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Effect of SRA on the expansive behaviour of mortars based on sulphoaluminate agent

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ABSTRACT

The effectiveness of an expansive admixture based on calcium sulphoaluminate, in the presence and in the absence of a shrinkage reducing admixture, is investigated in mortars manufactured with different water to binder ratios. The results show that the mortar free expansion increases with gypsum content, but the amount of mortar free contraction and the time to reach the mortar steady state remain unaffected. Moreover, the mortar expansion strongly depends on the adopted water to binder ratio; in particular the greatest expansion is reached when the highest water to binder ratio is used. A threshold water/binder value exists below which mortars do not expand. Addition of a shrinkage reducing admixture strongly amplifies the free expansion of mortars but not the mortar expansion loss that follows. On the other hand, the shrinkage reducing admixture does not affect restrained mortar expansion but only reduces restrained mortar shrinkage. The presence of the shrinkage reducing admixture slightly delays cement hydration, always decreases the mortar compressive strength and it changes dramatically the morphology of the ettringite fibers.

1. Introduction

It is well known that in cementitious materials the low tensile strength and the cement paste drying shrinkage can cause the formation of cracks [1–4].

Many efforts have been made to overcome this problem and some good results have been attained by using expansive admixtures [5]. In restrained mortars, the expansion can induce compressive stresses which approximately offset tensile stresses caused by drying shrinkage. Expansive agents have been recommended to compensate the drying shrinkage of reinforced concrete structures too. If adequate reinforcements are present, the concrete expansion induces a tensile stress in the reinforcement structure and a compressive stress in concrete both of which are going to decrease by shrinkage. However, when the expansion occurs too early in a deformable plastic concrete, it can not provoke a sufficient compressive stress. On the other hand an expansion occurring too late requires a prolonged wet curing to avoid the appearance of cracks [6,7].

A shrinkage reducing admixture (SRA) is another admixture traditionally used to reduce both autogenous and drying shrinkage of cementitious materials by reducing the surface tension of pore solution. This reduction decreases the capillary tension of menisci in the capillary pores responsible for the cement paste shrinkage [4,8–10].

The best results against shrinkage have been reached by a combined use of SRA and an expansive agent based on CaO [11]. In this case, the expansion occurs even in a relatively low humidity environment and SRA seems to avoid special precautions to maintain wet curing conditions during early ages. The SRA dramatically increases the expansion induced by CaO, and the expansion is maintained independently of the environmental R.H. [6,7,12–14].

Another family of expansive admixtures generally used in cementitious materials is based on calcium sulphoaluminate (CSA) that produces expansive ettringite [15–21]. CSA produces an expansion at later ages than that induced by CaO; processes related to lime hydration occur within 1–2 days, those based on ettringite formation need about 5–6 days to completely develop their potential expansion [6]. Expansion can usefully occur only if it develops mainly after the concrete compressive strength starts to grow and consequently the steel–concrete bond starts to develop [6]. The expansion rate related to lime hydration process agrees very well only with early strength development concretes whereas when concretes with 1-day compressive strengths of 5–10 MPa are used only the ettringite formation process can be a useful expansion [6]. However, since CSA produces an expansion at later ages than that induced by CaO, it requires a prolonged wet curing period that could be a problem for the use of this expansive admixture. The effects of various curing methods on the expansion of CSA based shrinkage-compensating concretes are reported in Ref.

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[22]. The strong dependence of expansive cement performance, expressed in terms of crack reduction, on continuous wet curing for several days together with the danger of possible disruptive later expansion (in the case of deficient curing) are two concerns which have strongly limited the diffusion of CSA based shrinkage-compensating concretes in the manufacturing of reinforced concrete structures.

Since with SRA the CaO expansion is maintained independently of the environmental R.U. [6,7,12–14], in this work, the effect of SRA on the performance of cementitious materials based on CSA has been investigated in order to outline possible benefits by a combined use of these admixtures.

2. Materials and testing

An expansive agent obtained by mixing Aluminate Cement (HAC), calcium sulphate dihydrate (gypsum) and calcium hydroxide (lime) in order to produce expansive ettringite, has been used in this study. A mixture of Portland Cement (CEM I 52.5R according to UNI-EN 197/1) and Aluminate Cement in the same percentage was used as the binder. The excess HAC, than the amount necessary for the formation of ettringite [15], was useful for developing strength at very short curing times. Natural sand with a 5 mm maximum size, density of 2620 kg/m^3 and water absorption of 3.0% by mass was used. A shrinkage reducing admixture (SRA) based on propylene-glycol ether was added at the dosage rate of 1% by binder weight.

