

**INFLUENCE OF BLENDS OF FLY ASH AND LIMESTONE POWDER
ON THE PROPERTIES OF SELF-COMPACTING PASTE SYSTEMS
UNDER VARIABLE MIXING WATER TEMPERATURE**



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This is to certify that the
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ABSTRACT

Self-Compacting Concrete (SCC) is a modern concrete system characterized by high flowability and high segregation resistance without mechanical vibration. ACI 237R – 07 defines SCC as “a highly flowable, non-segregating concrete that can spread into place, fill the formwork, and encapsulate the reinforcement without any mechanical consolidation”. SCC incorporates higher percentage of cementitious powders, lower w/p, and a super-plasticizer. This yields a workable, denser, durable and stronger concrete. There are various applications of SCC based on these characteristics, namely the heavy raft foundations, bridge piers, pavements, runways, tunnel linings, immersed tunnels and heavily reinforced sections.

In this study, the Self-Compacting Paste Systems (SCPS) have been studied as they control the properties of SCC both in fresh and hardened states and act as vehicles for the transport of aggregate during its flow. The study gives the comparative response of partial replacement of Ordinary Portland Cement grade-53 by the Secondary Raw Materials (SRMs) i.e. Fly ash (FA) and Limestone Powder (LSP) in Self-Compacting Paste Systems (SCPS) and the influence of mixing water temperature variation on the response of such systems. The parameters studied were the water demand (WD), super-plasticizer demand (SPD) for target flow of (30 ± 1) cm, flow times, initial and final setting times, calorimetry, and strengths of $4*4*16$ cm prisms at 1, 3, 7, and 28 days of age.

There were two aspects of this research. First was to determine the optimum replacement percentage of cement by equal mass blends of FA and LSP; second was to study the response of SCP systems to mixing water temperature variation at optimum replacement percentage.

The five mix proportions were considered; a neat self-compacting paste and 10, 20, 30 and 40 percent of cement replacement with equal mass blends of FA and LSP. The water requirement of the system increased if calculated as the w/c, while it decreased when calculated as w/p. SPD increased with increase in quantity of finer SRMs in system. Setting times and calorimetry showed that hydration was retarded by addition of blend of SRMs. Mixes were cast at WD of the respective system, cured and tested as per EN 196-1 in dual chamber. SRMs increased the 28-day strength of SCP systems up to 30 percent replacement, while 40 percent OPC replacement gave lower strength than

the neat SCP. The highest strengths of SCP system were at 20 and 30 % cement replacement by blend of FA and LSP because of finer particle size of SRMs and the better packing density.

These systems were then studied for the three mixing water temperature variations i.e. 10, 20 and 30 °C. While, water demand, w/c stayed constant for neat paste system, it increased for 20% and 30% cement replacement by blend of SRMs at 30 °C water. SPD increased for the systems as the mixing water temperature was lowered; there was a considerable high SP demand at 10 °C mixing water. A linear trend was observed with setting times; decrease in water temperature retards the initial and final setting. Using mixing water at higher temperature, the rate of release of heat of hydration in all systems increased. Consequently, peaks in the calorimetry curves occurred much earlier for the higher mixing water temperature. However, compressive strengths reduced for the paste systems as the water mixing temperature was varied from the (20 ± 1) °C.

Flowable and self-compacting concrete can be thus produced, conserving the natural resources, utilizing industrial waste, minimizing the compaction labor and costs and having a workable, pumpable, segregation-resistant mix with high strength and durability.

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LIST OF ACRONYMS

SCCS	Self-Compacting Cementitious System
SCP	Self-Compacting Paste
SCM	Self-Compacting Mortar
SCC	Self-Compacting Concrete
SRM	Secondary Raw Materials
OPC	Ordinary Portland Cement
FA	Fly Ash
LSP	Limestone Powder
WD	Water Demand
w/c	Water to Cement Ratio
w/p	Water to Powder Ratio
SP	Super-plasticizer
SPD	Super-plasticizer Demand
IST	Initial Setting Time
FST	Final Setting Time
PSD	Particle Size Distribution
BET	Brunauer, Emmett and Teller
XRD	X-Ray Diffraction
XRF	X-Ray Fluorescence
CH	Calcium Hydroxide
CSH	Calcium Silicate Hydrate
ACI	American Concrete Institute
ASTM	American Society for Testing and Materials

Chapter - 1

INTRODUCTION

1.1 General

Concrete is the most commonly used construction material. However, with the passage of time, conventional concrete is being replaced with high strength concrete (HSC), high performance concrete (HPC) and Self compacting concrete (SCC).

