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# Dredged sediments used as novel supply of raw material to produce Portland cement clinker

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## ABSTRACT

The maintenance of waterways generates large amounts of dredged sediments that are an environmental issue. This paper focuses on the use of fluvial sediment to replace a portion of the raw materials of Portland cement clinker, which would otherwise come from natural resources. The mineralogy of the synthetic cement was characterised using X-ray diffraction and scanning electron microscopy and its reactivity was followed by isothermal calorimetry. Comparisons were made to a commercial ordinary Portland cement (CEM I 52.5). Compressive strength measurements were conducted on cement pastes at 1, 2, 4, 7, 14, 28 and 56 days to study strength development. The results showed that Portland cement clinker can be successfully synthesised by using up to 39% sediment. The compressive strengths developed by the cement made from sediment were equivalent to those obtained with the reference at early ages and 20% higher at long term.

## 1. Introduction

With more than 10 billion metric tons of concrete being produced worldwide per year, the cement industry has a large environmental footprint. The tools and strategies to meet the environmental challenges should involve the use of recycled materials in place of natural resources. This study focuses on the recycling of sediment to replace a portion of raw materials in conventional Portland cement manufacture. Waterway sediments are dredged either to maintain navigability, prevent flooding or, more recently, to improve the environmental quality of the aquatic ecosystem. Presently, in France about 60% of waterway-dredged sediments are deposed in dedicated sites. However, this style of management is unacceptable to most local communities. In addition, several technologies, including the thermal elimination of organic matter, the stabilisation of heavy metals [1], mineral processing [2] and phytoremediation [3] have been explored either to treat sediments or to manage the risk of pollution transfer to surrounding environments. Most of these treatments are under validation but are either too expensive or not available at this stage.

From the viewpoint of resource recovery and recycling, the use of by-products or wastes as raw materials for cement production is of interest and has been widely investigated [4–13]. Judging by the chemical composition, sediment could also be used as substitute for cement raw materials. Dredged sediments present the advantages of a renewable resource and are available in huge quantities, easily transportable through waterways, which is in accordance with cement industry needs. Research conducted on the valorisation of sediment in cement-based materials has mostly focused on the classical sand substitution for mortar production [1 and citations therein]. This type of recovery requires treated or unpolluted sediment. Dalton et al. [14] demonstrate the potential of using contaminated marine sediment as feedstock replacement in Portland cement manufacture. A bench scale manufacture was carried out with feedstock mixtures containing up to 12% of dredged material from the New York/New Jersey harbour. Results showed a decrease of alite and increase of belite for contents higher than 6%. In addition, this study treated marine sediment with a high chloride content, which causes two difficulties. First, chloride trapped in the clinker is well known to reduce the concrete strength and may speed up the corrosion of reinforcing steel in concrete [15]. Second, large fractions of chloride are volatilised at approximately 980 °C and travel up the kiln to cooler regions to finally precipitate along the kiln walls. Thus, periodic maintenance is needed, and production time is lost [16].

Here we present the use of fluvial contaminated sediment to replace a portion of raw materials in conventional Portland cement manufacture. The sediment is classified as contaminated because of the presence of organic and inorganic contaminants. At the high temperature required for cement manufacture (1450 °C), organic contaminants are degraded, and heavy metals are stabilised [7–19]. In addition, previous research indicates that the heavy

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metals present in wastes and alternative fuels used in clinker kilns are incorporated into the clinker crystalline phases [20,21], and cement producers have extensive experience with their impact on concrete quality and cement production [17,22].

This use of sediment is an advantageous use of existing technologies to manage dredged fluvial sediment and leads to a reduction of the cement industry demand for raw materials. In addition, cement producers can be paid for using the polluted sediments and developing a sustainable way to reuse significant amounts of sediment. The goal of this study was first to synthesise Portland cement with the highest possible content of polluted sediment and secondly to compare its reactivity to commercial Ordinary Portland Cement (OPC) CEM I 52.5. The crystalline phases of the resultant cement clinker and cement pastes were identified by X-ray powder diffraction. SEM–EDS was used to investigate the clinker. The hydration kinetics was followed by isothermal calorimetry and by measurements of the compressive strength of hardened cement pastes.

