

# Mechanical and Microstructural Characteristics of Ternary Blended Mortars Incorporating GGBS and Alccofine Subjected to Acid and Sulphate Exposure

Arnab Mondal, Kushal Ghosh, Partha Ghosh

**Abstract:** *This experimental study has been carried out to investigate the behavior of ordinary cement mortars and mortars containing ternary mixtures of supplementary cementitious materials (SCM's) like Ground Granulated Blast Furnace Slag (GGBS), Alccofine along with cement, when subjected to exposure in magnesium sulphate and sulphuric acid solution. Mixes were prepared by replacing OPC with 30% and 50% by GGBS along with 5%, 10% and 15% by Alccofine as additives. Compressive strength test were performed on different samples to observe SCMs behavior in different exposure conditions (acid and sulphate) after 56 days. Microstructure studies using scanning electron microscopy (SEM) and electron dispersive X-ray (EDX) analysis were performed on samples after different exposure conditions.*

**Keywords-** Alccofine, Electron dispersive X-ray analysis, Ground Granulated Blast Furnace slag (GGBS), Scanning electron microscopy.

## I. INTRODUCTION

The recent trend towards sustainability becomes inevitable in construction industries as large scale production of cement causing environmental impact and depletion of natural resources. Emission of SO<sub>2</sub> and NO<sub>x</sub> along with CO<sub>2</sub> released from cement industry can cause the greenhouse effect and acid rain [14, 22] raising issues related to climate change, enhances serious consequences of rise in sea level. Thus importance has been given on optimum utilization of OPC by partially replacing it with various industrial by products such as Ground Granulated Blast Furnace Slag (GGBS), Alccofine and Silica fume [21]. These are the industrial produced from thermal power plants, electric arc furnaces etc and are finer (micro and nano) in nature. Due to their fineness they are capable of filling the pore space causing refinement of the concrete and making it more impermeable. The use of different (SCMs) leads to reduction in rate of hydration process in concrete and thus minimizing the heat formation during hydration process at the same time they produce more workable mixes having low w/b, rectification is not possible.

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Ratio. The formation of Calcium Silicate Hydrate (C-S-H), which leads to attain ultimate strength to concrete is enhanced due to addition of these by products. Many concrete structures are often exposed to different aggressive chemical (acid and sulphate) environment. Most common example is corrosion of concrete sewer due to formation of biogenic sulphuric acid. Other such examples are effluents from tannery, dairy firm, refinery industries and offshore concrete structures which often causes chemical degradation of concrete. Addition of different SCM's improve the microstructure of the concrete, thus improvement against chemical attack and corrosion resistance [20]. So durability against H<sub>2</sub>SO<sub>4</sub> and MgSO<sub>4</sub> of concrete is of major concern in this study. Therefore there is a substantial need for construction of durable concrete structures in such environments. This Paper investigates the durability properties of concrete by replacement of cement by Alccofine and GGBS in different proportions with natural sand

## II. LITERATURE REVIEW

Durability study of concrete is nowadays a major issue as concrete structures often subjected to harsh and aggressive environment. Addition of different SCM's enhances packing and extent of hydrated product generated during hydration. The secondary hydrated product formed due to pozzolanic and cementitious hydration reaction fills the pores and reduces the ingress of aggressive agents in concrete and extend the durability of concrete. **Vireen Limbachiya, Eshmaiel Ganjian, Peter Claisse** [1] reported that the water absorption of control mix is greater than the other SCMs mixes. Extreme fineness and greater formation of CSH gel providing greater matrix densification and decrease in water absorption. **D. Sivakumar T. Hemalatha (2015)** [2] conducted tests on the durability and mechanical characterization of concrete using Alccofines and concluded that with the inclusion of Alccofine reduction in the permeability significantly. **P.K Mehta Paulo J. M Monteiro** [3] described the sulfate attack in the form of expansive materials due production of gypsum leading to cracking and ingress of the aggressive water more easily into the interior. Presence of different cations related to the sulfate solution both Portlandite and C-S-H in the hydrated Portland cement paste may be converted to gypsum by sulfate attack. Magnesium sulfate attack, has serious consequences as the conversion of calcium hydroxide

