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Influence of mineral by-products on compressive strength and microstructure of concrete at high temperature

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Abstract. In the present work, Granulated Blast Furnace Slag (GBFS) and Fly ash (FA) were used as partial replacement of Natural Sand (NS) and Ordinary Portland Cement (OPC) by weight. One control mix, one with GBFS, three with FA and three with GBFS-FA combined mixes were prepared. Replacements were 50% GBFS with NS and 20%, 30% and 40% FA with OPC. Preliminary investigation on development of compressive strength was carried out at 7, 28 and 90 days to ensure sustainability of waste materials in concrete matrix at room temperature. After 90days, thermo-mechanical study was performed on the specimen for a temperature regime of 200°-1000°C followed by furnace cooling. Weight loss, visual inspection along with colour change, residual compressive strength and microstructure analysis were performed to investigate the effect of replacement of GBFS and FA. Although adding waste mineral by-products enhanced the weight loss, their pozzolanicity and formation history at high temperature played a significant role in retaining higher residual compressive strength even up to 800°C. On detail microstructural study, it has been found that addition of FA and GBFS in concrete mix improved the density of concrete by development of extra calcium silicate gel before fire and restricts the development of micro-cracks at high temperature as well. In general, the authors are in favour of combined replacement mix in view of high volume mineral by-products utilization as fire protection.

Keywords: fly ash; GBFS; elevated temperatures; weight loss; compressive strength; microstructure

1. Introduction

Sustainable concrete (SC) is defined as the concrete which consists of at least one of the constituent as waste and its production should not have negative impact on environment (Suhendro 2014). For the development of SC, various types of industrial wastes had been practiced from long time as Supplementary Cementitious Materials (SCM) such as fly ash (FA), silica fume (SF), ground blast furnace slag (Djamila *et al.* 2018, Cree *et al.* 2017) whereas palm oil fuel ash (POFA) has been included as new pozzolanic material in SCM group (Mohammadhosseini *et al*. 2018). Other than SCM, utilization of granular or coarser solid waste needs a greater attention as aggregate constitutes of about 70% of the volume of concrete. Although several solid wastes have been utilized up to suitable percentages such as granulated blast furnace slag (GBFS) (60%), steel slag (30%), copper slag (40%), class F fly ash (50%) and others as fine aggregates (Patra and Mukherjee 2018, Dash *et al.* 2016, Siddique 2003) by replacement method without compromising mechanical strength, greater amounts are either sent into land-fills outside the plants or accumulated in the plant premise. These materials are utilized in concrete as part replacement for the development of sustainable and durable concrete even for extreme environmental condition such as fire and corrosive exposure (Mohammadhosseini *et al.* 2018, Mohammadhosseini and Tahir 2018). Present work is based on GBFS and FA, used as single replacement and combined replacement for finding the scope for fire protection material.

GBFS is a non-metallic light weight material consists of silicates and alumina-silicates of calcium (Cheng *et al.* 2005), consists of similar chemical oxides constituent as in OPC, but may not be in the same proportion (Oner and Akyuz 2007) whereas NS consists of high content of silica (about 96.4%) and very low content of calcium oxide and alumina (Muttashar *et al.* 2018). Hence, it may be treated as calcareous fine aggregate. According to EN 1992:1-2 (2004) and CEB (1991), calcareous aggregate based concrete exhibits higher resistance against fire than siliceous based concrete. On the other hand, most of the Thermal power plants in India produce Class F type fly ash which consists of high percentage of alumina (Al_2O_3) and low percentage of calcium oxide (CaO) as compared to OPC (Pathak and Siddique 2012, Siddique 2003). Alumina is well known constituent of refractory products meant for enhancing the fire resistance. Also, slag and fly ash, consist of high percentage of Al_2O_3, will also effective in suppressing expansion at high temperature (Ramlochan *et al.* 2003) due to high percentage of ferric oxide which might have adverse effect on concrete in expansion and colour change. Therefore, combination of GBFS and FA might have advantage in enhancing the fire performance of

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concrete. Also, concrete made with light weight aggregates improves mechanical properties at high temperature in comparison to normal aggregates due to formation at high temperature and having low thermal conductivity (Go *et al.* 2012, Ma *et al.* 2015). Netinger *et al.* (2013) utilized fly ash as part replacement of OPC and steel slag as fine and coarse aggregate and found relatively higher residual strength (3-5%) than control mix based on OPC and dolomite as aggregate at temperature 200-600℃. Sancak *et al.* (2008) reported 10% and 20% higher residual strength for fly ash and pumice based Light weight concrete (LWC) than corresponding normal weight concrete (NWC) and pumice based LWC. Savya *et al.* (2005) reported about 4-8% higher residual strength for limestone aggregate based fly ash mix than siliceous aggregate based fly ash mix. Tanyildizi and Coskun (2008) developed LWC based on silica fume (10-30%) and pumice as coarse aggregates. LWC retained 28-38% residual strength at 800℃ which was 13-16% higher than NWC. Netinger *et al.* (2011) defined all aggregates developed at high temperature as fire resistant aggregate. But, the major hindrance in using GBFS is to get required workability as more water is absorbed by GBFS leaving behind lesser quantity for concrete matrix (Binci *et al*. 2008, Patra and Mukherjee 2017) which might be partially compensated by using fly ash known as water reducer and mobilizer for the fresh concrete. However, combined usage of high volume fly ash with GBFS affects the fresh property of concrete and need higher dosage of super-plasticizer (Sahani *et al.* 2018). Furthermore, GBFS based mortar or concrete improves strength (Patra and Mukherjee 2017, 2018) up to 60% after that strength starts to decrease due to increase in porous structure in concrete (Bilir 2012) and high content of glassy fine aggregates (Nataraja *et al.* 2013). However, pozzolanicity characteristics of GBFS and class F fly ash, even as fine aggregate, had been declared by many researchers (Binci*et al*. 2008, Patra and Mukherjee 2018, Siddique 2003).

Degradation in properties of fire exposed concrete is influenced by various factors such as types of binders, aggregates, water cement ratio (w/c), size and shape of the specimen, temperature and duration of fire, heating and cooling condition (Zhang *et al.* 2015, Neville 2011). In the first appearance, damage in fire exposed concrete is assessed by the change in colour, cracking and spalling. These parameters largely depend on the mineralogical composition of aggregates. Colour change is the easiest way for observer to understand the temperature effect of a specific range. Lee *et al.* (2010) found no change in the colour of the specimen up to 200℃, remains grey colour. Normal concrete containing silicate aggregates will turn red as temperature rises from 300℃ to 600℃ (Short *et al*. 2001). Carbonate aggregate decomposes at 700℃ and above, resulting concrete surface into lime whitish colour. Moreover, whitish grey colour appears between 600-900℃ and further increase in temperature gives a buff colour (Hager 2013).

