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Characterization of Calcium Sulfoaluminate Cement

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Abstract: Cement is a vital component of the construction industry. Modern society cannot develop without it. The Calcium Sulfoaluminate (CSA) cements are presently receiving a lot of interest, because their manufactures generate less CO₂ than Ordinary Portland Cement (OPC) and also the beneficial use of industrial by-products as raw materials. In this part my research, CSA cement clinker was synthesized and characterization has been performed through Thermogravimetry Analysis (TGA) and X-ray powder diffraction patterns (XRD).

Keywords: CSACement; RMS; TGA; Mass Loss; Phase Composition.

Introduction

Ettringite or CSA Cements are of high alumina cement that first came to prominence in the 1970s [1]. Over the years, much attention has been paid to the development of modified cement clinkers, giving rise to energy preservation as well as interests concerning its use as a binder for waste encapsulation [2]. The authors [3] discussed the laboratory invention of three aluminum-rich Belite Calcium Sulfoaluminate (BSA) clinkers with nominal mineralogical composition in the range 50–60% of C₂S, 20–30% of C₄A₃\$, 10% of Calcium mono aluminate (CA) and 10 % of mayenite (C₁₂A₇). Special cements based on CSA, can be successfully synthesized at a laboratory scale from raw mixes containing limestone, bauxite, Fluidized bed combustion (FBC) bottom and/or fly ash heated at 1200°-1300°C [4].

Materials

Raw Materials

In this study, CSA cement clinkers were produced from reagent-grade chemicals combining with RMS. Reagent-grade chemicals used were oxides of Calcium (96% CaO; Merck), Silica (95% SiO₂), Aluminum (98% Al₂O₃), Ferric (93% Fe₂O₃), and Calcium Sulfate Dihydrate (99% CaSO₄·2H₂O). The industrial waste RMS used is collected from Hindalco Industries Limited, Belgaum, Karnataka (India) and it is a solid waste from the Bauxite refining plant. Theraw material proportion and its Chemical analysis were tabulated in Table 1.

Table 1. Raw materials proportion and its Chemical analysis.

Parameter	Reagent-grade Proportion 1	Chemical analysis of 15% RMS (100%) 2	Final Raw material Proportion 1+2
CaO (wt. %)	42.00	0.22 (1.47)	42.22
Al ₂ O ₃ (wt. %)	20.00	3.24 (21.60)	23.24
CaSO ₄ (wt. %)	16.00	--	16.00
SiO ₂ (wt. %)	07.00	1.71 (11.40)	8.71
Fe ₂ O ₃ (wt. %)	--	5.745 (38.30)	5.745
MgO+Na ₂ O+TiO ₂ K ₂ O etc.,	--	2.36 (15.73)	2.36
LOI	--	1.725 (11.56)	1.725

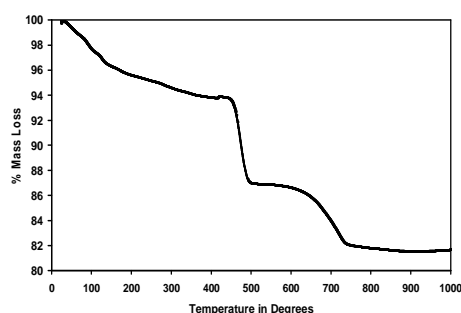
Synthesis

The proportioned raw materials were mixed with ultra pure water (1:2 ratio) by using an aluminum jar for 1 hour at 100 rpm. The solution was transferred to ceramic crucible and dried in a laboratory oven at 103°C for 24 hours. The resulted material was grinding into fine powder with a mortar and pestle. This powder material was submitted to TG for mass loss study during different calcination temperatures. The same raw materials were then collected in alumina crucibles and heated in a muffle furnace at varying temperatures of 500, 750 & 1250°C. Finally the resulted clinkers were submitted to XRD for Phase Composition.

Characterization

TGA Observations

Thermogravimetric analysis was used to observe the mass loss with temperature on raw materials during the calcination process. The instrument was operated from 25°C to 1000°C at a 10°C/min calcination rate was used for testing. The TGA provides information of the temperature ranges and corresponding mass losses. The TGA curves of the raw material are shown in Figure 1.

