

# Enhanced Durability and Reactivity of a Portland Cement with Low Phosphogypsum Content

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**ABSTRACT:** *Minimizing the environmental impact and optimizing the performance of the cement manufactured is the magic formula sought to define the glorious trio of performance, cost, and environmental impact. The use of industrial by-products such as phosphogypsum can help to achieve the sustainability of the cement industry. An industrial cement series was used to investigate the effect of low phosphogypsum content on Portland cement reactivity with percentages ranging from 1 to 4%. The raw materials, clinker, gypsum, and phosphogypsum, were characterized by X-ray fluorescence, and the clinker mineralogy was determined by X-ray powder diffraction coupled with the Rietveld method analysis. The reactivity of cement was followed by isothermal calorimetry, compressive strength, conductivity measurement, and thermal analysis. The results revealed that there is an influence of phosphogypsum in this range of composition on the early cement reactivity. However, it may enhance the long-term cement reactivity and the concrete performance. It has been found that the isothermal microcalorimetry analysis method, is able to detect the formation of an exothermic component due to the precipitation of phosphorus in the form of apatite even at very low phosphogypsum integration rates.*

**KEYWORDS:** *Industrial clinker, Mineralogy, Phosphogypsum, Reactivity.*

## INTRODUCTION

The cement sector is involved in the current context of environmental pollution, including global warming and its pathogenic effects on the environment[1, 2]. In fact, cement generates significant amounts of CO<sub>2</sub> during its production, more precisely during the clinker, which is the constituent material of cement. The latter involves the decarbonization of raw limestone, CaCO<sub>3</sub>, which is transformed into CaO, releasing CO<sub>2</sub> greenhouse gas

that causes climate change. For every ton of Portland cement, roughly 800 kg of CO<sub>2</sub> is emitted[3, 4]. In addition, another share of CO<sub>2</sub> emissions comes from the use of fuels for energy production, which intensifies air pollution.

This has generated a growing awareness around the world with the aim of having a stable quality cement product that is less harmful to the environment. So, in order to fight against the environmental pollution generated

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Table 1: Chemical composition of the raw materials

	Clinker	Gypsum	Phophogypsum	Clinker Phase minéralogique	
LOI	0.67			Alite	63.9
CaO	65.07	32.58	31.14	Bélite	23.6
SiO <sub>2</sub>	21.67	3.23	2.21	Aluminate cubique	1.3
Al <sub>2</sub> O <sub>3</sub>	4.54	4.64	0.36	Aluminate orthorhombique	0.8
Fe <sub>2</sub> O <sub>3</sub>	3.15	0.49	0.40	Ferrite	9.2
MgO	1.55	3.49	1.19	Lime	0.3
SO <sub>3</sub>	1.84	41.86	37.50	Périclase	0.1
Na <sub>2</sub> O	0.11			Hemihydrate	0.2
K <sub>2</sub> O	0.5	0.32	0.09	Calcite	0.4
P <sub>2</sub> O <sub>5</sub>	0.90	0.05	-	Syngenite	0.2
				Arcanite	0.0
				Total	100

by industrial development and its circumstances, the cement sector is concerned. One of several alternatives is the integration or the use of recycled solid waste, agricultural waste, and industrial by-products such as phosphogypsum manufacture of cement. Actually phosphogypsum management is one of the most serious problems currently facing the phosphate industry [5]. Only 15% of worldwide production is recycled, while 85% is stored in the vicinity of factories in coastal regions. Dumping of phosphogypsum into open land could have environmental and health concerns. This material is dominated by calcium sulphate dehydrate however it contains approximately 5-6% of impurities including heavy metals, fluoride and radionuclides [6]. These toxic substances can be transported by wind over long distances which could contaminate soil and or groundwater.

This attractive potential of phosphogypsum makes it at present a prodigious curiosity in the cement industry as an alternative raw material for many applications. It can be used as a replacement for gypsum with a certain percentage, which could give positive outcomes on the economic and environmental level. A large part of the literature is available on the use of phosphogypsum in the manufacture of cement instead of natural gypsum as a setting retarder [7-14]. On the one hand, phosphogypsum has a chemical composition similar to that of natural gypsum; on the other hand, it represents a significant industrial waste which produces harmful effects for the environment from which it must be recycled.

