Calcium Sulfoaluminate Based Concrete - Mechanical Characterization

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Abstract. In the last few years, sustainable cement manufacturing has been under the spotlight. Finding alternative cements while maintaining or improving current performance standards is scientist’s effort. In particular, calcium sulfoaluminate (CSA) cements represent a very promising alternative compared to ordinary Portland cement (OPC) because of their high early strength, excellent durability, good performance in aggressive conditions, low energy consumption as well as reduced CO₂ emissions. This paper presents the results of several experimental tests, planned in order to characterize the thermo-hygro-mechanical behaviour of CSA-based concrete since first minutes after casting. Twelve different tests were carried out: compressive strength and indirect tensile tests, stress-strain curve plot, rheology (creep and shrinkage) tests, water porosity, semi-adiabatic temperature evolution during hardening, fatigue test. Different curing periods were selected in order to analyse changes in strength. Experimental results show that CSA-based cements can be formulated to produce durable concrete with physical properties comparable to those of common classes of Portland cement concrete.

1. Introduction

Concrete is the most common construction material on earth and will continue to be required far into the future. It is well established that the production of Portland cement, typically used for cementitious material, is energy intensive as it involves the consumption of large quantities of raw materials, energy, and heat. Moreover, cement industry generates a significant amount of solid waste materials and gaseous emissions. For this reason, increasing attention is being paid to cementitious materials that can partially or completely substitute Portland cement.

Sulfoaluminate cement has been promoted in the last years as a very promising alternative to ordinary Portland cement for a more sustainable development [1], as it reduces the environmental impact of cement industry while preserving or even improving common performance standards. Indeed, the production of sulfoaluminate cements requires less limestone and less energy than the conventional clinkers, resulting in a reduction of CO₂ emissions per unit of clinker [2]. Valenti et al. [3] pointed out a number of environmental friendly aspects of calcium sulfoaluminate cements, as for instance the possibility to reuse industrial wastes and by-products, which are usually hard to dispose. Moreover, it has been shown that the hydration process of sulfoaluminate concretes involves a fast development of ettringite causing a rapid strength gain [4]. This property, together with the durability of concrete, makes it particularly suitable for civil engineering needs [5].
Sulfoaluminate clinkers can be obtained from the synthesis of a mixture of limestone, bauxite and calcium sulphate. The clinkering of that mix occurs at a lower temperature than Portland clinker, around 1250 –1300°C. The clinkering reactions of the various components of sulfoaluminate clinker are given in [6]. The details and the mass of carbon dioxide emitted for every ton of clinker component fabricated are given in [7].

Over the past decades, a number of studies have been conducted to characterize the chemical nature and mechanical properties of this type of sulfate-based binder. The use of calcium sulfoaluminate cement for concrete mixtures has been analysed [8] to solve the problem of strength development at low temperature. Gallardo et al. [9] analysed the mechanical (compressive strength) and chemical behaviour of sulfoaluminate concrete obtained from a mixture of fly ash, aluminium slag, and fluorogypsum.

However, although several research works have been conducted on sulfoaluminate concrete, they focus mostly on the chemical nature and composition of this type of mixture. Indeed, very few studies deal with the mechanical behaviour of sulfoaluminate concrete and, in particular, there is no relevant prior art that comprehensively describes the mechanical properties of the considered material.

The main goal of this study is to provide a comprehensive and exhaustive mechanical characterization of sulfoaluminate concretes. For this aim, the mechanical properties of concrete specimens obtained using a sulfoaluminate cement have been investigated experimentally. A series of destructive and non-destructive tests have been carried out: compressive strength tests, indirect tensile tests, three-point bending tests, fracture energy evaluation, shrinkage tests, creep tests, setting time measurement, stress (σ) – strain (ε) curves delineation, depth of penetration of water under pressure tests, semi-adiabatic curing tests, fatigue tests and freeze-thaw tests.

2. Tests description and results discussion

In this experimental program, twelve different types of testing have been carried out on specimens made with sulfoaluminate cement in order to characterize the thermo-hygro-mechanical behaviour of sulfoaluminate concrete mixtures.