**Figure 1.** TGA Curve

As can be seen in the figure, there are two different mass loss peaks in TGA curve at 470 and 730°C. The first mass loss at 458-500°C, with about 6 wt %, refers to dissociation of water from raw materials. The later, mass loss with about 2wt%, occurs in the temperature range of 645-750°C due to the release of CO₂ during calcination of the CaCO₃. Finally, a mass loss is stabilized beyond the 750°C. At higher temperatures, beyond the 750°C the calcination reactions occur to produce desired complex minerals C₄A₃S, C₂S, C₄AF, and C₃S.

XRD Observations

CSA clinkers obtained from High Temperature Muffle Furnace were submitted to XRD for phase composition analysis. The powder diffraction patterns were collected over the range of 10°– 80° 2θ and a step of 0.02° for a collection time of 1 sec with Cu Kα radiation at 40 kV and 40 mA and incident wavelength of λ=1.540 Å. All the measurements were performed using BRUKER D8 Advance.

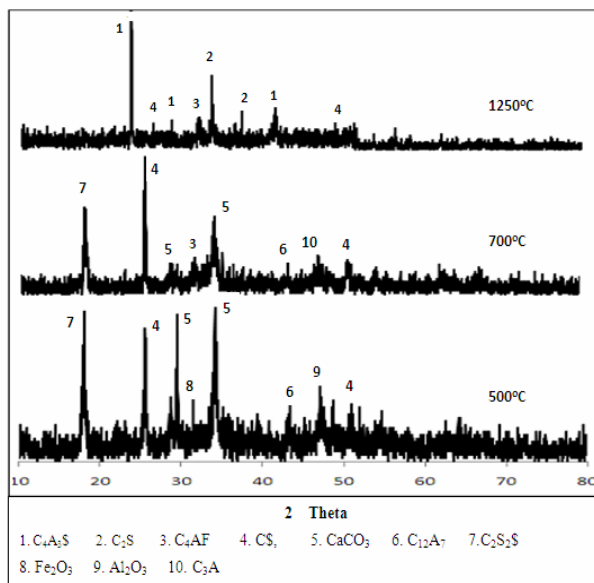


Figure 2. XRD Analysis

The phase composition result analyses of calcined temperatures (500, 750 & 1250°C) were shown in Figure 2. Once the calcination at 500°C was completed, the sample submitted to XRD and analysis showed the presence of CaCO_3 , CaSO_4 , Al_2O_3 , and Fe_2O_3 in the clinker, indicating that, dissociation of water content in the raw materials shown in Figure 2 (500°C). After burning of 750°C XRD analysis showed the presence of Tricalcium Aluminate (C_3A), Gehlenite ($\text{C}_2\text{S}_2\text{S}$), CaSO_4 , C_4AF and C_3A in the clinker, indicating that, release of CO_2 and Oxidation-reduction due to the calcination shown in Figure 2 (750°C). Finally, the absence of C_3A and $\text{C}_2\text{S}_2\text{S}$, indicating that, the final calcined temperature of 1250°C was sufficient for formation of new compounds such as $\text{C}_4\text{A}_3\text{S}$, C_2S , C_2AS and C_4AF . The high amount of $\text{C}_4\text{A}_3\text{S}$ and C_2S in clinkers indicates the high durability and rapid hardening of CSA cement.

Conclusions

1. Characterization technique allowed the identification of the compounds that appear in the clinkers.
2. Utilization of RMS as a substituent of raw material derived to produce CSA cement clinkers.
3. CSA cements with RMS were produced successfully at lower temperature (1250°C) compared to OPC driving to less energy consumption.
4. Optimum calcined temperature for the formation of $\text{C}_4\text{A}_3\text{S}$ and C_2S in CSA cement clinker was found to be 1250 °C for the raw materials containing 15 wt % RMS.
5. Use of similar raw materials and production possible in existing industrial by-products.

References

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