Microstructural behaviour and flowing ability of self-compacting concrete using micro- and nano-silica

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Concrete appears to be the strongest backbone for the building industry as construction material. The chief constituent of concrete is the cement which has a major negative environmental issue causing huge carbon dioxide contribution during the cement production. The cement can be partly replaced by various wastes and by-products like fly ash, ground granulated blast-furnace slag, micro-silica etc. This study is about the utilisation of micro- and nano-silica in self-compacting concrete (SCC). The replacement level of cement by silica purely depends on its silica content and its structural composition. This is identified solely by the material characterisation techniques in terms of the physical and chemical composition of that particular substitute material. The flowability property as suggested by the European Federation of National Associations Representing for Concrete guidelines was tested on slump flow, J-ring, V-funnel, Orimet and L-Box. The strength parameters are studied for SCC with micro-silica of 5, 10, 15% and nano-silica of 1, 2 and 3%. The important study on the microstructural aspect of scanning electron microscope and energy dispersive X-ray analysis was conducted on concrete at 28 days. Hence, using the by-products like micro-silica, the environment can be saved to some extent against the disposal.

1. Introduction: Concrete is the universally preferred structural material by the construction sector. The use of conventional concrete in areas of highly congested reinforcements is most challenging, which gave rise to the development of SCC in the late 1980s. This concrete was then well established and most preferred for reinforced areas. SCC has turned out to produce high strength and good workability with the usage of mineral admixtures. This trendy concrete consists of materials such as cement, water, aggregates and also engineered materials such as fly ash, micro-silica, ground granulated blast-furnace slag (GGBFS) etc. [1].

Presently, the construction industries are updated with SCC than conventional concrete. SCC possesses the ability to flow under its self-weight. The highly congested reinforcements can be stuffed with SCC easily [2]. SCC is being treated as an energy efficient material, requires less on-site task. It incorporates only low-energy material as the mineral additives in concrete. The fusion of fillers along with the mineral admixtures discovered an innovative way to enhance the hardened properties of SCC [3].

It was identified that SCC possesses little resistance to concrete flow, which can be utilised for placing and compacting with self-weight requiring minimal vibrations. SCC proved to be an efficient, concrete providing better durability and good quality of concrete compared to normal concrete [4]. To achieve a good flowability of concrete, there occurred a need to reduce the aggregate content and also by increasing the volume of paste. The paste volume can be increased by an effective adoption of chemical and/or mineral admixtures.

The superplasticiser dispersed the cement particles by decreasing the particle to particle friction and enabled the decrease in water quantity to purposely attain the flowing ability of concrete [5]. The stability can be obtained by incorporating viscosity-modifying admixture along with the appropriate dosage of superplasticiser to retain the flowability [6]. The mineral admixtures such as fly ash, GGBFS or limestone powder can be adopted to uplift the particle–particle interaction and grain-size distribution providing great cohesiveness to concrete [7].

The mineral admixtures were used for producing SCC. The rheological properties, workability of concrete were evaluated and the performance of hardened concrete was also determined [8, 9].

European Federation of National Associations Representing for Concrete guidelines [10] framed the SCC system with all design parameters and suggested the advisable mineral admixtures to be employed, established the measures for fresh concrete properties with three primary parameters: filling ability, passing ability and segregation resistance.

Plentiful reports stated that by employing mineral admixtures to concrete have improved the strength characteristics and drying shrinkage also. The compressive strength and drying shrinkage were tested for SCC using various mineral additions such as fly ash, GGBFS, micro-silica, metakaolin [11]. Micro-silica is a by-product of the silicon industries and it is the widely used silica in amorphous form. When micro-silica used as the cement replacement enhances the pozzolanic activity and tends to fill the pores in concrete [12].