Weight loss of the concrete under fire exposure is the most influencing parameter for development of thermal cracks and deterioration in strength. Normal concrete mainly consists of free water, capillary water, physically absorbed water, interlayer and chemically bound water in Calcium Silicate Gel (C-S-H) and Calcium hydroxide (CH) which is (Arioz 2007). However, types of aggregate and their replacement percentages do not influence weight loss significantly at high temperature 800℃ (Yüksel *et al*. 2011). For the low temperature zone, Savya *et al.* (2005) reported that type of binder and aggregate does not affect the change in compressive strength significantly between 100-300℃. But, fall and rise in strength within this temperature range may take place due to material softening or hardening due to loss of moisture (Lankard 1971, Cheng *et al*. 2004) for calcareous aggregate and siliceous aggregate based concrete (Savya *et al*. 2005). It was noticed that the water loss for the specimens heated at 110℃ was three times lower than that measured on the specimens heated at 210 and 310℃ (Noumowé 2009). It means that moisture loss has significant bearing on loss in strength in lower temperature zone. However, cement paste behave in different manner as it loss free water and physically absorbed water completely, and start to lose their chemically bonded water at about 100℃ (Khoury *et al.* 2002).

Investigators have different opinion regarding the changes in the properties of concretes, particularly in the range of 100-300℃. But, there is a uniformity of opinion regarding decrease in mechanical strength above 300℃ regarding reduction in strength. But, an interesting fact was concluded by Cree *et al.* (2013) that concrete heated below 300℃ does not loose significant strength and loss can be recovered through re-hydration; however, above 300℃, the concrete loses considerable strength and is considered unrecoverable for structural purposes. At 600℃, the strength of mixtures is reduced to about the half; at 750℃, the reduction is from 75% to 93%. Calcium hydroxide, which is one of the most important compounds in cement paste, dissociates at around 530℃ resulting in the shrinkage of concrete (Georgali and Tsakiridis 2005). Therefore, reduction in the compressive strength of concrete was significantly larger for specimens exposed to 600℃, and the decomposition of CH and C-S-H gel, especially at 800℃, resulted in the total deterioration of concrete (Demirel and Kelestemur 2010). It was observed that normal and high performance concrete loses 75-85% of initial compressive strength when they are exposed to 800℃ (Chan *et al.* 2000).

Very few works have been carried out, utilizing GBFS as fine aggregate in concrete for fire exposure. Shoaib *et al.* (2000) found the water quenched slag, also known as GBFS, exhibited an increase in compressive strength of mortar at 300℃ followed by gradual decrease up to 500℃, and a sharp decrease at 600℃. They further concluded that granular blast furnace slag consumes calcium hydroxide (CH) released and led to the formation of C-S-H at elevated temperature. Yüksel *et al.* (2011) and Sahani *et al*. (2015) reported that utilization of GBFS up to 50% in concrete, exposed to high temperature (800℃), performed similar or better properties than normal concretes for heat curing of 1h and 3h, respectively. Rashad *et al.* (2016) prepared alkali activated slag mortar in the ratio of 1:2, replacing NS with by 25-100% by weight. They reported that mortar strength increased with increasing GBFS sand content before and after exposure to 200, 400, 600 and 800℃ for 2 h. On the

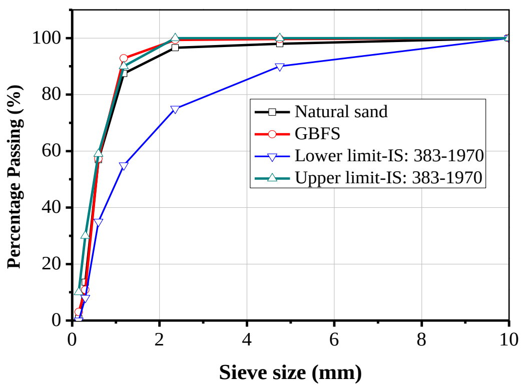


Fig. 1 Particle size analysis of granulated blast furnace slag and natural sand

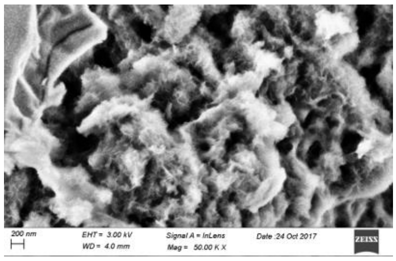
other hand, utilizing pozzolanic materials such as fly ash and other pozzolanic materials such as GGBFS, SF etc. in the concrete appreciably enhances its fire resistive properties due to consumption of calcium hydroxide (CH) in considerable amount during pozzolanic reactions as CH is assumed as a main source of thermal instability (Dias *et al.* 1990, Tanyildizi and Coskun 2008, Hosam *et al.* 2011).

Past research works indicated boundless investigations about natural pozzolana as part replacement of Portland cement. However, utilization of manufactured sand as NS along with natural pozzolana as Portland cement for fire exposed environment is lagging and needs experimental investigation. This will not only be a novel scope for enhancing the volume of utilization of industrial by-products but also will increase the weightage of alternative material for special industrial applications such as concrete structure near furnaces, ovens in steel plants. Therefore, the objective of present study is to investigate the influence of addition of GBFS and fly ash on the residual properties of concrete and its microstructure at elevated temperature. In this study, comparative study was also made to check sustainability of mineral based mixes at elevated temperature with other research works.

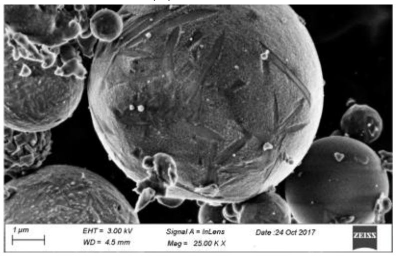
2. Experimental work

2.1 Materials

The Ordinary Portland cement (53 Grade) cement supplied by India Cement, conforming to IS 12269:1987, was used in this work where as Class F Fly ash, conforming to IS: 3812-2003, was procured from local thermal power plant in Durgapur. The physical properties of cement and fly ash are shown in Table 1. Natural sand (NS) and GBFS having maximum size of 4.75 mm were used as fine aggregate whereas coarse aggregates (Granite) of maximum size 20 mm and 12.5 mm in the proportion of 60:40 were used in the concrete mix. Both the fine aggregate and coarse aggregates conformed to the grading requirement of IS383:1970 as shown in the Table 2. Particle size analysis of both NS and GBFS is shown in Fig. 1. Modified Lignosulfonate based Sika Plastiment-100, water reducing super plasticizer, having density of 1.16 kg/l, was



(a) GBFS



(b) Fly ash

Fig. 2 FESEM images of GBFS (a) and fly ash (b)

Table 1 Physical properties of cement and fly ash

Material type	Colour	Specific surface (m^2/Kg)	Specific gravity	Standard consistency (%)	Initial setting time (min.)	Final setting time (min.)	Soundness (mm)	Compressive strength (MPa) 3 days	7 days	28 days
OPC	Grey	310	3.15	32	115	285	3	30.6	42.3	54.2
Fly ash	Dark grey	320	2.1	--	--	--	--	4.2 MPa (Lime reactivity at 28days)		

Table 2 Physical properties of natural sand, GBFS and coarse aggregate

Material	Fine aggregate NS	GBFS	Coarse aggregate (CA)	Granite
Maximum size(mm)	4.75	4.75	20	12.5
Fineness modulus	3.46	3.34	4.62	4.19
Specific gravity	2.62	2.32	2.87	2.87
Bulk density (Kg/m^3)	1560	1310	1800	1800
Water absorption (%)	0.28	4.1	0.2	0.38

used in suitable dosages to achieve required workability. Chemical composition of cement, fly ash and GBFS were determined by X-ray florescence by PANalytical AxiosmAX instrument as presented in Table 3, which indicates that main oxide components were CaO (63.16%, 0.95%,31.7%), SiO_2 (20.39%, 57.75%, 31.4%) and Al_2O_3 (5.6%, 30.9%, 23.7%) respectively.

