

## Alite hydration mechanism in presence of ye'elimite related to BAY cements

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### ABSTRACT

One alternative to decrease CO<sub>2</sub> emissions from ordinary Portland Cement (OPC) production consists on the development of a new kind of eco-cements composed by less calcite demanding phases. That is the case of Belite-Ye'elimite-Ferrite (BYF) cements, which have  $\beta$ -belite as a main phase and ye'elimite as a secondary phase. But the low reactivity of  $\beta$ -belite is well known, consequently these materials develop low mechanical strengths at early/intermediate hydration ages. A possible solution proposed to solve this problem consists on the production of cements which contain belite, alite and ye'elimite together, known as Belite-Alite-Ye'elimite (BAY) cements. Consequently, the reaction of alite and ye'elimite with water would develop cements with higher mechanical strengths at early ages, while  $\beta$ -belite will contribute to later ages.

The main objective of this work is to better understand the hydration behavior of monoclinic alite in presence of ye'elimite (both stoichiometric and pseudo-cubic polymorphs), in order to be compared with a BAY eco-cement systems. The hydration of systems with selected alite/ye'elimite ratios were studied for the first 24 hours. The main techniques used for this study were in-situ synchrotron X-ray powder diffraction combined with Rietveld methodology and isothermal calorimetry.

## 1. INTRODUCTION

Calcium sulfoaluminate (CSA) cements are more environmentally friendly materials than Portland cement (PC) and are characterized by low CO<sub>2</sub> emissions, low alkalinity, good early strength, good dimensional stability, and good durability. In the past few decades, many types of CSA cements have been developed for a wide range of applications (Bullerjahn, Boehm-Courjault, Zajac, Ben Haha, & Scrivener, 2019; Chen & Juenger, 2011; Gartner, 2004; Gartner & Sui, 2017; Péra & Ambroise, 2004; Winnefeld & Barlag, 2010). Moreover some fundamental studies for calcium sulfoaluminate phase (C<sub>4</sub>A<sub>3</sub>\$ or ye'elimite), which is the dominant mineral in CSA cement clinker, have been reported (Cuesta, Álvarez-Pinazo, et al., 2014; Hargis, Telesca, & Monteiro, 2014; Jansen, Spies, Neubauer, Ectors, & Goetz-Neunhoeffler, 2017; Khessaimi et al., 2018; Puertas, Blanco-Varela, & Giménez-Molina, 1995; Zhao & Chang, 2017). However, its production is considered expensive due to the need of high purity bauxite as a rich-source of Al<sub>2</sub>O<sub>3</sub> (Angeles G. De la Torre et al., 2018; June et al., 2010; Ludwig & Zhang, 2015; Scrivener, John, & Gartner, 2018).

In recent studies, Belite-Ye'elimite-Ferrite (BYF) cements, have been thought as the potential substitute of PC at large scales (Cuberos et al., 2010; Koga, Albert, Roche, & Pereira Nogueira, 2018; X. Lu et al., 2018; Senff, Castela, Hajjaji, Hotza, & Labrincha, 2011). Nevertheless, the mechanical strength of mortar shows a slow increase owing to the low reactivity of C<sub>2</sub>S at medium hydration ages (between 7 d and 28 d). On the other hand, C<sub>3</sub>S is responsible for early and medium mechanical strengths in Portland cements. Hence, a possible approach is to bring in C<sub>3</sub>S minerals in the production of the CSA clinker. Recently, extensive researches on the formation and coexistence of C<sub>4</sub>A<sub>3</sub>\$ and C<sub>3</sub>S minerals with doping minor elements has been carried out, which could lower the viscosity and the formation temperature of liquid phase, and promote the formation of C<sub>3</sub>S (Chitvoranund, Lothenbach, Winnefeld, & Hargis, 2017; Duvallat, Zhou, Robl, & Andrews, 2014; Hu et al., 2018; Li, Xu, Wang, Tang, & Shen, 2014; Liu, Li, Qi, Liu, & Li, 2009; Londono-Zuluaga, Tobon, Aranda, Santacruz, & De la Torre, 2017; L. Lu, Chang, Cheng, Liu, & Yuan, 2005; Pérez-Bravo et al., 2014). However, its hydration mechanism and mineralogy are strictly dependent on C<sub>3</sub>S and C<sub>4</sub>A<sub>3</sub>\$ ratio. Thus, the main objective of this study is focused on the interactions that occur between C<sub>3</sub>S and different polymorph of C<sub>4</sub>A<sub>3</sub>\$ during early hydration.