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to gypsum along with formation of magnesium hydroxide, which is insoluble and in turn reduces the formation of further C-S-H gel. **Ahmet Benli Mehmet Karatas , Elif Gurses** [4] explained the addition of different pozzolans like blast furnace slag and silica fume attributes to significant reduction in portlandite and alumina responsible for producing gypsum and ettringite, thus improvement in pore structure. **Mohammed Qureshi ,Yogendra Tandel, Bhavin, Patel (2014)** [5] investigated the effect of Alccofine on durability against acidic environment and suggested that with increase in fly ash content and decrease in Alccofine reduces the weight loss as well as the compressive strength after 28 days immersion in 5% sulfuric acid solution. **M.M Magdum, V. V. Karjinni (2017)**[16] have suggested that permeability of concrete blended with Alccofine reduce to great extent and has moderate resistance against thermal exposure. Another durability issue is chloride permeability in concrete.

## III. EXPERIMENTAL PROGRAM

The main objective of this investigation is to develop an eco friendly sustainable mortar mix by using varying percentages of GGBS as well as ALCCOFINE as partial replacement of OPC and to make a comparative study between the conventional concrete and concrete prepared with GGBS and ALCCOFINE micro materials in terms of durability exposures.

### A. Materials used and specimens preparation

#### B. Ordinary Portland cement

Ordinary Portland cement of 43 Grade (Ultratech) conforming to IS 8112-2013 has been used for this investigation..

#### C. Fine aggregates

River sand of grading Zone-II as per codal provision of IS-383-1970(Reaffirmed 2002) has been used in this experimental study.

#### D. Ground Granulated Blast Furnace Slag(GGBS)

GGBS is a by-product formed in the iron industries when molten iron blast furnace slag of about 1500°C temperature is rapidly chilled by quenching it in water and it turns into a fine, granular, almost fully noncrystalline, glassy form [13,30] . It is also called slag cement having average particle size of 45 microns, and specific surface area of about 400 to 600 m<sup>2</sup>/kg using Blaine method[6]. GGBS so far used in this investigation has been acquired from M/S Owndust India Ltd. Fig. 1.(a) represents the normal and surface morphology at microstructure level of GGBS powder by SEM. SEM image clearly indicates the GGBS grains characterized by their angular and flakey shapes.

#### E. Alccofine 1203

Alccofine 1203 is a new generation micro fine materials specially produced by product from slag having high glass content and high reactivity obtained through the process of controlled granulation[18]. Owing to its unique ultra fine particle size, having fineness greater than 12000 cm<sup>2</sup> /gm, Alccofine 1203 provides reduced water demand for a given workability[15,17,26]. Alccofine 1203 used in this

investigation has been acquired from M/S Counto Microfine Products Pvt. Ltd.

### F. Superplasticisers

MasterGlenium ACE30 a brown coloured modified polycarboxylic Ether based high range liquid super plasticizer was used to prepare workable mix in the laboratory. After addition of sufficient quantity of water say 70-90% to the mix a suitable quantity of superplasticizer of about 1% of total cementitious materials has been used for preparation of different mixes in each batches. Depending on the particles size and specific surface area of the mineral admixtures the quantity of super plasticizers may increase .

### G. Specimen preparation

The samples have been made by preparing total 7 separate mixes (including control mix ) in which 6 mixes were prepared using 30% & 50% replacement levels of cement by GGBS incorporating 5%,10% and 15% Alccofine 1203 by weight of total cementitious materials . The different mixes employed in this investigation are tabulated as shown in the table-1.Mixing of cementitious materials and sand were carried out using cementitious materials and sand ratio of 1:2.75 conforming to ASTM C 109[7] and stirred with the help of mechanical stirrer after adding water maintaining a w/b ratio of 0.36. It was critical to maintain such w/b ratio as the mineral admixtures used were ultrafine in nature and absorbed water due to high surface area. So adding modified polycarboxylate ether based superplasticizer(MasterGlenium ACE 30 ) at the rate of 1% by weight of total cementitious materials. Special care should be taken while adding superplasticiser and it was recommended to mix superplasticizer after adding some water say about 70-90% of total water required for each batch to the mix and stirred for 3 min. Each mix of desired slump flow diameter values were produced before pouring in to the molds different mixes. After preparing the mixes samples were casted in 50mm x 50mm x 50mm cube molds in layers with trowel on a vibrating table. After demolding the cubes were kept immerse in water for curing for 7 and 28 days respectively. Some samples after curing for 28 days were kept for another 56 days curing in 4% H<sub>2</sub>SO<sub>4</sub> and 4% MgSO<sub>4</sub> solution.

