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Micro-Structural Study on Hydration Process of Self-Compacting Concrete

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Abstract:- This research work focuses on the experimental investigation on compressive strength and micro-structural property of Self-Compacting Concrete (SCC) of various mix proportion of fly ash (FA) and ground granulated blast furnace slag (GGBFS). Moreover, utilization of mineral admixtures in SCC not only reduces cost but also reduces the heat of hydration and gives a dense structure. Initially, optimization is done using compressive strength as parameter to finalize the proportion of fly ash and GGBFS for micro-structural analysis. Samples of 14th and 28th day cubes were collected from the optimized mix proportion and subjected to Scanning Electron Microscope (SEM), X-Ray Diffraction (XRD) and Thermogravimetric Analysis (TGA). The images were interpreted and the result from micro-structural analysis were compared with their compressive strength. The experimental result showed that mix with 20% fly ash had better compressive strength and formation of hydrated products compared to other mixes.

***Keywords**:- Self-Compacting Concrete, Fly Ash, Ground Granulated Blast Furnace Slag, Scanning Electron Microscope X-Ray Diffraction and Thermogravimetric Analysis.*

I. INTRODUCTION

The most widespread composite building material used is concrete, which is a combination of aggregates (mostly gravel and sand) and cement binder. In present scenario with improved technology, there are different types of concrete with different composition mixtures which possess different properties. Based on their properties, their application varies.

SCC was first developed by Prof. Okamura and his team in 1988 at University of Tokyo, Japan. The original concept of developing SCC was to enhance long-term durability of structures having congested reinforcements, but it eventually developed to be user-friendly concrete. Self-compacting concrete is highly flowable concrete, which fills the formwork and encapsulate the reinforcement by its self-weight and eliminate the need for mechanical vibrator. SCC can be achieved from mixing Portland cement, aggregates and water along with mineral admixture such as fly ash, ground granulated blast furnace slag and condensed silica fumes and chemical admixtures such as superplasticizer and viscosity modifying agents.

Many techniques have been in co-operated into SCC for the betterment purpose, in such way this research work is done to study the microstructure of the concrete using Scanning Electron Microscope (SEM), X-Ray Diffraction (XRD) and Thermogravimetric Analysis (TGA) at different stages such as 14th day and 28th day for different types of mixes where cement is partially replaced with Fly Ash and GGBFS.

A. Research Aim and Objective

The ultimate aim of this research work is to infer the micro-structural characteristics of Self-Compacting Concrete during the hydration process, using mineral admixture fly ash (FA) and ground granulated blast furnace slag (GGBFS) in different proportion as cement replacement material, along with chemical admixture. The following analysis were made to study the micro-structural characteristics of concrete.

➢ *Scanning Electron Microscope (SEM)*

It is the study of particle morphology in microscopic level where morphology is the study of form, shape or structure of a particular thing. SEM is used to find the degree of hydration of cement, formation and unreacted particles.

➢ *X-Ray Diffraction (XRD)*

It is used to study the structure, composition, and physical properties of materials. They are used to find phase composition of cements and clinkers. It gives the phase identification for the SEM results.

➢ Thermogravimetric analysis (TGA)

It gives physical and chemical properties of materials which are measured as a function of increasing temperature with constant heating rate, or as a function of time with constant temperature. Thermal stability of a compound is measured.

B. Objective:

- To develop SCC using fly ash and GGBFS of different proportion with superplasticizer and viscosity modifying agent.
- To investigate the compressive strength of SCC with different proportion of admixture.

II. METHODOLOGY

A. Materilas

Ordinary Portland cement of Grade 53 conforming to Indian Standard 12269:1987 is said to be used in this project work with specific gravity of 3.14. Portable water is used for mixing the concrete and curing of the specimen. River sand was used as fine aggregate passing through IS sieve 4.75mm and gravel of size 10-12.5mm was used as coarse aggregate. Mineral admixtures used are Fly Ash of class C and Ground Granulated Blast Furnace Slag. Cement was replaced partially in various levels of 10, 20 and 30% with mineral admixtures. To increase the workability of concrete superplasticizer is used and to improve the flow of concrete Viscosity Modifying Agent from BASF is used.