Morphology of GBFS and FA, extracted from Field Emission Scanning Electron Microscopy (FESEM) indicated smooth spherical shaped fly ash particle and glassy, rough and angular shaped GBFS particle (Patra and Mukherjee 2018, Sahani *et al.* 2018). There was enough cotton type shaped silicates in the GBFS, as shown in Fig. 2 which may enhance the strength.

There were enough cotton type shaped silicates in the GBFS, as shown in Fig. 2 which may enhance the strength. But, it’s usage in higher percentage may affect the strength of concrete because of glassiness nature as cited in the literature.

Table 3 Chemical composition of cement, fly ash and GBFS

Chemical constituents	OPC	Fly ash	GBFS
CaO	63.16	1.95	31.7
SiO_2	20.7	57.75	31.4
Al_2O_3	5.6	30.9	23.7
FeO or Fe_2O_3	3.43	4.93	0.6
MgO	2.91	-	9.8
MnO	-	0.08	0.24
S or SO_3	0.7	0.19	0.6
Alkalis			
Na_20	-	-	-
K_2O	-	0.68	0.65
Loss of ignition (LOI)	2.16	3.5	-

Table 4 Mix proportion of concrete for 1 cubic meter

Ingredients	Types of Concrete Mixes							
	Control	GBS50	FA20	FA30	FA40	FA20 GBFS50	FA30 GBFS50	FA40 GBFS50
	M-0	M-1	M-2	M-3	M-4	M-5	M-6	M-7
FA (%)	0	0	20	30	40	20	30	40
GBFS (%)	0	50	0	0	0	50	50	50
Water (Kg)	202.5	202.5	202.5	202.5	202.5	202.5	202.5	202.5
Cement (Kg)	450	450	360	315	180	360	315	180
Fly ash (Kg)	0	0	90	135	270	90	135	270
Binder (Kg)	450	450	450	450	450	450	450	450
Water/binder	0.45	0.45	0.45	0.45	0.45	0.45	0.45	0.45
Fine agg.(Kg)	702	351	702	702	702	351	351	351
GBFS (Kg)	0	351	0	0	0	351	351	351
CA1 (Kg)	780.3	780.3	780.3	780.3	780.3	780.3	780.3	780.3
CA2 12mm(Kg)	520.2	520.2	520.2	520.2	520.2	520.2	520.2	520.2
Ch.Admix. (ltr.)	0	2.15	0	1.07	2.15	1.07	4.3	4.3
Slump (mm)	30	35	32	35	35	35	37	25

Note*- CA1 and CA2 denotes 12mm and 20mm down coarse aggregate, respectively

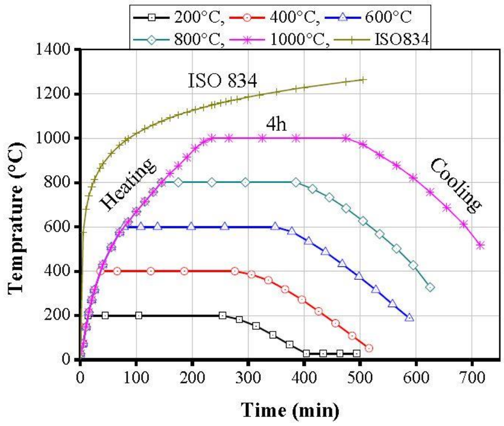


Fig. 3 Temperature time curve of electric furnace

2.2 Methodology

To achieve the objective, preliminary investigations were carried out to assure sustainability performance by checking compressive strength at ages of 7, 28 and 90days before fire exposure. Then, fire performances were assessed in terms of weight loss, degradation in compressive strength, change in colour, macro cracks on the surface of the specimen and development of micro cracks in the concrete matrix after exposed to elevated temperatures (200°C, 400°C, 600°C, 800°C and 1000°C) for 4h of duration. Visual inspection of surface colour and development of thermal cracks on the surface were observed through visual inspection. FESEM investigations were also carried out on selected specimen to corroborate the findings of test results at room temperature (RT) i.e., 27°C and 800°C.

2.3 Details of mix proportion, specimen and test

Eight concrete mixes including control mix were prepared as per IS456:2000 and IS 10262-2009 to explore the influence of elevated temperature on GBFS and fly ash based concrete. Detail of mix proportions are presented in Table 4. Control mix of M25 grade of concrete with proportion of 1:1.56:2.89 (cement: sand: aggregate) was designed for 25-50 mm slump using water cement ratio (w/c) of 0.45 with aim of achieving cubic target mean strength of 31.1 MPa. All aggregates are used in saturated surface dry conditions. Weight batching was adopted for all type of mixes. Replacement percentages were 50% GBFS as fine aggregate, 20%, 30% and 40% FA as OPC for single and combined mixes. For each case of mix, a 130 kg batch corresponding to 22 Kg of binder were prepared. Fresh property of the concrete was adversely affected by addition of GBFS due to rough surface texture and high water absorption capacity which was partially compensated by addition of fly ash. Even, high volume utilization of fly ash in mix demanded suitable dosage of chemical admixture due to increase in fine power content in the fly ash based mixes. Slump and adopted dosage of the super-plasticizer are shown in the Table 4.

In whole experimental work, cubic specimen of size 150 mm and cylindrical specimens of sizes 150×300 mm were prepared in accordance with IS: 516-1959 to obtain compressive strength at 27°C. For thermo-mechanical study, 100 mm cube specimens were casted for each mixes. Specimen were demoulded after 24h and kept in water curing tank till testing time at a temperature of 20°C±2°C and a relative humidity ≥95%. Compression test for larger size specimen were performed at 7, 28, 90days. Small size cubic specimen were also removed from curing tank at 90days and dried for next 10days in open air to remove surface moisture to avoid the deviation in test results at lower temperature range. An extended duration of moist curing was adopted for heat curing specimen to get assured of achieving target mean strength of 31.6 MPa before subjected to elevated temperature. All the mechanical loading tests were performed taking three specimens in 300MT UTM (Universal testing machine) keeping constant loading rate for the entire specimen.

2.4 Heating and cooling regime

All the specimens were heated in electric furnace (260×260×680 mm) of rated capacity of 1200°C, capable of maintaining constant temperature with ±1°C accuracy. Typical

