Investigating the use of wollastonite micro fiber in yielding SCC

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Abstract. Self compacting concrete (SCC) has good flowability, passability and segregation resistance because of voluminous cementitious material & high coarse aggregate to fine aggregate ratio, and high free water availability. But these factors make it highly susceptible to shrinkage. Fibers are known to reduce shrinkage in concrete mixes. Until now for conserving cement, only pozzolanic materials are admixed in concrete to yield a SCC. Hence, this study compares the use of wollastonite micro fiber (WMF), a cheap pozzolanic easily processed raw mineral fiber, and flyash in yielding economical SCC for rigid pavement. Microsilica was used as a complimentary material with both admixtures. Since WMF has large surface area (827 m²/kg), is acicular in nature; therefore its use in yielding SCC was dubious. Binary and ternary mixes were constituted for WMF and flyash, respectively. Paste mixes were tested for compatibility with superplasticizer and trials were performed on a normal concrete mix of flexural strength 4.5 MPa to yield SCC. Flexural strength test and restrained shrinkage test were performed on those mixes, which qualified self compacting criteria. Results revealed that WMF admixed pastes have high water demand, and comparable setting times to flyash mixes. Workability tests showed that 20% WMF with microsilica (5-7.5%) is efficient enough in achieving SCC and higher flexural strength than normal concrete at 90 days. Also, stress rate due to shrinkage was lesser and time duration for final strain was higher in WMF admixed SCC which encourages its use in yielding a SCC than pozzolanic materials.

Keywords: self compacting concrete; wollastonite micro fiber; admixtures; compatibility; restrained shrinkage

1. Introduction

Cement consists of lime (CaO), silica (SiO₂), alumina (Al₂O₃) and iron oxide (Fe₂O₃). They diffuse together to form tri calcium silicate (C₃S), di calcium silicate (C₂S) and tri calcium aluminate (C₃A), each having different rate of hydration and strength development. Thus, it is the percentage composition of these compounds which determine the strength and durability of cement composites. Hydration process could be changed by the introduction of admixtures containing lime, silicates and aluminates. The rate of reaction of these admixtures depends upon their surface area as well as their crystal structure, which could be glassy, amorphous or

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crystalline. The active phase consists of glassy and amorphous state. The use of flyash, microsilica and slag reduces pollution apart from improving the strength and durability. The improvement in workability by flyash is ascribed to its spherical shaped particles as well as the negative charge carried by it. The positively charged calcium ions from the alkaline solution get adsorbed over these particles and deflocculate the cement particles. This liberates entrapped water between the cement particles. Microsilica being small and having negative charge does the same job, but it also adsorbs large amount of water over its high surface area particles, and causes higher hydration rates thereby increasing the water demand (Langan *et al.* 2002). Superplasticizers containing negatively charged ions also reduce water demand in similar fashion, but the C₃A in the mix gets fixed over the negatively charged ions and neutralize the superplasticizer. Thus the ratio of aluminates and their crystal structure present in the admixtures also affect the compatibility of cement mix with the superplasticizer. Even if an admixture does not carry aluminates, the positive charge carried by it neutralizes the negatively charged superplaticizer's ions after the adsorption of calcium ions, thereby making its dosage insufficient for the cement paste mix (Drazan and Zelic 2006).

Bouzoubaa and Lachemi (2001) investigated the feasibility of producing SCC with high volumes of Class F flyash (40-60%) and found that economical medium strength SCC could be made by substituting cement with high volumes of flyash. Jalal et al. (2015) also emphasized their use stating the improvement in rheological and mechanical properties of flyash and nanoparticle admixed self compacting concrete. Kostrzanowska-Siedlarz and Gołaszewski (2016) stressed that Water: binder ratio and paste content (volume) are important parameters in yielding a high performance concrete. Lomboy et al. (2011) investigated the differences in the strength and shrinkage properties of semi flowable self-compacting concrete (SFSCC) and ordinary pavement concrete containing 20% Class F flyash. Compressive strength, splitting tensile strength, modulus of elasticity and fracture strength of both the concretes were evaluated at 1,3,7,14 and 28 days and were found to have negligible differences except modulus of elasticity, which got reduced for SFSCC. Also shrinkage induced cracking was found higher in SCC. Altoubat et al. (2017) introduced flyash in SCC to study restrained shrinkage behavior. It was found that 35% of flyash highly controlled the shrinkage whereas 50% cement substitution with flyash could be achieved with respect to control concrete. SCC mixtures shrink on water loss either due to surface evaporation or temperature gradient due to hydration (temperature difference in top and middle layers of mixes). Therefore fiber addition is sought in SCC mixes. Hossain et al. (2012) studied the influences of fiber types/size/dosages and fiber combinations on workability and rheological properties of SCC. It was found that the maximum dosage of Polyvinyl Alcohol (PAV) is limited to 0.125% compared to 0.3% of metallic ones in developed FRSCC mixtures due to PVA's higher workability reduction/viscosity enhancing capability. Study indicated that a homogenous fiber distribution can be obtained up to critical fiber content and once that is surpassed, a stiff structure of the granular skeleton made self compaction impossible as has been proved in the study by Grunewald & Walraven (2001). It is important for FRSCC to have optimal viscosity to ensure required flowability and to prevent segregation. Fathi et al. (2017) also testified that fibers reduce the slump and compressive strength of SCC but increase its flexural tensile strength. Talking about mechanical properties, Ahmad et al. (2017) compared the mechanical properties of a normal concrete (NC), SCC and glass fiber reinforced SCC. It was clarified that compressive & tensile strength increases in both types of SCC mixes, but modulus of rupture & elasticity are comparable for fiber reinforced SCC and NC, whereas these are lowest for simple SCC.

