The Pozzolanic Activity of the Sediment Treated by the Flash Calcination Method

DUC CHINH CHU (✉ duc.chinh.chu@imt-lille-douai.fr)

IMT Lille Douai <https://orcid.org/0000-0002-7142-0497>

Mouhamadou AMAR

IMT Lille - Douai

Joelle KLEIB

IMT Lille - Douai

Mahfoud BENZERZOUR

IMT Lille - Douai

Damien BETRANCOURT

IMT Lille Douai

Nor-Edine ABRIAK

IMT Lille Douai

Jaouad NADAH

EQIOM

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The pozzolanic activity of the sediment treated by the Flash calcination method

Duc Chinh CHU⁽¹⁾, Mouhamadou AMAR⁽¹⁾, Joelle KLEIB⁽¹⁾, Mahfoud BENZERZOUR⁽¹⁾, Damien BETRANCOURT⁽¹⁾,
Nor-Edine ABRIAK⁽¹⁾, Jaouad NADAH⁽²⁾,

⁽¹⁾ Univ.Lille, IMT Lille Douai, Univ.Artois, Yncrea Hauts-de-France, ULR 4515-LGCgE, 6 Laboratoire de Génie civil et
géo-Environnement, F-59000, Lille, France

⁽²⁾ EQIOM Le LAB, CRT 1 Parc Vendôme – 460 Allée de l'Innovation, 59810 LESQUIN, France.

^(*) Corresponding author:

duc.chinh.chu@imt-nord-europe.fr

Abstract

The dredged sediment has been positioned for years as alternative materials in the construction field. However, it is often necessary to apply a treatment to improve their reactivity and performance. This article aims to study the pozzolanic reactivity of fluvial sediment treated by flash calcination method at different temperatures 650 °C, 750 °C, and 800 °C. The physico-chemical, mineralogical, and environmental characteristics were studied for treated (flash-calcined sediment) and raw sediment. The pozzolanic reactivity of the flash-calcined sediments was estimated with Frattini's test, isothermal calorimetry test, lime consumption analysis and compressive strength then compared to metakaolin which is considered as the reference. The results of the compressive strength of mortars show the detrimental effect of raw sediment on the development of resistance. Contrary to the raw sediment, the treatment of the sediments by flash calcination activates the pozzolanic reactivity of the clay phases and considerably improves the contribution of the sediments to the development of resistance and the porous structure. Moreover, the sediment calcined at 750 °C gives better properties than those obtained at 650 °C and 800 °C. The result demonstrates the feasibility of using calcined sediments as a pozzolanic mineral addition in a cementitious material.

Graphic Abstract

Keywords: Sediment, Flash calcination, Pozzolanic, Reactivity, Cement, Compressive strength.

Statement of Novelty

The originality of this research is the use of an innovative calcination technique to treat the sediment. The raw sediment is a polluted sediment and has harmful effects on the hydration of the cement. However, Flash calcination significantly improved the hydration behavior of sediment compared to raw sediment. The different methods were used to demonstrate the beneficial effects provided by sediments calcined at different temperatures and to choose an optimal calcination temperature.

1 Introduction

Nowadays, concrete is the most used material in the construction field and public works [1]. However, the production of this material has a great impact on the environment due to the requirement of significant extraction of raw materials. With a global cement production of 4.6 billion tonnes in 2016 and an average annual growth of 6.9% [2], the global cement production process releases between 652 kg and 894 kg of CO₂ for each tonne of cement produced [3]. Above 7% of the worldwide CO₂ emissions are related to the cement manufacturing [2]. To achieve the goal of reducing CO₂ emissions by 2050 and to attend carbon neutrality, the use of mineral additions to replace part of the cement is considered one of the most promising solutions [4]. To this day, metakaolin, slag, fly ash, silica fume, and limestone fillers are the most widely used mineral additions in the construction industry. Thanks to their pozzolanic reactivity, the durability properties and the mechanical performances of concrete have improved [5]. However, one of the main disadvantages of large scale application of metakaolin, is the limitation of the high purity

kaolinite deposits [6]. In addition, environmental restrictions are increasingly strict (2006/21/EC) for an ecological transition and the protection of biodiversity.

Each year, the dredging operations for maintenance at ports and inland waterways generate around 300 million tonnes of sediment in Europe [6], with 56 million m³ are in France. Several studies have demonstrated the potential for sediment recovery in various fields: road technology [7, 8] as a mineral addition in mortar and concrete [4, 9], [10, 11] and as a raw material in the manufacture of cement [12, 13]. Nevertheless, most studies of valorization of sediments as an addition are based on the traditional calcination method to activate their pozzolanic reactivity. This method requires significant energy consumption and arises a significant ecological problem [14].

Snellings et al [15] studied the effect of the flash calcination of dredged sediment, from the port of Antwerp (Belgium), at different temperatures (820 °C, 865 °C and 905 °C) on their physico-chemical properties and on their pozzolanic reactivity. The treatment reduced the total organic carbon content by 85% or more. The clay minerals in the sediment were completely dehydroxylated. The amorphous phase formed during calcination constitutes the major part of the calcined material. In addition, the result of the calorimetric analysis showed that the calcined sediments had pozzolanic reactivity more than that of siliceous fly ash, however less than that of metakaolin. Snellings et al [6], showed also in another study that increasing the amount of cement replacement from 20 wt% to 40 wt.% by calcined sediment slowed down the strength development. Whereas, at 91 days of hydration, the mortar with 20 wt.% calcined sediment substitution gives similar strength as the reference mortar.