## 2. Traditional raw materials and cement production process

The clinker/cement production contains three main stages: raw material milling, clinkerisation and the final milling of the clinker mixed with gypsum and other additives [15]. Limestone rock (∼80%) and clay (∼20%) are the main raw materials. Limestone decomposes to CaO and $CO_2$ during firing. Clay contains mainly three oxides: $SiO_2$, $Al_2O_3$ and $Fe_2O_3$, which react with CaO at high temperature. In addition, depending on the raw material compositions, correction materials could be needed. High purity limestone, silica and iron-rich clays are the most widely used materials [12]. High temperatures (1450 °C) are required to convert raw materials to the clinker composed of alite ($3CaO{\cdot}SiO_2$), belite ($2CaO{\cdot}SiO_2$), tricalcium aluminate ($3CaO{\cdot}Al_2O_3$) and tetracalcium aluminoferrite ($4CaO{\cdot}Al_2O_3{\cdot}Fe_2O_3$), abbreviated respectively as $C_3S$, $C_2S$, $C_3A$ and $C_4AF$.

Clinker can be manufactured using either a wet or dry process. With the wet process, the raw materials enter the kiln in a wet slurry form. This process requires more energy and a large amount of water and, consequently, currently is used less than the dry process. Therefore, wet process plants may be better suited than dry process plants for accepting dredged sediments due to their water content.

## 3. Materials and methods

### 3.1. Materials

Sediment was collected by dredging the Scarpe Canal, located in the industrial basin of the North of France. This area was largely impacted by past mining activities and pyrometallurgical industries that emitted particles and slags enriched in metals. Emissions resulted in diffuse and localised contaminations of the riverbed of the canal, for example, with cadmium (from 2 to 643 mg $kg^{-1}$ dry weight) or zinc in sediments (from 138 to 9847 mg $kg^{-1}$ dry weight) [23]. The sampling area was selected to obtain sediments representative of the industrial area that contained about 150 mg $kg^{-1}$ dry weight of Cd and 4000 mg $kg^{-1}$ dry weight of Zn.

The sediment was mixed and stored for draining under cover for several months to reduce its water content. Thereafter, the sediment was mechanically homogenised several times.

Sediment was dried to constant weight at 105 °C and then sieved. For this study, the fraction with particles smaller than 2 mm was used. This fraction was crushed by a jaw crusher and ground to reach a particle size smaller than 100 μm with a centrifugal ball mill. The loss on ignition was determined at 550 °C. The mineralogical composition was studied by X-ray diffraction (XRD Bruker D8 with Co Kα radiation 1.78 Å). The chemical composition of the sediment was analysed by microwave-assisted digestion followed by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES Varian 720-ES).

### 3.2. Sediment substitution and raw mix preparation

The substitution rate of sediment for traditional raw materials was determined by taking into account the sediment composition, the Bogue calculation, the lime saturation factor (LSF), the silica ratio (SR) and the alumina ratio (AR) [15]. The chemical analysis of sediment showed that the main part of calcium oxide was missing to produce a Portland cement. A previous study [24] pointed out that, for the best clinkering reaction at the laboratory scale, the clinker should be rich with $C_3A$ and $C_4AF$ to minimise free lime. Thus, 1% of $Fe_2O_3$ and 1.5% of $Al_2O_3$ were added. For the present study, the raw materials were polluted fluvial sediment and high purity calcium carbonate, and analytical grade $Fe_2O_3$ and $Al_2O_3$ were used as correction materials. To produce 1 kg of clinker from polluted sediment, the raw mix was composed of 0.391 kg of sediment, 1.103 kg of $CaCO_3$, 0.01 kg of $Fe_2O_3$ and 0.015 kg of $Al_2O_3$.

