

# CALCINED FINE DAM SEDIMENTS AS INNOVATIVE BINDERS: OPTIMIZATION OF THE THERMAL TREATMENT AND POZZOLANIC ACTIVITY ASSESSMENT

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## ABSTRACT

Sedimentation processes occur in hydroelectric dams. The accumulated sediments have to be managed in a reasoned way. For environmental and/or technical reasons on land management could be required, it could be necessary to find innovative and beneficial reuse solutions. In this work, the potential use of two dam fine-grained sediment as pozzolanic binders is studied. After physical and mineralogical characterizations, dredged materials are fired at ten different temperatures between 550 °C and 1000 °C. These calcined powders are also physically and mineralogically characterized. The characterization is related to pozzolanic activity assessment by Frattini test. Results show that a marked pozzolanicity is developed only for one sediment, with an optimum calcination temperature of 850 °C. Pozzolanic reactivity after thermal treatment depends on the mineral characteristics of the raw material: carbonates and clay minerals contents. These preliminary results suggest a potential development of innovative and environmental-friendly binders.

*Keywords: Fine dam sediment, Beneficial reuse, Characterization, Thermal treatment, Pozzolanic activity*

## INTRODUCTION

Sediment management is required in order to ensure good operation of harbor facilities, waterways and reservoirs. The latter might be used as water storage, recreational activity spaces and energy production sources. As a hydroelectric dam manager, EDF Group is concerned with sediment management. Due to soils and rocks erosion of the watershed, particles are carried to the watercourse where they are transported in suspension. Because of the dam, the speed of the current diminishes, leading to siltation phenomenon and accumulation of sediment.

According to European and French legislation on water and aquatic environment, most of the sediment stays in the watercourse and is transferred from one side of the dam to the other. Such techniques ensure ecological and sediment continuity along the river.

Nevertheless, due to environmental and/or technical reasons, it could become necessary to dredge a part of dam fine sediments. If the dredged material has to be managed on land, it becomes part of the European list of waste materials. This status implies that beneficial reuse solutions should be found in order to avoid waste disposal by landfill.

In this work, the studied material mainly contains particles with  $d_{50} < 63 \mu\text{m}$ , that is to say silt and clay fractions.

In the literature, several reuse options have been considered for fine-grained sediments such as: (i) spreading on agricultural lands and raw material in anthropogenic soil construction [1], [2]; (ii) sub-base layer in road construction [3], [4]; (iii) raw material in the ceramics industry [5], [6]; (iv) aggregates or filler in concrete formulations [7]; and (v) raw material for ordinary Portland cement (OPC) synthesis [8], [9].

These different beneficial reuse solutions were investigated by Anger [10] for sediments sampled in hydroelectric reservoirs. The study presented in this paper follows the work of [10].

Besides these five primary recovery options, another one could be considered: pozzolanic activation after thermal treatment. According to the European Standard EN 197-1, pozzolanic materials are “substances of siliceous or silico-aluminous composition or a combination thereof” [11]. Contrary to hydraulic binders, they do not react in themselves when mixed with water. Pozzolanic materials need to be finely ground and mixed with calcium hydroxide ( $\text{Ca}(\text{OH})_2$ ) to react and form strength-developing phases: hydrated calcium silicates and hydrated calcium aluminates compounds. These hydrates are similar to those formed in the hardening hydraulic materials mixed with water.

Pozzolanic materials were already known in Greco-Roman eras: natural (mainly volcanic

deposits) and artificial pozzolanas (crushed fired-clay bricks and tiles) were used at these times to synthesize high-strength concrete [12]. When artificial Portland cement was discovered in the nineteenth century and then expanded worldwide, the interest for pozzolanas diminished. Mainly due to decarbonation reactions during Portland clinker synthesis, the cement industry is a major contributor to man-made carbon dioxide emissions: 5 % of the total in 2009 [13]. In a sustainable development approach, the interest for low-carbon cements is increasing. Pozzolanic materials can be suggested. Indeed, portlandite ( $\text{Ca(OH)}_2$ ) is formed during the hydration reactions of the anhydrous calcium silicates species of Portland cement. If Portland cement is partially replaced by pozzolana, a pozzolanic reaction is expected between  $\text{Ca(OH)}_2$  and the supplementary material forming additional hydrated phases containing silica and alumina. As the calcined clays processes require less energy than Portland cement production and emit less greenhouse effect gases, these blended cements should be considered as low-carbon cement.

