



# Preparation of calcium sulfoaluminate-belite cement from marble sludge waste



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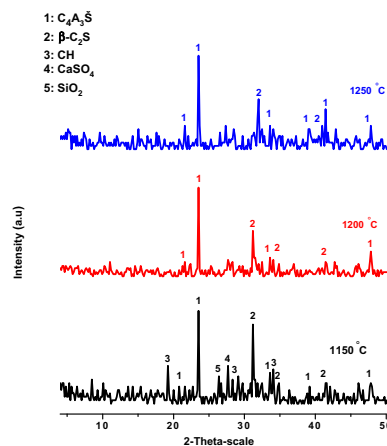
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## HIGHLIGHTS

- The recycling of waste material was successfully carried out leading to the production of less CO<sub>2</sub> emission cement known as sulfoaluminate-belite cement.
- The influences of the cement raw mix composition and the different burning temperature were investigated to evaluate the reuse feasibility of marble sludge waste material in cement production.
- Calcium sulfoaluminate-belite cement, environmentally cement, can be produced by raw mix contains in weight percent 25% kaolin, 20% gypsum and 55% marble sludge waste at firing temperature 1200 °C.

## GRAPHICAL ABSTRACT

In this study, the feasibility of recycling marble sludge waste in the production of calcium sulfoaluminate-belite cement was investigated.



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## ABSTRACT

Marble sludge waste was used as a major cement raw material in sintering sulfoaluminate cement clinker successfully in the laboratory. The influences of raw mix composition as well as different burning temperatures were investigated. Starting materials and prepared cement were characterized through different techniques including; Fourier transform infrared spectroscopy (FTIR); X-ray diffraction (XRD) and scanning electron microscopy (SEM). The results reveal that calcium sulfoaluminate-belite cement can be produced by burning a raw mixture contains in weight percent (25% kaolin, 20% gypsum and 55% marble sludge waste) at firing temperature ranging between 1200 and 1250 °C.

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## 1. Introduction

Calcium sulfoaluminate-belite (CSAB) cement is promoted as a sustainable alternative to Portland cement (PC) because of lower energy consumption as well as reduction in CO<sub>2</sub> emission during

Abbreviations: C, CaO; S, SiO<sub>2</sub>; Š, SO<sub>3</sub>; A, Al<sub>2</sub>O<sub>3</sub>; F, Fe<sub>2</sub>O<sub>3</sub>; H, H<sub>2</sub>O.

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production. The calcium sulfoaluminate–belite cement phases can form and are stable at a temperature of approximately 1250 °C, which is lower than the temperature used for Portland cement production. Further, calcium sulfoaluminate–belite clinker is more friable and soft than Portland cement clinker, which reduces the energy needed for grinding process [1]. Calcium sulfoaluminate–belite cement principally utilizes Ye’elimite phase ( $C_4A_3\bar{S}$ ), instead of tricalcium silicate (Alite,  $C_3S$ ) phase, as the primary early-age strength gaining phase and utilizes dicalcium silicate (Belite,  $\beta$ - $C_2S$ ) phase to develop additional long-term strength.

Ye’elimite phase ( $C_4A_3\bar{S}$ ) has lower calcium oxide content than Portland cement phases, making it an attractive option for developing sustainable cement product [2]. Calcium sulfoaluminate–belite cement has shown rapid setting, high early strength, and shrinkage compensating properties due to the fast reacting of Ye’elimite phase ( $C_4A_3\bar{S}$ ) and the expansive nature of ettringite [3]. In field practices, this kind of cement have been used mainly in precast concrete applications in cold environments and have shown good dimensional stability, low permeability, low alkalinity, good durability, and comparable compressive strength to Portland cement [4–7].

Raw mixes for calcium sulfoaluminate cement differ from those for Portland cement in that they contain a significant amount of sulfate. Therefore, the reactions and products are quite different from those normally found in Portland cement production [8]. The raw mix composition of this cement can be based on conventional raw materials (limestone, clay, bauxite and iron ores) but industrial by-products or wastes can be used as well [9,10] (for example fly ash, pyrite ash, galvanic sludge, metallurgical slags, phosphogypsum, etc...). Furthermore, this new class of cement can be produced in existing installations using a conventional cement kiln system.

Several types of industrial wastes can be used as raw materials to obtain the calcium sulfoaluminate (CSA) cement clinker. Wu et al. obtained a clinker of this cement from a mixture of municipal solid incineration wastes–limestone–bauxite–gypsum at 1250 °C, reaching more than 73.2 Mpa after 28 days of curing [11]. Li et al. [12] obtained cement by firing a mixture of fly ash, bauxite and calcium carbonate at 1300 °C. Li et al. [13] obtained the calcium sulfoaluminate (CSA) cement at a temperature as low as 1150 °C using fly ash and sludge as raw materials. Singh et al. [14,15] reported the formation of a ferric calcium sulfoaluminate cement from a mixture of calcium oxide, red mud and bauxite at 1250 °C, as well as using waste from a fertilizer industry, bauxite and iron mineral ore at 1250 °C. In general, these works showed the feasibility to obtain calcium sulfoaluminate (CSA) cement using many industrial wastes as raw materials. However, there is a great variety of industrial wastes that have the potential to be used as a source of the main components to fabricate this kind of cement [16].

