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# Characterization of calcined sewage sludge for its incorporation in cement

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## Abstract

The production of enormous quantities of sewage sludge is a compulsory result of wastewater treatment. This sludge is massive and can contain toxic substances; consequently, it needs to be valorized suitably. However, an appropriate treatment can lead to a successful reuse of this sludge in cement manufacturing. This investigation studies the impact of a thermal treatment on sewage sludge, for its reuse in cement, and presents the results of the characterization of calcined sludges, at different temperatures, by Atomic Emission Spectroscopy Inductively Coupled Plasma, X-ray Diffraction and Fourier Transform Infrared analysis. The results show that the calcined sludges contain oxides of different sizes, quartz, calcite, dolomite and lime; and several metals Ca, Mn, Mg, Na, P, Fe and Si. The compositions of calcined sludges recommend their use in cement manufacturing.

*Key-words*: Characterization; Sewage sludge; Thermal treatment; Cement.

## 1.Introduction

The biological treatment of wastewater leads to a purified water respecting the norm of rejection ordered by Morocco [1]. However, the treatment process produces continuously large quantities of a sludge that presents risks for the environment and human health [2, 3]. This sludge needs to be managed in an ecological and economical way.

The most common sewage sludge disposal methods are landfilling, sea dumping and soil application [4, 5]. In the meantime, space limitations on existing landfill sites and increasing environmental concerns have resulted in limitations on sewage sludge disposal options [6].

Yet, the technology of manufacturing cement needs to conserve the resources, to protect the environment and to use appropriately the energies. Consequently, it gives a lot of importance to the use of wastes and by-products in cement and concrete manufacturing. Thus, we have studied, in a previous work [7], the valorization of sewage sludge in cement manufacturing. The sludge was used with no preliminary treatment. The results weren't encouraging for the setting time and the mechanical strength of cements. These results can be improved by a thermal treatment of the sludge in order to reduce its organic matter.

This investigation provides the results of the calcinations of sludge at different temperatures.

## 2. Material and methods

### *2.1. Material: sewage sludge*

The samples of sewage sludge were collected from a company situated in the city of Fez, which treats its wastewater by the biological method of activated sludge, according to standardized conditions of AFNOR [8].

### *2.2. Methods*

The sludge was homogenate, dried up in the free air, grounded and sieved to a particle size smaller than 1 mm. The calcination was carried out by the introduction of 5 g of sludge in an electrical muffle, type Nbfertherm GMbH, in order to be calcined at fix temperatures (400 °C, 500 °C, 600 °C, 700 °C et 800 °C) at 50 minutes [9]. After that, the calcined sludge was cooled down under the atmospheric pressure and at ambiance temperature.

The characterization of incinerated sludge was provided by inductivity coupled plasma-atomic emission spectrometer (ICP-AES), X-ray diffractometer (XRD) and Fourier transform infrared spectrometer (FTIR).

## 3. Results

*3.1. Weight loss of sewage sludge during the calcination*

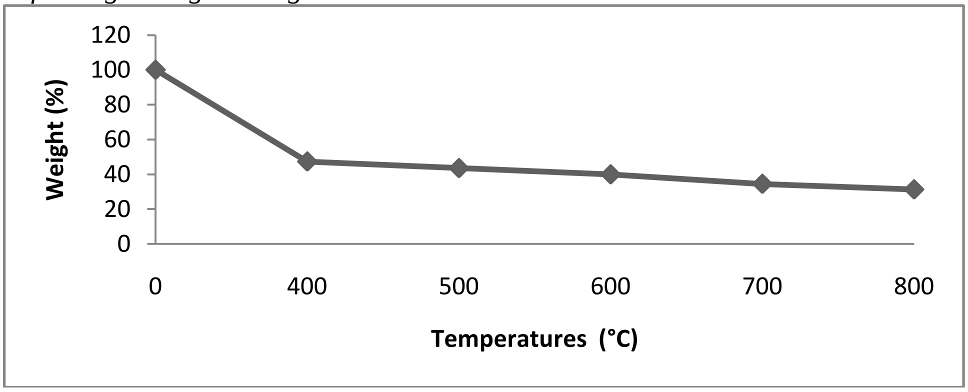



**Figure 1:** Weight loss of sewage sludge calcined at 400–800 ◦C

*3.2. Metallic composition of calcined sludge*

Metals analyzed in the calcined sludge by ICP-AES are calcium Ca, magnesium Mg, manganese Mn, sodium Na, phosphorus P, iron Fe and silicon Si (figures 2 to 7).

