Effects of the different atmospheric steam curing processes on the properties of self-compacting-concrete containing microsilica

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Abstract. In this paper an attempt has been made to study the use of microsilica on the properties of self-compacting-concrete (SCC) such as compressive strength, splitting tensile strength, flexural strength, ultrasonic pulse velocity (UPV) and microhardness when exposed to different atmospheric steam curing temperatures. The influence of microsilica as partial replacement of cement on the properties of SCC is investigated. In this study, mixes were prepared with three percentages of microsilica ranging from 5% to 10% and one controlled mixture without microsilica was also prepared for comparison. The specimens of each concrete mixture were heated up to different temperatures (65°C, 70°C and 75°C). The variables included were the temperature effects (65°C, 70°C and 75°C) using Cem I 42.5. SCC mixes enhanced atmospheric steam curing compressive strength ranging from 29.20 to 38.50 MPa, flexural strength ranging from 4.95 to 6.56 MPa and splitting tensile strength ranging from 1.18 to 1.63 MPa. Test results clearly show that there is little improvement in the compressive strength within temperature range of 70°C as compared to 65 and 75°C, although there is little reduction in splitting tensile strength ranging from 65 to 75°C and with the increase in percentage of microsilica. However, the rate of splitting tensile strength and flexural strength was higher than that of the compressive strength at elevated temperatures and with the increase in percentage of microsilica. In this paper, scanning electron microscopic (SEM) observations were also made to explain the observed residual compressive strength increase between 65°C, 70°C and 75°C.

Keywords. Self-compacting concrete; UPV; strength; microsilica; microhardness.

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1. Introduction

Use of self-compacting concretes (SCCs) lowered the noise level on the construction site and diminished the effect on the environment and has improved the quality of concrete in situ. SCC is a new category of high-performance concrete (HPC), which is characterized by its ability to spread into place under its own weight without the need of vibration and self-compact without any segregation and blocking. The introduction of SCC represents a major technological advance, which leads to a better quality of concrete produced and a faster and more economical concrete construction process. The use of SCC in civil engineering has gradually increased over the past few years. In addition, the increasing proportion of fine material is required and anything smaller than 0.125 mm should be considered as the fine grains of fine material. For this purpose, fly ash, stone dust (limestone powder), slag (ground) and silica fume can be used (Persson 2001; Xie *et al* 2015; Türkmen & Kantarcı 2006; Sonebi 2004; Aydın 2007; Aydın & Gül 2007; Bonavetti *et al* 2003; Bosiljkov 2003).

In order to accelerate the cement hydration reactions with saturated steam under atmospheric pressure is applied with heat. To increase concrete strength as well as temperature, the environment has to be saturated with enough moisture. Heat required to heat the media is achieved by steam curing. This process occurs under 100°C with atmospheric pressure, it is considered as a special case of humidity curing (Neville 1997).

Although maximum temperature limit values in curing locations should be from 40 to 100°C, the temperature is applied between 65 and 80°C. While the maximum temperature applied to the circuit of steam curing increases, the compressive strength of concrete will increase. The most effective and most important parameter on the degree of economic efficiency and successful application of steam curing is the steam curing cycle and cement type (Öztekin 1980).

In this study, normal aggregate was used to as aggregate. Three different atmospheric Steam Curing Cycles were applied to investigate the effect of microsilica on the properties of SCC. The cement type used was CEM I 42.5. The properties of SCC under the different atmospheric Steam Curing Cycles, i.e. the compressive strength and ultrasonic pulse velocity (UPV) were investigated. In addition to slump flow, V-Funnel and L box tests were performed to assess workability.

There are many studies related to the effect of mineral admixtures on the mechanical properties of SCC, although the effect of microsilica in the different atmospheric steam curing on the relationship between microhardness and compressive strength of SCC has not been reported previously. Therefore, the effects of pozzolanic activity of microsilica admixture on the mechanical properties of SCC also need to be investigated. Slump flow tests were performed to assess workability. Furthermore, the compressive strength, microhardness and UPV of hardened concretes are also determined for three different atmospheric steam curing cycles.

2. Experimental program

The chemical composition, physical and mechanical properties of the cement and microsilica used in this study are summarized in table 1. Furthermore, the microsilica is used as a replacement of total binder and proper for the standards ASTM C 618, ASTM C 1240/95, AASHTO M 307, and CAN/CSA23.5-M86.