Amar et al [16] compared the pozzolanic reactivity of sediment treated by flash calcination at 820 °C and by traditional calcination method. The results showed that the flash calcination has strongly modified the physico-chemical and mineralogical properties and improved the pozzolanic reactivity. The study showed also that the activity index (X_A) of mortars containing 25% wt of flash sediments as cement substitution, is higher than the one containing traditional calcined sediment at 28 days.

This study focuses on optimizing the flash calcination process of sediment on an semi-industrial scale. First, the sediment was calcined at three different temperatures (650 °C, 750 °C and 800 °C). Then, the pozzolanic reactivity of the calcined sediments and the raw sediment was examined by several methods in order to assess the performance of the method and the effect of the sediments on the hydration of the cementitious material. Finally, these results were used to choose an optimal calcination temperature.

1.1 Flash calcination method

The Flash calcination method is a technique of heat treatment of finely ground materials under high temperature in the short term. It has been applied to the activation of clays [17, 18]. Such calcination aims to produce metastable mineral phases or complex morphology [19]. Besides, high-temperature calcination and thermal shock increase the surface defects of crystallites and increase the number of reactive sites [20].

In a calciner, mineral particles get heated up and then undergoes a series of conversion processes. During calcination, the mineral materials occurs the flowing reactions Eq(1) -> Eq (5) [21]:

In addition, under the thermal effect, the other clay minerals will be dehydroxylated according to the following equations (Table 1):

The above reactions are influenced by several parameters such as the kaolinite structure, the rate of heating, and the particle size [22].

1.2 Pozzolanic reaction in the cementitious material

A constituent having pozzolanic properties, contains silicate and aluminate phases allowing hydrate production of ternary system CaO-SiO₂-Al₂O₃-H₂O in the presence of lime and water. The pozzolanic addition used as a substitute for the clinker, will react with portlandite (CH) to form the hydrated calcium silicate "pozzolanic" (pozzolanic C-S-H (II)). This hydrate generally has a C/S lower ratio than that of C-S-H formed from the hydration of cement without pouzzolanic addition. Unlike C-S-H (II), the C/S ratio of C-S-H (I) is independent of temperature and the Ca²⁺ concentration of the solution in pores. The pozzolanic reaction can be described by the following equations [23]:

- Dissolution of SiO₂:

- Pozzolanic C-S-H precipitation:

In general, the pozzolanic C-S-H stoichiometry is $\text{C}_{1.1}\text{SH}_{3.9}$. In addition, the presence of a large amount of aluminate can allow the formation of other phases such as C_2ASH_8 (stratlingite); C_4AH_{13} or hydrogrenat (C_3AH_6).

2 Materials and experimental methods

2.1 Presentation of the kiln and the Flash calcination process

The flash calcination tests are carried out in a flash furnace available in research center of IMT Nord-Europe (France). The product output can reach 20 kg of material per hour. A propane gas burner providing a power between 10 - 50 kW allows materials to be calcined up to a temperature of 850 °C. Fig. 1 illustrates the schematic of the flash calcination furnace used in this study.

The principle and operation of the flash oven was early described by Teklay et al. [17]. However, several parameters are adjustable such as burner power, air-flow, material flow and the calcination temperature. These parameters should be adapted regarding the material properties. In this present study, the sediment was calcined at three different temperatures of 650 °C, 750 °C, and 800 °C as previously tested for other types of materials [24, 25]. Except for the calcination temperature, all the other parameters were maintained constant during calcination. The optimum calcination temperature must meet the following requirements:

- Considerable removal of organic matter in the sediment.
- Improvement in the pozzolanic reactivity of the products (degree of dehydroxylation of clay minerals, in particular, kaolinite).
- Mechanical performance of calcined sediments based mortars compared to reference mortars (without sediments).
- Quality of the fumes rejected during calcination (analysis of the chemical composition of the rejected fumes).
- The lowest possible energy consumption.

2.2 Materials and methods

The sediment used in this study is the fluvial sediment collected from the Noyelles-Sous-Lens disposal site in the Haut de France region in France. First, this sediment was homogenized and dried at 105 °C to constant weight, then finely ground before flash calcination. Sediments calcined at the three different temperatures 650 °C, 750 °C, and 800 °C are referred as SF 650, SF 750, and SF 800 respectively.

The cement used is the Ordinary Portland Cement CEM I 52.5N from EQIOM specified in European standard NF EN 196-1 [26]. The sand used for mortars preparation is the standardized sand with a particle size between 0.08 and 2 mm according to the standard NF EN 196-1 [26]. A commercial product metakaolin (MK) was used as the reference for pozzolanic reactivity. Table 2 presents the nomenclatures used in this study.

2.2.1 Material characterization methods

The physicochemical, mineralogical and environmental properties of materials are characterized by several laboratory tests.

- The particle size distribution of the materials was measured by a COULTER laser diffractometry, LS 13 320 type device. The samples were dispersed in ethanol before measurements. The Blaine surface was determined according to the standard NF EN 196-6 [27]. Brunauer-Emmett-Teller (BET) specific surface was measured using the N_2 multipoint adsorption method. The organic matter content of the sediments was determined using the loss on ignition method according to the standard XP P94-047 [35]. The density of the materials is measured according to NF EN 1097-7 [28] using Micromeritics ACCUPYC 1330 Helium Pycnometer. The chemical composition of the materials was determined using X-ray fluorescence analysis (XRF) according to standard NF EN 196-2 [29] with an S4 POINEER equipped with a 4-kW generator and a rhodium anode.
- The mineralogical composition of the materials is identified by X-ray diffraction (XDR) analysis using a Bruker apparatus equipped with a D2 diffractometer with a Cu anode ($\lambda = 1.5406 \text{ \AA}$). To specify the nature of the argillaceous phases of the sediment, a specific analysis was carried out on the fraction less than 2 μm of the sediments [8, 11, 30]. For the study of clay species, the angular range explored is between 4° and 35° 2 θ with a step size reduced to 0.008 ° 2 θ and a step time of 0.84 s/step.
- Thermogravimetric analysis (TGA) is performed using the Netzsch STA 409 device. About 100 mg of the finely ground material was placed in an alumina crucible and heated from 40 °C to 1000 °C with a heating rate of 5° C/min.

