Efficacy of supplementary cementitious material and hybrid fiber to develop the ultra high performance hybrid fiber reinforced concrete

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Abstract. The rich recipe of ultra high performance concrete (UHPC) offers the higher mechanical, durability and dense microstructure property. The variable like cement/sand ratio, amount of supplementary cementitious material, water/binder ratio, amount of fiber etc. alters the UHPC hardened properties to any extent. Therefore, to understand the effects of these variables on the performance of UHPC, inevitably a stage-wise development is required. In the present experimental study, the effect of sand/cement ratio, the addition of finer material (fly ash and quartz powder) and, hybrid fiber on the fresh, compressive and microstructural property of UHPC is evaluated. The experiment is conducted in three phases; the first phase evaluates the flow value and strength attainment of ingredients, the second phase evaluates the efficiency of finer materials (fly ash and quartz powder) to develop the UHPC and the third phase evaluate the effect of hybrid fiber on the flow value and strength of ultra high performance hybrid fiber reinforced concrete (UHP-HFRC). It has been seen that the addition of fly ash improves the flow value and compressive strength of UHPC as compared to quartz powder. Further, the usage of hybrid fiber in fly ash contained matrix decreases the flow value and improves the strength of the UHP-HFRC matrix. The dense interface between matrix and fiber and, a higher amount of calcium silicate hydrate (CSH) in fly ash contained UHP-HFRC is revealed by SEM and XRD respectively. The dense interface (bond between the fiber and the UHPC matrix) and the higher CSH formation are the reason for the improvement in the compressive strength of fly ash based UHP-HFRC. The differential thermal analysis (DTA/TGA) shows the similar type of mass loss pattern, however, the amount of mass loss differs in fly ash and quartz powder contained UHP-HFRC.

Keywords: Ultra High Performance Concrete (UHPC); Steel Fiber; Crimped Fiber; Hooked Fiber; Hybrid Fiber; Fly Ash; Quartz Powder

1. Introduction

In past, the ultra high performance concrete (UHPC) has been developed using various types of supplementary cementitious material (SCM) (Yu et al. 2014a, b, Staquet and Espion 2004, Yu et al. 2015, Wang et al. 2012). The pozzolanic activity, finer particle size, and texture of SCM significantly improve the microstructure of matrix and consequently help in the development of denser matrix. Further, the categorization of calcium silicate hydrates (CSH) such as high-density CSH (HD-CSH) and lowdensity CSH (LD-CSH) help to categorize the compound formation of UHPC (Ulm et al. 2010, Lura et al. 2011, Sorelli et al. 2008). Therefore, the UHPC recipe is significantly distinct than normal concrete such as fine particle size lower than 1 mm, water cement ratio less than 0.24, the fiber of more than 2% by volume, ranging 10 to 20 mm in length and 0.1 to 0.25 mm in diameter. Various models, such as Modified Andreasen and Andersen model, Linear Packing Density model, Solid Suspension model (Fennis and Walraven 2012, Yu et al. 2014c, Funk and

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Copyright © 2019 Techno-Press, Ltd. http://www.techno-press.org/?journal=acc&subpage=7 Dinger 1994) are also available in the literature for design the UHPC. Although the first design rules for UHPC were published in France in 2002, thereafter, Japan's guideline appeared in 2004. It has been seen that the performance of UHPC is highly sensitive to the various parameter i.e., cement/sand ratio, water/binder ratio, amount of SCM, interfacial transition zone, type of fiber, state of fiber (mono or hybrid foam) and fiber matrix interaction. The lower cement/sand ratio increases the flowability and viscosity and vice versa. Simultaneously, the water/binder ratio affects the fresh and hardened property of the mix. At a constant sand/cement ratio of 1.25, the decrease in the flow is found to be 60% when the w/c ratio is decreased from 0.30 to 0.25. The strength of the mix increases with cement/sand ratio from 0.5 to 1.25 and then decreases (Rangaraju et al. 2014). Further, the fresh and hardened properties of the matrix are largely dependent upon the type of SCM. The nano-silica and rice husk ash, due to the higher surface area, absorbs a significant amount of water in the green stage of UHPC, as a result, the amount of water available for lubrication reduces. Hence, there is a considerable decrease in workability (slump flow values) of UHPC (Tuan et al. 2011, Ghafari et al. 2014, Korpa and Trettin 2008). Whereas, the inclusion of palm oil fuel ash (POFA) improves the workability of UHPC because of the lower specific gravity of POFA in comparison to the OPC. Due to the lower specific gravity of POFA, higher binder paste volume obtained at higher percentage replacement

