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Micro-Structural Characteristics of Self-Compacting Concrete With High Volume Fly Ash

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Abstract

Paper presents a study on the interfacial transition zone (ITZ) of self compacting concrete (SCC) in comparison to the conventionally vibrated concrete (CVC) based on scanning electron microscope (SEM). The SEM images were analysed using CLEMEX-4 (image analysis software). Results on the image analysis of SEM taken after 28 and 90 days of curing confirmed the absence of voids and micro-cracks in the transition zone of SCC compared to CVC. It is also observed that the high volume fly ash in SCC effectively replaces the cement content and fills the voids to form a thick non porous matrix and the higher cement content used in CVC is not fully utilized.

Keywords: Self Compacting Concrete, Conventionally vibrated concrete, microstructure, interfacial transition zone, scanning electron microscope, backscattered electron

Introduction

Self-Compacting Concrete (SCC) represents a milestone in concrete research. SCC is a highly flowable, non-segregating concrete that can spread over easily, fill the formwork and encapsulate the reinforcement without application of any mechanical vibration. Though the concept originally was thought to be a tool to enhance long-term durability of structures having members with congested reinforcements, the excellent user-friendly characteristics of SCC are of great attraction today in traditional construction industry as well. SCC has reached the status of being an outstanding advancement in the sphere of concrete technology with a promising future. There is an in-built assurance of uniform placement and fully consolidated concrete when SCC is used at site. This ensures high durability since air voids and other flaws are likely to be absent.

This new technology has been of interest to researchers, practicing engineers and several industrial sectors including the cement and admixture manufacturers due to the higher performance achieved in both fresh and hardened states, increased productivity, decreased labor requirements and improved working environment. With this revolutionary development, the construction engineer is now relieved of two annoying problems such as, the difficulty in ensuring thorough compaction employing unskilled labor, and the necessity to deal with repairs and making good finishes.

Time, cost and quality are the three important factors which assume significance in construction due to their impact on the industry as a whole. Any development, which has positive impact on the factors mentioned above are always of interest in Civil Engineering construction. When these developments have direct, desirable impact on social obligation of the industry, a thorough analysis of the pros and cons has to be done and all efforts needed to implement the concept in the field of construction are essential. The concept of SCC is one such tool to be seen with holistic view in the interests of modern construction industry in view of its advantages

Microstructure

The term macrostructure is generally used for the gross structure, visible to the human eye. The gross elements of the structure of a material can readily be seen, whereas the finer elements are usually resolved with the help of a microscope. The limit of resolution of the unaided human eye is approximately one-fifth of a millimeter (200 µm). The term microstructure is used for the microscopically magnified portion of a macrostructure. Microstructure is defined as the structure that is observed when viewed in an optical microscope or scanning electron microscope in the range of approximately 25 X to 2000 X magnification

At macroscopic level concrete is generally considered to be a two-phase material, consists of aggregate phase and hydrated cement paste. But at microscopic level, a third phase, the interfacial transition zone (ITZ), which represents the interfacial region between the particles of coarse aggregate and the hydrated cement paste may be identified. The microstructure of the interfacial transition zone is indicative of its mechanical properties and durability. Mehta and Monterio [1997] reported that ITZ exists as a thin shell typically 10 to 50 µm thick around large aggregate, the transition zone is generally weaker than either of the two main components of concrete, and therefore it exercises a far greater influence on the mechanical behavior of concrete than is reflected by its size.

Each of the three phases is itself a multiphase in nature. For instance, each aggregate particle may contain several minerals, in addition to micro-cracks and voids. Similarly both the bulk hydrated cement paste and the transition zone generally contain a heterogeneous distribution of different types and amounts of solid phases, pores and micro-cracks. A detailed investigation on the micro-structural characteristics of SCC is vital as the internal structures of hydrated cement pastes and ITZ in SCC gives more information about the strength and durability characteristics.