Table 5 Compressive strength of concrete mixes

Mixes. ID	Unit Weight (Kg/m^3)	Types of Concrete Mixes						
		Cube	Cylinder	Cube	Cylinder	Strength Ratio	Cube	Cylinder
		7 days		28days			90days	
M-0	2612	24.5	17.33	38.65	31.05	0.80	48.5	39.14
M-1	2577	26.14 [6.7%]	18.64 [7.56%]	37.5 [-2.97%]	29.7 [-4.34%]	0.79	48.21 [-0.6%]	39 [-0.36%]
M-2	2564	24.35 [-0.6%]	16.87 [-2.65%]	38.71 [0.15%]	30.15 [-2.89%]	0.78	45.1 [-7%]	35.7 [-8.79%]
M-3	2555	20.3 [-17.14%]	14.6 [-15.75%]	36.41 [-5.79%]	28.61 [-7.85%]	0.79	40.61 [-16.28%]	30.93 [-21%]
M-4	2475	14.76 [-39.75%]	10.93 [-36.93%]	28.9 [-25.225]	20.5 [-34%]	0.71	35.77 [-26.24%]	26.07 [-33.4%]
M-5	2507	25.13 [2.57%] (3.20%)	17.7 [2.13%] (4.91%	40.37 [4.45%] (4.28%)	31.5 [1.45%] (4.47%)	0.78	48.53 [0.06%] (7.60%)	37.46 [-4.29%] (4.92%)
M-6	2505	21.12 [-13.75%] (4.04%)	15.8 [-8.82%] (8.22%)	38.62 [0.007%] (6.07%)	29.75 [-4.18%] (3.98%)	0.77	43.87 [-9.54%] (8.03%)	33.5 [-14.4%] (8.31%)
M-7	2498	16.39 [-33.1%] (11.04%)	12.2 [-29.6%] (11.62%)	31.85 [-17.6%] (10.21%)	23.7 [-23.67%] (15.61%)	0.74	38.89 [-19.81%] (8.72%)	28.8 [-26.41%] (10.47%)

[± Δ1]; Percentage increase or decrease in strength of mixes with respect to control mix
(± Δ2%); Percentage increase or decrease in strength of combined mix (M-5 to M-7) with respect to corresponding fly ash mix (M-2 to M-4)

heating profiles for the various thermal exposures are given in Fig. 3. For a given target temperature, the rise in furnace temperature was recorded at every 5 minutes till reaching the desired temperature. After 4h of heat curing, furnace was switched off and specimens were allowed to cool in the furnace. Next daymorning, specimens were removed from the furnace and again allowed to cool in lab temperature before compression test. Longer duration was chosen with aim to reach target temperature at the core of specimen (Bastami *et al*. 2011, Singharoy and Sai Krishna 2012, Li *et al.* 2012). Then the specimens were removed from furnace and allowed to cool naturally at room temperature. The rate of heat development in the furnace is compared to a standard rating recommended by ISO 834. Although heating rate is milder in the furnace, temperature versus time curve revealed comparable behavior, as reported by other researchers (Mohammadhosseini and Yatim 2017, Chan *et al*. 1999, Peng *et al*. 2008).

3. Result and discussion

3.1 Compressive strength at room temperature

Cube and cylindrical compressive strength of all the concrete mixes was evaluated at ages of 7, 28 and 90days of moist curing to check whether FA and GBFS from particular source are suitable or not for normal exposure. Test results of compressive strength at various ages are shown in Table 5. Both the types of specimen exhibited relatively similar trend of variation in results for all type of concrete mixes. Hence, cubical specimen has been considered for discussion. The 7days compressive strength of mixes (M-0, M-1, M-2 to M-4 and M-5 to M-7) are 24.5, 26.14, 32, 25.49, (24.35, 20.3, 14.76) and (25.13, 21.12, 16.39MPa), which enhances to 38.65, 37.50, (38.71, 36.41, 28.9) and (40.37, 38.62 and 31.85) at 28 days, respectively. However, high replacement mixes could not be able to achieve target mean strength of 31.1MPa at 28days. But, consistent improvement in strength was with age of concrete. Compressive strength of the mixes was highly influenced with percentage increase in sustainable material which prolonged the development of strength towards higher ages. Patra and Mukherjee 2015 found 9.22%, 15.02% and 24.79% higher strength for 20, 40 and 60% GBFS concrete than control mix at 7 days. Binici *et al.* (2008) observed higher compressive strength development ratio for GBFS concrete at early ages with respect to control mix whereas Hemalatha and Sasmal (2017) found lower early strength of FA mixes with gradual improvement till 90 days. When combined replacements of GBFS and FA were executed, improvement in strength was observed. Combined mixes (M-5, M-6 and M-7) improved the cube compressive strength by 3.2-11% at 7days, 4.3-10.2% at 28days and 7.6-8.7% at 90days with respect to 20%, 30% and 40% FA mixes (M-2, M-3 and M-4), respectively while there was degradation in strength with increase in replacement percentages. Replacement of GBFS or FA decreases the strength which was marginal for M-1(GBFS50) and M-2(FA20) which may be attributed to weakening of mortar bond due to glassiness nature of high percentage GBFS addition and the late pozzolanic reaction of Class F fly ash for strength development. On the other hand, improvement in strength of combined mixes (M-5, M-6 and M-7) with respect to single mixes (M-2, M-3 and M-4) which may be attributed to enhancement in packing density of mortar along with increase in friction resistance between smooth and

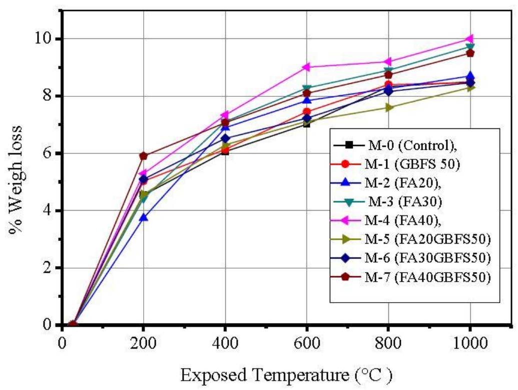


Fig. 4 Percentage weight loss of concrete mixes with elevated temperature

spherical shaped FA particle and rough and angular shaped GBFS, which strengthen the mortar strength of concrete matrix.

The ratios of the cylindrical and cube compressive strength of concrete specimen at 28 days were obtained as 0.8, 0.79, (0.78, 0.786, 0.71) and (0.78, 0.77, 0.74) for the concrete mixes (M-0, M-1, M-2 to M-4, M-5 to M-7), respectively.

Strength ratio of test results revealed that the ratio of cylindrical compressive strength and cube compressive strength were mostly influenced by their trend of strength. As the ratio is adopted as 0.8 (Al-Sahawneh 2013, Nevellie 2012), sustainable mixes M-1, M-2, M-3, M-5 and M-6 exhibited close approximated value. Hence, combined concrete mixes performed well among the mixes which can be explained by uniformity of concrete matrix and densification after combined replacement of GBFS and FA, as depicted in FESEM analysis (Fig. 9).

3.2 Properties of concrete at elevated temperature

3.2.1 Weight Loss

Percentage weight loss (%WL) increases with the rise in temperature for all the concrete mixes irrespective of type of mixes, as shown in Fig. 4. It shows that as the interval of temperature increases, the gradient of weight loss decreases. These losses were about 3.74-5.9%, 6.06-7.34%, 6.62-9.01%, 8.06-9.2% and 8.3-10% after subjecting to 200, 400, 600, 800 and 1000℃, respectively. It represents three zones for losses defined as steep, moderate and mild in the temperature zone of 27-400℃, 400-800℃ and 800-1000℃. Between 27-200℃, about 50-60% of Total Weight Loss (TWL) was observed which may be attributed to capillary water, physically absorbed which comprises most of the water. It is evaporable water could be get completely released when heated up to 400℃. It is known fact that TWL includes capillary water, physically absorbed water (Gel water) and chemically bound water in calcium silicate hydrate (CSH) and calcium hydroxide (CH). Hanaa *et al.* (2009) found 70% of the water contained in the concrete is evaporated at 300℃. Siddique and Kaur (2012) and Noumowe *et al*. (2009) also found weight loss of about 6% when the temperature was below 400℃. Initial weight loss was influenced with high absorption capacity of GBFS and water holding capacity of fly ash in the mix as water to reactive binder ratio in fly ash mixes is very high due to late pozzolanic reaction. Mixes M1, M4 and M7 had lossed water about 5.03%, 5.29% and 5.9%, respectively at 200℃ and 6.14, 7.11, 7.0 % at 400℃.