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increases the workability of concrete (Johari et al. 2012, Mohammed et al. 2014). In fact, the effect of SCMs on workability affects the mechanical and durability properties of UHPC also. Therefore, it is important to study the effect of SCMs on the fresh property as well as the mechanical property of UHPC. It has also been recognizing that the ITZ phase and the paste phase are the weakest phases in the concrete, particularly, during loading the crack initiation has taken place either in the paste phase or ITZ phase. This observation leads to the researchers on the next paradigm of improving the concrete performance through the improvement of the paste phase and ITZ. The porosity in the vicinity of aggregate is higher in normal concrete but usage of superplasticizer and finer material the (Supplementary cementitious material) reduce the space by nucleation all around. The microstructural differences between the pastes in the ITZ and that in the bulk will be lowered (Ollivier et al. 1995). In addition, the matrix itself improved through the densification and higher CSH formation through the use of supplementary cementitious material (SCM). It has been observed that the removal of the aggregate phase leads to the removal of the ITZ phase and the addition of SCM improve the performance of the paste phase consequently, the ultra high strength concrete obtained. However, the large-scale construction by UHPC is not possible only by achieving the aforementioned parameters. The failure pattern i.e., brittle failure of ultra high strength concrete losing the serviceability criteria. To overcome this limitation, the fibers are being used to impart the ductility in ultra high strength concrete. The addition of fibers in UHPC mix increases the compressive strength of more than 25% (Prem et al. 2012). Abbas et al. (2015) have investigated the effect of steel fiber length and content on the mechanical and durability properties of UHPC. The fibers length used were 8 mm, 12 mm, 16 mm and contents 1%, 3% and 6% of the volume of concrete. It has been observed that there is a slight increase in compressive strength with the addition of steel fibers along with that fiber length had no appreciable influence on the compressive strength of the UHPC specimen. Another important observation was that the sudden explosive behavior of UHPC without fibers changes to ductile behavior with the addition of steel fibers. Further categorization of the effect of various types of fiber demonstrates that the short, crimped or hooked fibers are more effective in compression than long straight fiber But at the higher content of steel fibers compressive strength was not affected much (Schmidt and Fehling 2005, Reda et al. 1999). The other properties like tensile and flexural strength are drastically improved, strain hardening and strain softening behavior is observed between onsets of cracking to ultimate failure (Park et al. 2012, Yang et al. 2011). The criteria of serviceability are fulfilled by the addition of fibers in the ultra high strength concrete. The efficient interaction between the dense matrix and fiber imparts the excellent advantages i.e., higher energy absorption, higher ductility and stress retention period of fiber-matrix bond increase.

Nowadays, the development in UHPC results in ultra high performance fiber reinforced concrete and ultra high performance hybrid fiber reinforced concrete. The usage of fiber in the mono state significantly affects the fresh and hardened properties of concrete. However, it is difficult to obtain high strain capacity (ductility), high tensile strength and high energy absorption with the use of single type fiber. To overcome these issues, researchers commenced the use of hybrid fiber to produce the UHPC, which is termed as ultra high performance hybrid fiber reinforced concrete (UHP-HFRC) (Park et al. 2012, Kwon et al. 2014, Nguyen et al, 2013). It is reported that the use of hybrid fibers improves the compressive strength of UHP-HFRC as compared to UHP-FRC. The addition of 1.5 vol.% long fiber+0.5 vol.% short steel fibers in the reference mix of UHPC achieved the 28 days compressive strength of 141.5 MPa, whereas, the reference mix achieved 99.0 MPa. The 43% improvement is observed after the addition of hybrid fibers (1.5 vol. % long fiber+0.5 vol.% short steel fibers). The addition of only short steel fibers 2 vol. % in UHPC the compressive strength improved from 99 MPa (reference mix) to 120.8 MPa. Hence, the addition of only short steel fibers is not as efficient as compared to hybrid fibers (long steel fibers+short steel fibers) in improving the compressive strength of UHP-HFRC (Yu et al. 2014c). In addition, the fibers alter the failure pattern and resulted in the ductile failure of concrete (Kwon et al. 2013). Currently, the ultra high performance hybrid fiber reinforced concrete (UHP-HFRC) is the advanced evolvement in the field of concrete and specifically preferred for the seismic resistance structures.

