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The possibility of using dredging sludge in manufacturing cements: Optimization of heat treatment cycle and ratio replacement

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HIGHLIGHTS

- Valorization of dredged sludge based on illite/muscovite minerals.
- The effects of heat treatment and heating rate on the activation of the sludge and on its pozzolanic activity.
- The effect of the mineralogical composition on pozzolanic activity of the sludge.
- Hydration heat and setting time of activated sludge.
- Compressive strength and pozzolanic activity of activated sludge.

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ABSTRACT

This study aims to produce an eco-cement by using heat treated dam's sludge as an additive to clinker. Different heat treatment cycles were applied to the dam's sludge: three temperatures selected in the range between the dehydroxylation and decarbonation temperatures (600, 700 and 800 °C) reached by adopting different heating rates (5, 10 and 20 °C/min). The analysis of DRX, DSC–TG, FTIR results show that the heat treatment cycles let to the change of the sludge structure allowing its use as a pozzolanic additive to produce cements. The optimal heat treatment cycle is 600 °C with a holding time of 5 h and a heating rate 20 °C/min. Normalized mortars have been designed using modified cements constituted by 5% of gypsum, different percentage of heat treated sludge (5–10% and 15%) and clinker. The compressive strengths at 28 days lead to the choice of blending 85% clinker, 10% heat treated sludge and 5% gypsum to produce modified cements. The influence of the thermal activation of the dam's sludge on the technical properties such as normal consistency, fineness, setting time, heat of hydration, compressive strength have been investigated and tests were conducted according European standards. Results reveal that heat treatment at 600 °C with a rate of 20 °C/min conducts to an activated sludge with no emission of carbon dioxide and the higher strength resistance when added to clinker at 10%. However, whatever the fineness or the rate of heat treatment, the percentage of kaolinite (11%) existing in the natural sludge is not sufficient to develop by heat treatment at 600 °C hydration products allowing significant improve of mechanical resistance of modified cements by comparison to CEMI.

1. Introduction

In Algeria, water is essential for economic development, but its insufficiency requires the use of dams. In 1999, 64 dams were in operation for a capacity of around 7 billion m^3. By 2016, Algeria will have 96 dams with a total capacity of 9 billion m^3 according to the Ministry of Water Resources. Besides, Algeria's many dams have to deal with the problem of sedimentation generated by stripped soils, violent rains, erosion. Much of this sediment is often trapped behind dams causing the reduction in storage capacity of the dam and the decrease of its useful life. Even the newer dams are not avoided. Hence, nine news dams are annually subjected to 45 Mm^3 of deposited sediment causing a loss of their storage capacity and a significant reduction of their useful life. Presently, 18 dams are seriously threatened due to the acceleration of silting otherwise they will reach the end of their lifetime [1]. Among these, four dams (Zardezas, Foum El Gherza, Fergoug and K'sob) are currently in the process of being de-silted using dredging techniques. Consequently, large amounts of dredged sludge disposed

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must be reused otherwise they will be stored in landfills causing negative impacts on environment. Thus several studies have been conducted to explore the possibilities of valorizing the dredged sludge in civil engineering fields in order to minimize the impact of their storage on environment and to increase the life time of dams. Among these opportunities the reuse of sludge in the bricks manufacturing [2] or as road materials [3] may be mentioned.

At the same time it must be noted that concrete, the most widely used material on Earth, is based mainly on Portland cement which is responsible of total CO_2 emissions of about 1.126 Gt [4]. Of this almost 50% are derived from carbon compounds in the raw materials that converted to CO_2 and 50% produced from the combustion of fossil fuels necessary to drive the manufacturing process.

Hence, the production of blended Portland cements using additional materials that are principally based on industrial by-products, such as blast-furnace slags and fly ashes contributes to the reduction of the outrageous use of natural resources and to the protection of the environment by lowering CO_2 emissions associated with cement manufacturing [4,5]. For example, producing one tone of kaolin based-geopolymeric cement generates 0.180 tones of CO_2, from combustion carbon-fuel, compared with one tone of CO_2 for Portland cements [5] and fly ash based-geopolymeric cement emits CO_2 to nine times less than Portland cements [4]. Moreover, the introduction of such additions in cement production minimizes the cost. Indeed, adding fly ash induces a decrease in price of about 10–30% compared to OPC [6].

Hence, new artificial pozzolans have emerged and have been used as a substitution of cement or of clinker. Among these, paper mill sludge [7–10], ceramic waste [11–13], heat treated clays like the kaolinite [14,15], illite or montmorillonite [16], etc. have been widely investigated and their pozzolanic activity assessed. These artificial pozzolans are based on materials rich in silica and alumina that become active pozzolans when treated at a given thermal cycle. The pozzolanic reaction is between the calcium hydroxide and aluminosilicate materials [17] and is based on disso lution–precipitation mechanism in aqueous medium causing the formation of additional C–S–H. This reaction enhances the strengths of cementitious materials and improves their durability [18]. This study is conducted to explore the possibilities of valorizing the dredged sludge trapped behind Ksob's dam which is currently being de-silted. In particular it aims on the optimization of a thermal cycle to activate this dredged sludge so that it can be used as a partial substitution of clinker for manufacturing blended Portland cement.