## 2. MATERIALS AND METHODS

### 2.1 Materials

Alite was supplied by Sarl Mineral Research Processing (France). C<sub>4</sub>A<sub>3</sub>\$ samples were synthesized following the methodology reported by Cuesta et al (Cuesta et al., 2013; Cuesta, De la Torre, Losilla, Santacruz, & Aranda, 2014). The orthorhombic phase, oC<sub>4</sub>A<sub>3</sub>\$ was obtained by heating a stoichiometric blend of finely-ground reagent grade of Al<sub>2</sub>O<sub>3</sub> (99.997%, AlfaAesar), CaCO<sub>3</sub> (99.95%, AlfaAesar) and Gypsum (98%, Sigma-Aldrich) in an electric furnace at 1300 °C for 4 h. While the pseudo-cubic phase, cC<sub>4</sub>A<sub>3</sub>\$, was synthesized by heating the same reagent grade blend with additional Fe<sub>2</sub>O<sub>3</sub> (99.945%, AlfaAesar), SiO<sub>2</sub> (99.5%, AlfaAesar) and Na<sub>2</sub>CO<sub>3</sub> (99.999%, Sigma-Aldrich) at 1250 °C for 4h. After cooling, both samples were ground in a microdeval ball mill to pass a 75 µm (#200) mesh sieve. Finally, anhydrite (C\$) was previously prepared following the methodology reported by Londono-Zuluaga et al (Londono-Zuluaga et al., 2017) by heating a commercial micronized gypsum (C\$H<sub>2</sub>), marketed by BELITH S.P.R.L. (Belgium), at 700 °C for 60 min. Their particle size distribution were measured in an isopropyl alcohol suspension using a MALVERN MASTERSIZER S Laser Diffraction Particle Size Distribution Analyzer. The D<sub>v,50</sub> were 13.8, 9.0, 5.6, and 8.9 µm for C<sub>3</sub>S, oC<sub>4</sub>A<sub>3</sub>\$, cC<sub>4</sub>A<sub>3</sub>\$ and C\$ respectively, as can be seen in Figure 1.

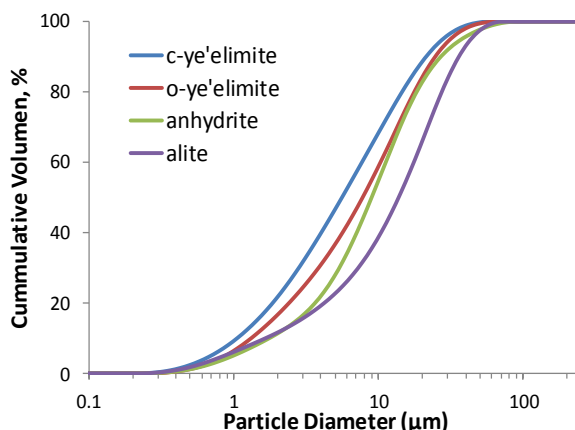


Figure 1. Particle size distribution of  $cC_4A_3$ ,  $oC_4A_3$ ,  $C\$\$  and  $C_3S$ .

The mix designs of the investigated systems are given in Table 1, including water/binder (w/b) ratio. Freshly twice-boiled deionized water was mixed with all blended samples at room temperature for 2 min to make the pastes used for Synchrotron X-ray Powder Diffraction (SXRPD) and Isothermal Calorimetry (IC) *in-situ* studies.

Table 1. Paste compositions, in weight percentages (wt%).

Mixture nomenclature	$oC_4A_3$	$cC_4A_3$	$C_3S$	$C\$\$	w/s
$o2.74\_0.59^*$	23.9	-	65.4	10.7	0.59
$o2.74\_1.19$	23.9	-	65.4	10.7	1.19
$c2.74\_0.59$	-	23.9	65.4	10.7	0.59
$c2.74\_1.19$	-	23.9	65.4	10.7	1.19

\* The nomenclature of the mixtures is referred to  $C_3S/C_4A_3=2.74$  and the second number is the w/s ratio.