The present experimental study conducted to evaluate the effects of cement/sand ratio, the effect of the addition of fly ash and quartz powder and, hybrid fiber to develop the ultra high performance concrete. The entire research program is conducted in three Phases. The phase I and phase II is prepared to find the optimum dosage of ingredient (sand/cement ratio, water/binder ratio, superplasticizer dosage, the effect of the addition of fly ash and quartz powder) by putting the selection criteria of workability and compressive strength. Further, the bestperformed UHPC matrix from phase II is cast with hybrid fiber in Phase III. It has been concluded that the use of fly ash leads to developing the dense matrix then quartz powder contained UHPC matrix and presence of hybrid fiber enhance the strength properties and improve the crack behavior.

2. Experimental program

2.1 Material

The OPC - 53 grade (Ultra Tech) with specific gravity 3.12, normal consistency 32% and 28 days compressive strength of 57 MPa was used. The Ennore sand available from Tamilnadu (India) with specific gravity 2.64, water absorption 0.88% with size range 1 mm - 90 μ m was used for UHPC production.

The grey color silica fume, specific gravity 2.2 and a mean particle size 0.15 μ m and off-white color metakaolin with a mean particle size range of 3 μ m were used as a



Fig. 1 Scanning electron microscopic images of (a) Cement, (b) Fly ash, (c) Metakaolin, (d) Quartz powder, (e) Ennore sand, (f) Silica fume

Table 1 Properties of fibers

Types	Length (mm)	Diameter (mm)	Aspect ratio	Ultimate tensile strength (N/mm ²)
Crimped steel fibers	15	0.6	25	1200
Hooked steel fibers	60	0.75	80	1200

mineral admixture. The specific gravity of metakaolin and quartz powder was 2.50 and 2.65, respectively. The morphology of the used material is shown in Fig. 1. A polycarboxylic ether based superplasticizer was used to adjust the workability of concrete.

2.2 Fiber

The crimped steel fiber and hooked steel fiber is used with a distinct aspect ratio. The aspect ratio of 25 and 80 and the tensile strength of 1200 MPa are used in the study as shown in Table 1. Two types of fibers, Crimped (15 mm×0.6 mm) and hooked steel fiber (60 mm×0.75 mm), as shown in Fig. 2. The short crimped fiber has undulation along the length and hooked fiber contained the mechanically deformed hook at both the ends. The undulation of crimped fiber provide the anchorage of the fibers and resist the growth of crack whereas, the long length hooked fiber control the propagation of macro crack through the hooked end configuration. Therefore, multilevel reinforcement system is used in this study.

2.3 Mix design and mixture proportions

The UHP-HFRC was developed in three phases (Phase I, Phase II and, Phase III). In phase I, the effect of



Fig. 2 Crimped and Hooked Fiber

sand/cement ratio, superplasticizer dosage and water/binder ratio on flowability and compressive strength were evaluated. Three superplasticizer dosages 0.50%, 1.5% and 2.53% by weight of the binder, three water/binder ratios 0.25, 0.225 and 0.172, and two sand/cement ratios 1.38 and 1.05 were considered in the study. The details of the various mixes are shown in Table 1. The best mix in terms of workability and compressive strength in phase I selected for the phase II study. In phase II, the effect of the addition of finer materials i.e., fly ash and quartz powder, on the workability and compressive strength of the mix was evaluated. The best-performed mix in phase II was, further, chosen for evaluating the efficacy of hybrid fiber in phase III. The fibers in a hybrid state (1% crimped fiber+1.5% hooked fiber) were selected based on the previous published literature to prepare the UHP-HFRC (Kwon et al. 2013). The detail of the various mixes is shown in Tables 2-4.

