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APPLICATION OF SEM METHOD TO INVESTIGATE THE CAUSE OF EFFECT OF ELEVATED TEMPERATURES ON COMPRESSIVE STRENGTH FOR TERNARY BLENDED CONCRETE USING METAKAOLIN AND MICRO SILICA

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ABSTRACT

The paper aims at using SEM as tool for studying the effect of the elevated temperatures on compressive strength of Ternary Blended Concrete (TBC) applying Scanning Electron Microscopy (SEM) method on binders' hydration process and its microstructure. During investigation, the ternary blended concrete with Ordinary Portland Cement (OPC), Micro Silica (MS) and Metakaolin (MK) was prepared and subjected to elevated temperature ranging from ambient to 800°C. The study has revealed the optimum proportions of constituents of Ternary Blended Concrete (TBC) for designing the structure to resist elevated temperatures. The results of the investigations through the SEM method demonstrated the potential of TBC (with 70% of OPC, 20% of MK and 10% of MS) for use as fire resistant structural material and subsequent analysis using SEM has indicated the possible causes for the observed effect of elevated temperatures on compressive strength of Ternary Blended Concrete. The studies also threw adequate light on the possibility of optimisation of natural resources in the construction industry in the form of savings in the cement usage and reduction in the emission of CO_2 (carbon credits).

Keywords: elevated temperatures, ternary blended concrete, admixtures, OPC, metakaolin, micro silica, compressive strength, scanning electron microscopy.

INTRODUCTION

The construction industry needs the special concretes utilizing the industrial wastes and natural minerals available abundantly to enhance the strength as well as performance for its use in the special conditions when the structures exposed to high temperatures. The microstructure and chemical reactions of the hardened mass formed by the hydration of the cements is essential to understand for the optimal use of binders in the building materials.

The main binding phases in all Portland cement-based systems are Calcium silicate hydrates (C-S-H) (Richardson, 1999) and the nature of C-S-H is vital to the chemistry of cement and concrete. This paper presents data for the C-S-H phases present in Ordinary Portland cements, in blends of Ordinary Portland Cement with Metakaolin and Micro Silica; data are also reported which illustrate the similarity of the C-S-H phases present in blends of Ordinary Portland Cement with metakaolin and Micro Silica exposed to elevated temperatures from ambient to 800°C. The replacement materials participate in the hydraulic reactions, contributing significantly to the composition and microstructure of the hydrated product.

The aim of the present work was to investigate the microstructure of composite cements incorporating 10% to 30% of MK and 10% of MS. Data obtained using Scanning Electron Microscopy (SEM) to study the hydration products of these composite cements exposed to elevated temperatures from ambient to 800°C are compared with similar data from a control sample, based on the same Portland cement paste that was used to make the blended materials.

One of the important and useful properties of concrete is the compressive strength and need to be estimated as a construction material to resist compressive stresses. The effects on residual compressive strength were studied in this investigation and SEM method was applied to analyse the hydration process of binding materials. The use of SEM method reveals more accurate information with regards to the behaviour of binding materials paste during hydration and gives a more realistic knowledge of the mechanisms that generate properties such as strength and durability (Jumate Elena, 2012) which are important for selection of binding materials.

OBJECTIVE

The objective of the present research work is to subject the ternary blended concrete to elevated temperatures and to analyse, applying SEM method, the after effects with respect to the Compressive Strength of the concrete for suggesting an optimal proportion of the Micro Silica and Metakaolin for safe assessing and designing of concrete structures exposed to elevated temperatures.

LITERATURE REVIEW

Partial replacement of OPC with metakaolin (Muthupriya *et al*, 2011) improved strength with increased percentage of admixtures. The packing was improved due to the finer particles of metakaolin and thus bond between metakaolin and fly ash and the substrata was observed which causes more cohesive and less prone to segregation. The compressive strength of metakaolin and fly ash concrete was higher than normal concrete at optimal

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percentage. Also the compressive strength of concrete increases with the age increases the brittleness. The denser micro structure and durability of concrete was observed with the addition of fly ash and metakaolin admixtures when compared to normal concrete.

