additional readings were taken at 7, 14, 21, and 28 days (of air storage). At each testing time, the drying shrinkage (of a series) was computed as the average strain of the 4 test prisms. The drying shrinkage strain at any time of air storage is computed as:

$$\varepsilon_x = \frac{(L_x - L_i)}{L_o} \quad (2)$$

where ε_x is the shrinkage strain of a specimen at time x, l_x is the reading of the specimen (stored under air conditions) at time x, Li is the initial reading of a specimen, and L_o is the nominal gauge length, i.e. the distance between studs (250mm, see Figure 3).

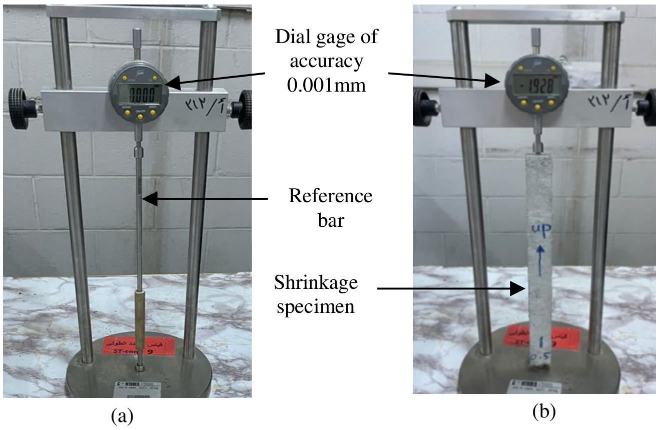

Fig. 5. Length change measuring instrument. (a) Adjusting the dial gauge to zero before taking a reading, (b) taking the reading of a specimen.

III. RESULTS AND DISCUSSION

A. Flexural Strength Results

The experimental results of the flexural strength tests are shown in Figure 6. Each curve represents a series where each point represents the average strength of 3 prisms tested at a given age. It could be seen that there is not a big difference between the flexural strength from one series to another. However, the series containing accelerator (S0.5, S1.0, and S1.5) have slightly higher flexural strengths at the very early ages (1 and 3 days). Inversely, at late ages, the strengths of the control series become the highest. This result corresponds to the results (performed on cement type I) of [32-34]. These studies agree that the hardening accelerator increases the flexural strength of the cement matrix at early ages while at the late age of 28 days, the flexural strength becomes lower than (or about identical to that) of the control mix. It seems that, at late ages, the mix containing more accelerator becomes more brittle, thus exhibiting lower flexural strength. In [14, 35, 36], it was found that the chloride-free hardening accelerator primarily speeds up the hydration of the tricalcium aluminate where ettringite forms. Hence, for the present study (where a chloride-free hardening accelerator is used), at early ages, the ettringite fills out the capillary pores and thus increases the strength of the cement matrix. Therefore, the mix that includes the hardening accelerator will have less pores and higher strength. With time and in the presence of water, the hydration reactions of the other cement phases proceed to form the main cement product of Calcium Silicate Hydrate (C-S-H). This product begins to fill the capillary pores that have not been filled by ettringite. C-S-H is the densest component of the hydrated cement paste [1]. In contrast, ettringite is an expansive compound, and its formation increases the porosity of the cement matrix as a whole [1, 38]. Porous materials are generally considered as brittle materials and have low tensile and flexural strength [38, 39]. Accordingly, at late ages, the mix that does not contain or contains a smaller amount of the accelerator will be denser, less brittle, and will have higher flexural strength than the other mixes.

The relative flexural strength (strength of a hardened mortar containing the accelerator to the reference one at a given age) was calculated and is presented in Figure 7 to clarify this result in another way. It is clear that S0.5 has the highest relative flexural strength, i.e. the strength of the mix with the lowest percentage of the accelerator (0.5%) is higher than the other two mixes with accelerator for all the cement mortar ages.