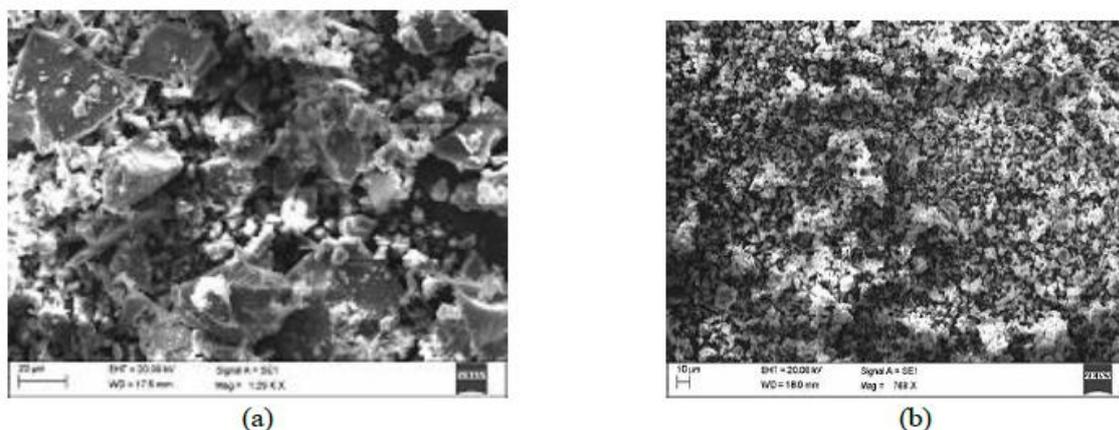


Fig 1: SEM morphology of (a) GGBS (b) Alccofine grains used in this investigation.

### III. TEST METHODS

#### A. Sorptivity test

This test was introduced by Hall in 1970 studying the importance of unidirectional flow of water in concrete specimen. The cumulative quantity of absorbed water per unit area of inflow surface are determined and relate to the square root of the elapsed time .The relationship is given by

$$I = S_i \cdot t^{0.5}$$

Where,

$I$ =The cumulative absorbed volume of water per unit area of inflow surface , in mm.

$S_i$ =Sorptivity or coefficient of sorptivity, in mm/ $t^{0.5}$ .

$t$  = Time elapsed expressed in square root .

For evaluating sorptivity test each mixture containing three identical samples for each specimen mix for different proportion of SCMs were prepared and cured for 28 days. After curing for 28 days the immersed samples were taken out from water and wiped the surface off with the help of a piece of dampened cloth and kept in air until saturated surface dry condition. Then put them in to oven at  $110 \pm 5^\circ\text{C}$  and dried for not less than 24 hours .Samples were dried until reached a constant weight . To reduce evaporation and obtain unidirectional water flow, samples were coated with water proofing paint on four sides and remaining opposite sides kept open. Immediate after drying the paint the initial weight of the samples were taken .Placed the support device at the bottom of the pan and filled the pan in such a way that the top of the water level was 1 to 3mm above the support devices and maintained the water level for the total duration of the test . Put the samples on the support devices and weighed the samples at regular intervals as mentioned in ASTM C 1585-04.[8].During each measurements samples were taken out and wiped the wetted surface with a damp cloth .Immediate after taking measurements the samples were placed on the support devices. Any tolerances during weighing and recording time was maintained conforming to ASTM C 1585-04.[8]