The performance of SCC replacing various mineral admixtures such as fly ash, silica fume and blast furnace slag was used as cement replacement material at 30, 40 and 50% [13]. The compressive strength of silica fume at 30% was found to be high for 28 days compared to fly ash and blast furnace slag. The percentage increase of fly ash concrete strength to silica fume was 27.19% and blast furnace slag concrete with silica fume was 40.43%. Comparative results satisfied the workability range suggested by EFNARC and proved that silica fume possessed greater strength.

The SCC with binary blending materials like GGBFS, silica fume was examined for the fresh and hardened state properties of concrete. The cement was partially replaced by silica varying from 5 to 25%. The flowability properties were satisfied for all mixes and strength was observed to be greater for the range of 5–10% micro-silica [14].

The effect of addition of micro- and nano-silica in SCC at sulphuric medium was investigated. The compressive strength of the combination of micro- and nano-silica at 5 and 1%, respectively, yielded greater strength of 15.41% compared to control concrete at 28 days [15]. The fresh properties of SCC with the nano-silica of different sizes of 5, 17 and 35 nm were investigated. The rheological and flow characteristics were evaluated and it was concluded that lesser the average particle size of nano-silica resulted in greater compressive strength [16].

This Letter focused to determine the effect of micro-silica as a mineral admixture on terms of freshness, hardened and microstructural properties of concrete. The workability test for SCC such as slump-flow, V-funnel, L-Box and Orimet tests was done to study the characteristics of SCC. The hardened concrete tests such as compressive strength, flexural strength and microstructural analysis were determined.

2. Materials and mix design

2.1. Material characterisation: All through the experimental study, ordinary Portland cement of 53 grades conforming to IS:12267-1987 was used. Micro-silica (MS) obtained from Corniche chemicals and nano-silica (NS) from Astraa chemicals was used as cement substitute materials. Locally available river sand with a fineness modulus of 2.91 was used as fine aggregates. The coarse aggregate with a maximum size of 12.5 mm was used. Superplasticiser (SP) used in this Letter is BASF Master Glenium 8233, poly-carboxylate ether type. The viscosity modifying agent (VMA) used is FOSROC Auramix 200 that alter the viscous property of concrete. Potable water was used for mixing and curing of concrete. The chemical composition of cement, micro- and nano-silica is listed in Table 1. The specific gravity and bulk density of aggregates are listed in Table 2. The scanning electron microscope (SEM) and X-ray diffraction (XRD) of cement, micro-and nano-silica are presented in Figs. 1*a*–*c* and 2. SEM analysis represents the structural morphology of particle size identification of cement, micro- and nano-silica. Fig. 2 shows the amorphous silica content present in the samples.

The SEM image of cement exhibited clinker crystals with the size ranging from 150 to 500 nm. The morphology of micro-silica was spherical in shape with the average particle size of 150 nm. The nano-silica was spherical in shape with the average size of 17 nm.

The XRD report of cement with peak intensity indicated the presence of tricalcium silicate, dicalcium silicate, tricalcium aluminate and gypsum in the tested cement sample. XRD of micro- and nano-silica with the peak indicated the presence of the silicon dioxide content.

Table 1 Chemical composition of cement, micro- and nano-silica

Chemical analysis, wt.%	Cement	Micro-silica	Nano-silica
SiO_2	22.350	99.886	99.888
Al_2O_3	10.880	0.043	0.005
Fe_2O_3	5.870	0.040	0.001
CaO	55.710	0.001	0.060
TiO_2	0.490	0.012	0.004
MgO	1.150	—	—
Na_2O	0.340	0.003	—
K_2O	0.170	—	—
SO_3	1.590	—	—
loss on ignition	1.230	0.015	0.660
insoluble residue	0.220	—	—

Table 2 Specific gravity and bulk density of the fine and coarse aggregate

Material test	Results
specific gravity of cement	3.1
specific gravity of fine aggregate	2.61
specific gravity of coarse aggregate	2.66
bulk density of fine aggregate	1.71 kg/m^3
bulk density of coarse aggregate	1.56 kg/m^3