Between 400-600℃, weight loss was about 70-90% of TWL, might be due to the dissociation of calcium silicate gel (C-S-H) at 400℃ and dissociation of CH at 600℃. After exposure to 600℃ and above, the difference in %WL was very low about 0.2-1.3%, might be due to loss of chemically bonded water of silicates gel (Arioz 2007, Kodur 2015). Fly ash mixes (M-2 to M-4) indicated that weight loss increased with the increase in fly ash percentage, as observed by many investigators, utilized fly ash or others pozzolanic material as mineral additive (Siddique and Kaur 2012, Li *et al.* 2012, Khan *et al.* 2013, Mohammadhosseini and Yatim 2017). There was marginal increment in %WL after 600℃. Also, FA40 exhibited highest loss of water which indicates that much of the fly ash in mix could not formed bonds with water to form secondary C-S-H gel at the age of 90days.

At 800℃, weight loss may be considered because of rapid loss of initial moisture along with water loss of C-S-H gel along with dissociation of C-S-H which might have caused thermal cracks in the specimen, clearly observed in the specimen after 800℃. At 1000℃, the highest weight loss was for 40% FA mix (M-4) while lowest for control mix (M-0) followed by GBFS 50 (M-1). Combined mixes have higher percentage loss than control mix but lower than parent fly ash mixes which is streamline with Ahn *et al.* (2016), used expanded shale, bottom ash as aggregates with fly ash. Sancak *et al.* (2008) utilized pumice aggregate and silica fume in place of conventional ingredients, found higher weight loss for Normal weight concrete (NWC) than light weight concrete (LWC) and mild changes in percentage weight loss after 800-1000℃. They reasoned that diffusion of heat in concrete is less in LWC than NWC because of lower thermal conductivity of LWC than NWC.

3.2.2 Colour change and crack appearance

Visual inspection of the specimen of all the concrete mixes indicated no change in colour up to 400℃ as compared to conventional colour at RT (27℃). Control mix (M-0) specimen slightly changes from its original grey colour (27℃ to 400℃) to partially reddish at 600℃ followed by off whitish pink colour after exposed to 800℃ while GBFS mix changed into white at high temperature. Fly ash mixes changed its blackish grey color into reddish black at 600℃ and reddish white at 800℃. Although it was difficult to identify the superimposed color resulting from grey, red, black and white, presence of red, white and reddish white were might be attributed to siliceous aggregates of natural sand, high content of silica and ferric oxide in fly ash and high lime content in GBFS, respectively. At 800℃ and 1000℃, control mix (M-0), GBFS mix (M-1), fly ash mix (M-2 to M-4) and combined mixes (M-5 to M-7) changed to white, red and reddish white. Hence, colour change of heated concrete is primarily influenced with binding paste and fine aggregate of the mortar as it exist on the outer surface of the specimen. Overall, three types of basic changes in colour changes in the specimen were observed in the concrete specimen, as shown in Fig. 5. Colour changes in the specimens follow the observation made by (Hager 2013, Lee *et al*. 2010).

In the present study, no crack was observed through visual

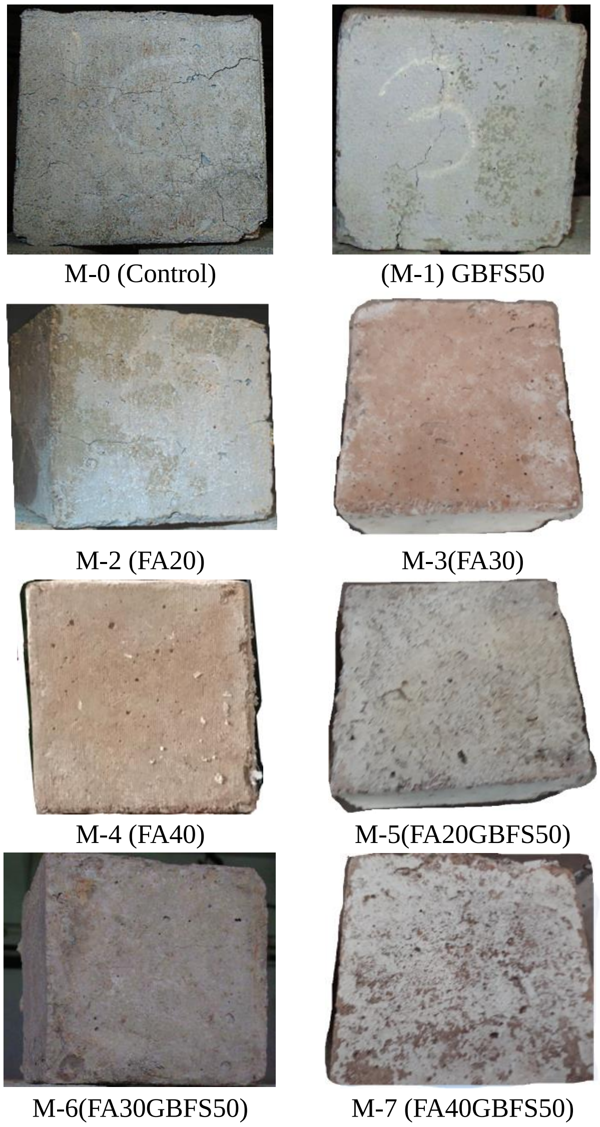


Fig. 5 Surface appearance of the concrete specimen after exposed to 800°C

inspection in specimens after exposure to 200°C and 400°C; a few of hairline cracks were initiated at 600°C, clearly visible at 800°C and continued to widen up till 1000°C. However, combined mixes (M-5, M-6 and M-7) were more thermally stable in view of cracks, corner spalling than other mixes at all level of temperature. During the whole heat curing period, no explosion and spalling were observed that might be due to lower heating curve as compared to ISO 834 and pores in light weight concrete (Singharoy and Sai Krishna 2012, Chen *et al.* 2014). It is mostly observed in high strength concrete at high temperature due to high density causing high build up vapour pressure.

