“EFFECT OF SILICA FUME AND FLY ASH ON THE PROPERTIES OF SELF COMPACTING CEMENTITIOUS SYSTEMS”

EFFECT OF SILICA FUME AND FLY ASH ON SELF COMPACTING MORTARS

By

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ABSTRACT

Self-compacting concrete does not require manual compaction and is capable to flow under its own weight. Self-compacting concrete has found diverse applications in modern engineering practices including placements in heavily reinforced sections such as bridge piers, prestressed concrete members; high rise buildings especially beam column joints etc. Self-compacting cementitious systems afford uniform durability and compaction at sufficiently low mixing water content containing suitable SRMs to improve its response in both fresh and hardened concrete.

The principal aim of the study is to determine the suitability of powdered silica fume in “as packaged” condition without first sieving it through finest sieve. In addition, feasibility of inclusion of Fly ash (FA) and two grades of powdered silica fume namely Elkem SF (920) and SF (971) in 25 and 50 kg bags have been employed. The study commenced with the response of SCP systems and thereafter, SCM formulations were also studied.

Comparative study of results revealed that Silica fume if used in “as packaged” condition has higher water demand and requires higher super-plasticizer content (compared with those of FA systems) to achieve target flow of 30±1 cm. Mixing water content has been taken equal to water demand of each system to impart cost effectiveness and durability.

It is believed that inclusion of powdered and sieved SF in self-compacting cementitious systems generally enhances strength and improves microstructure, strength and durability. However, in this investigation FA based formulation gave much better strength results than those having “as packaged” SF. The reduction in strength can be attributed to agglomeration of SF particles prevalent in “as Packaged” condition. The agglomeration effect adds to the porosity of the system. It is recommended that powdered SF when used in HP cementitious systems must be firstly sieved or a high shear rate should be employed in the mixing regime. A part of experimental work of present study was also carried out in Technical University Bergakademie, Freiberg, Germany under DAAD sponsorship.

TABLE OF CONTENTS

Chapter-1 INTRODUCTION

Chapter-2 LITERATURE REVIEW

Chapter-3 EXPERIMENTAL PROGRAM

Chapter-4 RESULTS

Chapter-5 DISCUSSIONS

LIST OF NOTATIONS

ASTM	American Society for Testing and Materials
FA	Fly Ash
SF	Silica Fume
C_2S	Di – Calcium Silicate
C_3A	Tri – Calcium Aluminate
C_3S	Tri – Calcium Silicate
C_4AF	Tetra – Calcium Alumino Ferrite
CH	Calcium Hydroxide
CSH	Calcium Silicate Hydrate
HPC	High Performance Concrete
MIP	Mercury Intrusion Porosimetry
OPC	Ordinary Portland Cement
SCC	Self- Compacting Concrete
SCCS	Self -Compacting Cementitious Systems
SCM	Self -Compacting Mortar
SCP	Self -Compacting Paste
SP	Superplasticizer
w/c ratio	Water Cement Ratio
w/p ratio	Water Powder Ratio
XRD	X – Ray Diffraction

LIST OF TABLES

LIST OF FIGURES

CHAPTER-1 INTRODUCTION

1. INTRODUCTION:

1.1 Self -Compacting Concrete (SCC):

Self-compacting Concrete (SCC) depicts the most influential and vibrant innovation in concrete technology. This type of high performance concrete is being used in modern engineering practices since late 1980's [1, 2]. Self- compacting concrete is considered as the concrete which can be placed and compacted under its own weight without segregation and bleeding while maintaining homogeneity. Compaction and vibration is not possible when congestion of reinforcement is encountered.

The European Guidelines for Self Compacting Concrete [3] defines SCC as "the concrete that is able to flow and consolidate under its own weight, completely fill the formwork even in the presence of dense reinforcement, whilst maintaining homogeneity and without the need for additional compaction." ACI 237 defines SCC a highly flowable, non-segregating concrete that can spread into place, fill the formwork, and encapsulate the reinforcement without any mechanical consolidation. It is a kind of high performance concrete (HPC) that is highly fluid, able to consolidate under its own weight and is more durable, stronger and easy to place in congested reinforcement.

The highly flowable nature of SCC is achieved by careful mix proportioning and reducing the content and size of coarse aggregate content by fines and chemical admixtures. Moreover, an optimum balance must also be attained between plastic viscosity and yield stress while ensuring stability and durability.

1.2 Supplementary Cementitious Materials:

Supplementary cementitious materials (SCMs) also called mineral additives contribute to the properties of fresh and hardened concrete through nucleation, filler and chemical pozzolanic activity. Typical examples are Silica fume, Fly ash, Metakaolin, Marble powder etc. which are used in concrete usually as cement replacement. SRMs possess in themselves little or no cementitious value but will in finely divided form and in the presence of moisture react with cement to form compounds possessing cementitious properties. Pozzolanic SRMs react chemically with calcium hydroxide released from the hydration of Portland cement to form cement compounds.

Secondary raw materials contribute to the properties of hardened concrete through physical and chemical processes including hydraulic or pozzolanic activity. They can be used individually as well as in different combinations to impart volume stability, enhance transportation properties, reduce permeability, increase strength, improve microstructure etc. Moreover, replacement of cement content and the use of industrial by products like silica fume, fly ash etc. leads indirectly to lower CO_2 emissions during cement manufacture and makes concrete environment friendly.

Addition of SRM's helps in the advancement of hydration process and especially on the improvement of hydration products [4]. SRM's particle size, shape, surface morphology, internal porosity has a direct influence on flow behavior, strength, durability and microstructure of concrete. One should be very careful in the selection of type and amount of SRM to be used since they have positive as well as negative impact upon strength development, water demand, heat of hydration, shrinkage behavior, volume stability and other properties of concrete.

1.2.1 Silica Fume:

Silica fume is an industrial by product, attained during the production of Silicon, ferrosilicon and other silicon alloys by reduction of quartz with coke or coal at very high temperatures (2000^{o}C) in electric arc furnaces. Silica fume is a fine powder, which comes in various forms including slurried SF, densified SF, undensified SF and pelletized SF. One of the most beneficial uses for silica fume is in concrete. Because of its chemical and physical properties, it is a very reactive pozzolan. The pozzolonic activity is due to the reaction between silica of SF and calcium hydroxide produced due to hydration of cement.

Silica fume has high surface area due to which water demand of concretes containing silica fume increases with the increase in silica fume content [4]. Moreover, silica fume can be used as an anti-bleeding agent. Silica fume can impart in concrete, resistance to sulphate attack, improve microstructure, control alkali silica reaction, enhance durability and high early strength development provided careful proportioning is done.

In the current study, silica fumes of grades 971-U and 920-U of Elkem Microsilica, Germany have been employed at TU Bergakademie Freiberg and at NUST. The term **U** corresponds to undensification. An undensified powder has a relatively low bulk density and often appears quite lumpy. The densification process increases bulk density of microsilica by producing spherical agglomerates.

1.2.1.1 Silica Fume Grade 971- U:

Elkem microsilica grade 971 is a standard high quality grade offered to the refractory industry. It is light grey in colour. It is globally available both in densified and undensified forms. Particle size (d50) of silica fume 971-U was found out to be 11.61 µm by laser Granulometry though this technique does not give the correct particle size due to agglomeration.

Table – 1.1 Standard specifications of Silica Fumes.

Analysis/ Properties	SF(971)	SF (920)
Silica (SiO_2)	98.4%	92.09%
Carbon (C)	0.50%	
Iron oxide (Fe_2O_3)	0.01%	0.50%
Alumina (Al_2O_3)	0.20%	0.70%
Calcium oxide (CaO)	0.20%	0.50%
Magnesium oxide (MgO)	0.10%	0.50%
Potassium oxide (K_2O)	0.20%	0.70%
Sodium oxide (Na_2O)	0.15%	0.42%
Chloride (Cl)	0.01%	0.02%
H_2O (Moisture) (when packed)	0.20%	0.40%
Loss on ignition (L.O.I.) @950^oC	0.50%	1.60%
Coarse particles > 45 µm	0.20%	4.70%
pH- value	6.8	6.8
Bulk density (when packed)	300 kg/m^3	200-350 kg/m^3

1.2.1.2 Silica Fume Grade 920– U:

Silica fume of grade 920-U conforms to the mandatory requirmements of **ASTM C1240**. It is also globally available both in densified and undensified forms. It is dark grey in colour. Particle size (d50) of silica fume 920-U was determined as 11.27 µm. The standard specifications are given in table 1.1.

1.2.2 Fly Ash:

Fly ash is a residue obtained from the combustion of pulverized coal in furnaces of thermal power plants. The characteristics of fly ash vary according to combustion operation system as well as coal composition. The major chemical constituents in fly ash are silica, aluminum and oxides of calcium and iron. The pozzolonic activity of fly ash is largely due to glass content which reacts with lime in the presence of water. Specific gravity of fly ash generally ranges between 1.9 and 2.8 and its color is grey. Fly ash benefits the rheological properties of concrete because of its spherical shape. It also improves the filling ability of concrete but leads to low early strength. Moreover, inclusion of fly ash in concrete depresses its setting time.

Fly ash is classified to Class C, Class N and Class F as per ASTM C - 618. If oxides of silica, iron and alumina exceed 70% then the ash is categorized as Class F and they belong to the burning of anthracite coal and bituminous coal. In Class C sum of all the oxides exceeds 50% and it basically originates from sub-bituminous coal. Raw or calcined natural puzzolans, volcanic ash, calcined kaolin clay, laterite, shale etc fall in the category of Class N fly ash [4].

Table – 1.2 Standard specifications of Fly Ash (ASTM C – 618)

Analysis/ Properties	Typical
Silica (SiO_2)	51.44%
Sodium oxide (Na_2O)	1.23%
Potassium oxide (K_2O)	2.63%
Magnesium oxide (MgO)	2.51%

Iron oxide (Fe_2O_3)	5.55%
Loss on ignition (L.O.I.)	2.71%
Calcium oxide (CaO)	4.03%
Phosphorous Oxide (P_2O_5)	1.89%

1.2.3 Pozzolan:

As per ASTM C 125 [8], Pozzolan may be defined as “A siliceous or siliceous and aluminous material, which in itself possesses little or no cementitious value but will, in finely divided form and in the presence of moisture, chemically react with calcium hydroxide (CH) produced by cement hydration at ordinary temperatures to form compounds possessing cementitious properties.

Ordinary Portland cement when mixed with water forms calcium silicate hydrate (CSH) gel along with calcium hydroxide and ettringite. However, when a pozzolan is present, its silica component reacts with liberated calcium hydroxide in hydrated cement paste in the presence of water to forms another form of calcium silicate hydrate. The reaction is generally slow, resulting in a slow rate of heat liberation and strength development. In addition to reactive silica, pozzolans also contributes reactive alumina, which in presence of calcium hydroxide present in the system, forms cementitious products such as tetra calcium aluminate hydrate (of the form C_4AH_x, in which x varies from 9 to 13), tricalcium aluminate hydrate (C_3AH_6), dicalcium aluminate silicate hydrate (C_2ASH_8), calcium carboaluminate ($C_3A.CaCO_3.H_{12}O$), high sulfoaluminate ettringite ($C_3A.3CaSO_3.32H_{12}O$) and low sulfoaluminate ($C_3A.CaSO_3.12H_{12}O$) [5,6].

1.3 Research Objective:

The research focuses on the influence of use of “as packaged” powdered SF of grades 971-U and 920-U and FA on self- compacting paste and mortar systems to find out possible response differences in comparison to use of sieved powdered SF . It is worth mentioning here that as per literature inclusion of SF even in replacement mode generally enhances the strength as well as improves microstructure, which is subject to its fineness.

CHAPTER- 2 LITERATURE REVIEW

2. LITERATURE ON SCCS:

The work focuses on compatibility and suitability of SRM's on self-compacting paste and mortars systems. In this chapter a comprehensive overview is being provided regarding work done by various researchers in the field of self- compacting concrete using silica fume and fly ash as secondary raw materials.

2.1 Self- Compacting Cementitious System:

Successful production and application of self-compacting concrete necessitates optimization of paste component since mechanical properties, volume stability and durability of mortars and concretes greatly depends upon the durability and compatibility of paste component.

Self compacting concrete was first developed in Japan and Prof. H. Okamura of Tokyo University was the first to achieve the necessity of development of SCC after considering problems in concreting such as the lack of uniformity of compaction resulting in inadequate compaction of concrete and non availability of skilled workers. After which extensive studies have been done to develop self compacting concrete at the university of Tokyo, Japan, in 1980's and by early 1990's Japan had formulated as well as applied the concrete which did not necessitate any vibration to achieve full compaction and flowed under its own weight [7,8]. Khayat [9] states that "Self compacting concrete is a special type of concrete that should flow into place and around obstructions under its own weight without segregation and flow blockage and with no significant separation of material constituents thereafter until the setting". Ulucan [10] defines "Self compacting concrete as a concrete that has excellent deformability and high resistance to segregation and can be used to fill a heavily reinforced area without applying vibrations". Successful and flawless production and application of self compacting concrete necessitates optimization of paste component. Emergence of SCC diminished the necessity of any kind of compaction, leading to reduced labor cost and thus affording cost effective construction [3] in developed countries. Also it imparted ease of flow around the congested reinforcement while maintaining high level of homogeneity with uniform concrete strength and durability of structure. Moreover, it provided superior level of finish, easy de-molding and reduced exposure of workers to noise and vibration at construction sites. In addition, its uses also include light weight reinforced concrete, pre-placed aggregate concrete and for rapid repairs and constructions.

Since the technology is constantly being analyzed and developed, numerous studies have been made on self compacting cementitious systems. Few significant, fundamental and imperative studies can be summarized as follows:-

Rizwan [11] reported that shape, size, surface morphology and porosity of secondary raw materials play a very significant role in determining the water and super plasticizer demands of the system. Also physical properties, the chemical composition of the secondary raw materials is of great significance. An example is that of addition of rice husk ash and silica fume in same proportions to fly ash based self compacting mortar formulations. A significantly low super-plasticizer demand is anticipated by the formulation containing 20% rice husk ash than the one containing silica fume. Thus suitable blends of secondary raw materials can provide an improved overall response of self compacting mortar systems using well graded aggregates. The study also suggests the relation between viscosity and yield stress in a way that T25 cms time is thought to be a function of viscosity of a system, whereas the total spread of mini slump cone is a function of the yield stress of such systems. Also high performance self compacting cementitious system is dependent upon shrinkage of first 24 hours.

Thixotopy can be defined as the property exhibited by certain gels of becoming fluid when stirred or shaken and returning to the semisolid state upon standing**.** Khayat [12] reports that self compacting concrete can exhibit a greater degree of thixotropy arising from the inclusion of a set accelerating admixture and higher binder content which promote a faster development of cohesion resulting from cement hydration. In addition, an increase in w/c ratio and lower coarse aggregate volume reduces the degree of internal friction, thus contributing to the ability of concrete to flow through congested reinforcement. Also it is important to note that increased water content can reduce the cohesiveness and viscosity of mix.

Rizwan [13] reports that "paste serves as the vehicle for transport of aggregate phase. Increased paste volume is beneficial in reducing internal friction. Also good workability can be achieved by reducing the aggregate content." Further it is recommended that the sand content may be considerably increased to impart cohesiveness and stability to the concrete mix. This may also result in higher requirement of super plasticizer content to meet the selected flow target. Regarding the aggregate requirement, the study specifies that fine aggregate content should be restricted to 50% of the total aggregate content as it may lead to enhanced internal friction, bigger voids and bleeding, thus necessitating a higher paste volume. The study also describes the effect of water temperature while mixing, on the flow response of self compacting cementitious systems and suggests that any addition of even small quantity of water after the chemical admixtures have been added could significantly reduce the mix cohesion and could yield slightly inaccurate results which appears contradictory to the literature [3]. Brouwers [14] reports that a sand of about 50% of the total aggregate mass has been found satisfactory for enhancement of certain properties of self compacting concrete especially stability.