Metakaolin optimum levels improve transport characteristics and performance of durability (Sherkarchi *et al*, 2010). The initial setting time has accelerated while the final setting time remained unchanged, with the addition of metakaolin. Partial replacement of cement with metakaolin increased the compressive strength and all other transport properties improved while ASR expansion reduced significantly.

The effect of temperature (up to 800ºC) on the mechanical properties and potential for explosive spalling of high strength concrete with and without silica fume was investigated (Bastami *et al*, 2011). The elevated temperatures influenced the effect of water cement ratio on compressive strength and spalling ratio. The increase in w/c ratio that decreased compressive strength at room temperature changed its trend to increase compressive strength at elevated temperatures. It was also reported that the addition of silica fume did not affect the relative strength of heated specimens but controlled the spalling ratio. Silica fume decreased the extent of spalling with increased w/c ratio. Reduction in compressive strength was high when the specimens heated up to 800ºC. The optimal level of process parameters were found corresponding to w/b ratio, sand ratio, silica fume ratio and the amount of silica fume which followed Taguchi parameter design technique.

The effect of elevated temperatures (100ºC - 800ºC) on mechanical properties and microstructure of silica flour concrete using OPC and silica flour (SF) in varying percentages was investigated (Morsy *et al*, 2010). After 28 days of curing, the specimens were cooled naturally to room temperature, and tested for compressive strength and indirect tensile strength. It was reported that, the exclusion of water at 100ºC has resulted in a reduction of residual compressive strength. Silica flour concrete unlike control concrete between 100 and 400ºC resulted in increase in compressive strength with the hydrothermal interaction of the silica flour particles liberating free lime. Decomposition of the hydration products at 800ºC caused the formation of micro cracks and decrease in compressive strength and indirect tensile strength was observed. Dehydration of concrete causes decrease in its strength, elastic modulus, coefficient of thermal expansion and thermal conductivity.

The experimental investigation (Phan L.T. *et al*, 2001) on the effect of elevated temperatures(100 - 450ºC) on spalling and residual properties of HPC using the Type I Portland cement, crushed limestone aggregate, natural sand, silica fume in the form of a slurry with a concentration of 54% (by mass) and a high range water reducing admixture (HRWRA) based on a sulponated naphthalene indicated the increased potential of silica fume for explosive spalling of concrete elevated to temperature of 300ºC - 400ºC.The explosive spalling mechanism was explained as the spike in internal pore pressure causing high restriction heat induced mass loss process coupled with sudden and drastic disintegration of concrete in to small fragments. HPC with higher original compressive strength has exhibited lesser loss of strength and dynamic modulus of elasticity than HPC with lower original compressive strength which was attributed to the presence of silica fume up to the temperature of 200ºC. Rate of decrease in modulus of elasticity was more in temperature range of 23ºC - 300ºC than in temperature range of 300ºC - 450ºC. HPC mixtures which experienced explosive spalling had a more restrictive process of capillary pore and capillary bound water loss than those which did not experience spalling. Explosive spalling of specimens containing silica fume was said to be due to the internal pressure developed and increased build up of thermally induced strain energy that might cause thermal stress in the failure.

Heavy weight high strength concrete using Portland cement, magnetite as both coarse aggregate and in some mixes, as fine aggregate, Silica fume slurry as mineral admixture and chemical admixture SP6 liquid super plasticizers (SPB) with w/c ratio of 0.5 exposed to transient temperatures (100ºC - 700ºC) for different exposure durations of 0, 1hr and 2 hrs. Both NSC and HSC showed decrease in compressive strengths and dynamic modulus at 100ºC and increase at 200ºC exposure with further sharp drop to 500ºC and 700ºC compared to room temperature (Mahdy *et al*, 2002). The addition of silica fume and magnetite as fine aggregate enhanced residual compressive strength when compared to HSC using natural sand. The variation in strengths was attributed to the shrinkage of cement paste with sand, as absorbed and hydration of water driven out more than that with magnetite paste and also bond loss resulted in the aggregate expansion and exposure durations.