3.2.3 Residual compressive strength

Compressive strength of concrete specimen (100 mm) for concrete mixes (M-0 to M-7) was obtained as 50.5, 49.74, 44.53, 41.22, 39.18, 45.76, 42 and 42.34 MPa, respectively at RT (27°C). For distinguishing the role of additives in concrete mixes, compressive strength versus temperature and residual compressive strength (%) versus

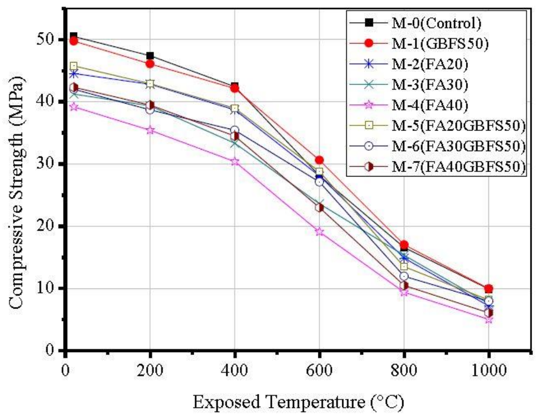


Fig. 6 Compressive strength of mixes at elevated temp

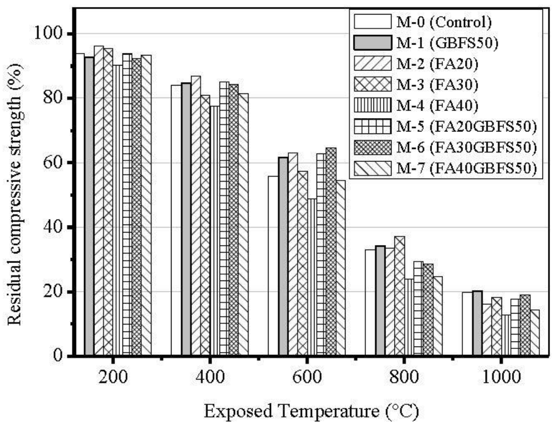


Fig. 7 Residual compressive strength of mixes at elevated temperature

rise in temperature have been shown in Fig. 6 and Fig. 7 respectively. From the viewpoint of strength loss, three temperature ranges: 27-400°C, 400-800°C and 800-1000°C have been considered for analysis.

At 200°C, decrement of 3.9-7.8% in compressive strength as compared to RT strength were observed for all the mixes which is not remarkable as strength could be regained after water curing (De Souza and Moreno 2010). As longer time of heat curing (4h) was adopted, it might have removed most of the evaporable and fraction of hydrated water. Percentage weight loss (%WL) and percentage loss in strength (%SL) were 3.74-5.9% and 3.9-7.83%, respectively, with respect to all mixes. It indicated that initial losses in strength were influenced with weight loss at 200°C. All other mixes performed more or less similar to control mix with marginal change in percentage strength retention at 400°C. Till 400°C, fly ash based mixes retained either higher or similar residual strength which may be attributed to pozzolanic nature of fly ash combines with free CH prior to its decomposition and form secondary cementing products C-S-H thereby, sustaining the strength (Rashad 2015).

Between 400°C to 800°C, steep fall in strength were observed for all mixes, especially for high fly ash based mixes (M-4 and M-7). Binder thermal stability is the main reason of deterioration after 600°C (Savya *et al.* 2005). Observed losses may also be related to loss of chemically

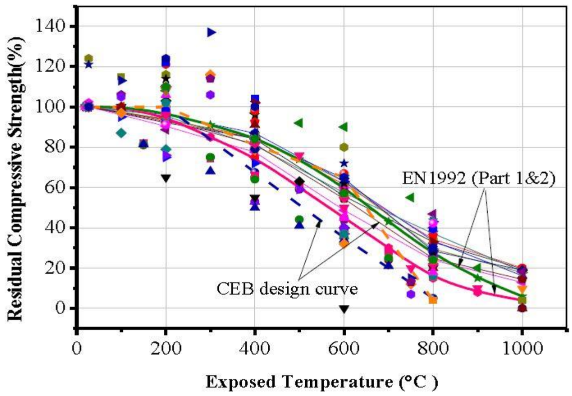
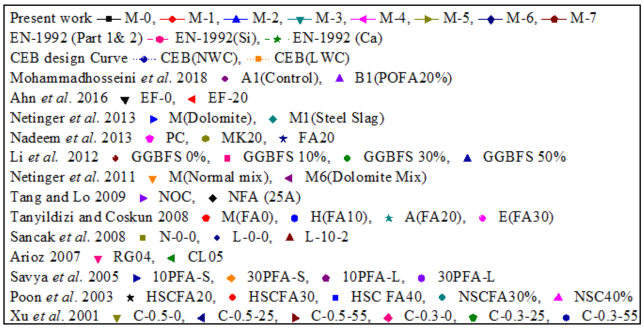


Fig. 8 Comparative study of residual compressive strength at elevated temperature



bounded water, increase in porosity which might have caused further degradation. It might be also due to contraction of binding paste following water loss and aggregate expansion (Fares *et al*. 2010) and decomposition of C-S-H which causes cementing ability. On the other hand, GBFS mix and combined mixes exhibited higher retention of strength, indicated thermal stability of aggregate, protecting the interfacial bond between aggregate and binding material. As GBFS is limestone and dolomite based calcareous aggregate, it has higher fire resistance than local river sand (Netinger *et al.* 2015) as it restricts thermal degradation due to the hindrance in diffusion of heat into concrete. Compressive strength retention of the mixes at 600°C were 55.73%, 61.54%, (63.06%, 57.33%, 48.72%) and (62.89%, 64.52%, 54.35%), which further degraded to 32.9%, 34.26%, (33.46%,37.2%, 24.02%) and (29.5%, 28.47%, 24.75%), respectively for the mixes (M-0, M-1, M-2 to M-4 and M-5 to M-7) at 800°C. Hence, additions of pozzolanic material along with fire resistant aggregate GBFS greatly influence the performance of concrete. The above combined effect of GBFS and FA were also observed at 800°C and above, with severe deterioration due to decomposition of CSH gel and complete dehydration of CH into free lime which can easily observed as whitish scalp over the specimen of control mix and GBFS mix while reddish white in fly ash mixes and combined mixes. Xiuli *et al*. (2014) also reported that fly ash reacts with remaining CaO (free lime) to produce to bermorite gel, a more stable product than C-S-H which contributes strength after exposure to 800°C as difference in residual strength between control and fly ash based mixes is marginal.

Exposing the concrete specimens to the highest level of temperature (1000°C) led to further degradation in the compressive strength along with development of wide cracks irrespective of concrete mixes. At this stage, residual strength is not sufficient even up to sustain its integrity against free fall as observed in some of the specimen irrespective of ingredients of mixes. Body crack clearly indicated the total loss of binding properties. It is streamlined with Sancak *et al.* (2008), reported similar fire performance of NWC and LWC at 1000°C.

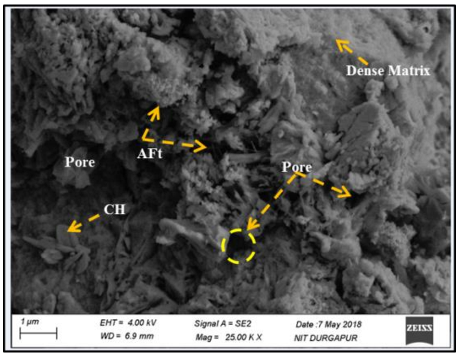
Li *et al.* (2012) obtained 35% and 21% residual compressive strength for concrete made of 50% ground granulated blast furnace slag (GGBFS) at 600°C and 700°C while concrete mix (M-1) having 50% GBFS as naturals and retained about 61.54% and 34.26% at 600°C and 800°C, respectively in the present work. Even though there is difference in residual compressive strength due to difference in methodology, it might be a promising concept to use blast furnace slag in granular form than in grinded form because grinding also imposed additional costs as also reported by Ö zkan *et al.* (2007).

In order to compare the behaviour of concrete mixes with other works, research data are extracted from original source and represented in the Fig. 8. The residual compressive strength data corresponds to the mixes having various forms of ashes, light weight aggregate, calcareous and siliceous aggregates and granite aggregate. It is important to note that variation in residual strength with respect to others is wider in the temperature range 200-400°Cwhich tends to concentered toward higher temperature at 600°C and higher which might be attributed to the influence of moisture losses which streamlined with Li *et al.* (2012), Savya *et al.* (2005), Poon *et al.* (2003) and others.

