

# Calcium Sulfoaluminate Cement and Fly Ash-Based Geopolymer as Sustainable Binders for Mortars

Antonio Telesca<sup>a,\*</sup>, Alessandra Mobili<sup>b</sup>, Francesca Tittarelli<sup>b</sup>, Milena Marroccoli<sup>a</sup>

<sup>a</sup> Scuola di Ingegneria, Università degli Studi della Basilicata, Viale dell'Ateneo Lucano 10, Potenza 85100, Italia

<sup>b</sup> Dipartimento di Scienze e Ingegneria della Materia, dell'Ambiente ed Urbanistica, Università Politecnica delle Marche, UdR INSTM, via Brecce Bianche 12, Ancona 60131, Italia  
[antonio.telesca@unibas.it](mailto:antonio.telesca@unibas.it)

This work investigates the hydration behaviour and the physico-mechanical properties of mortars based on calcium sulfoaluminate (CSA) cements and fly ash-based geopolymers (GEO) as alternatives to ordinary Portland cement. According to the EN 1504-3, mortars were prepared in order to reach three compressive strength classes, namely R1, R2 and R3 (R1 with  $R_c \geq 10$  MPa, R2 with  $R_c \geq 15$  MPa and R3 with  $R_c \geq 25$  MPa). CSA mortars were prepared by using sulfoaluminate cement alone (R3) or in mixture with a limestone filler (R1 and R2); GEO mortars were manufactured by alkali-activation of coal fly ash and calcium aluminate cement with a sodium silicate and potassium hydroxide water solution. The hydration behaviour was evaluated on pastes submitted to differential thermal-thermogravimetric and X-ray diffraction analyses. Mortars was analysed through mercury intrusion porosimetry; their mechanical properties were evaluated in terms of compressive strength and dynamic modulus of elasticity. Furthermore, capillary water absorption and drying shrinkage tests were carried out in order to evaluate their durability. Due to the rapid ettringite formation, CSA-based mixtures reached their maximum compressive strength values faster than the corresponding GEO mortars. Results showed that the lower modulus of elasticity of GEO mortars causes the higher drying shrinkage. Moreover, the lower porosity exhibited by GEO mortars was responsible for the lower water capillary absorption.

## 1. Introduction

The manufacturing process of cement is one of the largest consumers of fuel and raw materials; it is also one of the main industries responsible for generating carbon dioxide emissions (Gartner and Macphee, 2011; Gartner and Hirao, 2015). There are many ways to reduce the CO<sub>2</sub> produced by the Portland cement (PC) industry, namely: 1) the use of alternative fuels and/or raw materials, 2) the replacement of Portland clinker with "low-carbon" supplementary cementitious materials (Telesca et al., 2017), and 3) the development of alternative low-carbon binders (Coppola et al., 2018a, b). During the last ten years, an increasing attention has been paid by researchers and engineers toward calcium sulfoaluminate (CSA) cements (Glasser and Zhang, 2001; Juenger et al., 2011) and geopolymeric binders (Mobili et al., 2018). CSA cements contain C<sub>4</sub>A<sub>3</sub>S as main component and, depending on the synthesis temperature as well type and proportioning of raw materials, calcium sulfates, C<sub>2</sub>S, C<sub>4</sub>AF, C<sub>5</sub>S<sub>2</sub> and various calcium aluminates (Marroccoli, 2010a). Furthermore, their peculiar composition can be exploited not only for achieving valuable technical properties (e.g. rapid hardening, shrinkage compensation and chemical resistance (Winnefeld and Lothenbach, 2010; Telesca et al., 2014a)) but also for giving a more pronounced environmentally friendly feature to their manufacturing process. In this regard, compared to PC, CSA cements are characterized by: a) lower synthesis temperatures (<1350°C); b) reduced limestone requirement in the clinker-generating raw mix (Marroccoli et al., 2007); c) lower grinding energy required by the cement milling; and d) larger utilization of industrial wastes (García-Maté et al., 2013; Martin et al., 2017; Bertola et al., 2019) especially as kiln feed components (Marroccoli et al., 2009, 2010b; Guo et al., 2014; Telesca et al., 2016; Xu et al., 2017).