Dehydroxylation of kaolinite occurs at the temperature range of 400 °C to 600 °C during calcination [31]. The amount of kaolinite could be determined from the mass loss of water (M_{H_2O}) on the TGA curve according to the following equation (Eq (12)):

$$Q_{\text{mineral kaolinite}} = m_{H_2O} * \left(\frac{M_{\text{mineral kaolinite}}}{n_{H_2O} * M_{H_2O}} \right) \quad (12)$$

with :

$Q_{\text{mineral kaolinite}}$: Quantity of the mineral kaolinite.

m_{H_2O} : Loss of water due to the dehydroxylation of kaolinite between 400 °C and 600 °C.

$M_{\text{mineral kaolinite}}$: Molar weight of kaolinite

M_{H_2O} : Molar weight of water.

n_{H_2O} : Number of moles of water released during the dehydroxylation of kaolinite.

For $M_{\text{kaolinite}}$ and n_{H_2O} values, the stoichiometry of kaolinite in Eq(2) was retained for the dehydroxylation.

The degree of dehydroxylation of kaolinite D_{TG} was made by comparing the amount of kaolinite in the calcined materials and on the raw material according to the following Eq(13):

$$D_{TG} = \left(\frac{Q_{\text{mineral kaolinite}} - Q_{\text{calcined kaolinite}}}{Q_{\text{mineral kaolinite}}} \right) * 100\% \quad (13)$$

With:

$Q_{\text{mineral kaolinite}}$: Amount of the mineral kaolinite in the raw material.

$Q_{\text{calcined kaolinite}}$: Amount of kaolinite in the calcined material.

- The metallic trace elements in the materials is measured after leaching test according to the standard NF EN 12457-2 [32]. A fraction of aggregates (0-4 mm) was mixed with water with a liquid/solid ratio = 10 L/kg. After 24 hours of leaching, the leachate was filtered at 45 µm, then 2% of HNO_3 acid at a concentration of 63% were added. The chemical analysis is performed by inductively coupled plasma optical emission spectrometer (IPC - OES 5100 Agilent Technologies). Anionic elements were analyzed by chromatography and without acidification. The leaching limit values for inert waste (IW) and non-hazardous waste (NHW) specified in Directive 1999/31/EC were used to verify material compliance.

2.2.2 Methods for evaluating the pozzolanic reactivity of materials

The evaluation of pozzolanic reactivity of calcination products can be carried out using various chemical and mechanical methods. The principle consists in determining the content of $Ca(OH)_2$ consumed by reactive components in pozzolanic materials [33].

a. Frattini's test (NF EN 196-5 [34])

The Frattini's test was used in previous studies [11, 30, 35] to assess the pozzolanic reactivity of materials. The essay consists to react 16 grs of CEM I cement and 4 grs of material in 100 ml of distilled water. Then, the mixture was kept tightly at 40 °C for at least 8 days or 15 days. The samples were filtered under vacuum and the filtrates were subsequently analyzed. First, the OH^- ions content was determined using the HCl 0.1N hydrochloric acid . After adjusting the pH to 12.5, the Ca^{2+} ion content was determined by the use of an EDTA complexometric test. The Ca^{2+} content (expressed as CaO equivalent) function of OH^- content are shown relative to the limit of solubility of $Ca(OH)_2$. A position under the curve of $Ca(OH)_2$ solubility suggests a consumption of CaO by pozzolanic reaction [30].

b. Thermogravimetric analysis of mixture with $Ca(OH)_2$

In this method, the consumption of lime by the pozzolanic reaction in the lime paste is evaluated by thermogravimetric analysis (TGA) for the differents materials (flash calcinated sediments as well as metakaolin and the raw sediment) . The $Ca(OH)_2$ /mineral addition mass ratio is 4 and the amount of water was adjusted to have a good consistency of the pastes. Table 3 shows the composition of the different lime pastes studied.

The lime pastes were stored hermetically at 20 °C until the test was measured at 7, 14, and 28 days. Before testing, the hydration of the samples should be stopped. Therefore, the pastes were immersed in acetone solution for 4 days, then filtered under vacuum before the thermogravimetrie analysis.

Determination of the amount of chemically bound water and the $Ca(OH)_2$ content by the TGA curve

The quantity of chemically bound water (wt %) could be calculated from the TGA curve according to the following Eq (14):

$$Q_{\text{chemical bound water}} = M_{\text{sample}}(40^\circ C) - M_{\text{sample}}(400^\circ C) \quad (14)$$

With:

$Q_{\text{chemical bound water}}$: Amount of chemical bound water (wt %).

M_{sample (40°C)}: Mass of the sample at 40°C.

M_{sample (400°C)}: Mass of sample at 400°C.

The amount of Ca(OH)₂ can be calculated from the TGA curve. In this study, the dehydroxylation of portlandite occurs at the temperature range of 450 °C to 550 °C. The amount of portlandite can be calculated according to the following Eq (15):

$$Q_{Ca(OH)_2} = \frac{\Delta m_{450^\circ C-550^\circ C(t)} \cdot M_{Ca(OH)_2}}{MH_2O} \quad (15)$$

with:

Q_{Ca(OH)₂}: Amount of Ca(OH)₂ in the paste (wt %).