#### B. Samples subjected to acid and sulphate exposure

After curing in water for 28 days three identical samples containing specific proportions of SCMs for each test were chosen and subjected to 4%  $\text{H}_2\text{SO}_4$  and 4%  $\text{MgSO}_4$  exposure condition for another 56 days in separate plastic containers. For this acid and sulfate solutions were prepared in the laboratory by adding  $\text{H}_2\text{SO}_4$  (concentration level of 98% ) and  $\text{MgSO}_4$  to water and diluted to obtain 4%  $\text{H}_2\text{SO}_4$  and  $\text{MgSO}_4$  solution . Then the samples were immersed in the solution in separate container. The pH level of the sample was maintained at an interval of one week.

#### C. Microstructural studies at different unexposed and exposed condition

The microstructure of the hydrated products change phases depending upon different factors such as chemical composition , fineness of the cementitious materials ,water binder ratio ,use of chemical admixtures, variation in hydration condition and nature of curing [9]. So the samples need to be investigated at microstructure level before and after exposed condition. Microstructural study also helpful in determining the chemical components as well as phase determination at different ages of the sample [3] .So far two type of studies conducted namely scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDX) for microstructural studies. First one was used to determine the phase composition at different ages and the later one for determining the chemical composition of the samples. Secondary Electron (SE) Dispersion mode of SEM is one of the method of different SEM imaging techniques. For obtaining better effects generally samples were coated with carbon or platinum coating as coated samples provide a thin surface for receiving better images.In microstructural studies sample were categorised into different distinct exposure classes viz. normal or unexposed and chemically(acid and sulphate) exposed condition for better understanding of the effect of different environment the samples were underwent.. Special care must be taken for samples collected for chemical durability ,as it requires the inner and outer core of the affected samples separately for microstructural studies and collected in separate polythene packet.

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Table -I:Details of proportion of different mixes with different replacement levels of OPC,GGBS and Alccofine.

SI No	Mix	Cementitious materials			Binder/sand	SP* (%)	W/b
		Cement(%)	GGBS(%)	Alccofine(%)			
1	MOPC	100	0	0	1:2.75	1	0.36
2	GG30AF5	70	30	5	1:2.75	1	0.36
3	GG30AF10	70	30	10	1:2.75	1	0.36
4	GG30AF15	70	30	15	1:2.75	1	0.36
5	GG50AF5	50	50	5	1:2.75	1	0.36
6	GG50AF10	50	50	10	1:2.75	1	0.36
7	GG50AF15	50	50	15	1:2.75	1	0.36

\*SP=Superplasticiser.

## IV. RESULTS AND DISCUSSION

### A. Sorptivity test

Among durability related tests, sorptivity test is one of the most effective test to determine ingress of moisture and nature of the pore network of concrete in terms of capillary action. Sorptivity co-efficient values for different mixes were determined as described in ASTM C 1585-04. As discussed earlier that higher sorptivity coefficient value indicates considerable damage potential at early age [10,29]. It can be seen from Fig :2 that GG30AF15 and GG50AF5 were subjected to considerable ingress of water having sorptivity coefficient values  $31.7 \times 10^{-4} \text{ cm/s}^{0.5}$  and  $38 \times 10^{-4} \text{ cm/s}^{0.5}$  respectively. On the other hand higher values of sorptivity co-efficient signifies lower resistance to water penetration which is an indicative measure of lesser pore size causing greater capillary suction in case of alccofine. Increase in percentage replacement both by GGBS and alccofine improved matrix densification to some extent

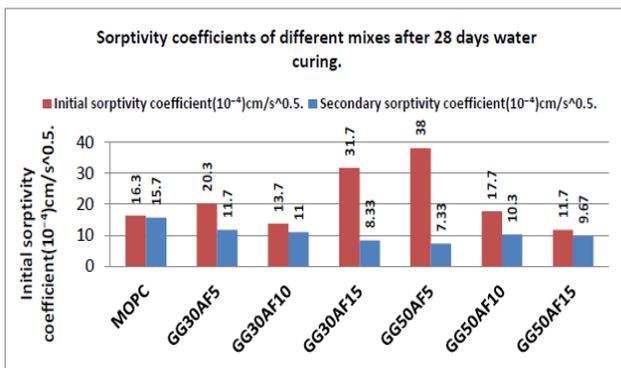


Fig:2: Sorptivity coefficients of different mixes after 28 days water curing.

