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Does a tiny amount of dispersant make any change to refractory castable properties?

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Abstract

A growing demand for refractory castables with specific behaviors has given rise to a continuous technological evolution, mainly due to the broad knowledge of hydraulic binders available nowadays. The high alumina cements remain as the most important hydraulic binders for castables. Nevertheless, calcium aluminate bound castables still show a characteristic drop of strength at intermediate temperatures, which could also be affected by the castable chemical additive. Thus, this paper aims to highlight the influence of dispersants on the refractory castable properties with the firing temperature. It was noticed that the hydrates formed during the curing process of castable depends on the dispersing additive used. The FS60, a polycarboxylate ether, induced the AH_3 formation and its decomposition resulted in a more stable hydrate (AH), which increased the splitting strength with the thermal treatment temperature. At a high temperature, the CA_2 and CA_6 formation is also favored in the presence of this additive. However, it did not bring benefits to the castables creep behavior, resulting in a less tough structure.

Keywords: A. Firing; B. Porosity; C. Creep; C. Mechanical properties; D. Al_2O_3; E. Refractories

1. Introduction

Despite the steady decrease in the amount of cement used in high performance refractory castables, the high alumina cements, CAC (calcium aluminate cement) in particular, remain as the most important hydraulic binders for castables, mainly because they render high strength within 6–24 h after placement. Usually, high alumina castables require only 24 h to develop 70–80% of their ultimate strength if properly cured, conversely to the 28 days for the ordinary Portland cement concretes [1,2].

Typical calcium containing phases presented in the commercial CAC – $CaO{\cdot}Al_2O_3$ (CA), $CaO{\cdot}2Al_2O_3$ (CA_2) and $12CaO{\cdot}7Al_2O_3$ ($C_{12}A_7$), for instance – result in hydrated cement phases after curing the castable in a humid environment. CA is the most important component of CAC due to its higher content (50–70 wt%) and is responsible for developing the highest mechanical strength among the hydrated phases. CA_2 is a secondary phase in CAC (30–45 wt%), its refractoriness is higher than CA and it also takes longer to hydrate. $C_{12}A_7$ (2 wt% or less) rapidly hydrates and can be used to control the CAC setting rate, when added in small amounts [3–5].

When the cement is mixed with water the anhydrous phases begin to quickly dissolve generating a saturated solution of Ca^{2+} and $Al(OH)_4^-$ ions. Nucleation of hydration compounds and, thereafter, their crystal growth, result in an interlocked network responsible for setting and providing the pre-firing strength [1,6]. Thus, the hydration process is carried out through an initial dissolution and subsequent precipitation of hydrates from the supersaturated solution. An induction or incubation period is detected before this precipitation due to the nucleation barrier.

The initial step of the CAC reaction with water is the formation of a surface layer of hydrated calcium aluminate and an apparently amorphous aluminum hydroxide. In the induction period, the hydration rate is low and the thickness of the hydrated surface layer grows slowly. When the layer reaches a critical thickness, the stress caused by the intruded water molecules breaks it and the induction period ends with the formation of crystalline nuclei which grow by a dissolution–crystallization mechanism [1,3].

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Ions dissolution and hydrates precipitation rates are strongly dependent on the material composition and the crystalline structure of the products formed during precipitation. The latter is particularly influenced by the available water amount, reaction time, temperature, and the ratio between the concentrations of Ca^{2+} and $Al(OH)_4^-$ ions in the solution during the anhydrous phase dissociation [1,7]. In general, CAH_{10}, C_2AH_8, C_3AH_6 and AH_3 ($Al_2O_3{\cdot}3H_2O$) are known as the main calcium aluminate hydrates.

The hydrate CAH_{10} ($CaO{\cdot}Al_2O_3{\cdot}10H_2O$) is formed at the beginning of the precipitation and it is the main product when the cure proceeds either at low temperatures (5–10 °C) or with enough water amount. The hydrate C_2AH_8 ($2CaO{\cdot}Al_2O_3{\cdot}8H_2O$) is formed at temperatures in the range of 22 up to 35 °C. The C_3AH_6 ($3CaO{\cdot}Al_2O_3{\cdot}6H_2O$) is the main hydrate formed at temperatures higher than 35 °C or in the presence of low water quantities. The hydrate AH_3 ($Al_2O_3{\cdot}3H_2O$) is formed at all temperature ranges and water contents [4,7,8].