Geopolymers (GEO) represent a subclass of alkali-activated cements (AAC); they are obtained by the chemical reaction between a powdered aluminosilicate (precursor) with a low CaO content and an alkaline

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solution (activator) (Bondar et al., 2011; Carabba et al., 2017). Precursors include also industrial by-products such as blast furnace slag and coal fly ash (Huseien et al., 2018) or calcined clays and natural pozzolans; sodium or potassium hydroxides and silicates are the commonly employed alkali-activators (Provis, 2013). Depending on their composition, GEO materials can exhibit either high drying shrinkage (Mobili et al., 2016) or low drying shrinkage (Ma and Ye, 2015), fast or slow setting (Lee and Lee, 2013) and low capillary water absorption (Mobili et al., 2016).

In this paper, CSA cement and fly ash-based GEO are employed as alternative binders to ordinary Portland cement for the manufacture of mortars belonging to non-structural classes (R1 with  $R_c \geq 10$  MPa and R2 with  $R_c \geq 15$  MPa) and structural class (R3 with  $R_c \geq 25$  MPa), according to UNI EN 1504-3. The binders were tested and compared in terms of hydration behaviour, compressive strength, dynamic modulus of elasticity, drying shrinkage, porosity values and water capillary absorption.

## 2. Experimental program

### 2.1 Materials

A commercial CSA cement was supplied by an Italian cement manufacture. Mixture R3 was based only on CSA cement (R3 CSA); in R1 and R2 mixtures (R1 CSA and R2 CSA) CSA cement was partially replaced with a 30% and 60% by mass of limestone filler (LF, with particle size smaller than 100  $\mu\text{m}$ ), respectively.

GEO binders were prepared with a class F fly ash (FA), generated in an Italian coal-fired power plant, as aluminosilicate precursor. Calcium aluminate cement (CAC) was added as external source of aluminium. A blend of a sodium silicate (SS) solution ( $\text{SiO}_2=29.86\%$  and  $\text{Na}_2\text{O}=14.64\%$  by weight) and a potassium hydroxide solution (prepared by dissolving KOH pellets in demineralized water) was used as alkaline activator. Mortars were prepared by adding a commercial calcareous sand (with maximum grain size 8.0 mm) to each binding mixture.

### 2.2 Methods

Binder pastes were hydrated for curing periods ranging from 2 to 28 days. A 0.50 w/b mass ratio was employed for R1, R2 and R3 CSA-based mixtures; w/b equal to 0.42, 0.38 and 0.30 were used for R1, R2 and R3 GEO-based binders, respectively. Paste samples were placed in polyethylene bags inside a thermostatic bath at 20°C. At the end of a fixed curing time, each sample was pulverized for differential thermal-thermogravimetric (DT-TG) and X-ray diffraction (XRD) analyses, after treatment with acetone (to stop hydration) and diethyl ether (to remove water), followed by storing in a desiccator over silica gel-soda lime (to ensure protection against  $\text{H}_2\text{O}$  and  $\text{CO}_2$ ).

Table 1 reports the compositions of the investigated mortars; R3 CSA mortar was prepared with a/b (aggregate/binder) ratio equal to 3 and a 0.50 w/b (water/binder) ratio. R1 CSA and R2 CSA mortars were prepared with the same a/b ratio and with 0.54 and 0.48 w/b ratios, respectively. R1, R2 and R3 GEO were prepared with an aggregate/precursor ratio equal to 2.7 by weight and different w/b ratios (Table 1).

Table 1: Compositions of CSA- and GEO-based mortars

Mortar	CSA (g)	LF (g)	Water (g)	Sand (g)	FA (g)	CAC (g)	Alkaline solution			w/b
							SS (g)	KOH (g)	Demin. Water (g)	
R1 CSA	180	270	245	1350	-	-	-	-	-	0.54
R2 CSA	315	135	217	1350	-	-	-	-	-	0.48
R3 CSA	450	-	225	1350	-	-	-	-	-	0.50
R1 GEO	-	-	-	1374	468	41	153	17	163	0.42
R2 GEO	-	-	-	1386	472	41	154	35	147	0.38
R3 GEO	-	-	-	1417	483	42	158	80	117	0.30

After mixing, mortars, depending on the test, were poured into different moulds and cured at  $T=20^\circ\pm 1^\circ\text{C}$  ( $\text{RH}=90\pm 5\%$ ) for the first week and at  $T=20^\circ\pm 1^\circ\text{C}$  ( $\text{RH}=50\pm 5\%$ ) until testing.