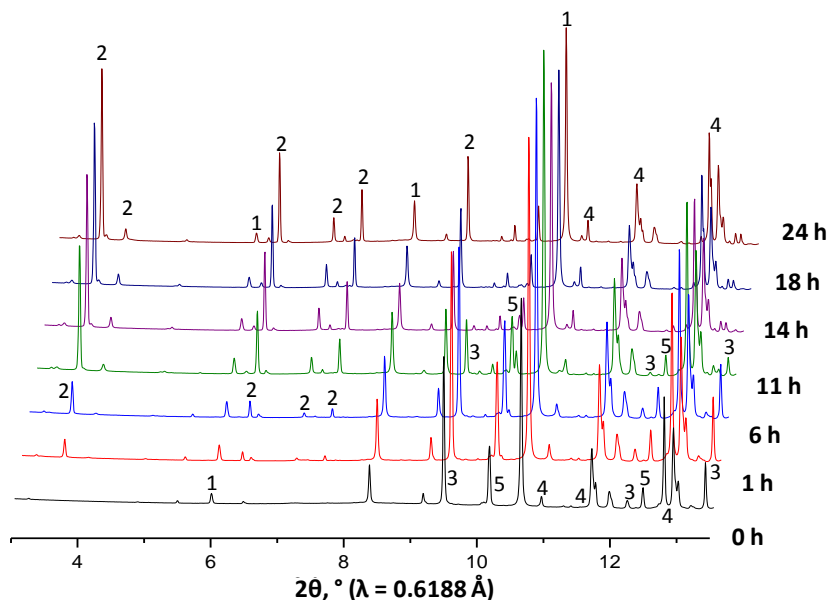
## 2.2 Methods

*In-situ* SXRPD investigations on the pastes were carried out at the Materials Science and Powder Diffraction (MSPD) beamline in the Spanish Synchrotron Light Source - ALBA at Barcelona (Spain). All patterns were collected in Debye-Scherrer (transmission) mode, utilizing a wavelength of 0.61878(3) Å selected with a double-crystal Si(111) monochromator. The diffractometer is equipped with a MYTHEN detector especially suited for time-resolved experiments. Previously, all the anhydrous mixtures were mixed with ~20 wt%  $SiO_2$  (99.5%, AlfaAesar) as an internal standard, and powder diffraction data were collected to obtain the initial phase assemblage ( $t_0$ ), including amorphous and non-crystalline (ACn) content (A.G. De la Torre, Bruque, & Aranda, 2001). Pastes were ex-situ prepared (mixing with fresh twice-boiled water by hand for 2 min) and immediately poured into glass capillaries of 0.7 mm of diameter with a syringe. Afterward, they were sealed with grease to avoid any water loss. The capillaries were rotated during data collection to improve diffracting particle statistics. The data acquisition time was ~6 min per pattern to attain very good signal-to-noise ratio over the angular range 2–40° (2θ), with the aim to follow the hydration products formation.