The structural quality of high strength concrete with silica fume using OPC, silica fume, super plasticiser and sand with w/c ratio of 0.32 and the mechanical properties were experimentally studied (Ivan Janotka *et al*, 2003) when exposed to temperatures of 40ºC, 60ºC, 100ºC and 200ºC. Reference concrete proportion was studied curing in a wet air. The strength, elasticity modulus and deformation of concrete were irreversibly influenced by temperature elevation to 100ºC and 200ºC. The effect of pore structure coarsening was attributed to the significant strength decrease of concrete and cement paste. Rapid cooling after temperature elevations evokes equal and irreversible deterioration in the quality of the structures of concrete and cement paste. The self curing of concrete and cement paste at 20ºC does not attribute to the structural integrity improvement. Concrete and cement paste persistently deteriorated, and the impossibility to acquire their original physical state before temperature attack was observed. The quick release of bound water from hydrated cement paste caused expansion of concrete and the contribution of sudden elevation of temperature had deteriorated structural integrity of the specimens.

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EXPERIMENT DETAILS

Materials and Methods

Materials for concrete making

Ordinary Portland cement of 53 grade confirming to IS: 12269-1987 having specific gravity 3.24 and fineness of 8.53 was used. Crushed angular granite metal of 20 mm size coarse aggregate of specific gravity of 2.64 and fineness modulus 7.35 and River sand confirming to zone-II of IS 383-1970 having the specific gravity of 2.62 and fineness modulus 2.48 were used . Also Metakaolin having specific gravity of 2.40 to 2.60 and Micro Silica having the specific gravity 2.2 and SiO_2 more than 92% were used. Super plasticizer CONPLAST 430 was used as water reducing admixture for increasing workability.

Mix proportion

Mix proportion for w/b ratio:
For w/b ratio of 0.35, the designed concrete mix is 1.00:1.55:2.35.

The various concrete specimens of experiment are designated as follows:

Sample	Proportion of materials
M_0	100% of OPC + 0% of Metakaolin and 0% of Micro Silica
M_1	800% of OPC + 10% of Metakaolin and 10% of Micro Silica
M_2	70% of OPC + 20% of Metakaolin and 10% of Micro Silica
M_3	60% of OPC + 30% of Metakaolin and 10% of Micro Silica

METHODOLOGY

Casting of specimens

Concrete cubes of size 100mm were cast with Ternary Blended Concrete using 10 to 30% of Metakaolin and 10% of Micro Silica as replacement of Ordinary Portland Cement and control concrete with w/b of 0.35 designated as M_0, M_1, M_2 and M_3 (as given above). The specimens were cured in water for 28 days.

Testing

The concrete specimens were exposed to temperature of 100°C to 800°C for duration of 1hour in furnace and allowed to cool to room temperature in the furnace. The concrete specimens were tested for compressive strength using 2000 kN compression testing machine.

RESULTS AND DISCUSSIONS

The present investigation was carried out to study after effects of elevated temperatures on mechanical properties of control and ternary blended concrete. During the above investigation, material like Ordinary Portland Cement, Metakaolin, Micro Silica and super plasticizer were used as ingredients of control and ternary blended concrete as the case may be.

- Tools like SEM studies were employed for qualitative analysis of structural changes of concrete.
- The result of the above investigations is discussed in the following sections.

Effect of elevated temperatures on compressive strength of control and ternary blended concrete

The result of experimental investigation on after effects of elevated temperatures on compressive strength on samples of concrete viz., M0, M1, M2 and M3 is presented in Figure-1. From the figure it can be seen that the compressive strength of all the samples of concrete exposed to temperature elevated up to 100°C, independent of mixes and type of admixtures have invariably decreased over the ambient temperature.

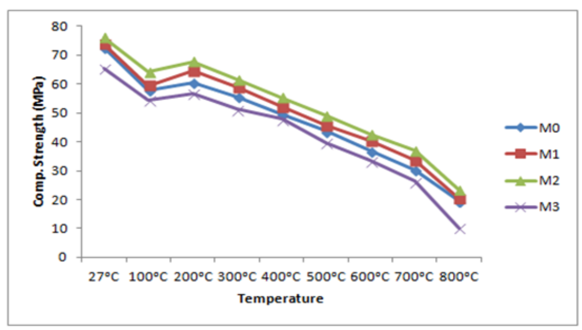

Figure-1. Compressive strength strength of control and TB concrete.