M_{CH}: Molar mass of Ca(OH)₂.

M_{H₂O}: Molar mass of water.

The amount of Ca(OH)₂ reacted was estimated using the following Eq (16) and Eq (17):

$$Ca(OH)_2 \text{ reacted} = Ca(OH)_2 \text{ total} - Ca(OH)_2 \text{ non reacted} \quad (16)$$

$$Ca(OH)_2 \text{ non reacted} = \frac{\Delta m_{450^\circ C-550^\circ C(t)} \cdot M_{Ca(OH)_2}}{MH_2O} + Ca(OH)_2 \text{ carbonated} \quad (17)$$

Determination of the quantity of hydrated phases (C-S-H, CASH) from the DTG derived curve

The hydrated phases as C-S-H, calcium aluminate hydrate (CAH) or calcium silico-aluminate hydrate (CASH) can be calculated from the DTG derived curve [36, 37]. However, it is difficult to distinguish two phases CAH and CASH. The spectra obtained from the curves derived from dTG(%) / dt = f(T °C) were deconvolved in order to determine the area of each hydrate decomposition according to the Lorentzian's area deconvolution method with the amplitude a₀, center a₁ and width a₂ according to the following Eq (18) [38]:

$$\frac{dTG(\%)}{dt} = \frac{a_0}{1 + \left(\frac{T - a_1}{a_2}\right)^2} \quad (18)$$

with:

dTG(%) / dt: Derived value of TG (%) at time t.

T (°C): Temperature (°C).

a₀, a₁ and a₂ : amplitude, centre and width (>0) of the spectrum respectively.

Fig. 2 illustrates the method of quantification for C-S-H and CASH.

c. Heat of hydration of cement pastes

The pozzolanic reaction is an exothermic reaction. Isothermal calorimetry analysis is used to follow the evolution of heat from exothermic hydration reactions in different cement paste mixes. Cement paste mixtures with a 10% wt substitution rate of the addition, are made with a water/binder ratio equal to 0.5. The internal temperature of the calorimeter is set at 20 °C.

d. Mechanical performance of mortars

Prismatic samples mortars 4x4x16 cm³ are made in accordance with the standard NF EN 196-1 [26]. The compressive strength of the mortars are measured at 1, 2, 28, 56, and 90 days of curing by an INSTRON 5500 R 4206 - 006 Press. Table 4 shows the different mortar formulas with a 10% wt substitution rate made in this study.

In order to demonstrate the effect of mineral additions on the compressive strength of mortars, we used Féret's law [39] presented in Eq(19), which predicts the compressive strength of a cementitious material as a function of the content of the components (aggregates, sands, cement, mineral additions, water) and the content of the occluded air.

$$R_c(t) = G \cdot R_{cm} \cdot \left(\frac{1}{1 + p_c \cdot \left(\frac{E_{eff} + p_w \cdot V_a}{C + kA} \right)} \right)^2 \quad (19)$$

with:

G : Aggregate coefficient.

R_{cm} : Cement class (Mpa)

p_c : Cement density (t/m³)

E_{eff}: Amount of water in the formulation (kg/m³)

C : Amount of cement in the formulation (kg/m³)

A : Amount of minerale addition in the formulation (kg/m³)

V_a: Air content of occluded in 1 m³

K : Activity coefficient of mineral addition

The objective is to establish a dilution curve of the compressive strength of the mortars that we consider only the cement participating in the development of the compressive strength and without the contribution of the effect of mineral additions (neither physical effect, nor chemical effect). In this case, we assume the activity coefficients of mineral additions are equal to 0 ($k = 0$) and the compressive strength of MR mortar was used in order to establish the dilution curve of the compressive strength of mortars (MMK, MRS, MSF 650, MSF 750, and MSF 800) in hydration time.

The difference between the resistance measured on the sample (experimental result) and the theoretical resistance calculated according to Fret's law gives the effect of the additions in the formulation.

- A negative deviation means that the mineral additions would have a detrimental effect on the development of compressive strength.
- A zero deviation signifies mineral additions are inert and do not contribute to the development of compressive strength.
- A positive deviation means that the mineral additions would have an effect on the development of resistance. An increase in the time difference could be due to the effect of the pozzolanic reaction.

e. Measurement of dynamic modulus of elasticity E_{dyn} :

The dynamic modulus of elasticity of the mortars was determined by the equipment GrindoSonic Mk5 "Industrial". It is a non-destructive test that assesses microcracking, homogeneity, and compactness [40]. The dynamic modulus of elasticity is measured according to the following Eq(20):

$$E_{dyn} = \frac{\rho(1+\mu)(1-2\mu)}{(1-\mu)} * f^2 \quad (20)$$

With:

ρ : Density of the mortar (kg/m^3)

μ : Dynamic Poisson's ratio

f : Pulse velocity of mortar (m/s)

f. Porosity of mortars

The microstructure of the mortars was studied by measuring the porosity of the mortars in order to assess the effect of incorporating mineral additions. Indeed, the porosity can have an influence on the durability [41] and on the mechanical resistance[42]. In particular, the larger capillary pores strongly influence the transfer properties of concrete [43]. The porosity and the pore size distribution of the mortars were measured at 56 days of hydration using Mercury Intrusion Porosimetry (MIP) technique (Micromeritics Autopore IV type).

2.2.3 Leach analysis of mortars

Leach analysis was performed on particles (0–4 mm) from the mortars after 90 days to measure the content of metallic trace elements and anionic elements. The analysis process is identical to that used in the material characterization part.