### B. Acid and sulphate exposure

### C. Change in weight

Acid or sulphate diffusion in to concrete causes depletion of C-S-H gel accompanied by formation of gypsum which eventually results in change in weight. In addition to gypsum, formation of ettringite takes place .Both of them are expansive products and responsible for significant change in weight in terms of weight loss or gain[24,27]. Fig. 3. depicted the percentage change in weight of different mixes in comparison with control mix after 4%  $\text{H}_2\text{SO}_4$  and  $\text{MgSO}_4$

exposure for 56 days. Observation made so far revealed that the trend in weight loss was significant both for control mix as well as mixes blended with different percentage level of alccofine and GGBS. GG30AF15 suffered greater weight loss (9.07%) as compared to control mix(4.81%) except for GG50AF15 where weight gain(shown as negative values) occurred to the extent of 4.07% due to formation of expansive product This may be due to the fact that depletion of CH or portlandite content in SCM's mixes attributed to the formation of secondary C-S-H gel, thus contribution of CH in the formation of gypsum is secondary rather than decomposition of C-S-H gel leads to dissolved solution (Calcium Sulphate Hydrate ) when exposed to severe sulphuric acid attack [28]. Conversely greater formation of expansive products occurred in case of  $\text{MgSO}_4$  exposure compared to sulphuric acid exposure leading to weight gain (Refer Fig 3(b) ). In comparison to other mixes control mix got marginal weight gain to the tune of 0.29%. Ternary mixes incorporating GGBS and alccofine also have marginal weight gain varying from 0.15 % to 0.42 % except for mix GG50AF10 where slight loss in weight was observed [25] having value 0.46%. Contribution to the formation of expansive materials was relatively greater in case of  $\text{MgSO}_4$  exposure as the formation of MH or Brucite which is insoluble in nature and in turn increases the weight as observed in almost all the mixes[28].

### D.Strength Deterioration factor after acid and sulphate attack

This is an indicative measure of damage in terms of compressive strength loss in concrete during acid and sulphate exposure[23] .

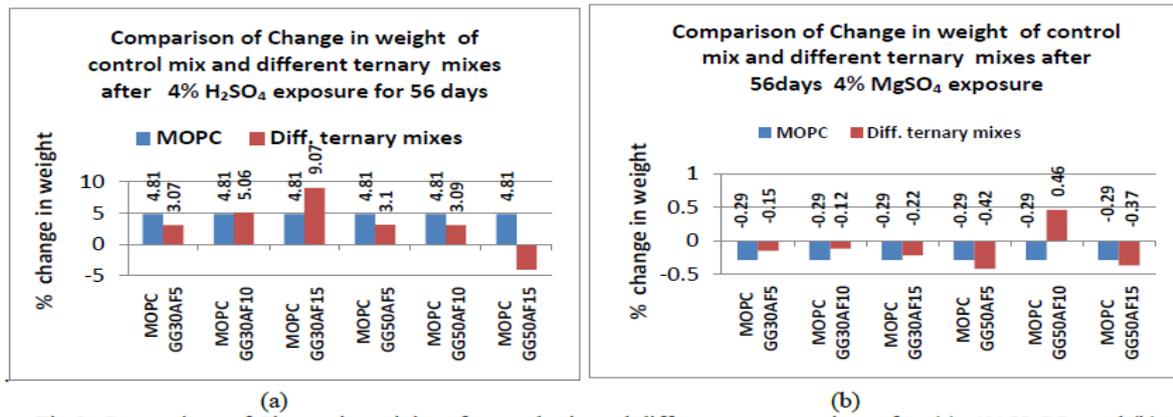


Fig.3: Comparison of Change in weight of control mix and different ternary mixes after (a) 4% H<sub>2</sub>SO<sub>4</sub> and (b) 4% MgSO<sub>4</sub> exposure for 56 days.