Scanning Electron Microscope (SEM)

SEM is now used extensively in material science and has many applications in cement and concrete petrography. An electron beam is formed from an electron gun, which is successively condensed by the projecting lens and the objective (magnetic lens) to a spot about 5-100 nm in diameter at the specimen plane. The entire system is tightly sealed so that when it is in operational state, the microscope column can be evacuated to about 1.33×10^{-4} Pa. A scan generator simultaneously drives the X- and Y-scan coils in the microscope column and in the cathode ray tube (CRT). Among the different types of responses produced, the secondary electrons (SE) and the backscattered electron (BSE) are collected by the photo- multiplier to form an image. The SE mode is dominated to topographic contrast, whereas the BSE mode is much more oriented to detect atomic density, which can be related to atomic number and density of grains forming the object.

Wong H.S. et al. [2006 a] characterized the pore structure of cement mortar with different water/cement ratio using quantitative backscattered electron imaging in terms of simple morphological parameters such as resolvable porosity and the specific surface area. A technique to segment pores from a normal backscattered electron (BSE) images has been reported by Wong. H.S. et al. [2006 b] to quantify porosity.

SEM is only one method of observation, and does not by itself provide a complete characterization of cement paste microstructure. The major limitation in SEM is that only a smaller portion of the surface exposed in a given specimen is usually documented with micrographs. Selecting and imaging the areas to be documented generally involves some interpretations of what is present; such interpretations may vary among different investigators. Nearly all backscatter SEM instruments are equipped with Energy-Dispersive X-ray spectroscopy (EDX) systems, which can almost instantly provide specific chemical compositional information on any desired spot or area in the image being observed. This feature helps make backscatter SEM an extremely powerful and informative technique in concrete investigations. Figure1 shows the Scanning Electron Microscope used in this investigation.

BSE images were taken at a relatively low magnification (approximately 500 x). It displays an area of about 40,000 μm^2. Images were taken for different samples of SCC and compared with that of conventionally vibrated concrete (CVC). Visual observation of different images based on the morphological characteristics derives the following inference. The bright white areas of different sizes represent un-hydrated cement grains and fly ash particles. Most of these particles are surrounded by, and are in close contact with, smooth-textured uniformly grey hydration product shells of varying thickness. These hydration shells appear non porous. Actually there is no guarantee that a grain that appears fully hydrated on the plane of observation is in fact completely hydrated; there may be residual un-hydrated core existing above or below the plane being imaged.

Figure 1: Scanning Electron Microscope

Experimental Investigations

The following materials were used in the experimental investigation based on the mix proportions given in Table 1.

1) Cement: Ordinary Portland cement (53 Grade) with specific gravity of 3.14 confirms to IS 12269:1987 (ASTM C 150-85A).
2) Fine aggregate: Locally available river sand of specific gravity 2.64, fineness modulus of 2.17, bulk density of 1320 kg/m3 which confirms to Zone II as per IS: 2386 (Part I).
3) Coarse aggregate: Crushed granite coarse aggregate of 12 mm down size with specific gravity of 2.79 and bulk density of 1480 kg/m3 confirms to ASTM C 33-86.
4) Water: Potable water confirms to ASTM D 1129, for mixing the concrete and curing of the specimens.
5) Fly ash: Class F fly ash obtained from Ennore Thermal Power Plant in Chennai with a specific gravity of 2.10 and fineness of 428 m2/kg determined as per IS 1727:1967 confirms to (ASTM C 618).
6) High range water reducing admixtures (HRWRA): Poly carboxylic ether (PCE) based super-plasticiser (SP) confirms to ASTM C 494-92 Type A and Type F in aqueous form to enhance workability and water retention.
7) Viscosity modifying admixture (VMA): A polysaccharide based VMA, to enhance segregation resistance, to improve the viscosity and to modify cohesiveness of the mix