The work was performed on a concrete produced industrially in a 8 m³ truck mixer; three aggregates have been used for the casting of the concrete (sand 0 – 2 mm, sand 0 – 4 mm and gravel); the concrete recipe contains 400 kg/m³ of binder and a w/c ratio of 0.44; the density of the concrete was 2351 kg/m³. A superplasticizer PV 39 NX from Sika was added for adjusting the consistency of the concrete to a class S4 and 0.5% of citric acid was used as set retarder. The binder used for the tests was a commercial sulfoaluminate cement (Next Binder SL05 NF) consisting of sulfoaluminate clinker, anhydrite and CEM I 52.5 R and containing as main mineral phase C₃S (36 %), C₄A₃$ (17%), C₂S (15%), C$ (10%).

A total of 173 specimens have been cast within 50 minutes. Among these specimens, 20 have been used for the calibration phases, and 153 for the test phases. They were all cast from the same batch of mixed concrete, which ensured the material properties of all the specimens to be the same. The casting included a number of cubic, prismatic and cylindrical specimens of different sizes.

2.1. Compressive strength test

Compressive strength tests have been carried out on cubic specimens 150 mm square, according to the UNI EN 12390-3 methodology [10]. Different curing periods have been selected in order to analyse variations in compressive strength with respect to time periods from about 3 hours to 199 days. A total number of 53 specimens have been tested.

Compressive strength data from concrete specimens made using sulfoaluminate cement have been compared with theoretical data given in the new fib Model Code 2010 [11]. The mean strength values are reported in Table 1.

\[1\] C = CaO, S = SiO₂, $ = SO₃, A = Al₂O₃
2.2. Indirect tensile test

Indirect tensile tests have been performed on cylindrical specimens of 150 mm diameter and 300 mm length. A Baldwin-Zwick testing machine with a loading capacity of 500 KN has been used. Cylindrical specimens were loaded diametrically across the circular cross-section until a tensile failure occurred, according with the UNI EN 12390-6 [12] test methodology. Tests have been performed at different ages, ranging from 3 hours to 203 days. A total number of 30 specimens have been tested.

As for the compressive strength, results from concrete specimens made using sulfoaluminate cement have been compared with theoretical data given in the new fib Model Code 2010 [11]. The mean strength values are presented in Table 2.

| Table 1. Compressive strength test. |
|-------------------------------------|
| Sample No. | Time from casting [hours] | Mean cubic compressive strength Rcm [MPa] | MC2010 compressive strength fcm [MPa] |
| 1-5 | 3.3 | 15.6 | 4.27 |
| 6-10 | 5.7 | 19.6 | 8.58 |
| 11-15 | 10.4 | 25.1 | 15.20 |
| 16-20 | 22.0 | 29.8 | 25.08 |
| 21-25 | 46.3 | 33.0 | 35.35 |
| 26-28 | 69.3 | 33.7 | 40.64 |
| 29-33 | 165.9 | 38.3 | 50.66 |
| 34-38 | 333.9 | 45.7 | 57.05 |
| 39-43 | 669.8 | 62.0 | 62.01 |
| 44-48 | 1532.9 | 64.4 | 66.36 |
| 49-53 | 4773.8 | 65.6 | 70.28 |

| Table 2. Indirect tensile strength test. |
|-----------------------------------------|
| Sample No. | Time from casting [hours] | Mean indirect tensile strength fctm [MPa] | MC2010 tensile strength fctm [MPa] |
| 1-3 | 3.6 | 3.0 | 0.99 |
| 4-6 | 6.0 | 3.2 | 1.51 |
| 7-9 | 11.2 | 3.6 | 2.23 |
| 10-12 | 22.6 | 3.9 | 3.06 |
| 13-15 | 46.3 | 4.7 | 3.83 |
| 16-18 | 165.8 | 5.1 | 4.90 |
| 19-21 | 340.1 | 5.5 | 5.33 |
| 22-24 | 669.9 | 5.6 | 5.63 |
| 25-27 | 1534.1 | 5.9 | 5.90 |
| 28-30 | 4845.9 | 7.3 | 6.14 |
2.3. Three-point bending test
Three-point bending tests have been carried out on specimens 480 mm long, 160 mm wide and 160 mm tall. A Baldwin-Zwick testing machine with a loading capacity of 500 KN has been used. Specimens have been placed on two parallel supporting pins and a loading force has been applied in the mid-span by means of a loading pin, according to UNI EN 12390-5 [13] test methodology. Testing took place at an age of 28 days.

Five specimens have been tested and load-displacement plots have been obtained for each test specimen.