High strength concrete (HSC) is made possible by reducing the porosity, inhomogeneity, and micro cracks in the hydrated cement paste and the transition zone. The advancement in HSC lead to high performance concrete (HPC) which was designed not only to achieve even higher strengths than HSC but also to achieve enhanced performance. Mehta and Aitcin used the term, high-performance concrete (HPC) for concrete mixtures possessing high workability, high durability and high ultimate strength.

1.2 Self-Compacting Concrete (SCC)

Self-Compacting Concrete (SCC) is a kind of high performance concrete (HPC) which is more efficient in workability and resistant to segregation.

The European Guidelines for Self Compacting Concrete 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.”

In late 1980's and early 1990's SCC was developed in Japan by Professor Okamura and his PhD student Kazamasa Ozawa. Their study resulted in proposing a concrete that did not require any vibration to achieve full compaction and flowed under its own weight. The three basic requirements for SCC with respect to its state are as under:-

1. In fresh state it should be self-compactable and resistant to segregation and bleeding.
2. At early age should avoid initial defects like shrinkage.
3. After hardening should possess sufficient resistance against external factors

1.2.1 Self-Compacting Paste System

Self-Compacting Paste (SCP) is a component of SCC which define the properties of SCC such as bleeding, permeability, volume stability, strength and durability. SCP comprises the powder-water interface and mineral and chemical admixtures. SCP is responsible for transporting the aggregates and greatly influence the physical and chemical properties in fresh and hardened state of SCC.

This study is focused on SCP as it is the root of characteristics of SCC. Powders are those particles having size less than 125 μm . In SCP system the powder consists of Ordinary Portland Cement and the Secondary Raw Materials. SCP is characterized with the lower water-to-powder ratio (w/c) to minimize bleeding and segregation and/or addition of Viscosity Enhancing Agent (VEA) to have adequate workability and resistance to segregation. A superplasticizer is a chemical admixture, added to SCP to achieve the deformability and flow.

1.3 Secondary Raw Materials (SRMs)

A Pozzolan is defined as “A siliceous or siliceous and aluminous material, which in itself possess little or no cementitious property but it will, in finely divided form and in the presence of moisture; chemically reacts with calcium hydroxide at ordinary temperatures to form compounds possessing cementitious properties.”

Secondary Raw materials are pozzolanic or inert materials which can partially replace the cement and react with calcium hydroxide to form cementitious compounds. In this way, the micro-structure of the paste has less porosity and gives better packing arrangements. SRMs can be used to modify the properties of concrete such as early heat of evolution, shrinkage, packing, and flow.

Thence, SRMs enhance strength, improve microstructure, achieve economy, and the durability of cement based systems. Some of the commonly used SRMs are silica fume (SF), fly-ash (FA), limestone powder (LSP), rice-husk ash (RHA), and ground-granulated blast furnace slag (GGBFS).

The SRMs used in the following research work are Limestone Powder and Fly Ash.

1.3.1 Fly Ash (FA)

Fly ash is one of coal combustion products in the electricity-generating power plants. The fly ash is pozzolanic and contains high quantity of silicates, alumina and ferric oxides. When used as a Supplementary Cementitious Materials (SCM), it helps in the hydration phases of hydraulic cement and with its fine size, it enhances the packing, workability, and durability of concrete.

1.3.2 Limestone Powder (LSP)

Limestone Powder is a natural occurring secondary raw material. Uses of Limestone range from an ingredient in cement clinker production to aggregate in concrete. But the composition of Limestone allows its usage as Supplementary Cementitious Material because of high Calcium Carbonate content. It may have low level of pozzolanic activity but finer the powder, it becomes active and provide the nucleation sites for hydration of cement and donates calcium hydroxide in the early ages.

1.3.3 Blend of FA and LSP:

A binary system has a cement replacement by a single SCM, this has some limitations and undesirable influences on properties of concrete. The combination of two SCMs forms a ternary system and it is postulated that one SCM can counter the negative traits of the other SCM.

Fly ash retards the setting while Limestone reduces the compressive strength at higher OPC replacement levels. However, using FA and LSP as a blend gives higher strength, reduces permeability and improves workability.

At the same time the availability of FA is reducing and quality of FA may be different from different coal burning power plants. Limestone particle size may be coarser. The ternary system may yield economy and quality to the concrete.

1.4 Super-plasticizers (SPs)

SPs or the High-Range Water Reducers (HRWRs) are chemical admixtures which impart flowability and workability to SCC systems at lower w/c ratio. Basically, SPs disperse the cement grains and reduce inter-particle dispersion, allowing the reduction of water content.

SPs have effect on both fresh and hardened properties of system. They affect the setting times of system; can be used as Accelerator or Retarder in a hydration reaction. HRWRs also improve the microstructure and the durability of the concrete as the workability requirement is fulfilled at lower W/C ratio and hence lesser free water, capillary pores and empty spaces in the system.