Wollastonite is a natural, inert, acicular, white silicate mineral of high modulus of elasticity and

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occurs in contact metamorphic zones in some schists and gneisses and limestone in volcanic rocks. It is abundantly available in the mines of Khera Tarla and Belka Pahar in Udaipur of Rajasthan State. Its cost component is constituted mainly of packing, grounding into fiber, and transportation charges which vary from 2-4 INR/Kg. Ransinchung et al. (2010) studied the effect on strength and durability of cement composites admixed with wollastonite micro fibers (WMF) and microsilica. It was found that WMF and microsilica together increased the flexural strength of cement mortar and reduced its water sorptivity; that too at higher cement substitution rates (greater than 20%). Thus cement substitution with WMF is beneficial in yielding SCC. Wollastonite has high modulus of elasticity (Low and Beaudoin 1992) and is thus anticipated to improve the strength of the SCC, but its effect on workability of SCC due to combined contribution of its acicular nature, high surface area and small particle size is not known. Its compatibility with superplasticizer is further not known. Furthermore, the effect on shrinkage of concrete is also not apparent as shrinkage depends upon both hydration rate and particle size of an admixture. Hence this study aims to find out whether a self compacting concrete could be made out by substituting cement at higher dosages with WMF, and if yes, then what would be the effect on all the desired properties of concrete, because WMF is both a frictional micro fiber and a pozzolan. Substituting cement in high volume is essential as this will reduce the cost of concrete and save the environment from pollution, caused by the manufacture of cement.

2. Materials and methods

2.1 Materials used

In this study Ordinary Portland cement (OPC) 43 grade conforming to Indian standard code IS: 8112 (2013) was used since IRC 44 (2008) recommends its use to mitigate shrinkage, which is nevertheless still present due to unavoidable curing conditions. Graded river sand conforming to Zone-II having fineness modulus of 3.23 and specific gravity of 2.58 was used as fine aggregate. Crushed graded coarse aggregates of 20 mm and 10 mm conforming to IS 383 (1970) and having specific gravity of 2.62 and 2.58 respectively were used in the proportion of 60:40 in normal concrete mix. Fine crystalline wollastonite powder supplied by Wolkem India Limited and Densified 920D grade microsilica supplied by KGR Agrofusions Pvt. Limited were used as cementitious materials and their specific surface areas were 827m²/kg and 18000 m²/kg. Specific gravity values of 2.9 and 2.05 were recorded for WMF and microsilica respectively. In addition, medium lime Class F Flyash as per ASTM C 618 (1985), obtained from National Thermal Power Plant Ghaziabad, India was also incorporated. Its specific surface area and specific gravity values were 350 m²/kg and 2.52 respectively. High water reducing poly carboxylate ether (PCE) based superplasticizer was used to enhance workability of concrete mixes.

Particle size analysis study was performed for WMF, cement, fly ash and microsilica using Ankersmid laser based analyzer and the results are shown in Table 1 and Fig. 1. Specific surface area of cement was found by using Blain's method, and that of WMF, flyash and microsilica were found using BET's surface area analyzer. Chemical compositions of WMF, flyash and microsilica were analyzed by X-Ray fluorescence spectrometer in accordance with Indian standard IS: 12803 (1989) and the same is presented in Table 2.



Fig. 1 Particle size distribution of WMF, flyash cement and microsilica

Table 1 Cumulative percentage sizes of various particles

All sizes in micron	Microsilica	Cement	Flyash	WMF
Median Size	0.087	17.093	9.639	1.181
Mean Size	0.145	20.055	25.695	1.830
D10	0.029	0.843	3.056	0.409
D45	0.089	17.093	8.661	1.064
D90	0.284	35.298	63.146	2.723

Table 2 Chemical properties of cementitious materials including OPC

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Compound	Cement	Flyash	WMF	Microsilica
SiO ₂	20.2	35	48	92.9
Al_2O_3	5.2	26	1.4	0.9
Fe_2O_3	3	8.7	0.6	0.72
MgO	1.51	5	0.2	0.57
SO_3	2.2	3	-	0.16
Na ₂ O	0.08	1.5	-	0.32
Chloride	0.014	0.005	-	0.037
Loss on ignition	4.3	5	4	2.6
CaO	62.9	15.3	45.9	1.4
K_2O	0.6	0.5	-	0.4

2.2 Mixture proportions

The mixes for paste and concrete were prepared by substituting cement with WMF/flyash in major quantities, and with microsilica comparably in smaller quantities. Binary mixes have been designated as CW1-CW3 for three percentage substitution 10%, 20% and 30% with WMF, likewise for flyash as CF1-CF3. Keeping similar substitution level of WMF and flyash, microsilica was used in levels of 2.5-10%, thus making twelve more mixes, each for WMF and flyash, respectively. Table 3 shows the various mixes, their designations, and the percentage of each material in the mix. Table 4 shows the mix proportions of normal concrete mix having design flexural strength of 4.5 MPa prepared in accordance with the specifications laid down in IRC 44

(2008) as the present study emphasized on obtaining flowable pavement quality concrete (PQC). Subsequent trials for obtaining self-compacting concrete mixes have been made by sequentially performing the following steps if needed: substituting the binder material, correspondingly decreasing the coarse aggregate to fine aggregate content ratio of the normal concrete mix and