The maximum size of coarse aggregates used was 16 mm, while the smallest particles used as fine aggregate were 0–4 mm. All the natural aggregates used for this study were in dry form. A polycarboxylic ether based high range water reducer (HRWR), density between 1.07

Table 1. Chemical analysis and physical properties of Components.

Component	CEM I 42.5 (%)	Microsilica (%)
SiO ₂	20.08	90.02
Fe ₂ O ₃	6.01	–
Al ₂ O ₃	2.05	0.3
CaO	64.14	0.33
MgO	0.93	2.36
SO ₃	0.74	0.85
Sulphide (S ⁻²)	0.21	–
Chlor (Cl ⁻)	0.0124	–
Undetermined	0.32	–
Free CaO	0.34	–
LOI	1.55	–
Specific gravity (g/cm ³)	3.13	2.17
Specific surface (cm ² /g)	3751	–
Specific surface (BET) (m ² /kg)	–	20,000
Compressive strength (MPa) day 2	23.6	
day 7	37.9	
day 28	48.0	

and 1.10 g/cm³ (at 20°C) was used to enhance the flow-ability of all the mixtures. Furthermore, the effect of SP percentage on the compressive strength is tested during pre-tests and its applicability observed by the insignificant compressive strength variation at the pre-test stage.

3. Method

In this study, three different atmospheric steam curing temperatures, namely 65°C, 70°C, 75°C and CEM I 42.5 as cement type were used. Therefore, a total of four different mixtures were cast in this study. The percentages of microsilica are decided according to the literature, sensitively. The full details of these mixtures are given in table 2. The self-compacting concrete mixtures were prepared in a laboratory mixer. The fine and coarse aggregate was initially dry mixed for approximately 30 s. This was followed by the addition of cement, microsilica and 1/3 of the total mixing water.

Table 2. Mixture proportions and fresh concrete properties of SCC mixes*.

Mix	Cement (kg/m ³)	Microsilica (%)	FA (kg/m ³)	CA (kg/m ³)	w/b	SP (%)	Slump flow (mm)	U-box (mm)	L-box (mm)	V-Funnel (sn)
SCC1	400	0	1186	620	0.41	1.7	550	40	0.80	12
SCC2	380	5	1181	617	0.42	1.8	700	20	0.90	9
SCC3	370	7.5	1178	616	0.42	1.9	660	30	0.85	10
SCC4	360	10	1168	611	0.44	2.0	680	30	0.90	11

*FA stands for fine aggregates; CA for coarse aggregates; w/b for water-binder ratio; and SP for Super plasticizer).

Table 3. Atmospheric steam cure cycles and characteristics.

Applied steam cure cycles	Pre-pending period		Heating period		Heat impregnating period		Cooling period		Total steam cure cycle Time (h)
	Temp. °C	Time (h)	Temp. °C	Time (h)	Temp. °C	Time (h)	Temp. °C	Time (h)	
65°C h 24	25	4	25–70	4	70	24	70–32	4.5	36 h 30 min
70°C h 24	27	4	27–75	4	75	24	75–32	4.5	36 h 30 min
75°C h 24	29	4	29–80	4.5	80	24	80–32	5	37 h 30 min

After 1.5 min of mixing, the rest of the mixing water together with the SP was added. The SP is used as a percentage of binder. All batches were mixed for a total mixing time of 5 min. Specimens for the testing of the hardened properties were prepared by direct pouring of concrete into moulds without compaction. The self-compactability of the mixtures was examined according to standards of Self Compacting Concrete Committee of EFNARC (2002). For each mixture, nine samples of 100×200 mm cylinders and three 70×70×280 mm prisms (totally 144 specimens) were prepared and the smooth finishes on the surfaces of 70×70×280 mm prisms without exposure of aggregates have been observed sensitively. The specimens have placed into the curing cabin with their moulds without delay after finishing of setting time for cement (190 min) and cured for 36.5 h in atmospheric steam cure at 65°C, 70°C and 75°C. After curing period, the specimens are demoulded and stored till testing, for 24 h at 20 ± 3°C. The full details of these concrete mixes are given in table 2. The atmospheric steam curing cycles and characteristics of mixes are presented in table 3. At 36.5 h, samples were tested for compressive strength, splitting-tensile strength and flexural strength (centre point loading) in accordance with ASTM C-192, ASTM C-496, and ASTM C-293, respectively. The atmospheric steam cure cycles and characteristics of mixes are presented in table 3.