The conversion of metastable hydrates CAH_{10} and C_2AH_8 into more stable compounds, C_3AH_6 and AH_3 with time and/or temperature is followed by some mechanical strength change. As the temperature increases, the dehydration process continues until all the hydrated phases lose their crystallization water. Therefore, these castables show a characteristic strength drop at intermediate temperatures (often quoted to be in the range of 550–950 °C), when the hydraulic bond has already decomposed, but the still sluggish sintering process has not yet allowed the development of a ceramic bond. The precise temperature range where strength deteriorates is not clear, but it may depend on various factors, such as the type and proportion of hydrates, the curing temperature and the heating rate [1].

In practice, high temperatures (>600 °C) may actually be necessary for the complete dehydration of the compounds, because the heating up process increases the water vapor pressure, particularly inside the closed pores, and therefore further delaying the dehydration process. Although decomposed hydrates may start to interact with the castable matrix at temperatures as low as 900 °C, a significant reaction of the calcium aluminate crystallites themselves and with the neighboring matrix compounds, only occurs at temperatures close to 1100 °C [1]. Thus, the cementitious phase of refractory castables also plays a major role in the processes carried out at high temperatures [1,9].

Moreover, all reactions on hydration/dehydration/firing occurring in refractories, where calcium aluminate cement is the main binder, are influenced by the presence of additives because of their effect on both the dispersion of fine particles and the cement hydration process.

Bearing this in mind, the aim of this article is to evaluate the properties of refractory castables – mechanical strength and porosity at intermediate temperatures, and thermo-mechanical behavior at high temperature – in the presence of different chemical dispersants.

2. Experimental procedure

High-alumina self-flow castable compositions were formulated using the software PSDesigner [10]. The particle-size distribution was adjusted to the theoretical curves based on Andreasen's packing model [11], with a distribution coefficient (q) equal to 0.21. White fused aluminas were used as aggregates (69 wt%). The matrix mainly comprised of calcined aluminas, A17NE (22 wt%) and CT3000SG (3 wt%). Calcium aluminate cements of two different suppliers, CA14M (Almatis-US) and Secar 71 (Kerneos-France), were separately added up to 6 wt%. The characteristics of these raw materials and binders are presented in Table 1.

Different compounds were evaluated as dispersant agents: anhydrous citric acid (Labsynth, 192 g/mol, 99.5% purity, named CA) and the polymers of the polycarboxylate ethers family developed by BASF, FS10 and FS60. The content of additives and water needed to prepare each composition were previously set, as shown in Table 2.

The mixing of raw materials was carried out under constant rotation (44 rpm) in a castable rheometer [12]. Water was added continuously to improve the mixing efficiency and the flowability of castables [13,14]. After 60 s of dry mixing (to homogenize the raw materials), the water amount added was enough to attain the castable turning point (75 wt% of total water). At this stage the castable behaves as a continuous medium. Thereafter, the remaining water was added keeping the rotation until the complete mixing of the system was attained (approximately 5 min).

Castable compositions were cast into 40 mm × 40 mm cylindrical molds for both the splitting strength and apparent porosity measurements. For the refractoriness under load (RUL) and creep measurements, cylinders with 50 mm of external diameter and height, with a 12.4 mm central passing hole were cast. Samples were cured at 50 °C in an acclimatized chamber (Vötsch, model 2020), under a moisture-saturated environment (~100% RH, for 72 h) in order to provide enough hydration.

After the curing period, the samples subjected to splitting strength and porosity tests were first dried at 110 °C for 72 h, and were later submitted to the thermal treatment at 200, 400, 600, 800 and 1000 °C (1 °C/min) with a dwell time of 5 h.

The dewatering tests were performed in a TG device comprising an electric furnace coupled to a digital scale. The temperature and mass data were automatically recorded at 5 s intervals throughout the tests. Results correlating the sample's temperature with its mass loss profile were attained using the data from two tests performed with the same heating schedule on specimens of the same batch (one for mass data and the other for temperature acquisition). Applied heating schedules consisted of continuously heating from room temperature to 800 °C at 2.5 °C/min, with a dwell time of 20 min at that temperature.

The diametral splitting strength was measured according to the ASTM C496-90 standard in a MTS Universal Testing System, Model 810. A constant loading rate of 42 N/s (1000 kPa/min) was used. The splitting tensile strength was calculated by:

$$\sigma_f = 2\left(\frac{P}{\pi h d}\right) \tag{1}$$

Table 1
Characterization of raw materials and binders used for the preparation of the refractory castables.

