## **Research Article**

# A spectroscopic study on ternary blended cement concrete

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#### **ABSTRACT**

This works aims to study the effect of Silica Fume (SF) on the properties of High Alumina Cement (HAC), Ordinary Portland Cement (OPC) and the addition of 5, 10, 15 and 20%SF were prepared using Groundwater. The nature of the reactions was dependent on the amount of water (W/C=0.4) present in the paste. The grinded samples were characterised by using XRD analysis. The experimental results of the compressive strength and setting time were measured. The highest increase of compressive strength was achieved in the early period of setting which indicates an improvement in the early hydration of the mixture. The kinetics of hydration is well explained and evidenced that 10% addition of SF is optimum for this blend.

Key words: High alumina cement, XRD, Compressive strength, Silica fume

#### INTRODUCTION

The mechanism of hydration of HAC is via solution, where an anhydrous phase dissolves and is followed by the precipitation of the hydrates from solution. Three distinct phases can be identified; dissolution, nucleation and precipitation. The hydration process is initiated by the hydroxylation of the cement surface followed by dissolution of cement in water and the liberation of calcium and aluminium ions. A small amount of gel like hydrates will form at this point if the solution concentration rises over the super saturation limit level of the hydrates  $\rm C_2AH_8$  and  $\rm AH_3$ . The dissolution will continue with a consequent increase in the concentration of calcium and aluminium ions until a saturation point is reached. After the dissolution phase there follows an induction period during which nuclei attain a critical size and quantity.

$$3CA+12H \longrightarrow C_3AH_6 + 2AH_3$$

When ordinary Portland cement is hydrated its main components are tricalcium silicate and dicalcium silicate are transformed into hydration products mainly CSH gel and portlandite (Amer, 2007). An AF phase is also formed in the presence of gypsum and in other compounds. The use of siliceous by-products such as fly ash and silica fume in making mortars to modifies the compressive strength, pore structure and permeability of hardened material. The addition of silica fume in Portland cement to reduce the thermal cracking caused by the heat of cement hydration, improve durability to attack by sulphate and acidic waters and increase in strength. Blended cements are produced by intergrading different amount of various supplementary cementing materials. The blended materials may be either pozzolanic in nature like SF etc., the performance of blended cement in many respects is superior to alumina cement (Barnes et al., 1978). The present investigation it has been planned to mix HAC, OPC and SF in different concentrations. The hydration process can be identified and explained using any analytical tool.

## **MATERIAL AND METHODS**

Ordinary Portland Cement (OPC), High Alumina Cement (HAC) and Silica Fume (SF) supplied by Coromondal Ltd, Chennai, ACE refractory, Chennai and Polygon Chemicals Ltd, Mumbai of the chemical composition is given in Table 1. The hydrated samples were stored at the given relative humidity, RH at 300C. The blended cements were prepared with 85%HAC+10%OPC+5%SF, 80%HAC+10%OPC+10%SF, 85%HAC+10%OPC+15%SF, 85%HAC+10%OPC+20%SF and well mixed with the help of minivibrator at water/cement ratio=0.4. The samples were hydrated at various time intervals. After dehydration the samples were powdered and analyzed using XRD analysis.

#### **RESULTS AND DISCUSSIONS**

X-ray powder diffraction (XRD) measures the angular dependent X-ray reflection from crystalline phases within a powder sample. If there are crystallographic structural models for the crystalline materials being analyzed. The process of estimating mass fractions using the crystallographic structural models is part of the Rietveld analysis is the basis of X-ray powder diffraction. If there are only crystalline phase present in the sample and structural analysis for each phase can be obtained by the Rietveld analysis can be used to determine the mass fraction of each phase. The XRD studies of cement hydration products demand a careful presentation with samples as the studies are related to discrete ages of hydration and the specimen may often interact with various other reactants in the atmosphere.

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|     |                  |          |        |        |          |         |          |  |

| Materials |       | Oxides    |                  |                                     |      |                   |                 |        |  |  |  |  |
|-----------|-------|-----------|------------------|-------------------------------------|------|-------------------|-----------------|--------|--|--|--|--|
|           | Cao   | $Al_2O_3$ | SiO <sub>2</sub> | Fe <sub>2</sub> O <sub>3</sub> +Feo | MgO  | Na <sub>2</sub> O | SO <sub>3</sub> | Others |  |  |  |  |
| HAC       | 38.5  | 39        | 4.5              | 14                                  | 0.4  | 0.1               | 0.15            | 3.15   |  |  |  |  |
| OPC       | 63.41 | 5.45      | 21.45            | 3.42                                | 2.59 | 0.38              | 2.39            | 0.91   |  |  |  |  |
| SF        | 0.45  | 1.17      | 93.14            | 3.13                                | .98  | 0.30              | 0.10            | 0.73   |  |  |  |  |

On hydration the recorded XRD patterns are observed with a reduction in intensity of main phases but accompanied by an increase in intensity of their hydration products. It is also observed that at any moment the intensity of these peaks depends upon the relative concentration of their own constituents. A higher intense peaks of main hydration by products of hydrated samples are CAH<sub>10</sub> peak at d=7.10 Å (101),  $C_2AH_8$  peaks at d=2.88 Å (110) are observed (Barathan *et al.*, (2009). The overlapped peaks at d=2.78 Å (003) belong to  $C_2AS_8$ . The more intense peaks at d=2.87 Å belongs to stratlingite. The new peaks seen at d=3.27Å (231) are due to  $C_4AH_{13}$ . In addition to calcite peaks at d=3.03 Å (104) the vaterite peaks at d= 3.29(112) is also seen with lesser intensity.