The percentage loss in strength is shown in Table-1. This phenomenon of loss of strength may be attributed to evaporation of capillary free water and shrinkage of concrete (Sri Ravindrarajah *et al*., 2002).

Table-1. Percentage of loss of compressive strength exposed to different temp.

Sample	**% of Loss residual compressive strength** (MPa)			
	27°C - 100°C	**27°C - 200°C**	**27°C - 400°C**	**27°C - 800°C**
M_0	20	17	33	73
M_1	19	14	32	72
M_2	16	9	25	70
M_3	17	13	35	84

SEM images of samples of Control Concrete and Ternary Blended Concrete subjected to elevated temperature of 100°C are depicted in Figure-2 through 9.

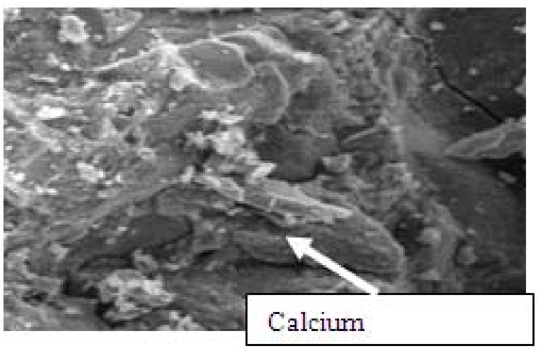

Figure-2. SEM image of M_0 concrete at ambient temp.

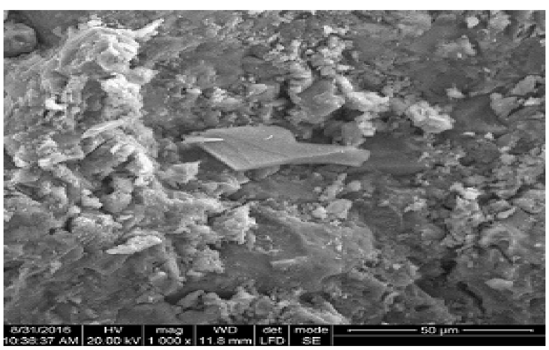

Figure-3. SEM image of M_0 concrete at 100°C.

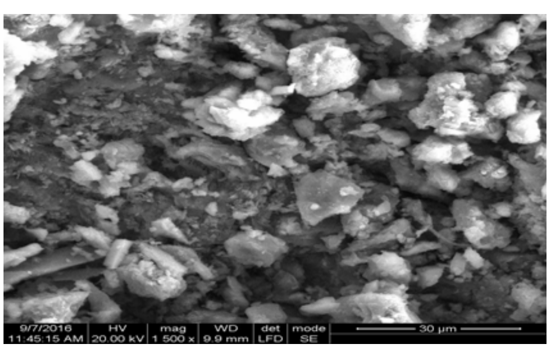

Figure-4. SEM image of M_1 concrete at ambient temp.

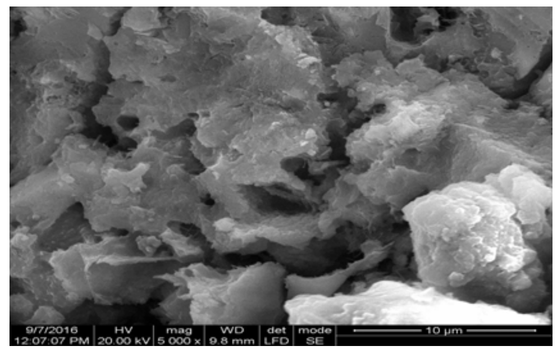

Figure-5. SEM image of M_1 concrete at 100°C.