B. Mix Design

This research work is carried out based on Nan Su from National Yunlin University of Science and Technology, Taiwan.

➢ *Design requirements*

Packing factor (PF) = 1.17
Specific gravity of cement = 3.15
Specific gravity of Coarse Aggregates = 2.73
Specific gravity of Fine Aggregates = 2.64
Bulk Density of Coarse Aggregates (Wgl) = 1510 kg/m^3
Bulk Density of Fine Aggregates (Wsl) = 1403 kg/m^3
Ratio of fine aggregate to total mass aggregate (s/a) = 0.56

- *Step 1: Design strength*

Fck_1 = fck + (1.65 X 5)
= 30 + (1.65 X 5) = 38.25 N/mm^2

- *Step 2: Determine the coarse and fine aggregate content*

Wg = PF X WgL (1 - S/a)
= 1.17 X 1510 (1 - 0.56) = 777.34 kg/m^3
Ws = PF X WsL (S/a)
= 1.17 X 1403 (0.56) = 919.24 kg/m^3

- *Step 3: Calculation of cement content*

C = fck_1 / 0.110
= 38.25 / 0.110 = 347.72 kg/m^3

- *Step 4: Calculation of water content required by cement*

W/C = 0.40 (assume)
Wwc =(W/C) X C
= 0.4 X 347.72 = 139 kg/m^3

- *Step 5: Calculation of fa and GGBFS contents*

Fly Ash
Wf = F% X C
Wf_1 = 0.1 X 347.72 = 34.772 kg/m^3
Wf_2= 0.2 X 347.72 = 69.542 kg/m^3
Wf_3= 0.3 X 347.72 = 104.31 kg/m^3
GGBFS
Wg = G% X C
Wg_1 = 0.1 X 347.72 = 34.772 kg/m^3
Wg_2= 0.2 X 347.72 = 69.542 kg/m^3
Wg_3= 0.3 X 347.72 = 104.31 kg/m^3

- *Step 6: Determine the sp and vma dosage*

SP Dosage
W_{SP} = n% X (C + W) n = 2%
= 0.02 X 486.72 = 9.73 kg/m^3
VMA Dosage
W_{VMA} = n% X (C + W) n = 1%
= 0.01 X 486.72 = 4.86 kg/m^3
SP and VMA is adjusted on site based on experience.

The calculated mix design is shown in table Ⅰ, with mix id A1-0% admixture, A2-10% FA, A3-20% FA, A4-30% FA, A5-10% GGBFS, A6-20% GGBFS and A7-30% GGBFS.

Mix ID	Cement Kg/m^3	Fly ash Kg/m^3	GGBFS Kg/m^3	Coarse Aggr. Kg/m^3	Fine Aggr. Kg/m^3	Water Kg/m^3
A1	347.72	-	-	919.24	777.34	139
A2	312.9	34.772	-	919.24	777.34	139
A3	278.18	69.54	-	919.24	777.34	139
A4	243.41	104.31	-	919.24	777.34	139
A5	312.9	-	34.772	919.24	777.34	139
A6	278.18	-	69.54	919.24	777.34	139
A7	243.41	-	104.31	919.24	777.34	139

Table 1. Mix proportions of grade 30MPa with 2% SP and 1.5% VMA

C. Experimental Procedure

The cubes upon demolding were subjected to curing over a period of 28 days. Curing process took place in the curing tank. Meanwhile samples were collected at an interval of 14th and 28thday and were subjected to oven heating for a period of 24 hours. After the proposed curing period the cubes were tested for its compressive strength. From the results, the optimum value for each mineral admixture was found and that particular 14th and 28thday concrete samples were tested for SEM, XRD and TGA and the corresponding results were compared. Samples for micro-structural test include traditional SCC, optimum values from FA and GGBFS.

III. RESULT AND DISCUSSION

A. Fresh Properties of SCC

The experimental results of workability of SCC are listed in table Ⅱ.