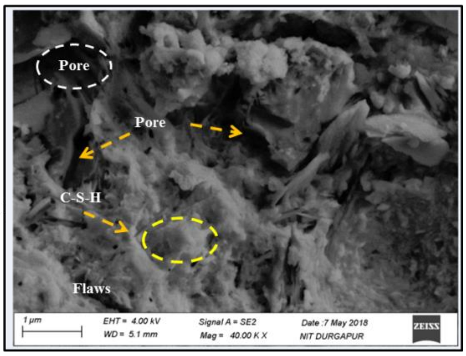
At 600°C, test results (61-64%) for the mixes (M1, M5 and M6) is streamlined and having close approximation with EN: 1992(1-2) (Ca), CEB (1991)(LWC), Neitinger *et al.* 2013 (Dolomite, steel slag), Ahn *et al.* (2016) (EF20) and Poon *et al.* 2003 (HSCFA 20 and HSCFA 30) whereas under estimates Li *et al.* (2012) and Savya *et al.* (2005) and overestimates Nadeem *et al.* (2013), Arioz (2007), Tanyildizi and Coskun (2008). It can be seen that concrete made with granular blast furnace slag as fine aggregate along with or without fly ash in concrete show comparable results.

3.2.4 Microstructure analysis through FESEM study

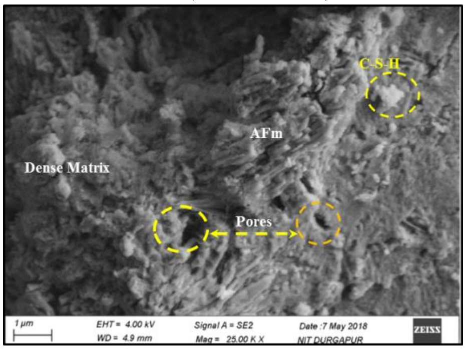
FESEM investigations demonstrated variation in the morphology of concrete specimen at 27°C and 800°C (Fig. 9 and Fig. 10) for control mix concrete (M-0), sustainable mixes having 50% GBFS (M-1), FA 40% (M-4) and 50% GBFS with 40% FA mix (M-7). Here, selected specimen were chosen for FESEM analysis as it could be expected that micro-structural changes due to partial replacement of natural sand and Portland cement with GBFS and FA, can be enlightened. The basic features for investigating the scanned images were fraction of hydrated products such as cotton type calcium silicate hydrate (C-S-H gel), needle-shaped ettringite (Aft), hexagonal cylinder or rode shaped mono-sulphate (AFm) and plate shaped calcium hydroxide (CH) in matrix (Li and Zhao 2003, Lv *et al.* 2017) along with formation of pores and flaws, cracks and coarsening of



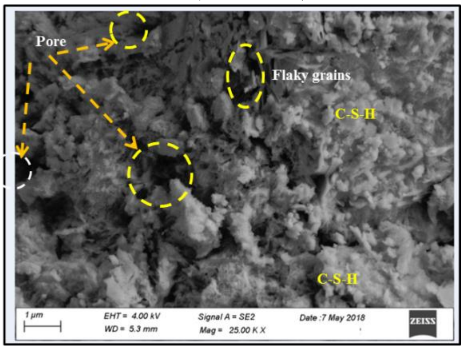
M-0 (Control mix)



M-1(GBFS 50)

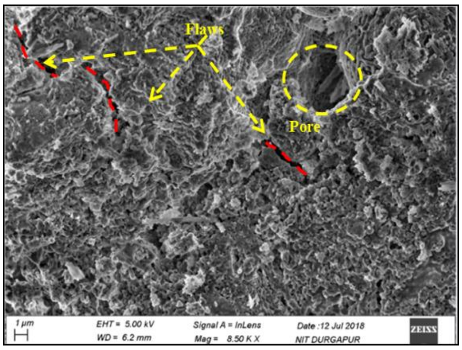


M-4(FA40)

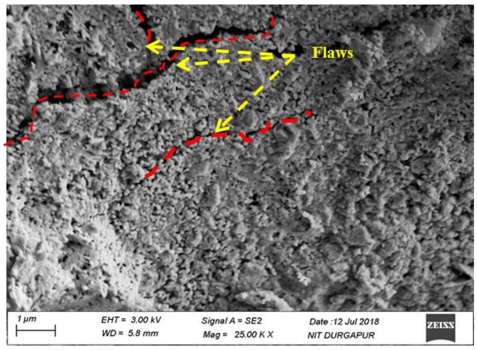


M-7 (FA40GBFS50)

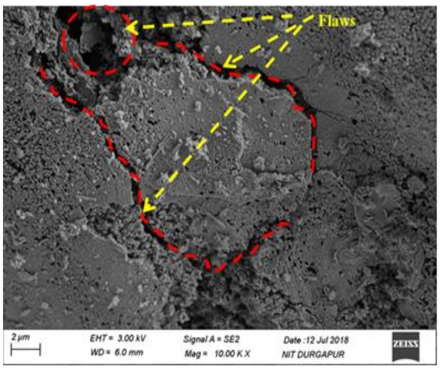
Fig. 9 Scan electron microscope image for typical concrete mixes at RT (27℃)



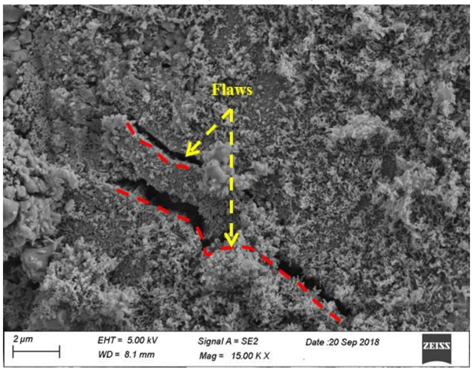
M-0 (Control mix)



M-1(GBFS 50)



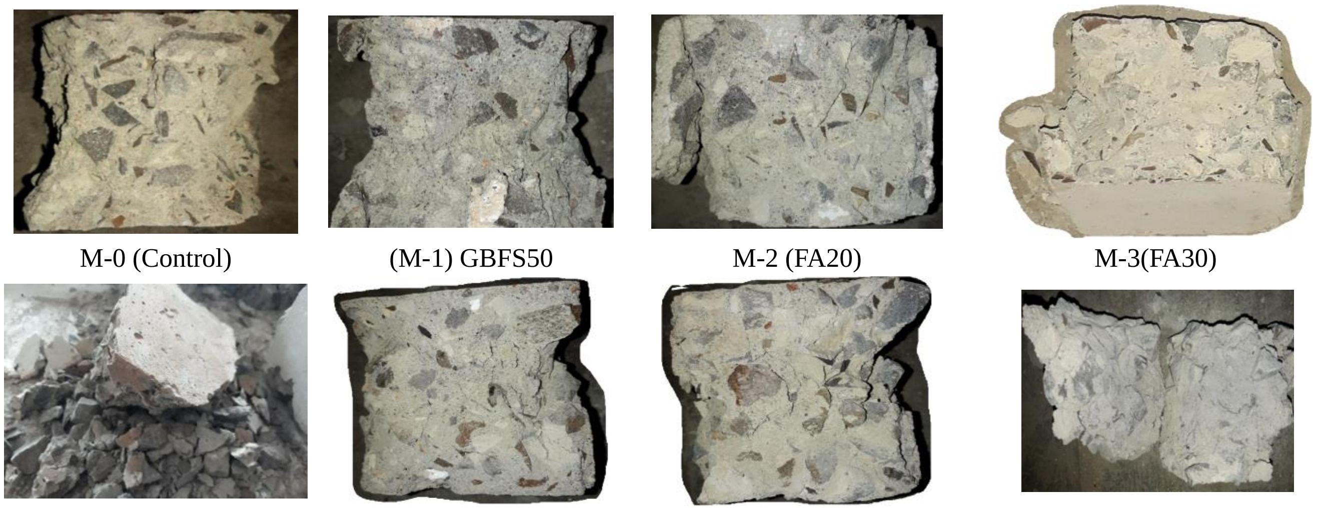
M-4(FA40)