The raw mixes were homogenised and pressed at 5 kN into pellets (height = 30 mm, diameter = 40 mm) to obtain a more regular clinkering process. The temperature cycle, based on literature data and free lime minimisation, had a ramp of 20 °C/min to 1450 °C, dwelled at 1450 °C for 45 min, ramped at 15 °C/min to 1350 °C and was finally quenched to ambient temperature. The quenching step is important to simulate the industrial process and to prevent the decomposition of $C_3S$. The quantities of cement produced in the laboratory are limited to the size of the furnace available. The cement made from polluted sediment (CFPS) was produced by mixing pure gypsum with clinkers to reach 3% $SO_3$ and grinding to reach particle sizes smaller than 60 μm with a centrifugal ball mill.

### 3.3. Methods

The chemical composition of clinker was checked by X-ray fluorescence. The mineralogy of the cement and cement paste was studied by means of X-ray diffraction (XRD).

The chemical composition of the clinker phases was investigated on polished sections with a Hitachi S-4300SE/N Scanning Electron Microscope (SEM) operating in the backscattered electron mode (20 keV) and equipped with an energy dispersive X-ray spectrometer (EDS). To make the polished section, a piece of clinker was impregnated under vacuum in epoxy resin and then polished with ethanol to avoid reaction with water. Finally, the samples were carbon coated before observation.

The reactivity of the cements was determined through isothermal calorimetry measurements done at 20 °C with a water/cement (W/C) ratio of 0.5. 5 g of cement and 2.5 g of water previously stored at 20 °C were mixed manually for a few seconds outside the calorimeter directly in cells that were then placed inside the calorimeter. The calorimeter used was a homemade calorimeter using fluxmeters that allowed the calorimeter to equilibrate in less than two minutes. Thus, it was possible to record the heat flux released at the beginning of hydration even if the mixing of the cement paste was carried out in the calorimeter.

The cement paste microstructure was also observed through SEM analyses on polished sections after 14 days of curing. The hydration was stopped with isopropanol, and the samples were then dried for 12 h at 60 °C.

Because of the limited quantities of clinker produced in the laboratory, the compressive strength was determined on small cylinders (height = 12 mm, diameter = 6 mm) made by cement pastes with a W/C ratio equal to 0.45 according to a slightly modified method reported by Perez et al. [25,26]. Cement and water were manually mixed for 3 min. The paste was loaded into polytetrafluoroethylene (PTFE) moulds. The filling of the moulds was performed in two steps on a vibrating table. The moulds were kept for 24 h at 100% relative humidity at 20 °C. The paste cylinders were demoulded after 24 h, and curing was continued at 100% relative humidity and 20 °C. The compressive strength was measured on 10 samples at 1, 2, 4, 7, 14, 28 and 56 days. The tests were performed at a constant stress of 0.3 MPa/s using an uniaxial press. Thus, the values of the compressive strength obtained for the paste cylinders were not comparable to mortars made according to EN-206.

## 4. Results and discussion

### 4.1. Material compositions

Sediment X-ray diffraction analysis indicated that the most abundant crystalline minerals were quartz, calcite and illite. The loss on ignition of the sediment was 10.2%.

The chemical compositions of the sediment, the cement made from sediment (CFPS) and the commercial OPC are given in Table 1. The sediment and the two cements contained calcium, silicon, aluminium and iron. In this study, the silicon content in the sediment was larger, whereas the calcium content was smaller, than a typical cement raw mix. Thus, as described in Section 4.2, the CFPS contained 39.1 wt.% of sediment and 62.8% of limestone added as calcium carbonate. For an industrial application, this proportion can be modified to take into account the variability of sediment. Cement producers have extensive experience with the variability of a raw material and the use of correction products.

Table 1 shows that, apart from zinc, the chemical composition of CFPS is comparable to that of the commercial cement. This finding is still valid when compared to the average composition of Portland cement [20]. It is important to note that currently the average Zn concentration in Ordinary Portland Cement is equal to 0.0164%. Given that zinc induces a delay in cement hydration [27,28], one might ask about the hydration of CFPS, this phenomenon will be discussed later in Section 4.2. Only the mineralogical characterization and the SEM micrograph of the synthesized cement are presented in this section.