Hence, self compacting concrete is characterized with higher powder content, low w/p ratio and use of super plasticizer to produce an adequate paste to act as lubricant for the aggregate phase [2].

2.2 Supplementary Raw Materials:

The environmental aspects associated with the production and application of cement, concrete and other building materials are of utmost importance. The manufacture of cement leads to the production of 0.8-1.3 ton of CO_2 per ton of cement produced making it highly energy intensive and environment unfriendly. Necessary steps are being taken to formulate replacements of cement, thus leading to the inclusion of secondary raw materials (SRM’s). Since secondary raw materials are industrial by products and they also reduce the cement content hence they are beneficial in producing cost effective and environment friendly concrete. Such environmental improvements have also been reported using blends of fly ash and bottom ash in self compacting cementitious systems [15].

According to Cordeiro [16], capability of pozzolanic materials to enhance the strength of concrete includes the physical as well as chemical effects. The physical effects are dependent upon size, shape and texture of the particles and influence the packing characteristics of the mixture. The chemical effects are associated with chemical reaction with calcium hydroxide in presence of water. Few other studies [17, 18] suggest that the physical effects of fly ash and silica fume contribute towards the strength development upto 28 days. Thereafter chemical effects become significant. Tangpagsit [17] reports that the smaller particle size of pozzolanic materials tends to produce higher compressive strength than the coarse one. SRM’s are basically incorporated in concrete to reduce WD and shrinkage and at the same time to impart volume stability and ease of placement. Hence complete understanding and appropriate selection of SRM for a desired placement must be ensured. Rizwan [4] suggested that in addition to cement grains, secondary raw material particles also adsorb super plasticizer and water. The internal powder pores of bottle neck type usually result in higher super plasticizer demand.

Cyr [20] reported that an optimum efficiency and a definite degree of enhancement in the compressive strength is attributed to a minimum inclusion of SRMs in self compacting cemenntitious systems. Hence, in the present study SRMs are incorporated at 10 % replacement of cement mass.

2.2.1 Fly Ash:

Rizwan [4] suggested that particle characterization of fly ash revealed that its particles are of spherical shape, glassy texture and have small circular voids. It can be expected that fly ash would require minimum super-plasticizer dosage because whatever is added remains available in the solution for dispersion of cement grains. Also combination of high value of maximum pore size and low mercury intruded volume may lead to lesser super-plasticizer demand to achieve target flow. Increase in effective water content for given mixing water is the reason for decrease in the yield stress. Fly ash also reduces shrinkage of self compacting cementitious systems. In covered conditions expansion is encountered which is due to the formation of expansive species like calcium hydroxide. Also fly ash retards hydration by depressing calcium ion concentration during early age. Carbon content in fly ash would also have a retarding effect. SEM of fly ash SCM systems at 28 days of strength reveals the phenomenon of ‘nucleation’ and deposition of hydration products on fly ash particles, which along with filler effect are key factors for improved strength. Pozzolonic activity of fly ash gets activated after 28 days thus mixes containing fly ash show rapid increase in compressive strength after 28 days.

Slump flow test and V-funnel tests have revealed that fly ash systems show better flowability and deformability [21]. The spherical shape of fly ash particles and their fineness have beneficial effects on workability. Also fly ash prolongs initial and final setting times, which kept on increasing by increasing fly ash content.

Yield stress of the mix increases as the fly ash content increases [22]. Moreover, spherical shape of fly ash particles reduces the frictional force among the angular particles of ordinary Portland cement, thus contributing flowability to the mix.

The present work focuses on 10% replacement of cement by fly ash in SCP systems. However, increased percentages of fly ash have also been used [24]. In such cases compressive strength increased with the decrease in fly ash content. Also by increasing the amount of fly ash, reduction in shrinkage is observed. Moreover, influence of increasing percentages of fly ash on shrinkage capacity of the mixes is also suggested by Khatib [25].

2.2.2 Silica Fume:

Because of extreme fineness and very high amorphous silica content, SF is a very reactive pozzolonic material. Rizwan [4] suggests that when SF is incorporated in the mix, there is corresponding increase in the water demand of the system. Also due to high internal porosity and large surface area, mixes containing SF have the highest super-plasticizer demand to achieve target flow. Moreover, MIP results show that SF has very small size of internal pores with very high mercury intruded volume. It has also been reported that higher SF replacement levels will require more super-plasticizer content to achieve target flow [26]. Also, as the proportion of SF in the system increases, autogenous shrinkage increases with a corresponding decrease in the drying shrinkage. The same effect on shrinkage with the increase in SF content has also been reported in the literature [24]. Moreover, it is also suggested that SCC with SF exhibits satisfactory results in workability because of small particle size and large surface area.

Effect of increased dosage of SF on plastic viscosity and yield stress has been reported in the literature [22]. According to which, there is an abrupt increase in yield stress and plastic viscosity with the corresponding increase in SF content. Enhanced concrete strength due to inclusion of SF in the mix is reported in the literature [23]. Also improvement in microstructure and antibleed properties is associated with the pozzolanic, filler and nucleation effects of SF.

As far as use of SF in SCM systems is concerned, it is not used alone keeping in view that higher level of SF replacements require higher dosages of supeplasticizer at a relatively low w/c ratio. So it has been used in combination with FA in SCM in order to depress the super-plasticizer demand for the target flow. Rizwan [4] used SF in combination with FA (by 20% mass of FA) and suggested that by doing such improvisation super-plasticizer demand increased when compared to the formulation containing FA only. Also, it gives the lowest viscosity and yield stress which is due to compatibility of their shapes and highly variable sizes which enhance their internal packing and result in reduced internal friction. Moreover, an increased shrinkage is encountered, since hydration process is accelerated due to inclusion of SF, thus releasing heat faster and leading to high early shrinkage.

It has been reported in the literature [27] that inclusion of SF alone, in SCM systems for more than 30% by mass of cement results in very high super-plasticizer demand for target flow, thus leading to cost ineffectiveness. Also, in the same literature, SF has been used in combination with FA leading to lower super-plasticizer content. Moreover, it can also be used in varying proportions with other SRM’s namely ground granulated blast furnace slag (GGBFS) [22].

2.3 Shrinkage:

Rizwan [4] suggests that the most important factor after flow is volume stability. Also addition of SP reduces early shrinkage in uncovered conditions due to decrease in surface tension. Moreover, shrinkage recovery is encountered after about 8 hours due to thermal expansion corresponding to heat of hydration and formation of expansive species such as calcium hydroxide and rod shaped ettringite crystals. Addition of SRM’s in the mix increased shrinkage for the first 24 hours due to consumption of water by these SRM’s. But when increased water cement ratio has been used, shrinkage reduces due to dilution of cement content. Since SF accelerates hydration and setting processes leading to rapid release of heat, thus resulting in high early shrinkage. On the contrary, system containing FA gives the lowest shrinkage due to delayed setting. The 24 hour value of shrinkage depends upon evaporation rate, specimen surface exposure conditions, type of cement and its contents, mixing water, admixture content, temperature, relative humidity and internal crystals growth. Controlling or minimizing shrinkage at the paste level may ensure satisfactory performance of resulting mortars and concretes.

In cement based composites, cement paste is the major source of shrinkage [28]. Also, in cement pastes having low w/c ratio, an increased shrinkage is encountered due to reduction in internal relative humidity by inclusion of SRM’s especially SF [29]. Moreover, it has also been suggested by Morin that increase in super-plasticizer content is accompained with increase in shrinkage and retardation in chemical activity [30].

2.4 Calorimetry:

Calorimetry was done to estimate the amount of heat released as well as influence on the hydration kinetics of the system by incorporating secondary raw materials. Water content was taken equal to the water demand of the system. Moreover, super-plasticizer content required to achieve target flow was used to determine the hydration kinetics of SCP and SCM systems with and without inclusion of silica fumes and fly ash, extending upto a period of 72 hours.

2.5 Studies of Microstructure:

2.5.1 Scanning Electron Microscopy (SEM):

Rizwan [7, 18] suggests that scanning electron microscopy is a versatile tool for study of microstructure, interfacial transition zone (ITZ) and hydration products including calcium silicate hydrate, calcium hydroxide and ettringite. Calcium hydroxide has influence on strength and durability of cementitious systems. The quantity of calcium hydroxide produced depends upon the water content, available space, cement clinker type and its content. Higher mixing water and clinker content give higher calcium hydroxide content. The addition of pozzolanic secondary raw material in the system reduces the calcium hydroxide content compared with plain cement formulation due to dilution of clinker content and simultaneous pozzolanic reaction of secondary raw materials. Being a weaker crystal, calcium hydroxide generally reduces the strength and durability of the concrete composite. It is important that its content should be reduced to improve the performance of cement based systems. The calcium hydroxide crystals can be detected after about 8 – 10 hours of water mixing and about 90% of total calcium hydroxide is in place after the end of first 24 hours.

2.5.2 Mercury Intrusion Porosimetry (MIP):

Rizwan [4] reported that samples up till 7 days can give sufficient details regarding pore structure refinement of Portland cement based formulations by mercury intrusion porosimetry since after three days there is only a very little improvement in the pore structure refinement. It was also reported that most of the porosity of cement-based systems relates to the paste phase and the aggregate phase possesses little porosity and offers internal resistance to shrinkage.

Diamond [31] suggests that the mercury intrusion porosimetry cannot provide a true pore size distribution as the pore system in concrete is not cylindrical (as assumed by Washburn equation model) as well as continuous. It also has the problem of accessibility of interior pores from the outer surface. Then there is contribution of air voids which magnifies the results and finally, the mercury must pass through the narrowest pores connecting the pore network, to get an accurate data. The threshold pore width, however, may provide a better indicator of material durability as it has an important influence on the permeability and diffusion characteristics of cement paste.

Cook [32] stated that increased curing time and decreased w/c ratio, results in lower total porosities, as it is widely accepted that as hydration proceeds, hydration products grow into the pore spaces of a hardened cement paste. In addition the rate of degree of hydration decreases with decrease in w/c ratio, however for all w/c ratios around 35% degree of hydration is achieved during initial 24 hours and thereafter the rate varies according to w/c ratio. Moreover, the significance and importance of first 24 hours for shrinkage measurement of SCC is also observed and reported by Rizwan [11].

Maintaining lower water contents while achieving an acceptable level of workability, results in higher strengths for given cement content. Also it imparts lower permeability and reduced shrinkage to the mixes [33]. However, Laskar et al [34] recommends the minimum number of tests must be conducted to estimate the values of mercury intrusion porosimetry parameters for an accuracy of 95%, basing on certain statistical formulas supported by the experimental data.

CHAPTER- 3 EXPERIMENTAL PROGRAMME

3. EXPERIMENTAL PROGRAMME:

3.1 General:

Some of the experimental work pertaining to this study has been performed in sophisticated laboratories of Technical University Bergakademie, Freiberg, Germany, affording temperature controlled environment in order to impart authenticity to the results attained.Some work was also done at NICE Labs of NUST.

In depth description is being provided in this portion, regarding the equipments employed for experimental work as well as the methods adopted.

3.2 Investigation on SCP & SCM Systems:

3.2.1 Materials for SCP Systems:

CEM 1 42.5 R (Lafarge, Germany), in accordance with ASTM C150 [35], was used in the experimental work. Both grades of Silica Fume have been attained from Elkem Microsilica, Germany. Fly Ash was obtained from Opole, Poland. The super-plasticizer used in the experimental work was powder type third generation PCE based, Melflux 2651 F. Mixing water content in all the formulations was as per the water demand (WD) of the systems which is well below 0.44, conforming to the literature [36]. Fly Ash and both grades of Silica Fumes are incorporated at 10% of cement mass in replacement mode. Both grades of SF were used in the fresh “as packaged” state, as were procured from the supplier to determine variation in their response in comparison to that of sieved powdered SF.

3.2.2 Materials for SCM Systems:

SCM systems comprised of CEM I 42.5R (Lafarge, Germany) and sand used was Roba sand 0-2mm size with fineness modulus of 2.39. Super-plasticizer employed was PCE Type Melflux 2510L with 45.7% total solids content. SCM systems had 1:1:2 mix proportions (cement: SRM: sand) by mass with 40% w/c and 20% w/p ratio. Such a low w/p ratio was considered to ensure adequate segregation resistance. SRM’s used were FA and 10% mass blends of FA with both grades of SF namely 971 & 920.

3.2.3 SRM Shapes:

The surface morphology, shape and size of secondary raw materials are very important in terms of water demand, SP demand, shrinkage and strength development in cement based composites. Scanning electron microscopy was executed to attain shapes of SRM’s. Particle shapes of SF and FA are expected to be circular. In case of SF, particles are of submicron size if viewed through SEM and BET d50 measurement gives the size of primary agglomerates as SF particles are interwound and interpenetrated due to some electrical bonds. Due to extreme fineness and high internal porosity, high super-plasticizer demand to achieve the target flow can be anticipated. Moreover, in case of fly ash, particles are of spherical shape, glassy texture and have small circular voids. Also super-plasticizer dosage for fly ash would be minimal because whatever is added remains available in the solution for dispersion of cement grains.

3.2.4 Water Demands & Setting Times:

Water demand calculation is a pre-requisite to all the experiments in formulation of self compacting cementitious systems which is coherent to the literature [3]. For a durable self compacting concrete mix, it is important that the water content should not significantly exceed the water demand of the system.

Water demands and setting times of all the formulations have been determined by standard Vicat apparatus (Fig. 3.1) at 20 ± 1^{O} C and at 60 ± 5 % RH in laboratory in accordance with EN 196-3: 1994 D. Due to high pozzolonic activity, fineness and large specific area of Silica fume, an increase in water demand has been observed when it is added in the mix. Moreover, it is also observed that an increase in SF content results in the corresponding increase in the water demand of the system [36]. Also, an early setting is achieved due to high heat of hydration resulting from inclusion of SF. A contrasting behavior is encountered when FA is incorporated in the cement based composites. An appreciable increase in setting time has been observed in FA systems due to glassy texture and less water absorption tendency leading to delayed hydration as compared to SF.

Fig. 3.1 Automatic VICAT apparatus used to determine Initial & Final setting times

3.2.5 Flow Measurements:

Flow measurements of self compacting paste systems were obtained by determining super-plasticizer content required to achieve a target spread of 30±1 cms using Hagermann cone (fig. 3.2) at respective water demands determined earlier, using Vicat apparatus. In case of self compacting mortar systems, super-plasticizer content was first attained using Hagermann cone and then flow times were calculated using as well as V-funnel as shown in fig. 3.3.

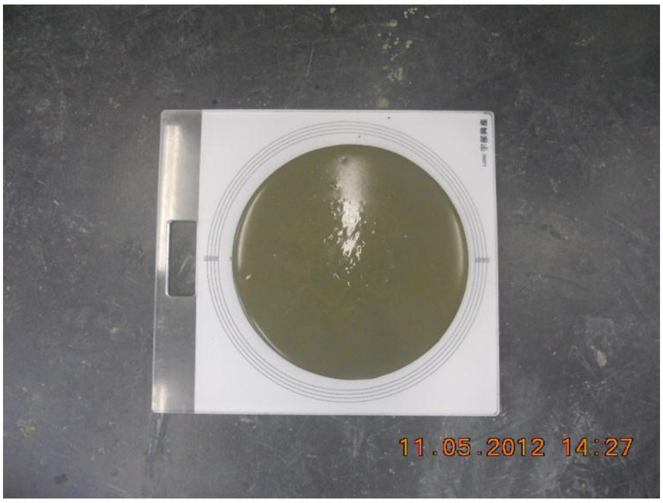

Fig. 3.2 Target spread of 30±1 cms achieved using Hagermann cone.