Mir Designation	Percentage of material in mix				Paste Composition (%age)
Mix Designationn	Cementt	Flyash Wollastonite micro fiber		Microsilicaa	C+F/WMF+MS
С	100	-	-	-	100
W	-		100	-	100
F	-	100	-	-	100
MS	-	-	-	100	100
CW1	90	-	10	-	90+10
CW2	80	-	20	-	80+20
CW3	70	-	30	-	70+30
CWS1	87.5	-	10	2.5	87.5+10+2.5
CWS2	85	-	10	5	85+10+5
CWS3	82.5	-	10	7.5	82.5+10+7.5
CWS4	80	-	10	10	80+10+10
CWS5	77.5	-	20	2.5	77.5+20+2.5
CWS6	75	-	20	5	75+20+5
CWS7	72.5	-	20	7.5	72.5+20+7.5
CWS8	70	-	20	10	70+20+10
CWS9	67.5	-	30	2.5	67.5+30+2.5
CWS10	65	-	30	5	65+30+5
CWS11	62.5	-	30	7.5	62.5+30+7.5
CWS12	60	-	30	10	60+30+10
CF1	90	10	-	-	90+10
CF2	80	20	-	-	80+20
CF3	70	30	-	-	70+30
CFS1	87.5	10	-	2.5	87.5+10+2.5
CFS2	85	10	-	5	85+10+5
CFS3	82.5	10	-	7.5	82.5+10+7.5
CFS4	80	10	-	10	80+10+10
CFS5	77.5	20	-	2.5	77.5+20+2.5
CFS6	75	20	-	5	75+20+5
CFS7	72.5	20	-	7.5	72.5+20+7.5
CFS8	70	20	-	10	70+20+10
CFS9	67.5	30	-	2.5	67.5+30+2.5
CFS10	65	30	-	5	65+30+5
CFS11	62.5	30	-	7.5	62.5+30+7.5
CFS12	60	30	-	10	60+30+10

Table 3 Mixes chosen for the tests and their material composition

Mix design for Normal M-40 concrete used to derive SCC for rehabilitation as per IRC 44							
Cement	Sand	Coarse aggregates	Water	Nominal MSA=16mm			
450 Kg	711 Kg	1057 Kg	165 Kg	Superplasticizer 0.20/ 1.221t/au m			
1	1.58	2.35	0.36	Superplasticizer 0.5% ~1.250/cu ili.			
		20mm 10mm		CA:FA=60:40			
		740 Kg 317 Kg		C.A. 20mm:10mm=70:30			

Table 4 Mix design of normal PQC

Assuming that 20% cement reduction takes place on addition of superplasticizer



Fig. 2 Sequential steps in yielding a SCC from a normal concrete

addition of superplasticizer for each changed ratio. All three parameters i.e. binder composition, fine aggregate to coarse aggregate ratio (FA:CA) and water to cementitious material ratio are required to be changed to increase the flowability and passability of trial concrete. But the important thing is the order in which they have to be changed. Superplasticizer could not be introduced abundantly to normal concrete initially, or FA: CA can't be changed without knowing the volumetric change in binder content.

Viscosity enhancing admixture needs to be added in the binder material in order to balance the decrease in segregation resistance caused by the change in other two parameters, otherwise the concrete will segregate, and will leave only water separated from other constituents. Trials indicate that the increase in FA:CA is proportional to the volumetric increase in binder material. Increasing binder volume without FA:CA increase causes lesser passability, whereas if FA:CA is increased for a constant binder composition, then a segregated mix is obtained having cement laitance. Increasing water to cementitious material ratio by superplasticizer addition increases both flowability and passability, but it imposes a high danger of segregation (coarse aggregates leave the mortar). Fig. 2 shows the sequence of obtaining a SCC from a normal concrete.

2.3 Study on paste and concrete mixes



Fig. 3 Workability test for (a) flowability, (b) passability and (c) segregation resistance

Compatibility test for cement slurries was conducted adopting Marsh cone test concept in accordance with ASTM D 6910 (2009). This test enables to ascertain the optimum dosage of a superplasticizer; marked by that dosage beyond which there is no change in flow time of cement slurry. The study was performed for 0.3, 0.35 and 0.4 Water/Cement ratios by weight. Here the cement ratio has been taken instead of cementitious materials in order to avail equal amount of water for a certain amount of cement in all mixes, so as to keep the study of this test in line with the trial method used for yielding SCC mixes from normal concrete mix. For fresh concrete mixes, workability tests like Abram's flow test, V funnel test, J ring test and Probe ring test were performed and checked according to EFNARC guidelines (2005). Fig. 3 shows the workability tests performed in the lab to obtain SCC from a normal concrete. In order to assess the compressive & flexural strength of the qualified mixes cubes (15×15×15 cu. cm.) & beams $(10 \times 10 \times 50 \text{ cu. cm.})$, respectively were prepared and tested in accordance with IS:516 (1959) after 30, 60 and 90 days of curing. Restrained shrinkage test was performed as per ASTM C1581 using a 50 mm thick concrete specimen, 150 mm in height, cast around a steel ring of 25 mm thickness and 300 mm outside diameter. Altoubat et al. (2017) have performed similar test on flyash admixed SCC. The compressive strain developed in the steel ring caused by shrinkage of the mortar or concrete specimen was measured from the time of casting. Cracking of the test specimen is indicated by a sudden decrease in the steel ring's strain. The age at cracking and the rate of tensile stress development in the test specimen are indicators of the material's resistance to cracking under restrained shrinkage. Strain gauges were used to measure the strain development in the inner steel ring at an interval of not more than 30 minutes.

3. Results

3.1 Results for binder materials

Fig. 1 clearly illustrates that microsilica is finest among all considered powdery materials followed by WMF, fly ash and cement respectively. Microsilica has particle size ranging from 0.01-0.5 microns and about 80 percent of WMF particles are in the range of 0.5-4.47 microns. Nearly 80 percent of OPC and flyash particles are larger than 4.47 microns. The peak of WMF lies exactly in between the peaks of microsilica and OPC. This interpretation clearly infers that WMF used was median size to both microsilica and OPC, and hence, fair degree of interlocking within the fine matrix has taken place.