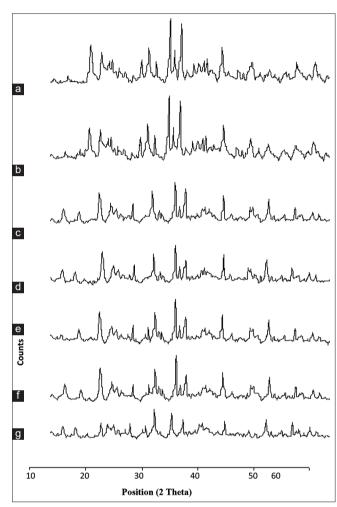
With increasing the time of hydration a gypsum peak at d=2.87 Å (200) can be found. The gypsum peak decreases with an increasing intensity of the peak at d=3.88 Å (114) which suggests the formation of ettringite. The intensity of the C<sub>3</sub>AH<sub>6</sub> peak increases until all gypsum is consumed. More free gypsum existing in the system corresponds to an acceleration of the system compared to pure HAC and control. This may be due to the higher surface area of silica fume (Jianmiao & Shizong, 2005). When gypsum reacts with C<sub>3</sub>AH<sub>6</sub> calcium sulphoaluminate hydrate and monosulphate hydrate are formed (Midgley & Rao, 1978). This makes the paste from stiffen to hardened state and is referred as final setting time and these results are confirmed by Vicat's test measurements.

An increase in intensity of the peak due to CAH<sub>10</sub>, C<sub>2</sub>AH<sub>8</sub>, C<sub>2</sub>S and C<sub>3</sub>S are the characteristic peaks of HAC and OPC respectively. The peaks at C<sub>3</sub>AH<sub>6</sub> and AH<sub>3</sub> are lesser intensity. This is probable evidence that the presence of SF prevent the conversion process of hexagonal calcium aluminate phases (CAH<sub>10</sub>, C<sub>2</sub>AH<sub>8</sub>) to a stable cubic form of hydrogarnet (C<sub>3</sub>AH<sub>6</sub>). The peaks at d = 4.84Å (002), 4.71 Å (001), 4.37 Å (110) and 2.22 Å (201) can be assigned to polymorphs of aluminium hydroxide. As time ellapses, the bayerite peak increases whereas the gibbsite band increases reducing the strength than the HAC. At 4<sup>th</sup> week, the peaks at d=4.84 Å (002) and 4.37 Å (110) reduce probably due to the addition of SF. As time eludes, the Ca (OH)<sub>2</sub> phase decreases with increase in C<sub>2</sub>ASH<sub>8</sub> implying the strength to be higher. The generation of these products can be well explained by the following equations.

$$2(3\text{CaO.SiO}_2) + 6\text{H}_2\text{O} \longrightarrow 3\text{CaO.2SiO}_2.3\text{H}_2\text{O} + 3\text{Ca} \text{ (OH)}_2$$

$$3\text{Ca} \text{ (OH)}_2 + 2\text{SiO}_2 + \text{H}_2\text{O} \longrightarrow 3\text{CaO.2SiO}_2.3\text{H}_2\text{O}$$
For ternary system
$$C_2\text{AH}_8 + 2\text{CSH} + \text{AH}_3 + 3\text{H} \longrightarrow 2C_2\text{ASH}_8$$

$$C_3\text{AH}_9 + \text{AH}_2 \longrightarrow C_3\text{ASH}_9 + 3\text{H}$$



**Figure 1:** XRD spectra of hydrated ternary sample treated with GW containing 80%HAC + 10%OPC + 10% SF (a) 1 hour (b) IST (c) FST (d) 1 day (e) 1 week (f) 4 week (g) 12 week

From 4<sup>th</sup> week the intensity of the stratlingite peak is higher with a decrease in intensity of the other peaks indicate the strength is higher. The total porosities of cement paste containing SF are lower that the control. In Figure 1, the addition of 5%SF to HAC the strength shows a progressive increase during the different periods of hydration. When compared to pure HAC, the strength is slightly lesser. This is due to the effect of amorphous silica in SF on the kinetics of formation of stratlingite at the expense of the cubic hydrate of C<sub>3</sub>AH<sub>6</sub>. During the progress of the reaction the XRD due to bayerite completely disappears at higher order increasing of the gibbsite peaks. Due to this strength falls off rapidly than the GW paste (Zhang *et al.*, 1997; Yogendran *et al.*, 1991)

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When the addition of SF increases to 15 and 20% due to pozzolanic reaction the setting time advances whereas the strength decreases and its almost equal to 5%SF additions in character at later time.

#### CONCLUSIONS

- As percentage of SF increases the stratlingite amount increases, this stratlingite is responsible for strength.
- At 4<sup>th</sup> week the carbonate polymorph will be in the order for GW paste aragonite calcite vaterite
- In the initial period of hydration the amount of bayerite and gibbsite is higher at later period of hydration

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