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Fig. 3.3 V-funnel apparatus used for determining flow times of SCM formulations.

Hagermann cone had an upper diameter equal to 70 mm, a lower diameter equal to 100 mm with a height of 60 mm. Also V-funnel consisted of a rectangular cross section with top dimension as 270 x 30 mm and bottom opening as 30 x 30 mm. The total height of funnel is 315 mm with a 75 mm long straight bottom section with a gate.

Methodology adopted involves dry mixing of the constituents thoroughly. Thereafter required water content is introduced in the mix and fed into the mixer. Mixer employed for mixing was Hobart Toni Technik as shown in fig. 3.4. First of all, slow mixing at 145rpm was done for 30 seconds and after cleaning interior of the bowl, fast mixing at 285rpm was excuted for the next 150 seconds. Mix obtained is filled in Hagermann cone. After lifting it, time required by the mix to reach 250 mm and 300±10 mm are carefully noted which give us T25 and T30 flow times. Mean of two orthogonal diameters is considered as total spread of the mix. Moreover, it requires ample trials to attain the desired results.

Fig. 3.4 Hobart mixer used for mixing.

Determination of flow times using V-funnel necessitates it to be completely filled with the formulation and then gate is opened. Time required by the mix to flow out of the funnel is precisely noted which is the required flow time.

Total spread as well as T25 and T30 flow times are calculated for self compacting paste systems using hagermann cone. In case of self compacting mortar systems, T25 and V-funnel flow times are calculated and plotted against each other in later chapters. T25 is basically a measure for determining both plastic viscosity and yield stress. It was determined on an assumption involving cone diameters and the standard total spreads determined for concrete. Slump cone used to determine total spread of 50 cm has an internal diameter of 20 cm. So ratio of internal diameter of cone to total spread is 2.5. Similarly, same ratio was implemented while determining flow values using hagermann cone which has an internal diameter of 10cm. Hence, time required by the mix to reach 25cm i.e T25 alongwith T30 was also determined.

3.2.6 Strength:

Prisms for flexural and compressive strength measurements comprised of 4x4x16cm dimensions and were cast and cured as per EN 196-1. Strengths of self compacting paste and mortar systems are determined at the age of 1, 3, 7 and 28 days conforming to EN 196-1: 1994. The flexural strength at any age was an average of three specimens while compressive strength was an average of five specimens. The specimens were cured at 20±1^{o}C and relative humidity of more than 90% for the first 24 hours and then these were demoulded and put underwater till the respective ages of testing.

3.2.7 Calorimetry:

Calorimetry is done to determine the amount of heat released and influence on the hydration kinetics of the system. In the present study, a very sophisticated and user friendly equipment namely Calmetrix (F -CAL 8000) as used for the purpose as shown in fig. 3.5 and 3.6 . It was a portable equipment, thus can be used at construction sites more effectively. It had two batteries each operating four channels, which makes eight channels in total. First of all, equipment was logged on so that it can regulate the temperature within the equipment. Samples can be kept in all the eight channels simultaneously. Only modification to be done by the user is to enter the time of introduction of sample into the calorimeter. After 72hrs, results are attained and well defined curves are automatically plotted by the calorimeter as per the data incorporated in the system.

Fig. 3.5. Semi-adiabatic Calorimeter used for determining hydration kinetics.

3.2.8 Heat of Hydration:

In the present study heat of hydration is calculated by “Testo” as shown in fig. 3.6, using software namely Testo Comfort Software Basic 5.0. This equipment was used in combination with shrinkage apparatus which resulted in the easy and speedy extraction of results for both the shrinkage and heat of hydration measurements for any two formulations in a single trial. It had two wires protruding outwards with the sensors (which recorded the temperature) at the end. Procedure adopted includes pouring of fresh specimen for shrinkage measurement and then sensors are immersed in the sample. The equipment records temperature after every five seconds. It is important to note here that once the equipment is logged on, it starts determining temperature by both the sensors. So, time lapse between pouring of both the shrinkage samples must be noted. After 24 hours, results are attained, which gives gradual change in temperature with the passage of time. Also very fine curves for both the specimens are plotted by the said software.

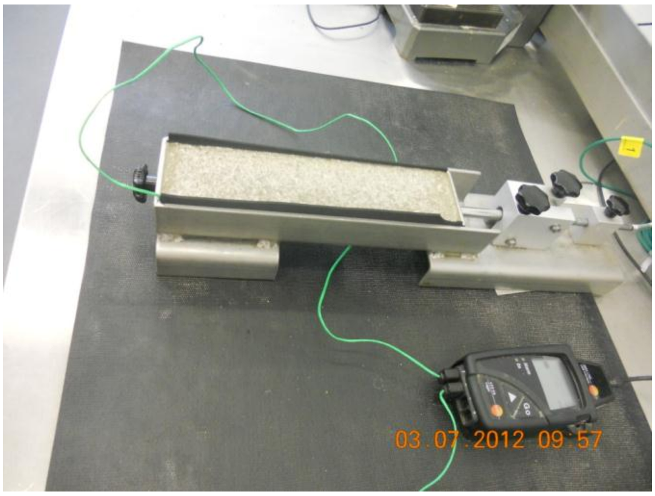

Fig. 3.6 Sensors of “Testo”, immersed in the fresh shrinkage sample

3.2.9 Scanning Electron Microscopy:

Scanning electron microscopy was done using FEI XL 30 Environmental Scanning Electron Microscope with field emission gun (ESEM FEG). The specimens of powdered secondary raw materials as well as the self compacting paste samples were studied at specified ages after stopping hydration by immersing samples in acetone for about 24 hours. The purpose was to study the microstructure, morphology and the hydration products at various ages.

3.2.10 Mercury Intrusion Porosimetry:

Mercury Intrusion Porosimetry is a widely used method for measuring the pore size distribution of porous media. It is based on the fact that for squeezing out a non-wetting fluid in a pore of diameter d, a pressure P inversely proportional to the diameter of this pore must be applied. For a cylindrical pore this pressure is given by Washburn (Laplace) relation as given below;

$$P = -4 \gamma \cos \theta / d \qquad \textbf{(3.1)}$$

Where γ is the surface tension of the fluid and θ is the contact angle in degrees.

In this study, Pascal 440 mercury intrusion porosimeter was used for the purpose. The contact angle between mercury and the pore wall was 140^o and mercury surface tension was 480 dyne / cm. Samples were prepared after being subjected to compression. Thereafter, hydration was stopped by immersing them in a mixture of acetone for about 24 hours.

3.2.11 Shrinkage:

Total shrinkage may be considered as the sum of the chemical, plastic and drying shrinkages. Autogenous shrinkage occurs due to internal consumption of water during hydration at a constantly maintained temperature without loss of moisture to the environment. Drying shrinkage is caused by the loss of water to the environment [4].

This study emphasis on the estimation of total early shrinkage values attained in self compacting paste and mortar systems with and without the inclusion of SRM's in said proportions. Shrinkage measurements are attained at a temperature of $20\pm1\ ^{O}C$ and relative humidity of $60\pm5\ \%$ by a modified version of German classical "Schwinderinne" which means shrinkage channel apparatus of dimensions 4x6x25 cm. It is connected with computer & capable of recording translations of the order of 0.31 microns due to shrinkage or expansion of cement based systems. The measurements were started ten minutes after the water addition to the constituents of self-compacting cementitious systems, which lasted for the first 24 hours only. It is important to note that shrinkage apparatus is very sensitive and readings get disturbed readily, thus immense care must be taken once the fresh sample is poured in the shrinkage channel. The computer software recorded the shrinkage readings automatically with one minute interval. At the end of 24 hours, the shrinkage strains were picked up and imported into Excel sheets. The details of the measuring device can be seen in literature [4].

3.3 Specimens Designation:

In the present study, SCP formulations are generally designated in the format i.e. CI – 10FA – WD, where CI denotes CEM I. Since in all the SCP formulations water content is equal to the water demand of the system, hence WD denotes water demand. SRM's are used in 10% replacement by mass of cement so this proportion is designated as 10SRM eg. 10FA.

Similarly, in the later portion for SCM formulations, it has been mentioned that formulations have been used in 1:1:2 proportions representing two parts of fine aggregate and one part of cement and SRM. Water content has been fixed equal to 40% of cement which was later adjusted with super-plasticizer content for achieving target flow. So the formulations are designated as CI – SRM e.g. CI – FA. For SF containing formulations, improvisation has been done providing respective percentages of FA and SF like CI – 0.9FA+ 0.1SF.

CHAPTER- 4 RESULTS

4. RESULTS:

In this chapter results of tests for self compacting paste and mortar systems are incorporated.

4.1 Tests on SCP Systems:

Following tests were conducted on self compacting paste systems;

- Particle characterization of secondary raw materials
- Determination of water demands using VICAT apparatus
- Initial and final setting times using VICAT apparatus
- Super-plasticizer demand for target flow of 30± 1 cm
- Flow times using Hagermann Cone
- Strength determination using stiffer displacement controlled strength testing equipment
- Shrinkage response
- Calorimetric investigations
- Measurement of heats of hydration
- Study of microstructure using mercury intrusion porosimetry, scanning electron microscopy and X-ray diffraction.

4.1.1 Particle Characterization of SRM’s:

Particle size, shape and surface morphology of SRM’s have a direct influence on flow, shrinkage, strength and microstructure of self compacting paste systems. Particle characterization of SRM’s has been done using scanning electron microscope. Hence, resulting SEM images are listed in Fig 4.1.

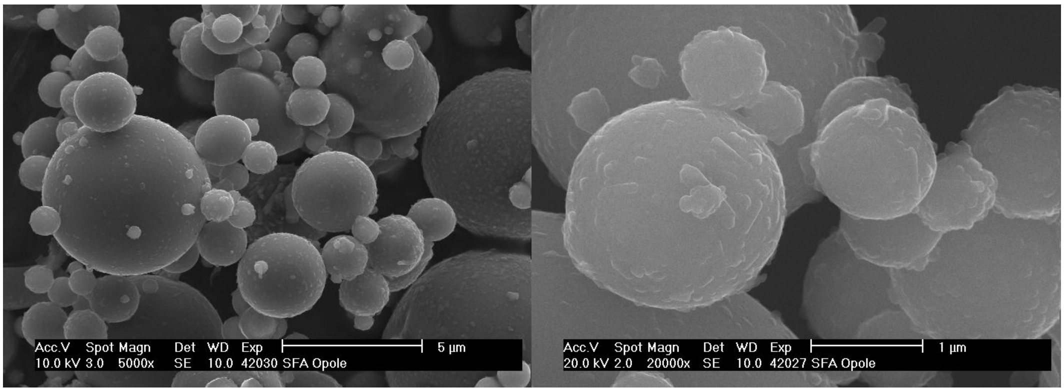

Fig. 4.1 (a). SEM images of FA particles

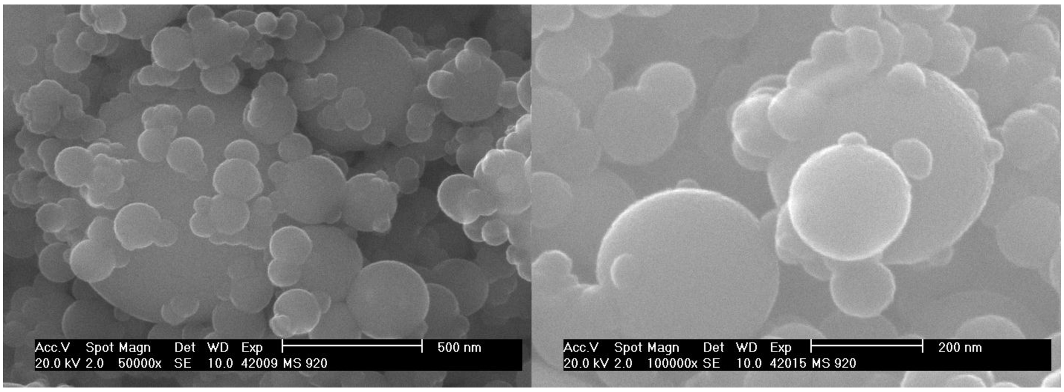

Fig.4.1 (b). SEM images of SF(920) particles

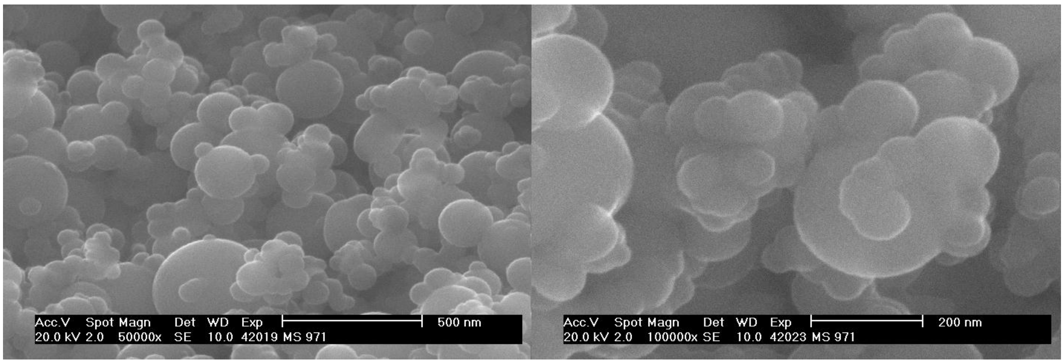

Fig.4.1 (c). SEM images of SF(971) particles

4.1.2 Water Demand & Setting Time:

Water demands and setting times of self compacting paste systems are determined using VICAT apparatus. Water demands and setting times of CEM I using SF and FA in 10% replacement mode are shown in Figures 4.2 and 4.3

respectively.