It is well-known that some clay minerals are able to react with portlandite when thermally treated, for instance by transformation of kaolinite into metakaolinite. Since fine-grained sediments contain a noteworthy proportion of clay minerals, pozzolanic activity can be expected after a thermal treatment.

Indeed, Dang *et al.* [14] calcined at 650 °C and 850 °C a fine-grained sediment sampled in Rance estuary in France, made of quartz, calcite and phyllosilicates (illite, kaolinite, muscovite and montmorillonite). This marine dredged material was blended with OPC at various weight ratios: 8, 16 and 33 %. They showed that the mechanical strength of standard mortars is higher when the sediment calcined at 650 °C is used compared to the sediment heated at 850 °C (above decarbonation temperature). The sediment treated at 650 °C is also better than a calcareous filler from a mechanical strength point of view.

Semcha [15] suggested blended cements containing a thermally treated sediment from an Algerian reservoir. Only one phyllosilicate is identified by XRD analysis: kaolinite mineral. As kaolinite is highly reactive after a heating process at 750 °C, this temperature was chosen. Compression strength tests on cement pastes showed that blended cement with 30 % replacement of OPC by artificial pozzolana had performances similar to control cement pastes made with 100 % OPC. Sediments from the same reservoir were also studied by Belas [16]: replacement percentages were lower compared to Semcha (10, 15 and 20 %).

Concerning the durability of blended cements, Rabehi demonstrated that sediment from Choufra reservoir (Algeria) fired at 750 °C could increase the resistance of mortars to sulfatic and chloride attacks. Denser structure mainly explains this higher

durability. 10 % replacement of OPC seems to combine both mechanical strength and durability of mortars [17].

Therefore, according to this literature review, interesting results were obtained when calcined sediments are used as pozzolanic additions to concrete. However, generally speaking, the listed references only study a reduced number of calcination temperatures.

According to the watershed contexts, sediments necessarily demonstrate various mineralogical and physicochemical characteristics. Thus, the response to thermal treatment is expected to be different.

The aim of this study is to investigate the optimal temperature for pozzolanic activation according to the raw sediment characteristics in order to synthesize innovative and sustainable binders.

## MATERIALS AND METHODS

### Dredged Materials

#### *Origin of the studied fine-grained sediments*

Two reservoir sediments, called ISE and RHI, were sampled by EDF Group. Sampling locations are given in Fig. 1.

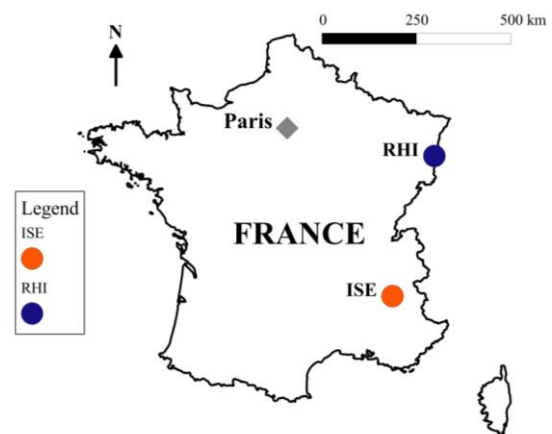


Fig. 1 Map of the sampling sites

### *Characterizations*

All the analyses were performed on a previously dried material. Therefore, it is interesting to measure the water content before the oven-drying operation. This was done according to the European standard NF EN 12880 [18]. In this protocol, samples are dried at 105 °C until reaching a constant mass.