Compressive strength (EN 1015-11) and dynamic modulus of elasticity (EN 12504-4) measurements were carried out on 40x40x160 mm specimens cured for 28 days. Mercury intrusion porosimetry (MIP) analysis was employed on samples mortar cured at 28 days. Free drying shrinkage measurements were carried out on 40x40x160 mm specimens during 2 hydration months; moreover, capillary water absorption measurements were carried on 40x40x40 mm specimens after 28 days of curing (EN 1015-18).

### 3. Results and discussion

Figure 1 shows the DT (left)–TG (right) thermograms for CSA-based cement pastes (up) and GEO-based systems (down) cured at 2 and 28 days. With reference to CSA cement pastes, the DT-TG apparatus was able to identify (Taylor, 1997) ettringite, calcium monosulfoaluminate (dodeka)hydrate ( $3\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot\text{CaSO}_4\cdot 12\text{H}_2\text{O}$ ) and calcium carbonate ( $\text{CaCO}_3$ ) through the following dehydration endothermic peaks:  $152^\circ\pm 7^\circ\text{C}$ ;  $203^\circ\pm 5^\circ\text{C}$ ; and  $855^\circ\pm 16^\circ\text{C}$ , respectively.

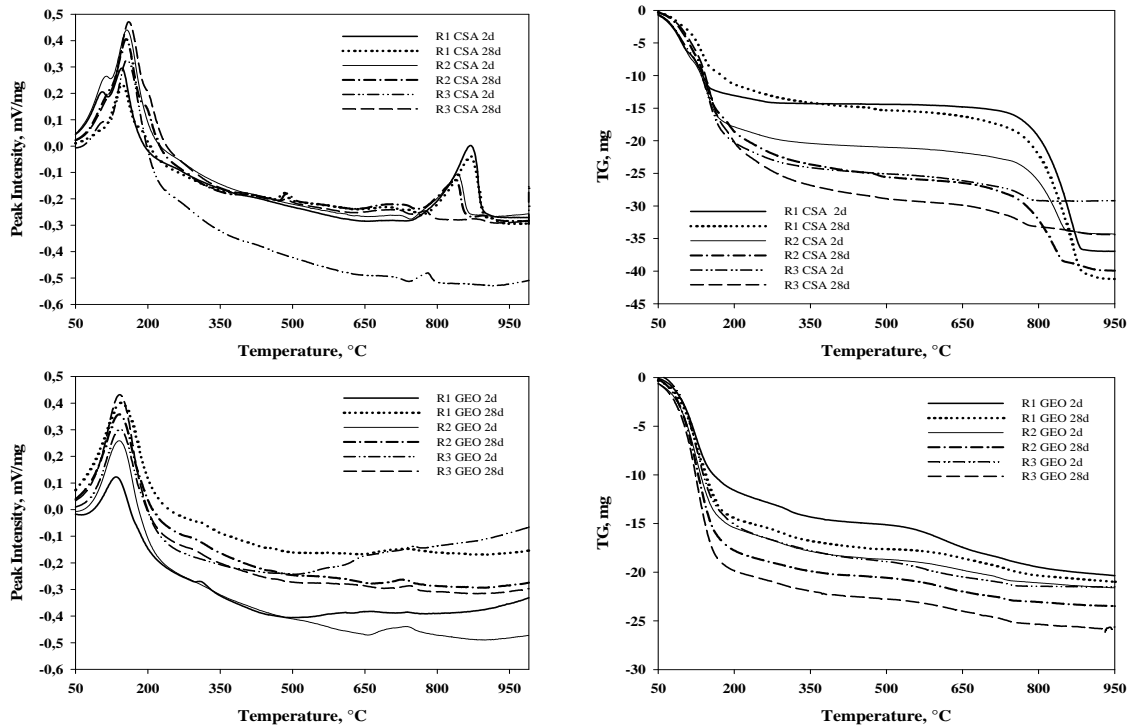


Figure 1: DT (left)–TG (right) thermograms for CSA-based cements (up) and GEO-based systems (down) hydrated for 2 and 28 days