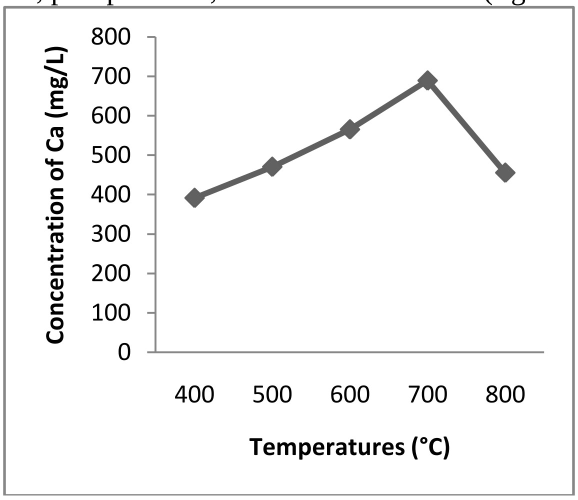



**Figure 2**: [Ca] according to the calcination temperatures

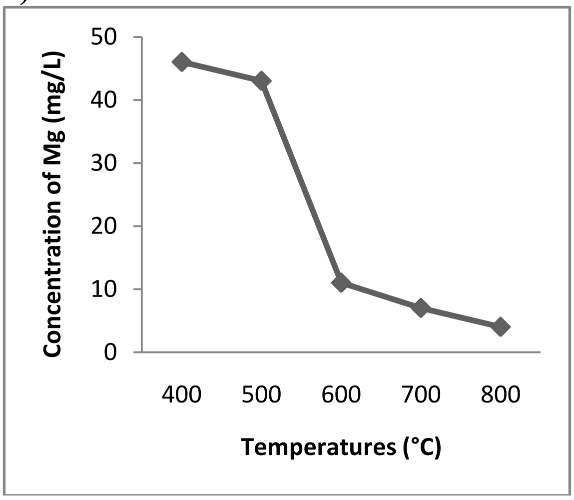



**Figure 3**: [Mg] according to the calcination temperatures

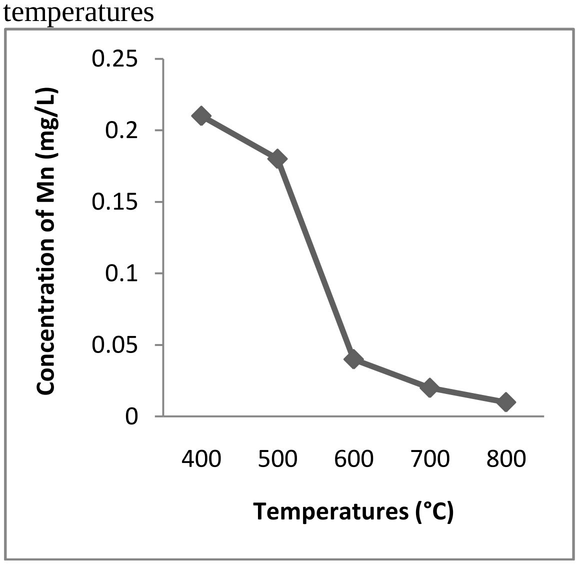



**Figure 4:** [Mn] according to the calcination