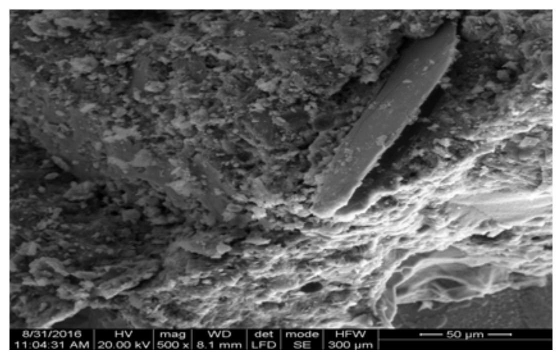

Figure-6. SEM image of M_2 concrete at ambient temp.

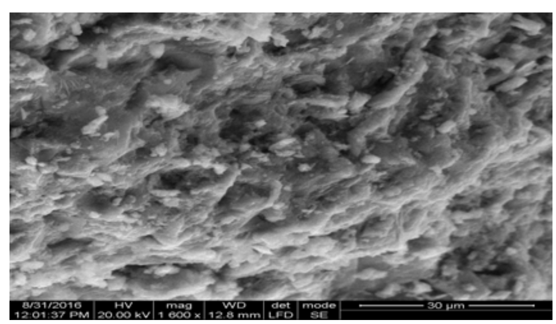

Figure-7. SEM image of M_2 concrete at 100°C.

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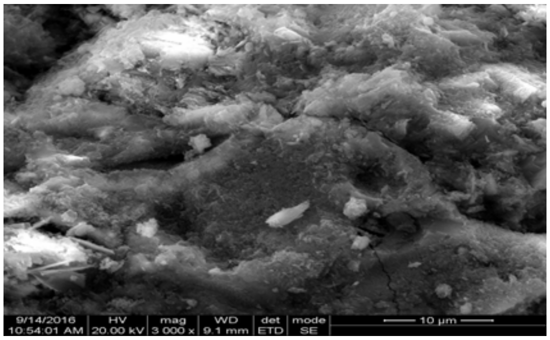

Figure-8. SEM image of M_3 concrete at ambient temp.

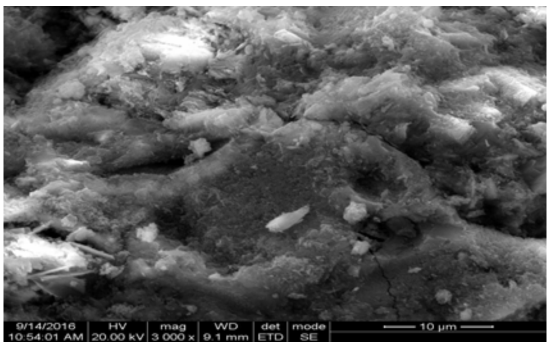

Figure-9. SEM image of M_3 concrete at 100ºC.

From the above figures C-S-H and CH though appear to be present both at ambient and at 100ºC, the variation in cloudiness of the images indicate changes in the chemical composition.

Further elevation of temperature beyond 100ºC and up to 200ºC resulted in a small gain of the compressive strength as shown in Table-1 for all the samples. SEM images of the samples of Control and Ternary Blended Concrete subjected to elevated temperature beyond 100ºC and up to 200ºC are depicted in Figure-10 through 13.

Figure-10. SEM image of M_0 concrete at 200ºC.

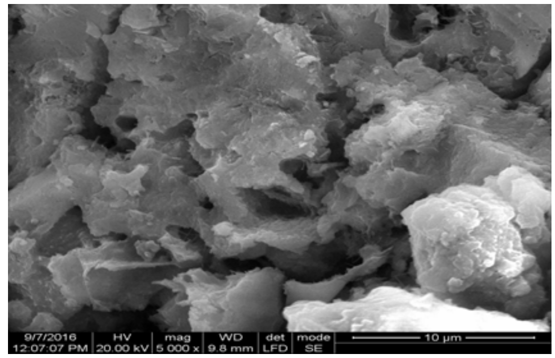

Figure-11. SEM image of M_1 concrete at 200ºC.