Method	Unit	Property	Typical ranges of values		Result
			Minimum	Maximum	
Slump flow	mm	Filling ability	650	800	720
J-ring	mm	Passing ability	0	10	6
V-funnel	sec	Filling ability	8	12	9
L-box	h2/h1	Passing ability	0.8	1.0	0.87

Table 2. Self Compactability test as per EFNARC Specifications

Workability test done satisfies the norms of EFNARC specifications.

B. Hardened Properties of SCC

The experimental results of compressive strength of SCC are listed in table Ⅲ.

Mix ID	Average compressive strength of 3 samples (N/mm2)
A1	33.2
A2	28.8
A3	37.1
A4	32
A5	24.5
A6	26.9
A7	30.7

Table 3. Result for Compressive Strength of SCC

Samples of mix A1, A3 and A7 are used for micro-structural study.

C. Micro-Sturctural Analysis of SCC

➢ *Scanning Electron Microscope (SEM)*

SEM is the study of particle morphology. The compounds usually present during the hydration process CH, C-S-H and ettringite. CH gel have hexagon shape like crystal C-S-H gel have small flower shaped structure and ettringite have needle like structure. C-S-H and CH gel forms in 2 to 3 days whereas ettringite takes a bit longer time. SEM is also used to find unreacted particles which does not have any structure formation like hexagon or needle shaped.

For SEM analysis samples of 14th day and 28th day of mix ID A1, A3 and A7 were taken to show the compound transition. C-S-H is formed due to the hydration of C_3S and C_2S which is also responsible for the strength of the concrete and ettringite is formed due to the hydration of C_3A and C_4AF. At the end of 28^{th} day C-S-H gel is completely formed which makes up to 50-60% of the volume of solids in a completely hydrated cement paste, and ettringite becomes unstable and gets gradually converted into monosulphate, which is the final hydrated product of cement.

The microstructure obtained from the SEM images indicates the distribution of the hydrated and unhydrated cement paste along with the pores and void spaces. The interpretation is done through the visual observation of the SEM images with standard journals as reference.