temperatures

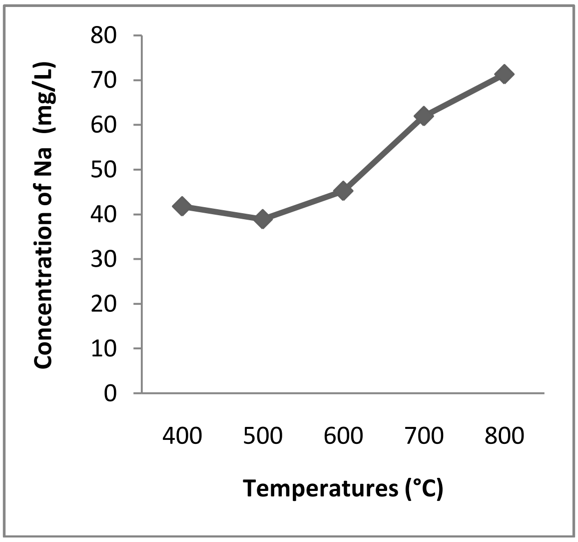



**Figure 5**: [Na] according to the calcination temperatures

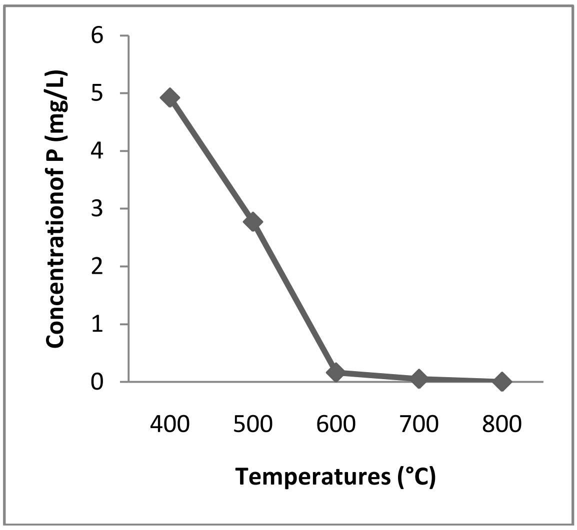



**Figure 6**: [P] according to the calcination temperatures

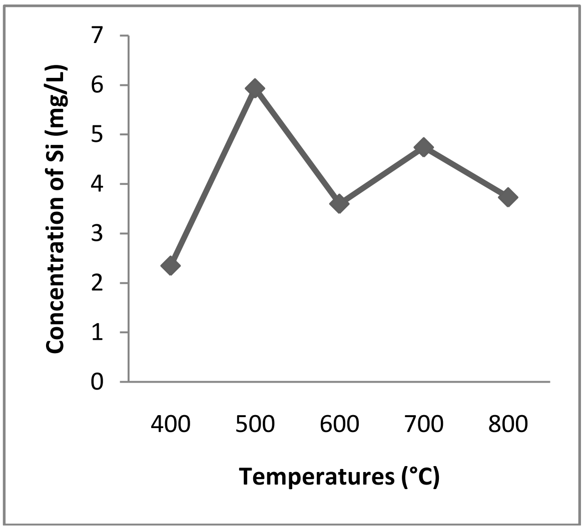
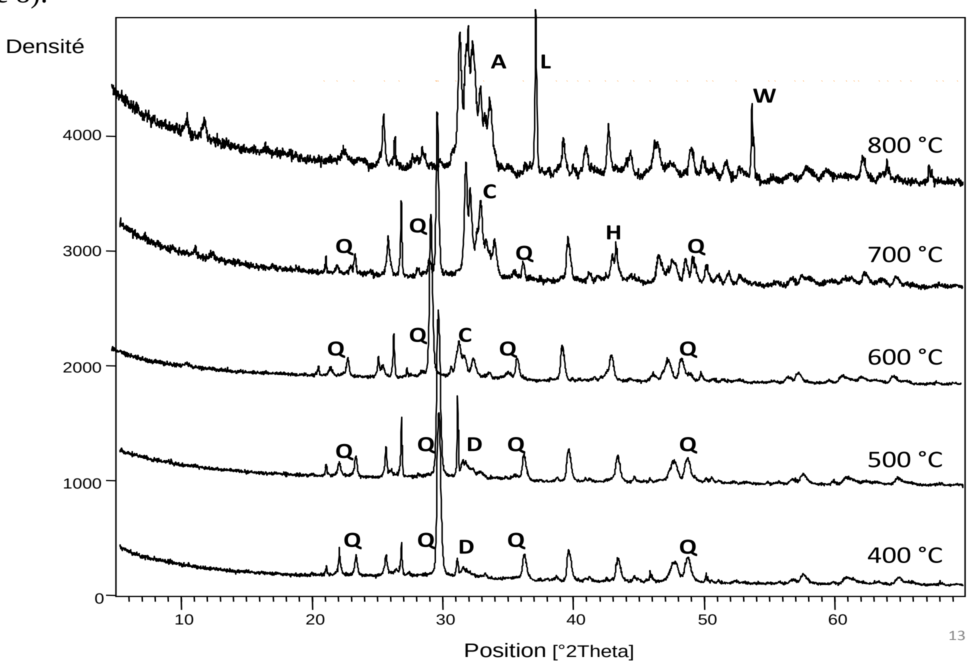