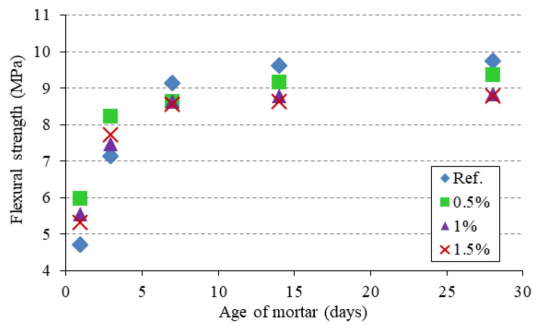

Fig. 6. Flexural strength development with age for the four series.

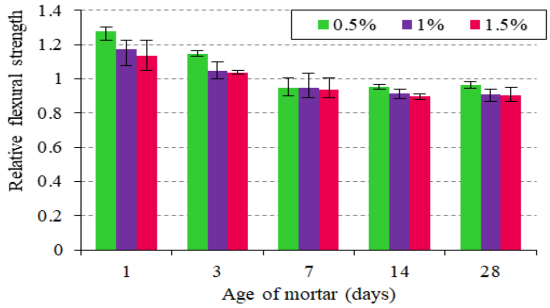

Fig. 7. Relative flexural strengths of the series with accelerator vs. mortar age.

B. Compressive Strength Results

The results of the compressive strength tests are shown in Figure 8. Each point in a separate series represents the average compressive strength of 6 halves (of the 3 broken prisms at the flexural test). It could be seen that the control series has lower compressive strength than the other series for all mortar ages. This result indicates that the hardening accelerator has a beneficial effect on the development of compressive strength at early as well as at late ages (until 28 days) of the mortar. This result agrees with the findings in [32, 33], where the hardening accelerator increases the compressive strength at early ages and maintains it higher than the mix without accelerator even at the late age of at 28 days. The results of [34] also confirm that the hardening accelerator increases the compressive strength of the cement matrix at the early ages, while at the late age of 28 days, the strength of the mix with the accelerator becomes the lowest. The hardening accelerator increases the reaction rate of the cement components, especially C_3A (to form ettringite), and leads to the early completion of this cementation phase. Since ettringite is an expansive material, it fills the capillary pores, increasing the strength of the cement matrix at early ages [1]. Due to the low amount of C_3A in the SR-cement, and with the presence of the accelerator, this compound appears to be consumed early. Therefore, it is not expected to continue producing ettringite which has a detrimental effect on the cement matrix when it is formed at a later age, i.e. when the cement has become sufficiently hard. Thus, the mix with accelerator has higher compressive strength than the control one, even at late ages. To evaluate the best percentage of the accelerator, the relative strength was computed, i.e. the compressive strength of a series to that of the control series (Figure 9). From this Figure, it is clear that the different percentages of the accelerator (0.5, 1, and 1.5%) have about the same effect on the strength of a given age of mortar, even though it seems that S1.0 has the best performance since its relative strengths are higher than those of S0.5 and S1.5 for almost all the mortar ages. That means 1% is the best percentage of the accelerator to be added in order to enhance compressive strength.

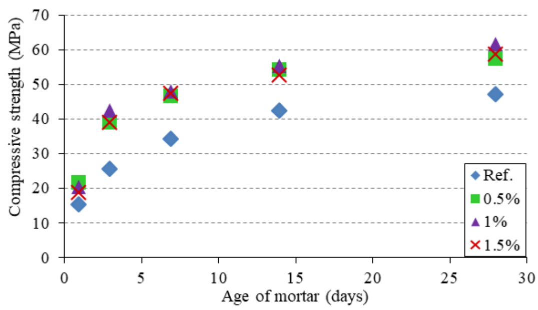

Fig. 8. Compressive strength development with age of the four series.

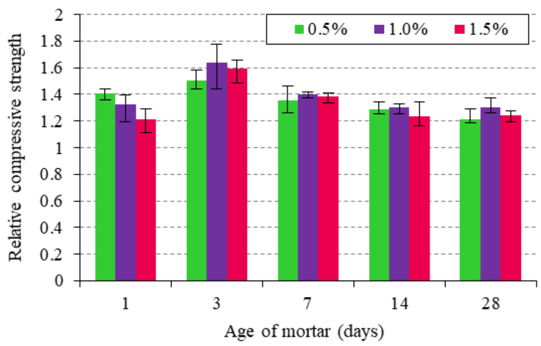

Fig. 9. Relative compressive strengths of the series with accelerator vs. mortar age.