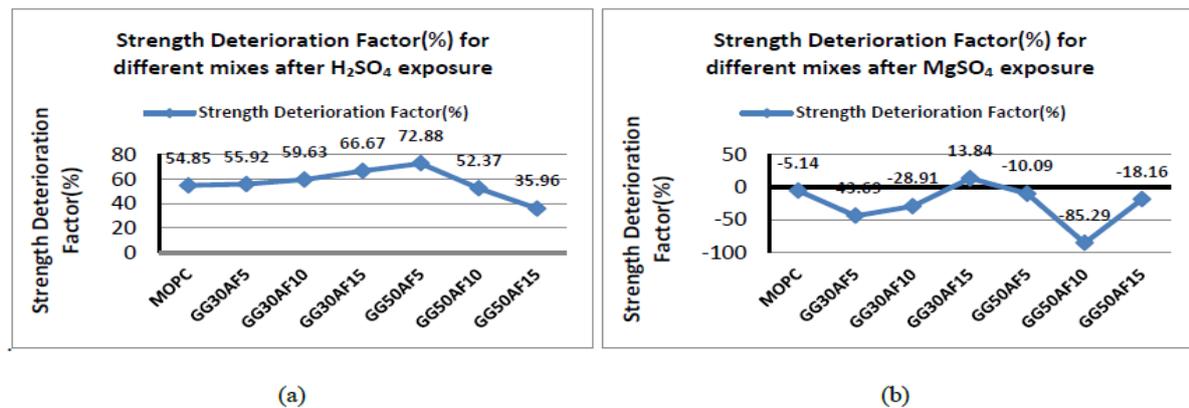


Fig. 4. Strength Deterioration Factor(%) for of control mix and different ternary mixes after (a) 4% H<sub>2</sub>SO<sub>4</sub> and (b) 4% MgSO<sub>4</sub> exposure for 56 days.

$$\text{Strength Deterioration Factor (\%)} = \frac{(f_{c28} - f_{\text{expose}})}{f_{c28}} \times 100$$

Where,

$f_{c28}$  = Average compressive strength of cube specimens curing in water for 28 days in (MPa).

$f_{\text{expose}}$  = Average compressive strength of cube specimens immersed in acid or sulfate solution for 56 days in (MPa).

Observation made so far indicates that GG50AF5 suffered significantly with an SDF value of 72.88%. In contrast mix GG50AF15 offered greater resistance to sulphuric acid exposure having a minimum SDF value of 35.96%. In ternary mixes incorporating GGBS and alccofine the presence of GGBS influences the resistivity significantly. Results confirmed that with lower percentage replacement level of GGBS(30%) accompanied by incremental percentages levels of alccofine there is an increasing trend in SDF values ranging from 55.92% to 66.67%. But with increase percentage level both in GGBS(50%) and alccofine offered better resistance against acid attack as indicated in the Fig.4(a). There is clear evidence that the increase in SCM's in mixes triggering steep depletion in Ca(OH)<sub>2</sub> or portlandite level which exacerbate the gypsum formation predominantly

by decalcification of C-S-H gel. Control mix shows better performance compare to SCM's mixes after 56 days of acid exposure. This may be due to lower depletion level of Ca(OH)<sub>2</sub> or portlandite contributing primarily to the formation of gypsum rather than C-S-H gel. The graphical representation of the SDF values of different mixes as shown in Fig:4(b). Massive strength gain observed for mix GG50AF10(85.29%). For other mixes incorporating GGBS(30%) and alccofine the lower percentage replacement of alccofine inevitably produced better result (43.69%) for mix GG30AF5 but with increase in alccofine content consequent increase in SDF values as observed from fig:4.32. Finally an SDF value of 13.84% obtained with 15% replacement of alccofine. A reverse trend observed with increase in GGBS (50%) percentage which implies that GGBS reduced the pozzolanic activity causing ample Ca(OH)<sub>2</sub> or portlandite available in the solution and consequently increase in the extent of Mg(OH)<sub>2</sub> or Brucite formed as a result of Ca(OH)<sub>2</sub> reacting with MgSO<sub>4</sub>, is relatively insoluble product clogs the pores and prohibits the further intrusion of deleterious materials, thus protects the C-S-H gel from MgSO<sub>4</sub> attack leading to increase in strength. Mix GG50AF10 achieved an increase in strength of 85.29% whereas further increase in alccofine causes reduction in strength up to 18.16% for mix GG50AF15.