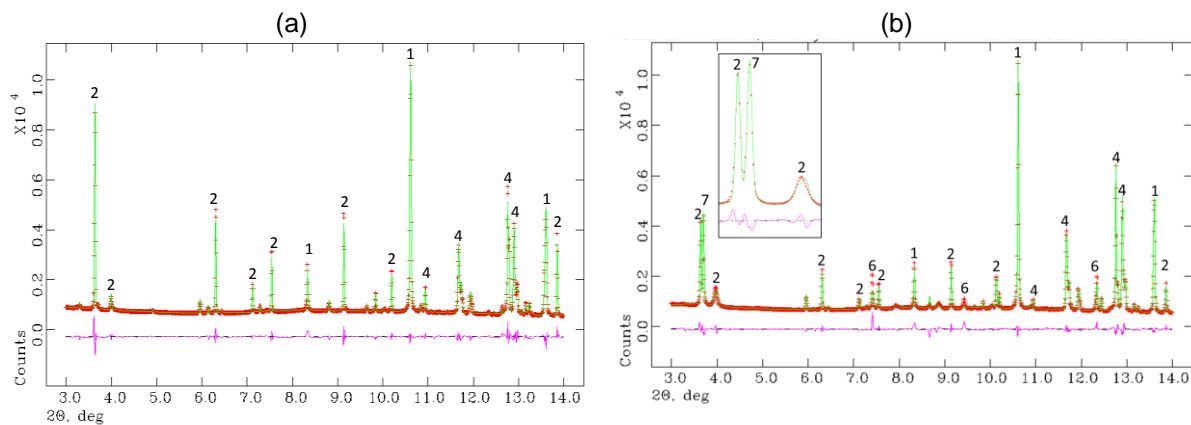
For *in-situ* IC, two grams of mixture with its corresponding mix water/solid ratio (Table 1) were prepared by internal mixing for 2 min using an Admix automatic device. The pastes were tested for heat evolution using a Thermometric TAM Air® 8 (TA Instruments) during 72 hours at 25 °C. The heat flow of three separate runs for each paste was recorded following ASTM C1679 – 13 methodology. No significant differences were observed between the three runs and the arithmetic mean was used.

### 3. RESULTS AND DISCUSSION

The hydration mechanism of mixtures of pure phases was studied during the first 24 hours. The effect of w/s ratio and ye'elimite polymorphism jointly with anhydrite on the alite hydration were studied. Figure 2 depicts the raw SXRPD patterns at different times of hydration of c2.74\_1.19 mixtures. Figure 3 shows Rietveld plots of c1.37\_1.32 and o1.37\_1.32 mixtures after 24 hours of hydration, as representative examples.



**Figure 2.** In-situ SXRPD hydration evolution of c2.74\_1.19. 1 (Quartz as internal standard), 2 (ettringite), 3 (pseudocubic ye'elimite), 4 (alite) and 5 (anhydrite).



**Figure 3.** Rietveld plots of (a) c2.74\_1.19 and (b) o2.74\_1.19 at 24 hours of hydration. 1 (Quartz as internal standard), 2 (ettringite), 4 (alite), 6 (stratlingite) and 7 (monosulfoaluminate).

Table 2, Table 3, Table 4 and Table 5 give RQPA including the ACn and free water (FW) contents determined by internal standard methodology. Figure 4 provides the full phase content evolution and heat flow curves of c2.74 and o2.74 at different w/b ratios. From all these Figures and Tables, two phenomena can be clearly described. On the one hand, the orthorhombic ye'elimite mixture has totally reacted at 5 hours of hydration independently of w/b ratio. While in pastes prepared with the pseudocubic ye'elimite has only partially reacted during the same time. This is in agreement with Cuesta et al (Cuesta, Álvarez-Pinazo, et al., 2014) and Jansen et al (Jansen et al., 2017) where orthorhombic ye'elimite with gypsum or anhydrite reacted at a higher speed than cubic one. On the other hand, alite did not start to react until ye'elimite was completely consumed, independently of the polymorphism. The main crystalline hydration products of these systems were AFt, monosulfoaluminate and stratlingite.

Moreover, the phase assemblage was not strongly affected by water-to-solid ratio or polymorphism of ye'elimite.

**Table 2.** RQPA results (wt%) of the in-situ hydration study of c2.74\_0.59.

Phases						
Age (h)	C <sub>4</sub> A <sub>3</sub> \$	C\$	AFt	C <sub>2</sub> ASH <sub>8</sub>	C <sub>3</sub> S	ACn+FW
0	15.8	6.8	-	-	38.1	39.3
1	13.9	6.3	2.2	-	38.1	39.5
2	13.3	6.2	2.7	-	38.1	39.6
3	12.4	6.2	3.4	-	38.1	39.9
4	11.1	6.0	4.5	-	38.0	40.3
6	8.4	5.4	7.8	0.4	38.0	40.1
8	4.2	3.9	14.1	0.4	38.0	39.3
10	1.7	2.4	18.4	0.5	38.0	39.1
14	1.0	2.0	20.6	0.6	37.7	38.1
18	0.1	1.0	24.1	0.7	35.8	38.3
24	-	0.2	26.8	1.1	33.6	38.3
47	-	-	27.8	1.8	31.3	38.9

