# The use of flash calcined dredging sediments in cementitious matrix

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#### **Abstract**

Significant volumes of sediment are annually dredged in France (50 million m<sup>3</sup>), about 300 million tons in Europe, and all over the world. These materials may, in fact, be used beneficially as supplementary cementitious material.

In this paper, several mineral addition are used in substitution to cement in order to make comparison : raw sediment (RS), sediments treated with the flash calcination method (STFC) based on a low energy consumption process and Limestone filler (LF80). The physical, chemical and mineralogical characterization performed show that heat treatment has interesting impact on the final characteristics of the sediments. The mechanical characterization evidenced that the flash calcination based mortar has an equivalent or better performances to the reference mortar for a substitution rate below 10%. Moreover, the presence of the calcined materials generates additional heat release during hydration process.

Key words: Sediment, Characterization, Flash calcination, Substitution, Pozzolanic activity.

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## **1** Introduction

In the early 21<sup>st</sup> century, the environment issue become a major concern. The 21<sup>st</sup> United Nations conference on climate change held in 1992 in Brazil inspired by the famous Brundtland report entitled "Our Common Future" in 1987, officially introduced the new environmental global founding politics principles. In the construction sector, aggregate need was 366 million tonnes in France in 2013, of which 96% were of natural origin. The turnover of the aggregates sector has reached in 2015 the sum of 3.66 billion euros [1] and the ready-mixed concrete (RMC) sector turnover was 3.77 billion euros. Cement consumption in France meanwhile has reached almost 19 million tonnes in 2013. This represents a considerable financial wealth and environmental weight. Sediments can potentially be used as materials in the building sector [2–6].

In order to save natural resources and consider the increase in environmental regulations, the use of alternative materials in the construction sector, recycling and the use of sediment as a resource (USAR project, SETARMS project, etc.) is considered a suitable solution for sustainable development [7,8].

However the beneficial use of dredged material is strongly controlled by several factors: mineral composition, grain size distribution and physicochemical characteristics [9]. Moreover, in this purpose, a number of scientific challenges need to be solved. Among these is the possible content of inorganic contaminants in sediment, such as heavy metals (lead, copper, chromium, etc), salts, cyanides, etc, as well as organic hydrocarbons :PAHs, PCBs and TBT [10]. In France, about 50 million m<sup>3</sup> [11] of sediments are annually dredged, stored, treated or salted overseas; with about 300 million [12] m<sup>3</sup> for whole Europe. In Brazil, this volume have reached 80,3 million m<sup>3</sup> [13] of sediment were dredged from ports in 2009.

The use of heat treatment by direct calcination using a laboratory furnace has already been applied to sediments in order to improve some properties [14–18] but it seems to have high cost and high energy consumption. However the use of the flash heat treatment, was applied several study for the activation of clays. For example to transform kaolin into Metakaolins [19–22] and reduces energy consumption and gas release by the rapid time process it require. This process involves dehydroxylation to activate a material by thermal excitation between 700°C and 850°C which initiates an amorphous phase [20]. Recent studies have targeted activation of the clay fractions present in sediments [12]. This present paper will focus on the characteristics of STFC based mortars in the form of a comparative study.

This study is based partially on results established on a previous study conducted by [23]. The latter had proven, by using different pouzzolanic test that these calcination process have positive impact on pozzolanic activity of sediments.

## 2 Material and methods

A Portland cement CEM I 52.5N (OPC) was been used which complies with European standard EN 197-1 (2012). The sand used for the mortar formulation was a siliceous standardised ISO 679: 2009, with rounded grains, river origin and a coarse aggregate Dmax = 2 mm.

The marine sediments were dredged from the Grand Port Maritime de Dunkerque (GPMD, France). Materials were dried in an oven at 40°C then crushed and sieved 120 $\mu$ m. In this study, sediments treated using direct calcination method were designated STDC and STFC for those which were treated using flash method, RS for raw sediment, NS for natural sand. MK80 is a metakaolin with Dmax=80 $\mu$ m and LF80 is a limestone filler with Dmax=80 $\mu$ m.