The XRD pattern of the sintered (CFPS) cement is shown in Fig. 1 together with the pattern for the commercial Ordinary Portland Cement (OPC). $C_3S$, $C_2S$, $C_3A$ and $C_4AF$, the major crystalline phases in OPC, are all present in the CFPS. These results indicate that the control of compositional parameters produced the target crystalline phases in our experimental conditions.

**Table 1**
Chemical compositions of sediment, cement from polluted sediment (CFPS) and commercial OPC.

| Chemical composition (%) | Sediment | CFPS | OPC |
|---|---|---|---|
| Ca0 | 5.77 | 65.28 | 62.05 |
| $SiO_2$ | 55.51 | 21.96 | 20.80 |
| $Al_2O_3$ | 6.52 | 4.21 | 3.91 |
| $Fe_2O_3$ | 3.29 | 2.25 | 2.16 |
| $Na_2O$ | 0.59 | 0.13 | 0.32 |
| MgO | 0.63 | 0.27 | 1.99 |
| $K_2O$ | 1.81 | 0.05 | 0.88 |
| P | 0.31 | 0.10 | 0.13 |
| S | 0.77 | 1.76 | 1.68 |
| Ni | 0.01 | ND | ND |
| Cu | 0.02 | 0.016 | ND |
| Zn | 0.41 | 0.158 | ND |
| Ba | 0.13 | ND | ND |
| Cd | 0.01 | ND | ND |
| Pb | 0.05 | ND | ND |
| ND < 0.01% | | | |

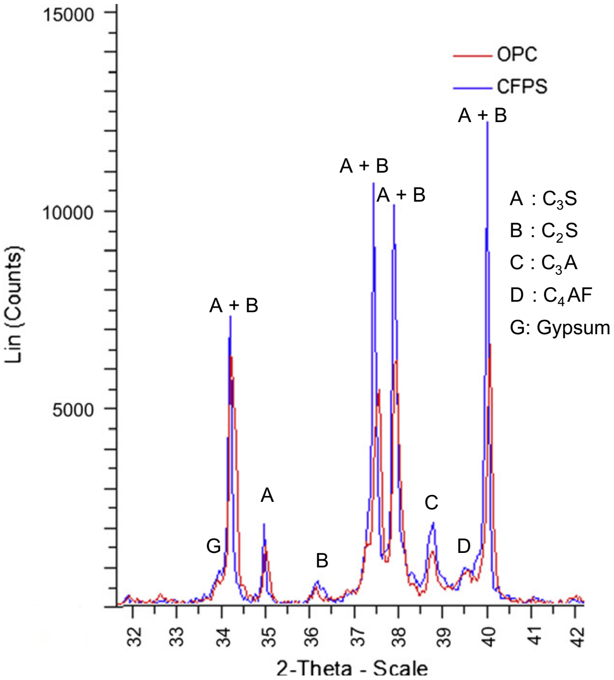

**Fig. 1.** XRD patterns of the cement made from sediment (CFPS) and the commercial Ordinary Portland Cement (OPC) used as reference.

The CFPS contained approximately 10 times higher Zn than OPC and did not show any modification of the major crystalline phases. This finding is in accordance with the literature. Stephan et al. [29] pointed out that heavy metals such as Ni, Cr and Zn have no influence on the formation of clinker phases, even at concentrations that are 10–20 times higher than the concentrations observed in normal clinkers. However, very high intakes (approximate 2.5%) of heavy metals could cause changes in the clinker phases. In conclusion, a cement containing more than 39 wt.% of polluted sediment was successfully synthesised.

SEM was used to locate the Zn in the CFPS clinker. Fig. 2 shows zones with high zinc contents, identified as white areas on the SEM micrograph and red areas on the elemental X-ray image. The exact chemical composition of these areas could not be determined accurately by EDS measurements because of their small size. However, these areas were located in the aluminate phases ($C_3A$ and $C_4AF$). For higher concentrations of Zn, Bolio-Arceo and Glasser [30,31] identified the exact composition of an equivalent phase as $Ca_6Zn_3Al_4O_{15}$. Recently, the maximum of Zn that could be incorporated in a laboratory clinker has been determined experimentally. This threshold limit is found to be 0.7 wt.% for Zn [24]. These threshold limits were not reached in the CFPS.