3 Results and Discussion

3.1 The physical characteristics of materials

Table 5 shows the physical characteristics of the materials used in the study. The RS sediment has a high content of organic matter (16.1%). Flash calcination significantly removes organic matter from products, especially in SF 750 and SF 800 where a reduction of 88% and 93% is respectively observed.

The increase in density was also observed for the calcination products. This is due to the elimination of organic matter (density 1 g/cm^3) and the decomposition of limestone (density $2.6\text{-}2.8 \text{ g/m}^3$) to form calcium oxide (density approximately 3.3 g/cm^3). However, a decrease in the density of SF 800 upon increasing the calcination temperature could be due to the recrystallization of the mineral phases [44]. The flash calcination also induces a slight increase in particle size without a reduction in specific surfaces. This is due to an agglomeration of clay particles without sintering during calcination [31]. The increase in particle size in the calcined sediment was also observed in previous studies [11, 15, 45]. Concerning the reactivity of the addition, the latter increases when the BET surface increases, in contrary to the water demand. However, an increase in calcination temperature to 800°C led to a decrease in the specific surface. This could be explained by the melting of certain particles that contain elements such as Na, K, and Fe reacting like fluxes and sintering during calcination at high temperatures [15, 31].

From the TGA - DTG analysis of the sediments (Fig.3 and Fig.4), several operations can be carried out in order to follow the phase transformation:

- **Calcite content (CaCO_3)**

The CaCO_3 content can be calculated from the TGA analysis according to the following Eq (21) and Eq (22):

$$\% \text{CaCO}_3 = (m_{\text{sample } 600^\circ\text{C}} - m_{\text{sample } 800^\circ\text{C}}) * (M_{\text{CaCO}_3} / M_{\text{CO}_2}) \quad (22)$$

With:

$M_{\text{sample } 600^\circ\text{C}}$: Mass of sample at 600 °C

$M_{\text{sample } 800^\circ\text{C}}$: Mass of the sample at 800 °C

M_{CaCO_3} : Molar mass of CaCO_3

M_{CO_2} : Molar mass of CO_2

Table 6 shows the content as well as the degree of decarbonation of calcite in the sediments.

The presence of CaCO_3 and CaO in the calcined products could have impacts on the hydration of calcined sediments based mixtures. In fact, mineral additions with a high CaCO_3 content promote hydration of C_3S [46], but are very unfavorable for the C_3A phases hydration [47].

On contact with water, CaO turns into Ca(OH)_2 according to the following reaction Eq (23) [48]:

This reaction is exothermic and this could cause a strong degradation due to the heat generated in the core of the structure. In addition, an increase in the Ca(OH)_2 volume could be the cause of cracking which influenced the durability and the mechanical performance of the structure.

- **Degree of dehydroxylation of kaolinite**

The content and degree of dehydroxylation of kaolinite (Table 7) can be estimated from the results of TGA analysis according to Eq (12) and Eq (13).

The result shows that the kaolinite was transformed into metakaolin Eq(2) from calcination at 650 °C, however the degree of dehydroxylation is relatively low (31%). The increase in calcination temperature significantly improves the dehydroxylation of kaolinite. The degree of dehydroxylation of kaonilite is an important index for evaluating the quality of calcination because the metakaolin formation promotes the pozzolanic reaction.

3.2 Mineralogical and chemical characterizations

The XRD analysis shows the presence of the main mineral phases in the RS, SF 650, SF 750, and SF 800 sediments (Fig.5). The results indicate that :

- The quartz is the major crystalline phase identified in the RS sediment as well as in the calcined sediments.
- The presence of calcite is well identified in the RS sediment. An increase in calcination temperature led to a decrease in the calcite content in the calcined products due to the decomposition of this phase.
- The formation of anhydrite phase in the calcined sediments has been well identified. This could be explained by the reaction between the CaO released from the decomposition of CaCO_3 with the sulfate to form the anhydrous calcium sulfate (CaSO_4) [22].

The result of the analysis on oriented slides showed that the clay phases of the RS sediment consist mainly of kaolinite and illite (Fig.6). After calcination, the kaolinite transforms into metakaolin due to a dehydroxylation reaction. The degree of dehydroxylation at 800 °C is the highest and this seems in accordance with the result calculated from the TGA analysis (Table 7). Despite calcination temperatures are higher than the dehydroxylation temperature of kaolinite but the calcination time is very short, this is explained by the incomplete reaction during calcination of the sediment at 650 °C, 750 °C and 800 °C.

The chemical composition of sediments as well as OPC cement and MK is given in Table 8.

- The major oxides of materials are SiO_2 , Al_2O_3 , CaO , Fe_2O_3 .
- The chemical composition of calcined sediments is relatively similar. Therefore the increase in the calcination temperature did not change the composition in the calcined products. Fig.7 shows the content of major oxides in the materials in the $\text{CaO-SiO}_2\text{-Al}_2\text{O}_3$ ternary diagram.

The result of the leaching analysis shows the presence of metallic trace elements as well as anionic elements in sediments (Table 9 and Table 10). The content of certain metallic trace elements in the RS sediment exceeds the value specified for inert waste (IW), in particular, the content of zinc.

By comparing the calcined sediments to the raw sediment, it can be seen that the content of some metals such as Cu, Ni and Zn has decreased. This could have beneficial impacts on the hydration of the cementitious matrix incorporating calcined sediments, in particular for zinc. The presence of Zn decreases the compressive strength and reduces the density of the material [49]. In fact Zn^{2+} ions combined with OH^- ions to form an impermeable Zn(OH)_2 film which inhibits the hydration of C_3S and C_3A with a more pronounced effect on C_3S . In addition, Zn(OH)^{2-} and Zn(OH)^{3-} prevent adsorption to the electronegative surface of C-S-H at high pH.