The sediments used in this study are dredged from the Grand Port Maritime de Dunkerque (GPMD) located in the North of France (GPS coordinates: 51°02'24.8"N 2°22'26.0"E). This port dredges about 4 million m<sup>3</sup> per year. A sample of 500kg was taken in February 2015 in a settling lagoon located inside the port where nonsubmersible dredged sediments are stored. This area is composed of pools of lagoons and settling ponds where sediments are initially placed to optimize drying process. This technique of active lagooning is mainly used for dredged material extracted by hydraulic dredging technique. At the end of the process, the water content reduction can reaches 65%; what facilitates transport or landfilling.

## 2.1 Experimental program: Characterization techniques

All used materials were characterised using physical and chemical methods. The granulometry analysis was performed using a COULTER LS12330. This laser device allows the determination of granular particle distribution under 1µm., Mechanical strength were asssesed using the three prismatic test sample 4x4x16 cm (NF EN 196-1 [24]). The BET (Brunauer-Emmett-Teller) method and Blaine (NF EN 196-6 [25]) test are an estimation of surface area and allow the finesse to be appreciated (NF EN ISO 18757 [26]) by using a Micromeritics Autopore IV 9505. The organic fraction was also determined (XP P94-047 [27]) and total organic carbon (TOC) content was evaluated. The specific density of the materials was determined by a MICROMETRICS AccuPyc1330 helium pycnometer(NF EN 1097-7[28]). The apparatus was 400\*160mm with a Dewar thermally sealed container. The initial setting time test was performed which the aim of which is to monitor the initial setting of the cementitious matrix using a VICATRONIC automatic device in accordance with the NF EN 196-3 [29] standard.

The thermogravimetric analysis (TGA) and Differential Scanning Calorimetry (DSC) where carried out on an NETZSCH STA 449 apparatus using nitrogen gas.

Mineralogical characterization through X-ray diffraction (XRD) analysis was conducted. These tests identify and qualify the mineralogical nature and crystalline phases present. The settings were 40 kV and 40 mA voltage, as already described in earlier studies in the case of sediment-based mortar [2,30,31]. In addition, an XRD bulk mineralogical composition was investigated using the Rietveld refinement method specifically for the quantitative measurements of clay phases. SEM analysis was performed using a Hitachi S-4300SE/N Scanning Electron Microscope (SEM) with a field emission gun; the acceleration voltage was regulated at 5 kV.

## 2.2 FLASH calcination method

Flash calcining is a heat treatment technique for the rapid exposure of finely crushed materials in the presence of air, under high temperatures, using a technique which was initiated and developed by Professor Salvador in 1992 [21]. The flash calcination technique was originally applied to chemically activate some clays such as Kaolinite and to confer pozzolanic properties. When applying this technique to Kaolin clays, a dehydroxylation process following the dehydration was noted [20,32]. This fits with the elimination of a hydroxyl bond (-OH) which occurs between 450°C and 750°C [37]. Direct calcination can help to achieve such results. However, energy costs and CO<sub>2</sub> footprint in particular are often exorbitant. In addition, due to the instantaneousness character of the flash process, the flash calcination causes the emergence of glassy phases, a destructuration of the matter, amorphous state and therefore a potential reactivity of the product [38]. Two parameters are however predominant: the exposure time (in the order of a few tenths (1/10)<sup>th</sup> of seconds), and the temperature, which is around 1200°C inside the calciner unit [20].

The aim of our study was to apply this process to the treatment of dredged sediments, which will be adapted to activate some of the sediment phases (including clay phases). The calcining unit, flash tower and pilot principle are shown in Fig. 1.



Fig. 1 a) Flash calciner unit ; b) Semi mobile flash tower [20] ; c) Pilot principle

#### 3 **Results and discussion**

#### 3.1 **Physical characterization**

#### 3.1.1 Granulometry

a)

The laser granulometry results are given in Fig. 2. The calcination process has greater the coarse fraction (D50=2.46 $\mu m$  for RS,  $D_{50}{=}5.24 \mu m$  for STDC and  $D_{50}{=}5.93 \mu m$  for STFC ).



Fig. 2 Particle size distribution for OPC, MK80, LF80, NS, RS, STDC and STFC.