2.4 Mix procedure and sample preparation

The W/B ratio and SP dosage were varying to attain the required flow i.e., 130 mm-180 mm. The procedure for preparing the UHPC and UHP-FRC/UHP-HFRC is described below;

- Binder and sand in the dry state was mixed in a mortar mixer for 2 minutes (to obtain uniformity)
- The steel fibers added in the dry mix
- Part of water (80%) added in the dry mix and mixing was done for 3 minutes.
- After 3 minutes, the water+Poly carboxylate ether based superplasticizer added in the mix and mixing was done for 8 minutes.
- The prepared mix was filled in $70.5 \times 70.5 \times 70.5$ mm and 150 mm×300 mm (diameter×length) size mould. The specimens were de-moulded after 24±1 hour and cured in water at 21° Celsius throughout the curing period.
- Three specimens of each mix were cast and tested at 3, 7, 28 and 90 days. The reported compressive strength of the sample for each mix is the average of three specimens.
- The specimen sizes of 160 mm×40 mm×40 mm were cast and tested as per EN: 196 to evaluate the flexural strength of the entire specimens. The three specimens for each mix are tested and average of three specimens is reported as the flexural strength of the sample.

3. Results and discussion

Designation	Cement	Sand/Cement ratio (2 mm-1 mm)	Sand/Cement ratio (1 mm-500 µm)	Sand/Cement ratio (500 µm-90 µm)	Total sand/ cement ratio	Superplasticizer (%)	Water/ Binder
TM-1	1	0.46	0.46	0.46	1.38	0.50	0.25
TM-2	1	0.46	0.46	0.46	1.38	1.5	0.25
TM-3	1	0.46	0.46	0.46	1.38	2.53	0.25
TM-4	1	0.46	0.46	0.46	1.38	0.50	0.225
TM-5	1	0.46	0.46	0.46	1.38	1.5	0.225
TM-6	1	0.46	0.46	0.46	1.38	2.53	0.225
TM-7	1	0.35	0.35	0.35	1.05	0.50	0.172
TM-8	1	0.35	0.35	0.35	1.05	1.5	0.172
TM-9	1	0.35	0.35	0.35	1.05	2.53	0.172

Table 2 Detail of various mixes for UHPC formation (Phase I)

Table 3 Detail of various mixes, prepared with pozzolanic material for the UHPC formation (Phase II)

Designation	Cement	Ennore Sand	Silica Fume	Quartz Powder	Fly Ash	Metakaolin	Superplasticizer (%)	Water/ Binder
UHPC-1	1	1.05	0.25	0.1	-	0.15	2.53	0.172
UHPC-2	1	1.05	0.25	-	0.1	0.15	2.53	0.172

Table 4 Detail of selected mix prepared for UHP-HFRC with hybrid fiber (Phase III)

Designation	Cement	Ennore Sand	Silica Fume	Quartz Powder	Fly Ash	Metakaolin	Crimped Fiber + Hooked Fiber (%)	Superplasticizer (%)	Water/ Binder
UHP-HFRC1	1	1.05	0.25	0.1	-	0.15	1+1.5	2.53	0.172
UHPC- HFRC2	1	1.05	0.25	-	0.1	0.15	1+1.5	2.53	0.172

