USE OF TARTARIC ACID FOR THE PRODUCTION OF SUSTAINABLE PORTLAND-FREE CSA-BASED MORTARS

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ABSTRACT

The purpose of the study is to evaluate the effect of a tartaric acid-based set retarding admixture on rheological, elastic and physical performances of sustainable mortars manufactured with calcium sulfoaluminate, anhydrite and Supplementary Cementitious Materials replacing totally OPC. Experimental results indicated that the tartaric acid acts as superplasticizer and it is effective to extend the pot-life of mortars up to about 2 hours. On the other hand, the set-retarding admixture provides a strong retardation of binder hydration resulting in a reduction of initial expansion and compressive strength at early ages. However, this retarding effect disappears at long ages.

KEYWORDS
Calcium sulfoaluminate cement; supplementary cementitious materials; tartaric acid; set-retarding admixtures; sustainability

HIGHLIGHTS

• Sustainable OPC-free mortars with CSA and SCMs were investigated
• Tartaric acid acts as superplasticizer and set-retarding admixture
• Shrinkage and mechanical properties are influenced by tartaric acid addition
• About 60% reduction in Gross Energy Requirement and CO₂ emissions was achieved
The topics of sustainability and environmental protection in construction have become very important when Kyoto protocol was adopted in 1997 and, particularly, after the Paris Agreement (COP21, 2015) which was aimed to limit the temperature increase even further to 1.5°C above pre-industrial levels.

In this context, a sharp reduction of CO$_2$ emissions and in consumption of natural resources in production of construction materials is needed.

In 2016, about 4.2 billion tons of Portland-based hydraulic cement were used globally – about 2.4 billion tons in China – (1), and CO$_2$ emissions from the cement industry exceeded 7% of global anthropogenic greenhouse gases emission (2). Concrete industry is not eco-friendly since it consumes great amount of cement, aggregates and water.

In order to reduce the environmental impact of concrete industry, several authors (3,4) have identified different strategies: a) using alternative fuels and raw materials to reduce greenhouse gases emissions to produce Portland cement (5–7); b) replacing Portland cement clinker with low-carbon supplementary cementitious materials (SCM) (8–11); developing alternative low-carbon binders, such as alkali-activated materials, geopolymers and calcium sulfoaluminate cements (12,13); c) reducing natural resource consumption through to waste management and waste recovery (14–21); increasing durability of concrete structures by means of high-performance admixtures (22,23).

Between these strategies, calcium sulfoaluminate cements (CSA) are actually receiving increasing attention because they promise to provide a low-CO$_2$ and low-embodied energy alternative to Portland cement. In fact, compared to alite, which releases 0.578 CO$_2$ g/g, emissions of greenhouse gases, calcium sulfoaluminate clinker contributes only for 0.216 CO$_2$ g/g. In addition, the maximum kiln temperature to produce CSA clinker is typically 1250 °C, about 200°C lower than
that used for Portland cement clinker. Finally, the grinding process requires lower energy due to the lower hardness of CSA clinker compared to Portland clinker (24–26).

In general, about 15-25 wt. % of calcium sulfate, in form of gypsum (G) or anhydrite (C₅S), is blended with CSA-clinker to control setting time, strength development and volume stability. Several authors (27–29) have shown a close correlation between the main properties of CSA-based composites and calcium sulfate dosage. Nowadays, the use of calcium sulfoaluminate cement is widespread for calcium sulfoaluminate – ordinary Portland cement – calcium sulfates ternary binders in which OPC is present up to 60% with respect to binder mass (30,31).

One of the main issues related to the use of CSA for the production of concrete is the setting time; indeed, it is well known that mixtures containing calcium sulfoaluminate cement are characterized by a short pot-life and a pronounced workability loss over time (32–34). In order to face this practical problem, several set-retarding admixtures were proposed by different authors. Sugama proposed the use of citric acid as a set retarder (35). Velazco et al. (36) showed that the addition of citric acid increased the setting time, modified the morphology of the ettringite needles, changed the microstructural configuration and prevented the decreasing in compressive strength caused by delayed ettringite formation. Moreover, Bishop et al. synthetized a novel retarding admixture based on calcium nitritoltris(methylene)-triphosphonate (37) and investigated mechanisms responsible for set and hardening retardation promoted by sucrose, tartaric acid and lignosulfonate in CSA-based mortars (38). Results indicated that tartaric acid is the most effective in retarding C₃A hydration and ettringite formation.