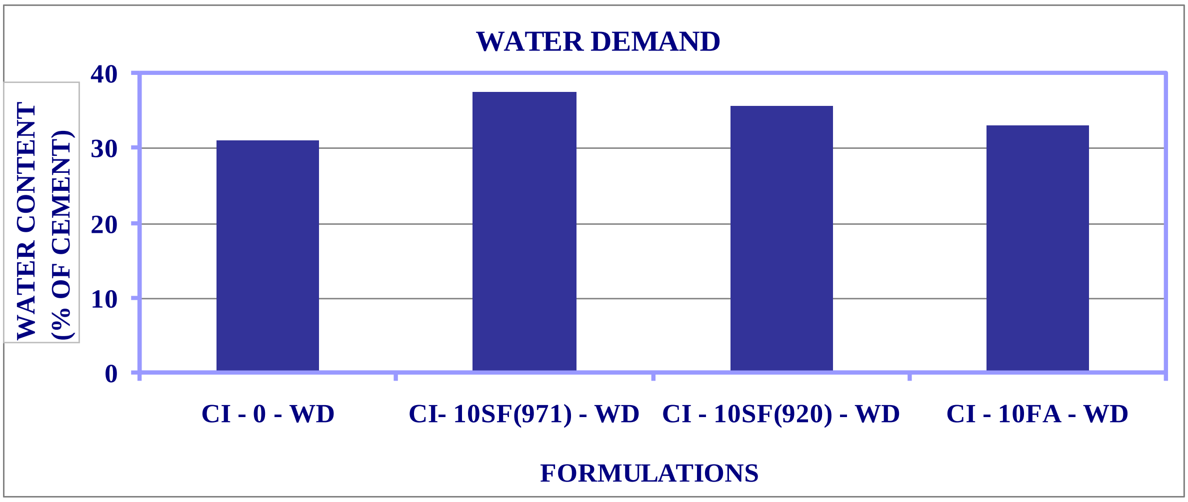

Fig. 4.2 Water demands of CEM- I using SF and FA in 10% replacement mode

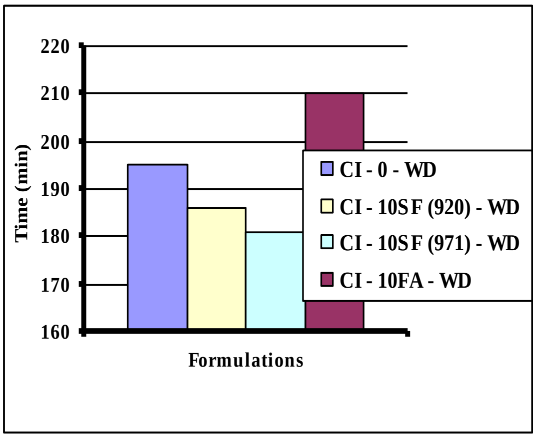

Fig. 4.3(a) Initial setting times

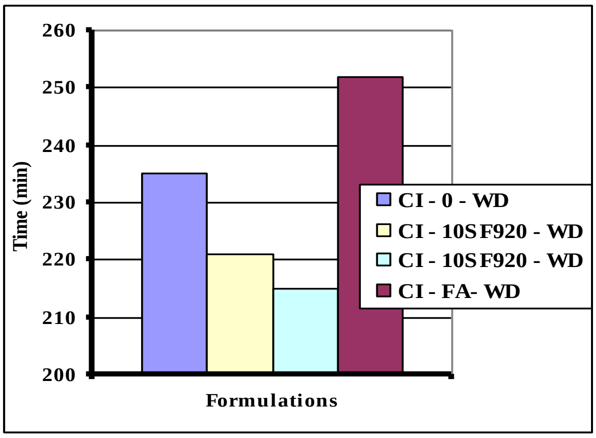

Fig. 4.3 (b) Final setting times

4.1.3 Super-plasticizer Demand:

Super-plasticizer demands for self compacting paste systems are shown in fig 4.4. Total spread of the mix as well as T25 & T30 flow times are also calculated and shown in fig. 4.5.

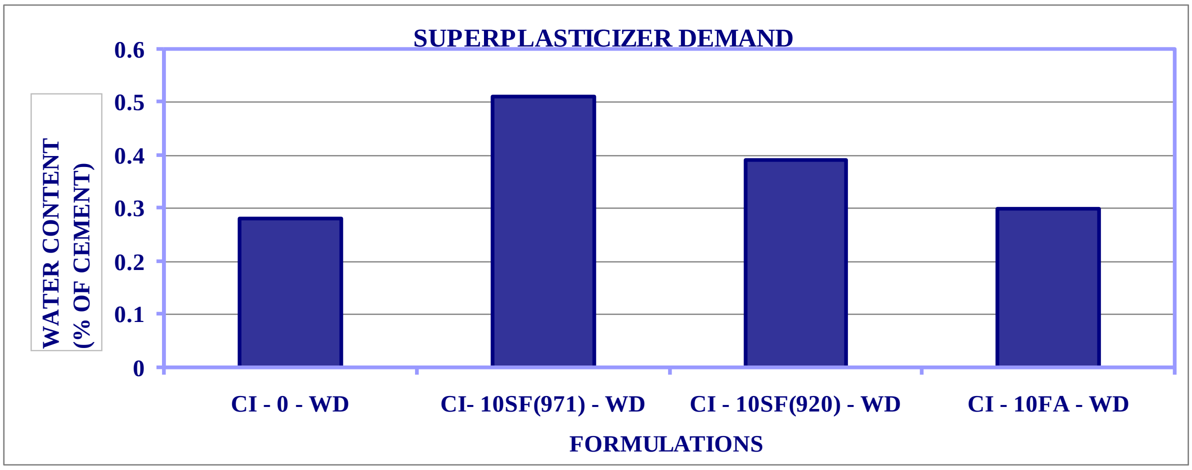

Fig. 4.4 Super-plasticizer demand for achieving target flow

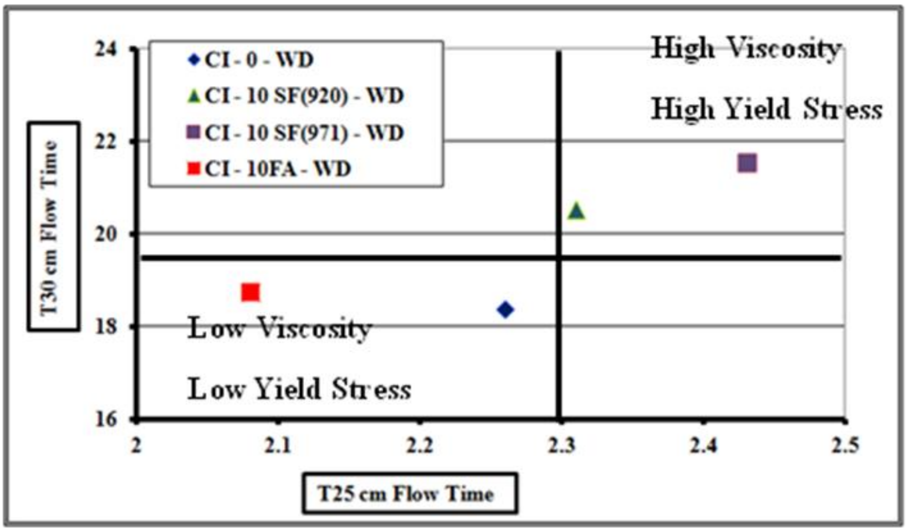

Fig. 4.5 T25 and T30 flow times

Flow data can also be used to make a comparison between super-plasticizer demand against total spread and total time required by the mix to achieve target flow. These illustrations are provided in fig 4.6 and 4.7.

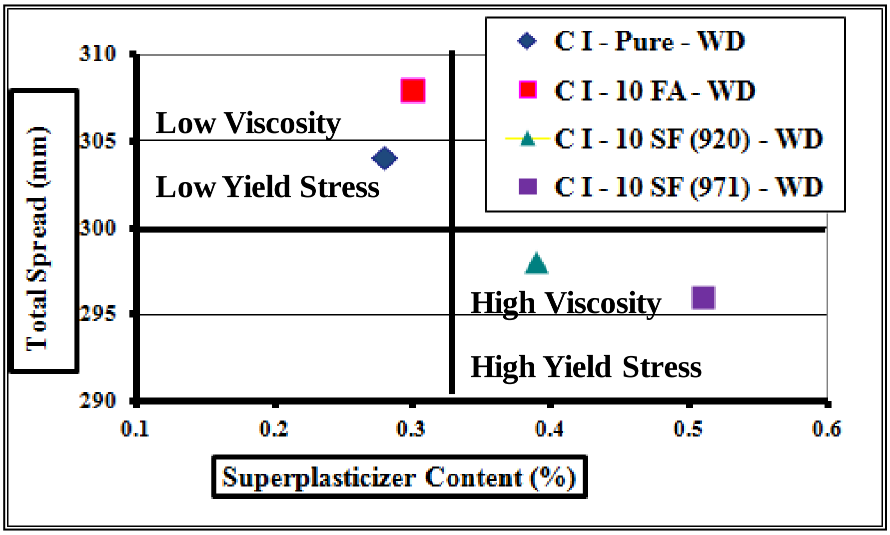

Fig. 4.6 Relation between super-plasticizer content and total spread

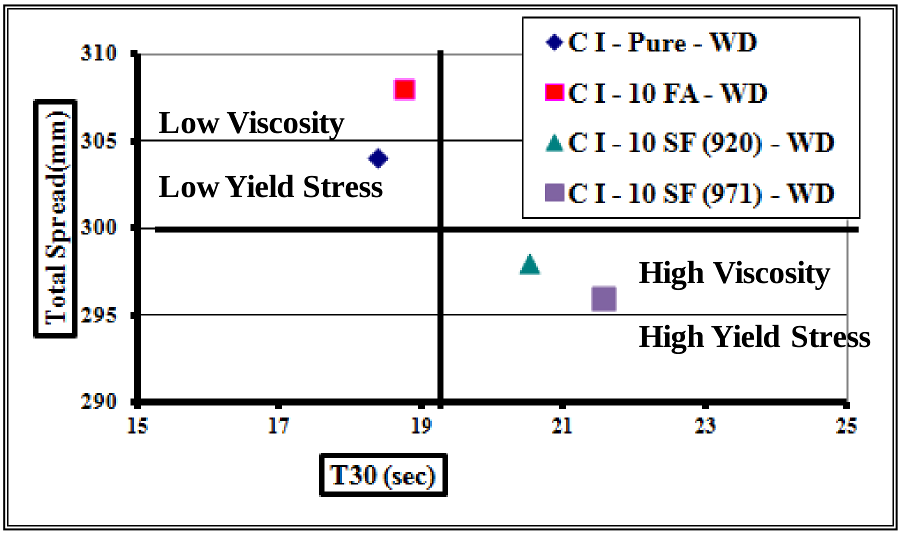

Fig. 4.7 Relation between T30 and total spread

4.1.4 Strength:

Strength development (both flexure and compression) for all the SCP formulations at specified ages is given in fig 4.8 and fig 4.9 as follows;

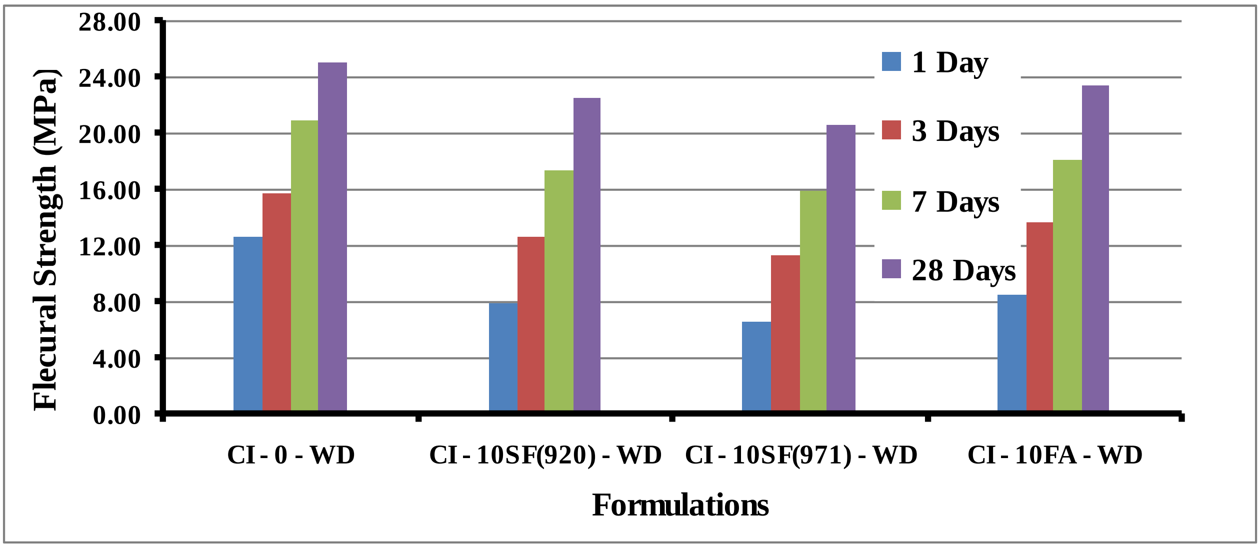

Fig. 4.8 Flexural strength of SCP formulations at 1, 3, 7 & 28 days

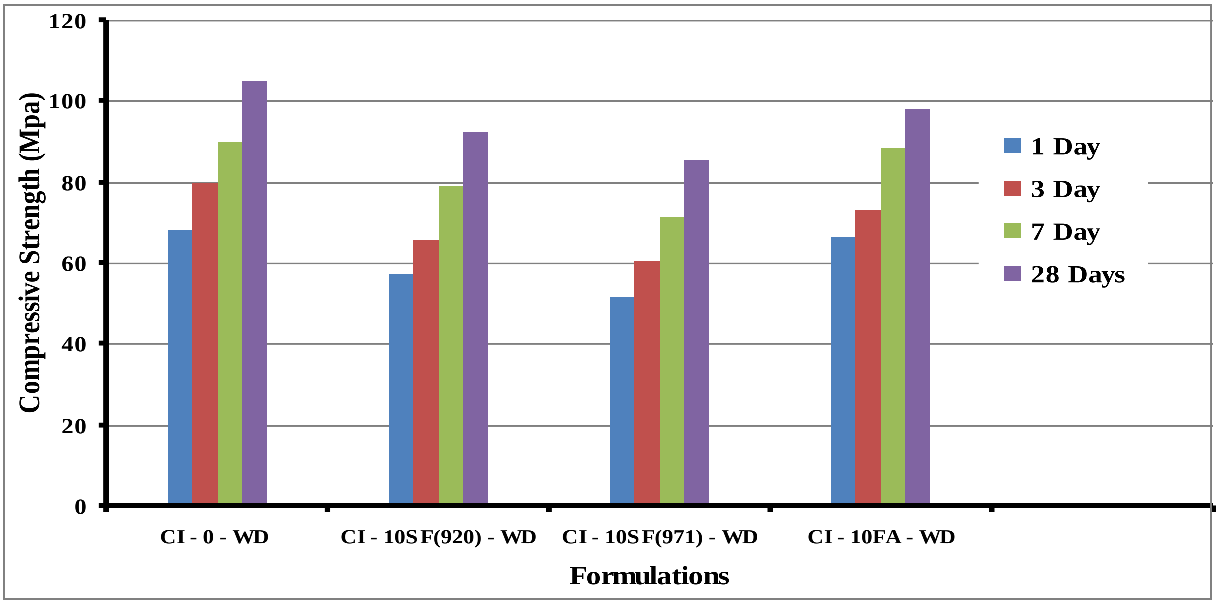

Fig. 4.9 Compressive strength of SCP formulations at 1, 3, 7 & 28 days

4.1.5 Calorimetry:

The key objective of calorimetry is to determine the influence of inclusion of secondary raw materials on the hydration kinetics as well as pattern and amount of heat released. Fig 4.10 represents the effect of addition of secondary raw materials in SCP. In other words, the following figure represents hydration kinetics of SCP systems with and without SRM’s.