Mortar mixtures with two different gypsum dosages (3% and 4% by solid weight), as reported in Table 1, and four water/binder (w/b = 0.65, 0.67, 0.70, 0.75) were manufactured. These w/b ratios are commonly used for self-leveling screeds in residential and industrial sub-flooring where relatively low mechanical performances are required. An acrylic super-plasticizer was added in the mixtures at doses ranging from 0% to 0.8% by weight of binder to obtain the same consistency (slump flow = 135–150 mm as measured by a flow table according to UNI EN 1015-3:2000).

Prismatic mortar specimens (40 × 40 × 160 mm) were manufactured to evaluate the free expansion/shrinkage according to UNI 6687 (Cement mortar. Hydraulic shrinkage determination. Laboratory test) and the relative weight loss. Moreover, prismatic specimens (50 × 50 × 250 mm) reinforced with steel bar were manufactured to evaluate the restrained expansion/shrinkage behaviour according to UNI 8147:1980 (Not metallic expansive agents for cement mixings. Determination of restrained expansion of a mortar containing the expansive agent). All beams were demoulded 7 h after casting and kept at T = 20 ± 1 °C and R.H. = 50% ± 3% during the testing time. Seven hours corresponds approximately to the final setting time of the cement paste; this was also the time at which the measurements were started.

Measurements of compressive strength were carried out on cubic specimens (100 × 100 × 100 mm) de-moulded at 1 day and wet cured at T = 20 ± 1 °C at curing times of 1, 3, 7, 14 and 28 days.

Finally, the effect of SRA on the hydration of shrinkage-compensating cement pastes was investigated by thermo-gravimetric analysis, carried out on cement pastes with water/binder = 0.70 containing gypsum (4% by solid weight) in the absence or in the presence of SRA (1% by binder weight), after 2, 6, 24 and 168 h of hydration, respectively. For each evaluation time, the cement paste hydration was stopped by removing unbound water through the use of acetone and the resulting dried powder kept in a vacuum dryer until being tested. The weight loss due to ettringite and gypsum thermal decomposition (dehydration) was determined between 50–120 °C and 130–165 °C, respectively. Scanning Electron Microscopy (SEM) morphological observations on the same cement pastes were carried out after 6 h of hydration.

Table 1
Solid ingredients dosages (% by solid weight) in mortar mixtures.

Mixture	G3	G4
Portland Cement	10	10
Aluminate Cement	10	10
Gypsum	3	4
Lime	1	1
Limestone	12	11
Sand	64	64

3. Results and discussion

The reported results are averaged among those obtained in three specimens of the same type.

Fig. 1 shows the free expansive–shrinkage behaviour of two mortars with w/b = 0.70 differing in gypsum content (3% and 4% by solid weight). The gypsum percentage determines the amount of expansive agent produced and consequently the obtained expansion value. In both cases drying shrinkage begins immediately after expansion. It should be noted that the amount of mortar contraction (intended as the difference between the maximum value and the final asymptotic value) is the same in both specimens and after 20 days a more or less steady state is achieved.

Mortars with the same gypsum content (3% by solid weight), but manufactured with different w/b (Fig. 2) show very different performances; different expansion value or absolutely no expansion can be observed depending on the w/b adopted. A threshold value of w/b exists, below which no expansion can be detected; as a matter of fact, only a shrinkage is indicated by the curve related to w/b = 0.65. A possible explanation may be the little water available to form ettringite, a compound that needs much more water to grow [17], when a low w/b is adopted. Consequently, mortars show greater expansion when manufactured with higher amounts of water. This threshold value in w/b or w/c ratio has already been observed in mortars without expansive agents [23,24].

When SRA is added in the mixture to preserve the expansion for longer time as when used previously with CaO, a strong enhancement of the first expansion is achieved as already observed in CaO based mortars (Fig. 3a) [6,7,13]. However, the contraction starts at the same very early time and continues at the same rate (Fig. 3a). With SRA, the amount of free contraction is about the same, but a portion of the whole expansion is preserved. The SRA agent affects only the mortar expansion phase enhancing it to values three times higher with respect to those observed in mortars without SRA, but it does not affect the stability of the system. With and without SRA, a steady state is reached after about 20 days of exposure in the drying environment. The weight loss of the specimens is reported in