Fresh and Hardened Concrete Properties

Different ingredients were batched by weight as per the mix proportions given in Table 1 and mixed well in a pan mixer of capacity 60 kg and the workability tests such as slump flow test, V-funnel test, L-box test and GTM screen stability test as per specifications were carried out to test the flowability, filling ability, passing ability and segregation resistance as per EFNARC specifications [2002]. Table 2 and 3 shows the fresh and hardened concrete properties of SCC. The workability properties are found to be within the prescribed limits as per specifications and guidelines given by EFNARC [2002], the European guidelines [2005] and Brite-Euram Project [1998]. It is observed that all the required rheological characteristics and self-compactability are within the permissible limit as per specifications. Standard cubes were tested for compressive strength after 28 days and 90 days of curing. Binu et al., [2008] evaluated and reported on the strength of self compacting concrete with high volume fly ash at early ages of curing.

Table 1: Mix proportions of SCC and CVC

Materials Used	Quantity Used in SCC	Quantity Used in CVC
Cement (kg/m^3)	250	385
Fly ash (kg/m^3)	275	-
Fine Aggregate (kg/m^3)	842	693
Coarse Aggregate (kg/m^3)	772	1232
w/p ratio	0.34	0.52
SP % of binder (HRWRA)	0.40	-
VMA % of binder	0.10	-

Table 2: Fresh concrete properties of SCC

Workability characteristics	Measured Values	Recommended limits
Slump flow (mm)	743	600-800
T50cm slump flow (s)	2	< 3 s
V-funnel flow at T_f (s)	4	< 6 s
V-funnel at T_{5min} (s)	5	$<T_f + 3$
L-Box T_{20}, T_{40} (s)	1.0, 1.5	1 ± 0.5, 2 ± 0.5
L-Box h_2/h_1	1.0	>0.8
GTM segregation ratio	6.0	<15

Table 3: Comparison of compressive strength

Compressive strength	**CVC N/mm^2**	**SCC N/mm^2**
28 days of curing	36.25	39.62
90 days of curing	41.53	65.37

Studies on SEM Images

Samples were prepared from cubes of different grades of SCC and CVC. Cubes of 1cm size were cut from different specimens of SCC using a concrete cutting machine. Samples were derived almost from the centre of cube specimens after 28 days and 90 days of curing. The samples were washed with acetone to stop further hydration before undergoing scanning electron microscope (SEM) studies. Cut surfaces of samples were polished using different grades of silicon carbide polishing paper with the help of a polishing machine. Polished specimens were subjected to SEM study in the backscattered electron mode (BSE). Totally, ten samples were analyzed.

The microstructure obtained from SEM images indicates the distribution of the hydrated and unhydrated cement paste along with the pores and void spaces. The interpretation is done through the intensity of grey levels exhibited by the constituents of the image. In backscatter SEM images the available range of black to white is customarily divided into 256 grey levels or shades of darkness. The lowest level, usually recorded as grey level 0, is fully black; the highest level, usually recorded as grey level 255, is fully white. One speaks of the darker areas on the image as areas of lower grey levels and conversely of the brighter areas as areas of higher grey levels.

From Figure 2 and 3, it is obvious that the unhydrated particles of the cement grains exhibit much higher grey levels than the hydrated components surrounding them. The hydration products found in most hardened Portland cement pastes or concretes primarily consists of C-S-H gel and calcium hydroxide accompanied by smaller amounts of ettringite. These hydration products are intermingled with pore spaces. The calcium silicate hydrate phase (C-S-H) makes up 50 to 60 percent of the volume of solids in a completely hydrated Portland cement paste and is, therefore, the most important in determining the properties of the paste. The morphology of C-S-H also varies from poorly crystalline fibres to reticular network. Significant contents of pore space can usually be detected by backscatter SEM in most cement pastes. The actual content depends mostly on w/c ratio and degree of hydration. It is represented as dark regions or spots in the SEM images. Analysis of the images was done using the image analysis software to quantify the content of detectable pore space in a given paste.