In fact, phosphogypsum is an acidic by-product from the phosphate fertilizer industry. It is mainly calcium sulfate dihydrate, but it contains elevated levels of impurities which originate primarily from the phosphate source. It is a powdery

material that has little or no plasticity [7]. Nevertheless, previous studies found that the substitution of natural gypsum by raw phosphogypsum residue in the preparation of industrial cement, causes a considerable delay in the cement hardening due to the presence of minor elements which may modify the kinetics of the cement hydration reactions and introduce some modification [15-18]. The nature of these penalizing elements depends closely on the basic product, which is phosphate, its composition and its quality. These disadvantages associated with the presence of impurities in phosphogypsum can be overcome when, instead of adding it directly to portland cement as a set-retarding additive, it had to be treated. Which the hydration process was significantly accelerated and the setting time was shortened [19].

This paper investigates the effect of a small amount addition of untreated phosphogypsum on the Portland cement reactivity and its hydration kinetics, through a comparative study between cements made from the same clinker specific to the Bizerte cement plant with 1-2% addition of gypsum and phosphogypsum as a setting retarder. The chemical compositions of the raw materials are determined using X-ray fluorescence, the mineralogical composition of clinker was checked by X-ray diffraction coupled with Reitveld analysis [20] and the monitoring of the cement reactivity was followed by isothermal calorimetry, compressive strength, conductivity measurement, and thermal analysis.

## EXPERIMENTAL SECTION

### Sample Preparation

Some properties and major constituents of the Portland clinker, gypsum, and phosphogypsum used in the study are reported in Table 1. The chemical composition was determined by X-ray fluorescence using the ARL ADVANT X

**Table 2: Raw materials composition**

Raw material	integration rate of setting retarder (%)	Clinker weight	Setting retarder weight
Gypsum	0.0	5000	0
	1.0	4950	50
	2.0	4900	100
	3.1	4850	150
	4.2	4800	200
	5.3	4750	250
	6.4	4700	300
	7.5	4650	350
Phosphogypsum	0.5	4975	25
	1.0	4950	50
	1.5	4925	75
	2.0	4900	100
	2.6	4875	125
	3.1	4850	150
	4.2	4800	200

series spectrometer. The measurement was made on pellets using the fusion technique. This technique consists of heating at a high temperature (1100 °C–1200 °C) a mixture of sample and lithium tetraborate, a melting product. The dilution and cooling have to be chosen in a certain way in order to get a glass containing single phases. This is the best technique to eliminate the effects of particle size and mineralogy.

After the chemical analysis of the raw materials, a quartering was made by introducing them into a mixer for 5 minutes in order to ensure a homogeneous, simple and harmonic composition. Finally the specimen are prepared with well-defined quantities as indicated in table 2, and then the mixtures are introduced into a mini ball mill for one hour to obtain the cement samples.

### **Compressive strength**

To perform durability test of cement samples, prismatic specimens having dimensions of 40 mm \*40 mm\*160 mm were used to measure the strength resistance[21]. The mortars were prepared by mixing 450 g cement, 1350 g normalized sand and 225 g distilled water with w/c equal to 0.5, then stored in a chamber especially designed to maintain a constant temperature at 22° C and humidity equal to 98%.

### **Isothermal calorimetry**

The reactivity of the cement was estimated by differential isothermal calorimetry. The isothermal calorimeter was programmed to maintain a temperature of 23°C in its cells.

After thermal equilibration of the cells, the cement pastes were prepared in plastic ampoules by mixing 1 g of cement and water with a water to cement ratio of 0.5. Mixing was performed for two minutes with a Dremel equipped with a spatula. The ampoules were then introduced into the measurement cells of the calorimeter and the heat released was recorded for up to 24 h. For each sample, the measured heat flow was normalized relative to the cement mass.

### **Conductivity measurement**

The evolution of the ionic concentration of the cement constituents during the hydration phase was carried out using the solution conductivity measurement. All the experiments were done on a conductimeter using a thermostated cell at 25°C, pre-calibrated with a KCl solution (0.1M,  $\lambda=12.88(\text{mS}/\text{Cm})$ ).