**Figure 7**: [Si] according to the calcination temperatures

The concentration of Fe in the sludge is lower than 0.01 mg/L for all the calcination temperatures.

*3.3. Mineralogical phases of calcined sludge*

The mineralogical analysis of calcined sludge, at the studied temperatures, reveals the presence of the following phases (figure 8):




A: Actinolite; C: Calcite; D: Dolomite; H: Hydroxyapatite; L: Lime; Q: Quartz; W: Wilkeite.

**Figure 8**: X-ray diffractogram of sewage sludge calcined at 400–800 ◦C

*3.4. Infrared spectra of calcined sludge*

The infrared spectra of calcined sewage sludge, at the studied temperatures, are presented in figure 9:

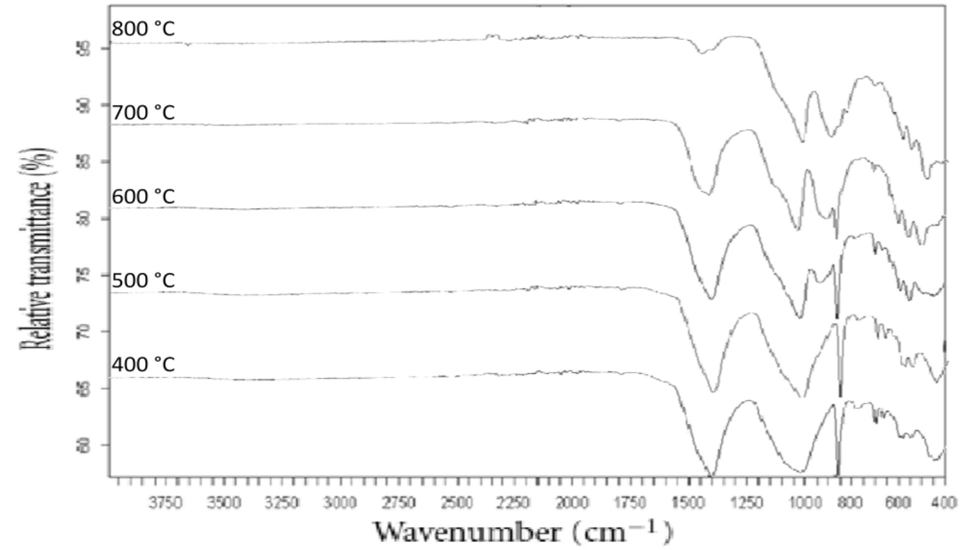



**Figure 9:** The FTIR spectra of sewage sludge calcined at 400–800 ◦C

## 4. Discussion

The weight of sludge decreases widely between 0 and 400 °C; with a loss beyond 50 % of its initial weight. Weight loss which occurs in temperature range 50–130 ◦C is due to elimination of moisture and absorbed water. Weight losses which occur in temperature range 200–320 ◦C and 330–390 ◦C are due to emission of volatile organic matter and combustion of complex nonvolatile organic matter, respectively. This is because sewage sludge is composed of various species of hydrocarbons with a wide range in boiling point [10]. From 400 °C to 800 °C, the weight of sludge decreases gradually and regularly from 47,4 % to 31,4 %. Weight loss which occurs in temperature range 400–670 ◦C is due to combustion of fixed carbon captured by inorganic materials, elimination of structural water of the clay mineral, and decomposition of the carbonaceous matter present in sewage sludge. From 700 °C to 800 °C the weight of sludge become more or less constant (figure 1).