The sulfates content in the RS sediment is higher than the limit value for IW. In addition, it can also be seen from the results that the RS sediment has a higher sulfate content compared to the calcined sediments, where the sulfate

content decreases with the increase in temperature. The presence of sulfate under certain conditions can cause degradation of materials, such as: significant swelling, cracking, and a decrease in mechanical strength. The presence of chlorides accelerates the setting and hardening of the cementitious matrix. Chlorides can also combine with C₃A in the cement to form chloro-aluminate causing a swelling. The chloride content in the sediments seems to remain unchanged during calcination. Similar results were found in the study of Ruben et al [15].

3.3 Results of pozzolanic reactivity analyzes

3.3.1 The Frattini test

The Frattini's test was performed on samples after 15 days of hydration. The result (Fig.8) shows that, after 15 days, the calcined sediments and the MK have pozzolanic reactivity while the RS sediment does not exhibit pozzolanic reactivity. This is due to the formation of metakaolin in the calcined sediments during the dehydroxylation of kaolinite. This transformation was demonstrated in the XRD analysis (Fig.5) and the analysis on oriented slides (Fig.6). In addition, Fig.8 shows that for MK, the pozzolanic reaction could be clearly detected after 8 days.

3.3.2 Heat hydration of cement pastes

The heat of hydration of cement pastes is shown in the Fig. 9. The first exothermic peak in the acceleration period (point 2) is mainly related to the hydration of C₃S and C₃A to form C-S-H and ettringite respectively. In the deceleration period, the second exothermic peak is observed, which corresponds to the additional dissolution of C₃A and the accelerated precipitation of ettringite [50]. In addition, the depletion of calcium sulfate CaSO₄ led to the transformation of ettringite to Afm (C₃A.CaSO₄.12H₂O). The results show also that in the mixtures OPC 52.5, (OPC 52.5 + MK), (OPC 52.5 + SF 650), (OPC 52.5 + SF 750) and (OPC 52.5 + SF 800), the induction period begins after 3h while this period begins only after 6h in the mixture (OPC 52.5 + RS). This delay could be explained by the high organic matter content in the RS sediment that can disturb the hydration of the cement. Indeed, Young [51] indicated that the formation of complexes between Ca²⁺ ions and groups such as hydroxyl, carboxyl, amine, benzene releases protons that provokes the acidifying of the matrix according to the following reaction Eq (24):

In addition, Pollard et al. [52] showed that the formation of a physical barrier altered the growth and/or the morphology of the crystals and prevented the normal hydration of the cement. Indeed, the reduction of Ca²⁺ ions retarded the nucleation of Ca(OH)₂ and generated an unstable C-S-H gel with a low Ca/Si ratio which slowly transformed into a stable C-S-H gel [53]. Agglomeration of sediment may be the cause of delay in hydration because it trapped water in the mix and reduced the water available for hydration [54]. Removal of organic matter from calcined sediment is beneficial by reducing the delay in hydration of the mixture. Metakaolin has the highest pozzolanic reactivity while the three calcined sediments have relatively identical hydration kinetics. This result appears to be consistent with that of Frattini's test.

The incorporation of the calcined sediment reduces the heat of hydration of the mixtures. This result is contrary to that reported in the study by Benzerzour et al [9]. However, a similar result was obtained in the study of Safhi et al [55]. This contradiction could be explained by the coarser size (d₅₀ about 15 µm) of the sediments in this study and the study conducted by Safhi et al [55] compared to that in the study of Benzerzour et al [9] (d₅₀ = 5 µm). Therefore, this could lead to higher hydration kinetics.

3.3.3 Determination of pozzolanic reactivity by thermal analysis

a. Quantification of chemically bound water and Ca(OH)₂ from TGA analysis

Fig.10 shows, an example for the method of calculating the content of bound water in the (Ca(OH)₂+SF 750) mixture by TGA analysis.

From the TGA analysis results of (Ca(OH)₂+SF 750) mixture, the following conclusions can be extract :

- The content of Ca(OH)₂ in the mixture decreases with time, due to the pozzolanic reaction between the sediment and the portlandite.
- An increase in the bound water content in the mixture corresponds to hydrates formed over the time of hydration process. This is consistent with the observed Ca(OH)₂ consumption.
- The CaCO₃ content in the mixture at each test time (7, 14 and 28 days) is constant. This stability means that the mixture was well homogenized and that the used sample of the mixture is well representative.

The amount of chemically bound water of the mixtures during hydration calculated using Eq (14) is shown in Fig.11. The (Ca(OH)₂+MK) mixture showed the highest amount of bound water at all measured times. This is related to the amount of hydrates formed in the paste over the time of hydration. At 7 days, the amount of water bound in mixtures containing sediment is relatively the same. This could be due to the slow pozzolanic reactivity of the calcined sediments. Indeed, after 28 days of hydration, the content of bound water increases in all mixtures. Pastes containing calcined sediment exhibited a higher amount of bound water than the paste incorporating raw sediment.

Among the calcined products, the mixture with SF 750 sediment leads to a higher bounded water content at short term as well as at long term. This result is in accordance with previous tests on the pozzolanic reactivity of sediments.

b. Ca(OH)_2 amount in lime paste

The pozzolanic reaction is a reaction that consumes Ca(OH)_2 , therefore this pozzolanic reaction can be evaluated by measuring the Ca(OH)_2 over time of hydration. Fig.12 shows the evolution of Ca(OH)_2 in the different mixtures. It can be seen from the results that the MK consumes a greater quantity of Ca(OH)_2 than the sediments. Among the sediments, SF 750 seems to be the most reactive when comparing the quantity of Ca(OH)_2 consumed at short term as well as at long term. In addition, at short term, the Ca(OH)_2 content is relatively stable. This is consistent with the result of the amount of water chemically bound. In fact, the more portlandite is consumed, the more hydrates are formed.