### 4.2. Early-age hydration behaviour

To determine whether the synthetic cement (CFPS) exhibited behaviour similar to that of traditional OPC cement, the rate of hydration was evaluated. The reaction between cement and water is exothermic, and the heat produced is a good indication of their early-age hydration behaviour. In this study, the CFPS was compared to a commercially produced OPC.

The reactivity of cement is affected by its particle size. Therefore, to compare hydration behaviour between commercial OPC and the synthesised CFPS cement, the synthesised clinker was first ground to a fineness comparable to that of OPC (with a size distribution verified by a laser granulometer) and tested for Blaine fineness. The grain size distribution of the CFPS was similar to OPC. The

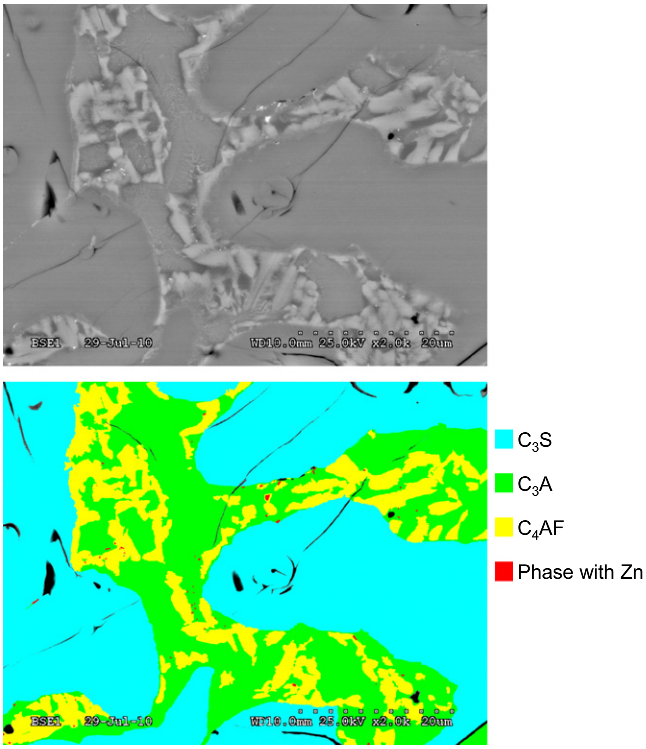

**Fig. 2.** SEM micrograph (top) and elemental X-ray mappings (bottom) of the clinker synthesised.

number of particles with a diameter between 0.6 and 1 μm was 5% higher in the case of CFPS. The Blaine fineness for OPC was 4400 $m^2$/kg, and it was about 13% higher for CFPS. This difference could be due to the grinding and sieving process used at the laboratory scale.

The gypsum addition content was determined by adding a range of gypsum content to the synthesised clinker and testing for early-age hydration behaviour using isothermal calorimetry. The addition of gypsum to reach 3% $SO_3$ was considered the optimum gypsum content. This content represents the lowest amount of gypsum that results in only one hydration peak and no significant change of the shape of the peak with additional amounts of gypsum added.

The rates of heat evolution for OPC and the CFPS are shown in Fig. 3. The rates of heat evolution show that the CFPS possessed hydration behaviour similar to the commercial OPC because the shapes and the maximum heat evolution rate of the curves were similar.

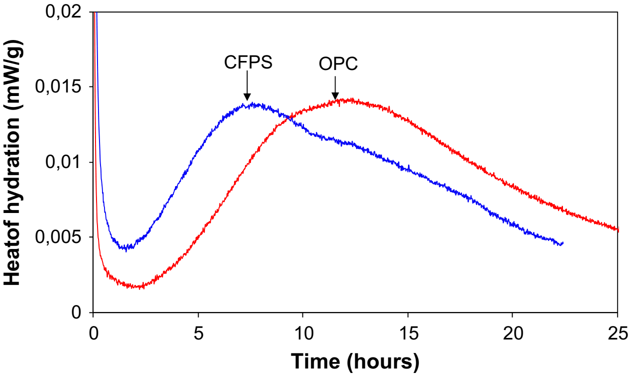

**Fig. 3.** Heat flow generated during the hydration of CFPS and OPC cement pastes.

The results show that the heat flow released during the synthetic cement hydration occurred about one hour earlier than the commercial OPC. This finding was perhaps caused by the differences in the fineness, which was higher for the CFPS than for the OPC.