The concentration of Ca rises according to the temperature (figure 2). At 400 °C and 500 °C the Ca joins the Mg to form dolomite (figure 8). At 600 °C and 700 °C, a formation of calcite took place. At 800 °C, the $CO_2$ was vaporized to form lime, this creation go with a decrease in the concentration of Ca in the sludge. In fact, the presence of lime in sludge is very favorable for its incorporation in cement, because during the mixture of cements with water, the sludge consumes lime before cement to create portlandite $Ca(OH)_2$, silicates calciques hydrates (CSH) and ettringite ($3CaO.Al_2O_3.3CaSO_4.32H_2O$) [11].

The concentration of Mg decreases lightly between 400 °C and 500 °C (figure 3), with a formation of dolomite (figure 8). However, it drops rapidly at advanced temperatures with a formation of actinolite. This result could be attributed to the evaporation of Mg at higher temperatures [12]. The disappearance of Mg from the calcined sludge promotes its incorporation in cement, since the presence of Mg increases the expansion of cement and causes cracks in the concretes [13].

The sludge content of Mn is insignificant and varies in the same manner as Mg, with no registration of mineralogical phases containing Mn in the incinerated sludge (figures 4 and 8). Moreover, the presence of Mn has no influence on the hardening or the mechanical properties of cements [13].

The rate of Na rises regularly from 500 °C to 800 °C (figure 5). An elevated concentration of Na could affect inadequately the mechanical strength of cements at young age [14].

The concentration of phosphorus starts to decrease from 400 °C until nearly its disappearance above 600 °C (figure 6). This result encourages the incorporation of sludge in the cement. Sure enough, the presence of phosphorus in cement precipitates the calcium in the form of hydroxylapatite ($Ca_{10}(PO_4)_6(OH)_2$), which slow down the setting time of cements [11].

The concentration of Si fluctuates according to the temperature. From 400 °C to 700 °C a formation of quartz took place and at 800 °C we have the creation of actinolite (figure 8). Besides, the presence of Si in the sludge is accepted as long as the ratio CaO/SiO2 >> 1 [15].

Figure 9 illustrates the FTIR spectra of sewage sludge calcined at 400–800 ◦C. The absorption bands of silica appear at 1101, 796, and 467 $cm^{-1}$ corresponding to the asymmetric stretching vibration of Si–O–Si, symmetric stretching vibration of Si–O–Si, and bending vibration of O–Si–O, respectively [16]. The incineration of sewage sludge at 800 ◦C leads to a decrease in the intensity of the absorption bands of silica. This is an indication of increasing the degree of polymerization of silica network as a result of the crystallization of silica into quartz. The absorption at 1426 and 873 $cm^{-1}$ is due to $CaCO_3$ presence [17]. The decrease of the intensities of calcite peaks and the appearance of new peaks in the samples heated to 800 °C corroborate that the decomposition of calcium carbonate in the sludge is succeeded by formation of new phases, involving CaO. The absorption band of P-containing compounds appears at 567 $cm^{-1}$ due to P-O bending vibration [18]. The samples exhibit sharp around 1600 $cm^{-1}$ may be due to C=C vibration of aromatic ring and O–H stretching vibration due to absorbed water.

## Conclusion

The thermal treatment of sewage sludge for 50 minutes at different temperatures reveals:

- A decrease in the metals (Mg, Mn, P) affecting unconstructively the quality of cement in an average temperature of incineration of 700 °C (table 1).
- An increase in the concentration of calcium during the incineration of sludge.
- A formation of several mineralogical phases: dolomite, calcite, quartz, lime…, proving the reduction of the organic matter present in the raw sludge and the formation of new mineralogical phases according to the temperature of calcination.

The results aroused support the use of calcined sludge at 700 °C for 50 minutes in cement manufacturing as a primary or secondary admixture.

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