The purpose of the present study is to evaluate the effect of addition of a tartaric acid-based set retarding admixture on rheological, elastic and physical performances of sustainable mortars manufactured with CSA, anhydrite and supplementary cementitious materials (fly ash, metakaolin and ground granulated blast furnace slag) replacing totally OPC.
MATERIALS AND METHODS

Materials

A ternary binder based on ordinary Portland cement CEM I 52.5R (OPC: according to EN 197-1), commercial CSA clinker and technical grade anhydrite (CŚ) was used to manufacture the reference mortar (CSA:OPC:CŚ = 40:40:20). Ground granulated blast furnace slag (S: according to EN 15167-1), metakaolin (MK: according to ASTM C618), V class fly ash (FA: according to EN 450-1 and EN 197-1) and hydrated lime CL90-S class (CH: according to EN 459-1) were employed to replace totally OPC in sustainable mortars (CSA:SCM:CH:CŚ = 40:35:5:20). CH was added to the mix in order to improve the pozzolanic reaction of SCM amount not consumed in the reaction with CSA; furthermore, sand/binder ratio was fixed equal to 3 (maximum diameter of natural siliceous aggregates equal to 2.5 mm). Tartaric acid-based set-retarding admixture was added up to 1.2% with respect to binder mass, while the mixing water was adjusted in order to attain, in absence of set retarding admixture, the same workability at the end of the mixing procedure, equal to 160 mm ± 10 mm spreading.

Table 1 – Physical properties of binders

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Table 2 – Composition of mortars

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<th>CH</th>
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Tests on mortars

At the end of the mixing procedure, workability was measured by means of flow table according to EN 1015-3. In addition, specific mass was evaluated on fresh mortars according to EN 1015-6 standard. Moreover, the pot-life of the mixture, which corresponds to time during which workability by flow table is higher than 140 mm, was also detected. Specimens 40x40x160 mm³ were produced and cured under water at 20°C; in addition, only for mixture containing 0.8% of tartaric acid, specimens were cured both under water at 20°C and in a climatic chamber at 20°C and R.H. 60%. Specific mass, compressive and flexural strength at 1, 7 and 28 days were also determined (EN 1015-11). Moreover, drying shrinkage was measured over time on specimens stored in dry environment (20°C, R.H. 60%) both in plastic and hardened (according to EN 12617-4) phase. Finally, SEM observation were performed on 28-day specimens cured under water.

RESULTS AND DISCUSSIONS

The amount of water to achieve the target workability (160 mm spreading) is, in the absence of set retarding admixture, variable due to the different specific surface, texture and shape of binders
(Tables 1 and 2). In particular, replacing OPC with GGBFS, no change in terms of mixing water was noticed. On the contrary, the use of FA determines an increase in water demand equal to 7% due to the high unburnt carbon content (L.O.I. = 4.9% according to EN 196-2 and ISO 10694), while employing MK in place of OPC water amount rise up about 18% respect to reference mortars due to the higher fineness of metakaolin compared to Portland cement. Furthermore, tartaric acid-based set-retarding admixture acts as a superplasticizer (Figure 1). The super-plasticizing effect is more pronounced in fly-ash based mortars (+45% spreading with respect to mixture without set-retarding admixture), while is almost the same for mortars manufactured with the other binders employed (+20%).