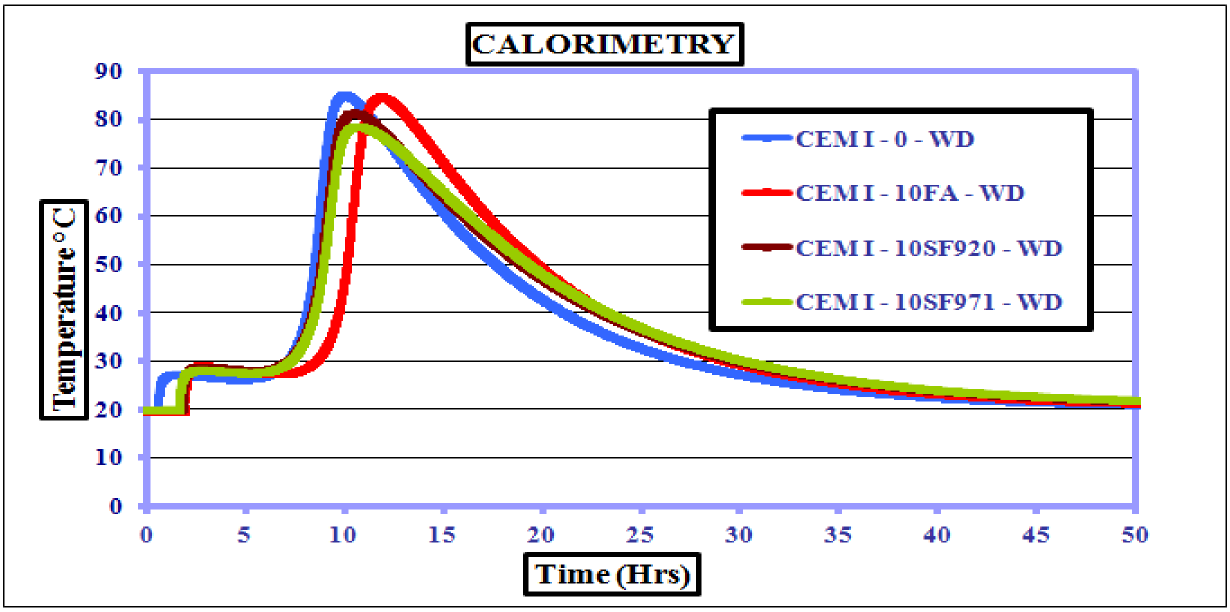

Fig. 4.10 Calorimetric investigation for analyzing heat generation in SCP systems

4.1.6 Shrinkage:

Shrinkage measurements were done for all the formulations of SCP systems i.e with and without SRM. Fig 4.11 represents a merged representation of all the mixes to have a clear understanding of basic concepts pertaining to behavior of SRM's. Moreover, all the shrinkage measurements were done in uncovered exposure conditions.

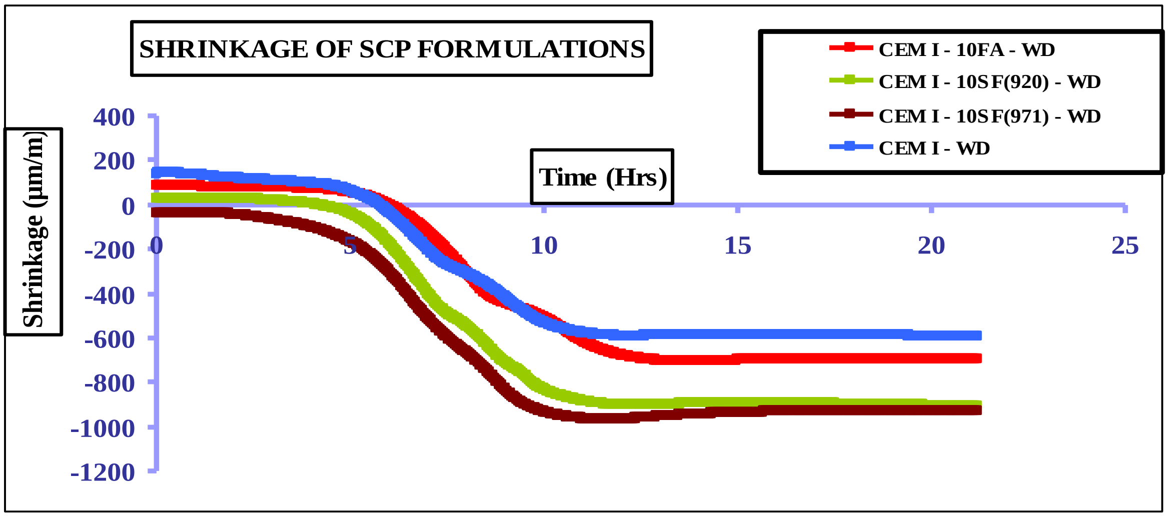

Fig. 4.11 Shrinkage response of SCP formulations

4.1.7 Heat of Hydration:

Heat of hydration of fresh SCP formulations is determined by equipment namely 'Testo'.

Care must be taken while introducing sensors of the said equipment in the fresh specimen. Fig. 4.12 represents the increase or decrease in the internal temperature of the SCP samples with the passage of time. These readings were taken for not more than 24 hours.

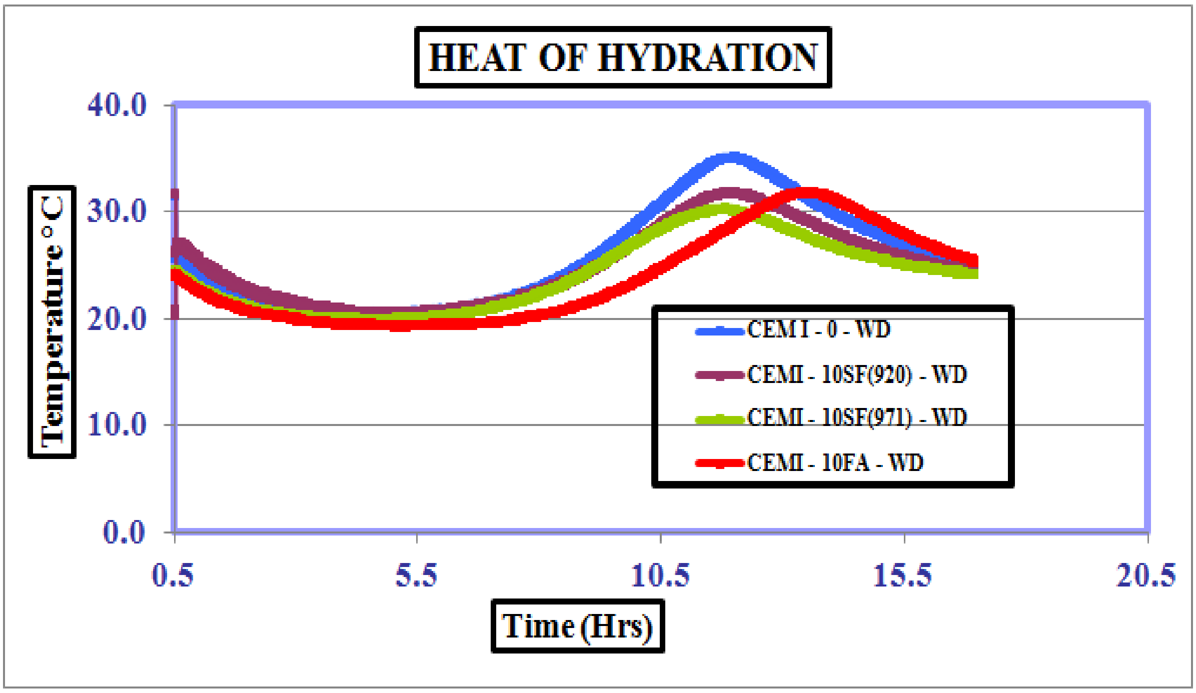

Fig. 4.12 Heats of hydration of SCP formulations

4.1.8 Mercury Intrusion Porosimetry:

Following figures represent the relation between pore size and intruded mercury volume for all the SCP formulations.

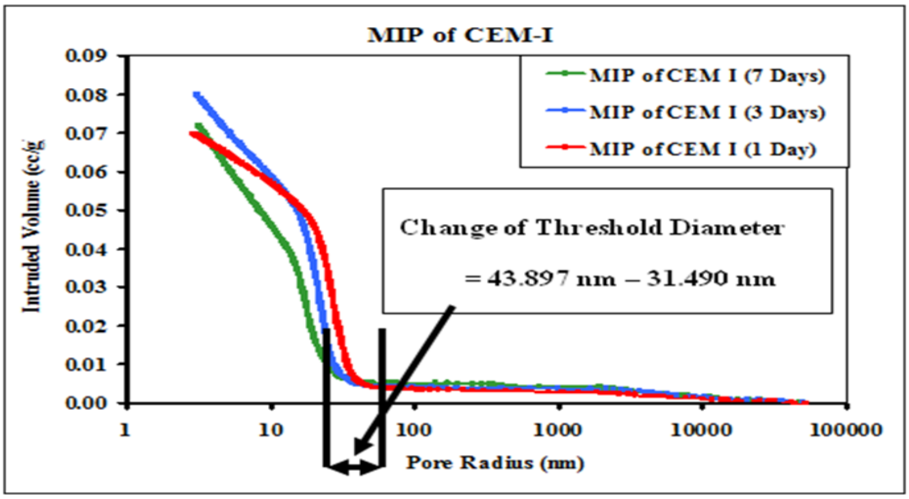

Fig. 4.13 Cumulative diagram of CEM-1 without SRM's

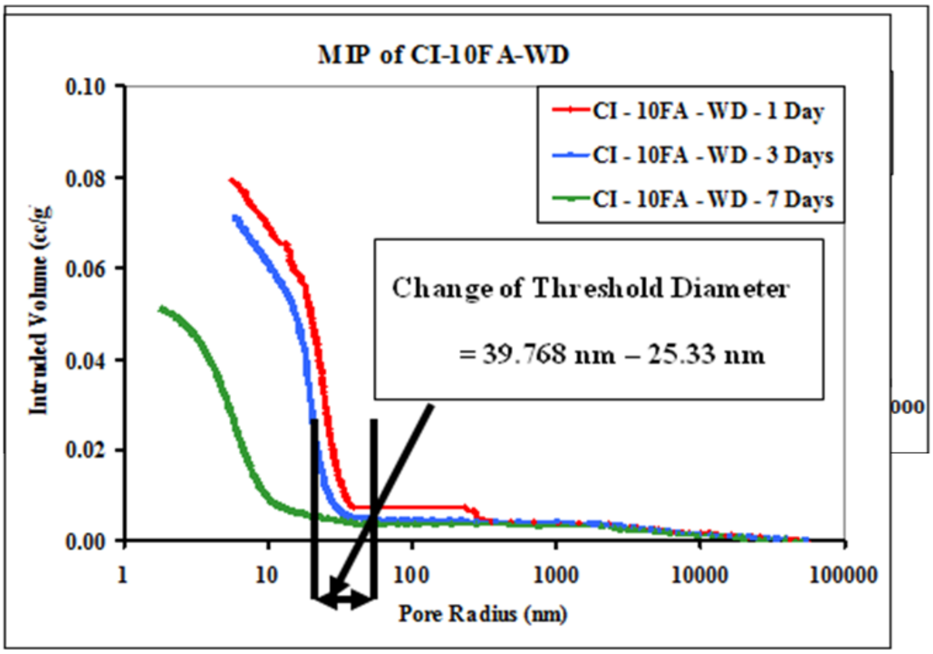

Fig. 4.14 Cumulative diagram of FA containing SCP formulation.

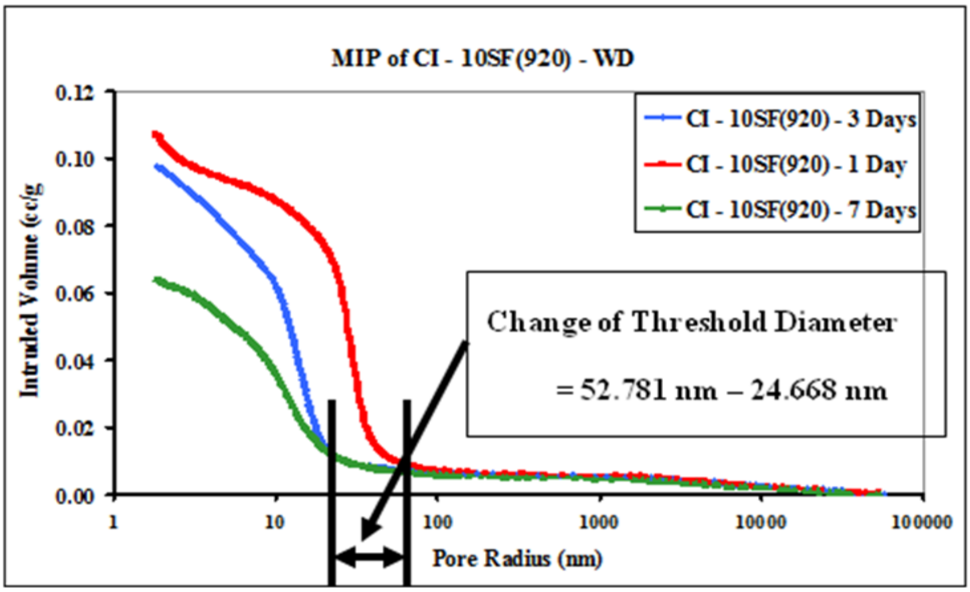

Fig. 4.15 Cumulative diagram of SF(920) containing SCP formulation.

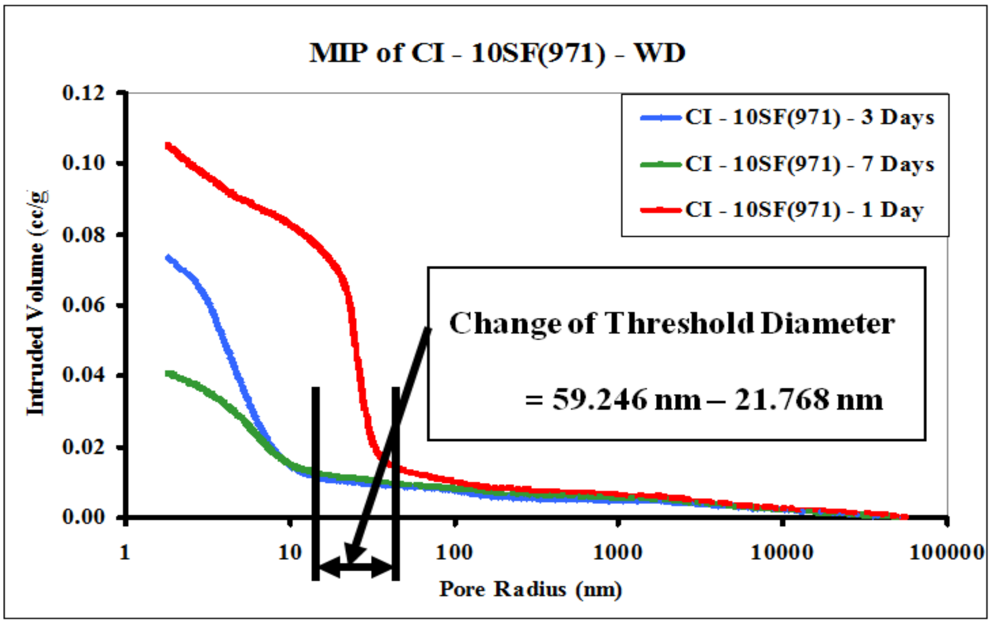

Fig. 4.16 Cumulative diagram of SF(971) containing SCP formulation.