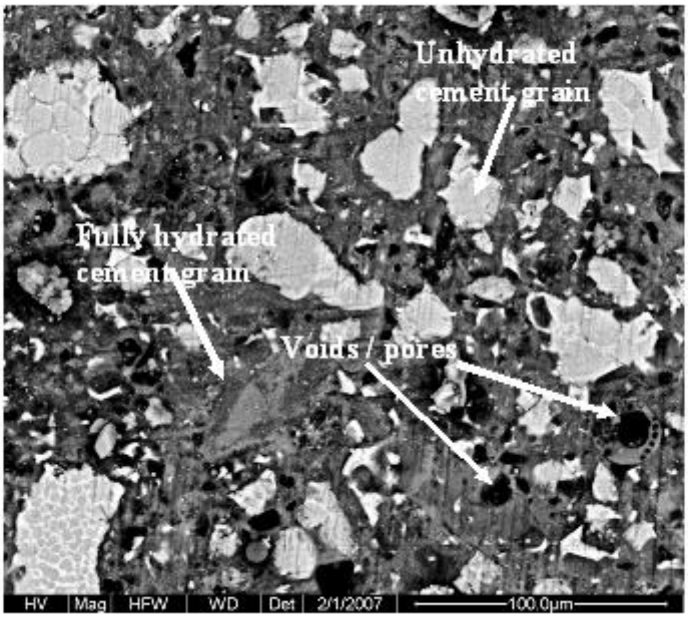


Figure 2: Micro-structure of CVC at 28 days of curing

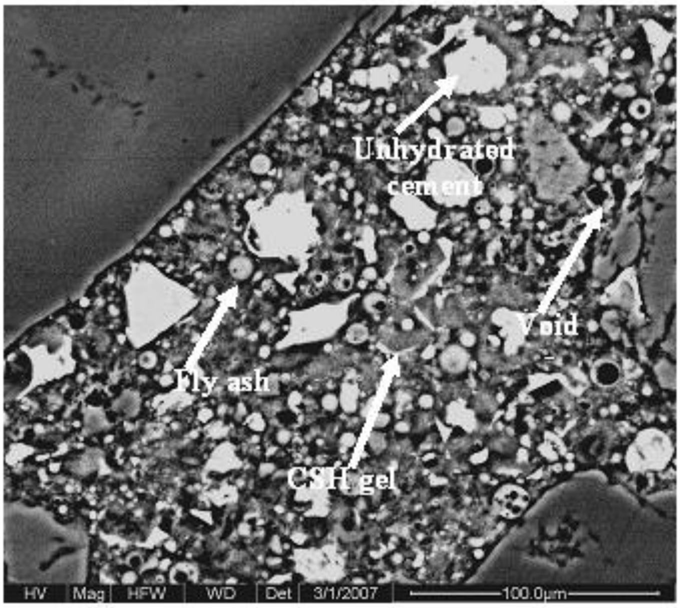


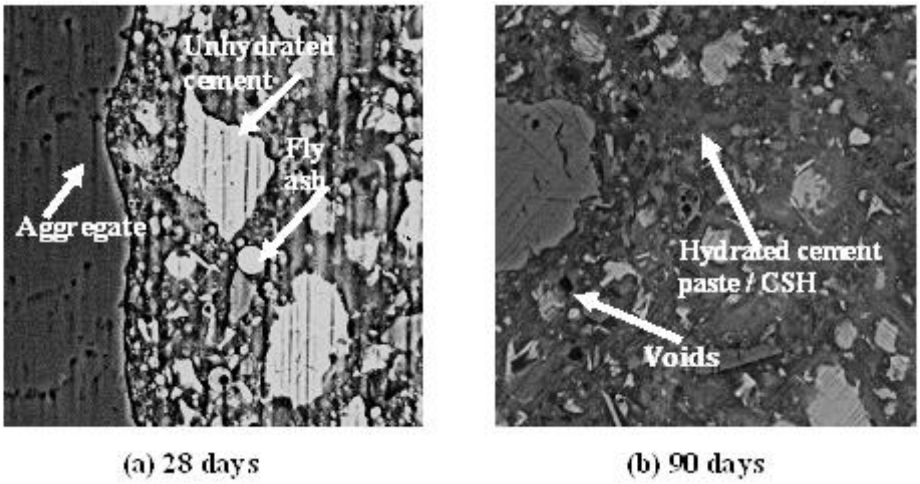
Figure 3: Micro-structure of SCC at 28 days of curing