The heat treatment (HT) cycle applied to the dredged sludge consists on applying different temperatures (600–700–800 °C) reached by varying the heating rate (5–10–20 °C/mn). The objective of such heat treatment is to destabilize the structure of the phyllosilicates and to create an active pozzolan. Hence this active created pozzolan interacts with calcium hydroxide to develop a supplementary calcium silicate hydrate (C–S–H) in addition to C–S–H provided during the hydration of tricalcium silicate (C_3S) and dicalcium silicates (C_2S) in basic medium. The cements were made with 10% of treated sludge, 85% clinker and 5% gypsum. The properties of sludge (before and after heat treatment) and cements were studied with using different techniques: differential scanning calorimetry (DSC), thermogravimetric analysis (TG), Fourier Transform InfraRed (FTIR), setting time, consistency, hydration temperature and compression tests.

2. Experimental methods

X-ray fluorescence (XRF) data of cement samples were generated on Philips PW2404 XRF spectrometer. The loss of ignition was determined according to European standard EN 196-2 [19].

The X-ray diffractograms of different samples were recorded on PANalytical X'PertPro X-ray diffractometer. The wavelength of X-rays used in this work was Cu Kα radiation with λ = 1.5405 Å. The data was collected for each sample over 2θ values ranging from 4° to 60°.

The thermal gravimetric analysis (TGA) and the differential scanning calorimetry (DSC) have been recorded using Netzsch STA 449F1 machine. Tests were carried out from 20 °C up to 1000 °C under nitrogen with a constant heat rate (10 °C/min). Simultaneous DSC–TGA measures both the heat flows (DSC) and weight changes (TGA) associated with phase transitions in the different cements as a function of temperature and time.

The density of the cement samples was determined using Le Chatelier flask according to EN 196-6 standard [20].

The fineness of cement was measured according to EN 196-6 Standard [20], using Air Permeability Method (Blaine Method). Consistence test was conducted on cement pastes using Vicat apparatus, performed according to EN 196-3 Standard [21] at a temperature of 20 ± 2 °C and a relative humidity of 65%. After mixing a quantity of water W, with 500 g of cement, C, the Vicat mold resting upon a glass plate is completely filled with this cement paste then the surface of the paste is smoothed. The standard consistency corresponds to the water quantity allowing to the Vicat plunger to penetrate to a point 5–7 mm from the bottom of the Vicat mold.

The Vicat apparatus is, also, used to determine setting times. The cement paste of standard consistency is used for this test.

The Hydration heat tests were carried out according to EN 196-9 [22] by means of semi-adiabatic calorimetry known as the Langavant method. This method consists of quantifying heat generated by cement paste during the first few days. The test was carried out at a temperature of 20 ± 2 °C and a relative humidity of 65%. The heat of hydration Q [J/g] was obtained by the Formula 1:

$$Q\ (\mathrm{J/g}) = \frac{C}{m_c} \cdot \theta_t + \frac{1}{m_c} \int_0^t \alpha \cdot \theta_t \cdot dt \qquad \text{(Formula 1)}$$

where $t(h)$ is the hydration time, C (J/°C) is the total thermal capacity of calorimeter as well as the mortar sample depending on heat capacity of the empty calorimeter and the masses of cement, sand, aggregates and water of the specimen itself as well as the mass of the empty calorimeter, m_c (g) is the cement mass, θ_t (°C) is the heating of the sample at the hydration time t(h) and α(J/h/°C) is the coefficient of the calorimeter heat loss.

Fourier transform infrared spectroscopy (FTIR) tests were recorded in the range of 400–4000 cm^{-1} using a Bruker Optik (Tensor 27 model).

All normalized mortars were prepared by mixing 1350 g of normalized sand, 450 g of cement and 225 g of water. These samples were demolded after 24 h and placed in water bath maintained at 23 °C. Compressive tests were carried out on mortars at 2, 7, 14, 28 and 90 days according to EN 196-1 [23].

In the present work, the hydration reactions were stopped by immersing small samples of cement paste in liquid nitrogen at a temperature of −50 °C. To prevent the hydration reactions starting again when the samples are reheated to room temperature, the water was removed by placing the frozen samples in a low-pressure chamber (500 Pa) and by continuously pumping to remove free water.

After freeze-drying, the samples are in dry solid form. Consequently, further cement hydration cannot takes place. The samples were, then, stored in a dry environment so that additional analysis can be conducted.

3. Materials

The clinker and the gypsum used to manufacture cements are provided by LAFARGE Group established in M'sila – Algeria. The raw materials employed are clay, limestone, sand of dune from Bousaada-M'sila and iron ore.

Sludge was dredged from sediment trapped behind K'sob dam which covers an area of 1460 km^2 with a perimeter of about 180 km with an elongated shape. The K'sob dam is located at a place called Hamman between mountains of Kef El Ouerad and Djebel El Gruon at 15 km north of the city of M'sila. It was built at this location on the river K'sob between years 1934 and 1940 for irrigation of agricultural area. In 2011 according to the ANB (national agency of dams), the K'sob dam recorded 59.90% of siltation rate, the amount of sludge deposited annually was approximately $8 \times 10^5\ m^3$. This sludge was dredged to surface and evacuated downstream using pumps in settling basins built on uncultivated land.

3.1. Clinker and gypsum

The clinker has a regular chemical and mineralogical composition (Table 1). Bogue's compounds are acceptable, tricalcium