C. *Drying Shrinkage Results*

The readings (4 prisms per series) were taken during the air storage period of 4, 7, 11, 14, 18, 21, 25, and 28 days. The average shrinkage strain of the 4 prisms was computed at each test time (Figure 10). As shown in Figure 10, for about all the test times, the drying shrinkage strain of the control series is higher than those of the other series. Also, as the percentage of the hardening accelerator is increased, the shrinkage strain decreases. That could be related to the fact that the drying shrinkage decreases with increasing strength of the cement paste [40]. Since the strength of SR-cement mortar has been enhanced due to the hardening accelerator (Figure 8), the specimens that include the accelerator exhibit lower shrinkage than the control ones. However, in [41], it was found that the drying shrinkage strain increases with increasing percentage of accelerator. To clarify the extent of the effect of the accelerator, the relative shrinkage strain (the strain of a mortar with the accelerator to the reference one at a specific age) was calculated (Figure 11). At the end of the shrinkage test (after 28 days of air storage), the strains of S0.5, S1.0, and S1.5, respectively, became 0.7, 0.62, and 0.55 of that of the control series.

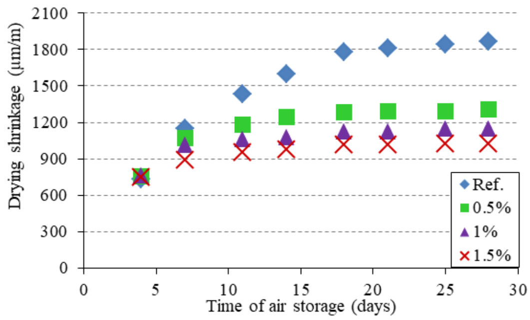

Fig. 10. Drying shrinkage strain vs. time of air storage.

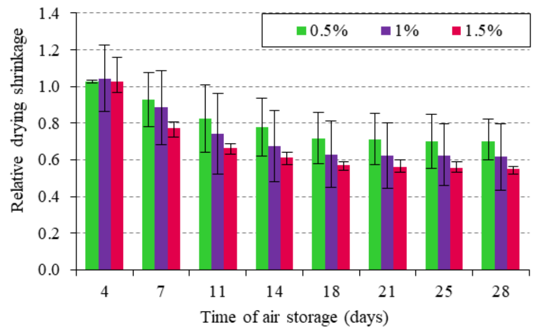

Fig. 11. Relative drying shrinkage of mixes with accelerator vs. time of air storage.

IV. CONCLUSIONS

Since the sulfate-resisting cement has a slow rate of early strength development, this study aimed to increase the strength at early ages by adding a hardening accelerator. To evaluate the effects of the accelerator, four series of cement mortar mixes were implemented (using standard sand). The accelerator was not added to the mix of the control series, while for the other three mixes, it was added with different percentages of 0.5, 1, and 1.5%. Flexural and compressive strength tests were carried out at different mortar ages (from 1 day to 28 days). Drying shrinkage test was also performed to check the cracking potential. From the obtained results, the following points could be drawn:

- For the flexural strength, the results were approximately close. However, at the early ages of cement mortar, the strengths of the series with the hardening accelerator are somewhat higher than that of the control. Inversely, at late ages, the flexural strength of the control series becomes the highest. Comparing the series containing the accelerator, it was observed that the mix with 0.5% accelerator has the best performance since its relative strength is higher than those of the other series for all mortar ages.

- The compressive strength of the control series is lower than those of the series with the hardening accelerator for all mortar ages. Although the relative compressive strength decreases with age, it has never been less than one, i.e. the compressive strength of the series with the accelerator never becomes less than that of the control. This indicates that the hardening accelerator enhances the compressive strength at the early age without damaging effects at later ages. It was also found that, for all mortar ages, the highest compressive strength is obtained by adding 1% accelerator.
- The hardening accelerator strongly decreases the drying shrinkage strain. As the percentage of the hardening accelerator increases, the drying shrinkage values decrease. After 28 days of air storage, the strain of S1.5 became about half (0.55) of that of the control series.

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