From an overall examination of the DT curves, it is recalled here: i) ettringite is detected both at 2 and 28 days of curing; ii) calcium monosulfoaluminate (dodeka)hydrate, originating from the decomposition of ettringite in presence of calcium aluminate hydrates and in lack of sulfates, is observed only at 28 days of hydration; iii)  $\text{CaCO}_3$  is also seen in R3 systems due to a partial carbonation of lime (already present in the CSA cement). With reference to GEO pastes, the DT-TG technique was mainly used for the evaluation of the adsorbed water dehydration as well as the hydroxyl groups condensation; in this regard all DT curves are very similar to each other and reveal the presence of a sharp endothermic peak between  $134^\circ\text{C}$  and  $143^\circ\text{C}$ . Furthermore, a mass loss, ranging from about 15% to 22% is highlighted by TG thermograms. Few amounts of carbonates, related to carbonation phenomena, are also identified. XRD results confirm the DT-TG findings; in the CSA binders, ettringite represents the main hydration product already at two days of hydration, while monosulfate is detected only at 28 days; peaks for anhydrous phases, namely dicalcium silicate and calcium carbonate, do not show any significant change of their XRD signals. XRD patterns for GEO systems confirm their amorphous nature as one of the major structural characteristics of geopolymers; in fact, the presence of an amorphous halo (comprised in the range  $27^\circ\text{--}38^\circ 2\theta$ ) is evident at all the investigated curing periods. Furthermore, crystalline phases (e.g. quartz and calcite), belonging to fly ash, and other signals, corresponding to zeolite structures, are also detected. Table 2 shows the compressive strength values until 28 days curing. CSA and GEO values are higher than those indicated in the UNI EN 1504-3 at 28 days for the three strength classes. Furthermore, as expected, the replacement of CSA cement with limestone filler reduces the compressive strength values owing to the lower amount of hydration products (Martin et al., 2015). In addition, CSA mortars show a different mechanical strength development depending on their strength class; in fact, at 2 days of curing, while R1 CSA shows a mechanical compressive strength value equal to only 1 MPa, R2 and R3 CSA-based mortars already display strength values equal and higher to those required by the UNI EN 1504-3. For GEO mortars, compressive strength values are positively affected by both the concentration of the alkaline solution and the reduction of w/b ratio (Mobili et al., 2016). In addition, GEO mortars show a gradual

development in strength from between 2 and 28 days of curing. Compared to CSA mortars, the slower development in strength of GEO systems is caused by fly ash reaction mechanisms that continue to reorganize leading up to strength development even after long periods of time (Lloyd, 2009).

Table 2: Compressive strengths ( $R_c$ ), Dynamic modulus of elasticity ( $E_d$ ), total porosity ( $V_p$ ) and capillary water absorption coefficient ( $C$ ) of mortars

Mortar	$R_c$ after 2 days (MPa)	$R_c$ after 7 days (MPa)	$R_c$ after 28 days (MPa)	$E_d$ (GPa)	$V_p$ (%)	$C$ ( $\text{kg}/(\text{m}^2\text{min}^{0.5})$ )
R1 CSA	1	4	10	16	21	0.573
R2 CSA	15	22	24	24	17	0.441
R3 CSA	30	39	36	30	13	0.271
R1 GEO	3	6	13	12	17	0.502
R2 GEO	4	9	19	18	14	0.473
R3 GEO	6	18	32	25	13	0.355

The dynamic modulus of elasticity ( $E_d$ ) of mortars after 28 days of curing is also reported in Table 2. As expected, with the increase of the strength class the  $E_d$  value increases (Mobili et al., 2015); the  $E_d$  values for GEO systems were lower than those exhibited by the corresponding CSA mortars (i.e. 25%, 25% and 17% for R1, R2 and R3 strength classes). The results confirm that geopolymers are less rigid than ordinary cement-based mortars (Nath and Sarker, 2017) and CSA-based mortars (Mobili et al., 2017).

Table 2 shows the total porosity values ( $V_p$ ) for mortar samples cured at 28 days. These results confirm that the total porosity values decrease with the reduction of w/b ratio for all the systems. When moving from R3 to R2 and from R2 to R1 systems, the aforementioned trend was also due to the reduction of hydration products, without being counterbalanced by the filler effect of addition of limestone powder for CSA mortar samples. Due to their low w/b ratios (Mobili et al., 2016), GEO mortars show the lowest  $V_p$  values (Tittarelli et al., 2018), when compared to CSA mortars belonging to the same strength class.