![Fig. 1 – Workability by flow table of mortars vs set-retarding admixture dosage](image)

Tartaric acid addition does not produce any abnormal air entrapment; in fact, regardless of set-retarding admixture dosage, specific mass of mortars is substantially the same both in fresh and hardened state (Figure 2). On the contrary, the total substitution of OPC with SCMs modifies density of mortars; in fact, FA and MK-based mixtures showed specific mass (both in fresh and hardened state) lower than that detected on reference (containing OPC) and S-based mortars as a consequence of the increase of mixing water to achieve the target workability.
Figure 2 – Specific mass at fresh (continuous line) and hardened (dashed line) state of mortars vs set-retarding admixture dosage

Figure 3 shows that the effectiveness of admixture with different binders is almost the same. Regardless to the binder used, pot-life of mortars without tartaric acid is about 20 minutes, which is not suitable for placing in the job-site; the addition of tartaric acid-based admixture extends the pot life of mortars up to 110 minutes from mixing. The average time between mixing and placing a mortar in the job-site is generally close to 60 minutes. Based on this target, it is possible to conclude that the ideal set-retarding admixture dosage is equal to 0.8% vs. binder mass (Figure 3).

Figure 3 – Pot-life of mortars vs set-retarding admixture dosage
Results (Table 3) indicate that addition of tartaric acid-based admixture determines a strong retardation of binder hydration and, consequently, a general reduction of 24-hour compressive strength of both reference and SCMs mortars, except for MK-mixtures. The higher the tartaric acid dosage, the stronger the decrease of compressive strength (Figure 4).

### Table 3 – Mechanical properties of hardened mortars wet cured

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<tr>
<th>Composition</th>
<th>w/b ratio</th>
<th>$R_c$ at 24 h [MPa]</th>
<th>$R_c$ at 7 days [MPa]</th>
<th>$R_c$ at 28 days [MPa]</th>
<th>$R_c$ at 24 h [MPa]</th>
<th>$R_c$ at 7 days [MPa]</th>
<th>$R_c$ at 28 days [MPa]</th>
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<td>7.52</td>
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<td>7.03</td>
<td>13.03</td>
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Since the retarding effect of tartaric acid is more pronounced on OPC (39), the decrease of compressive strength in reference mortars, when the dosage of the set-retarding admixture increases, was significantly higher compared to slag and fly ash based mortars. Another effect responsible for the lower reduction of compressive strength of SCMs-based mortars should be attributed to a partial adsorption of tartaric acid by S, FA and MK particles. The lower amount of tartaric acid in the aqueous phase could justify the lower retardation of these mortars.

This retarding effect totally disappears at 28 days for all mixtures (Figure 5). In general, the total replacement of OPC with SCMs causes a reduction in compressive strength ranging from 30% (for slag and fly ash) to 60% (for MK-based mortars). The effect of curing conditions was also investigated; specimens cured at 20°C under water evidenced compressive strength values about
25% lower than that measured on mortars stored in dry environment (20°C, R.H. 60%), independently of the age and the SCMs used (Figure 6).

**Fig. 4** – Compressive strength of mortars after 24 hours vs set-retarding admixture dosage

**Fig. 5** – Compressive strength of mortars after 28 days vs set-retarding admixture dosage (specimens under water at \( T = 20°C \))
The images collected by means of scanning electron microscope (SEM) on 28-day specimens showed a different microstructure between mortar containing Portland cement and those manufactured with SCMs (Figure 7 - 8). In particular, SCM-based mixtures are characterized by a homogeneous microstructure with rich amounts of needles covered by small Ca-rich particles of size 0.1-0.5 μm. The prismatic needles had a thickness of about 2-5 μm and a length approximately equal to 5-20 μm. On the contrary, the microstructure of CSA/OPC/CS mortar seems to be composed of denser crystals than those of SCM-based mixtures. In addition, the presence of Ca-rich particles highlighted in Portland-free mortars was not observed.

**Fig. 6** – Development of compressive strength over time with different curing: under water (WET) and on dry environment at R.H. 60% (DRY)

**Fig. 7** – SEM images of reference mortar (on the left) and S-based mortar (on the right)
Shrinkage tests were performed up to 270 days on prismatic 40x40x160 mm specimens stored at 20°C and R.H. 60% (Figure 9). Reference mortar manufactured without tartaric acid shows a marked expansive behavior (up to +800 μm/m at 24 hours from casting) followed by shrinkage (-400 μm/m at 270 days). Total replacement of OPC with SCMs and hydrated lime determines – in absence of set-retarding admixture – a sharp reduction of the initial expansion followed by shrinkage (about -500 μm/m after 270 days), independently of the nature of SCM replacing OPC.