3.1 Flow behavior of UHPC

The flow behavior of each phase has been observed during the experiment. The compatibility between the flow value and compressive strength is evaluated in Phase I mix. In phase I, nine concrete mixes TM-I to TM-9, as described in Table 2, were tested for flow using Hagerman's minislump cone of size 6×7×10 cm (see Fig. 3). The range of flow value i.e., 130 mm-180 mm is selected to develop further the phase I mix to UHP-HFRC. Subsequently, to find the compressive strength of the phase I mix, cubes size of 70.5×70.5×70.5 mm were cast and tested as per C1856. From the test results, it can be observed that the flow value of TM-1, TM-2 and TM-3 increase up to 146% with the increase of superplasticizer content from 0.50 to 2.53%. The flow value of TM-4, TM-5 and TM-6 shows the 98.63% improvement in flow value with the increase of superplasticizer content from 0.50 to 2.53%. Whereas, the flow value of TM-7, TM-8 and TM-9 shows the improvement in flow value by 90.78% for increased superplasticizer content from 0.50 to 2.53%. It has been observed that the TM-6 and TM-9 are the mixes which have flow value in the desired range (130-180 mm) and compressive strength 70.31 and 76.00 MPa, respectively. The equal flow value of TM-6 and TM-9 and higher compressive strength of TM-9 can be attributed due to the lower sand/cement ratio in TM-9. The lower amount of sand in TM-9 requires less amount of water to maintain the flow value even at lower water/binder ratio. Also, the surface area of sand particle has been reduced resultant in the efficient coating of cement particle on the surface of sand. Therefore, the higher compressive strength of TM9 is observed. So, TM-9 is considered for the next phase of studies (see Fig. 4). The flow behavior of TM-1 to TM-9 mix and obtained compressive strength test results are presented in Fig. 3 and Table 5 respectively.

In Phase II, the effect of the addition of finer materials such as silica fume, quartz, and metakaolin in mix TM-9, on the flow value and compressive strength is studied. The addition of finer material (silica fume, quartz, and metakaolin) is 50% of cement to prepare the UHPC recipe. The addition of fly ash in UHPC-2 improved the flow as compared to quartz powder contained UHPC (see Fig. 5). The glassy spherical shape of fly ash observed through the SEM images, reduced the inter-particle friction as compared to the quartz powder contained UHPC mix (see Fig. 1) (Kondraivendhan and Bhattacharjee 2015, Joshi and Nagaraj 1990).

In Phase II, the UHPC-2 exhibited the maximum flow values as well as the higher compressive strength. Further, the hybrid fibers were added in UHPC-1 and UHPC-2. In the phase III study, the higher reduction of 34.66% and 25.92% was observed in flow value of hybrid fiber mix UHP-HFRC-1 and UHP-HFRC-2 (see Table 6). The increment of fiber dosage significantly affects the flowability of the UHPC. The previously reported effect of fiber on the workability of the matrix is mainly due to the following reasons (Grünewald 2004, Markovic 2006): (1) the shape of the fibers is more elongated compared to aggregates, so the surface area at the same volume is higher; (2) stiff fiber change the structure of the granular skeleton, while flexible fiber fill the space between aggregates, which both can increase the porosity of concrete; (3) surface characteristics of fibers differ from



Fig. 3 Flow Table behavior of Phase I, II and III specimens

Table 5 Flow value and compressive strength of trial mixes (Phase I)

Designation	Flow Value	Compressive Strength			
Designation	(mm)	7 days	28 days		
TM-1	77	59.39	63.21		
TM-2	120	53.927	62.23		
TM-3	190	53.724	63.12		
TM-4	73	71.33	73.67		
TM-5	110	53.77	72.34		
TM-6	145	50.76	70.31		
TM-7	76	54.76	65.66		
TM-8	115	50.37	63.4		
TM-9	145	54.30	76		



that of cement and aggregates. The flow behavior of Phase I (Trial mix), Phase -II (UHPC) and Phase III (UHP-HFRC) mixes are shown in Fig. 3.