2.2. Mix proportion: The mix design of M40 grade of SCC was worked out based on the EFNARC guidelines. The estimated quantities for the study were listed in Table 3. The notations mentioned in the work are C represents the control concrete, 5MS, 10MS, 15MS that indicates the 5, 10, 15% micro-silica as cement replacement and 1NS, 2NS, 3NS represents the 1, 2 and 3% nano-silica as partial cement substitute material. This Letter concentrates the SCC with the replacement of cement with micro- and nano-silica with the varying percentage weight of cement from 5 to 15 for micro-silica and 1 to 3% for nano-silica, respectively. In addition, SCC was produced with optimum of micro- and nano-silica combination was also used.

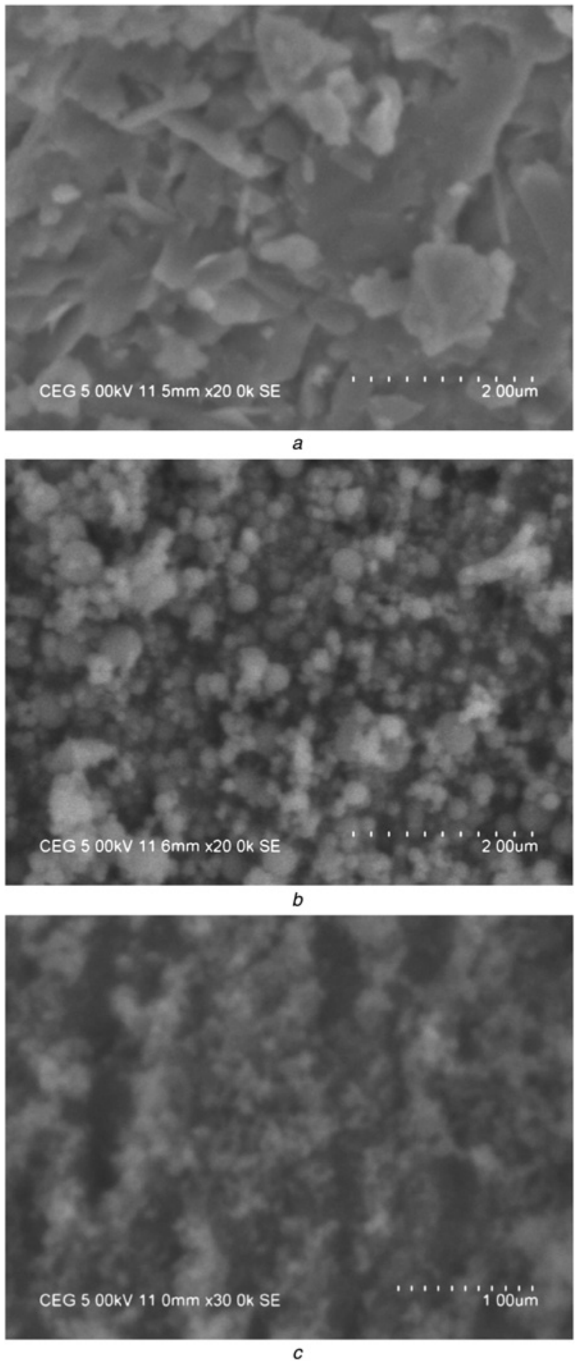

Fig. 1 *SEM analysis of materials used*
a SEM analysis of cement,
b SEM analysis of micro-silica,
c SEM analysis of nano-silica