**Fig. 9** – Free shrinkage over time in dry environment (T = 20°C, R.H. 60%)
By using tartaric acid, CSA-based mortars (both that containing OPC and those manufactured with SCMs) did not evidenced the initial expansion, but they began to shrink just after the final setting time has occurred. This could be ascribed to the delay in the development of ettringite when the tartaric acid is used. These mixtures, however, despite the absence of initial expansion, present a quite stable behavior over time, with a free shrinkage lower than -500 μm/m after 270 days.

The role of tartaric acid is also evident on the expansive behavior in the plastic phase (Figure 10); reference mortar (containing OPC) manufacturing without set-retarding admixture achieves an expansion equal to 1’000 μm/m already after 6 hours from casting while the reference mortar manufactured with 0.2% set-retarding admixture is essentially stable over time. In general, use of tartaric acid-based set-retarding admixture, independently of the presence of OPC and the nature of SCM, eliminates the initial expansion typical of reference CSA-OPC-C\(\alpha\) without admixture (Fig. 11). In other words, the "expansive/shrinkage" behavior of the mortar in the absence of tartaric acid is transformed in an "exclusively shrinkage" behavior. However, the final contraction detected for mortars containing set-retarding admixture was substantially the same of that measured in reference mortar (containing OPC) without tartaric acid.
From a practical point of view, mixtures with “exclusively shrinkage” behavior - at the same value of 270-day shrinkage of expansive/shrinkage mortar - seems to be more promising in repair of existing concrete structures since the tensile stress induced by restrained contraction is lower. As a consequence of more stable behavior of SCM mortars manufactured with the tartaric acid, although the sharp decrease detected for 28-days compressive strength when OPC is replaced by SCMs, FA- and S-based mortars showed mechanical performances suitable for mixtures devoted to “cosmetic repair” of existing reinforced concrete structures, where shrinkage is the main design parameter. In particular, CSA-Portland free mortar can be classified as R3 strength class ($R_c \geq 25$ MPa at 28 days) according to EN 1504-3 and, hence, suitable for repair of existing concrete structures that present corrosion of rebars and spalling of concrete cover. Furthermore, the worsening in mechanical strength of SCMs mortars is fully offset by a marked improvement in environmental sustainability. Indeed, at equal strength class, gross energy requirement (GER) and global warming potential (GWP) parameters of Portland-free mortars are lower than those of both high volume fly ash (HVFA) mortars (40), traditional mixtures manufactured with OPC and ternary binders based on...
CSA. In particular, starting from data on CO₂ emissions and energy production of binders (CEM I, CEM II/AL, CSA, CS and CH), supplementary cementitious materials (S, FA and MK) and aggregates (Table 4), the reduction of GER and GWP deriving from the use of limestone Portland cement, HVFA, CSA-OPC- CS ternary mixtures and CSA-SCMs-CH-CS blends in place of Portland cement was determined. It is possible to conclude that, at the same strength class, the use of traditional CSA-based ternary mixture in place of Portland cement or limestone Portland cement determines a reduction in terms of GER and CO₂ emissions ranging from 25% to 35% while the total replacement of OPC with SCMs slumps GER and GWP of about 60% (Figure 12). Finally, GER and GWP of Portland-free CSA-based mixtures reach lower values than those shown by HVFA mortars.