The dredged materials were characterized by combined thermogravimetry and differential thermal analysis (TGA/DTA). These experiments were performed on a Setaram SetSys 16/18 TG-DTA apparatus. The samples were heated up to 1000 °C

with a heating rate of 5 °C/min. A flow of dry air was used inside the chamber during the analysis.

In addition, the calcite contents of the samples were determined by the “Bernard calcimeter” method according to the French standard NF P94-048. Hydrochloric acid reacts with calcium carbonate of the sample, forming carbon dioxide. The released CO<sub>2</sub> volume is measured, allowing the calculation of the initial calcium carbonate content [19].

Mineralogical compositions were assessed by X-ray diffraction technique (XRD). Powders were analyzed on a Bruker D8 ADVANCE device using a Bragg-Brentano geometry: Cu anode ( $\lambda = 1,5406 \text{ \AA}$ ), angles between 5 and 70 °2 $\theta$ , 0.02 °2 $\theta$  step, operation at 40 kV and 40 mA). Crystalline phases identifications were performed using Bruker DIFFRAC<sup>plus</sup> EVA software.

Three types of microstructural characterizations were carried out:

- Particle size distribution (PSD): by laser diffraction using a Coulter LS230 apparatus;
- True density: by helium pycnometry using a Micromeritics AccuPyc 1330 device;
- Brunauer-Emmett-Teller (BET) specific surface area: using a Micromeritics Tristar II apparatus. Degassing temperature and duration were 120 °C and 17 hours respectively. The relatively low temperature was chosen in order to limit the thermal alteration of the raw material.

Chemical composition analyses were also performed on raw sediments using a Varian ICP-AES after alkali fusion.

### Calcination Process

Each sample was homogenized and dried for a week at 40 °C. It was checked that 100 % of the dredged materials passes through a 100 microns sieve. For RHI, around 5 % by mass was a coarser fraction. It had to be crushed in a mortar to reach diameters lower than 100  $\mu\text{m}$ .

Ten firing temperature were studied: 550 °C, 600 °, 650 °C, [...], 1000 °C. For each thermal cycle, 50 g of dredged material was calcined in a programmable electric elevator furnace. To reach the calcination temperature, a ramp of 5°C/min was used, followed by a dwell of 5 hours at this maximum temperature. When the dwell period was completed, sample was quenched to room temperature. This process should preserve the metastable phases formed during the firing cycle.

### Calcined Powders Characterizations

Microstructural characterisations were carried out on the fine-grained sediments calcined at different temperatures. Protocols were similar to those used on raw materials, except for the degassing procedure in

BET specific surface area analyses. The selected temperature is 200 °C: as sediments are calcined above 550 °C, there is no risk of degradation and the degassing process is more efficient. Moreover, no chemical analysis of calcined powder was performed.

### Pozzolanic Activity Assessment

Pozzolanic activity assessments were performed according to EN 196-5 protocol [20] also called Frattini tests. In this method, blended cements were prepared in polyethylene (PE) plastic containers mixing 16 g of OPC, 4 g of calcined sediment – 20 % by mass – and 100 mL of freshly deionized water. According to EN 197-1, the OPC used in the tests is referenced as CEM I 52.5 R. These prepared samples remained sealed at 40 °C until the analysis date. Tests were carried out after 8, 15 and 28 days of storage. At the deadline, they were vacuum-filtered and the filtrate was successively titrated by 0.1 M HCl and 0.03 M EDTA to determine HO<sup>-</sup> and Ca<sup>2+</sup> concentrations, respectively. The obtained concentrations were compared to the calcium hydroxide saturation curve as a function of pH.

## RESULTS AND DISCUSSION

### Raw sediments characterizations

#### *Water content of the samples*

The water contents of the studied samples are given in Table 1.

Table 1 Water contents according to NF EN 12880

Sediment	ISE	RHI
Water content (% by mass)	1.2	4.2

It can be seen that the free water content is already relatively low before the drying procedure. It is related to a natural air drying effect between the sampling operation in reservoirs and the laboratory analyses.