BET's study results on surface area show that microsilica, WMF and fly ash are finer than OPC in the order of 60, 2.8 and 1.3 times, respectively. X Ray fluorescence spectrometer test indicates that fly ash used in the present study contains appreciable amount of silica and highest amount of alumina when compared to the rest of the materials. Microsilica mainly consists of silica and rest other oxides are in meagre amount. WMF has equal proportions of lime and silica, which together constitute more than 90% of its mineral content.

3.2 Results for pastes

It was found, that whether PCE based superplasticizer is mixed initially or after mixing of water, there is no comparative difference in the workability of fresh concrete, which suggests that cement contains good ratio of aluminates and sulphates in it. This finding was substantiated by the X-Ray fluorescence test, which gives the amount of aluminates and sulphates in the cement as 5.2 and 2.2 percent respectively. Flyash introduction, which contains 26 percent aluminates and 3% sulphates, also did not alter the superplasticizer behaviour. This suggests that the aluminates present in flyash are not highly reactive and have a higher inert crystalline content.

3.2.1 Compatibility at W/C=0.3

The water content is low and therefore the activity of microsilica and WMF is retarded. This retardation is not similar to the retardation in hydration caused by the admixtures at dormant period; it is related to time period just after the addition of water. This retardation effect is lower for lower contents of these admixtures and gets amplified at their larger contents.

Flyash mixes have normal consistency in this range because of the workability created by the repulsive forces of adsorbed particles of flyash carrying negatively charged sulphate ions. These ions repel the cement particles far apart, thus releasing more water. Therefore no retardation was observed in the effects of flyash and it kept reducing the water demand at its increased content by filling the voids in cement particles.

As far as increment in superplasticizer is concerned, it has been observed that fine admixtures start behaving normally with an increment in effective water content caused by superplasticizer. These admixtures easily infiltrate into the voids between cement particles and get adsorbed over their surfaces thereby repelling them and causing more increase in flow. Thus, for WMF and microsilica, flow time reduced up to 20% WMF and 5% microsilica content and thereafter it increased due to the water demand created by excess WMF and microsilica, as their further adsorption was not possible on cement particles beyond that content. No behaviour change was observed in the flyash mixes with an increment in superplasticizer because the behaviour of flyash did not vary much with a small increment in the effective water content of mix.

3.2.2 Compatibility at W/C=0.35

The water content is high for flyash admixed pastes, whereas it approaches normal consistency for pastes containing lower contents of WMF and microsilica. Hence, at medium W/C ratios, flyash admixed pastes were observed to increase flow up to 20% content and then it remained constant. WMF and microsilica acceleration effect was enhanced at this stage. At higher content, WMF and microsilica were found to increase the flow time. Obviously, this increment in flow time was lower in comparison to that with W/C ratio (0.3%). Hence, for pastes having higher content of WMF and microsilica, the W/C ratio of 0.35 is low, and they behave as being retarded.

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Mix CA	CA	Abram's	V Funnel	V Funnel	J Ring	Probe ring	Super plasticizer
	CA. FΔ	flow (600-	time6-	after 5 min.	difference (0-	penetration (0-7	7)C.M.) % by weight
	IA	750) mm	12sec	(9-15 sec)	10 mm)	mm)	of cement
С	60:40	360	17	24	24	2	0.30
CW1	55:45	560	10	12	21	3	0.45
CW2	50:50	580	8	10	19	5	0.45
CW3	50:50	540	12	12	21	4	0.6
CWS1	55:45	580	9	11	15	3	0.45
CWS2	50:50	630	8	10	9	5	0.45
CWS3	50:50	620	9	10	10	4	0.45
CWS4	50:50	605	10	11	13	3	0.45
CWS5	50:50	646	7	11	11	5	0.45
CWS6	50:50	660	7	9	5	7	0.6
CWS7	50:50	620	8	11	8	5	0.6
CWS8	50:50	590	9	14	10	4	0.6
CWS9	50:50	570	10	12	14	5	0.6
CWS10	45:55	630	7	9	12	3	0.6
CWS11	45:55	575	9	13	17	2	0.45
CWS12	45:55	530	13	18	22	2	0.45
CF1	55:45	570	10	16	15	5	0.3
CF2	55:45	581	9	17	13	7	0.3
CF3	55:45	604	7	18	11	9	0.3
CFS1	55:45	585	9	16	12	5	0.45
CFS2	50:50	615	8	14	11	8	0.45
CFS3	50:50	600	9	10	15	6	0.45
CFS4	50:50	573	10	12	18	3	0.45
CFS5	50:50	625	7	9	9	7	0.45
CFS6	50:50	650	6	9	8	9	0.45
CFS7	50:50	618	8	9	10	6	0.45
CFS8	50:50	587	10	11	13	6	0.45
CFS9	50:50	633	6	15	11	10	0.45
CFS10	50:50	663	5	17	8	12	0.3
CFS11	45:55	623	7	17	13	9	0.3
CFS12	45:55	594	8	21	15	7	0.3

Table 5 Results obtained from the workability tests conducted on SCC in the fresh state as per EFNARC guidelines EFNARC (2005)

Bold text shows good workability parameters

3.2.3 Compatibility at W/C=0.4

The water content is high enough for flyash admixed pastes, whereas it approaches normal consistency for WMF and microsilica admixed pastes, especially those containing higher content of WMF and microsilica. Same effect for flyash was observed at this W/C ratio as was observed at 0.35% W/C. For WMF-microsilica mixes the effect of WMF and microsilica was unhindered up to

20% content of WMF and up to 5% content of microsilica, after which it registered a small decrement.