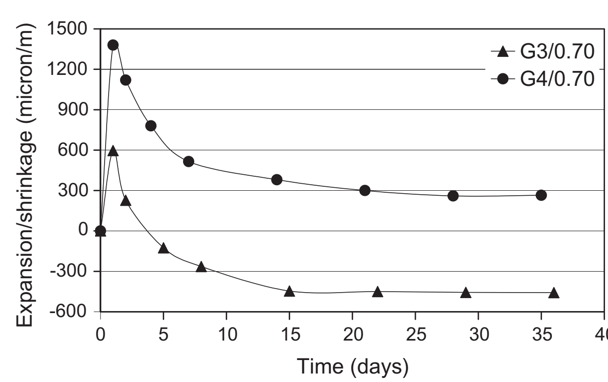

Fig. 1. Free expansion–shrinkage of mortars with the same w/b (0.70) and different gypsum content (3–4% by solid weight).

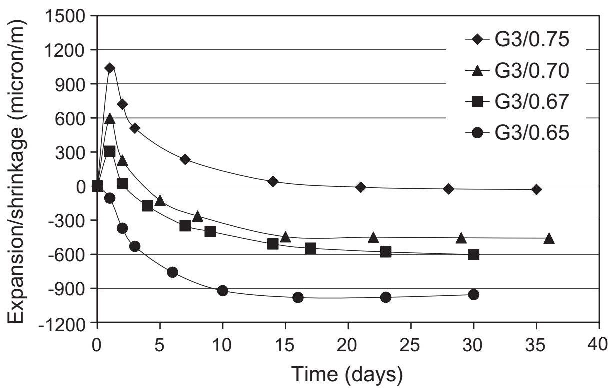

Fig. 2. Free expansion–shrinkage of mortars with the same gypsum content (3% by solid weight) and different *w*/*b* (0.65–0.67–0.70–0.75).

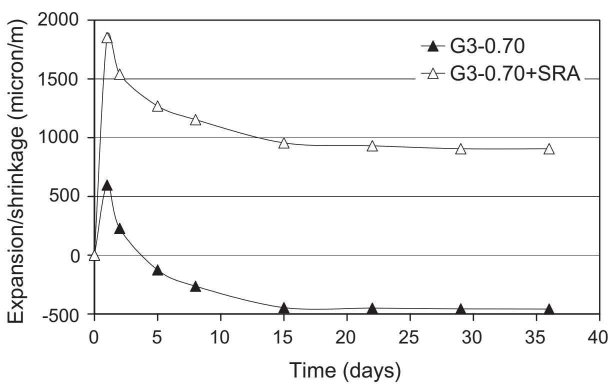

Fig. 3a. Free expansion–shrinkage of two mortars with the same composition (gypsum = 3% by solid weight, *w*/*b* = 0.70) with or without SRA.

Fig. 3b. It is evident that SRA does not reduce the water evaporation from mortars confirming the results already reported in Ref. [6], even if this depends somewhat on *w*/*c* ratio and drying R.H. [25].

The investigation of restrained expansion–shrinkage of mortars is reported in Figs. 4a–4c. They show that the differences observed in the free expansion–shrinkage of different mortars are reduced and, in particular, very similar trends are exhibited by mortars with or without SRA due also to the degree of restraint and the modulus of the system. When considerable restraint is provided, the system will not be allowed to expand. The SRA agent hardly modifies the performance during the expansion phase (Figs. 4a and 4b) but the contraction seems to decrease slightly if SRA is present even if the time to reach the steady state remains the same. When a low *w*/*b* (0.67) is adopted (Fig. 4c) no expansion is detected regardless of the presence of the SRA.

A moderate delay in the kinetics hydration in the presence of the SRA admixture is highlighted by TG analysis at 2 h (Fig. 5), since the amount of gypsum and ettringite is respectively lower and higher in cement pastes with SRA in comparison to that observed in cement pastes without SRA. However, this slight delay is recovered at 6 h. At 24 h, in the presence of SRA, the ettringite and gypsum content become even slightly higher and lower respectively. At 7 days gypsum disappears while the ettringite content remains slightly higher in the presence of SRA.