**Table 3.** RQPA results (wt%) of the in-situ hydration study of c2.74\_1.19.

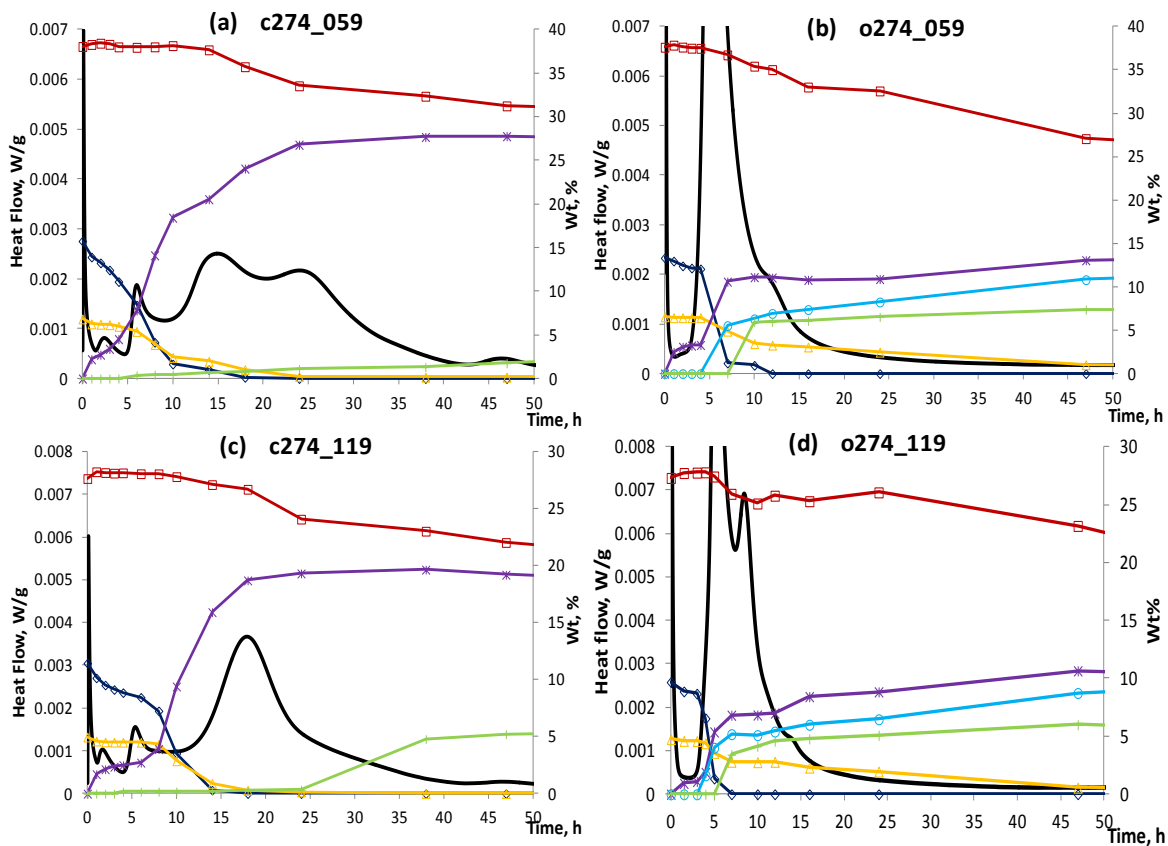
Phases							
Age (h)	C <sub>4</sub> A <sub>3</sub> \$	C\$	AFt	AFm	C <sub>2</sub> ASH <sub>8</sub>	C <sub>3</sub> S	ACn+FW
0	11.4	5.0	-	-	-	27.6	55.9
1	10.2	4.6	1.7	-	-	27.6	55.9
2	9.5	4.5	2.1	-	-	27.6	56.2
3	9.1	4.5	2.4	-	-	27.6	56.4
4	8.9	4.5	2.7	-	-	27.6	56.3
6	8.4	4.5	2.9	-	-	27.6	56.6
8	7.3	4.3	4.1	-	-	27.6	56.8
10	3.5	2.9	9.6	-	-	27.6	56.4
14	0.3	0.9	15.9	0.2	0.2	27.1	55.3
18	-	0.3	18.7	0.3	0.2	26.7	53.8
24	-	0.2	19.7	0.3	0.3	24.5	54.9
47	-	-	19.2	0.3	5.2	21.9	52.9