Table 3 lists the experimental results after the samples were broken under three-point bending tests.

| Sample No. | Load max [N] | Flexural strength $f_{ct,fl}$ [MPa] | Mean flexural strength $f_{ct,fl}$ [MPa] |
|------------|--------------|----------------------------------|----------------------------------|
| FL01       | 34000        | 5.98                             |                                  |
| FL02       | 34700        | 6.10                             |                                  |
| FL03       | 35500        | 6.24                             | 6.19                             |
| FL04       | 32500        | 5.71                             |                                  |
| FL05       | 39400        | 6.93                             |                                  |

Figure 1 illustrates results from three-point bending tests in terms of applied load versus mid-span displacement of the specimen under the loading pin of the electronic tester machine. Load-displacement curves have been obtained for each specimen.

2.4. Fracture energy evaluation
The analysis of the concrete fracture energy has been carried out on specimens 600 mm long, 150 mm wide and 150 mm tall. All tests have been performed with a servo-hydraulic test machine MTS 250 KN of load capacity, according to the test methodology described in RILEM TC-50-FMC1 [14]. Testing took place at about an age of 28 days on five specimens for each considered mixture.

The numerical results obtained for the two different mixtures are listed Table 4. Load-crack width diagrams have been obtained for each specimen, as shown in Figure 2.
Table 4. Fracture energy evaluation.

| Sample No. | Load max [KN] | Last vertical displacement [mm] | Fracture energy $G$ [J/m²] |
|------------|---------------|---------------------------------|----------------------------|
| E01        | 1.505         | 5.98                            | 264                        |
| E02        | 1.429         | 6.10                            | 254                        |
| E03        | 1.303         | 6.24                            | 225                        |
| E04        | 1.467         | 5.71                            | 234                        |
| E05        | 1.539         | 6.93                            | 234                        |

Figure 2. Crack widths comparison in function of load.

2.5. Shrinkage test
In order to assess the axial shrinkage strain of concrete experienced during hardening, shrinkage tests have been carried out following the test procedure defined in UNI 11307 [15].

Four cylindrical specimens of 150 mm diameter and 300 mm length have been used and three steel pins, spaced to each other 120° and called H1, H2, H3, have been glued on the head surfaces of the concrete specimens in order to measure shrinkage dimensional changes.

Figure 3. Shrinkage evolution in time.
For the first three specimens, exposed to a relative humidity (RH) of 50±2% and a temperature (T) of 20±1°C, shrinkage strain measurements started on the first day after casting, whereas for the fourth specimen the shrinkage has been measured starting after 28 days from casting.

Results from shrinkage tests are summarized in Figure 3, and compared with MC2010 shrinkage curves. The mean deformation values from the experimental data obtained for the four specimens loaded under identical conditions are reported.

2.6. Creep Test

Creep influence has been investigated by performing tests on cylinders of 150 mm diameter and 300 mm length, using contrast steel frames and a load system able to ensure a uniform and constant level of uniaxial compression, according to the test procedure described in ATSM C512 [16]. Specimens have been subjected to constant loads of 10.2 MPa, 12.7 MPa, 14.9 MPa, 15.7 MPa, after a curing periods of 2, 3, 7 and 28 days respectively.

Creep tests results are reported in a time-strain diagram for each curing period, as shown in Figure 4 to 7. The shrinkage strain obtained from shrinkage tests (see the previous section) has been subtracted in order to consider the creep contribution only.

![Figure 4. Creep and shrinkage curves (2 days curing period).](image1.png)

![Figure 5. Creep and shrinkage curves (3 days curing period).](image2.png)

![Figure 6. Creep and shrinkage curves (7 days curing period).](image3.png)

![Figure 7. Creep and shrinkage curves (28 days curing period).](image4.png)

2.7. Setting time

The analysis of the concrete setting time has been performed using cylindrical specimens of 150 mm diameter and 300 mm length. A 250 KN MTS testing machine has been used according to the test methodology described in UNI 7123 [17]. Results are listed in Table 5.
Table 5. Setting time evaluation

| Test No | hour | minute | Load [MPa] |
|---------|------|--------|------------|
| casting | 0    | 0      | 0          |
| 01      | 1    | 59     | 9          |
| 02      | 2    | 20     | 18         |
| 03      | 2    | 26     | 56         |

2.8. Stress ($\sigma$) – Strain ($\epsilon$) curves

The relationship between stress ($\sigma$) and strain ($\epsilon$) for the considered concrete mixture has been determined by means of cylindrical specimens of 150 mm diameter and 300 mm length.