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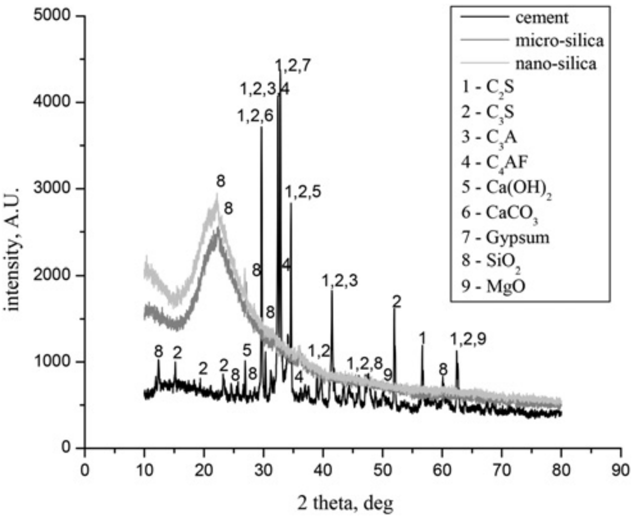

Fig. 2 *XRD analysis of materials used*

2.3. Experimental methods

2.3.1. Slump flow test: The fresh concrete test of SCC was conducted to evaluate the workability of concrete mixtures. The filling ability was determined by the Abrams cone (slump cone) as per EFNARC guidelines (2005). The slump flow diameter was measured at two different diameters at right angles and the average was noted as the final slump flow of concrete. Measure the elapsed time to achieve the 50 cm mean diameter and then it is notified as T_{50}cm.

2.3.2. J-ring test: The fresh concrete test was conducted with apparatus specifications given by EFNARC (2005). This J-ring test assessed the passing ability of fresh SCC. This test procedure involved the placing of the slump cone inside the J-ring apparatus. The fresh concrete is poured onto the slump cone, as the cone was lifted upwards and the concrete would flow through the J-ring. The slump flow through the J-ring was measured at two different locations and an average was taken. The slump flow and J-ring flow was mentioned in Fig. 3. The height difference of concrete in the J-ring was measured and notified in Table 4.

2.3.3. V-funnel test: The V-funnel apparatus is used to examine the unconfined flowability. The time grasped for the SCC to pass through the V-funnel was observed.

2.3.4. Orimet test: Orimet was used to detect the filling ability of fresh SCC. This apparatus has an orifice at the bottom and gate is provided at the end. Fresh concrete was poured on to the apparatus and the time elapsed since the SCC passing through Orimet was finally determined.

2.3.5. L-Box: L-Box was preferred to detect the capacity of filling fresh SCC in the areas of congested reinforcements. This test was conducted based in [10]. The fresh SCC was poured on the vertical portion of the L-Box and the gate was lifted. Fresh concrete passed through the rebars at the junction. The difference in the concrete level at the beginning and at the end of the box was noted. The height ratio (H_2/H_1) determined the L-Box ratio for fresh SCC and thus represented in Fig. 4.

2.3.6. Compression test: Cubes of size 150 × 150 × 150 mm were used to determine the compressive strength of 7, 28 and 90 days. This test was performed as specified by IS:516-1959 (2004). The compressive strength of the cube was tested with the compression testing machine of 2000 kN. The value of compressive strength is the ratio of crushing load by resisting area of a cross-section of the specimen.

2.3.7. Flexure test: The beams of size 100 × 100 × 500 mm were cast to investigate the flexural strength of 28 and 90 days. This test was carried out based on IS:516-1959 (2004). All the concrete specimens were cured in water and dried on the day of testing.

2.3.8. Microstructure analysis: The microstructural study of concrete was carried out by conducting SEM and Energy Dispersive Analysis of X-rays (EDAX) analysis of concrete specimens at 28 days.

3. Results and discussion

3.1. Slump flow test: The fresh concrete properties were tested as per European standards. The list of tests investigating the workability parameters with the specified limitations are mentioned in Table 5. The slump flow was tested for different mix proportions of micro- and nano-silica content as a cement replacement in SCC. The SCC with the greater slump flow took less time to fill 50 cm. The slump flow values (mm) and the nature of SCCs were compared with [10] and listed in Table 6.