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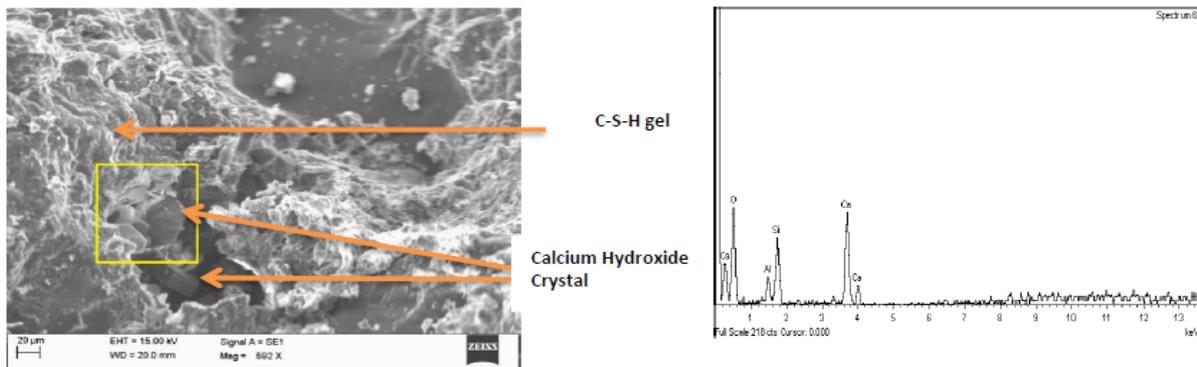


Fig 5 :SEM image and corresponding EDX profile of GG30AF10 mix after 28 days water curing.

## E. Microstructural studies at different unexposed and exposed condition

Microstructure studies using SEM and EDX were performed to investigate the change in hydration state at different ages as well as for better understanding of the surface morphology of different samples at normal and different exposure conditions. For mix GG30AF10 SEM image confirmed the traces of few portlandite or CH. In addition to the formation of C-S-H gel the presence of unreacted GGBS along with alccofine grains surrounded by C-S-H gel also observed as shown in Fig:5. This may be due to the fact that the hydration degree of GGBS is very low of even after 28 days. Similar observations have also been reported by **Banti A. Gedam et.al(2015)**[11]. EDX profile showing high peaks of Ca, Si which may indicate sufficient formation of C-S-H gel with limited occurrence of portlandite. The Ca/Si ratio confirms calcium deficiency clearly associated with the consumption by reaction with SCM's to form more C-S-H gel. In SCM's blended mixes calcium content decreased considerably, while silicon and aluminium contents increased causing significant change in ratios of Ca/Si and Al/Ca respectively. Observations made by **J.I. Escalante et.al (2004)**[12] revealed similar outcomes. Samples subjected to 4% H<sub>2</sub>SO<sub>4</sub> solution after 56 days underwent microstructural investigation found occurrence of C-S-H gel dissolution. SEM image of GG50AF15 (Fig:6) confirmed scarce dissolution of C-S-H gel accompanied by formation of gypsum. This may be the possible cause of weight gain due to formation of expansive materials as observed earlier in the experimental results section. There was rarely observed portlandite or CH content in the SEM image which may be clearly suggested the more formation of gypsum primarily by consuming CH rather than decalcification of C-S-H gel. SEM image of mix GG50AF10 revealed the dissociation of C-S-H gel over the entire surface when exposed to MgSO<sub>4</sub> solution for 56 days (Fig:7). In addition to the disintegration of C-S-H gel rare formation of ettringite (expansive product) was also observed causing weight loss of the sample as reported earlier in experimental result section. Formation of MH or Brucite was quite prominent as confirmed in EDX spectrum. Production of Brucite or MH as observed is relatively insoluble in nature and clogged the pores, thus prohibited the further intrusion of deleterious materials. It acted as protective shield for the C-S-H gel against further deterioration from MgSO<sub>4</sub> attack.