Table 6. Stress- strain curves results.

| Sample n° | Time from casting [hours] | Load max [KN] | Peak strength $f_{c2}$ [MPa] | Peak strain $\epsilon_{c2}$ [x10E-3] | Young modulus $E_{cm}$ (40%) [MPa] | Mean peak strength $f_{\bar{c}}$ [MPa] | Mean peak strain $\epsilon_{\bar{c}}$ [x10E-3] | Mean Young modulus $E_{cm}(40\%)$ [MPa] |
|-----------|---------------------------|---------------|-----------------------------|-------------------------------------|-----------------------------------|-----------------------------------|-----------------------------------|-----------------------------------|
| 1         | 5.4                       | 154           | 8.71                        | 0.27                                | 16418                             | 9                                 | 0.27                              | 16418                             |
| 2         | 6.4                       | 354           | 20.03                       | 2.52                                | 15926                             | 21                                | 2.51                              | 16370                             |
| 3         | 6.7                       | 379           | 21.42                       | 2.40                                | 17031                             | 27                                | 2.42                              | 17747                             |
| 4         | 7.0                       | 364           | 20.61                       | 2.61                                | 16154                             | 31                                | 2.51                              | 18008                             |
| 5         | 11.5                      | 490           | 27.74                       | 2.69                                | 16156                             | 27                                | 2.42                              | 17747                             |
| 6         | 11.7                      | 482           | 27.27                       | 2.26                                | 18714                             | 36                                | 3.22                              | 18733                             |
| 7         | 12.0                      | 465           | 26.34                       | 2.32                                | 18371                             | 31                                | 2.51                              | 18008                             |
| 8         | 23.4                      | 580           | 32.81                       | 2.72                                | 18288                             | 31                                | 2.51                              | 18008                             |
| 9         | 23.6                      | 551           | 31.19                       | 2.43                                | 18111                             | 31                                | 2.51                              | 18008                             |
| 10        | 23.8                      | 493           | 27.89                       | 2.38                                | 17626                             | 31                                | 2.51                              | 18008                             |
| 11        | 47.6                      | 655           | 37.04                       | 3.26                                | 19043                             | 36                                | 3.22                              | 18733                             |
| 12        | 48.0                      | 583           | 32.98                       | 3.01                                | 18190                             | 36                                | 3.22                              | 18733                             |
| 13        | 48.3                      | 685           | 38.76                       | 3.38                                | 18966                             | 36                                | 3.22                              | 18733                             |
| 14        | 167.5                     | 726           | 41.06                       | 5.39                                | 19465                             | 41                                | 5.24                              | 19331                             |
| 15        | 167.8                     | 731           | 41.36                       | 5.44                                | 19575                             | 41                                | 5.24                              | 19331                             |
| 16        | 168.0                     | 706           | 39.94                       | 4.88                                | 18953                             | 41                                | 5.24                              | 19331                             |
| 17        | 339.9                     | 843           | 47.72                       | 4.81                                | 21206                             | 48                                | 4.73                              | 21641                             |
| 18        | 340.3                     | 848           | 47.97                       | 4.79                                | 21725                             | 48                                | 4.73                              | 21641                             |
| 19        | 340.8                     | 848           | 48.01                       | 4.61                                | 21990                             | 48                                | 4.73                              | 21641                             |
| 20        | 672.2                     | 938           | 53.10                       | 4.11                                | 24943                             | 53                                | 4.14                              | 23058                             |
| 21        | 672.5                     | 922           | 52.18                       | 3.96                                | 20990                             | 53                                | 4.14                              | 23058                             |
| 22        | 673.1                     | 943           | 53.35                       | 4.36                                | 23241                             | 49                                | 2.72                              | 25582                             |
| 23        | 2019.4                    | 988           | 55.90                       | 3.68                                | 25824                             | 49                                | 2.72                              | 25582                             |
| 24        | 2020.0                    | 801           | 45.32                       | 2.27                                | 25064                             | 49                                | 2.72                              | 25582                             |
| 25        | 2020.7                    | 813           | 45.99                       | 2.22                                | 25857                             | 49                                | 2.72                              | 25582                             |
| 26        | 4772.3                    | 1173          | 66.38                       | 4.30                                | 25065                             | 60                                | 4.26                              | 25513                             |
| 27        | 4773.4                    | 1172          | 66.30                       | 4.68                                | 26917                             | 60                                | 4.26                              | 25513                             |
| 28        | 4774.9                    | 859           | 48.60                       | 3.81                                | 24558                             | 60                                | 4.26                              | 25513                             |
A 1000 KN MTS testing machine has been used, and an increasing monotonous loading has been applied with a test speed of 5 μm/s until the post-peak branch is reached. A total number of 28 specimens have been tested. Results are listed in Table 6 referring to each specimen.