Microhardness measurements were made using a Vickers microhardness tester. Measurements were obtained using a 0.01 kg test load and 10 s load duration. The first indentation was made 0 µm from the aggregate surface with the long axis of the indenter parallel to the aggregate surface. Subsequent indentations were made in dimensions of every 15–20 µm (average) from the previous indentation and the whole analysis covered a distance from the aggregate surface of 150 µm (Hussin & Poole 2011).

4. Results and discussion

The data on the slump flow test of specimens made using normal aggregate and microsilica is given in table 2. As can be seen from table 2, concretes made with normal aggregate and containing microsilica show a flow diameter higher than 500 mm. Thus, all concrete specimens are self-compacting concrete for shown slump flow diameter higher than 500 mm. Flow diameter values changed up to 700, 680, 660 and 550 mm for SCC (see table 2). The controlled mixture without microsilica made with normal aggregate was reduced workability of concrete specimens. Khayat (1999a, b) reported that an SCC often contains high-volume replacements of fly ash or BFS to enhance fluidity and cohesiveness and limit heat generation. Such materials are generally less reactive than cement and can reduce problems resulting from the fluidity loss of rich concrete.

The behaviour of SCC and vibrated concrete differs significantly between 65°C and 75°C. The compressive strength of concretes with or without microsilica at atmospheric steam curing to 65°C, 70°C and 75°C are shown in figure 1. At atmospheric steam curing 65°C with the

Figure 1. Relationship between compressive strength and curing temperatures.

increase in microsilica content from 5% to 10%, SCC mixes (SCC2–SCC4) developed compressive strengths between 34 and 29.2 MPa (table 4). Strength values were 36.1 MPa for the control mix (SCC1). An increase of strength was observed at atmospheric steam curing 65°C with the decrease of microsilica content from 10% to 5% (SCC4–SCC2) as compared with the control mix (SCC1).

At atmospheric steam curing 70°C, SCC mixes (SCC2–SCC4) developed compressive strength ranged between 38.5 and 33.4 MPa. An increase of strength was observed at 70°C, with the decrease of microsilica from 10% to 5% (SCC4–SCC2) as compared with the control mix (SCC1). At atmospheric steam curing 75°C, SCC mixes (SCC2–SCC4) developed compressive strength ranged between 35.1 and 30.6 MPa. An increase of strength was observed at 75°C, with the decrease of microsilica from 10% to 5% (SCC4–SCC2) as compared with the control mix (SCC1). The compressive strength increased with the decrease in microsilica content

Table 4. Hardened concrete properties.

Mixtures	Samples				
	Compressive strength (MPa)	Flexure strength (MPa)	Splitting tensile strength (MPa)	UPV (m/s)	Dry unit weight (kg/m ³)
SCC1					
65°C	36.1	5.5	1.3	4439	2350
70°C	33.5	5.4	1.5	4570	2368
75°C	32.7	6.5	1.3	4218	2380
SCC2					
65°C	29.2	6.6	1.6	4375	2355
70°C	38.5	6.1	1.6	4623	2385
75°C	35.1	7.4	1.3	4531	2332
SCC3					
65°C	34.0	6.0	1.3	4525	2367
70°C	35.3	6.9	1.4	4705	2399
75°C	31.8	6.0	1.4	4355	2372
SCC4					
65°C	33.0	5.8	1.6	4591	2376
70°C	33.4	5.7	1.6	4624	2360
75°C	30.6	5.0	1.2	4424	2369

Figure 3. Relationship between UPV and curing temperatures.

accepted for less than 100 μm in distance from aggregate surface, but the microhardness values are observed for above than 140 μm . Thus, the variation of microhardness through aggregate

Figure 4. (a) Microhardness test setup. (b) Typical distribution points of microhardness measurements including image of indent area and strips moved successively starting from the surface of aggregate extending outward to bulk paste (Xie *et al* 2015; Duan *et al* 2013; Igarashi *et al* 1996).

could be obtained. The reported microhardness values in the ITZ are results of concretes exposed to the atmospheric steam curing. Also reported is microhardness test data on bulk pastes in concretes exposed to the atmospheric steam curing. The microhardness test setup and typical distribution points of microhardness measurements including image of intent area and strips

Figure 5. Vickers microhardness distribution in SCC containing microsilica after atmospheric steam curing (a) 65°C, (b) 70°C and (c) 75°C.