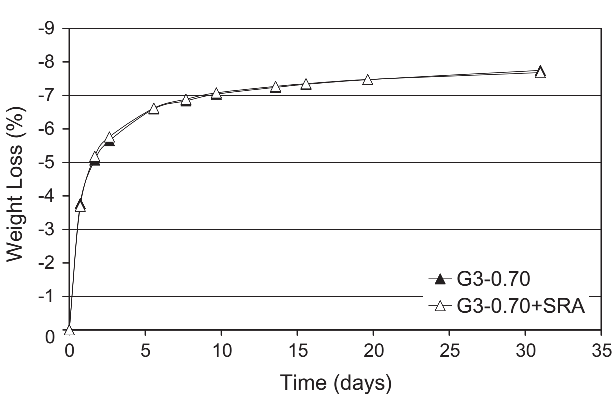

Fig. 3b. Weight loss of two mortars with the same composition (gypsum = 3% by solid weight, *w*/*b* = 0.70) with or without SRA during free expansion/shrinkage.

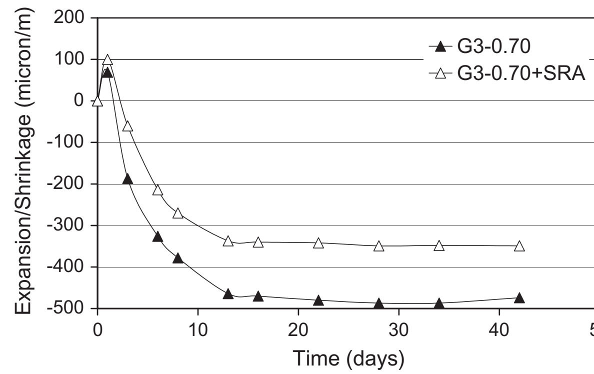

Fig. 4a. Restrained shrinkage of two mortars with the same composition (gypsum = 3% by solid weight, *w*/*b* = 0.70) with or without SRA.

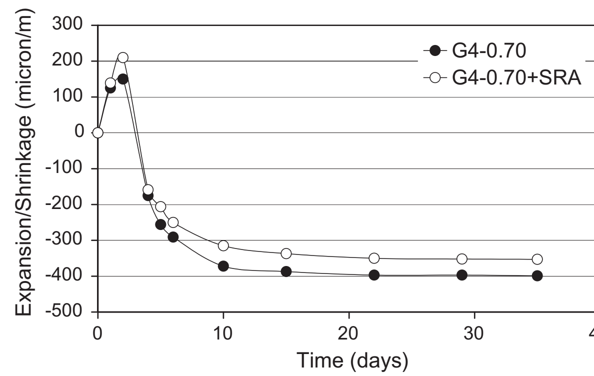

Fig. 4b. Restrained shrinkage of two mortars with the same composition (gypsum = 4% by solid weight, *w*/*b* = 0.70) with or without SRA.

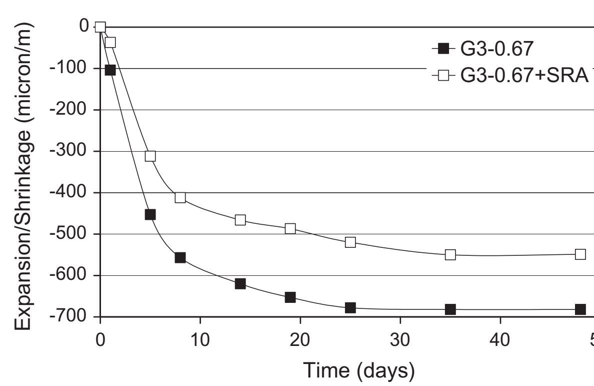

Fig. 4c. Restrained shrinkage of two mortars with the same composition (gypsum = 3% by solid weight, *w*/*b* = 0.67) with or without SRA.

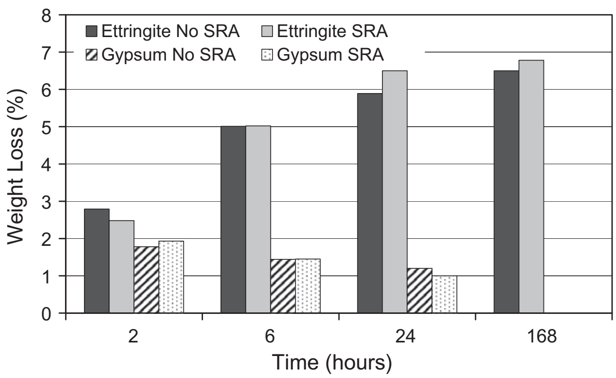

Fig. 5. Weight loss due to ettringite and gypsum thermal decomposition in cement pastes (gypsum = 3% by solid weight, w/b = 0.70) with and without SRA.