Raw materials		Particles size distribution (μm)	Density (g/cm^3)	Surface area (m^2/g)
White fused alumina (aggregates)	4/10	4750–2000	3.90	–
	8/20	2360–710	3.90	–
	20/40	710–355	3.90	–
	40 F	425–106	3.90	–
	200 F	106–11.3	3.90	–
Calcined alumina (matrix)	A17NE	11.30–0.21	3.94	2.90
	CT 3000SG	6.69–0.21	3.97	8.00
Binders	CA14M	63–5.6	2.96	1.87
	Secar 71	63–8.0	2.98	1.17

Table 2
Additive and water content for preparing the different compositions of refractory castables.

Composition	Additives	Additive content		Water content (wt%)
		mg/m^2	wt%	
CA14M or Secar 71	CA	0.7	0.07	5.5
	FS10	1.3	0.13	5.2
	FS60	0.9	0.09	4.5

where, σ_f is the splitting tensile stress (MPa); P is the maximum load (N); h (mm) and d (mm) are the height and diameter of samples, respectively.

Apparent porosity was evaluated according to the ASTM C830 standard, using kerosene as the immersion liquid.

Thermo-mechanical properties were measured according to the ASTM C832-89 standard by refractoriness under load (RUL) and creep tests in a NETZSCH-421E device. RUL measurements were performed in pre-fired samples (600 °C for 5 h) and the analysis was carried out under continuous heating (5 °C/min) up to 1500 °C, under a compressive load of 0.2 MPa. Creep samples were also pre-sintered at 1500 °C for 24 h and then subjected to a compressive load of 0.2 MPa at 1450 °C for 48 h.

Additionally, some of the dispersants used were heated up to 900 °C for 5 h and their residues were evaluated by X-ray diffraction.

3. Results and discussion

3.1. Splitting strength and porosity measurements

The diametral strength results with the firing temperature for castables prepared with CA14M or Secar 71 in the presence of different dispersing additives, are shown in Fig. 1. In the presence of the FS60 additive, the highest splitting strength values were attained for both binders, regardless of the firing temperature.

The highest strength developed by the castables in the presence of FS60 can be related, *a priori*, to the efficient dispersion of their particles resulting in a well packed structure with lower porosity, as well as to the lower water content necessary for their processing (Table 2).

The results also showed a loss in mechanical strength mainly after heating at temperatures in the range of 600–800 °C. Strength loss is well known to occur in refractory castables bound with aluminate cement due to the dehydration process of CAC phases, as the temperature is increased.

Both the hydration and the dehydration processes are complex and not fully understood. CAH_{10} loses part of its water

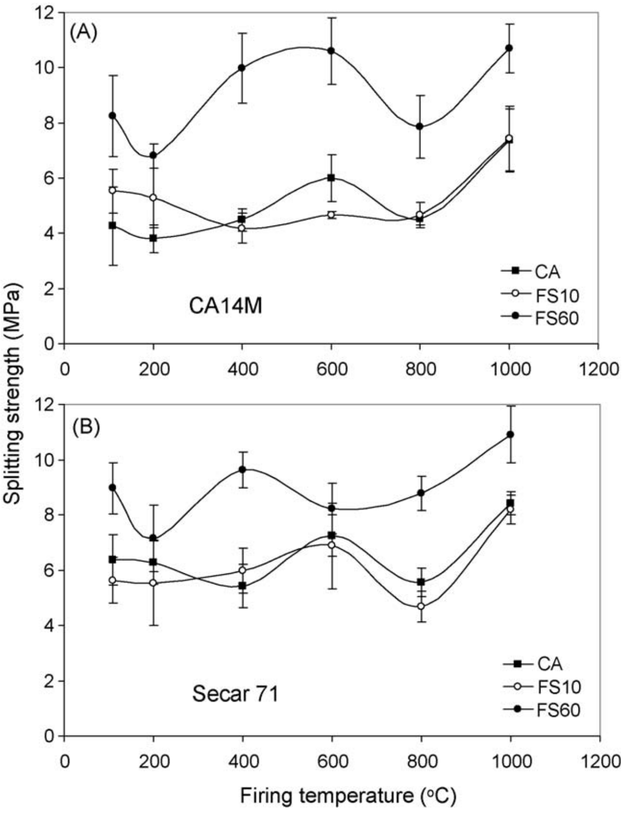

Fig. 1. Splitting strength as a function of firing temperature for castables prepared with CA14M or Secar 71 in the presence of different dispersing additives.