In order to demonstrate the consumption of Ca(OH)_2 in the mixtures, XRD analysis was performed on the samples after 28 days of hydration. The result has been presented in Fig.13. We can confirm that:

- The Ca(OH)_2 content in the (MK + Ca(OH)_2) mixture after 28 days of hydration is the lowest. This means that the Ca(OH)_2 content consumed in the mixture is the highest. This result is consistent with the result measured by the TGA analysis (Fig.12).
- The Ca(OH)_2 content in the mixtures contained in the calcined sediments is relatively similar and lower than the (RS + Ca(OH)_2) mixture. This result is also consistent with the result found by the TGA analysis (Fig.12).

c. Evaluation of hydrates formed from the DTG curve

The relative amount of hydrates formed over hydration time of the pastes was estimated from the DTG curves by the deconvolution method based on Eq (16). Fig.14 and Fig.15 show the relative amounts of the C-S-H and other phases such as CASH produced in pastes over time of hydration. By comparing the relative amount of hydrates formed (C-S-H and CASH), it can be concluded that the MK is the most reactive comparing to the other additions. This result appears to be in accordance with previous results, that demonstrated the higher reactivity of MK with respect to sediments. However, we note that the relative amount of C-S-H at 28 days in all mixtures are relatively the same. In addition, the CASH amount formed from the (Ca(OH)_2 +MK) mixture is much higher than the other mixtures incorporating sediments. This can be explained by the higher Al_2O_3 content of MK (31.18% wt) compared to sediment (12 wt.% in SF 650, SF 750, SF 800, and 9.64 wt.% in RS).

The reaction for C-S-H formation occurs relatively slowly in mixtures containing sediments for up to 14 days. This is well consistent with the quantification result of the Ca(OH)_2 amount consumed and the content of the chemically bound water by the TGA analysis (Fig. 11). For example, the amount of Ca(OH)_2 consumed in the (Ca(OH)_2 + SF 750) mixture incorporating SF 750 is 8.63% at 7 days and 10.90% at 14 days compared to 64.83% at 28 days. The result also showed that SF 750 was the most reactive among sediments by comparing the amount of hydrates formed.

3.4 Compressive strength of mortars

Fig.16 shows the evolution of the compressive strength of mortars in function of the hydration time (from 1 to 90 days of curing). The following observations can be dressed:

- MMK mortar exhibits good mechanical performance compared to MR mortar at all testing times. This proves the highest pozzolanic reactivity of MK.
- MRS mortars have the lowest compressive strengths. Whereas the calcined sediments appear to significantly improve the mechanical performance of mortars when compared with the strength of mortar MRS.
- Among the mortars containing calcined sediments, MSF 750 mortar shows the better mechanical performance. An increase in the calcination temperature seems to improve the mechanical performances of mortars by increasing the specific surface of the sediments. However, calcination at 800 °C reduces the specific surface of the SF 800 sediment compared to SF 750 and this explains the reduction in the strength of MSF 800 mortars compared to MSF 750.

Fig.17 and Fig.18 show the resistance measured on the specimen and the resistance calculated according to Fret's law (with the activity coefficient $k = 0$) of the mortars over time of hydration.

The result shows that:

- The compressive strength of MRS mortar is always below the theoretical strength curve. This shows a detrimental retardation effect of RS sediment on resistance development. Indeed, the delay effect of RS sediment was also shown in the isothermal calorimetry test (Fig.9).
- The difference in resistance of MSF 650, MSF 750 and MSF 800 mortars is almost zero after 1 day of hydration. This means that these calcined sediments are inert and do not contribute to the development of resistance. However, after 2 days of hydration, the difference is positive. This difference seems to get bigger and bigger with the time of hydration. This means that these sediments would have a chemical effect such as the pozzolanic reaction contributing to the development of resistance.

- The compressive strength of MMK mortar is always above the theoretical strength curve. An increase in this difference in hydration time has been observed. In addition, this difference is also greater than the difference in the case of mortars containing calcined sediment. This means the MK would have a higher pozzolanic reactivity than the calcined sediments. This result is consistent with the result shown in the analysis of isothermal calorimetry (Fig.9) and lime consumption (Fig.12).

3.5 Dynamic modulus of mortars

Fig.19 shows the evolution of the dynamic modulus of mortars calculated according to Eq (20) from 28 days to 90 days. Stabilization of the dynamic modulus after 56 days indicates that all of the portlandite has been consumed by the pozzolanic reaction [55]. Indeed, the consumption of portlandite makes it possible to form a more dense and coherent microstructure. In addition, a relatively large increase in dynamic modulus of sediment containing mortars between 28 and 56 days compared to MMK mortar indicates the long-term pozzolanic reactivity of the sediments.

3.6 Porosity of mortars

The mercury porosity of mortars measured at 56 days is shown in Table 11 and the pore distribution is also shown in the Fig.20.

The results show that:

- Substitution of cement by RS sediment increased the porosity of the mortar. Indeed, the result of the compressive strength indicated that the RS sediment was almost inert after 90 days of hydration. Replacing cement with an inert addition leads to an increase in the W/C ratio. This could lead to an increase in porosity. The pore distribution in the MRS mortar returns to the larger pores. A similar result was found in the research of Berodier et al [56] when replacing cement with quartz, considered inert.
- Contrary to RS sediment, replacing cement with active mineral additions (MK, SF 650, SF 750 and SF 800) reduced the porosity of the mortar. The pozzolanic reaction appears to overcome the replacement dilution effect. In addition, these mineral additions appear to reduce the size of the pores towards the finer pores. According to Zengfeng et al [57], the presence of the sediment allowed a finer distribution of the porosity, most often associated with a better texture of C-S-H.