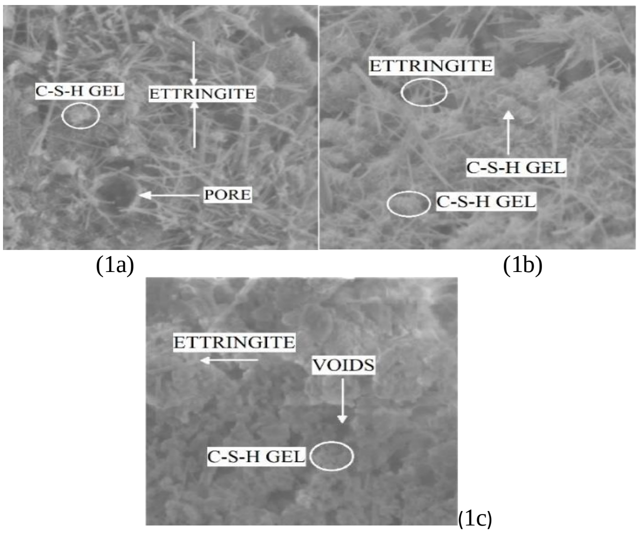

Fig 1:- (1a, 1b,1c are SEM images of A1, A3 and A7 from 14^{th}day

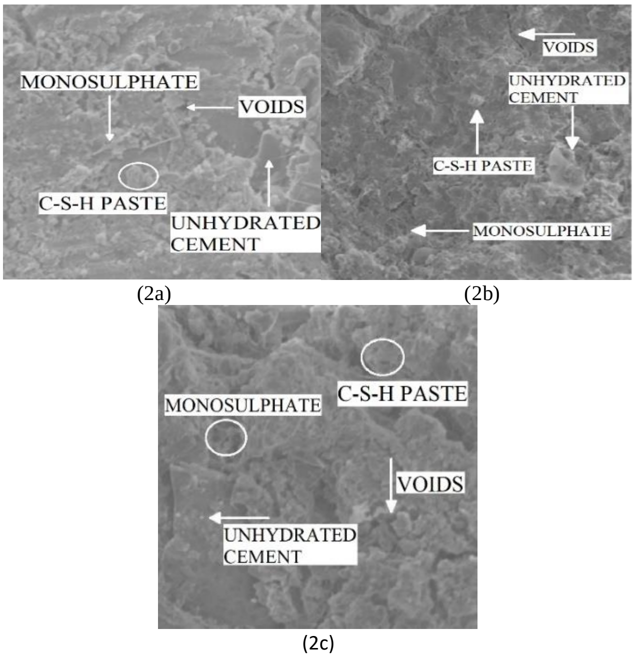

Fig 2:- 2a, 2b,2c are SEM images of A1, A3 and A7 from 28^{th}day

From the SEM images fig:1a, 1b, 1c of 14^{th} day and fig:2a 2b, 2c of 28^{th} day shown above compares the transition of the compound in the concrete during the hydration process. In both 14^{th} day and 28^{th} day SEM images voids, unhydrated cement and pores are shown in it. The 14^{th} day SEM images fig:1a, 1b, 1c of A1, A3 and A7 shows more of C-S-H gel which is small flower like structure and ettringite formation which is needle like structure than the 28^{th} day. A3(fig:1b) has more cluster formation when compared to A1 and A7. At the end of 28^{th} day the concrete undergoes 90% of its hydration. In 28^{th} day SEM images fig 2a, 2b, 2c of A1, A3 and A7 show the complete hydrated particles of concrete such as C-S-H paste, and mono sulphate can be seen. Comparing the 28-day images A3(fig:2b) show dense structure then A1 and A7. From the SEM images it can be concluded that A3 mix ID with 20% fly ash shows better result than A1 with 0% admixture and A7 with 30% GGBFS. C_3S and C_2S are responsible for the strength of concrete which hydrates to form C-S-H gel the formation of C-S-H gel is more in A3 sample, which shows the strength of A3 cube will be more. This is verified with the compressive strength test taken in the lab. The result was compared, and it is shown that A3 mix with 20% fly ash has higher compressive strength then all the other mix proportions.

➢ *X-Ray Diffraction (XRD)*

X-ray diffraction is used as a phase identification of the components formed during hydration of cement. SEM gives us the visual details of the compound, but to verify the compounds assumed by us whether its right or wrong XRD helps in identifying the compound. The compound is identified using it peaks. XRD is depicted in graphical form highlighted with distinguished peaks for each element. The cement constituents present in concrete are C_2S, C_3S, C_3A and C_4AF which undergoes hydration process. The most common hydrated products formed are CH, C-S-H, ettringite and mono sulphate. Apart from these hydrated

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products other products like hematite, mullite, sillimanite, magnetite and albite are also formed. These compounds can be identified using XRD peaks.

Samples used for XRD analysis was A1, A3 and A7 of 28th day. The samples used for XRD analysis fine powder of concrete up to 40 to 50 gm were submitted for the test. The fly ash samples by finely powdered by grinding in a pestle and mortar, and each powder was subjected to XRD analysis using a Philips x' Pert diffractometer system with Cu Ka radiation. Scans were run from 5 to 80' 2 theta, step scanning was used with a scan speed 4 degree/min and sampling interval of 0.005 degree. The peaks are plotted against 2 theta degree in X-axis with the scale of 0 to 80 degree to intensity CPS (counts per second) in the Y-axis. The interpretations of results are done by comparing with past research works.