of crystallization at temperatures lower than indicated by thermal analysis (50–70 °C). Part of this water is even lost at a low relative humidity environment at room temperature and, as CAH_7 is sometimes detected, it is believed that CAH_{10} may display three water molecules that are easily lost [1], although another study indicated dehydration in the range of 100–130 °C [1,15]. The C_2AH_8 usually decomposes in the temperature range of 170–195 °C. The C_3AH_6, often shows a stepwise dehydration process resulting in the formation of the metastable compound $C_3AH_{1.5}$, after losing 4.5 water molecules (300–360 °C). The remaining molecules are decomposed and released as water vapor at a higher temperature (≅450 °C). Finally, AH_3 gel and gibbsite, the calcium free phases, usually decompose in the range of 210–300 °C [1,15].

In a previous paper by the authors [16], it has been shown that the water content present in the castable affects the sort of hydrate formed, according to the phase diagram for CAC hydration reaction [1]. With the increase of water content necessary for the castable processing (CA > FS10 > FS60), the C_2AH_8 formation took place, highlighted by the characteristic dehydration peak at 200 °C measured by thermogravimetric analysis. Furthermore, the retarding effect of the CA additive on the cement hydration mechanism due to Ca^{2+} ions complexation, inhibited the formation of the C_3AH_6 hydrate.

Conversely, in the presence of the FS60 additive, the lower water content necessary for the castable processing explained the main formation of C_3AH_6 hydrate, at a curing temperature of 50 °C.

Thus, for the thermal treatment close to 400 °C, a complete dehydration and a strength reduction due to the increased porosity and pore growth for castables prepared mainly with the FS60 additive was expected. The dehydration of C_2AH_8 must also induce a more significant strength loss, due to the higher water vapor content being released when compared to the C_3AH_6.

However, the splitting strength results (Fig. 1), as well as the apparent porosity (Fig. 2) showed an opposite trend. Conversely to the expected strength loss, the results indicated that heating favored the formation of another type of hydrate thermally more stable, responsible for increasing the splitting strength, mainly in the FS60 presence.

In fact, matching with the literature [17], the present results also have shown [16] that the main formation of C_3AH_6 (a calcium rich phase) with the FS60 additive might have induced the AH_3 phase formation, because of the relative abundance of $AlOH_4^-$ ions in the castables mixing and curing steps. This phase, usually as a gel and, due to its high capacity to fill interparticle gaps, could be associated to the splitting strength increase.

Moreover, AH_3 gel usually dehydrates between 210 and 300 °C but may otherwise convert into boehmite (AH), which only decomposes between 530 and 550 °C. Thus, the CAC hydrated phases can be decomposed up to around 600 °C, depending on the heating rate and the sample size. It is important to highlight that these phases are difficult to be detected due to their low crystalinity, small amount, low chemical stability and their dependence on the reagents/oxides ratio. Thus, their study on systems containing only matrices is not valid and on actual castables, they are difficult to be detected by X-ray diffraction analysis.

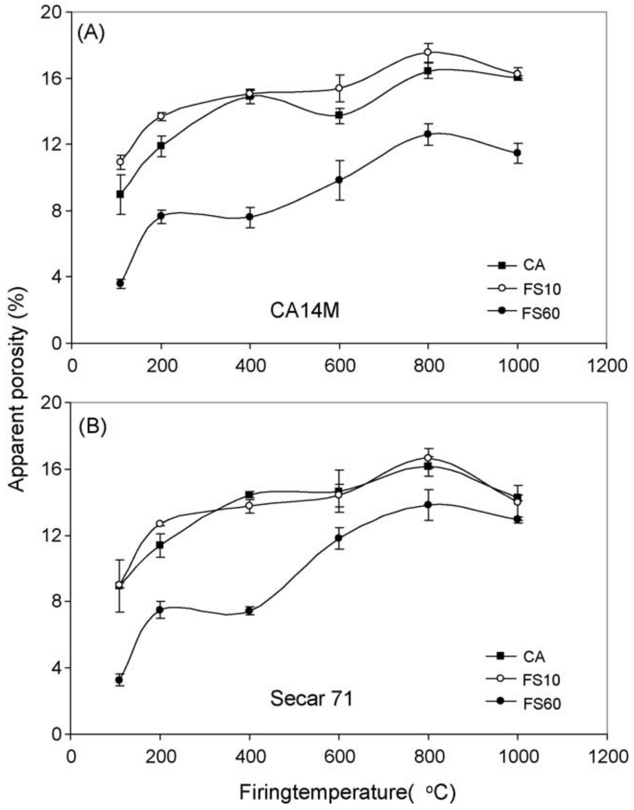

Fig. 2. Apparent porosity as a function of firing temperature for castables prepared with CA14M or Secar 71 in the presence of different dispersing additives.