4 Mechanical and microstructural testing

4.1 Compressive strength

Table 6 Flow value of SCM contained UHPC formation (Phase II) and, hybrid fiber contained UHP-HFRC (Phase III)

Phase	Ingredients	Flow value (mm)
II	UHPC-1	150
II	UHPC-2	162
III	UHP-HFRC-1	98
Ш	UHP-HFRC-2	120



Fig. 5 Flow value of Phase II and Phase III mix



Fig. 6 Compressive strength result of selected UHPC/UHP-FRC/UHP-HFRC mix

Figs. 6-8 shows the compressive strength and axial stress-strain behavior of best performed sample results from each phase. The additions of finer size SCM i.e., fly ash, silica fume and metakaolin in a mix TM-9 improves the 90 days compressive strength by 20.88%. Whereas the additions of quartz powder mix i.e., UHPC-1 (Phase II) improves the compressive strength by 14.68% at 90 days as compare to mix TM-9. Due to the very low reactivity of quartz powder, the improvement in strength of quartz powder mix UHPC is lower than the fly ash mix UHPC (Mosaberpanah and Eren 2013). The filler effect and pozzolanic reaction of fly ash in UHPC-2 (Phase II) enable the matrix to gain the compressive strength at the age of 90 days. In initial curing days the fly ash particles act as filler and later provide the additional nucleation sites on the surface for hydrates from the cement (called seeding effect). Therefore, the fly ash improves the denseness of the matrix by filler effect and pozzolanic activity (Descher et al. 2012,



Fig. 7 Axial stress strain behavior of phase I and phase II sample



Fig. 8 Axial stress strain behavior of phase III sample

Rahhal and Talero 2004, Baert et al. 2008, Fraay et al. 1989). In addition, the usage of fly ash with silica fume improves the mechanical properties of UHPC matrix (Zang et al. 2017). The axial strain i.e., 0.00357 has been observed in UHPC-2 (Phase II) which is maximum ultimate strain in without fiber category samples. However, brittle failure is observed in sample TM-9, and UHPC-2 (Phase II) at the ultimate failure stage. The rapid loss of strength has been observed after the peak stress attainment of the specimen consequently affects the post peak performance (see Figs. 7-8). The ductile post peak performance is significant for the field application of high strength concrete. To achieve the high strength and higher ductility the steel fiber in hybrid state has been added in the UHPC matrix. It has been observed that the usage of hvbrid fiber in UHP-HFRC-2 improves the compressive strength by 58.22% at the age of 90 days as compared to TM-9. The usage of fine size supplementary material develops the dense matrix which produces the dense bond interaction with hybrid fiber. The presence of the fiber small aspect ratio control the crack at early stages whereas, the higher aspect ratio steel fiber hold the large crack at the ultimate stage of failure (Grünewald 2004, Yu et al. 2015). Therefore, the UHP-HFRC-2 exhibits the ultimate axial strain of 0.0089 and 0.0139 at the age of



Fig. 9 Compressive strength testing of 28 days cured UHP-HFRC- 2 sample (Phase- III)

Table 7 Flexural strength of trail mixes, UHPC and UHP-HFRC mix

Sr. No	Particulars	28 days flexural strength (MPa)	90 days flexural strength (MPa)
II	TM-9 (Phase I)	6.94	7.8
II	UHPC-2 (Phase II)	8.2	8.35
III	UHP-HFRC-2 (Phase III)	12.68	19.36

28 and 90 days which shows the excellent improvement in axial stress strain post peak behavior compared with TM-9 and UHPC-2 (Phase II).The property of the hybrid fiber matrix bond to resist the width of crack opening led to absorbing the higher energy of UHP-HFRC-2 as compared to control samples. Based on the obtained axial stress strain behavior of all the samples, the sequence of sample in terms of excellent strain is UHP-HFRC-2>UHPC-2>TM-9. In addition, it can also be seen from Fig. 9 that the spalling of concrete is completely controlled and no brittle failure is obtained with the usage of hybrid fiber. This property enables the UHP-HFRC to attain the structure serviceability criteria.

4.2 Flexural strength

The flexural strength of the specimen has been tested under three point bending test as per EN 196-1 (1995). The TM-9 exhibit the flexural strength of 6.94 MPa and 7.8 MPa at the age of 28 and 90 days. The 18.15% and 7.05% improvement has been observed with the addition of supplementary cementitious material i.e., UHPC-2, Phase II at the age of 28 and 90 days respectively (see Table 7). The TM-9 and UHPC-2 exhibited linear pre peak behavior whereas brittle failure has been observed in post peak region (see Fig. 10(a)).