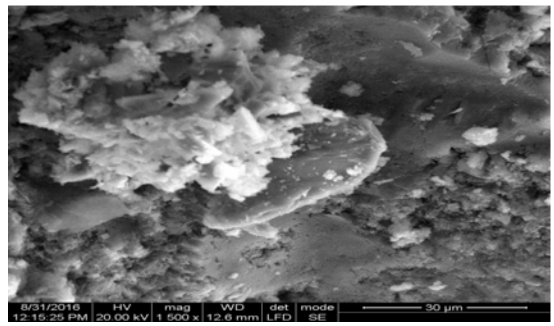

Figure-12. SEM image of M_2 concrete at 200ºC.

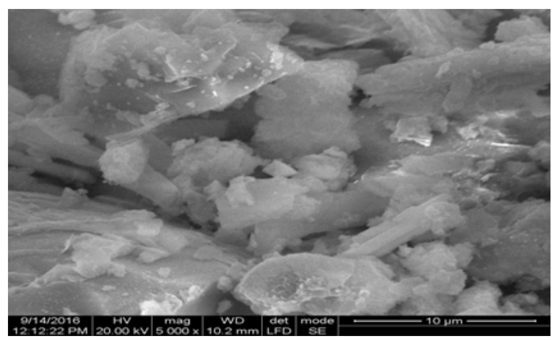

Figure-13. SEM image of M_3 concrete at 200ºC.

From the above images a dense mass of C-S-H was identified at a temperature of 200ºC in all samples. The temperature beyond 100ºC might have been favourable for such additional hydration that reaction of binders with water is rigorous (Morsy *et al*. 2007). This increase could be due to the hydration of unhydrated particles of Micro Silica and/or Metakaolin activated because of rise in temperature (Nimityongskul *et al*). However, the percentage of variation either decrease/increase was not constant for all samples but varied from sample to sample.

Further increase in temperature beyond 200ºC and up to 400ºC continued loss of strength as seen in Table-1. SEM images of the samples of Control and Ternary Blended Concrete subjected to elevated temperature beyond 200ºC and up to 400ºC are illustrated in Figure-14 through 17.

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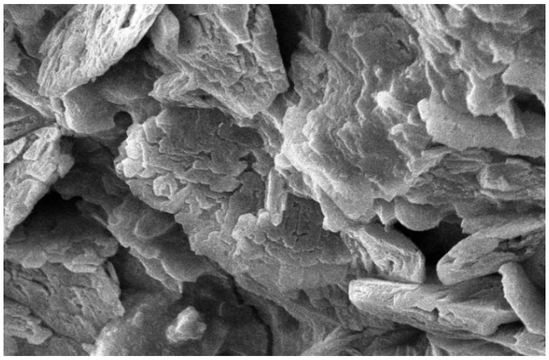

Figure-14. SEM image of M_0 concrete at 400°C.

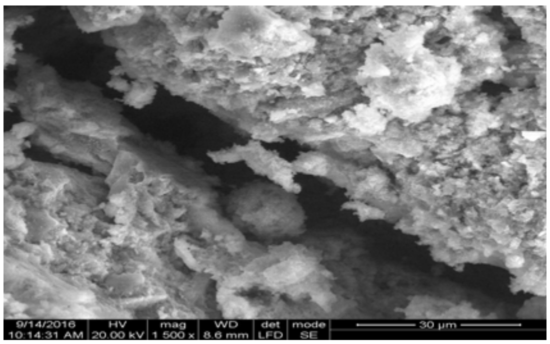

Figure-15. SEM image of M_1 concrete at 400°C.

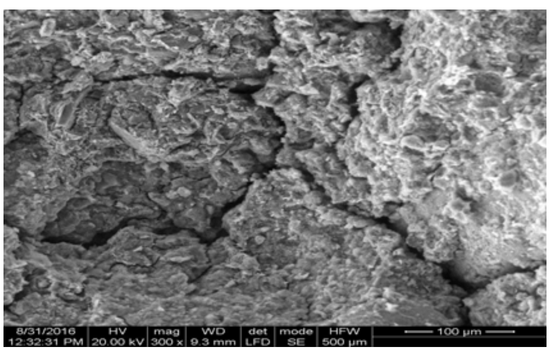

Figure-16. SEM image of M_2 concrete at 400°C.