The dehydration of higher amounts of AH_3 (210–300 °C) can explain the superior formation of AH, which is one of the most thermally stable hydrates. The latter is responsible for the splitting strength values in the temperature range from 400 up to 600 °C, detected in the presence of CA14M. The complete dehydration of AH, explains the drop in the splitting strength at a firing temperature of 600 °C.

The main formation of AH in the presence of FS60 was confirmed by drying tests carried out in a previous study by the authors [16], as shown in Fig. 3. Three well defined regions can be identified in this figure: (1) region A, below 200 °C, related to the presence of free-water and hydrates with higher water content (C_2AH_8); (2) region B (200–400 °C) corresponding to the formation of hydrates C_3AH_6 and AH_3; (3) region C (400–600 °C) due to the AH formation. In the presence of both CA and FS10 additives, the dehydration is mainly carried out in region A, and the peaks in region C are less noticed mainly in the presence of CA. When the FS60 additive is used, the main formation of hydrates is detected in region B, although the formation of hydrates in region C is also favored.

On the other hand, above 900 °C, CA_2 crystallization and sintering led to a modest decrease of the porosity and improved the strength, as shown in Figs. 1 and 2.

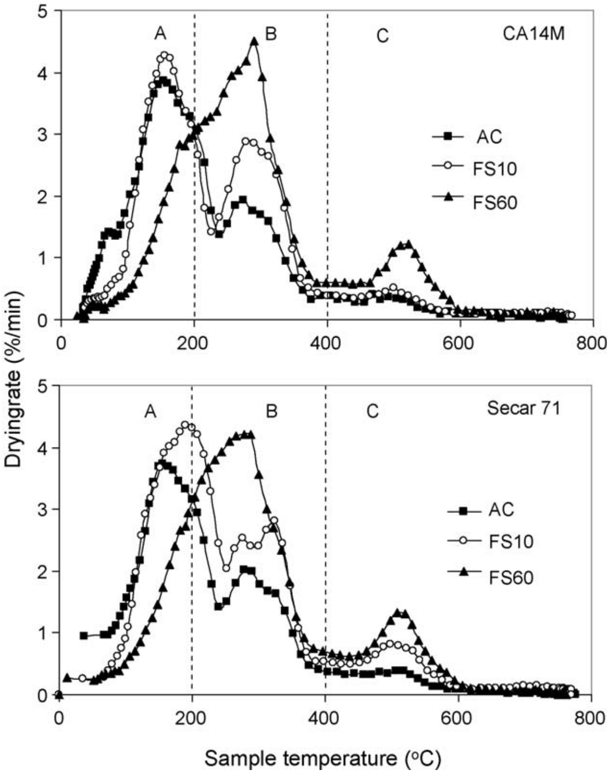

Fig. 3. Drying rate as a function of sample temperature for castables prepared with CA14M or Secar 71 containing different dispersing additives.

Therefore, the additives evaluated in this work dispersed the particles of the matrix and the binder and also influenced the type of formed hydrate which affected the castable properties such as splitting strength and porosity at intermediate temperatures.

3.2. Thermo-mechanical behavior

Because refractories usually work under compressive loads at a high temperature, it is important to evaluate their thermo-mechanical (RUL and creep) properties to predict their behavior on similar practical conditions.

The RUL test is used to evaluate the sample deformation under a load with increasing temperature. The deformation of the castable with the time under a constant load and temperature is evaluated by the creep test [18–20].

The results of deformation curves with the temperature and the time are shown in Figs. 4 and 5, respectively. The presence of additives also influenced the high temperature properties of castables.

The process of hydration, followed by dehydration, and the 30 wt% of fine alumina added by the producers in both binders generate a very reactive anhydrous material. After the CAC dehydration, lime and alumina recombine leading to CA_2, and eventually, if enough free alumina is present, to CA_6 ($CaO{\cdot}6Al_2O_3$). In the 1000–1200 °C range, CA reacts with alumina to form CA_2, whereas above 1300 °C, CA_2 reacts with alumina to form CA_6.