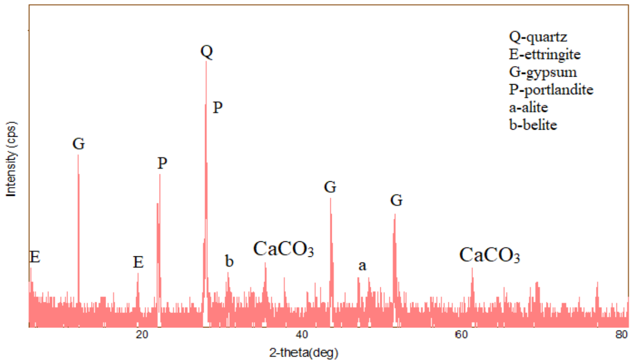

Fig 3:- XRD pattern of A1 from 28th day

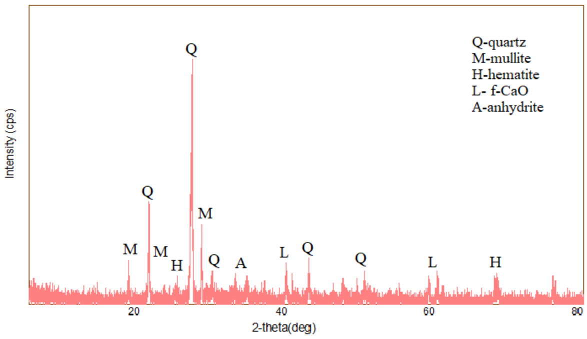

Fig 4:- XRD pattern of A3 from 28th day

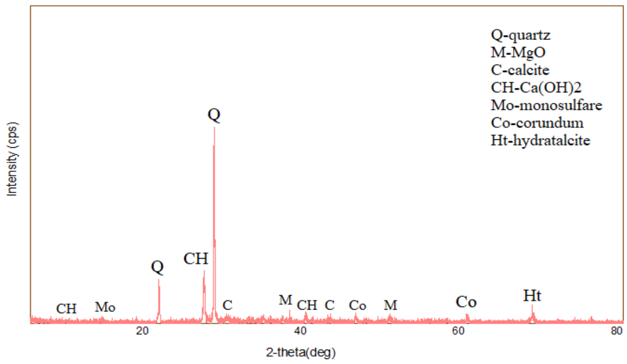

Fig 5:- XRD pattern of A7 from 28th day

Fig.3. Shows the XRD patterns at 28days for A1 mix design. The diffraction spectra show that alite and belite is found in the sample, that part of it is hydrated and the calcium silicate hydrate is produced. Quartz present in concrete has the highest peak followed by gypsum which is produced during the hydration. Ettringite and portlandite are present in hydration stage. Changes in the mineral compounds during hydration were highlighted where from silicate hydrates and aluminate hydrates appeared (portlandite and ettringite). The XRD pattern of A3 mix ID is shown in Fig.4. The main components of A3 is quartz (SiO_2), millite ($Al_6Si_2O_{13}$), L (CaO), anhydrite ($C_4H_6O_3$) and hematite (Fe_2O_3). The peak formed for quartz of A3 is higher than that of A1 is due to the presence of high content silica as Class C fly ash is used, comprising more silica compared to ordinary portland cement. XRD diffractograms for 28-day A7 mix ID is shown in Fig.5. The A7 specimens exhibits peaks of quartz, peaks of magnesium oxide are also observed. Other compounds such as monosulphate, corundum(Al_2O_3), calcite($CaCO_3$), calcium hydroxide and hydrotalcite ($Mg_6A_{l2}CO_3(OH)_{16}.4(H_2O)$) are observed as small peaks.

- *Thermogravimetric Analysis (TGA)*

TGA is a method of thermal analysis in which, changes in physical and chemical properties of materials are measured as a function of increasing temperature. It is used to find the change in weight of the material when exposed to increased temperature. The loss of weight gives an indication of the evaporation or decomposition of the material. Each compound has a standard mass loss, from the value we get from the test the compounds can be verified. From reference journals we can interpret which compound loses how much mass at which temperature and the identified compounds from SEM analysis and XRD can be verified.

In concrete there are two major components C-S-H and CH. When the sample is heated there is a mass loss between 100 to 200^{O}C indicating the compound as C-S-H. At 200^{O}C almost all the components losses their mass. The components which lose their mass between 100 to 200^{O}C are hydrated products. The samples are generally heated in the range of 0-2000^{O}C, for this project the samples were subjected to heat up to 800^{O}C. The result was plotted in graphical form with temperature along X-axis and mass loss in Y-axis.