For each measure 100 mL of deionized water is introduced into the cell which maintained under continuous stirring, once the baseline is stabilized, 5 g of specimen were added. The experiments are carried out in an environment diluted at a water - Portland cement ratio = 20. This system allows to a continuous follow of the conductivity evolution of as a function of time. Using this method we can determine the portlandite precipitation time, this is characterized by the maximum of the conductivity curve.

### **Thermal Analysis**

In order to study the evolution of cement hydration products by the thermal analysis we proceeded to the following steps: We mixed the cements and the distilled water using a water to cement ratio (w/c) equal to 0.4 using an automatic mixer during few minutes to obtain a paste. Then the paste sample were allowed to hydrate in sample holders in a desiccator at 20° C saturated with nitrogen and water vapor. After the cubes are broken and crushed at the desired times. The hydration of these powders is stopped by rinsing with anhydrous ethanol and vacuum filtration for 30 min. Finally, the sample is dried at 50° C, then ground and sieved to 80  $\mu\text{m}$ .

## **RESULTS AND DISCUSSION**

### **Compressive strength**

The compressive strength measurement is considered as a scale of appreciation which allows checking on the cement quality. The results of the compressive strength test of the prepared samples are summarized in Figs. 1 and 2.

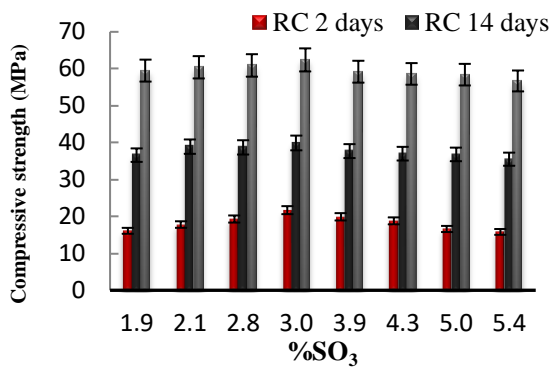


Fig. 1: Evolution of compressive strength made from clinker and gypsum as a cement retarder

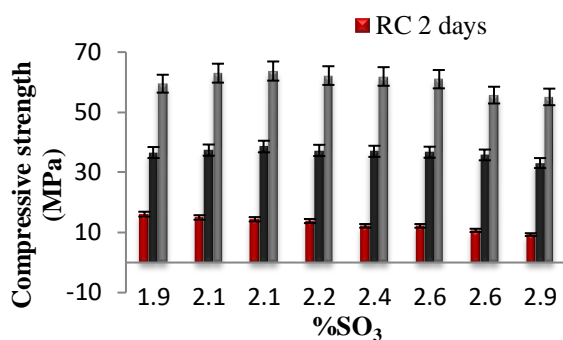


Fig. 2: Evolution of compressive strength made from clinker and phosphogypsum as a cement retarder

It is clear that the cement mortars present the same tendency for evolution. As seen in fig. 1, the resistance values increase with the rate of the integrated gypsum to reach an optimum for 3% of SO<sub>3</sub>. Beyond this optimum, the resistance decreases. Thus, the SO<sub>3</sub> rate for having the best resistance is 3%.

In fact, the anhydrous constituents of the cement on contact with water give rise to hydrated calcium silicates and aluminates, which are practically insoluble in water. Aluminate phase will be surrounded by a protective layer of Ettringite in the presence of gypsum. After consumption of the whole setting retarder and the saturation of the aqueous phase, hydrated calcium aluminates are formed. The development of crystals formed by the anhydrous constituents of cement over time explains the increase in strength.

When the gypsum acts with all of the C<sub>3</sub>A grains, we will thus have a maximum amount of Ettringite and thus, CAH allowing us to have the greatest resistance. Exceeding this rate generates an excess which weakens

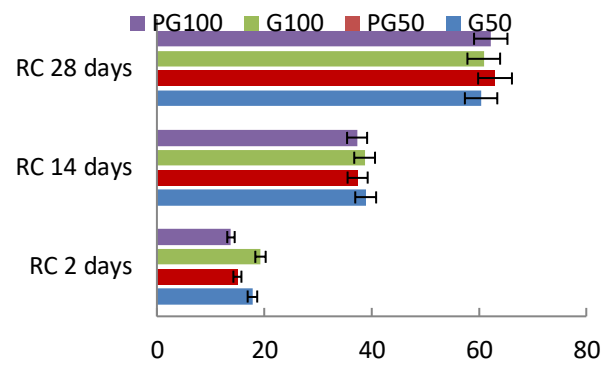


Fig. 3: Evolution of Compressive strength of cements prepared from the industrial clinker with 1%-2% of gypsum and phosphogypsum

the mechanical properties of the cement since the gypsum have neither mechanical resistance nor hydraulic properties. This explains the decrease in strength after a gypsum addition rate of 3%.