3.3 Results for fresh concrete

Table 5 summarizes the best possible design mix parameters obtained from trials performed for checking self compacting criteria of binary mixes and ternary mixes and their corresponding workability status. Following results have been drawn from the present investigation:

• By and large, it was observed that admixing of WMF led to reduction in flowability and passability of the mixes but segregation resistance increased with increment in WMF content.

• With microsilica addition at lower amount (0-5%), the flowability, passability and segregation resistance increased.

• Flyash at all contents improved the flowability and passability of mixes, but it decreased the segregation resistance of self compacting concrete.

3.4 Results for drying shrinkage resistance

This test corresponds to the behavior of concrete in the period before 28 days; literally the behavior of concrete in the first seven days has more prominent effect on the drying shrinkage of concrete. Typical concrete shrinkage has been measured at 520 to 780 millionths. Fig. 3 shows the



Fig. 3 Configuration of restrained SCC ring samples



Fig. 4 Compressive strain data with respect to square root of elapsed time for concrete mixes

		Initial	Age at	G ()	Avg.	F1 1.4	Stress	
Mix	Max.	age	max.	Strain rate (ε) at elapsed time T	strain	elapsed ti-me	rate	for
IVIIX	Strain (μ)	in	strain	$(\varepsilon = \alpha \times T/2 + K)$	rate $(\alpha) \times$	(<i>Tr</i> Davs)	(q)MPa at	cracking
	0.6	Days	(Days)		(10^{-6})	(=! = 0,j =)	$\frac{Tr}{2}$	
С	-96	0.24	12.31	Y=10.48X-58.35	10.48	12.06	0.109	Mod High
CWI	-70	0.03	18.85	Y = 6.8/2X - 3/.00	6.87	18.82	0.057	Mod Low
CW2	-55	0.25	16.81	Y= 4.836X-35.12	4.83	16.56	0.043	Mod Low
CW3	-96	0.24	23.69	Y= 9.069X-54.14	9.07	23.45	0.068	Mod Low
CWS1	-56	0.47	28.00	Y = 10.95X + 0.280	10.95	27.52	0.075	Low
CWS2	-64	0.34	19.54	Y = 5.266X - 42.20	5.27	19.19	0.043	Mod Low
CWS3	-68	0.34	22.51	Y= 9.956X-17.54	9.96	22.17	0.076	Mod Low
CWS4	-67	0.50	27.40	Y=11.13X-2.352	11.13	26.89	0.077	Low
CWS5	-72	0.26	18.78	Y= 4.316X-51.34	4.32	18.51	0.036	Mod Low
CWS6	-70	0.55	26.59	Y= 2.317X-56.68	2.32	26.03	0.016	Low
CWS7	-78	0.47	23.69	Y= 4.856X-51.09	4.86	23.21	0.036	Mod Low
CWS8	-76	0.27	20.50	Y= 5.390X-51.34	5.39	20.23	0.043	Mod Low
CWS9	-39	0.40	28.02	Y= 8.081X+6.033	8.08	27.63	0.056	Low
CWS10	-40	0.55	28.02	Y= 6.505X-3.328	6.51	27.47	0.045	Low
CWS11	-54	0.34	24.13	Y=10.69X-1.725	10.69	23.79	0.079	Mod Low
CWS12	-55	0.34	28.02	Y= 12.36X+3.933	12.36	27.68	0.085	Low
CF1	-120	0.32	14.44	Y=12.05X-68.02	12.05	14.13	0.116	Mod Low
CF2	-96	0.44	18.92	Y=15.07X-22.94	15.07	18.49	0.127	Mod Low
CF3	-113	0.42	16.01	Y=15.59X-52.78	15.59	15.59	0.143	Mod Low
CFS1	-117	0.37	19.17	Y=12.09X-58.50	12.09	18.80	0.101	Mod Low
CFS2	-116	0.45	20.53	Y=10.28X-66.31	10.28	20.08	0.083	Mod Low
CFS3	-119	0.29	16.64	Y=12.05X-69.14	12.05	16.35	0.108	Mod Low
CFS4	-116	0.27	15.04	Y=11.82X-67.93	11.82	14.77	0.111	Mod Low
CFS5	-84	0.55	28.02	Y= 16.22X+4.654	16.22	27.47	0.112	Mod Low
CFS6	-89	0.34	28.02	Y=16.03X-2.588	16.03	27.68	0.110	Mod Low
CFS7	-69	0.53	28.02	Y= 13.61X+12.02	13.61	27.49	0.094	Low
CFS8	-95	0.34	28.02	Y= 18.55X+5.899	18.55	27.68	0.127	Mod Low
CFS9	-96	0.50	28.02	Y= 16.42X+0.420	16.42	27.52	0.113	Mod Low
CFS10	-105	0.29	18.02	Y=11.88X-54.38	11.88	17.73	0.102	Mod Low
CFS11	-108	0.29	21.81	Y=16.85X-22.19	16.85	21.52	0.131	Mod Low
CFS12	-110	0.45	26.95	Y=18.88X-2.764	18.88	26.50	0.132	Mod Low

Table 6 Results obtained from the restrained shrinkage test

Bold text shows mixes with better properties

configuration of restrained concrete ring sample. Fig. 4 shows the compressive strain data with respect to square root of elapsed time for normal concrete and binary concrete mixes. Table 6 shows the cracking time and final stress rate for different mixes. While testing at a laboratory, shrinkage is measured by two parameters: stress rate due to shrinkage and time for crack development. Results from this study show that WMF mixes have the highest stress rate increment



Fig. 5 Compressive strength of normal and binary concrete mixes

capacity and its ability to enhance crack development time is followed by silica fume. Silica fume has maximum stress rate reduction capacity due to higher paste strength imparted by high rate of hydration. Flyash has lowest crack time increment capacity.