2.9. Depth of penetration of water under pressure
The depth of penetration test has been performed using cylindrical specimens of 150 mm diameter and 300 mm length. A 250 KN MTS testing machine has been used according to the test methodology described in UNI EN 12390-8 [18]. Results are shown in Table 7.

Table 7. Depth of penetration of water.

| Sample n° | Days from casting | Brazilian test, maximum load [MPa] | Brazilian test, tensile strength [MPa] | Penetration [mm] |
|-----------|-------------------|------------------------------------|----------------------------------------|-----------------|
| 01        | 35                | 187                                | 6.23                                   | 17              |
| 02        | 35                | 211                                | 7.03                                   | 16              |
| 03        | 35                | 199                                | 6.63                                   | 16              |

2.10. Semi-adiabatic Curing Test
Temperature rise evaluation in semi-adiabatic curing conditions has been carried out on two specimens 300 mm long, 300 mm wide and 800 mm tall.

The four lateral faces (30x80cm) and the bottom face of the specimens have been covered by polystyrene panels 5 cm thick; the upper face was in contact with the air.

Four thermocouples have been positioned inside the specimens as described below:

1. Upper thermocouple: the thermocouple has been positioned in the centre of the squared upper face, 5 cm below the maximum casting level.
2. Upper external thermocouple: the thermocouple has been positioned in the middle of one of the four sides of the squared upper face, 1 cm below the maximum casting level.
3. Intermediate thermocouple: the thermocouple has been positioned along the specimen longitudinal axis, 15 cm below the upper one.
4. Lower thermocouple: the thermocouple has been positioned along the specimen longitudinal axis, 20 cm below the intermediate one.

Specimens have been cured at 20°C for a week (168 h) in a temperature-controlled environment. The temperature of the four thermocouples has been continuously measured, as reported in Figure 8.

![Figure 8. Temperature evolution in time](image_url)
2.11. Fatigue test
Fatigue tests have been carried out on cylindrical specimens of 150 mm diameter and 300 mm length. The specimens have been tested under cyclic loadings with a frequency of 3 Hz, a minimum compressive strength of 1MPa and the strength variation $\Delta\sigma$, as shown in Table 8.
All specimens have reached 3 million cycles without showing damage, cracking or failure.

| Sample n° | $\Delta\sigma$ [MPa] | Cycles [-] |
|-----------|-------------------|-----------|
| 1         | 20                | 3M        |
| 2         | 20                | 3M        |
| 3         | 20                | 3M        |
| 4         | 25                | 3M        |
| 5         | 25                | 3M        |
| 6         | 30                | 3M        |
| 7         | 30                | 3M        |

2.12. Freeze-thaw test
Freeze-thaw testing is conducted by exposing the product to freezing temperatures (approximately -10°C) for 24 hours, and then allowing it to thaw at room temperature for 24 hours. The sample is then placed in a higher temperature (approximately 45°C) for 24 hours and then placed at room temperature again for 24 hours. The sample is analysed for significant changes. The test was conducted following the UNI 7087:2002 procedure [19]. The results of freeze-thaw test are presented in Table 9.

| Sample n° | Days from casting | weight [kg] | $\Delta$weight [%] |
|-----------|-------------------|-------------|--------------------|
| 01        | 28                | 9.345       | -                  |
| 02        | 28                | 9.254       | -                  |
| 03        | 28                | 9.478       | -                  |
| 01        | 29                | 9.436       | 0.964              |
| 02        | 29                | 9.335       | 0.868              |
| 03        | 29                | 9.569       | 0.951              |

3. Conclusions
Several tests have been carried out in order to describe a specific sulfoaluminate based concrete mixture. The results led to a mechanical characterization of the material, quite comparable to the typical characteristics of ordinary Portland cement, and even underlying a rapid strength gain and durability in aggressive conditions and much lower shrinkage. These aspects make it applicable to civil engineering needs.
It can be concluded that a range of innovative building materials can be developed using sulfoaluminate cement. Other materials are under development.

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