Table 2 also reports the values of capillary water absorption coefficient ( $C$ ) for the investigated mortars. The lower the mechanical strength class, the higher the  $C$  values inasmuch as they strictly depend on the  $V_p$ . For the same mechanical strength class, GEO mortars show (with the exception of R1 GEO) higher  $C$  values than CSA mortars due to the presence of a higher fraction of capillary pores with larger diameters able to absorb water faster than those with smaller dimensions (Mobili et al., 2017).

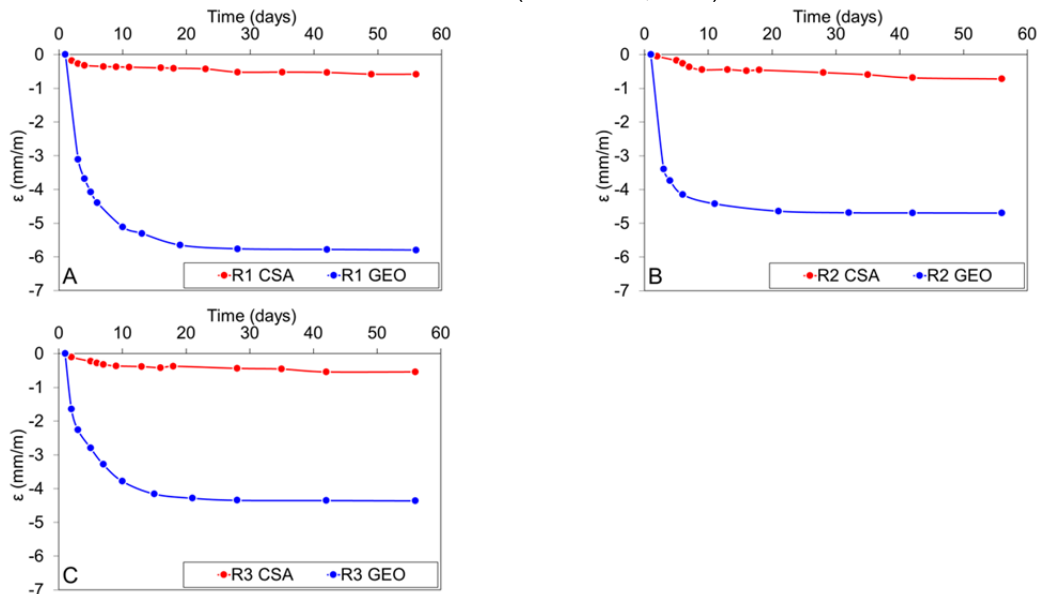


Figure 3: Drying shrinkage of mortars belonging to: A. R1 class, B. R2 class and C. R3 class

Drying shrinkage measurement results (Figure 3) show that GEO mortars, due to their lower modulus of elasticity (Mobili et al., 2016), shrink about 6 times more than CSA mortars; furthermore, moving up the higher strength classes, GEO mortars shrink less due the higher concentration of the alkaline solution which leads to (i) reduced porosity, (ii) improved mechanical strength and (iii) greater stiffness of the material (Thomas et al., 2017). All the CSA mortars exhibited almost zero shrinkage, most probably owing to suitable ettringite

nucleation and formation rates and a good compromise between deformability and the strength of the paste, as outlined in the introduction (Telesca et al., 2014b).

#### 4. Conclusions

This study demonstrates that the mechanical strength of both calcium sulfoaluminate- and geopolymers-based binders can be adjusted in order to obtain mortars fitting the three strength classes indicated in the UNI EN 1504-3 (R1 with  $R_c \geq 10$  MPa, R2 with  $R_c \geq 15$  MPa and R3 with  $R_c \geq 25$  MPa).

In particular, the following observations were made:

- the hydration behaviour of CSA pastes is clearly determined by the rapid formation of ettringite while no hydrated phases are clearly discernible in GEO systems;
- CSA mortars generally exhibited a higher strength development than GEO mortars where the geopolymerization of fly ash is relatively slow at room temperature (even though it continues at later curing periods);
- the dynamic modulus of elasticity of GEO mortars is about 20% lower than that of CSA mortars;
- the capillary water absorption of GEO mortars is slightly higher than that of CSA mortars;
- the drying shrinkage of GEO mortars is 6 times higher than that of CSA mortars, due to their lower modulus of elasticity.

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