Backscattered SEM Images

Backscattered mode SEM images were taken after 28 and 90 days of curing on the prepared samples of SCC and CVC. Figure 4 shows the images of SCC at 28 days and 90 days of curing. The photographs on the SEM images reveal the following details.

The images taken at 28 days (Figure 4a) of curing show larger percentage of white bands compared to the same taken at 90 days of curing (Figure 4b). This is due to the presence of unhydrated / partially hydrated cement or fly ash particles.

The inner part of the transition zone, between the aggregate surface and the C-S-H layer is rich in calcium hydroxide crystals. Un-reacted grains of fly ash are observed in this zone, instead of un-reacted cement grains usually encountered in normal concrete. The presence of high amounts of fly ash in SCC with a reduced cement content than that required for equivalent strength of CVC**,** leads to the absence of partially hydrated cement grains (Hadley Grains) in the transition zone.

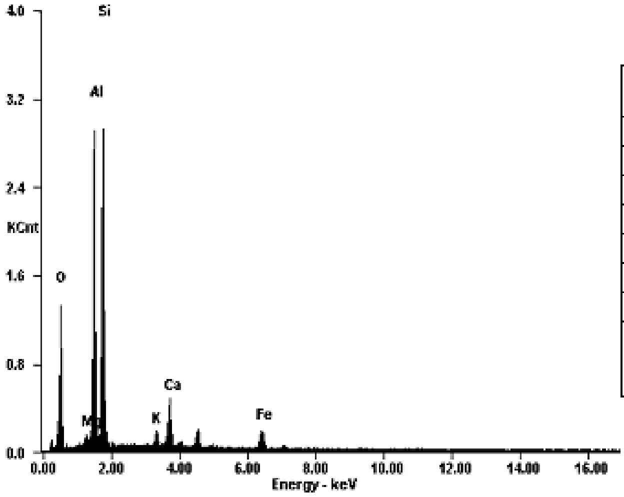


(a) 28 days (b) 90 days

Figure 4: SEM images on SCC

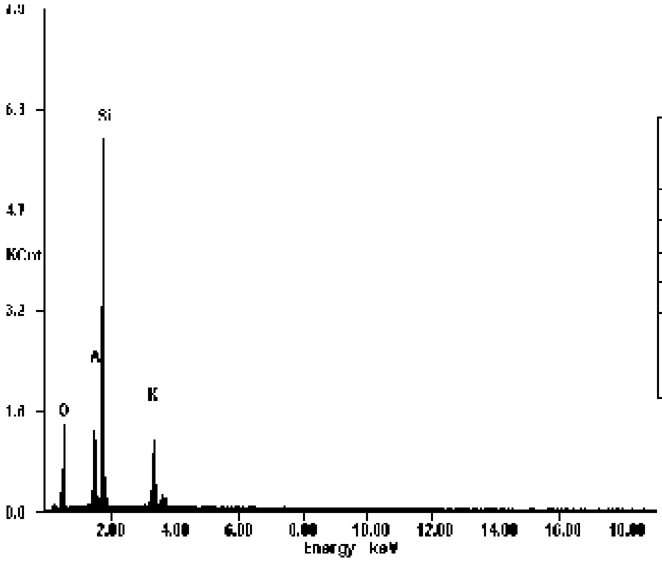
The presence of un-reacted particles of pozzolanic materials in ITZ is not harmful as compared to that of the un-reacted /partially reacted particles of cement, as there is no volume expansion associated with the possible reaction of fly ash grains at a later date unlike the "Hadley Grains" of cement. The strength of the interfacial zone in SCC is bound to increase with time as a consequence of a secondary reaction between the calcium hydroxide present in it and fly ash grains. The presence of fly ash particles in the ITZ densifies and reduces the porosity of this zone. The transition zone of SCC is free of micro cracks in contrast to normal concrete. This may be the reason for the higher long term strength of SCC with high volume fly ash compared to the same with low volume fly ash. Voids of more than one micron size are seen in ITZ of conventional concrete. It is found that the ITZ of conventional concrete is relatively porous near the aggregate surface than away from it.