**Table 4.** RQPA results (wt%) of the in-situ hydration study of o2.74\_0.59.

Phases Age (h)	C <sub>4</sub> A <sub>3</sub> \$	C\$	AFt	AFm	C <sub>2</sub> ASH <sub>8</sub>	C <sub>3</sub> S	ACn+FW
0	13.3	6.6	-	-	-	37.6	42.5
1	13.0	6.5	2.5	-	-	37.6	40.5
2	12.4	6.5	3.1	-	-	37.6	40.4
3	12.2	6.4	3.2	-	-	37.5	40.6
4	12.1	6.4	3.4	-	-	37.5	40.7
7	1.2	4.9	10.6	5.5	-	36.7	41.0
10	0.9	3.3	10.6	6.0	5.6	33.8	40.5
12	-	3.2	10.7	6.7	5.8	33.8	40.2
16	-	3.1	10.8	7.4	6.1	33.0	39.6
24	-	2.5	10.9	8.3	6.5	32.6	39.2
47	-	1.0	13.1	10.8	7.4	27.1	40.6

**Table 5.** RQPA results (wt%) of the in-situ hydration study of o2.74\_1.19.

Phases Age (h)	C <sub>4</sub> A <sub>3</sub> \$	C\$	AFt	AFm	C <sub>2</sub> ASH <sub>8</sub>	C <sub>3</sub> S	ACn+FW
0	9.7	4.8	-	-	-	27.3	58.2
1.5	8.9	4.6	0.9	-	-	27.3	58.2
3	8.7	4.6	1.1	-	-	27.3	58.3
4	6.6	4.4	1.9	1.6	-	27.3	58.2
5	1.4	3.6	5.4	4.0	-	27.3	58.3
7	-	2.8	6.8	5.2	3.4	26.0	55.8
10	-	2.8	6.8	5.2	4.1	25.1	55.9
12	-	2.8	7.0	5.4	4.6	25.1	55.1
16	-	2.3	8.4	6.0	4.8	25.1	53.3
24	-	2.0	8.8	6.5	5.1	24.3	52.3
47	-	0.6	11.7	8.7	8.0	17.2	53.8



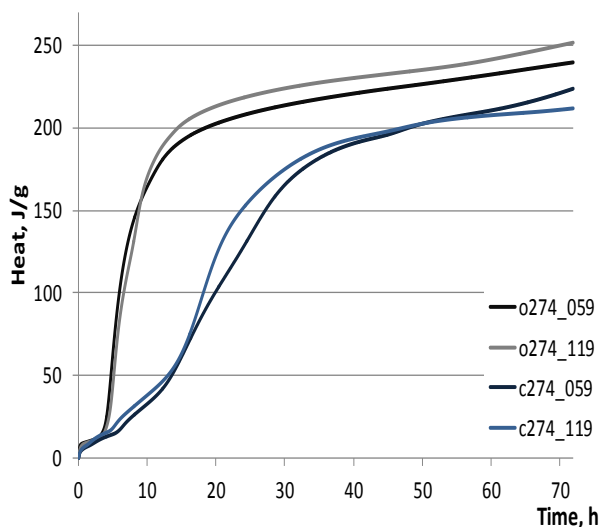
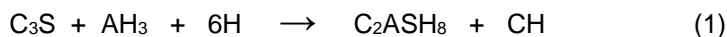
**Figure 4.** Heat flow and mineralogical in-situ hydration evolution of (a and c) c2.74 and (b and d) o2.74. In each plot, the left-hand vertical axis shows the rate of heat release, and the right-hand vertical axis shows the weight percent amount of the phases. Black line: Heat flow, blue diamond:  $C_4A_3\$$ , yellow triangle:  $C\$$ , red square:  $C_3S$ , purple asterisk: ettringite, light blue circle: monosulfoaluminate and green circle:  $C_2ASH_8$ .