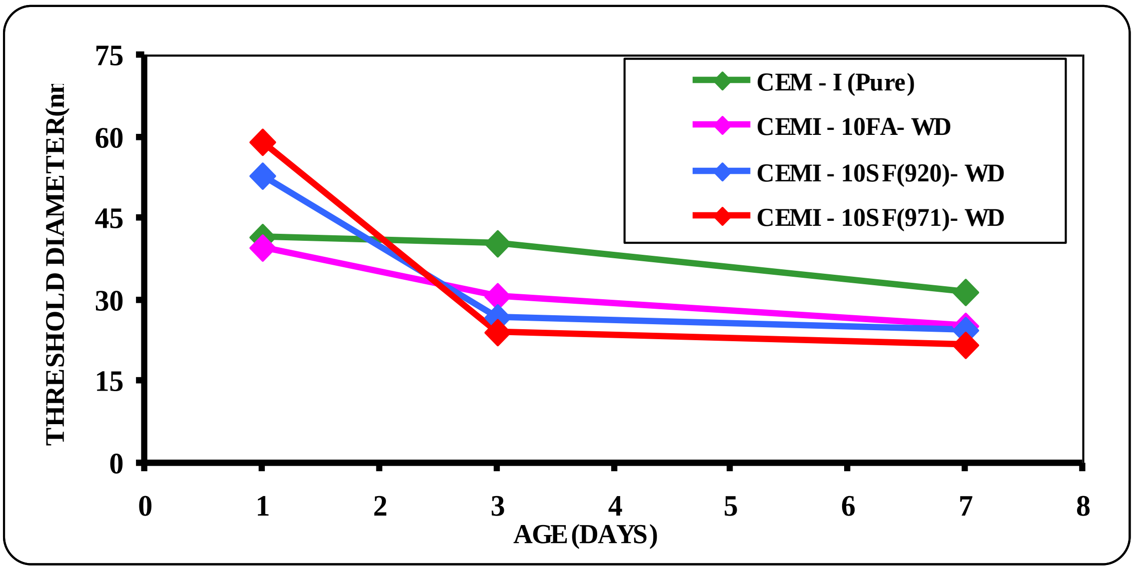

Fig. 4.17 Threshold Diameters of SCP formulation

4.2 Tests on SCM Systems:

Tests executed on self compacting mortar systems are as follows;

- Super-plasticizer demand and flow times using Hagermann Cone

- Flow times using V-funnel
- Strength determination using stiffer displacement controlled strength testing equipment
- Shrinkage response
- Calorimetric investigations
- Measurement of heats of hydration

4.2.1 Super-plasticizer Demand and flow times using Hagermann Cone:

Super-plasticizer content necessitating target flow of 30± 1 cm and T25 times were determined along with total spread of the mix. In the process, both grades of SF were used in combination with FA by replacing it 10% and 20% by mass in order to comment on the effect of superplastcizer content for respective formulations. Thereafter, SF was used in 10% replacement of FA was fixed and used to determined V-funnel times, shrinkage response, calorimetry and strength testing. Water content was taken equal to 40% of cement content. Fig.4.18 shows super-plasticizer demand of all the formulations of SCM systems. Fig. 4.19 hows the relation between mean diameter and T25 flow time.

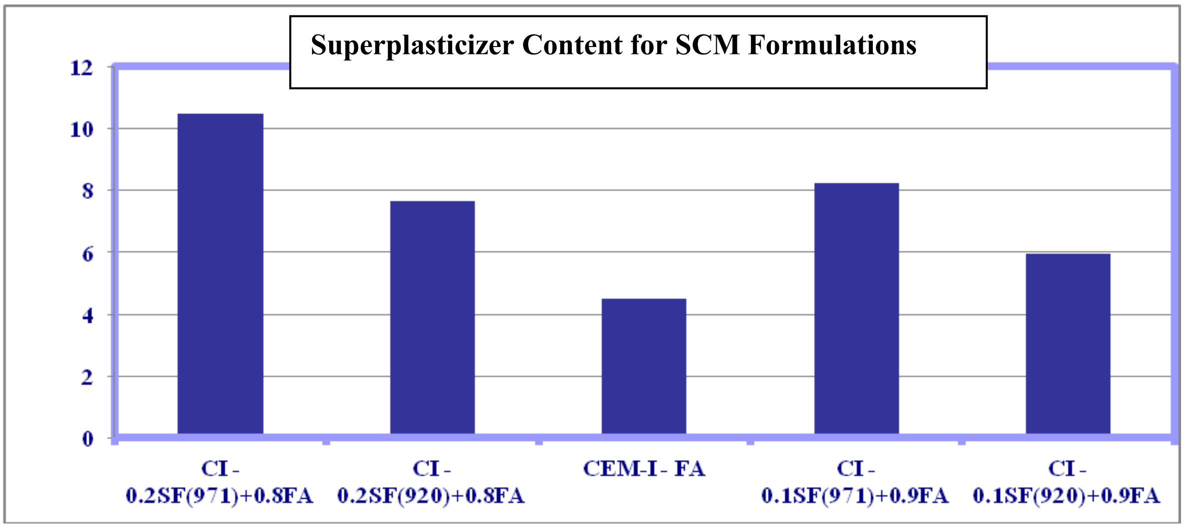

Fig. 4.18 Super-plasticizer content required for target flow of SCM formulations

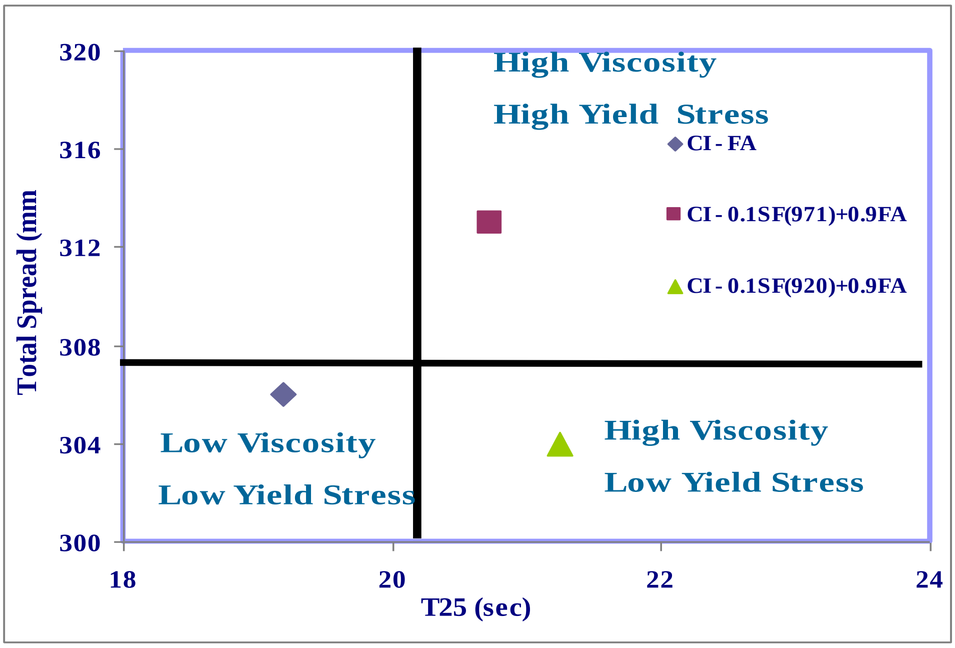

Fig. 4.19 Relation between mean diameter and T25 flow time of SCM formulation

4.2.2 Flow times using V-funnel:

Flow times were also determined using v-funnel apparatus once super-plasticizer content corresponding to target flow was fixed. Following figures 4.20 & 4.21 offer comparison between v-funnel flow time, total spread and T25 cm flow time.

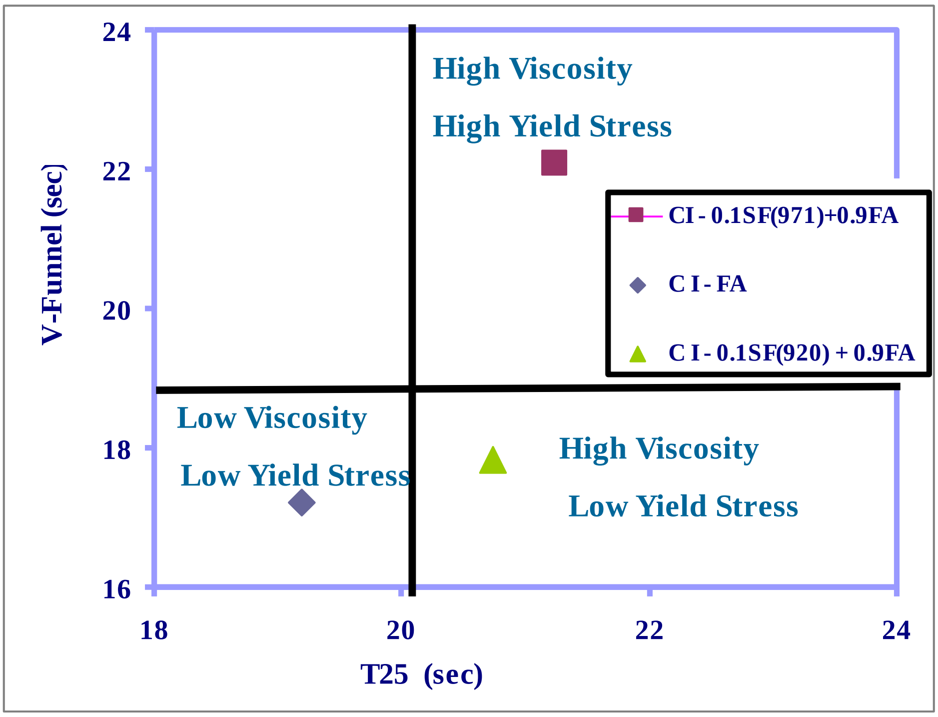

Fig. 4.20 Relation between V-funnel and T25 cm flow times

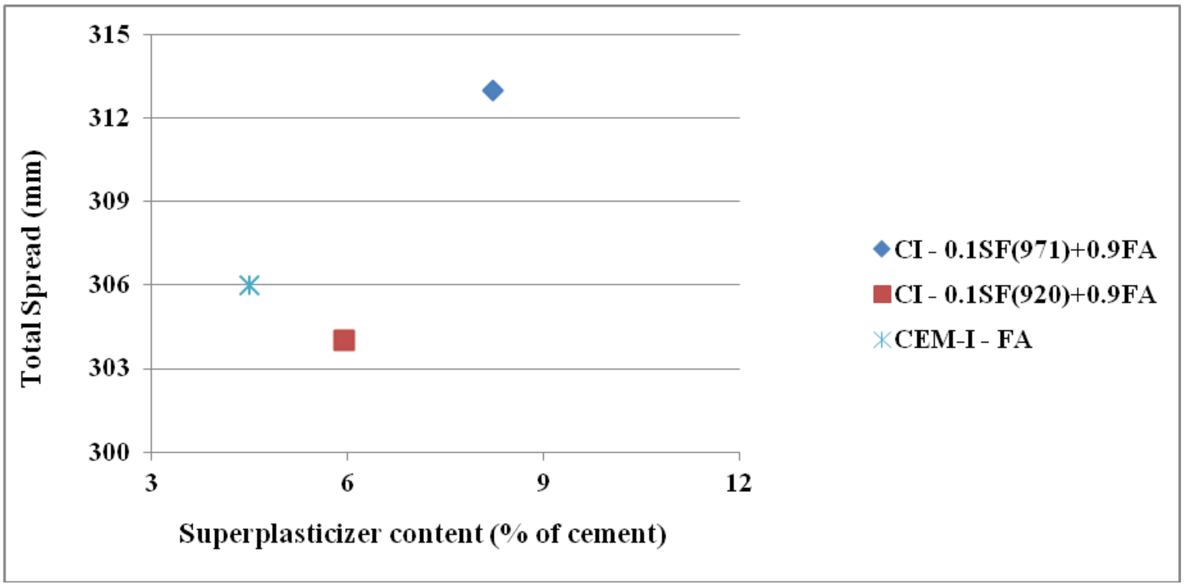

Fig. 4.21 Relation between super-plasticizer demand and total spread

4.2.3 Shrinkage:

SCM formulations were also subjected to shrinkage measurements. Equipment used for shrinkage measurements had already been discussed. All the measurements for SCM formulations also extended up to 24hours. Fig.4.22 gives the shrinkage response of all the mixes for uncovered exposure conditions.

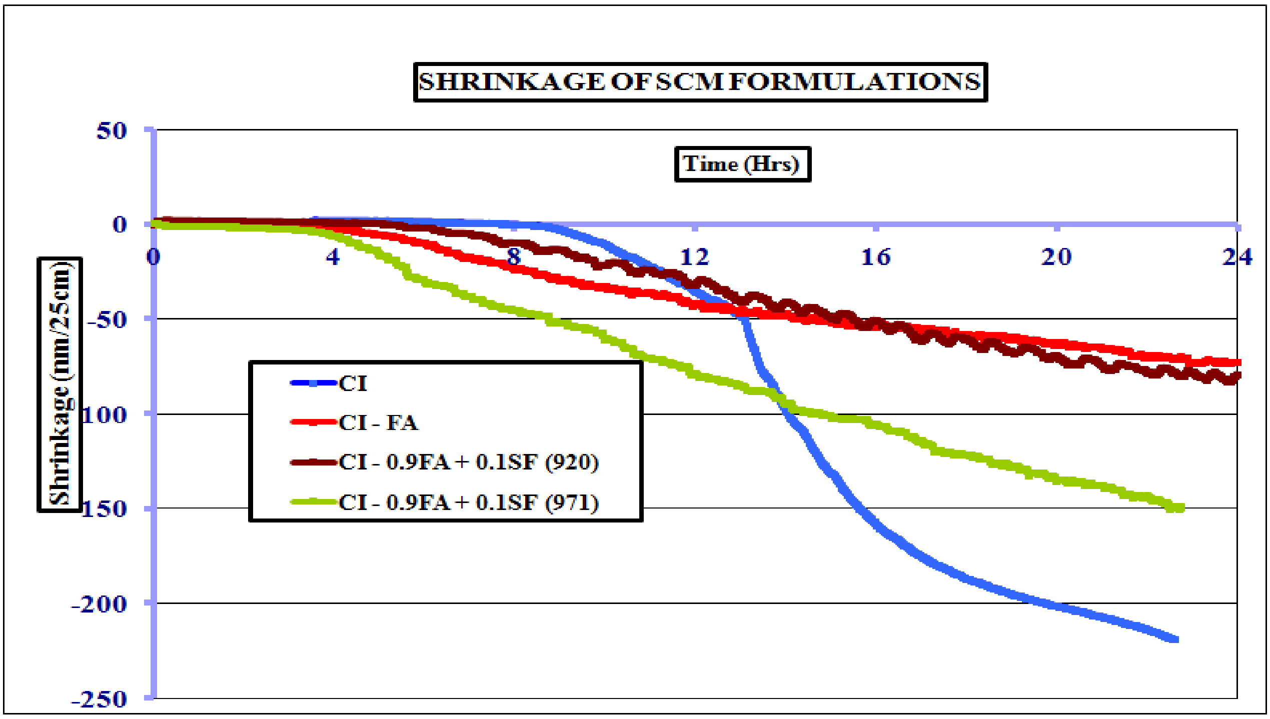

Fig. 4.22 Shrinkage response of SCM mixes

4.2.4 Calorimetry:

Fig. 4.23 represents the influence on hydration kinetics of SCM formulations by the addition of SRM's.