Fig. 2 provides the compressive strength variation as a function of hydration time for cement made with phosphogypsum as a setting retarder. It can be noticed that the compressive strength after 2 days hydration decrease accordingly with the rate of the integrated phosphogypsum. For the compressive strength after 7 and 28 days hydration the resistance values increase with the rate of the integrated phosphogypsum to reach an optimum for 2% of SO<sub>3</sub>. Beyond this optimum, the resistance decreases. Thus, the SO<sub>3</sub> rate for having the best resistance is 2%. The difference in behavior observed at short term hydration for these samples is attributed to the penalizing elements found in the composition of phosphogypsum.

A comparative study was carried out to estimate the effect of phosphogypsum at low integration rates as a setting retarder on Portland cement reactivity. Cements with respective additions of 1–2% of gypsum and phosphogypsum are selected. Cements with 1% addition are labeled G50 and PG50, while those with 2% addition are labeled G100 and PG100. The compressive strength evolution of the selected samples is presented in Fig. 3. Generally, the long-term mechanical properties are equivalent, but the short-term time is limited for the cements with phosphogypsum addition.

At the ages of 2 and 14 days after hydration, the samples with gypsum presented higher compressive strength than the samples with phosphogypsum. For filed condition,

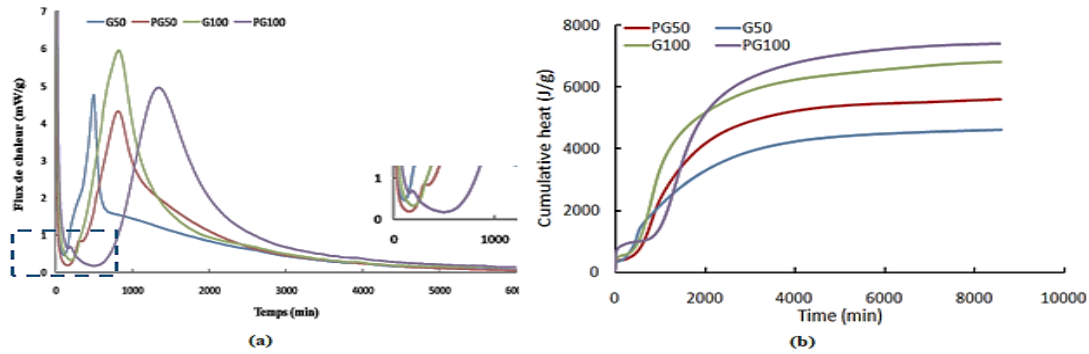


Fig. 4: Heat evolution rate curve recorded during the cement hydration(a) and Cumulative heat during the cement hydration(b)

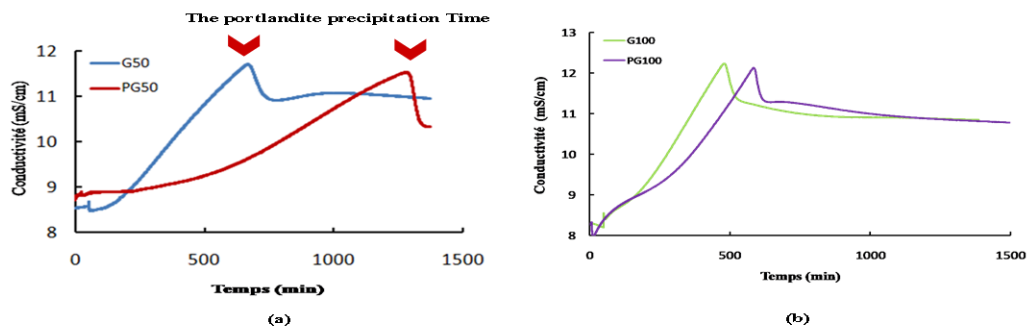


Fig. 5: Conductivity measurement of cements prepared from the industrial clinker with 1%(a)-2%(b) gypsum and phosphogypsum

A decreasing trend in strength with phosphogypsum addition was observed at this age. But at 28 days, these materials gave higher concrete strength than the control sample. Compared to the gypsum, phosphogypsum's presence in the cement increased the long-term mechanical strength rapidly up to 4-2% for corresponding additions 1-2% of the setting retarder.