#### 3.1.2 **Physical properties**

The results indicate that the direct calcination as a flash method modifies the material density. Probably, the density of material increases by a sintering or densification process. Through these stages, the coarser grains would sinter each other through a phenomenon that involves the welding of grains under the effect of heat [14,39]. The effect on the STFC, however, is relatively low, probably because of the duration of the treatment.

The results of Blaine and BET analyses are presented in Table 1. These parameter allowed a correlation to be found with the activity of the material and therefore its performance.

The measure of the Blaine-specific surface area (BSA) consists of confining material in a crossing cell by an air stream under the effect of a pressure variation. The results clearly show that the direct calcination process as a flash impacts the fineness of materials. Indeed, calcining systematically decreases the surface area and involves the density, but the most significant effect is in the BET analysis instead of the BSA test, which is more suitable for cement.

	Cement	Raw sed	Flash calcined	Limestone	Natural	
Materials	CEM I	120µm	sediment	fillers		
	52,5 N	(RS)	(STFC)	( <b>LF80</b> )	Sand (INS)	
Density (g/cm <sup>3</sup> )	3.15	2.48	2.65	2.70	2.65	
Blaine surface area (cm²/g)	3669.1	10 093,4	4106.31	7181	4548.9	
BET (cm <sup>2</sup> /g)	9194	86207	59930	9744	9507	
TOC (%)		6.1	0.99			
LOI (%)	1.9	9.92	1.70			

Table 1

Det

TOC= total organic carbon; LOI= loss of ignition

#### 3.1.3 Thermo gravimetric analysis (TGA) and DSC

This technique has been used by Xiao et al. [42] in order to study sticky rice–lime mortars properties. The TGA analysis (**Fig. 3**) highlights a peak in mass loss corresponding to the dehydration phase. This mass loss appears from  $380^{\circ}$ C to  $600^{\circ}$ C. This loss of H<sub>2</sub>O is probably due to intergranular water and constitution water from clay minerals as identified by [32]. The release of carbon monoxide and carbon dioxide between  $350^{\circ}$ C and  $550^{\circ}$ C could originate from the combustion reactions of organic matter and organic pollutants (PAHs, PCBs, TBT) present in sediments [43]. A CO<sub>2</sub> peak appears between  $600^{\circ}$ C and  $750^{\circ}$ C and is induced by the decarbonation of calcite [17]. This must originate from the shells of marine species. According to this analysis, the temperature which allows for optimum calcination sediment must be greater than  $730^{\circ}$ C.



Fig. 3 : a) TGA analysis on RS and b) TGA on pre flash-calcined sediment (STFC)

The choice of calcination temperature was guided by the results in Fig. 3-b. TGA and DSC were performed on flash calcined sediment at 765°C (STFC765) and a flash calcined sediment at 820°C (STFC820). It is noted that there is still a loss of relatively large mass (~ 10.6%) between 400°C and 700°C, compared to 16.5% in the RS (Fig. 3-a). The analysis revealed the presence of a recrystallisation peak at ~820°C. Due to a lack of calcined matter noted on this sample, the suitable choice was then to calcine material at 820°C. On the latter material, the mass loss between 400°C and 700°C decreases (~6.3%). In addition, the area of the recrystallisation peak at 810°C did not decrease sensitively which indicates that the recrystallisation process had not been completed (compared to at 765°C). There is also a peak at 400°C which might correspond to calcium hydroxide (Ca(OH<sub>2</sub>)). Indeed, there has been a partial production of CaO through the calcination process. Due to air humidity, it is brought back to

 $Ca(OH_2)$  during the cooling phase. Another part reacts with ambient carbon dioxide (CO<sub>2</sub>) to recreate CaCO<sub>3</sub> [12]. These analyses infer the benefit of applying 800°C as the temperature for flash calcination.

## 3.2 Chemical and mineralogical analysis

The chemical analysis consisted of determining the proportion of each chemical element by X-Ray Fluorescence (XRF) (Table 2 and Table 3). It revealed the presence of the following main chemical elements: oxygen (~50%), silicon (~16.6%) and calcium (~15%). These values are close to those identified in previous studies [44,45] conducted on GPMD sediments. For both calcined sediments, the results are quite similar, with similar values for the main oxide constituents SiO<sub>2</sub>, CaO, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub> and little difference from RS. These results confirm that the calcination processes should have moderate impacts on chemical composition regarding changes between RS and STDC-STFC material.