The excellent improvement in flexural strength has been observed in UHP-HFRC-2 (Phase III). The UHP-HFRC-2 exhibit 12.68 MPa and 19.36 MPa at the age of 28 and 90 days respectively. Also, the significant difference has been observed in post peak region between the plain UHPC samples (TM-9, UHPC-2) and hybrid fibers containing samples (UHP-HFRC-2, Phase III). The hybrid fibers containing sample (UHP-HFRC-2) exhibit the higher load carrying capacity then TM-9 and UHPC-2 (Phase II). The



Fig. 10 (a) Load deformation behavior of trial mixes and UHPC



Fig. 10 (b) Load deformation behavior of UHP-HFRC-2 (Phase III) mix

presence of micro fiber and macro fiber improves the preelastic and post-elastic performance of the specimen (Rossi *et al.* 1987). The presence of micro fiber retard the opening of micro crack hence improves the pre peak load deformation behavior. In addition, the delay in opening of macro cracks because of the presence of macro fibers improves the post-elastic deformation characteristics of the UHP-HFRC-2 consequently, imparts the ductility in the specimen. In addition the large amount of energy absorption has been observed in post peak region of UHP-HFRC-2 specimen (see Fig. 10(b)).

4.3 SEM/EDX

To study the fiber matrix interaction of fly ash mix UHP-HFRC and quartz powder mix UHP-HFRC, the images of scanning electron microscopy and elemental analysis by energy dispersive X-ray spectroscopy are compared. Fig. 11 depicts the interface between steel fiber and paste of UHP-HFRC-2 (Phase III). It can be concluded from Fig. 11 (a)-(d) that the dense interface is formed between the steel fiber and paste. The dense CSH formation leads to develop the bond between the steel fiber and paste. Also, the dense interface helps the matrix to sustained the



Fig. 11 Morphology Evaluation of UHP-HFRC-2 (Phase III)



Fig. 12 EDS of UHP-HFRC-2 (Phase III)



Fig. 13 Morphology evaluation of UHP-HFRC-1 (Phase III)

applied load for the higher stress and simultaneously utilize the mechanical property of fiber. The presence of fine pozzolanic material and low water-cement ratio develop a higher amount of CSH formation and lower amount of calcium hydroxide. However, the presence of voids is also observed in the UHP-HFRC paste which can be stated as the matrix can be improved further by optimizing the ingredient of UHP-HFRC. The EDS analysis of UHP-HFRC-2 (Phase III) depicts that the calcium is in higher amount followed by the silicon dioxide (see Fig. 12). The additional calcium provided by the SCM to the matrix enhances the compound formation consequently, higher strength is obtained.

It can be observed that the use of quartz powder in UHP-HFRC formation revealed the dense matrix (see Fig 13 (a)-(d)). However, the hairline crack is also shown in the



Fig.14 EDS analysis of UHP-HFRC-1 (Phase III)



rig. 15 AKD of OHF-HFKC-1 (Flase III)

paste. In Fig. 13 (b)-(c), the presence of the gap at interface attributed to the weak bond between the steel fiber and the matrix. The presence of quartz in UHP-HFRC matrix developed the dense CSH in a paste but unable to form the dense interface with fiber. These parameters responsible for the weak performance of UHP-HFRC prepared with quartz powder. The EDS of UHP-HFRC prepared with quartz powder shows the higher peak of silica oxide and less content of calcium oxide as compare to UHP-HFRC prepared with fly ash (see Fig. 14).