The isothermal calorimetric curves, Figure 4 (black line), present a strong peak within the first 25 minutes (0.4 hours) since mixing, known as the induction period. This peak is clearly associated with the dissolution (heat of wetting of the cement), and hydration of ye'elimite to form ettringite (purple asterisk) (Cuesta, Álvarez-Pinazo, et al., 2014; Jansen et al., 2017). The hydration of orthorhombic and pseudo-cubic ye'elimite was published elsewhere (Cuesta, Álvarez-Pinazo, et al., 2014) and the total heat evolved after 7 days was ~570 and ~550 J/g, respectively. The higher amount of heat released by the samples with the orthorhombic polymorph at 25 minutes corroborates the quicker reactivity of that phase when compared with the samples pseudo-cubic polymorph in spite of the amount of water. At 15 h, when both ye'elimites have completely reacted, the total heat released for c2.74 and o2.74 were ~70 and ~200 J/g respectively, Figure 5. While independently of the w/b used, the cumulative heat displayed after 48 hours, were about ~200 and ~230 J/g by c2.74 and o2.74 respectively.

During the induction peak a small amount of AFt crystallized without the dissolution of anhydrite, as previously reported (Cuesta, Álvarez-Pinazo, et al., 2014; Jansen et al., 2017; Pelletier-Chaignat et al., 2011). Monosulfoaluminate was not detected in the pseudo-cubic ye'elimite mixtures. However, in o2.74 mixtures, the precipitation of AFt increased considerably jointly with monosulfoaluminate co-precipitation, Figure 4 (Table 2, Table 3, Table 4 and Table 5). The raise of water/binder ratio from 0.59 to 1.19 did not affected the mixtures with orthorhombic ye'elimite. However, in the mixture with the pseudo-cubic form, both alite dissolution and precipitation of stratlingite are enhanced, were larger amounts of stratlingite were precipitated. This may be justified by the availability of water.

It is noticeable that crystalline portlandite and gibbsite were not detected in the first 48 h of hydration in any system (c2.74 and o2.74). The lack of precipitation of CH was expected and it is associated to the enhanced hydration of  $C_4A_3\$$  that favors the early-ettringite formation (Hargis, Kirchheim, Monteiro, & Gartner, 2013; Péra & Ambroise, 2004; Winnefeld & Barlag, 2009). Moreover, the lack of crystalline  $AH_3$

is associated with the formation of stratlingite (1) (Trauchessec, Mechling, Lecomte, Roux, & Le Rolland, 2015; Winnefeld & Lothenbach, 2016).



**Figure 5.** Total heat (cumulative) release curves of c2.74 and o2.74.

The pseudo-cubic ye'elimitite mixtures produced higher amounts of ettringite than orthorhombic ye'elimitite samples, under similar hydrating conditions. This is due to orthorhombic ye'elimitite presents a higher kinetic of hydration than pseudo-cubic ye'elimitite, consequently when orthorhombic ye'elimitite reacts with the anhydrite, which is slowly dissolved, favors the jointly formation of AFt and monosulfoaluminate.

#### 4. CONCLUSIONS

C<sub>3</sub>S hydration was influenced by the kinetic of ye'elimitite dissolution and reaction, and it began to react after ye'elimitite reacted completely. In mixtures with orthorhombic and pseudo-cubic ye'elimitite, alite depletion started after 10 and 20 hours respectively, since orthorhombic ye'elimitite reacts faster than the pseudo-cubic polymorph. The main products from alite reaction were stratlingite and C-S-H gel. The formation of calcium hydroxide (as portlandite or amorphous) was not detected, as expected. The increase of w/s ratio also affected the kinetic of hydration of alite. The mixtures with orthorhombic ye'elimitite and higher w/s ratio presented, after 48 hours, a higher hydration degree. Consequently, higher amounts of C-S-H (as amorphous phase) and stratlingite were obtained. The mixtures with pseudo-cubic ye'elimitite, even after 48 hours of hydration, presented high amounts of non-reacted C<sub>3</sub>S. Moreover, the main hydration product from the reaction of alite is stratlingite, while in the orthorhombic ye'elimitite mixtures, the main phase is also amorphous C-S-H gel.

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