The spot EDX (Energy Dispersive X-ray spectroscopy) on the white region in the image of SCC shows that most of the white region constitutes fly ash rather than un-reacted cement grains. The spot EDX on a small white region is shown in Figure 5(a). The element composition at the spot shows the presence of partially hydrated cement or fly ash. Figure 5(b) shows the spot EDX on a spherical particle. Element composition here confirms the presence of fly ash. Spot EDX on a larger white region is given in Figure 5(c). Element composition at that spot shows the presence of un-hydrated cement.



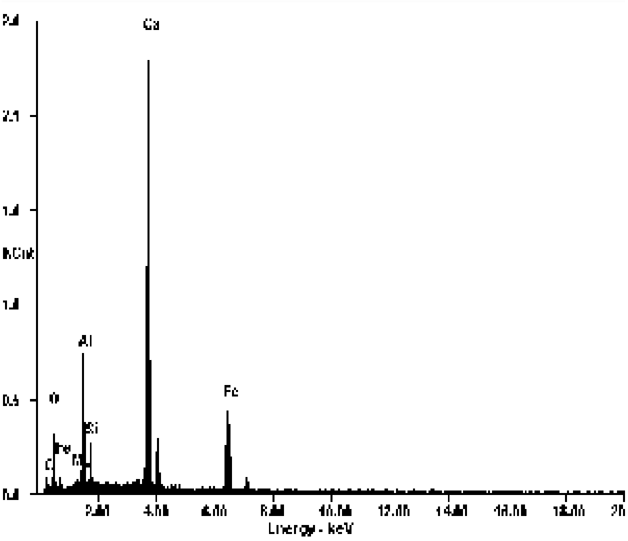
Element	Wt%	At%
OK	41.76	56.55
MgK	00.93	00.83
AlK	20.79	16.69
SiK	28.59	22.05
KK	01.30	00.72
CaK	03.84	02.08
FeK	02.78	01.08
Matrix	Correction	ZAF

Figure 5: (a) Spot EDX on a small white region



Element	**Wt%**	**At%**
OK	39.93	55.02
AlK	11.38	09.29
SiK	37.26	29.24
KK	11.43	06.44
Matrix	Correction	ZAF

Figure 5: (b) Spot EDX on a spherical particle



Element	Wt%	At%
CK	09.07	17.51
OK	31.44	45.60
MgK	01.77	01.69
AlK	11.24	09.66
SiK	03.43	02.84
CaK	29.45	17.05
FeK	13.60	05.65
Matrix	Correction	ZAF

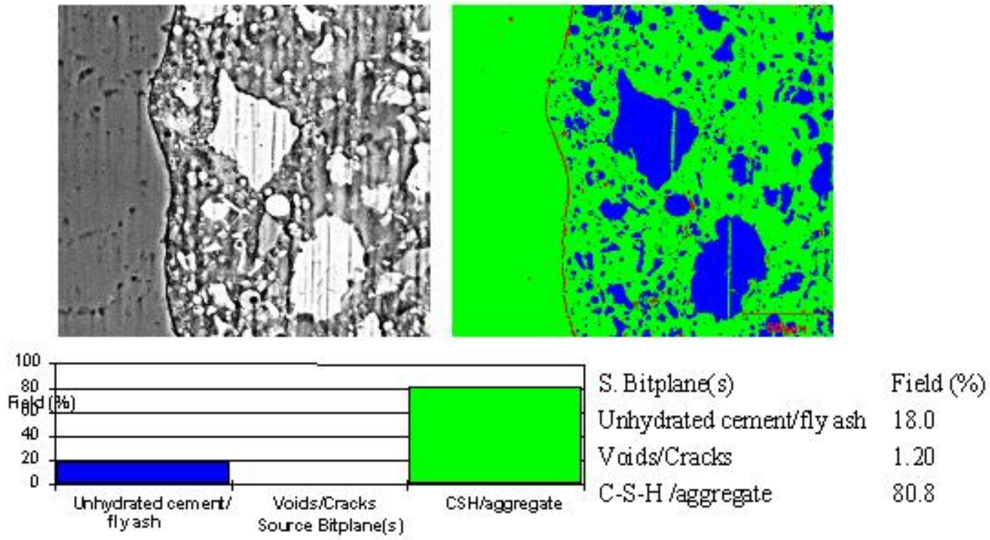
Figure 5: (c) Spot EDX on a larger white region