The aim of this section was to compare the reactivity of the synthesised cement to that of commercial OPC. We showed that, despite the enrichment of Zn, the CFPS possessed hydration behaviour similar to the commercial OPC.

### *4.3. XRD patterns and SEM micrograph of cement pastes*

Cement pastes were examined by XRD to identify the crystalline hydration products and to compare the behaviours of CFPS and OPC. Fig. 4 shows the XRD patterns of the cement pastes after 1, 7 and 28 days of hydration.

The following results show that the principal hydration products in CFPS are essentially similar to those found in OPC. For the two cement pastes, a gradual increase of Portlandite ($2\theta$ 33.4° and 39.8°) content, accompanied by a corresponding decrease in the anhydrous calcium silicate ($2\theta$ 34.2°, 35.0°, 36.2°, 37.5° and 38.0°) and calcium aluminate ($2\theta$ 38.7) compounds, was observed. The formation of Ettringite ($2\theta$ 26.7), which is the first reaction product between calcium aluminates and gypsum, was confirmed in pastes even after 1 day of hydration. After 28 days, the Ettringite was mostly consumed through the reaction with the excess of $C_3A$. The background observed in the range $2\theta$ 32–41° was attributed to the formation of calcium silicate hydrates (C–S–H).

The XRD patterns showed some differences between the CFPS and OPC pastes. After 7 days, the XRD pattern of cement pastes showed that the silicates ($C_3S$ and $C_2S$) were more reactive in the case of CFPS compared to commercial cement pastes. This finding is clearly established at $2\theta$ 34.2° and the evolution of $2\theta$ 37.5° and 38.0°. This difference in reactivity could be caused by two possibilities. First, the Blaine fineness of CFPS was 13% greater than that of OPC and might have caused the difference in reactivity at 28 days. Second, the incorporation of trace elements in cement phases is well known to influence their reactivity [32]. In the case of CFPS, this enrichment was due to the use of polluted sediment for its production.

The microstructures of the cement pastes made from CFPS and OPC were studied through SEM coupled with EDS after 14 days of hydration. The objectives were to compare microstructures and to locate Zn in the cement paste of CFPS. Indeed, if the 0.16% of Zn contained in CFPS were concentrated in one phase in the cement paste, SEM/EDS could be able to detect this phase. Neither significant