Table 4 – Environmental parameters of binders, SCMs and aggregates. Source: Ecoinvent 3.0 Database

<table>
<thead>
<tr>
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<th>GER [MJ/kg]</th>
<th>GWP [Kg CO₂/Kg]</th>
</tr>
</thead>
<tbody>
<tr>
<td>CEM I 52.5 R</td>
<td>5.50</td>
<td>9.8 \times 10^{-1}</td>
</tr>
<tr>
<td>CEM II/AL 42.5R</td>
<td>3.60</td>
<td>8.8 \times 10^{-1}</td>
</tr>
<tr>
<td>CSA</td>
<td>2.70</td>
<td>7.4 \times 10^{-1}</td>
</tr>
<tr>
<td>CS</td>
<td>1.30</td>
<td>2.4 \times 10^{-1}</td>
</tr>
<tr>
<td>CH</td>
<td>4.50</td>
<td>4.2 \times 10^{-1}</td>
</tr>
<tr>
<td>S</td>
<td>0.31</td>
<td>1.7 \times 10^{-2}</td>
</tr>
<tr>
<td>FA</td>
<td>0.10</td>
<td>5.3 \times 10^{-3}</td>
</tr>
<tr>
<td>MK</td>
<td>1.50</td>
<td>9.2 \times 10^{-2}</td>
</tr>
<tr>
<td>Aggregates</td>
<td>0.13</td>
<td>2.4 \times 10^{-3}</td>
</tr>
</tbody>
</table>
Further experimentations are needed to measure Young modulus and the adhesion properties of these eco-friendly ternary binders. In addition, durability issues of mortars and concretes manufactured with CSA-based Portland-free binders have to be thoroughly investigated, especially in chloride and sulfate-rich environments or in presence of freeze and thaw cycles.

CONCLUSIONS

In this paper, the influence of tartaric acid-based set-retarding admixture was evaluated on the performances of sustainable Portland-free CSA-based ternary mortars manufactured with supplementary cementitious materials. Results indicated that the tartaric acid, independently of the nature of SCMs replacing OPC, acts as superplasticizer and it is effective to extend the pot-life of mortars up to about 2 hours. No side-effects were detected on specific mass in fresh and hardened state by increasing the set-retarding admixture dosage; on the other hand, total substitution of OPC with fly ash or metakaolin cause a decrease in specific mass due to the higher water demand to attain the target workability at the end of the mixing procedure. The use of tartaric acid determines...
a strong retardation of binder hydration and, consequently, a reduction of compressive strength at 
early ages, especially in reference mortars containing OPC. No retarding effect was detected at 28 
days from casting. In general, the total replacement of OPC with supplementary cementitious 
materials and hydrated lime determines a sharp reduction in terms of compressive strength ranging 
from 30% (for mixture containing fly ash and slag) to 60% (for mortars manufactured with 
metakaolin). In addition, as opposed to OPC-based mixtures, mortars containing CSA, SCM, 
hydrated lime and anhydrite show higher compressive strength values when cured in dry 
environment respect to that measured on specimens stored under water.

Mortars without tartaric acid showed an initial expansion during the first 5-7 days as a consequence 
of the ettringite formation, than the mixtures shrink. When set-retarding admixture is used, the free-
expansion totally disappears and shrinkage begins immediately after final set has occurred. 
However, after 270 days shrinkage is substantially the same for mortars with and without tartaric 
acid. In other words, the “expansive/shrinkage” behavior of the mortar in the absence of tartaric acid 
is transformed in an “exclusively shrinkage” behavior. However, the final contraction detected for 
mortars containing set-retarding admixture was substantially the same of that measured in reference 
mortar (containing OPC) without tartaric acid. Although the compressive strength of SCM-based 
mortars is lower than the reference mixtures containing OPC, the more stable behavior evidenced 
by these Portland-free materials makes them suitable for “cosmetic repair” of existing reinforced 
concrete structures, where shrinkage is the main design parameter. In particular, CSA-Portland free 
mortar can be classified as R3 strength class (Rc ≥ 25 MPa at 28 days) according to EN 1504-3 and, 
hence, suitable for repair of existing concrete structures that present corrosion of rebars and spalling 
of concrete cover.

Finally, CSA-SCM-CH-CS mortars are characterized by a reduction both in energy requirement 
(GER) and greenhouse gases emissions (GWP) close to 60% respect to traditional OPC-based or 
CSA-OPC-CS mortars at equal strength class.
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