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The effectiveness of the incineration of sewage sludge on the evolution of physicochemical and mechanical properties of Portland cement

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HIGHLIGHTS

- Extensive and detailed characterization of sewage sludge treated thermally at temperatures between 300 and 800 °C.
- Monitor the evolution of the properties of sewage sludge during the thermal treatment.
- Study the influence of varying the calcination temperature of sewage sludge on Portland cement properties.

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ABSTRACT

The influence of sewage sludge, treated at temperatures between 300 and 800 °C, on cement-based materials has been studied in this paper. The clinker of CPJ_{45} cement was partially replaced by sewage sludge ash (SSA) admixture in mortar at various percentages. The sewage sludge ashes were characterized chemically, physically and mineralogically. In addition, the influences of SSA on mortar properties, including Blaine fineness, specific gravity, water demand, time of setting, compressive strength and degree of hydration were also investigated.

Results show that calcination influences the microstructure of sewage sludge and increases its pozzolanic activity that reaches its maximum at 800 °C. The presence of absorbed water and organic matter in sewage sludge calcined in temperature range 300–500 °C prolongs strongly the setting time and affects negatively the compressive strength and the hydration degree of mortars. While the high amounts of P_2O_5 and SO_3 in sewage sludge calcined in temperature range 700–800 °C increase also water demand and setting time compared to the control mortar, however, the compressive strengths and the degree of hydration increase with time and become superior to the control mortar at 90 days, for a replacement rate of 15%. The best mechanical results are obtained for the substitution of 5% of calcined sewage sludge at 28 days.

1. Introduction

The production of sewage sludge is one of the major issues in wastewater treatment that requires proper, effective and environmentally safe management. After several years of experience in handling with sewage sludge, many countries start to reconsider or minimize the use of this waste as natural fertilizer in landfilling or in agriculture because of the limited availability of suitable landfill space, concerns over the accumulation of heavy metals in soils, and the potentially pathogenic organisms [1–3]. In the mean time, incineration has become a growing option and an attractive disposal method of sewage sludge, especially for large cities, as it reduce the waste volume, eliminate the pathogenic organisms and the volatile contents, and can also produce energy recovery profits [1,4]. However, another environmental problem has emerged because of the substantial amounts of sewage sludge ashes (SSA) produced after the firing process, which must be disposed by other means [5].

To avoid eventual environmental impact, the use of SSA in building materials is therefore a smart solution, given that the technology of manufacturing cement needs to conserve the resources, reduce CO_2 emissions and preserve energy [6–8]; and that the cement matrix can immobilize the SSA contents [9,10]. Accordingly, SSA has been used in concrete mixtures ([11–13], in lightweight aggregates [14,15], in ceramic products [16–18], in asphaltic paving mixes [19], in cement mortars ([20–22]; and as raw material in clinker production [23,24].

The purpose of the present work is to deepen the characterization of SSA and to better understand the effect of varying the

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calcination temperature, between 300 and 800 °C, on the evolution of physical, chemical, mineralogical and micro-structural properties of sewage sludge. This work also aims to study the effect of the thermal treatment of sewage sludge on Portland cement properties, including Blaine fineness, specific gravity, water demand, setting time, mechanical strength and hydration degree of mortars.

2. Materials and methods

2.1. Preparation of SSA

Sewage sludge used in this investigation was sampled from an industrial wastewater treatment plant located in Fez (Morocco), operating according to the activated sludge method [25]. The sludge samples were dried at 40 °C, homogenized, grounded and sieved to a particle size smaller than 1 mm. The calcination of the dried sewage sludge was carried out at fixed temperatures (300, 400, 500, 600, 700 and 800 °C) during 50 min in an electrical muffle, type Nbfertherm GMbH [26]. Then, the calcined sludge was cooled down under the atmospheric pressure and at ambiance temperature.

2.2. Preparation of SSA mortar

The cement used was CPJ_{45} Portland cement according to NF EN 197-1 standard [27]. Cement compounds (68% of clinker, 27% of limestone and 5% of gypsum) were ground using a ball mill. The particle size of the cement was monitored during milling using sieves, to set the refusal 80 µm at 2% ±0.1, to eliminate its impact on mechanical properties. The addition of SSA was performed by replacing (5%, 10%, 15% and 20%) of clinker by each type of SSA separately, according to the following equation:

$$-CPJ45_{Y-X} = [(100-X)\%\ \text{clinker} + X\%\ SSA_Y] \times 68\% + 27\%\ \text{limestone} + 5\%\ \text{gypsum}$$

With: X = 5, 10, 15, 20.
Y = 300, 400, 500, 600, 700 and 800 °C.

The sand was from quartz with particle sizes between 0 and 2 mm in accordance with NF EN 196-1 standard [28]. The mortar mixtures were prepared according to the same standard NF EN 196-1 [28], and contained three parts of sand to one part of binder by mass, with a W/C ratio of 0.50. To study the influence of SSA, one batch of control mortar without adding any sewage sludge was also prepared. The mixtures were cast in 4 × 4 × 16 cm molds for the first 24 h. Then specimens were demoulded and stored in a temperature controlled room at 90% humidity and at 20 °C. Strength tests were performed at different ages in accordance to French standard NF EN 196-1 [28].

2.3. Tests for SSA and mortar properties

The different types of SSA were characterized by inductivity coupled plasma-atomic emission spectrometer (ICP-AES), X-ray Diffraction (XRD) and scanning electron microscopy (SEM) equipped with a probe EDX for surface microanalysis.