4.4 XRD analysis

The XRD study was conducted for selected sample based on the obtained higher compressive strength UHPC samples (quartz powder based UHP-HFRC and fly based UHP-HFRC). The UHP-HFRC-1 shows the maximum peak of quartz in the mix (Fig. 15). The presence of a higher amount of quartz in the diffraction pattern reflects the presence of the unreacted particle, consequently these particle acts as filler in the matrix. It can be concluded that the presence of filler particle decreases the void space in the matrix however do not significantly participates in nucleation. The product of hydration of the matrix can be confirmed by the presence of CSH and portlandite. The presence of ettringite in UHP-HFRC-1 is also observed. The higher presence of unreacted quartz particle revealed that the matrix of UHP-HFRC is dense, however, the formation of hydration products are less. Consequently, the weak fiber matrix interface bond is observed in SEM observation.

The XRD diffraction pattern of fly ash based UHP-



Fig. 17 DSC curve of UHP-HFRC paste after 56 days of hydration

HFRC (UHP-HFRC-2) is shown in Fig. 16. The majority of crystalline phases of silica and calcium silicate hydrate are observed. The small amount of portlandite, calcium silicate, calcium sulphate, anad tricalcium aluminate is observed. The fly ash contains a significant amount of silica and lime, therefore, the peak of CSH and silica is observed. The peak of CSH shows the presence of a stable hydration product in the matrix, whereas silica reflects the partly unreacted particle of fly ash. The formation of the cementitious compound can also be confirmed by portlandite also.

4.5 Thermal property analysis of UHP-HFRC

The DSC and TGA curves of 90 days cured UHP-HFRC-1 and UHP-HFRC-2 (Phase III) are illustrated in Figs. 17 and 18. It can be concluded from the DSC curve that the peak exists in the vicinity of 100° C, 400° C, 650° C, and 850° C. The evaporation of water takes place at the 100° C. The 30-105 °C is the range of where the evaporable water and part of the bound water escapes and generally at 120° C evaporable water is completely eliminated (Alarcon-Ruiz *et al.* 2005, Noumowe 1995).



Fig. 18 TG curves of UHP-HFRC paste after 56 days of hydration

The range of 450° C- 550° C dehydroxilation of the portlandite (Noumow 1995). In the vicinity of 450° C, the dehydroxylation of the portlandite was observed. The presence of portlandite in both the UHP-HFRC samples ware confirmed by XRD analysis. The decarbonation of calcium carbonate lies in the range of 700° C- 900° C (Alarcon-Ruiz *et al.* 2005, Noumowe 1995, Bellew 1996). The UHP-HFRC sample 1 and 2 both have shown a peak at the temperature of 850° C (see Fig. 17).

The Fig. 18 shows a mass loss of UHP-HFRC-1 and 2 of phase III sample. The pattern of mass loss for both the specimen is similar, however, the amount of mass loss is different. This could be attributed due to the reacted substance in UHP-HFRC which reacts differently at a different temperature. The significant mass loss is observed at the temperature of 450 °C and 700 °C which is attributed to the decomposition of portlandite and calcium carbonate.

5. Conclusions

The UHP-HFRC is developed through various stages in this experimental study. The SCM, fine materials and fibers are essentially required to develop the ultra high strength. The following conclusions are drawn from the experimental study.

• The low sand/cement ratio is favorable to produce the UHPC. The presence of fly ash and quartz powder improved the strength property of the matrix. However, the comparison between fly ash and quartz powder shows that the fly ash is a better ingredient to produce the UHPC.

• The presence of hybrid fiber in dense matrix improved the strength significantly. The presence of small-scale and long scale fibers sustained the load for a longer time and impart the crack bridging phenomenon.

• The fly ash imparts the significant contribution in developing the densest microstructure then quartz powder. The SEM image shows the good fiber matrix interaction in fly ash based UHP-HFRC then quartz powder contained UHP-HFRC.

• The presence of high peaks of CSH in fly ash

contained UHP-HFRC matrix depicts the formation of the dense matrix. The number of peaks of silica in quartz powder UHP-HFRC matrix shows the unreacted particle which acts as a filler particle in the mix. It is due to the usage of higher amount of finer material in developing the UHP-HFRC and the low amount of water/binder ratio. The high temperature behavior of fly ash contained UHPC and quartz powder contained UHPC show the similar mass loss pattern, however, the amount of mass loss was different.

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