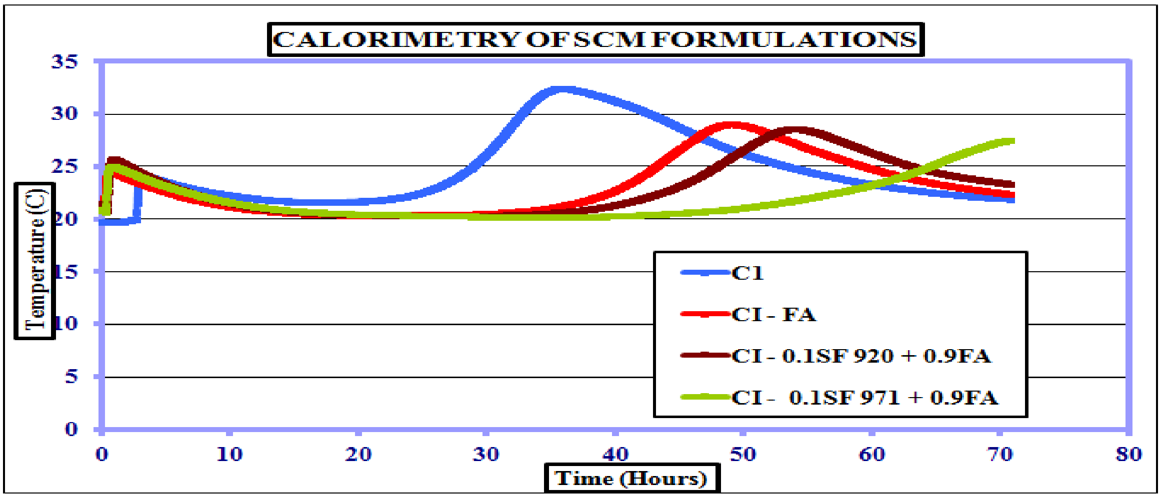

Fig. 4.23 Calorimetric investigations on SCM formulations

4.2.5 Heat of Hydration:

Heats of hydration were studied using the same equipment mentioned in chapter 3. Fig. 4.24 shows the heats of hydration of all the SCM formulations i.e with and without SRM's.

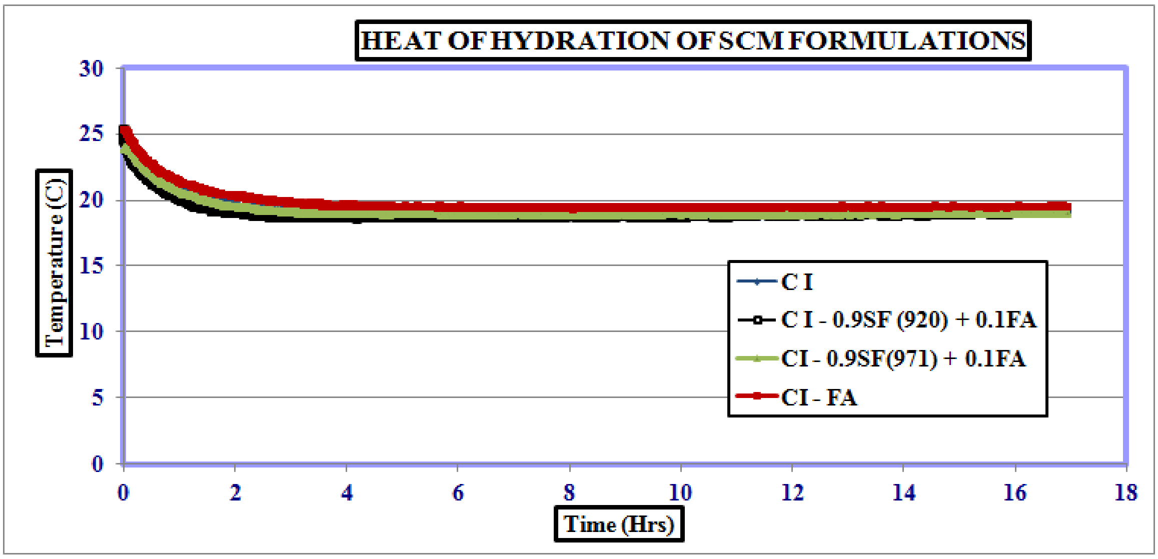

Fig. 4.24 Heats of hydration of SCM formulations

4.2.6 Strength:

Flexural and compressive strengths of SCM formulations after 28 days are given as follows;

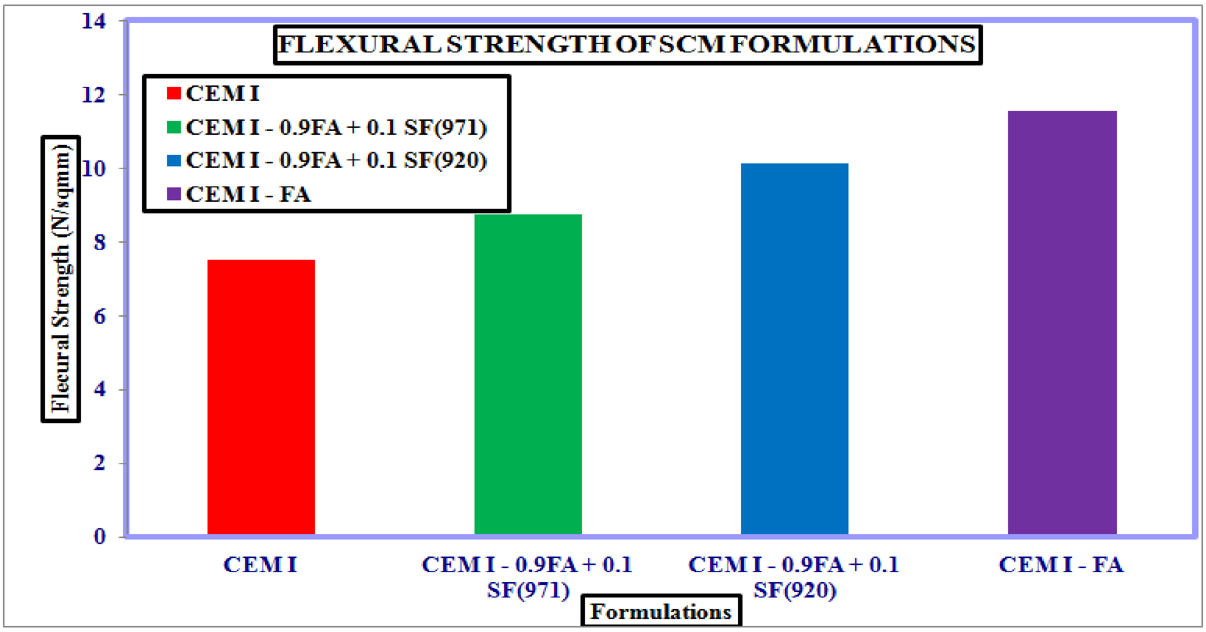

Fig. 4.25 Flexural strength of SCM formulations.

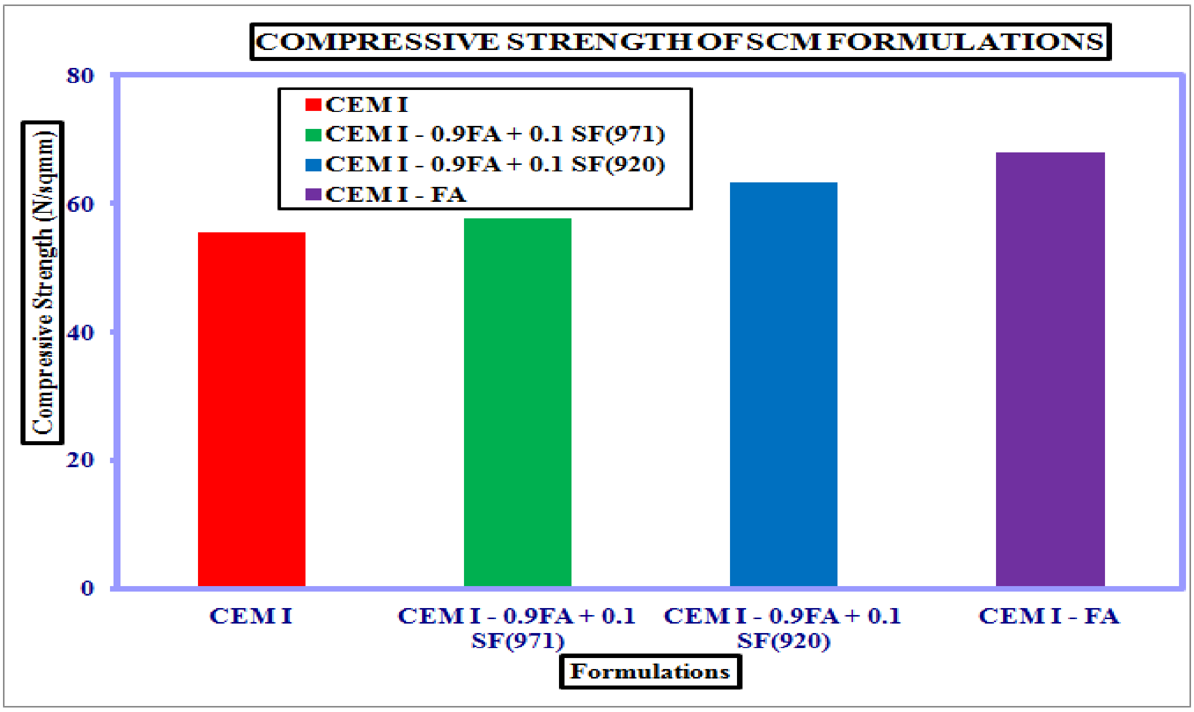

Fig.4.26 Compressive strength of SCM formulations

CHAPTER-5 DISCUSSIONS

5.1 DISCUSSIONS ON SCP SYSTEMS:

5.1.1 Particle Characterization of SRM's:

A clear understanding of particle shape, size, surface morphology, internal porosity etc. is beneficial in depicting their behavior when incorporated in the self compacting cementitious mixes. SRM's influence water demands, flow properties, strength development etc. of cement based composites, since in addition to cement grains, they also adsorb sufficient SP depending upon their size, shape, surface morphology, porosity etc.

Fig. 4.1shows SEM images of SRM's, used in the present study. Fig. 4.1(a) shows spherical particles of FA having smooth and glassy texture. Fig. 4.1(b) and (c) show the spherical particles of both grades of SF. We can also observe agglomeration which is linked with the direct use without sieving. The governing difference between the two is the silica content as shown in table 1.1. SF (971) having the greater percentage of silica ensures higher pozzolonic activity between the two.

5.1.2 Water Demand & Setting Times:

It can be observed from fig. 4.2 that for both the SF, comparable results were attained. As it has already been mentioned that both grades of SF were incorporated in the mixes in raw form as attained from the supplier, hence it led to relatively coarser particles and formation of agglomerates. Relatively higher water demand was anticipated for the SF containing formulations since coarser size of particles is also associated with larger voids, necessitating and accommodating more moisture within them. On the other hand, water content required to lubricate each particle in the mix containing fly ash is quite less than that containing silica fumes. Such behavior can be attributed to smooth and glassy texture of FA leading to lesser absorption of water, resulting in lower water demand.

From fig. 4.3 (a) and (b), variation between the setting times of formulations with and without SRM's can be clearly seen. Setting can be defined as 'conversion from liquid to solid phase'. The mixes for which accelerated hydration is achieved, thus early setting can be anticipated for the said formulations. Accelerated hydration is associated with high pozzolanic activity of powders. SF mixes having high silica content had much rapid setting as compared to FA formulations due to slow pozzolanic nature of FA.

5.1.3 Flow of SCP Systems:

Flowability of self compacting cementitious systems is of prime importance in order to eliminate the need for manual compaction. Flow properties of self compacting cementitious systems depend upon size and aggregate content, surface area, particle size, shape and surface morphology of powders and also time and pattern of inclusion of SP in the mix. So, after fixing water content for SCP formulations, super-plasticizer demand was determined for respective formulations using Hagermann cone. Since, both grades of SF were used without sieving, it led to relatively coarser primary particles of SF having higher capacity to consume locally available water which may be responsible of creating bigger sized voids in the formulations incorporating them. The influence of particle size can be seen from fig. 4.4, which shows that SF (971) has the highest super-plasticizer demand required for target flow, which kept on increasing with the increase in silica fume due to its coarser particle size & more surface area. Also flow times were calculated to get an idea on plastic viscosity and yield stress of formulations using SRMs in SCP formulations.

Fig.4.5 shows that formulation containing fly ash attains 250 mm and total target spread considerably earlier than the other mixes using SF. This behavior depicts the contribution of spherical particles of FA in reducing frictional force among angular particles of cement. Such adjustment of spherical particles of FA also decreases yield stress. Also, relatively quick flow time of FA formulations can be attributed to the increase in effective water cement ratio, which is related to the surface morphology and slow pozzolanic activity of FA. Addition of SF in the same %age sufficiently increases total flow time i.e T30 cm flow time due to corresponding increase in the yield stress and plastic viscosity, hence necessitating increased super-plasticizer dosage to achieve target flow.

Super-plasticizer plays a key role in enhancing flow properties of cement based composites. It is beneficial in increasing workability of the mix while reducing its mixing water content. Also it allows uniform dispersion of cement particles in the mix. As the super-plasticizer content in the mix increases, there is a subsequent increase in the total spread which is measured as ‘mean of two orthogonal diameters’. Moreover, it is also dependent upon the surface morphology of SRM’s incorporated. It can be seen from fig.4.6 that FA necessitating the least amount of super-plasticizer while both grades of SF require sufficiently more super-plasticizer is needed to achieve the target flow. Similarly as mentioned above inclusion of both grades of SF increase viscosity of the mix, thus leading to an increase in flow times.

Such trend can be observed in fig. 4.7 showing minimum time required by FA and CEM I (pure) and vice versa.

5.1.4 Strength:

SF was attained from Elkem Microsilica in bags weighing 25 kgs and 50kgs. Both grades were incorporated in the self compacting cementitious systems in the fresh state without being sieved. This led to relatively coarser size of SF particles. As mentioned above particle size along with texture and surface morphology of SRMs are of utmost importance in relation to the strength development. Furthermore, fineness of the SRM is also directly associated with the pozzolanic activity. The finer the particle more will be the pozzolanic activity and hence greater will be the strength. Hence, lesser strength is linked with the relative coarser size of particles of both grades of SF. Self compacting paste systems containing FA in 10% replacement mode unexpectedly showed much better strength results (fig. 4.8 and fig. 4.9) than both the SF (971) and SF (920).

5.1.5 Calorimetry:

Calorimetric investigations were done on SCP formulations to compare the amount of heat released by respective mixes, depending upon a number of factors including their water and super-plasticizer demands. Heat generation is basically anticipated with the cement content in the mix. The more the cement content, more will be the heat released. Since a part of cement content was replaced with SRMs, water content in respective formulations and pozzolanic nature of powders became vital for their hydration kinetics. As soon as water is incorporated in the mixes, an early first peak is attained within an hour which is due to initial temperature difference between the sample and the equipment. For comparison of results, second and third peaks are considered authentic. From fig. 4.10, it can be observed that CEM I (pure) attains relatively earlier and higher second peak as compared to FA containing formulations. This can be attributed to retarding/delaying effect of FA. On the other hand, for SF (920) and SF (971) second peak occurs at almost the same time but SF (920) liberates much more heat than the other. This can be attributed to dilution of cement due to relatively more water content in SF (971) containing mix. Same effect can be observed in FA containing formulation that although heat generation is a bit delayed but there isn't any significant reduction in the amount of heat released because water content in it does not dilute much of the cement content in the mix.

5.1.6 Shrinkage:

Accumulated shrinkage of self compacting paste formulations is the combination of various parallel and over lapping operating mechanisms namely drying shrinkage, chemical shrinkage, autogenous shrinkage and plastic shrinkage. Typically, shrinkage is aided by utilization of water during hydration process, absorption by both SRM and cement for their lubrication and external loss of water to environment. The measurement technique of shrinkage includes volumetric and linear method. Moreover, one can also get an idea about the fluctuations in setting times from graphical representation of shrinkage measurements.

Shrinkage and expansion in self compacting cementitious systems are controlled by the paste phase. The more the cement content, greater will be the shrinkage. Similarly, dilution in cement content by replacing it with SRM will lead to reduced shrinkage values depending upon their behavior. All the shrinkage measurements were taken for uncovered conditions. It is important to mention here that immense care must be exercised while using shrinkage apparatus. Even opening and closing of lab door may disturb the readings.