#### *Simultaneous DTA-TGA results*

Results of thermogravimetric and differential thermal analyses are given in **Erreur ! Source du renvoi introuvable.**

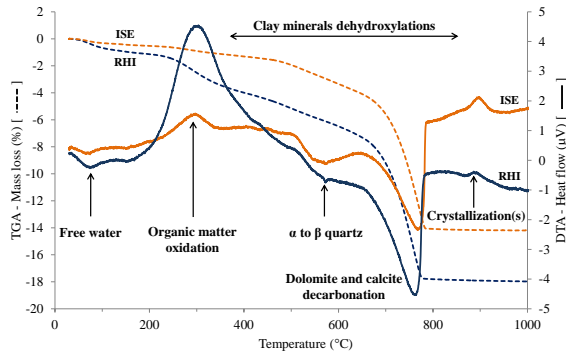


Fig. 2 DTA-TGA curves of ISE and RHI

Combined TG-DTA results show that free water content is reduced with respect to fine-grained sediment right after the sampling operation. Prior to performing the experiments, sediment needs to be dried and kept in dry conditions. Thus, there is no significant influence of the free water content on the total mass loss.

In contrast, a difference in behavior is observed between RHI and ISE in the 200 °C – 400 °C temperature range: the exothermic peak is stronger for RHI associated to a larger mass loss than for ISE. It can be explained by a higher content in organic matter for RHI. It can also be understood that calcination over 400 °C is necessary to use fine-grained sediment in order to remove the organic matter which is known for its negative impact on cement rheology and mechanical performance [10].

According to mass loss between 400 and 650 °C explained by dehydroxylation phenomena, phyllosilicates content should be higher for RHI sediment. Nevertheless, these mass losses have to be carefully interpreted since overlaps can occur like final oxidation of organic matter and beginning of kaolinite dehydroxylation around 500 °C [21].

The next strong endothermic peaks are attributed to carbonates (mainly calcite) decarbonation. The corresponding mass losses are also quite important and higher for RHI sediment than for ISE. Therefore, a higher production of calcium oxide can be expected above 800 °C for RHI. Between 800 °C and 1000 °C, mass losses are reduced and can be identified as final dehydroxylation of phyllosilicates, such as illite and muscovite. Also, a small exothermic peak on the DTA curve can be identified around 900 °C and could be associated to the crystallization of stable crystalline phases, such as wollastonite [22].

Thanks to these thermal analyses, the temperature of total dehydroxylation is located between 550 °C and 1000 °C. The effect of decarbonation can also be taken into account in this temperature range.

Carbonate content was also determined by the Bernard calcimeter technique. The obtained results on ISE and RHI samples are compiled in Table 2.

Table 2 Calcium carbonate content of the studied sample according to NF P94-048 protocol

Sediment	ISE	RHI
CaCO <sub>3</sub> content (% by mass)	25.7	27.4

Table 2 confirmed the TGA/DTA results: CaCO<sub>3</sub> content is higher for RHI than for ISE. Thus, over 800 °C, a higher production of lime after decarbonation could be expected for RHI.

#### Microstructural parameters

Physical characteristics are summarized in Table 3 for both the raw sediments.

Table 3 Microstructural characteristics of the raw sediments

Physical properties	ISE	RHI
S <sub>BET</sub> (m <sup>2</sup> /g)	11.7	7.8
Density (g/cm <sup>3</sup> )	2.70	2.59
d <sub>10</sub> (μm)	0.8	3.4
d <sub>50</sub> (μm)	6.1	24.4
d <sub>90</sub> (μm)	24.8	135.7

Particle size distributions showed that ISE is finer than RHI.

Furthermore, BET specific surface areas show slight differences: S<sub>BET</sub> for RHI appears lower than ISE S<sub>BET</sub>. It could be explained by the high carbonates content for RHI as deduced from TGA and the Bernard calcimeter results. This reduces the specific surface area of the whole material.

Finally, ISE density is higher than RHI's. The main explanation is probably the higher organic matter content of RHI that tends to diminish the material density.

#### X-ray diffraction analyses

Results of XRD analyses carried out on raw sediments are shown in Fig. 3.