3.5 Results from compressive strength test

In concrete mixes the inclusion of fly ash led to a reduction in compressive strength whereas WMF improved the compressive strength up to 20% substitution levels. Incorporation of flyash continued to decrease the compressive strength up to 60 days. But the concrete cured beyond 60 days showed considerable improvement in compressive strength as can be seen from Fig. 5. The trend of improvement of compressive strength clearly depicted that prolonged curing is more beneficial for admixed concrete as far as strength improvement is concerned.

In WMF admixed concrete mixes the compressive strength increased with cement substitution by WMF. Irrespective of days of moist curing, concrete mix containing 20% WMF produced better compressive strength than rest of the combinations. If we look at the strength development pattern obtained at 60 days moist curing, concrete mixes containing 20% WMF and 30% WMF produced same compressive strength but at 90 days moist curing, lower compressive strength was offered by the mix containing 30% WMF. Such phenomenon was observed mainly due to slower rate of hydration of cementitious system on account of significant amount of cement content reduction and on other hand presence of WMF particles at the interface of aggregate more than necessary, as a result of which additional advantage could not be drawn by adding extra amount of WMF into the concrete system. Considerable improvement in compressive strength of WMF admixed concrete upto 20% is primarily due to its excellent voids infilling capacity by virtue of its microfine size and silicious nature. It imparts better pozzolanic action particularly when silica from WMF comes in contact with CH liberated during the hydration of cement particles, as a result of which additional C-S-H compounds produce. As in the case of flyash admixed concrete, prolonged curing plays a significant role in the compressive strength development for WMF admixed concrete too.

For ternary mixes containing microsilica in addition to either of WMF or flyash there is an increment in compressive strength values of mixes with an increment in microsilica content as the duration of curing period increases. The rate of increase is quite significant for those mixes, which have flyash content equal to or greater than 20%, and microsilica content equal to or greater than 7.5%. This is because of the extra nucleation spaces provided by flyash to microsilica with enough



Fig. 6 Compressive strength of flyash admixed ternary concrete mixes



Fig. 7 Compressive strength of WMF admixed ternary concrete mixes

water, which otherwise would have been very small within the hydrated pore solutions adjoining the cement particles.

The trend of increase of compressive strength for WMF admixed ternary concrete mixes quite resembles to that of their binary concrete mixes. However, in the case of WMF admixed ternary concrete mixes, CWS12 offered the maximum compressive strength followed by CWS11, CWS8, CWS10, CWS7, CWS6, CWS9,CWS5,CWS4,CWS3,CWS1 and CWS2 respectively at 90 days moist curing. Very similar trend of increase of compressive strength is observed at 60 days moist curing. Whereas, at 7 & 28 days moist curing, compressive strength development pattern is slightly abrupt. Highest compressive strength is being offered by CWS8 at the age of 7 & 28 days but in the long run, this particular mix designation failed to prove to be the highest one. Such a phenomenon strongly inferred that for concrete mix containing WMF and WMF & microsilica together, compressive strength obtained either at 60 or 90 days moist curing should be considered for the design of pavement to draw the maximum possible advantage as their efficacy is being strongly influenced by the prolonged curing.

3.6 Results from flexural strength test

A decrease in flexural strength of all mixes at all durations was observed in Fig. 8 with respect to normal concrete mix which was obvious on account of cement substitution. This also indicates that the pozzolanic activity and the packing effect of the admixtures were not effective enough to



Fig. 10 Flexural strength of WMF admixed ternary concrete mixes

increase the flexural strength beyond that of normal concrete mix. Yet it was also observed that WMF at 20%-30% content gave comparable strength to normal concrete at 28 days and even beyond that. Comparing WMF with flyash, it was observed that percentage decrease in flexural strength was higher with progressive addition of flyash, whereas the rate of percentage increase in flexural strength was lower in WMF. Ternary mixes too followed the same trend with WMF and flyash addition, so it will be better to focus on the effect of microsilica in these mixes. As seen in Fig. 9, with microsilica addition there was an increase in the flexural strength of all mixes except those containing 30% flyash. With 10% microsilica a decrease in strength was in equal proportions for all mixes with microsilica addition at a given curing period. With the increase in curing period

there was an overall increase in this proportion for all mixes such that at 90 days there was highest increase in flexural strength with an increase in microsilica content in the mix. Fig. 10 shows the behaviour of WMF admixed ternary mixes, and indicates that, with the addition of microsilica there was an increase in the flexural strength of all mixes except those which have 10% microsilica and WMF in range of 20%-30%. But at 90 days curing no such exception was observed and flexural strength increased with microsilica substitution for all WMF contents.

4. Discussion

4.1 Materials' discussion

OPC used in the study, exhibits particle sizes comparable to flyash and a considerable fraction of flyash is even larger in size than OPC particles which may interfere the reaction process by retarding the reaction rate as well as bringing down the density of mix. On the other hand, a fair chance for improvement in workability is anticipated due to higher rate of dispersion of cement particles on account of high negative charge carried by their larger sizes. Strong tendency of pore infilling capacity is anticipated on admixing of ultra-fine microsilica and WMF.

Chemical composition of materials strongly suggests that microsilica would be prominently proactive in comparison to the rest, as it has inherent ability to contribute strength development through pozzolanic reactivity. By virtue of its amorphous microfine nature, microsilica is a promising material for infilling micro pores and apt for reduction of hydrated cement compounds like CH and ettringite considerably. All the admixtures are pozzolanic and the anticipated reactivity follows the order: microsilica, WMF and fly ash.