Image Analysis: Results and Discussion

A solid qualitative understanding of the internal features of the material is essential before the implications of quantitative studies can be meaningfully understood. SEM images obtained for different samples were analyzed using the image analysis software CLEMEX (version 4.0). All the above images were analyzed by assigning different colours for different regions. Green is assigned to C-S-H and aggregate, red for cracks/voids and blue for un-reacted / partially reacted cement grains and fly ash. The area occupied by each colour was represented as a percentage of the total area.

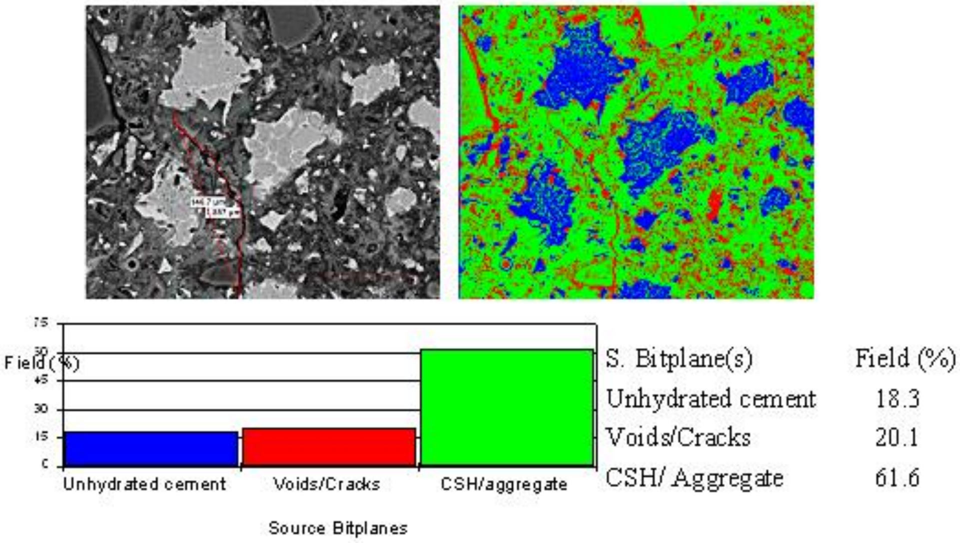
Figure 6(a) shows the images taken on SCC sample at 28-day curing, where un-hydrated cement grains and fly ash particles occupy an area of 18.0%, void space 1.2% and CSH/aggregate 80.8 % of the total field area. Figure 6(b) shows CVC at 28-day of curing in which, un-hydrated cement grains and fly ash particles occupy an area of 18.3%, void space 20.1% and CSH/aggregate 61.6 % of the total field area. Analysis shows that SCC contains very less voids/cracks at the order of 1.2% in comparison with CVC (20.1%). This is a clear indication of the better performance characteristics of SCC in comparison with CVC

When the results of SCC at 90 days (figure 6 c) is compared with 28 days, the area occupied by the unhydrated cement and fly ash particles is decreased to 2.6% with void space of 0.9%. Whereas the area occupied by C-S-H and aggregates is increased from 80.8% to 96.5% at 90 days of curing. It is understood that at the age of 90 days almost all cement and fly ash particles are converted to calcium hydrate gel which makes the concrete impervious and free of voids and cracks.