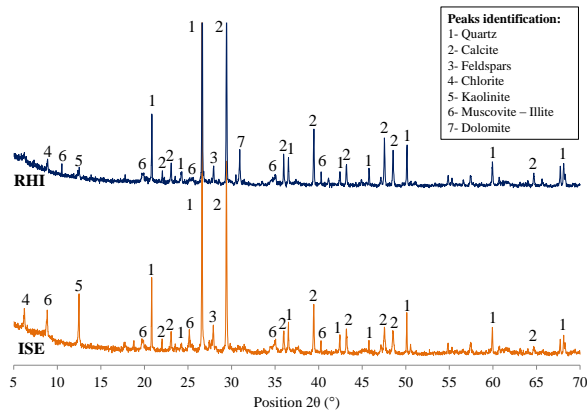


Fig. 3 XRD patterns of ISE and RHI

For both the studied sediments, the main mineral phases are quartz, carbonates, feldspars and phyllosilicates. However, the nature of the clay minerals differs. It is well-known that the XRD peak intensity of one phase in a mixture is directly proportional to its content. Thus, Fig. 3 shows that carbonate content is higher for RHI and clay minerals (chlorite and illite/muscovite) are more present in ISE.

### Calcined powders characterizations

#### Mineralogical study

The apparition and extinction of XRD peaks are summarized in Fig. 4.

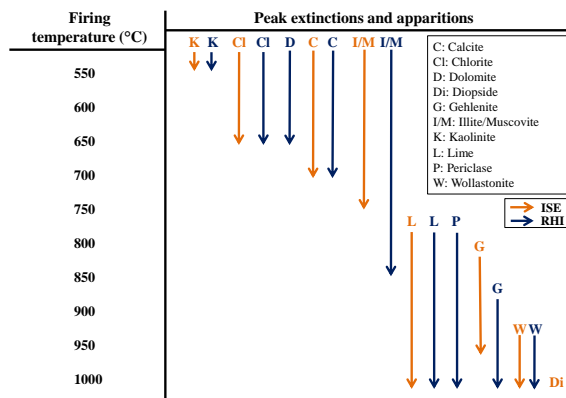


Fig. 4 Evolution of the mineral composition according to firing temperature

The examination of the mineral phases present after calcinations allows us to determine at which temperature the dehydroxylations of the clay minerals are complete: 800 °C and 900 °C for ISE and RHI, respectively. Moreover, calcite decarbonation (between 750 °C and 800 °C) and high temperature crystallizations (gehlenite, wollastonite and diopside) can be referenced.

#### Microstructural characteristics

The evolution of physical parameters *i.e.*  $S_{BET}$  and  $\rho$  according to firing temperature are presented in Fig. 5.

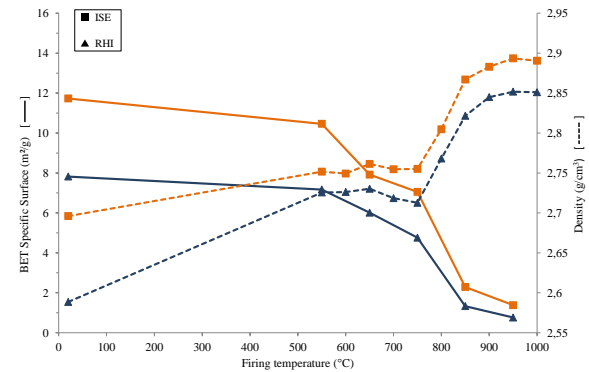


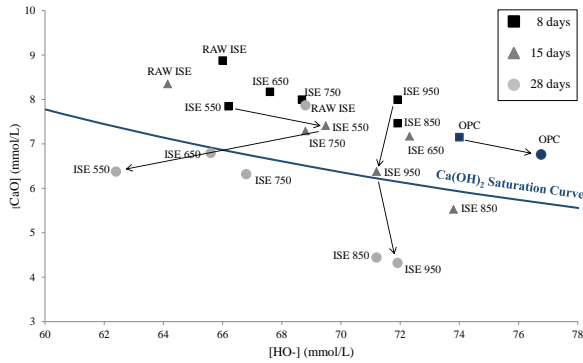
Fig. 5 Evolution of specific surface area areas and density of the sediments according to calcination temperature