#### Table 2

Concentrations (%) of the major chemical elements for OPC, RS, STDC and STFC

Elements	0	Na	Mg	Al	Si	Р	S	K	Ca	Ti	Mn	Fe	Zn
OPC	38.9	0.5	0.7	2.7	7.6	0.2	1.8	0.1	0.6	43.5	0.2	(*)MTE	2.8
RS	50.1	1.1	1.5	5.1	16.6	0.2	1.8	1.5	15.0	0.3	0.1	5.5	0.05
STFC	46.2	1.5	1.5	5.1	17	0.2	2.4	1.6	16.4	0.3	0.1	6.2	0.1

(\*)MTE = metallic trace elements

#### Table 3

Table 4

Concentrations (%) of the major oxide elements for OPC, RS, STDC and STFC

	Na <sub>2</sub> O	MgO	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	P2O5	SO <sub>3</sub>	K <sub>2</sub> O	CaO	TiO <sub>2</sub>	MnO	Fe <sub>2</sub> O <sub>3</sub>	ZnO
OPC	0.3	0.8	5.1	20	0.46	3.1	0.8	63.5	0.33	0.13	3.4	0.25
RS	2.0	2.0	8.2	51.9	0.4	0.2	1.9	22.1	0.4	0.2	9.3	0.1
STFC	1.9	2	8	52.8	0.4	0.2	1.8	21.6	0.4	0.2	9.3	0.1

(\*)MTE = metallic trace elements

#### 3.2.1 X-ray diffraction analysis (XRD)

These analyses were performed on a single oriented glass slide to identify clay minerals; the results are presented in Table 4 and **Fig. 4**. It is based on the determination of lattice spacings (001) and relative intensities. The calcination process transforms the majority of the clay minerals present to illite (86%). According to [17], the occurrence of newly created phases can be explained by the heat treatment effect.

Samples	Smectite	Illite	Kaolinite	Chlorite	Interlayered 10- 14S swellings	Additional minerals
RS	32%	36%	20%	12%		Quartz
STFC		86%	5%		9%	Quartz

Identification of clay mineral by the XRD method

The analysis revealed that the RS samples are composed mainly of illite (36%) and smectite (32%), but there is also kaolinite, which represents 20% of the clay phase, and chlorite (11-12%).

STFC is composed of 86% illite and 5% kaolinite. The presence of interlayered swelling illite-smectite (9%) is also noted. The XRD patterns clearly establish the effect of heat on minerals with a structural decomposition of the main clay minerals [12]. These transformations should explain a potential chemical activity of this product.



Fig. 4 Identification of the mineral clay phases on RS and STFC by XRD.

#### 3.2.2 Scanning Electron Microscopy (SEM)

The SEM analysis consists of an observation at very high resolution of the internal structure of material. SEM analysis provides powerful magnification, up to 500,000 times. That allows the majority of micron constituents of mortar to be observed, such as hydrates CSH ( $\approx 100$  Å), strips of clay particles ( $\approx 10$  Å), ettringite crystals ( $\approx (50 \,\mu\text{m})$ , crystals of portlandite Ca(OH)<sub>2</sub> ( $\approx 20 \,\mu\text{m}$ ), or the morphology of finely-ground material. The results in Fig. 5-a) and Fig. 5-b)) seems to show different particles of mineral or animal (sea shell) origin. These types of particles are common on sediments. Also, because of sedimentation and the presence of certain minerals such as limestone (CaCO<sub>3</sub>), the phenomenon of cementing between the particles may be produced (Fig. 5-a). For the RS material, the presence of pyrite particles and some plate-like particles can be clearly distinguished (Fig. 5-a). The presence of organic constituents or pollutants partly justifies the need for adequate treatment (using heat treatment for instance).