Figure 6. Aggregate–mortar ITZ modality in SCC containing microsilica as a partial replacement Portland cement (Mag=10.00 K X).

moved successively starting from the surface of aggregate extending outward to bulk paste (Xie *et al* 2015; Duan *et al* 2013; Igarashi *et al* 1996) are given in figure 4a and 4b.

Figure 5a–5c shows variation of ITZ ranges, i.e., where the microhardness test resulted in lower Vickers Hardness Numbers (Hv) than those in the middle of the hardened bulk cement pastes. It can be seen that the results display noticeable differences between the control mix and microsilica content. In the microsilica content concretes, within the ITZ region, the microhardness increases as the distance from the aggregate surface increases. No obvious increase in width of the weak zone (ITZ range) was observed when microsilica content was increased from 5 to 10%. Increased microhardness values in the vicinity of the aggregate were observed in all specimens and this may be due to the presence of stiff inclusions in the excited range around the indentation, which restrains the flow of material under the indentation (Igarashi & Kawamura 1994). These inclusions are the result of the significantly heterogeneous distribution of the hydration products in both the ITZ and bulk phase. Moreover, the porosity of the ITZ is generally higher than the bulk phase. Thus, hydration products and porous structure of ITZ results stiff inclusions.

Microhardness test results on bulk pastes are shown in figure 4a and 4b. The trend shows an increase in microhardness up to 65°C and 70°C and beyond 75°C, microhardness decreases. The trend for microhardness variation is quite similar to that of compressive strength versus % of microsilica as shown in table 4.

The ITZ morphology of SCC examined in the SEM varies with the different specific surface area and the weight fraction of microsilica. Figure 6 presents the SEM pictures of aggregate–mortar ITZ at the atmospheric steam curing. The study on SEM pattern of aggregation–mortar ITZ follows the regular results of the microhardness test. It is summarized as follows. At the same weight fraction of microsilica, the reaction products in the ITZ grow more compactly for the higher surface area material (compare figure 6a–6c). The ITZ shown in figure 6c appears to be denser than that in figure 6a. It is apparent that microsilica at the weight fraction of 5% improves the ITZ microstructure the most.

5. Conclusions

This study concerns the behaviour of SCC at atmospheric steam curing. Specimens of various concretes compositions were made and subjected to different temperatures. The effects of atmospheric steam curing on the properties of SCC containing microsilica were studied. The

compressive strength and splitting tensile strength, the reduction in UPV, microhardness and observation of surface characteristics of samples were investigated. Some conclusions can be drawn from the experimental results:

1. Although microsilica reduces the strength, it is still possible to produce SCC with adequate strength.
2. All the high volume microsilica concretes have satisfied the norms that were set to qualify them as SCC and fresh properties like workability were in good agreement with European guidelines. The developed SCC was highly segregation resistant and had good flow-ability and passing ability.
3. The SCC mixes developed compressive strengths ranging from 38.50 to 29.2 MPa. Splitting tensile strengths developed were from 1.18 to 1.63 MPa. The compressive strength increased with a decrease in the percentage of the microsilica and the water-to-cementitious materials ratio. Due to lower w/b ratio of control mixes, the compressive strength decrease could be contributed to lower performance of microsilica.
4. The UPV of heated SCC specimens decreased with the increasing temperature. Test results indicated that SCC mixtures were exposed to atmospheric steam curing, 65°C and 75°C series showed higher reduction of pulse velocity when compared to 70°C series. Furthermore, it was noticed that the reduction of the pulse velocity of SCC mixtures in which containing microsilica were significantly higher than without microsilica in all replacement ratios.
5. The trend shows an increase of microhardness up to 65°C and 70°C and beyond 75°C, microhardness decreases. The trend for microhardness variation is similar to that of compressive strength versus % of microsilica as shown in table 4.
6. It is apparent that microsilica at the weight fraction of 5% improves the ITZ microstructure the most.

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