#### Isothermal calorimetry

Heat evolution rate curves during the hydration of cements prepared from the clinker by adding 1-2% gypsum and phosphogypsum are plotted on Fig. 4. All the heat development curves of prepared cements show the typical five stages of the hydration reaction, which begin with an initial reaction, then a slow reaction period, an accelerating period, a decelerating period, and a reaction period slow continues. Usually, heat of hydration curves are characterized by the presence of two peaks. The first is attributed to the rapid dissolution of alite and the precipitation of hydrated calcium silicate and portlandite. The second peak is attributed to the hydration of the aluminate phase in AFm once the gypsum is consumed

to form ettringite [22, 23]. The latter is not visible, probably because it occurs simultaneously with the first one.

It is noticeable that there is a clear change in the heat evolution curves observed with the use of phosphogypsum as a setting retarder. A slight delay of the main hydration peak is determined. In addition, the presence of an identifiable exothermic component is noted with the presence of phosphogypsum even with an integration rate of 1%. These observations range in the direction of a slower precipitation of calcium phosphate [24]. Also, it can be reported that the samples which the setting retarder is phosphogypsum is characterized by a longer dormant period than those of gypsum.

Fig. 5 presents the total cumulative heat  $Q$  (J/g) which presented the enthalpy of each cement hydration process for cement pastes. The results show that cement with phosphogypsum addition presented the highest cumulative heat. We conclude that in this range, the increase of phosphogypsum addition increases always the cumulative heat and then the rate of hydration.

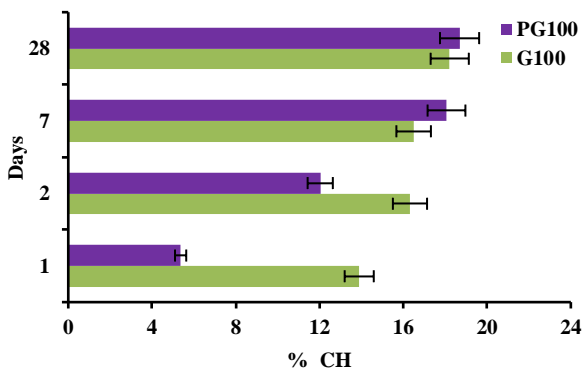


Fig. 6: Portlandite Content as a function of curing time for cement with 2% of setting retarder addition

### Conductivity measurement

As clearly seen in Fig. 6, the conductivity curves recorded during the hydration of the clinker samples show the typical three stages of the hydration reaction. Firstly, an instantaneous increase in the conductivity is observed due to the dissolution of the cement constituents ( $\text{Ca}^{2+}$ ,  $\text{OH}^-$ ...), then the second step occurs when the curve shape changes, reflecting the germination of the first hydrates of CSH that appear on the grains' surface. From this moment, the conductivity increases slowly and then accelerates. This phenomenon is due to the free-radical growth of the hydrates, when all the grains surfaces are covered, the kinetics of hydration becomes governed by the diffusion of the species through the hydrates layer. A competition between the dissolution of the anhydrous grains and the precipitation of the hydrates leads to a progressive decrease of the conductivity.

First, there is an instantaneous increase in conductivity, which corresponds to the step of dissolving the constituents of the cement. The second step takes place when the conductivity curve bends. It represents the germination of the first hydrates (CSH and ettringite). The CSH will appear punctually on the surface of the grains. From this point on, the conductivity increases very slowly and then accelerates.

This phenomenon is due to the free growth of hydrates which is limited only by the number of germs having precipitated. When the entire grain surface is covered, the hydration kinetics become governed by the diffusion of species through the layer hydrates. When the oversaturation with respect to portlandite is reached, portlandite precipitates and the conductivity drops. A competition between the dissolution of anhydrous grains and precipitation of hydrates causes a gradual decrease in conductivity. From the conductivity curves we can

determine the time necessary to reach the portlandite precipitation which corresponds to the maximum of the conductimetric curve. The time required to reach maximum conductivity is strongly affected by the presence of phosphogypsum.