3.2. J-ring test: SCC was designed and worked out in the laboratory. Initially, the actual slump flow diameter was measured. Next, the J-ring slump flow was noted. This test was used to find the J-ring slump flow by placing the slump cone inside the J-ring and measure the flow diameter. This showed the ability of each concrete mix passed through the rebars in the J-ring. The results are interpreted in Fig. 3.

The control SCC showed a difference in slump flow and J-ring flow of 15 mm which inferred that the viscosity was moderate with no segregation. This difference should be <30 mm. SCC

Table 3 Mix proportions of self-compacting concrete

Mix ID	Mix proportions, kg/m^3						Dosage, %	
	Cement	MS	NS	Water	Aggregates		SP	VMA
					Fine	Coarse		
C	450	—	—	200	1000	750	1.2	0.12
5MS	427.5	22.5	—	200	1000	750	1.5	0.15
10MS	405	45	—	200	1000	750	1.8	0.18
15MS	382.5	67.5	—	200	1000	750	2.0	0.20
1NS	445.5	—	4.5	200	1000	750	1.5	0.15
2NS	441	—	9	200	1000	750	1.8	0.18
3NS	436.5	—	13.5	200	1000	750	2.0	0.20
10MS + 2NS	396	45	9	200	1000	750	1.8	0.18

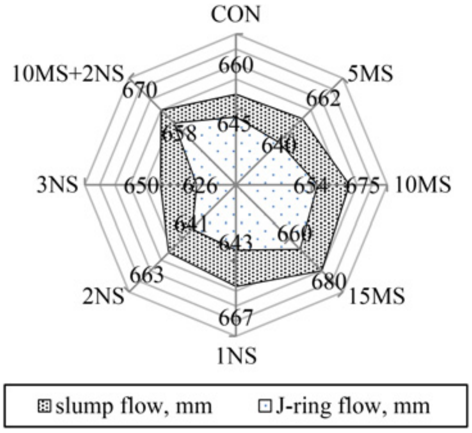

Fig. 3 *Representation of slump and J-ring flow*

Table 4 Standard deviation for fresh concrete properties

Test results	Slump flow	J-ring flow	L-Box ratio	V-funnel	Orimet
standard deviation	9.342	11.179	0.012	1.727	0.504

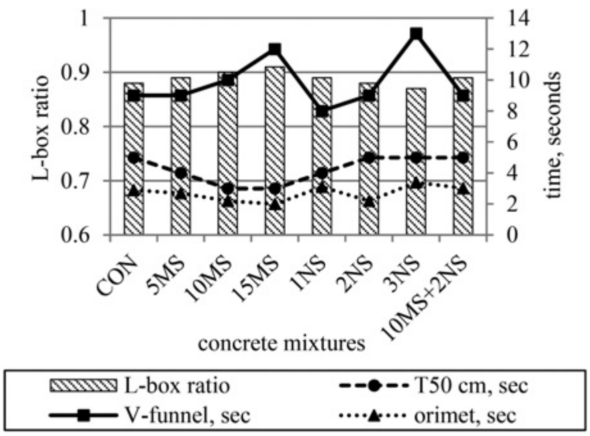

Fig. 4 *Fresh concrete tests on SCC and its flow characteristics*

Table 5 Acceptance limits as per EFNARC standard

S. no.	Method	Unit	Typical range of values	
			Minimum	Maximum
1	slump flow	mm	650	800
2	T_{50}, cm slump	second	2	5
3	V-funnel	second	6	12
4	L-Box	(H_2/H_1)	0.8	1.0
5	Orimet	second	0	5
6	J-ring slump flow difference	mm	0	30
7	J-ring height difference	mm	0	10

with 15% micro-silica showed 21 mm of flow difference which reflected the presence mild segregation. SCC with 3% nano-silica exhibited a horizontal flow difference of 24 mm with a minor blockage near rebars. All other mixes apart from this appeared to possess good workability without segregation and blockage. SCC with a combination of micro- and nano-silica expressed a flow difference of 12 mm which meant that concrete was more compact microstructure and ease flow of concrete through the reinforced bar was seen. As per investigations from the workability test of SCC, when the height difference is more in J-ring test the concrete fails to pass through the rebars present as shown in Fig. 5.

