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Use of tartaric acid for the production of sustainable Portland-free CSA-based mortars

L. Coppola, D. Coffetti *, E. Crotti

Department of Engineering and Applied Sciences, University of Bergamo, Italy
Consorzio INSTM, UdR "Materials and Corrosion", Florence, Italy

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 CO_2 emissions was achieved.

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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 h. 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.

1. Introduction

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 sulphoaluminate 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 sulphoaluminate 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 sulphoaluminate clinker contributes only for 0.216 CO_2 g/g. In addition, the

* Corresponding author at: Department of Engineering and Applied Sciences, University of Bergamo, Italy.
E-mail addresses: luigi.coppola@unibg.it (L. Coppola), denny.coffetti@unibg.it (D. Coffetti), elena.crotti@unibg.it (E. Crotti).

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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 (CS), 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 sulphoaluminate cement is widespread for calcium sulphoaluminate – 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 sulphoaluminate 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 nitrilotris(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 C_3A 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.

2. Materials and methods

2.1. 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 (CS) was used to manufacture the reference mortar (CSA:OPC:CS = 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:CS = 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.

2.2. 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 $40 \times 40 \times 160$ mm^3 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.

3. 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 (Fig. 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%).

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 (Fig. 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.

Fig. 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 min, 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 min from mixing. The average time between mixing and placing a mortar in the job-site is generally close to 60 min. Based on this target, it is possible to conclude that the ideal set-retarding admixture dosage is equal to 0.8% vs. binder mass (Fig. 3).

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-h 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 (Fig. 4).

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 justifies the lower retardation of these mortars.

This retarding effect totally disappears at 28 days for all mixtures (Fig. 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 (Fig. 6).

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 (Figs. 7 and 8). In particular, SCM-based mixtures are characterized by a homogeneous microstructure with rich