1.5 Research Objectives:

The objectives of this research are to study the production of a Self-Compacting Paste System. Economic and environmental friendly replacement of OPC in the system by incorporating the Secondary Raw Materials. Determination of a viable replacement percentage which can enhance the fresh and hardened properties of concrete. And studying the effect of mixing water temperature on the properties of SCCS.

Chapter - 2

LITERATURE REVIEW

2.1 Self Compacting Paste Systems

Self-compacting Paste (SCP) systems are the agents that transport the aggregates and define the properties of self-compacting mortar (SCM) systems and self-compacting concrete (SCC) systems [1]. Addition of different secondary raw materials improve the properties of the SCCS considerably and can only be achieved by the optimization of its major components self-compacting paste (SCP) and self-compacting mortar (SCM). Self-compacting cementitious system (SCCS) has three types:

Self-Compacting Paste (SCP) System or the Single Component System

Self-Compacting Mortars (SCM) System or the Two Component System

Self-Compacting Concrete (SCC) System the Three Component System

2.1.1 Development History

During early eighties there existed a problem of the durability of concrete structures. To make concrete structures durable, sufficient compaction by skilled workers and precise quality control was required. However, the high costs associated with the skilled workers led to the development of a concrete that can flow under its own weight and fill the form work without external work. Self-compacting concrete was proposed by Professor Hajime Okamura in 1986. Studies to develop self-compacting concrete, including a fundamental study on the workability of concrete by Ozawa and Maekawa at the University of Tokyo [2].

The first prototype was created in 1988 using materials already available in the market. The prototype performed satisfactorily with regard to drying, hardening shrinkage, heat of hydration and denseness after hardening along with other properties, was named “High Performance Concrete.”

Professor Aitcin had already defined “High Performance Concrete” as the one with high durability due to low water-cement ratio [3]. Since then, the term high performance concrete has been used to refer to high durability concrete. Therefore, Okamura has

changed the term for the proposed concrete to “Self-Compacting High Performance Concrete.” [4]

Through invention of self-compacting concrete, the need for skilled labor was considerably reduced and concrete could be placed at any area without the need of any mechanical device. This new technology presented economic and environmental benefits over traditional methods. Better travel rates of concrete placement, smooth and easy flow of concrete around heavy and dense reinforcement, faster construction and less noise due to the absence of mechanical devices are some of the associated benefits of SCC.

2.1.2 Characteristics of Self Compacting Concrete

Khayat et al [5] defines SCC as “Self Compacting Concrete is a special type of concrete that should flow into place and around obstacles under its own weight without segregation and flow blockage and with no significant separation of material constituents thereafter until the setting”. According to this definition SCC must have adequate flow ability along with the resistance to segregation in reaching into those places where normal and conventional concrete can't reach without the help of manual vibration.

SCC should meet the strength, durability and workability criteria. The SCC's mix design must be cooked in such a way that there is a good balance between deformability, stability and preventing the blockage of concrete flow.

SCC must have the high flow ability and high resistance to segregation that is only possible with the use of appropriate HRWR and the proper w/p ratio. Water-to-powder ratio (w/p) is actually the ratio of the water to powder that includes cementitious materials and various fillers, such as limestone filler. As the reduction of the water-to-powder ratio (w/p) can limit the deformability of the cement paste, an increase in w/p is needed; however, it can also reduce the cohesiveness of the paste and mortar ultimately leading to segregation of fine and coarse aggregate particles, hence causing blockage of the flow. When we talk about the deformity, inter particle friction between the various solids (coarse aggregate, sand, and powder materials) also comes into play. This friction increases the internal resistance to flow, thus limiting deformity and speed of concrete flow. The use of HRWR such as Melflux can spread the cement grains and

cause a reduction in the inter particle friction and maintain the high level of viscosity and flow ability by enabling the reduction in water content. Well graded aggregates and the cementitious material can also exhibit lower inter particle friction that can also be achieved by lowering the aggregate content and increasing the paste volume.

Stability of the SCC is another major parameter for the workability of SCC. Highly flow able concrete that might be stable once mixed may undergo segregation when placed or pumped because of the pseudo-plastic nature of the concrete. Concrete with low cohesiveness can segregate as it cannot maintain proper suspension of particles. As concrete deforms around a restriction, a portion of the coarse aggregate can begin to segregate, which results in arching of the aggregate, and hence, blockage of the flow [6]. This problem can be addressed by reducing the spacing of the aggregate by limiting aggregate content, reducing the MSA and increasing cohesion and viscosity as stated above. Moreover the bleeding can be reduced by lowering the water content, reduction in w/p ratio and using the powder with higher surface area.