### Thermal analysis

In order to further this study, a kinetic monitoring is undertaken. The reaction progress of the various hydrated cement pastes were estimated from their portlandite rate. The quantification of the portlandite content is carried out by thermogravimetric analysis.

As known the portlandite phase decompose in the temperature ranges:  $420^\circ\text{C}$  to  $550^\circ\text{C}$  [25, 26]. Therefore, it is possible to quantify this phase based on the decomposition reaction:



$$\% \text{Ca}(\text{OH})_2 = \frac{m_{420^\circ\text{C}} - m_{550^\circ\text{C}}}{m_{550^\circ\text{C}}} \times \frac{M_{\text{Ca}(\text{OH})_2}}{M_{\text{H}_2\text{O}}}$$

$m_{420^\circ\text{C}}$ : mass loss at  $420^\circ\text{C}$

$m_{550^\circ\text{C}}$ : mass loss at  $550^\circ\text{C}$

$M_{\text{Ca}(\text{OH})_2}$ : molar mass of portlandite phase

$M_{\text{H}_2\text{O}}$ : molar mass of water

Fig. 7 presents the evolution of the mass percentage of portlandite as a function of curing time. We note that the portlandite content increases continuously during the hydration. As known, the amount of portlandite in the cement pastes could be a first prediction of Portland cement durability at early age hydration. PG100 shows the lowest portlandite content comparing to the cement made from gypsum. But at the age of 28 days it giving better results.

In fact, the presence of impurities such as free phosphoric acid, phosphates, fluorides and organic matter in phosphogypsum inhibits the formation of portlandite from the first hours of hydration. The inhibitory effect is strengthened with increasing phosphogypsum mass concentration.

Thermogravimetry validates the increase in inhibition caused by the phosphogypsum presence, this is confirmed by the phenomenon of delayed portlandite precipitation observed in conductometry and the increase in the dormant period reflected in isothermal calorimetry.

### Discussion

During the hydration, the main mineralogical phases of cement, alite, belite, aluminate and ferrite, participate

independently with different kinetics in order to have the hydration products responsible for the development of the desired properties of cement. However, the presence of minor elements coming from the phosphogypsum affects the cement reactivity. This shows a dependency relationship between the mineralogy of the different cement phases and the concrete behavior. The different experiments show a direct correlation between the reactivity of the cements and the phosphogypsum content. Taking in count the portlandite precipitation time determined by the solution conductivity and the results from isothermal calorimetry measurements, there is a possible effect of very low contents of phosphogypsum above 1% on the cement early age reactivity, it plays the role of a hydration decelerator. One can note a clear influence of phosphogypsum impurities with regard to the intensity and position of the main peaks, duration of induction period, the accumulated heat and apparition of duplex in some cases. On the other hand, the phosphogypsum contributes to better long term compressive strength. This is also seen through the thermal analysis which proves that cements with phosphogypsum as a retarder form more portlandite. Its growth in the system, that fills the hollow pores in the mortar structures and makes it more denser, which increases the long term mechanical strength rapidly.

## CONCLUSIONS

The presence of impurities in phosphogypsum composition disrupts the balance of formation of cement hydration products by modifying their speed and leads to limiting the development of the mechanical properties of concrete at an early age. This limits the use of raw phosphogypsum in the manufacture of cements. But later it gives better results than gypsum.

Thanks to the isothermal microcalorimetry analysis method, we were able to detect the formation of an exothermic component due to the precipitation of phosphorus in the form of apatite, which explains the delay observed by the conductivity measurement of the formation of portlandite.

Phosphogypsum has a specific advantage in helping to solve present and future problems related to the management of natural resources. Gypsum and environmental concern is an important issue in this regard. A more detailed research and development work might

help to ensure better use of this material in construction industry and go deep into its mechanism during hydration.

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Throughout this paper, the following notation for the cement:

LOI = Loss in ignition, PG=phosphogypsum, G=gypsum CAH=hydrated calcium aluminates, CSH=hydrated calcium silicate, Ca (OH)<sub>2</sub>=portlandite, Na<sub>2</sub>SiF<sub>6</sub>=sodium fluorosilicate, CAH=hydrated calcium aluminates

C3A = Aluminuate phase