M-7 (FA40GBFS50)

Fig. 10 Scan electron microscope image for typical concrete mixes at 800℃

concrete matrix. Therefore, analyzing feature of the mortar matrix were cotton like, little hexagonal rod shape like, needle-like features and sheet-like crystals at RT whereas coarsening, uneven microstructure and the existence of cracks and larger pores and holes at 800℃, From Fig. 9, It is predicted that control mix concrete (M-0) has irregular microstructure having many pores but having enough amount of cotton ball shaped C-S-H gel and uniformly distributed needle shaped ettringite and a very few CH which contributed to gain high strength.



M-0 (Control) (M-1) GBFS50 M-2 (FA20) M-3(FA30)

M-4 (FA40) M-5(FA20GBFS50) M-6(FA30GBFS50) M-7 (FA40GBFS50)

Fig. 11 Specimen under compression failure after exposed to 800℃

Hence, main hydrated products are C-S-H gel and ettringite (Aft). Fig. 9 M-4 shows uniformly distribution of C-S-H gel and development of extra C-S-H gel, due to the consumption of portlandite by pozzolanic action of fly ash, resulted in improved concrete matrix. Also, it may be noted that addition of fly ash leads to the decrease of the surface area by filling pores and reduces the proportion of the smallest pores. Unreacted spherical fly ash particles were not clearly recognized at any other magnification also which indicates complete pozzolanic reaction has progressed. Moreover, pores and disarray of silicates gel in the GBFS mix (Fig. 9 M-1) could be attributed to insufficient compaction of fresh concrete because of glassy and angular shaped flaky particles and quick soaking of water from fresh mix due to high water absorption. Even though, sufficient C-S-H gel could be observed in the matrix and seems to be deposited over flaky grains of GBFS. Flaws or holes observed in the image might be due to space created by aggregates. In this case, the main hydration product is C-S-H but sheet like CH cannot be clearly distinguished. After combined replacement of 40%FA and 50% GBFS mix (M-7) filled the pores and improved the microstructure of concrete than GBFS-50 mix (M-1) as depicted in (Fig. 9 M-7). Even though, it's microstructure is poor than control mix in view of porosity and packing density, these features are crucial requirement for improving the fire performance, as covered in literature review as well-known fact for fire resistant concrete.

Fig. 10 predicts that microstructure of the concrete mixes was extremely damaged due to internal cracking and coarsening, caused by dissociation of C-S-H at 800℃ for mixes M-0, M-1, M-4 and M-7. It can be seen that concrete matrix of GBFS and Fly ash mix (M-7) was more compact than that of Fly ash 40% mix (M-4) and GBFS 50% (M-1) mix, even than OPC mix (M-0). Fractural damage in 40% FA mix was more than GBFS mix (M-1) and control mix (M-0). FA40% mix showed internal cracking in binding paste but coarsening is less observed while GBFS50mix showed internal cracking along coarsening. Furthermore, combined blended mix (M-7) exhibited coarsening but having insignificant crack in matrix which may be attributed to high thermal stability of porous GBFS because of formation history there by protection of (C-S-H) bond. Overall, it is observed that the micro-cracks observed in the control concrete are much more extensive and widened at cement mortar, compared to that of combined mix concrete.

3.2.5 Failure behaviour of fire exposed specimen under uniaxial loading

Conventional failure of cube transforms the specimen in pyramidal form or a cone which indicates uniformity of the concrete matrix at RT (27℃). But, this conventional failure ceases to brittle and collapsible failure at high temperature. Basically, two types of failure were observed after inspecting the fracture surface of the specimen: conventional failure of concrete as on RT i.e., mortar and coarse aggregate failure (Type1) and mortar failure (Type II) at higher temperature. Type I failure was observed for all the specimen exposed up to 600℃ while Type II failure started to dominate at 800℃ and above. At 800℃, pure Type II failure was observed for 40% fly ash mix (M-4) and 40% FA & 50% GBFS mix (M-7). Examples for both the type of failure can easily be distinguished from Fig. 11. Mixes M-4 and M-7 exhibited sudden failure at 800℃ after reaching failure load due to mortar failure prior to coarse aggregate could take load. Moisture losses due to evaporation of free water, absorbed water, chemical bounded water and dissociation of calcium silicate bond in concrete had influenced the failure. Overall, all the specimen (M-0 to M-7) exhibited Type II failure at 1000℃. No one specimen with or without mineral additives could prevent mortar bond failure at this temperature.

4. Conclusions

- Combined mixes, made with partial addition of GBFS and fly ash as fine aggregate and with Ordinary Portland Cement, enhanced compressive strength with respect to fly ash mixes.FESEM study revealed that density of concrete matrix improved after combined replacement of granular blast furnace slag and fly ash. In the images, extra C-S-H gel was found in the sustainable mixes as compared to control mix.

• Conventional changes in colour of control mix specimens were observed as original grey colour between (27℃ to 400℃) to partially reddish at600℃ followed by off whitish pink after exposed to 800℃. Visual observation indicates that percentages of fly ash and granular blast furnace slag in concrete directly affected the colour change.No appreciable change in colour up to 400℃ has been observed. After 600℃, reddishness appears with addition of fly ash, whereas it changes to whitish with addition of GBFS. Combined mixes specimen changes to reddish white with the rise in temperature beyond 600℃.

• Residual compressive strength of combined mixes made with 20-30% FA and 50% GBFS performed better at all range of temperature. Even, high replacement mixes (40%FA and 50% GBFS) exhibited close to control mix at 600℃ and 800℃. However, most of the mixes exhibited similar percentage of compressive strength retention at 1000℃ with close approximation. At 800℃, SEM images indicated flaws, pores and micro-crack in the concrete matrix of control mix, GBFS mix, fly ash mixes while there was no significant deterioration in concrete matrix for the combined mix.

• The entire specimen exhibited conventional failure (i.e. mortar bond and coarse aggregate failure) up to temperature 800℃ except M-4 (40% FA) and M-7 (40% FA and 50% GBFS) mixes. Hence, concrete of such type should be used carefully for the structure exposed to elevated temperature of 600℃ and above, because pure mortar failure of concrete may lead to sudden collapse at high temperature. However in the authors' opinion, concrete mixes made with 30% FA and 50% GBFS can be suitably recommended for fire protection in view of high volume utilization, residual strength, crack resistance and mode of failure.

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