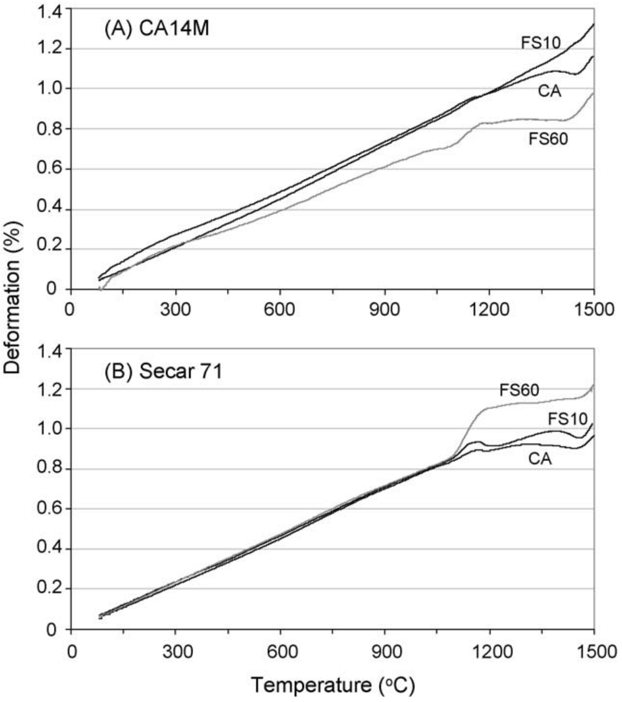

Fig. 4. Refractoriness under load for castables prepared with CA14M or Secar 71 containing different dispersing additives.

Thus, the CA_2 formation is detected by the linear expansion close to 1100 °C due to the different density among this resulting phase and the reacting ones (CA and alumina). This expansion is mainly detected for the FS60 containing castables, for both binders. The higher CA_2 formation using this additive could be associated to the calcium addition to this poly-carboxilate ether polymer for the charge density neutralization (information provided by BASF). Moreover, the reaction between CA_2 and alumina resulting CA_6 can also be detected by the expansion close to 1500 °C. The different behaviors when comparing the binders are related to the changes of the CA, $C_{12}A_7$ and CA_2 contents in the CAC.

The results presented so far indicated FS60 as the additive that mostly affected the CA_2 formation in the temperature range from 900 to 1200 °C. Nevertheless, when submitted to the creep test, it was the one that induced the highest deformation after 48 h at 1450 °C (0.2 MPa), as shown in Fig. 5.

The creep deformation of a given material depends on the load and the time at a certain temperature, and to the material

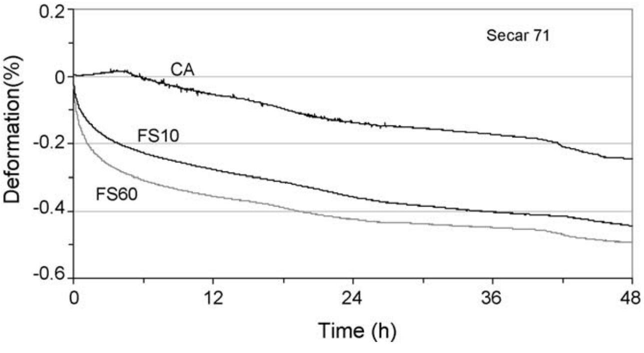

Fig. 5. Creep for castables prepared with Secar 71 containing different dispersing additives. The castables were pre-sintered at 1500 °C for 24 h and tested at 1450 °C for 48 h.

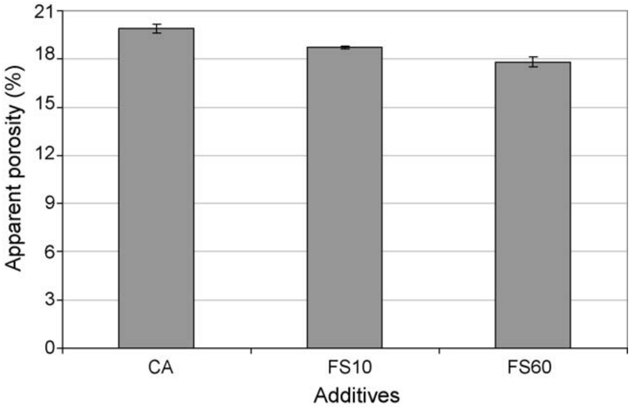

Fig. 6. Apparent porosity for castables prepared with Secar 71 containing different dispersing additives, pre-sintered at 1500 °C for 24 h.