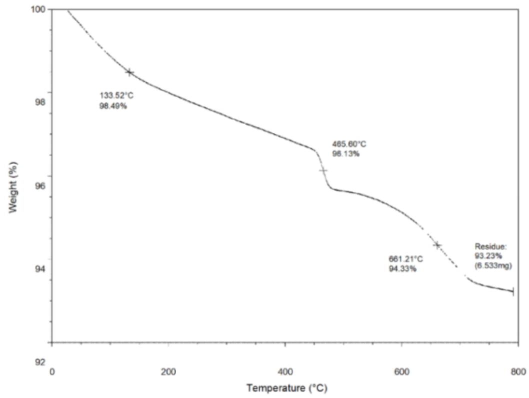

Fig 6:- TGA graph of A1 from 28th day

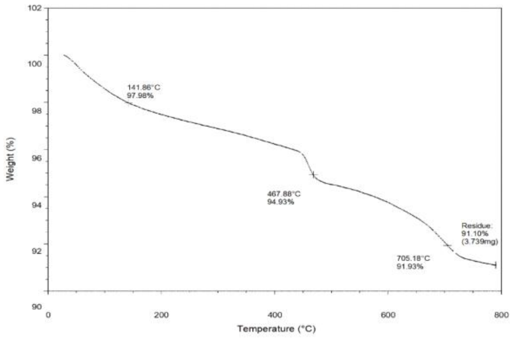

Fig 7:- TGA graph of A3 from 28th day

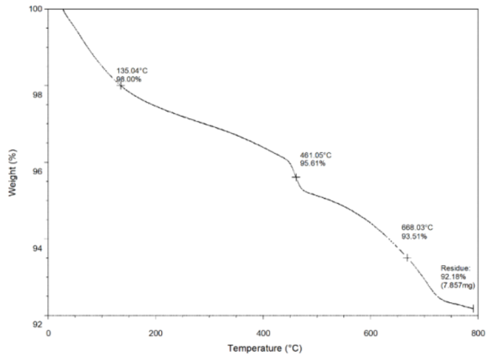

Fig 8:- TGA graph of A7 from 28th day

The fig 6,7&8 shows the thermogram of mix ID A1 with 0% admixture, A3 with 20% fly ash and A7 with 30% GGBFS. The TGA curve shows three significant weight loss steps. The initial one is about 100-150^{O}C has to do with the drying (capillary pore residual water) and / or with the dehydration of hydration products such as C-S-H gel and ettringite. This first weight loss step is usually associated with several minor steps that are likely to take place which includes capillary pore water, interlayer water and adsorbed water. The second weight loss step at about 400–500^{O}C is due to the dehydration of CH. Following chemical reaction usually takes place in this region:

$$Ca(OH)_2 \rightarrow CaO+H_2O \quad (1)$$

The third weight loss step was in the range 650-700^{O}C can be attributed to the de-carbonation of $CaCO_3$. The carbonate is however not present in the original mixture and must therefore arise from a carbonation reaction. Equation (2) shows the carbonation reaction, while equation (3) shows the subsequent de-carbonation equation which takes place when the temperature exceeded 700^{O}C

$$Ca(OH)_2 + CO_2 \rightarrow CaCO_3+H_2O \quad \text{(carbonation)} \quad (2)$$

$$CaCO_3 \rightarrow CaO+ CO_2 \quad \text{(de-carbonation)} \quad (3)$$

The value of mass loss for the A3 sample is higher than A1 and A7 samples in the temperature interval of 100–150^{O}C, which is due to the formation of more hydrated C–S–H gel. C-S-H gel is the hydrated product of C_2S and C_3S which is responsible for the strength of the concrete, where compressive strength test also shows the similar result.

IV. SUMMARY

SCC has reached the status of being an outstanding advancement in the sphere of concrete technology. The use of SCC is rapidly increased, due to its striking properties. This new technology has been of interest to researchers, practicing engineers and several industrial sectors including the cement and admixture manufacturers due to the higher performance achieved in both fresh and hardened state, increased productivity, lesser labor requirements and improved working environment. The necessity for the development of SCC is increasing gradually because of the demand imposed by the construction industry in recent times.