The pozzolanic activity of SSA was evaluated according to Chapelle test. One gram of SSA and One gram of CaO were added in 200 mL of distilled water. The mixture was hydrothermally treated at 100 °C for 16 h in a stainless steel capsule of 350 mL maximum capacity. Then, the solution was filtrated and titrated with 0.1 N HCl solution using phenolphthalein as indicator. Fixed lime content (mmol/L) was calculated from the difference between the amount of $Ca(OH)_2$ measured in absence (blank solution) and presence (sample solution) of sample [29].

The SSA-cement mixtures were characterized by X-ray fluorescence according to NF EN 196-2 standard, Blaine fineness and specific gravity according to the NF EN 196-6 standard, water requirement and the setting time according to the NF EN 196-3 standard [30].

For mortar specimens, the degree of hydration (α) was obtained by using the formula (1). The hardened mortars were tested at 28 days:

$$\alpha = [(W_{105} - W_{580}) + 0.41(W_{580} - W_{1007})/nW_{1007}] \times 100\% \quad (1)$$

where n is the amount of water evaporated in the cement paste when hydration is completed (for ordinary cement paste, n equals 0.24); W_{105}, W_{580}, W_{1007} are the weights of the specimens at 105 °C, 580 °C, and 1007 °C, respectively; The conversion factor, 0.41 represents the mass ratio of 1 mol H_2O to 1 mol CO_2 [31].

3. Results and discussions

3.1. Characterization of sewage sludge

The Chemical analyses (Table 1) of SSA calcined at different temperatures, provided by ICP-AES, revealed the presence of high amounts of calcium, iron, magnesium, sodium, phosphorus and potassium. Generally, the concentration of these elements in SSA increased with the increase of the temperature of calcination. This may be attributed to the fact that during the calcination these elements react with oxygen gas to form its oxides. Zn, Pb, Cu, Ti, Ba, Cr, Mn and Ni were present as minor constituents and the other metals were present in insignificant concentrations for all ashes. Moreover, some elements as Pb and Cd are highly volatile; therefore, its concentrations in SSA decrease from 300 °C to 800 °C and could be volatilized at a higher temperature [32]. While, other elements as Ni, Cr, Zn and Cu are moderately or lowly volatile, thus their concentrations in SSA stay stable or increase from 300 °C to 800 °C [33].

Table 1
Trace analysis of sewage sludge calcined at 300–800 °C.

Element (mg/L)	300 °C	400 °C	500 °C	600 °C	700 °C	800 °C
Ca	342.64	394.20	470.46	565.18	687.24	712.68
Fe	33.82	33.87	33.98	34.23	34.16	34.08
Mg	37.74	46.34	55.54	57.38	69.42	78.82
Mn	0.18	0.20	0.20	0.22	0.33	0.29
Na	40.64	41.76	61.44	61.36	79.23	37.78
P	43.91	43.95	43.26	44.28	55.82	69.78
K	16.82	17.03	17.25	15.04	18.96	14.55
Cd	0.07	0.07	0.08	0.07	0.04	0.02
Co	0.026	0.018	0.013	0.015	0.021	0.019
Cr	0.38	0.39	0.42	0.41	0.79	0.74
Ba	0.31	0.33	0.36	0.38	0.53	0.62
Cu	1.27	1.28	1.33	1.46	1.84	1.86
Ni	0.27	0.30	0.29	0.42	0.67	0.83
Pb	1.07	1.11	1.13	1.09	1.04	0.79
Ti	0.17	0.19	0.25	0.32	0.39	0.46
V	0.05	0.07	0.09	0.08	0.10	0.04
Zn	3.55	3.76	3.79	3.83	3.82	4.02

Fig. 1 presents the scanning electron microscopy (SEM) micrographs of ashes calcined at 300–800 °C. These micrographs show the presence of a large amount of organic matter which appears in black in case of ash calcined at 300 °C and disappear after incineration at 500 °C. The SEM micrographs also show the presence of some crystalline aggregates plus the amorphous phases. The morphology of particles is irregular for all ashes, especially those calcined at 300 and 400 °C. This morphology will have a decisive influence on the water demand, the porosity and the compressive strength of mortars [21].

XRD measurement (Fig. 2) of sewage sludge ashes showed the presence of quartz (SiO_2) and calcite ($CaCO_3$) as the main phases in addition to dolomite ($MgCaCO_3$), anhydrite ($CaSO_4$) and magnesium calcium carbonate ($(Mg_{.064}\ Ca_{.936})\ (CO_3)$). This may be credited to the fact that some calcium compounds might be combined with magnesium or sulfur produced from the combustion of the organic matter. In addition, some phases containing heavy metals (Cr, Zr and Sr) were found. After incineration of sewage sludge at 800 °C calcite decomposes into calcium oxide CaO [34]. Also, additional crystalline phases appeared in SSA composition ($Ca_2\ Fe_{1.2}\ Mg_{0.4}\ Si_{0.4}\ O_5$) and Ca5 (P, Si, $S)_3O_{12}$(Cl, O H, F) that have a chemical structure close to wilkeite.

The fixed lime contents of calcined sewage sludge increases by 779% between 300 °C and 800 °C (Fig. 3). This reveals the important changes in the microstructure of SSA caused by the firing process, and goes with a development of the pozzolanic activity and lime adsorption capacity of SSA. Some researches show that the calcination of sewage sludge at temperatures higher than 800 °C reduces the fixed lime content [35]. So the sewage sludge calcined at 800 °C exhibit the highest pozzolanic activity.