3.7 Leach analysis result of mortars at 90 days of curing

Table 12 shows the content of metallic trace elements in the mortars after 90 days of hydration. In addition, the content of anionic elements in the mortars was shown in Table 13. A considerable reduction in the sulfate content was observed. The result showed a stabilization of all the elements compared to the leaching limits of inert waste.

3.8 Analysis of the quality of the exhaust fumes

The quality of the gases is an important indicator to assess the feasibility of the calcination process. The gases emitted during calcination of the sediment were analyzed by the company Socor air. The results of certain elements in the flue gases are presented in Table 14.

The results in Table 14 leads to tell that the gas content (O_2 , CO_2) stabilizes during calcination. An increase in the calcination temperature allows the reduction of the CO gas emissions by reducing it by 97.6% for the temperature of 750 °C comparing to 650 °C . The similar trend with NO_x gases was observed with increasing calcination temperature.

4 Conclusion

The objective of this article is to study the influence of the calcination temperature of sediment on their properties. This consists to evaluate several criteria such as the activation of pozzolanic reactivity, mechanical performance, energy consumption, and the quality of the gases emitted. Besides, the effects of the use of calcined sediments on the kinetics of hydration were investigated. It can be concluded from this study that:

- Flash calcination method removes the fraction of organic matter already present in the raw sediment. This reduces the delay in hydration related to the presence of organic material.
- The calcination temperature modifies the physical properties comparing to the raw sediment. Indeed, calcined sediments have a greater BET specific surface and density than the raw sediment. The transformation of kaolinite to metakaolin and the formation of anhydrite in calcined sediments were observed by XRD analysis.
- The flash calcination also active the pozzolanic reactivity of the sediment. The calcined sediments showed a strong ability to react with portlandite to form hydrates compared to the raw sediment.
- The heat of hydration of binders containing calcined sediment is greater than that of the binder with raw sediment. However, the calcined sediment reduces the heat of hydration of the binder compared to the reference cement. This could have a positive effect in the case of concrete and a negative effect in the development of strength at an early age.
- The incorporation of the calcined sediment significantly improved the mechanical performance of the mortars compared to the raw sediment. Besides, the reducing in the hydration delay with calcined sediments

improves the development of mechanical strength at young ages comparing to the raw sediment. MSF mortars have similar mechanical strength as the reference mortar at long-term.

- The incorporation of calcined sediments reduces the porosity of the mortar due to the pozzolanic reaction.
- An increase in the calcination temperature from 650 °C to 750 °C improves the quality of the gases emitted by comparing the content of the elements in the emitted gases. An increase in the calcination temperature allows the CO gas content to be greatly reduced (97.6% reduction).

- The content of some metallic trace elements was reduced in calcined sediment, especially zinc content. In addition, the content of flourures, chlorides and sulfates tends to decrease with increasing calcination temperature.

Declaration of competing interest

The authors declare that they no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data Availability

The datasets generated during and/or analysed during the current study are available from the corresponding author on reasonable request.

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Figures

Figure 1

Flash calcination process representation (CERI MP at IMT Nord -Europe)

Figure 2

Deconvolution method for quantification the quantity of C-S-H and CASH phases of the (SF 750+ $Ca(OH)_2$) mixture after 28 days of hydration

Figure 3

TGA curves of sediments

Figure 4

DTG curves of sediments

Figure 5

XDR patterns of sediments (1) Quartz, (2) Kaolinite, (3) Illite, (4) Anhydrite, (5) Calcite, (6) Albite

Figure 6

XRD patterns of clay phases of sediments using the XRD analysis on the oriented slides (K: Kaolinite, I: Illite)

Figure 7

CaO-SiO₂-Al₂O₃ ternary diagram of materials

Figure 8

The Frattini's test result after 15 days of hydration

Figure 9

Heat of hydration generated during the hydration of cement pastes with the ratio $W/B = 0.5$

Figure 10

Result of the TGA analysis of the ($\text{Ca(OH)}_2 + \text{SF750}$) mixture over time of hydration

Figure 11

Content of chemically bound water in lime pastes during hydration using the TGA analysis

Figure 12

Evolution of the Ca(OH)₂ amount in lime pastes over time of hydration using the TGA analysis

Figure 13

XRD patterns of mixtures after 28 days of hydration (1: Stratlingite ($\text{Ca}_2\text{Al}_2\text{SiO}_7 \cdot 0.8\text{H}_2\text{O}$), 2: Ettringite, 3: Ca(OH)_2 , 4: Quartz, 5: Calcite)

Figure 14

Relative amount of C-S-H in the different pastes over time of hydration using the deconvolution method

Figure 15

Relative amount of CASH phases of the pastes over the time of hydration using the deconvolution method

Figure 16

Compressive strength of mortars over time of hydration

Figure 17

Compressive strength measured on the sample and the compressive strength calculated according to Fret's law with the activity coefficient $k = 0$ after 1 and 2 days of curing

Figure 18

Compressive strength measured on the sample and the compressive strength calculated according to Fret's law with the activity coefficient $k = 0$ after 28 and 90 days of curing

Figure 19

Evolution of the dynamic modulus of mortars over time of hydration

Figure 20

Pore size distribution in mortars measured by MIP at 56 days of curing