Marble processing lines in the Egyptian factories produce huge amounts of marble wastes either solid or liquid (slurry, resulted from sawing the blocks to slabs and grinding and polishing processes). The random disposal of these wastes, estimated as one million tons yearly, in the areas near the factories (irrespective of their possible economic values) and with the increasing production cause severe environmental problems, such as land degradation, increase wastage of minerals, air pollution, water pollution, damage to flora and fauna as well as human resources displacement [17]. Recycling process of industrial by-products and waste materials coming from industrial manufacture activates have become an urgent problem for the near future. In the light of environmental protection standards which aiming to limit the accumulated of the industrial dump, the development of recycling techniques capable of exploiting the industrial wastes into new marketable products acquires an increasing importance for the industrial and environmental sectors.

In this study, the feasibility of reusing the marble sludge waste as starting materials in the preparation of sulfoaluminate–belite cement was evaluated. This work shows the feasibility of producing calcium sulfoaluminate–belite cement from a mixture of kaolin; marble sludge waste as a source of calcium carbonate ( $CaCO_3$ ) and gypsum ( $CaSO_4 \cdot 1/2H_2O$ ). The sintering temperature in the laboratory muffle furnace could be controlled by the temperature of 1150, 1200 and 1250 °C. The cement quality was investigated to evaluate the reuse feasibility of marble sludge waste material in cement production.

## 2. Materials and methods

### 2.1. Starting raw materials

The raw materials used in this study were kaolin, marble sludge and gypsum. Kaolin material was supplied from the general company for ceramic & porcelain products (Sheeni); Marble sludge was collected from Shaq El Thoaban marble industrial zone; Egypt. The surface area of marble sludge waste was determined by using the method of  $N_2$  adsorption (BET) using automated gas sorption; model NOVA, Version 1.12 from the quantachrome. The measured surface area and specific gravity of marble sludge waste were 0.6695  $m^2/g$  and 2.67 respectively. Gypsum (hemihydrate,  $CaSO_4 \cdot 1/2H_2O$ ) was supplied from Gypsina company; Egypt. The main oxide compositions of starting raw materials chemically analyzed by X-ray fluorescence technique (Wavelength dispersive XRF; PANalytical) were reported in Table 1. The mineral composition phases of starting raw materials were identified by X-ray diffraction analysis technique using (XRD; X’Pert-PANalytical) diffractometer with Ni filter, with  $Cu K\alpha$  ( $\lambda = 1.5406 \text{ \AA}$ ) radiation at 40 kV, 30 mA at a scanning speed of 0.020°/s over the  $2\theta$  range of 4–60°. FTIR spectra of starting raw materials were acquired using a JASCO FT/IR-6100. The IR spectra were recorded between 400 and 4000  $cm^{-1}$  with a resolution of 4  $cm^{-1}$  at room temperature.

For preparing cement mixes, appropriate amounts of starting raw materials in predetermined wt.% proportions, as shown in Table 2, were taken and ball milled for 30 min using a top planetary ball mill (Fritsch planetary mono mill Pulverisette 6) for homogenization. The resultant powder samples with chemical oxides composition based on the XRF data of started materials, as shown in Table 3, were made into a thick paste using a low amount of water ( $\approx 5\%$ ) and moulded under a pressure of 50 Mpa into  $5 \times 5 \text{ cm}$  cubes [18]. The cubes were dried overnight in a hot air oven at 100 °C and then fired in an electric laboratory muffle furnace (Lenton) at different firing temperatures (1150, 1200 and 1250 °C by a heating rate of 10 °C/min) with sintering duration at the maximum temperature for 1 h in furnace for all cement clinker mixes.

The theoretically expected calcium sulfoaluminate–belite cement phases were determined by adapting the Bogue method, shown in Table 4. The Bogue method is a technique used in the cement industry to estimate phase composition in Portland cement clinker from its chemical oxide composition. It was adapted for calcium sulfoaluminate–belite cement by assuming a phase assemblage of  $C_4AF$ ,  $C_4A_3\bar{S}$ ,  $C_2S$ ,  $C\bar{S}$  and  $C$ , as shown in the following equations (1)–(5) [1].

$$\%C_4AF = 3.043(\% Fe_2O_3) \quad (1)$$

$$\%C_4A_3\bar{S} = 1.995(\%Al_2O_3) - 1.273(\% Fe_2O_3) \quad (2)$$

$$\%C_2S = 2.867(\% SiO_2) \quad (3)$$

$$\%C\bar{S} = 1.700(\% SO_3) - 0.445(\% Al_2O_3) + 0.284(\% Fe_2O_3) \quad (4)$$

$$\%C = 1.000(\% CaO) - 1.867(\% SiO_2) - 1.054(\% Fe_2O_3) - 0.550(\% Al_2O_3) - 0.700(\% SO_3) \quad (5)$$

The synthesized clinkers were ground using a top planetary ball mill (Fritsch planetary mono mill Pulverisette 6) for 20 min to reach a Blaine fineness of 4500 ( $\pm 100$ )  $m^2/kg$  [19]. All the cement samples were submitted to characterization by different techniques in order to assess the reactants conversion and evaluate the phases formed on clinkering process at different temperatures [20].

## 3. Results and discussions

### 3.1. Characterization of starting raw materials

The results of X-ray diffraction (XRD) analysis of supplied starting raw materials as shown in Fig. 1 indicates that the kaolin shows the typical diffraction patterns of a well-crystallized layer lattice mineral of kaolinite with muscovite impurities as Illite mineral.

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