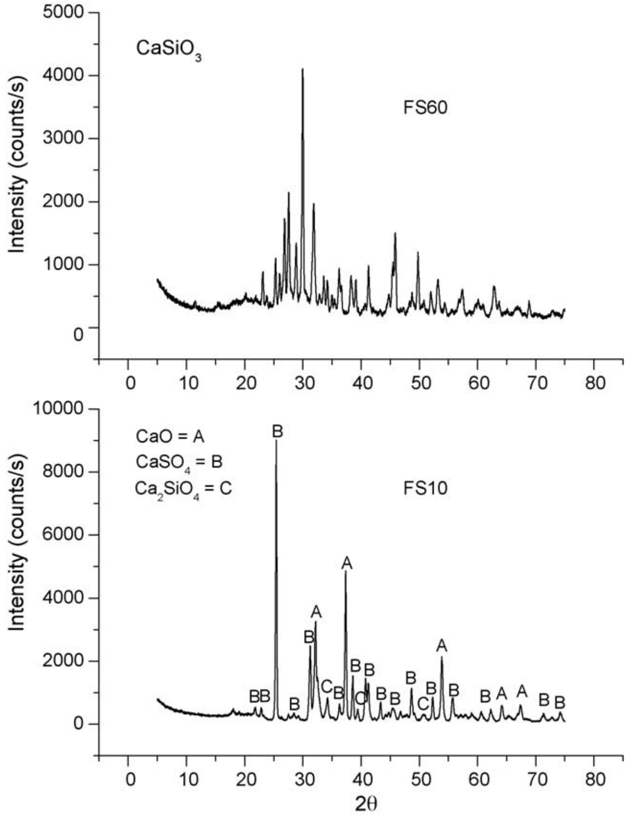

Fig. 7. X-ray profile for the FS60 and FS10 additive thermally treated at 900 °C for 5 h.

features such as grain size, porosity and glassy phase content, as well as its viscosity [18,21].

The glassy phase effect is greater when located at the grain boundary. If the wetting angle is low, the glassy phase weakens the intergrain bonds. When its concentration is high, all direct bonds between the grains are spoiled and a continuous network of low-melting phase boundaries is formed, increasing the material sensitivity to creep [21].

The porosity results for the castable containing Secar 71 just before the creep test are presented in Fig. 6. The lowest value attained for the FS60 additive, could not explain its larger sensitivity to creep.

Then, the possible formation of low-melting phases induced by additives was investigated. Additive powders were burned at 900 °C for 5 h and their residues were subjected to X-ray diffraction analysis, except for CA which burned completely. The residues content for FS10 and FS60 were 14.4 and 11.3 wt%, respectively.

The presence of the $CaSiO_3$ phase was detected for the FS60 residue (Fig. 7), indicating a possible formation of a glassy phase at high temperature ($CaO–SiO_2–Al_2O_3$ system) [22]. The formation of this glassy phase might have contributed to the castable sintering, resulting in its lower room temperature porosity, but a higher sensitivity to creep. This hypothesis was strengthened by BASF's information that the polycarboxylate ethers additives are Ca-salt polymers and that some silica is usually added to keep it as a free-flowing powder. This information also helps us understand why the drying behavior was also affected depending on the dispersing additive used.

4. Conclusions

The additives evaluated in this work not only act as dispersing particles for the matrix and binder, but also affected the type of formed hydrate which influenced castables properties at both, intermediate and high temperatures.

The additive FS60 is highlighted as an efficient dispersant, as it reduces the water consumption, inducing the C_3AH_6 formation and leaving more reactive alumina for the AH_3 formation. The decomposition of AH_3 results in a more stable hydrate (AH), which increases the splitting strength with the temperature. A characteristic drop in strength is only detected at higher temperatures due to dehydration of AH (>600 °C).

The CA_2 formation in the temperature range from 900 to 1200 °C was favored in the FS60 presence due to the calcium addition for the polymer charge density neutralization. Above 1300 °C, CA_2 reacts with alumina to form CA_6. On the other hand, the FS60 addition affected the creep behavior of the castables. The glassy phase formation due to the presence of $CaSiO_3$ resulted in a less tough structure.

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