Fig. 5 Electron microscopy of RS and treated STFC sediment

For STFC, some physical changes have been confirmed by the SEM. The shape of particles is clearly modified by the heat process, with the growth of regular spherical particles with a diameter of  $20\mu$ m (Fig. 5-d). In an earlier study conducted by [20] on pure Kaolin, these particles have been identified to account for 20% in the final product. In a similar context, [19] showed that these particles originated from an agglomeration process of submicron kaolin, which occur during flash calcination, as shown in Fig. 5-c. They are mainly composed of gases and nanometre-sized particles of aluminium silicates which cover the external surface and crystallinities (mullite) (Fig. 5-c). In a later investigation, [12] established the local melting process origin of this effect. Particles around spheres have been established to be nanometre-sized particles of aluminium silicates [19].

# 4 Impact of the presence of sediments on cementitious matrix

This study is based partially on result established on a previous study conducted by [23]. The latter proven by using different pouzzolanic test that these calcination process have positive impact on pozzolanic activity of sediments.

The physico-chemical composition of sediments have an impact on the cementitious matrix [17]. Indeed, the presence of lime, which comes from the thermal transformation of calcite (Care et al., 2000) can enhance hydration reactions. However, the presence of constituents such as Lead(Pb) [50], Zinc(Zn) [51], Cadnium(Cd) or Chromium(Cr) [50] for instance, can perturb the regular hydration processes and the setting of mortar [52,53]. Some constituents can also disrupt the mechanisms which habitually lead to the formation of hydrates (CSH) or modify the porous network [50,52,54].

Blended sediment based mortars are namely designated: RM = reference mortar with any substitution, MRS= mortar with raw sediment, MSTFC= mortar including treated sediment by flash calcination. The number after these notations stands for the substitution rate (MRS5=5% cement replacement for instance).

#### 4.1 Mechanical strength

The prismatic samples 4x4x16 cm (NF EN 196-1[24]) were tested in compression for all formulated mortars. The results of the compression tests demonstrate that treated sediment-based mortars have significant resistance. At 28 days, the most resistant mortars were MRS5 and MSTFC5 with respective compressive strengths of 59.2MPa and 62.2MPa (Fig. 6 and Fig. 7). Mortars substituted by 10% had similar strengths to RM at 28 days, while the 15%-substituted mortars had resistances lower than RM strength in the order of 5% and 10%.

These results seem to infer that the presence of materials such as sediment, adequately used in cementitious matrix, induces overall strength improvement. Benezet et al. [55] has demonstrated in an previous study that finely crushed quartz particle (under 5µm or BET>10000 cm<sup>2</sup>/g) are highly active. In this present study, one should have in mind that  $D_{50} \le 6\mu$ m for all calcined material and BET>20000 cm<sup>2</sup>/g (Fig. 2 and Table 1).



Fig. 6 Compression strength of RM and MRS, (5%,10% and 15%) mortar



Fig. 7 Compression strength of RM, MSTDC (5%, 10% and 15%) and MSTFC (5%, 10% and 15%) mortar

## **5** Conclusion

The aim of this paper was to study the effect of heat treatment processes on sediment. The purpose was to determine and compare the physical, mineralogical, morphological and chemical activity and the characteristics of flash calcined sediment, raw sediment, and direct calcined sediment using a lab kiln and also pure metakaolin and limestone filler. Sediment-based mortars are formulated and tested after 7, 14 and 28 days of curing water at 20°C. The important conclusions which can be drawn through this work are the following:

- Heat treatment modifies some physical parameters such as density (by removal of organic matter) or densification. However, the finesse is dropped by a swelling or sintering process.
- The calcination process leads to drying, dehydroxylation and decalcification processes. Mineralogical analysis evidenced that the calcination leads to the creation of new phases but the differences are fairly small between the flash-calcined and direct-calcined sediment.
- The XRD analysis of clay phases on oriented glass slide highlighted that during the flash calcination, the clayey phases present were transformed mainly to illite. Also, the SEM analysis leads to identify spherical particles constituted of gas and aluminium silicates which can enhance the hydration process.
- From the mechanical point of view, the presence of fine grounded sediments, treated in an optimal way, enhances the resistance until 10% substitution. The compressive strength of MSTFC mortars is 11.3% higher to the RM at 28 days for 5% substitution. Whereas the 10% substituted mortars are equivalent to RM at the same time.

A later study will comparatively determine the pozzolanicity of treated sediments using chemical tests. Also, an additional study relative to the durability and environmental impact of sediment-based mortars will be carried out in a near future.

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