Table 6 Fresh concrete properties

Mix ID	Slump flow	Nature of concrete
EFNARC	<650	viscosity too high
standards	>750	viscosity too low
C	665	good viscous flow
5MS	662	good viscous flow
10MS	675	better viscous flow
15MS	680	better viscous flow and mild segregation
1NS	667	good viscous flow
2NS	663	good viscous flow with dense concrete
3NS	650	mild blocking was observed
10MS + 2NS	670	dense concrete with a better viscous flow

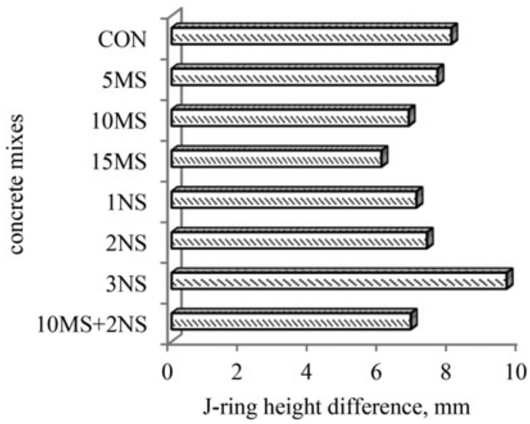

Fig. 5 *J-ring height difference*

3.3. V-funnel test: SCC with 15% micro-silica and SCC with 3% nano-silica took greater time to pass through the V-funnel which reflected their fresh workability characteristics like segregation and blockage, respectively. SCC with 1% nano-silica took 8 s in V-funnel test expressing the low viscous type of concrete. SCC with 10% micro-silica and 2% nano-silica reported that good viscosity, flowability flaws such as segregation, high yield value and blockage were not observed. Those results were represented in Fig. 4.

3.4. Orimet test: The time elapsed for the SCCs to pass through the Orimet was observed to be in the range 0–5 s. SCC with 15% micro-silica took less time to pass this test as shown in Fig. 6.

3.5. L-Box: The L-Box ratios for different SCCs were investigated as shown in Fig. 4. The addition of micro-silica from 5 to 15% increased the L-Box ratio due to the increasing contents of chemical admixtures. Though increasing the superplasticiser and viscosity modifying agent, the addition of nano-silica expressed a decrease in L-Box ratio. This showed that 3% nano-silica reported least passing ability compared to other mixes. The combination of 10% micro-silica and 2% nano-silica produced considerable ability of concrete to pass through the apparatus.

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Fig. 6 *Orimet test used for this study*

The standard deviation values for fresh concrete properties were listed in Table 4.

3.6. Compression test: The compressive strength of the cubes was tested for 7, 28 and 90 days and shown in Fig. 7. The strength results of control concrete and various proportions of SCC with micro- and nano-silica particles are shown below. The pore filling effect of micro-silica improved the strength compared to control concrete and the nano-silica played an effect of role in forming the dense concrete as the surface to volume ratio is high when nano-silica was used in concrete. The 10% micro-silica concrete was found to be 7.62% greater than control concrete, 2% nano-silica concrete was 8.63% greater than control concrete. The combination of micro- and nano-silica concrete achieved to be 12.83% greater than control concrete and proved as a substitute for cement, provided the best strength comparatively.

3.7. Flexure test: The flexural strength of SCC using micro- and nano-silica was determined for 28 and 90 days. The combination of 10% micro-silica and 2% nano-silica produced the best results compared to other proportions and represented in Fig. 8. The standard deviation results are shown in Table 7.