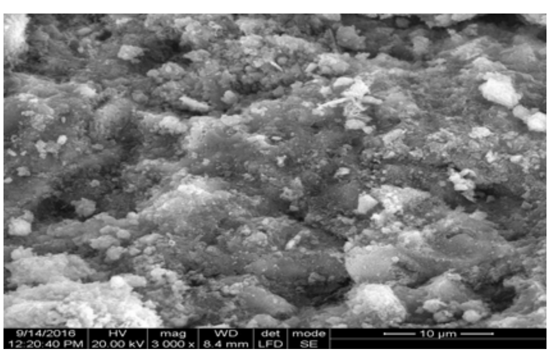

Figure-17. SEM image of M_3 concrete at 400°C.

This loss of strength may be due to dehydration of calcium hydroxide (Morsy *et al* 2006) in the concrete as evident from the hollow spaces in the form of black holes and platy form seen in SEM images of respective samples. Further loss of strength between 400°C and 800°C temperatures may be attributed to the disintegration of C-S-H and C-A-S and propagation of micro cracks (Morsy *et al* 2006). SEM images of the samples of Control and Ternary Blended Concrete subjected to elevated temperature beyond 400°C and up to 800°C are depicted in Figure-18 through 21.

This stage of concrete may be characterised by the collapse of its structural integrity and loss in compressive strength.

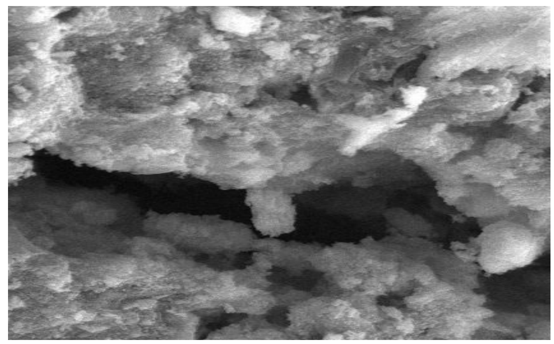

Figure-18. SEM image of M_0 concrete at 800°C.

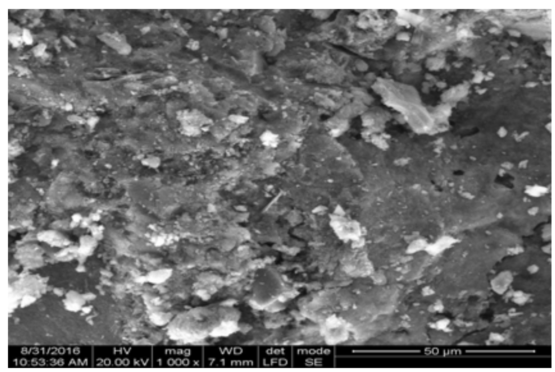

Figure-19. SEM image of M_1 concrete at 800°C.

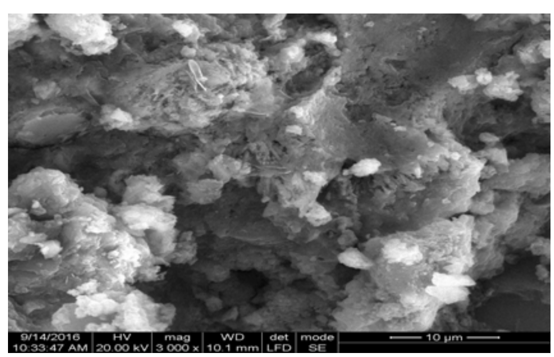

Figure-20. SEM image of M_2 concrete at 800°C.

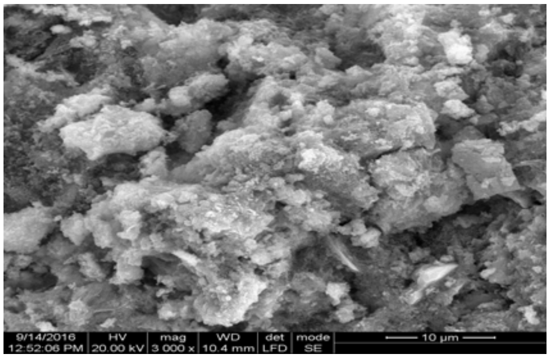

Figure-21. SEM image of M_3 concrete at 800°C.