Significant information is revealed by SEM observations. When SRA is added to the mixture (Fig. 6b) the ettringite morphology changes into finer fibers compared to those observed in the absence of SRA (Fig. 6a). This fact could be a reason contributing to the higher expansion observed in mortars manufactured with SRA.

Finally, compressive strength tests carried out on mortars with the same w/b, but with different gypsum dosages (3–4% by solid weight), show that a higher gypsum content causes lower strength values (Fig. 7). This can be ascribed to the Aluminate Cement reduction since it is involved in expansive product formation, and hence not available for strength development. Moreover, strength values registered on mortars with the same composition, but with or without SRA (Figs. 8a and 8b), show some penalizing effect correlated with the presence of SRA. At the first curing time, the SRA reduces strength by about 30–40% and at later ages by about 10–15%. Since the compressive strength decreases when the expansion rate increases at each curing time, it is possible that, besides a slight retarding effect and a change of ettringite morphology, with SRA a loosening of the mortar structure due to the excessive expansion exists and is responsible for the lowering of strength [5].

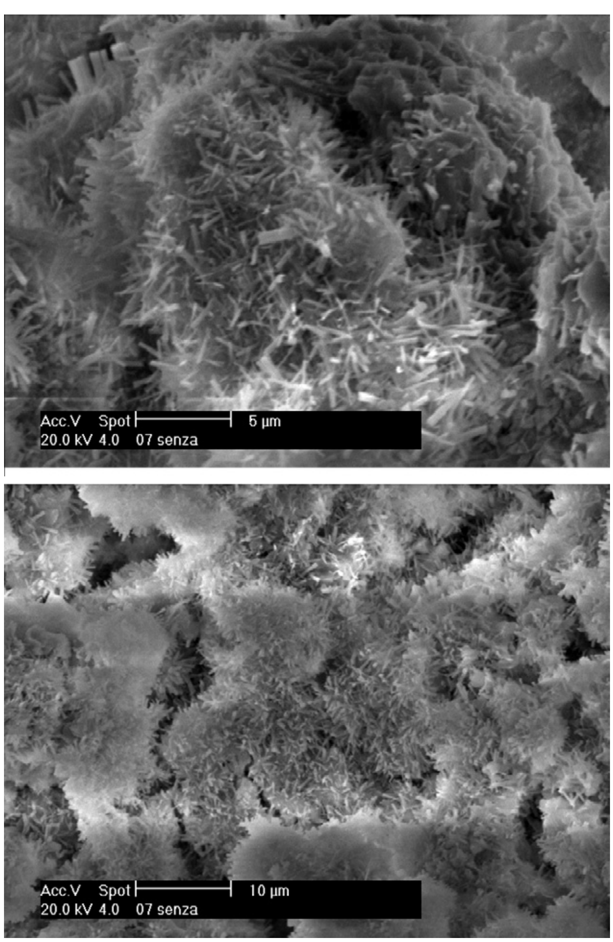

Fig. 6a. SEM micrographs of paste sample (gypsum = 3% by solid weight, w/b = 0.70) without SRA at t = 6 h.