This observation made is in agreement with the strength deterioration factor values as observed from the experimental data.

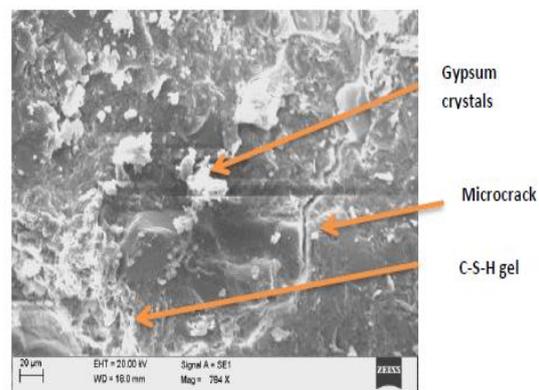


Fig 6:SEM image of GG50AF15 mix after 56 days of H<sub>2</sub>SO<sub>4</sub> exposure

## V.CONCLUSION

Based on observations and experimentally obtained results the following conclusions can be drawn:

1. Sorptivity test was carried out to assess the transport characteristics of deleterious materials in to the concrete. Initial sorptivity coefficient values observed for different mixes confirmed that lesser number of pore networks were present in GGBS, alccofine ternary mix series compared to control mix.
2. Behaviour of different mixes exposed to H<sub>2</sub>SO<sub>4</sub> and MgSO<sub>4</sub> solution were expressed in terms of both change in weight and also strength deterioration factor values. In both the cases GGBS alccofine mix series performed better with respect to control mix.
3. Mixes exposed to MgSO<sub>4</sub> solution underwent gain in weight in almost all the samples and also exhibited negative strength deterioration factor i.e strength gain instead of strength loss. GGBS, alccofine mix series showed superior performance compare to control mix.
4. SEM image revealed dense C-S-H matrix formation accompanied by presence of CH or portlandite in GG30AF10 after 28 days of water curing. Traces of gypsum and ettringite formation were found from SEM image of alccofine mix series after H<sub>2</sub>SO<sub>4</sub> exposure for 56 days. Deterioration of C-S-H gel coupled with the



formation of Brucite and Portlandite were quite prominent as observed from EDX spectrum for alccofine samples.

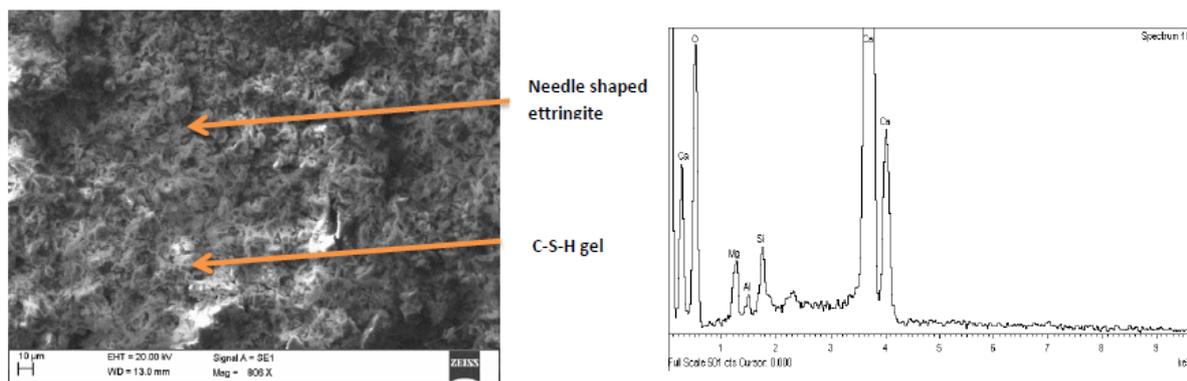


Fig 7:SEM image and EDX profile of GG50AF10 mix after 56 days of MgSO<sub>4</sub> exposure.

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