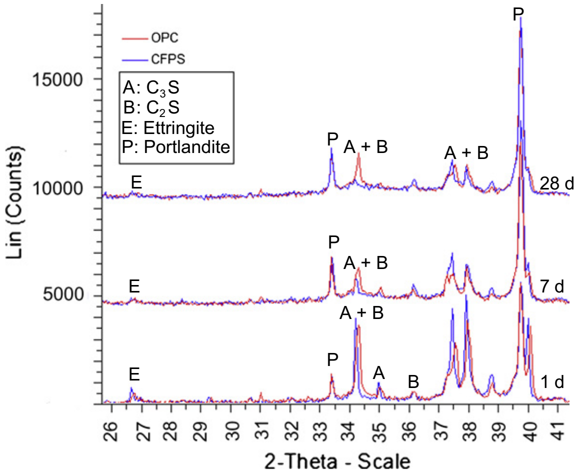

**Fig. 4.** XRD patterns of the cement pastes after 1, 7 and 28 days of hydration.

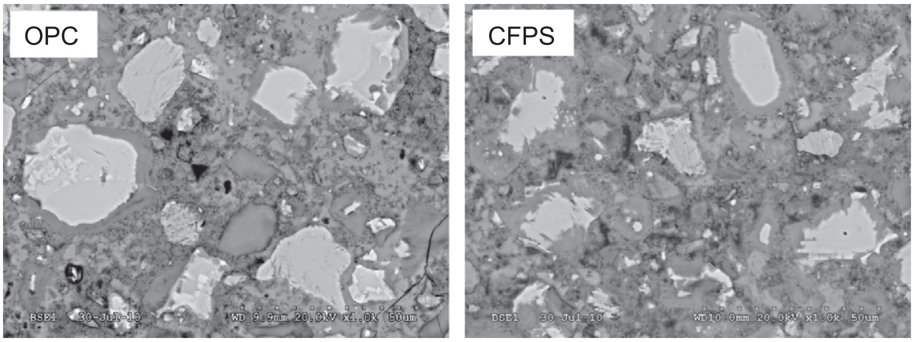

**Fig. 5.** SEM micrograph of CFPS and OPC cement pastes after 14 days of curing.

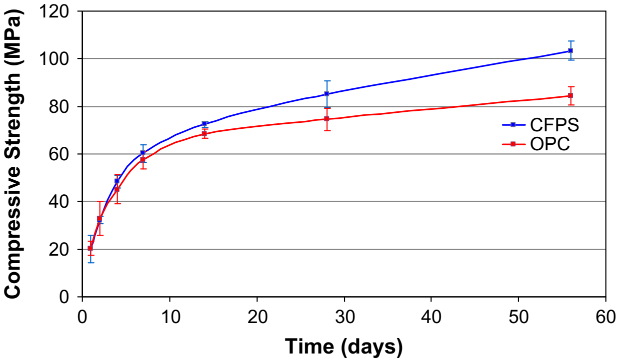

**Fig. 6.** Compressive strength of cement pastes obtained after 1, 2, 4, 7, 14, 28 and 56 days of curing.

differences in microstructure nor the formation of phases different than those of the reference (OPC) were detected in the sample of CFPS (Fig. 5). Thus, Zn was not identified in the cement paste by SEM/EDS.

### 4.4. Compressive strength

The compressive strength of cement pastes for the commercial cement (OPC) and the cement made from polluted sediment (CFPS) were assessed. At an early age, the cement synthesised from sediment produced a compressive strength equivalent to those obtained with OPC cement (Fig. 6). In addition, after 7 days, the CFPS develop higher compressive strength. This result is in accordance with previous XRD results showing that silicates ($C_3S$ and $C_2S$) are more reactive in CFPS than in commercial cement pastes.

## 5. Conclusions

The concrete industry is concerned by the principles of sustainable development because of its enormous environmental footprint. The tools and strategies to meet the environmental challenges should involve the use of recycled materials in place of natural resources. This article focuses on the use of fluvial contaminated sediment to replace a portion of raw materials in conventional Portland cement manufacture. The goal of this study was to produce a value-added product (cement) from wastes (sediment) and reduce the demand of the cement industry for raw materials. The three major conclusions of this paper are as follows:

(1) For each metric ton of Portland cement produced, 390 kg of polluted fluvial sediment can be recycled. This amount was never reached in previous studies on the substitution of cement raw materials.
(2) The synthesised CFPS cement was at least as reactive as the commercially produced OPC (CEM I 52.5).
(3) The results showed that the CFPS produced compressive strengths equivalent to those obtained with OPC at early ages, and 20% higher compressive strengths at 56 days. This result could be explained by the cement paste XRD patterns which suggested that silicates were more reactive in CFPS than in OPC.

The goal of this study was to synthesise Portland cement with the highest sediment content. Thus, the determination of this substitution rate of sediment for traditional raw materials was determined by considering the sediment composition and the Bogue formula. Consequently, only one composition has been studied. It would be interesting to test how different substitution rates affect the results.

In this paper, leaching tests were not performed. Except for the zinc, the chemical composition of CFPS was comparable to the average composition of Portland cement. Moreover, 0.15% of Zn is a low concentration, and the literature shows that this level does not induce environmental problems [33].

Finally, in contrast to most studies in the literature that regard synthesis or hydration, this paper presents a global approach from the synthesis to the hydration of cement made by using a by-product. This approach of recycling can be used for other by-products and/or wastes. Marine sediment should be included in this approach because of the large quantities produced during dredging. To use marine sediment, the problem of chlorides that interact with the kiln must be solved.

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