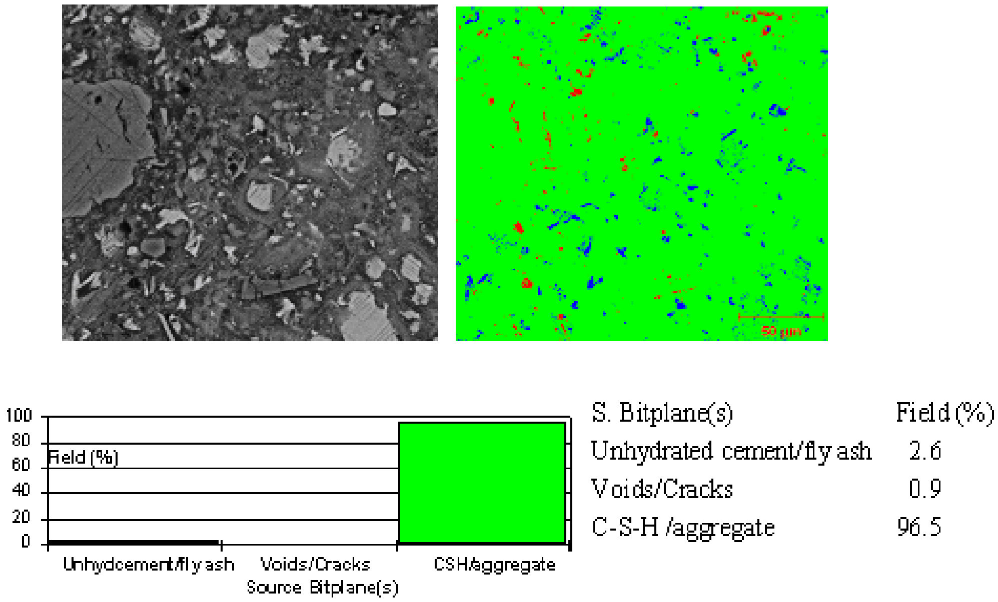
S. Bitplane(s)	Field (%)
Unhydrated cement/fly ash	18.0
Voids/Cracks	1.20
C-S-H /aggregate	80.8

Figure 6 (a): Result on the image analysis of SCC at 28 days



S. Bitplane(s)	Field (%)
Unhydrated cement	18.3
Voids/Cracks	20.1
CSH/ Aggregate	61.6

Figure 6 (b): Result on the image analysis of CVC at 28 days



S. Bitplane(s)	Field (%)
Unhydrated cement/fly ash	2.6
Voids/Cracks	0.9
C-S-H /aggregate	96.5

Figure 6 (c): Result on the image analysis of SCC at 90 days

In SCC an optimum combination of fly ash and cement that causes the reduction in the viscosity of the paste permitting better self-compactability resulting in lower voids and pores. This shows that the matrix is very dense and impervious even without the use of vibratory equipments for compaction. It is understood that the cement used in CVC is not fully utilized at the same time it is found to be highly porous even with the use of vibratory equipments for compaction.

Research Highlights

- A scanning electron microscope (SEM) based study was carried out to investigate the ITZ of SCC in comparison to the conventional concrete
- The microstructure of the interfacial transition zone (ITZ) in concrete is indicative of the mechanical and durability characteristics of concrete.
- Results on the analysis using scanning electron microscopy on samples of self-compacting concrete after 28 and 90 days of curing reveal the absence of voids and micro-cracks in the transition zone compared to the conventional concrete.
- It is observed that the higher cement content used in CVC is not fully utilized.
- The use of high volume fly ash in SCC, effectively replaces the cement content and fills the voids to form a thick non porous matrix.
- Long term strength of fly ash based concrete is more compared to concrete without fly ash.

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