It can be shown that  $S_{BET}$  diminishes when dredged materials are calcined. Until 750 °C, this diminution is moderate. Between 750 °C and 850 °C,  $S_{BET}$  collapses for both the sediments. The values measured at the highest temperature are in both cases lower than 1.5 m²/g. This constant reduction of BET specific surface area is also observed by Fernandez Lopez [23]: in this work, different commercial quarried clays were studied. Whatever the main clay mineral (kaolinite, illite or montmorillonite),  $S_{BET}$  decreased when the temperature of thermal treatment changed from 600 °C to 800 °C. This diminution was particularly noticeable for illite and montmorillonite.

Fig. 4 shows the density variations in the same temperature range. An increase in densities is seen from the raw sediment to the first calcination temperature (550 °C). This gain can be associated to the organic matter oxidation. As a result of this transformation of organic matter, density of the remaining material is relatively higher. This explanation is confirmed by the augmentation level: RHI density increases by 5.3 % whereas the increase of ISE density is only 2.1 %. It is well correlated to the higher organic matter content of raw RHI identified by TGA/DTA analysis. Until 750 °C, densities are stable or slightly reduced (from 650 °C to 750 °C for RHI). In this temperature range, two competitive phenomena can be identified: (i) a reduction of density due to decarbonation and (ii) an increase due to the beginning of sintering. Beyond 750 °C, that is to say after carbonate decomposition, densities are significantly increased.

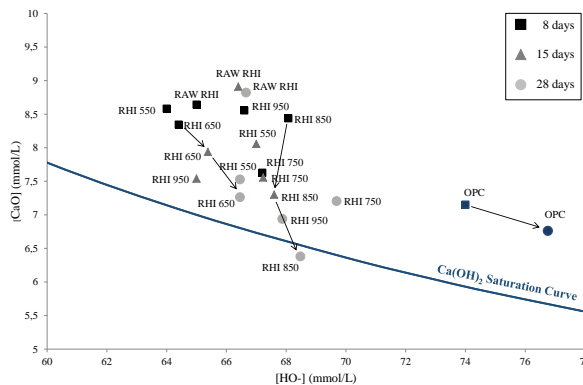
### Pozzolanic activity tests

Results of pozzolanic activity assessment by Frattini tests at 550 °C, 650 °C, 750 °C, 850 °C and 950 °C are given in Fig. 6 and Fig. 7. Evolutions with curing durations of blended cements with raw sediments and the evolution between 8 and 28 days of a control cement (100 % OPC) are also plotted.



Note: OPC: control Ordinary Portland Cement  
RAW ISE: Portland cement blended with non-thermally treated ISE sediment  
ISE 550: Portland cement blended with ISE sediment calcined at 550 °C (same format for other calcination temperatures)

Fig. 6 Results of Frattini tests for blended cements incorporating raw and calcined ISE sediment after different curing durations (8, 15 and 28 days)



Note: OPC: Control Ordinary Portland Cement  
RAW RHI: Portland cement blended with non-thermally treated RHI sediment  
RHI 550: Portland cement blended with RHI sediment calcined at 550 °C (same format for other calcination temperatures)

Fig. 7 Results of Frattini tests for blended cements incorporating raw and calcined RHI sediment after different curing durations (8, 15 and 28 days)

As a first interpretation, it can be seen that:

- (i) For OPC, concentrations in calcium and hydroxide ions are above the saturation curve, meaning that Portland cement continuously supplies the aqueous solutions in  $\text{HO}^-$  and  $\text{Ca}^{2+}$ .
- (ii) For all the blended cements incorporating fired sediments, calcium concentrations at a given ageing duration are lower than in blended cements with raw material. It suggests that the thermal treatment has a positive effect on the activation of powders.
- (iii) Whatever the blended cement,  $[\text{HO}^-]$  is lower than in OPC. It indicates that the addition of a sediment slightly acidifies the solution.
- (iv) Concerning calcium concentration, depending on the calcination temperature and the curing duration, the value can be either higher or lower than for OPC.
- (v) RHI values are less dispersed than for ISE, particularly on y-axis.