The present investigation is an attempt to develop a mix design method of SCC based on the material characteristics with fly ash and GGBFS and also to access the strength and microstructural characteristics of the developed mixes to evaluate the effectiveness of the designed mix. If the advantages of SCC can be combined with the use of an alternative material for cement, the resulting SCC could have a promising future.

Experimental investigations were carried out to validate the proposed mix design method and to check the accessibility of the designed mixes. Mix proportions were arrived as per the proposed mix design method based on the material characteristics for SCC of grade 30 MPa with various percentage replacement of fly ash and GGBFS. The performance of the designed mix in term of rheological characteristics in the fresh state was studied to check the acceptability of the designed concrete mixes as SCC. All the mixes were tested for some of the key properties such as flowability, filling ability and passing ability and compared with the acceptance criteria as per EFNARC specifications and guidelines.

The strength parameter was studied through compressive strength, determination at the end of 28 day curing period. With the results of compressive strength, the sample with the highest compressive strength of its category (fly ash and GGBFS) is selected as the optimum sample for the microstructural study of the concrete.

Microstructural study of concrete includes scanning electron microscope (SEM), x-ray diffraction (XRD) and thermogravimetric analysis (TGA). SEM is used for studying the particle morphology. By visual observation in microscopic level the compounds present in concrete are observed. The observed compounds are identified with the help of XRD. XRD gives the peak for each and every compound present at the concrete. TGA is used as a confirmation of the identified components. In TGA the samples are heated up to 0-1000^{O}C which are subjected to mass loss. From the mass loss the compounds can be verified. From the microstructural study compressive strength results are compared.

V. CONCLUSION

From this experimental study conducted, it can be concluded that the replacement of cement with mineral

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admixtures in SCC can have a great influence on fresh concrete, hardened concrete and micro-structural properties.

- A mix design method has been proposed for SCC with different proportions of fly ash and GGBFS based on the material characteristics.
- The proposed mix design accounts for the efficiency of fly ash and GGBFS in the self-compacting concrete. All the concretes have satisfied the norms that were set to qualify them as self-compacting concrete. The developed self-compacting concrete has good flowability and passing ability. Thus, the proposed methodology for the design of self-compacting concrete is found to be acceptable.
- Segregation and bleeding were controlled compared to the conventional vibrated concrete with increase in fineness content.
- Compressive test results show that SCC with 20% fly ash has 37.1 N/mm^2 higher strength when compared to SCC with no admixture of 33.2 N/mm^2 and SCC with 30% GGBFS of 30.7 N/mm^2.
- Results on the analysis using scanning electron microscope on samples of self-compacting concrete after 14 and 28 days of curing reveal that mix with fly ash has more C-S-H gel formation which is responsible for the strength of concrete then mix with no admixture and GGBFS.
- X-ray diffraction- it shows the presence of various components responsible for hydration.
- Thermogravimetric analysis shows value of mass loss for fly ash sample is 94.93% which is higher than mix with no admixture of 96.13% and GGBFS of 95.61% due to the formation of more hydrated C-S-H gel. C-S-H gel is responsible for strength showing that fly ash mix is better than other mixes.
- Replacement of cement with fly ash and GGBFS are said to be economical where fly ash and GGBFS cost are lesser than cement and eco-friendly as it reduces the thermal waste and slag waste which are being dumped.

➢ *Significant Contribution*

- Developed a mix design method for SCC with fly ash and GGBFS
- Studied the strength parameter, compressive strength which were conducted on samples after 28 days of curing.
- Suggested an alternative material in place of cement for SCC applications.
- Define the optimum percentage replacement of fly ash and GGBFS for cement of SCC.
- Studied the microstructural characteristics of concrete using scanning electron microscope, x-ray diffraction and thermogravimetric analysis.

➢ *Suggestion For Future Work*

The present research has been focused primarily on the development and evaluation of self-compacting concrete using fly ash and GGBFS. It can be suggested that further investigation can be carried out with different types of admixtures. In a broader perspective, structural applications of SCC need better understanding in terms of creep resistance and can be considered for further investigation. It can also be recommended to study the structural performance of SCC in column and thin wall elements with confined reinforcement.

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