4.2 Discussion on pastes and slurries

4.2.1 Compatibility at W/C =0.3

Both microsilica and WMF at their lower content did not get adsorb on the cement particles, thereby showing a raised water demand. This demand kept on increasing with their higher contents due to retardation, and owing to more water consumption by their particles' surfaces. Hence, flow time shows a small increment with microsilica and WMF addition at their lower content, and vice versa at higher contents. The reason for continuous reduction in water demand by flyash is the low reaction tendency of flyash. Higher fraction of large sized flyash particles in the paste further reduced the water demand due to dilution. The aluminates present in the flyash could have done two functions depending upon their reactivity: firstly they could have consumed the superplasticizer, and secondly, converted the ettringite and lime into monosulphates and C-S-H respectively. This somehow should have reduced the water content in pore solution. It is not sure that up to what extent the aluminates were reactive, but overall, there is a decrement in the flow time by the use of flyash.

4.2.2 Compatibility at W/C=0.35

Flyash was observed to release entrapped water at a higher rate, at its lower contents and as the flyash content increased, the rate decreased abruptly due to no further increment in adsorption of flyash at cement particles' surfaces. WMF and microsilica mixes released more water at their lower content in comparison to their water consumption by adsorption of water at their respective

surfaces. Hence they gave lower flow times. Further adsorption on cement particles reduced at their higher contents, thus flow times increased.

4.2.3 Compatibility at W/C=0.4

Even though the amount of water in the pore solution was higher for the free movement of WMF and microsilica particles, but the flow did not change at high content of WMF and microsilica, because there is a limit beyond which all the surfaces of cement particles get engulfed with admixtures and no further adsorption is possible.

Initially whatever may be the effect of admixture but at higher superplasticizer contents there is enough water in the paste and the paste behaviour becomes independent of the behaviour of its constituents. This generally happens at superplasticizer contents above 1.5%, 1.2%, and 0.9% for water/cement ratio of 0.3, 0.35, and 0.4, for both pure cement paste and admixed pastes.

4.3 Discussion on workability of fresh concrete

WMF is acicular and it has high adsorption tendency as well as the tendency to be a part of pore solution at its higher contents. Hence it increases the inter-particle friction by producing friction between cement particles on which it get adsorbed, and it also increases the viscosity of pore solution at its higher contents. Microsilica has smooth texture and spherical shape, which induces a ball bearing effect between cement particles on whom it get adsorbed. The ball bearing effect does not allow the cement particles to move away though it allows them to roll or slide over each other on account of the sticky nature of microsilica. Khayat and Aitcin (1992) also suggest that presence of microsilica affects the properties of fresh concrete by inducing cohesivity and thus reducing the bleeding of concrete. Both of these factors work contradictory as far as shrinkage is concerned; on one hand cohesivity also requires more slump for a given flow, with respect to a normal concrete. But it has one advantage i.e. homogeneity, and thus enables good passability with medium flowability, thereby enabling microsilica admixed concrete as a pumpable concrete (1998).

Hence at higher amount of microsilica, the flowability and passability decreased but the segregation resistance increased, because the pore solution between the cement particles get thicker and more viscous due to increased microsilica content.

4.4 Discussion on drying shrinkage

Initially when WMF is added in lower quantity (up to 20%), the cement particles are not far apart in the solution, and the voids in between them are easily filled or blocked by WMF due to its pore size or grain size refinement, represented by dense zigzag hydrated WMF particles in the SEM image shown in Fig. 11 for mix CW2 & CWS5. Though the water is released when WMF enters into the cement voids, but this water only is used in the accelerated hydration done by cement particles. As a result of which, the water is not able to escape from the pores (mainly gel pores) and shrinkage is reduced. With the increment in the content of WMF in the mix, the cement particles increase their distance, and more and more WMF particles enter into the pore solution present in between these voids (infiltration of WMF made possible by released water being provided by superplasticizer). As a result of which the number of capillary spaces increase whereas the gel pores reduce. This is verified from the SEM images of mixes containing WMF, as has been



Fig. 11 SEM image of (a) CW2 (b) CWS5



Fig. 12 SEM image of (a) CW3 (b) CWS9

shown for CW3 & CWS9 mix in Fig. 12. Since the void space is larger, the lower hydration rate due to dilution of cement, as well as insufficient capacity of WMF to block the voids is not able enough to stop the water from escaping from these capillary voids. Therefore, an increased shrinkage stress was reported for WMF content greater than 20%. As a reinforcer, WMF will increase the tensile strength and toughness of mortar, which would resist the crack development and also will delay the propagation of cracks by distributing the stresses over a local region. Thus, the overall effect observed for WMF addition, was the delay in crack development in spite of high stress rate. Since the time period of cracking also depends upon the stress rate development, therefore the variation trend for time period of cracking is similar to the stress development.

For flyash mixes, the hydration rate is higher than normal concrete uptil 7 days. This is apparent from the SEM images of flyash admixed pastes (CF2 & CF3) at 7 days which shows large amount of CSH formation (Fig. 13). The effect of this acceleration increases its crack development time in comparison to normal cement concrete. After 14 days the effect of acceleration diminishes, which the literature about flyash supports CUR Report (1991), and thus the samples start cracking. Though flyash contain alumina and calcium oxide, which promote expansive ettringite formation in presence of sulphates, which would otherwise reduce shrinkage stresses, but the reactivity of alumina depends upon its crystal morphology i.e., whether it is available in glass part of the compound or not (Langan *et al.* 2002). If it is present in glass part than it provides a long term source of lower rate of ettringite formation in presence of sulphates. SEM images in Fig. 13 indicate that ettringite formation is hindered in presence of flyash. Thus, the stress rate is equal to or a bit higher than normal concrete because effect of pore refinement is



Fig. 13 SEM image of (a) CF2 (b) CF3

nullified by presence of fine material (fraction of flyash much finer than cement) verified by particle size analysis. Hence with the increment in flyash content both the stress rate and the crack time increases to a small extent.