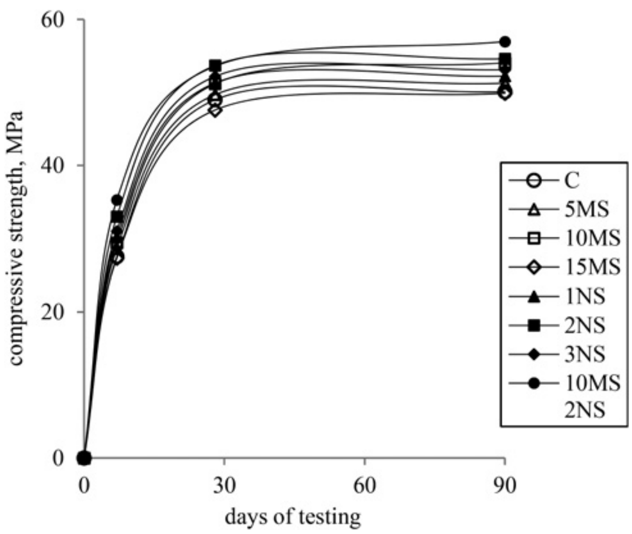

Fig. 7 *Compressive strength of SCC mixes used*

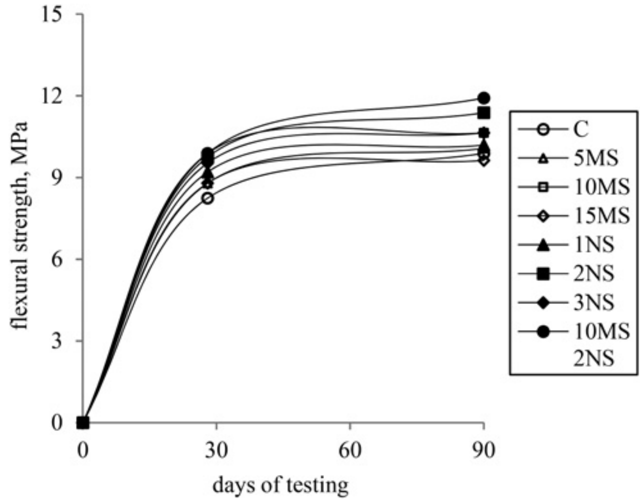

Fig. 8 *Flexural strength of SCC mixes used*

Table 7 Standard deviation values for hardened concrete properties

Experimental data	Compressive strength		Flexural strength	
	28 days	90 days	28 days	90 days
standard deviation	2.1666	2.412	0.606	0.777

3.8. Microstructure analysis: The microstructural analysis of SCC was done by taking SEM and EDAX analysis of the concrete specimens taken at 90 days. The SEM analysis of control SCC was shown in Fig. 9. The visible voids are seen in dark colour. The needle-like structures represent the presence of C–S–H gels and the crystal structures are calcium hydroxide. The addition of micro-silica improved the pore structure of concrete [17]. The formation of calcium silicate hydrate gel is very minimal in control SCC. Fig. 10 shows the SEM image of SCC with 10% micro-silica and 2% nano-silica at 90 days. The EDAX report of SCC control concrete provided a Ca/Si ratio of 1.44. The presence of voids is very minimal as the micro-silica tended to possess the pore filling effect when utilised in concrete. The nano-silica when added to concrete, it activated the cement hydration process and enriched the density of concrete.

The nano-silica additive in concrete helped to fill the voids between the micro-level particles. Nano-silica particles assisted to