Pozzolanic reactions change the microstructure of concrete and chemistry of hydration products by consuming the released Calcium Hydroxide (CH) and production of additional Calcium Silicate Hydrate (C-S-H), resulting in an increased strength (Shivram Bagade *et al*, 2007). The qualitative analysis figures of SEM indicate the changes in the microstructures of the samples.

On perusal of figures from Figure-1 through 4, it may be concluded that TBC (M_2) resulted in higher residual strengths at all temperatures compared to other samples viz., Mo, $M_1$and M_3. The above phenomenon may be attributed to the balanced hydration process for the formation of C-S-H and CAH (Morsy *et al,* 2008). Therefore, percentage of MK and MS in M_2 may be inferred as optimum for obtaining the maximum residual strength in TBC. Similarly lesser strengths were obtained in M_3 compared M_1 and M_2 probably due to un-reacted excess amount of MK. The increased percentage of MK in M_3 caused bursting of dense concrete at elevated temperatures.

CONCLUSIONS

The following conclusions are drawn from the present research work:

a) The residual compressive strength of samples using 0% of MK and 0% of MS (M_0) 10% of MK and 10% of MS (M_1), 20% of MK and 10% of MS (M_2) and 30% of MK and 10% of MS (M_3) decreased with increase in temperature from ambient to 100°C.The percentage in decrease however varied from 16% for M_2 to 20 % for M_0. These values for $M_1$and M_3 are 19% and 17% respectively.
b) The residual compressive strength of samples for M_0, M_1, M_2 and M_3 subjected to elevated temperatures of 100°C to 200°C, increased at varying percentages of 5, 7, 8 and 4 respectively which amount to 5%, 7%, 8% and 4% respectively. The above phenomenon of increasing in residual compressive strength could be attributed from SEM results to heat of hydration.
c) The residual compressive strength of samples for M0, M1, M2 and M3 when subjected to elevated temperatures between 200°C and 400°C decreased in varying percentages of 20%, 21%, 18% and 25% respectively. This phenomenon may be attributed to SEM results and to the fact that the loss in residual compressive strength may be due to the decomposition of hydration products in all cases.
d) The residual compressive strength of samples for M_0, M_1, M_2 and M_3 when subjected to elevated temperatures between 400°C and 800°C decreased in varying percentages of 56%, 50%, 4% and 78% respectively. This phenomenon may be attributed to SEM results and the higher losses in compressive strength may be due to disintegration of hydrated gel in all cases.
e) The Ternary Blended Concrete (M_2) with 70% of OPC, 20% of metakaolin and 10% of micro silica has performed better and observed an increased compressive strength when exposed to ambient temperature and 800°C over the control concrete.
f) The control concrete exposed to elevated temperatures has lost its compressive strength from 72.5MPa at ambient temperature to 22.45 MPa at 800°C. Thus the total percentage loss in compressive strength of control concrete with increase in temperature from ambient to 800°C was found to be 73%. Similarly, the total percentage loss in compressive strength for M_1 concrete was 72%, the total percentage loss in compressive strength for M_2 concrete was 70% and the total percentage loss in compressive strength for M_3 concrete was 84% when the temperature was raised from ambient to 800°C.
g) The SEM analysis on specimens of the test cubes reveals the formation of C-S-H gel and consumption of CH in the secondary hydration reacting with Al present in MK and forming CAH in all specimens of all mixtures of all types with an evidence of major minerals Ca, Si and Al along with minor minerals and the particle distribution which are responsible for the gain of strength for ternary blended concrete. It was also confirmed that from SEM that the loss of compressive strength was due to evaporation of free capillary water and shrinkage in the beginning and also can be attributed to the decomposition of hydrated products and disintegration of hydrated gel.

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