4.5 Discussion on compressive strength

Flyash, overall, is a low reactive pozzolana. The strength improvement due to flyash is due to its pozzolanicity and filler effect. The flyash particles fits into the voids of cement particles, and later on after hydration, nucleate hydrated products over its surface, thereby reducing the capillary voids or porosity, which imparts strength. It is only during the first two-three days, that its reactivity is a bit higher because of small quantity of reactive silica present in it (confirmed from chemical analysis done through X Ray fluorescence), but owing to its bigger size this reactive silica is not able to hydrate lime into C-S-H at the same rate as WMF. It does not have any fiber effect because of its spherical smooth particle shape. The increment in hydration because of flyash is on the account of water released due to its dispersion effect, the presence of poorly reactive aluminates which slowly convert the ettringite into hydrated aluminates, and the bigger sized particles which adsorb less water and provide nucleating surfaces for other admixtures like microsilica to hydrate owing to its smooth surface. Even though flyash is a low reactive material, but it does maintain continuous reactivity and hence more densifying action takes place which finally results in improvement of concrete strength to a greater extent.

Microsilica remains highly reactive until 28 days beyond which it starts dropping down due to depleting moisture content. Both WMF and flyash are able to hold moisture effectively at 30% & 20% and higher levels, respectively. Microsilica which was earlier adsorbed onto cement particles, and being reactive goes inside this pore solution which lies in the vicinity of these admixtures, due to the potential gradient developed on account of concentration difference. It starts hydrating on the nucleating surfaces provided by these admixtures. The hydration rate of microsilica is higher in this case due to lesser competition for moisture with respect to the situation when microsilica was competing with cement particles for moisture. The hydration done by flyash and, especially WMF, cause an additional improvement. WMF gave exceptional high improvement at 30% due to its fiber action on the hydrated paste bound to it.

4.6 Discussion on flexural strength

Flyash though reduced the ettringite and CH due to its pozzolanic activity, but its packing effect was very poor because of its large sized particles, whereas WMF did both. Though it was found from SEM test that WMF did not stabilize ettringite because of lesser aluminates present in it, yet it improved the flexural strength because of its good packing as well as reinforcing effect, as has been found by Low and Beaudoin (1992, 1993). The needle shaped particles of WMF filled the voids created in between CH and ettringite and oriented them to make a densely reinforced matrix. Also WMF did CH stabilization and produced CSH. Hence there was a reduction in the flexural strength of the concrete mix with flyash, whereas WMF increased it. The strength was found comparable to normal concrete at higher WMF contents i.e., 20%-30%, at 28 days and even beyond it.

As we know that flexural strength of concrete is mainly affected by the interfacial transition zone (ITZ), thus ITZ is considered as the strength-limiting phase in concrete. The introduction of microsilica at progressive levels improved the ITZ by highly stabilizing both CH and ettringite present in ample amounts in this zone. Thus it improved the bond between aggregates and mortar and densified the crack propagation path. Therefore, on an average an increment of 10% was found for an increase in microsilica content from 2.5%-10% for all twelve ternary mixes and at all durations irrespective of whether it is WMF or flyash. Also the maximum percentage rise in flexural strength with respect to normal concrete was recorded to be approximately 20% in case of CWS12 at 90 days.

Microsilica filled the voids created by large sized flyash particles, stabilized ettringite and CH and also filled the voids in between cement particles for flyash contents upto 20%. But beyond it, even microsilica could not fill the extra voids created in between excess flyash particles and thus the strength of the mix reduced on account of weak mortar present in it. In case of WMF ternary mixes, the flexural strength reduced at 10% microsilica and WMF contents higher than 20%, for all curing periods. This happened on account of water scarcity caused by the pozzolanic activity as well as water adsorption of fine sized WMF and silica fume which caused self-desiccation. Higher content of silica in flyash strongly indicates good potential to reduce formations of CH and ettringite.

5. Conclusions

Cement substituted with 20% WMF and 5% microsilica yields slurries having good compatibility with PCE based superplasticizer. For a given superplasticizer-cement ratio, these slurries at high water to cement ratios, achieve same compatibility with cementitious mix as in case of low water to cement ratio borne flyash admixed slurries. As far as workability of concrete is concerned, though flyash imparts high flowability but have poor passability and segregation resistance as the mix does not remain as a single unit. On the other hand WMF admixed concrete mixes show medium flow, good passability and better segregation resistance because of better homogeneity in the mix along with 5% microsilica.

The mixes CWS2, CWS3, CWS6, CWS7, CFS5 and CFS7 could yield SCC mixes which concludes that not more than 20% of flyash/WMF with 2.5-7.5% microsilica should be used. Furthermore, only CWS6 and CFS7 have low shrinkage which proves that good pore refinement with high tensile strength could be achieved only through 20% flyash or WMF along with 5-7.5% microsilica. But the increment in compressive and flexural strength with respect to normal concrete was achieved only by ternary mixes of WMF (20-30%) containing 7.5-10% microsilica.

Thus it is positively possible to substitute cement with WMF to yield self compacting concrete containing high values of compressive and flexural strength provided that WMF and microsilica are present at the rate of 20% and 7.5% respectively. An increment by nearly 5.25% and 10% could be expected in flexural strength and compressive strength at such substitution levels of cement. Also considering nearly 30% substitution of cement in this case with cheap WMF and costly microsilica in small contents (7.5%), this highly flowable concrete is more environmental friendly and atleast expected to bear same construction cost.

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