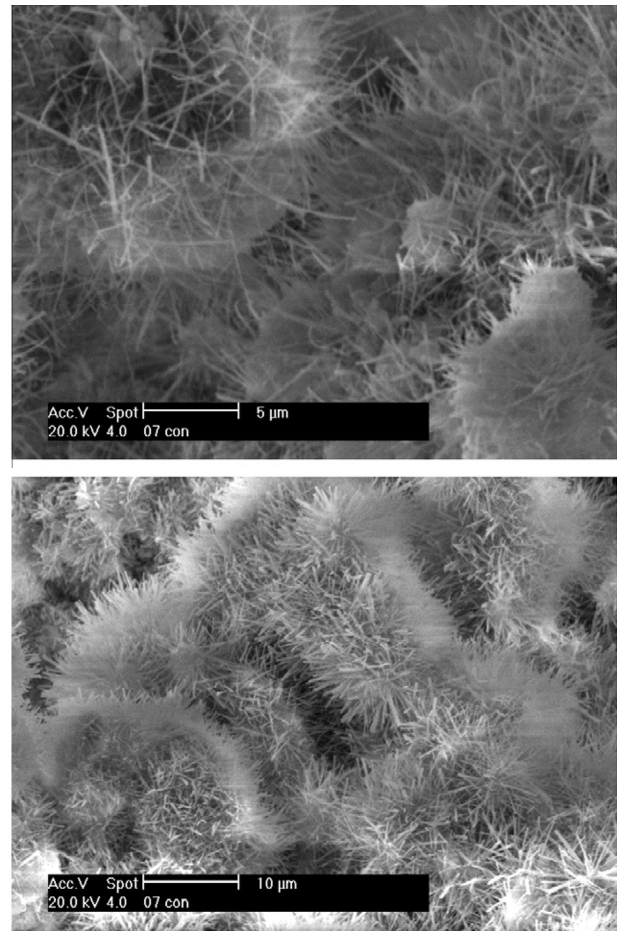

Fig. 6b. SEM micrograph of paste sample (gypsum = 3% by solid weight, w/b = 0.70) with SRA at t = 6 h.

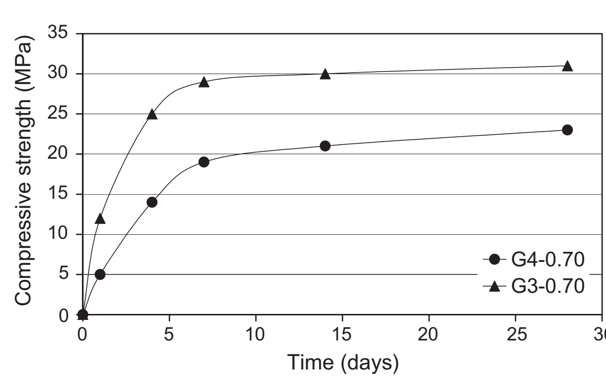

Fig. 7. Compressive strength of mortars with the same w/b (0.70) and different gypsum content (3–4% by solid weight).

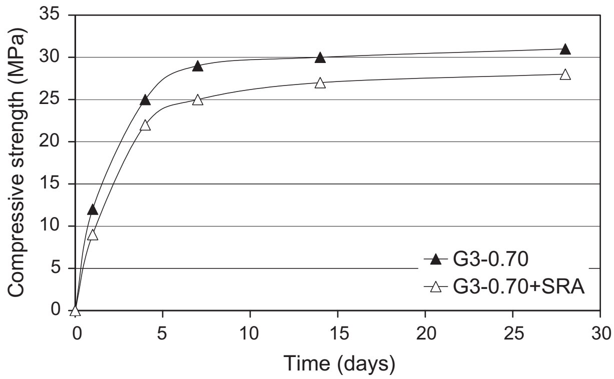

Fig. 8a. Compressive strength of mortars with the same composition (w/b = 0.70, gypsum content = 3%), with or without SRA.

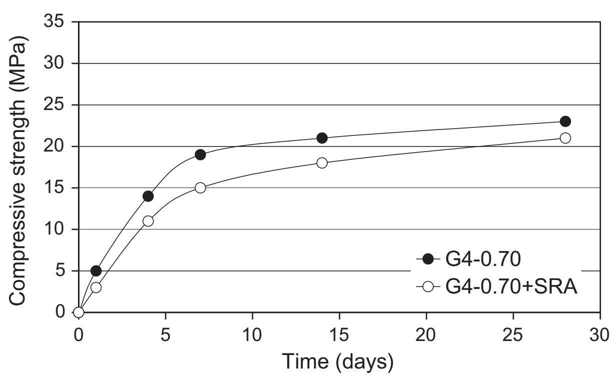

Fig. 8b. Compressive strength of mortars with the same composition (w/b = 0.70, gypsum content = 4%), with or without SRA.

4. Conclusions

The salient conclusions from this study on the effectiveness of an expansive admixture based on calcium sulphoaluminate (CSA) in the presence and in the absence of SRA in mortars are:

- The mortar free expansion increases with the gypsum content and with the water/binder used.
- The mortar freely expands when the water/binder is high enough; a threshold water/binder value exists below which only shrinkage is observed.
- The SRA admixture strongly amplifies the free mortar expansion but not the amount of free contraction nor the time to reach the steady state.
- In the presence of restraint, the SRA admixture does not affect the mortar expansion but it slightly reduces the amount of restrained shrinkage.
- The SRA slightly delays ettringite formation only at early age but it changes significantly the morphology of the ettringite fibers which result in a finer structure in the presence of SRA.
- SRA always reduces the compressive strength of mortars particularly at early ages.

Therefore from the obtained results, we can conclude that SRA does not really help much in the restrained scenario but it could be a useful addition to CSA based shrinkage-compensating cementitious materials, such as self-leveling screeds for industrial or residential sub-flooring, where reinforcements are absent.

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