After these preliminary observations, the main question concerns the utilization of this data to assess the pozzolanic activity of the addition. According to the standardized test protocol [20], an addition should be considered as pozzolanic if  $\text{CaO}$  and  $\text{HO}^-$  concentrations are below the saturation curve. It would mean that  $\text{Ca(OH)}_2$  generated by OPC hydration is consumed and incorporated into newly formed hydrated phases – probably C-A-H and C-S-H types. In the strict sense of the standard, this condition is fulfilled for calcined ISE after 28 days of ageing. For RHI, even if there is a  $[\text{CaO}]$  decrease for fired RHI blended cements with time, only one point (temperature of 850 °C) is below the  $\text{Ca(OH)}_2$  saturation curve after 28 days. As it is almost on the curve, only a reduced pozzolanic activity is proposed for this firing temperature.

Results for ISE can be divided in two: thermal treatments below and above 750 °C. For ISE 850 and ISE 950, there is no significant change in  $[\text{HO}^-]$  between 8 and 28 days and the  $[\text{CaO}]$  drop on the same period is much more important than at lower temperatures. If these results are related to mineralogical evolution according to temperature, it can be seen that 800 °C is the temperature at which complete destruction of illite occurs (peak disparition in XRD). Then it can be suggested that the loss in structure and organization of illite leads to a reactive material. Below this temperature, other phyllosilicates can be dehydroxylated by the thermal treatment and develop a reduced pozzolanic activity that could explain the smaller drop in  $[\text{CaO}]$ .

According to [10], illite is the main phyllosilicate of ISE: it is interesting to identify whether similar results are observed on quarried illitic clays.

A recent Argentinian study by Lemma *et al.* [24] concerning calcination of illitic clays extracted from quarries showed results close to ours. As less firing temperatures are studied (only 300, 600 and 950 °C),

it can only be said that better results are obtained for the 950 °C treatment. However, the Frattini kinetic patterns are very close to the ones we obtained. Moreover, [25] showed that industrial illitic clay can demonstrate low to moderate pozzolanic activity only if calcined at a high temperature (930 °C). The preliminary dehydroxylation of illite around 650 °C is not enough to thermally activate the material. Total destruction is required. This result is also in agreement with observations made on ISE.

Although minerals are qualitatively similar between raw ISE and raw RHI, the content in phyllosilicates has to be considered. Indeed, the clay minerals contents in RHI and ISE are 22 % and 35 % by mass respectively [10], while the total carbonate content (dolomite and calcite) is higher for RHI. According to our pozzolanic activity assessment a phyllosilicates threshold exists between 22 % and 35 %: the content has to be above this limit to observe a marked pozzolanic activity after thermal treatment.

Even if the dredged material densifies and loses specific surface area at temperatures higher than 800 °C, reactivity is increased in this range of temperature. Thus, it can be understood that physical parameters have less influence than mineralogical characteristics on pozzolanic activity. The lack of correlation between BET specific surface area is shown by [26] on rice husk ash: a pozzolana obtained after calcination of agriculture sub-product.

## CONCLUSIONS AND OUTLOOK

As a conclusion, it can be said that thermal treatment of fine-grained dredged material potentially leads to a reactive pozzolanic materials. According to phyllosilicates and calcite contents, the behavior of calcined sediment varies. ISE appears more suitable to this application than RHI. A relevant firing temperature – relevant for the activation and for energy consumption reasons – would be 850 °C corresponding to the complete destruction temperature of illite mineral. Innovative, promising and “greener” binders, using calcined sediments as a replacement of a part of clinker in blended cements can be expected.

Additional data concerning mechanical strengths and performances of hydrated blended cements will be obtained and connected to the results available in this paper.

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