Fig. 4.11 shows the variation in shrinkage behavior of SCP formulations containing FA, SF (920) and SF (971) in 10% replacements. FA containing mix undergoes expansion in the beginning since it does not observe water readily, thus leading to the formation of expansive species since sufficient space and moisture are available for their growth. Expansion in the beginning can also be linked with the precipitation or settling of excess moisture at the bottom of the fresh sample due to increase in effective water cement ratio. Moreover, its setting time was also relatively delayed due to slow pozzolonic activity of FA. On the contrary both grades of SF exhibited accelerated setting as well as more shrinkage which is related to their rapid consumption of moisture within the voids of primary agglomerates formed due to their direct use without sieving. Also, a clear demarcation can be observed among SCP mixes that both grades of SF having more accelerated pozzolanic nature leads to early drying shrinkage than others due to lesser silica content. Hence, pozzolonic activity and consumption of moisture by SRM’s as per their particle size, shape and surface morphology as well as heat generation during pozzolonic reaction can be considered as controlling factors for estimation of total shrinkage.

5.1.7 Heat of Hydration:

From fig. 4.12, it can be observed that both grades of SF have minor difference in gain in temperature with the passage of time. Reason behind less variation can be linked with almost the same percentage of silica in both the grades of SF. Moreover, temperature rise shown by FA formulation is relatively delayed and maximum temperature is reached after about 2hrs with respect to other formulations. Also, it can be seen that the interval at which maximum temperature is attained is identical for CEM I (pure) and both grades of SF. Convergence of temperature for all the formulations of SCP occurs after 16 hours.

5.1.8 Mercury Intrusion Porosimetry:

Mercury intrusion porosimetry is basically a pore structure characterization technique used to determine total porosity, threshold pore diameter & mean pore diameter. Cumulative diagrams are presented with intruded mercury volume in cc/g on y-axis and pore radius in nm on x-axis, to comment on the microstructure of SCP formulations. These illustrations are vital to comment on the pore refinement with the passage of time as well as threshold diameter. Threshold diameter can be referred to as the diameter at which a sudden increase in mercury intrusion is facilitated. Minimal difference was observed in the decrease in threshold diameter was observed for pure CEM I and FA containing mix. This can also be observed from strength results that these two mixes exhibited fairly better results. Since both grades of SF were incorporated ‘as packaged’, hence bigger voids accommodated more mercury intrusions. It was also observed that pore refinement was much better for these two mixes. Only factor was the mode used to include SF powders in mixing i.e without sieving. Moreover, it is important to mention here that this method is not a true representative of total mercury intrusion in the samples. Although, maximum mercury intruded has been mentioned in the cumulative graphs but at such high pressure of 400 MPa, pores of the sample are likely to be broken hence leading to imperfections in results.

5.2 DISCUSSIONS ON SCM SYSTEMS:

5.2.1 Flow times using Hagermann Cone:

Total spread and T25 cm flow time were determined using Hagermann cone. SCM formulations comprised of use of FA alone and in combination with both grades Of SF by replacing 10% by its mass. Moreover, results of replacement of both grades of SF upto 20%

by mass of FA are also presented in the present study to show decrease in super-plasticizer demand as SF content is reduced. Since FA depresses the super-plasticizer demand hence it was employed to arrive at an economical mix. Super-plasticizer used for mortar systems was Melflux 2510L with 45.7% solids. Also, it is important to mention here that usage of liquid super-plasticizer requires much more careful handling than the one in powder form, since its liquid content has to be adjusted with w/c ratio of the system otherwise imperfections like bleeding etc. appear in the mix. Fine aggregate must also be well graded to achieve the desired results. As mentioned, sand used in SCM formulations was Roba sand with 0-2mm particle size.

Fig. 4.18 shows the fluctuation in super-plasticizer demands for SCM formulations. As discussed above, SRMs particle size, shape and surface morphology significantly affect super-plasticizer demand of the system. It can be seen from the figure that super-plasticizer content increased with the increase in SF content. Also, replacement of both grades of SF was later fixed at 10% by mass of FA in order to further economize the mix.

Procedure adopted was determination of T25 cm flow time and then total spread i.e mean diameter was determined. It was observed that T25 cm flow time decreases with the increase in SF content with SF (971) showing least time to reach 250mm when it is used in 20% replacement by mass of FA. Such behavior can be attributed to reduction in resistance offered by fine aggregate against flow and good internal packing of FA and SF (971) particles. Also high super-plasticizer content can be the reason behind lesser flow time.

In fig. 4.19 comparison has been made between total spread and time taken by the mix to reach 250mm. It can be observed that FA containing formulation attains T25cm flow time earlier than the other mixes in which its proportion had been diluted. The reason behind such reduction in T25 cm flow time is the easy adjustment of spherical particles of FA, leading to decrease in yield stress. On the contrary, increase in viscosity can be attributed to the addition of SF in the remaining formulations containing blends of FA with both grades of SF.

5.2.2 Flow times using V-Funnel:

Fig. 4.20 is the relation between V-funnel time and T25cm flow time of SCM formulations. V-funnel apparatus can be treated as a measure of plastic viscosity. For FA containing formulation, T25 cm flow time is relatively less due to easy adjustment of spherical and glassy textured particles of FA in the mix. Also when FA is incorporated in SCP systems, effective water cement ratio increases since FA powder has a glassy texture and hence low absorption capacity and slow pozzolanic activity can be anticipated. So, availability of sufficient moisture in the mix aids towards its workability. On the other hand, the above mentioned formulation also gave the lowest V-funnel flow time. When SF was incorporated in the mix, it increased viscosity in the mix it led to the increase in both T25 cm flow time and V-funnel flow time as well for both the formulations. Since the surface area and silica content of SF(971) was more than that of SF(920), hence it required more super-plasticizer to produce target flow and the same was accommodated in between voids of coarser grains. Hence, with the increase in super-plasticizer content, viscosity reduces and thereby reducing both V-funnel and T25 flow times.

5.2.3 Shrinkage:

Shrinkage behavior of SCM formulations are shown in fig. 4.22. Mix containing FA and blend of SF (920) with FA showed almost the same amount of shrinkage extending up to the same amount of time i.e. 24 hours. In other words, it can be said that replacement of FA with 10% SF (920) reduced shrinkage to a certain degree. On the other hand, by the inclusion of SF (971) a considerable increase in shrinkage was observed. Moreover, its addition also leads to early setting due to accelerated hydration. As mentioned above, liquid super-plasticizer was employed for SCM formulations. It was observed that its inclusion lead to a retarding effect, since it can be seen from the amount of shrinkage. Also, it can be observed that for FA and FA+SF (920), the amount of super-plasticizer was quite less and the shrinkage curve becomes uniform after about 22 hours. On the other hand for SF (971) containing mix, super-plasticizer content was relatively higher hence its delaying influence can be clearly seen with the shrinkage continuously increasing for the first 24 hours. So it can be anticipated that its drying shrinkage prolongs for more than 24 hours.

5.2.4 Calorimetry:

Fig. 4.23 shows amount of heat released using Calmetrix (F-CAL 8000) of the SCM formulations. This equipment allows the user to check the development of calorimetric curves at any instant of time. Initially, calorimetric investigations were done for 72 hours as it is indicated for CEM-I (without SRM) for which second peak was attained for the first 24 hours. Although we are not interested in first peak, even though it can also be observed that it is relatively delayed. Conventionally, inclusion of SF in FA must increase the amount of heat released as well as it must also accelerate the occurrence of second peak but contradictory results were attained. The occurrence of second peaks of all the SCM formulations is coherent to the super-plasticizer demands arranged in the ascending order. So it can be anticipated that it has a certain role to play in the hydration kinetics of the system. It is also important to mention here that it should be ensured that weight of the samples must be kept constant. Otherwise it leads to undesirable results, since increase in weight results in an increase in amount of heat released. The maximum temperature attained by all the FA containing formulations is almost the same. It can be observed that FA system gave early second peak as compared to the one containing SF (920). When SF (971) was incorporated in the mix, an abrupt rise in super-plasticizer demand was mentioned, which resulted in the delay of second peak even beyond 72 hours. Such results can also be compared with those of shrinkage, for the mix containing SF (971) drying shrinkage prolonged beyond 24 hours due to delaying action of super-plasticizer. Similarly, mixes containing FA and blends of SF (920) with FA, showed almost the same shrinkage for the same amount of time.

5.2.5 Heat of Hydration:

Fig. 4.24 shows the heats of hydration for SCM formulations. It can be observed that temperature decreased gradually and became constant after about 5 hours. So it can be suggested that there isn't any increase in temperature once the samples are casted. For all the SCM formulations, the initial temperature bracket was between 23-25 ^{O}C. Thereafter it kept on decreasing till 19^{O}C uniformly, showing retarding action of super-plasticizer. If the curve is observed carefully, a slight increase in temperature is noted. Since readings by TESTO does not prolong beyond about 17 hours, hence an idea of the trend of heat of hydration can be taken from calorimetric curve. For SCP formulations, second peak was attained after about 12 hours whereas for SCM formulations time taken to attain maximum temperature is more than 30 hours. Variables in SCM formulations are fine aggregate and liquid super-plasticizer i.e. Melflux 2510L. This delaying action can also be attributed to the restraint offered by the fine aggregate in the mix.

5.2.6 Strength:

It can be clearly seen from Figs. 4.25 & 4.26 that maximum flexural and compressive strengths have been exhibited by FA containing formulations, which is directly related to SP dosage to attain the target flow since water cement is kept constant for all the SCM formulations. If calorimetric curves of SCM formulations are analyzed, clear indication of effect of super-plasticizer can be observed. Strength development is greatly dependent upon three phases i.e. hydration, setting and hardening. The accelerated the hydration, more will be the strength enhancement for that particular formulation. For instance, SCM formulation with 10% SF (971) + 90% FA had the highest super-plasticizer content to achieve the target flow. Hence the convergence of maximum heat of hydration for the said formulation was also delayed even after 70 hours which resulted in the least strength. Hence, one can clearly state that increase in SP content is inversely proportional to the strength development.

CHAPTER- 6 CONCLUSIONS AND RECOMMENDATIONS

. Test results depicted that one must be sure about behavior of SRM's, determined by means of series of experiments namely particle characterization, chemical analysis etc. otherwise addition of SRM's without any background study, may lead to detrimental effects. Also, it was found that particle size, shape, internal porosity, chemical composition have imperative influence on the water demands, super-plasticizer contents, hydration kinetics, volume stability and strength development of SCP and SCM systems. Conclusions and recommendations from the present study are as follows;

- Fineness of SRMs is very vital to achieve since, it is directly associated with their pozzolanic activity. Finer the powder more will be the pozzolanic activity.
- With the inclusion of FA in self compacting cementitious mixes, effective water content increases which enhances workability.
- SF was incorporated in the mixes in the raw form as attained from the supplier which led to bigger sized voids in relatively coarser grains. This depressed the pozzolanic activity to a certain degree due to formation of these primary agglomerates.
- Addition of FA depresses the setting time as its addition retards hydration process sue to its slow pozzolanic activity.
- Measurement of heat of hydration of shrinkage samples gave a fair idea about the difference of time interval in attaining drying shrinkage, with all the mixes reaching their maximum temperature at the same instant they approach drying shrinkage. This shows start of setting phase since it is accompanied by hydration.
- Both grades of SF & FA have exhibited fairly good results for SCP & SCM mixes. Their implementation in SCC formulations must also be analyzed.
- SF content must not exceed 10% of cement mass since it leads to sufficiently high super-plasticizer demand and hence an uneconomical mix.
- Studies should be made to find the optimum percentages of SF & FA that can be used for self compacting concrete.
- There is more room for improvement as well as criteria regarding selection for super-plasticizers suitable for self compacting cementitious systems. In order to achieve the purpose, characterization of materials is indispensable.
- Both grades of SF & FA have exhibited fairly good results for SCP & SCM mixes. Their implementation in SCC formulations must also be analyzed.

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ANNEXURE- A

Table.1 Water Demands and Setting Times of SCP formulations

Sr. No.	Formulations	Water Demand (% of cement)	Vicat Reading (mm)	IST (min)	FST (min)
1.	CEM-I (Pure)	31	6	195	235
2.	CI -10 SF(971)- WD	37.5	5	181	215
3.	CI -10 SF(920)- WD	35.5	4	186	221
4.	CI -10 FA- WD	33.5	5	210	252

Table.2 Super-plasticizer Demand, Flow Spread and Flow Times using Hagermann's Cone of SCP formulations

Sr. No.	Formulations	Super-plasticizer Demand (% of cement)	Flow Spread (mm)	Flow Time (sec)	
				T25 (sec)	T30(sec)
1.	CEM-I (Pure)	0.28	304	2.26	18.38
2.	CI -10 SF(971)- WD	0.51	296	2.43	21.56
3.	CI -10 SF(920)- WD	0.39	298	2.23	20.53
4.	CI -10 FA- WD	0.30	308	2.08	18.75

Table.3 Flexural Strength of SCP formulations

Sr. No.	Formulations	Flexural Strength (MPa)			
		1 Day	3 Days	7 Days	28 Days
1.	CEM-I (Pure)	12.62	15.68	20.92	25.02
2.	CI -10 SF(971)- WD	6.49	11.27	15.79	20.63
3.	CI -10 SF(920)- WD	7.81	12.54	17.38	22.46
4.	CI -10 FA- WD	8.37	13.67	18.12	23.39

Table.4 Compressive Strength of SCP formulations

Sr. No.	Formulations	Compressive Strength (MPa)			
		1 Day	3 Days	7 Days	28 Days
1.	CEM-I (Pure)	68.18	80	90.2	105.06
2.	CI -10 SF(971)- WD	51.42	60.22	71.46	85.67
3.	CI -10 SF(920)- WD	57.36	65.68	79.06	91.34
4.	CI -10 FA- WD	66.58	73.14	88.56	98.27

ANNEXURE- B

Table.1 Super-plasticizer Demand, Flow Spread and Flow Times using Hagermann's Cone of SCM formulations

Sr. No.	Formulations	Super-plasticizer Demand (% of cement)	Flow Spread (mm)	Flow Time T25 (sec) (Hagermann)
1.	CEM-I	2.7	304	4.94
2.	CI – 0.9FA + 0.1SF(971)	8.23	313	20.73
3.	CI – 0.9FA + 0.1SF(920)	5.94	304	21.24
4.	CI – FA	4.5	306	19.19
5.	CI – 0.8FA + 0.2SF(971)	10.5	309	18.45
6.	CI – 0.8FA + 0.2SF(920)	7.65	302	19.14

Table.2 V-funnel flow times of SCM formulations

Sr. No.	Formulations	V-funnel Flow Time (sec)
1.	CEM-I	3.97
2.	CI – 0.9FA + 0.1SF(971)	17.83
3.	CI – 0.9FA + 0.1SF(920)	22.07
4.	CI – FA	17.23

Table.3 Flexural strength of SCM formulations

Sr. No.	Formulations	28 days Flexural strength (MPa)
1.	CEM-I	7.5
2.	CI – 0.9FA + 0.1SF(971)	8.72
3.	CI – 0.9FA + 0.1SF(920)	10.13
4.	CI – FA	11.55

Table.4 Compressive strength of SCM formulations

Sr. No.	Formulations	28 days Compressive strength
1.	CEM-I	55.52
2.	CI – 0.9FA + 0.1SF(971)	57.61
3.	CI – 0.9FA + 0.1SF(920)	63.33
4.	CI – FA	68.12