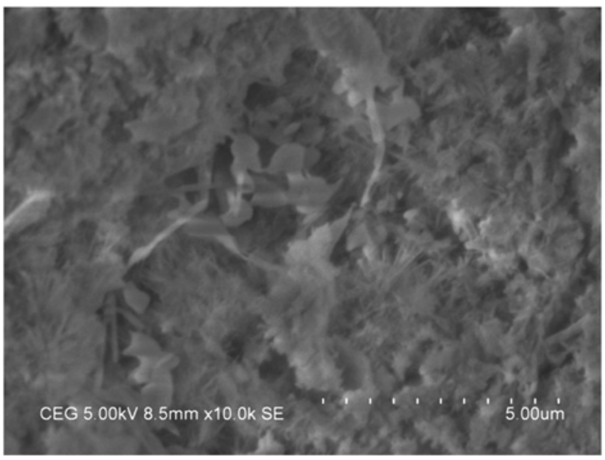

Fig. 9 *SEM image of SCC-control at 90 days*

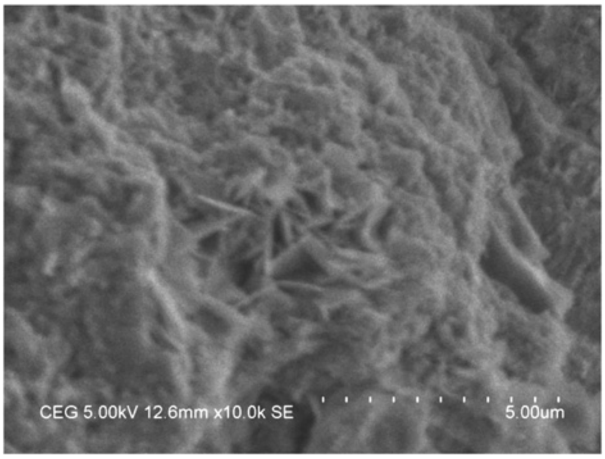

Fig. 10 *SEM image of SCC-10% micro-silica and 2% nano-silica at 90 days*

retrieve the packing of particles in the dense microstructure of cement paste in concrete. This nano-silica accelerated the cement hydration thus by reducing the $Ca(OH)_2$ crystals. The C–S–H gels were present in most of the regions which played a vital role in improving the bond between inter-facial zone (ITZ). The enhancement of calcium silicate hydrate (CSH) gel led to form a greater volume of reaction products near the ITZ. This enhanced CSH gel formation reduced the CH crystals and EDAX analysis of this concrete at 90 days produced a Ca/Si ratio of 1.27. The combination 10% micro- and 2% nano-silica particles refined the pore structure and activated the reactivity of hydration products, thus leading to the more compact microstructure of concrete at 90 days.

A dense concrete without pores was obtained by the inclusion of micro and nanoparticles in SCC.

4. Conclusions: In order to reduce the CO_2 emission to some extent due to the production of cement for the construction industry, the utilisation of micro- and nano-silica was proved effective. As SCC is being preferred in most congested reinforcements, these materials can be used to improve the workability and strength at the site. Micro- and nano-silica showed better workability through these test parameters such as slump cone, J-ring, V-funnel, Orimet and L-Box. All the mix proportions satisfied the European standards. SCC containing 10% micro-silica showed better workability and yielded good strength compared to 5 and 15% micro-silica. Similarly, SCC with 2% nano-silica exhibited better compressive and flexural strength at 28 and 90 days. Therefore the combination tried for the SCC with micro-silica at 10% and nano-silica at 2% yielded the best strength and the workability was also within the acceptable limits. The microstructural analysis through SEM analysis of concrete at 90 days for control and SCC with a combination of 10% micro-silica and 2% nano-silica were discussed in detail. Thus, by the pore filling nature of micro- and nano-silica in concrete the formation of C–S–H gel increased which relatively led to the increase in strength. The nano-silica improved the particle packing density of cement in concrete in microscopic level and it is evident with SEM analysis. The percentage variation of those two mixes in the aspects of strength was about 12.5% greater at 90 days. The EDAX report projected that the percentage difference in Ca/Si ratio of SCC control and optimum SCC with micro- and nano-silica was 12.5% lower. The Ca/Si ratio could be related to the compressive strength indirectly. The decrease in Ca/Si ratio increases the strength of concrete. A greater percentage of the densification was obtained in the ITZ at the level of microstructural study. Thus the need for adopting the micro- and nano-silica particles in SCC was met successfully.

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