2.2 Secondary Raw Materials

2.2.1 Fly Ash

Fly ash also known as pozzolana is a byproduct from burning pulverized coal in electric power generating plants [7]. The exhaust gases from the power plants are the major source of fly ash that are formed by the burning of the pulverized coal with or without combustion materials. Chemically, fly ash comprises of alumina, ferrous oxides and high contents of silica and is collected through electron precipitator. Canadian Centre for Mineral Technology (CANMET) and Energy first developed High-volume fly ash concrete for structural in late 1980s. [8].

ASTM C 618-89 classifies the fly ash into two main classes

Class F: Lime content of class F fly ash is usually under 15 percent and contains the combination of silica, alumina and iron oxides greater than 70 percent of total composition. It is available in the largest quantities.

Class C: Lime content of class C fly ash is generally more than 15 percent often as high as 30 percent and contains the combination of silica, alumina and iron oxides greater than 50 percent but less than 70 percent of total composition. Elevated CaO may give Class C unique self-hardening and pozzolanic characteristics.

European standard, EN-197-1 also classifies fly ash into two classes

Siliceous: Lime content is less than 10 percent

Calcareous: Lime content is more than 10 percent

Fly ash is composed of extremely fine spherical, glassy and hydrophilic particles that have retarding effect on setting time [9, 10, and 11]. The fly ash used in our study was Class F that is produced from burning of harder, older anthracite and bituminous coal.

Particle size, specific surface and particle shape of the fly ash have significant effect on the performance of the fly ash. On average the size of the particle is lesser than the cement thus having larger specific surface. The smaller sized particle of the fly ash fills the voids created in the cement particles causing a decrease in the porosity and increase in the durability of the concrete. Also fly ash reduces the water demand by filling the pores that required to be filled by water. Finer the fly ash, the larger the specific surface

area is, thus the more their surfaces layer water demand is. Research shows that fly ash particles are like small balls that play a vital role in lubrication in the flow process of the mortar. [12] Traditionally, fly ash used in structural concrete is limited to 15% to 25% cement replacement [13, 14].

2.2.2 Lime stone Powder

Filler

A filler is a very finely-ground inert material, of about the same fineness as Portland cement. According to Neville [15] filler can be naturally occurring materials or processed inorganic mineral materials. Fillers are typically recommended for improving both physical and chemical properties of hydrated products in concrete such as workability, density, permeability, structure bleeding, and cracking tendency. [16].

Limestone

Limestone is a sedimentary rocks primarily of calcium carbonate. Lime stones are generally obtained from the calcareous remains of marine or fresh water organisms embedded in calcareous mud. They change from the soft chalks to hard crystalline rocks [17]. It is used as non pozzolanic filler material in the SSCS.

According to Livesy [18] limestone-filled cements have been developed in Europe during the last twenty years. There have been numerous studies in Thailand in the 1990s on the durability of limestone powder cement concrete [19]

ENV 197-1: 1992 limits the filler content to 5 per cent but allows the use of limestone up to 35 per cent, provided the remaining cementitious material is Portland cement only. Adding the limestone is both environmentally and economically beneficial as it will cost lesser to create and ground less clinker.

Carlsward et al [20], reported that Limestone powder (LSP) usually increases the yield stress but has negligible effect on the plastic viscosity and the slump flow of the mix. LSP only acts as a filler in the SCC mix, which do not take part in any cement hydration reaction [21]. However research shows that although limestone doesn't have cementitious properties but can still contribute to strength development [22], because the very finely grounded limestone particles provide the nucleation site to be used by

products of cement hydration. This accelerates the strength development during the early age.

LSP increases the early strength, controls bleeding in concrete with low cement content [23]. Limestone reacts with cement hydrate products producing a cementitious reactant. Limestone powder may reduce the drying shrinkage of the concrete and decrease the water absorption [24]. Limestone powder (LSP), a chemically inert filler, have advantages like increased segregation resistance, reduced cost, lower heat evolution, reduced carbonation [Tanaka et al (1993)] and reduced shrinkage [Ogawa et al (1995)]. Limestone powder accelerates the cement hydration, especially the C3S phase, by acting as nucleation site for portlandite and CSH precipitation [5-7 art 04n58]. The accelerating effect can give rise to a slightly higher compressive strength at early age at moderate cement replacements (<10%). At later age, replacing part of the OPC with limestone powder may result in a strength reduction, due to the replacement of the more reactive component, OPC, with less or non-reactive one [8 art04 n 58]. This effect is referred to as the dilution effect.

Chemical factors include the effect of limestone filler in supplying ions into the phase solution, thus modifying the kinetics of hydration and the morphology of hydration products (Daimon & Sakai 1998). Researchers have also found that partial replacement of cement by an equal volume of limestone powder varying from 5% to 20% resulted in an enhancement of the fluidity of high-performance concrete having a W/C ratio ranging between 0.35 and 0.41 (Nehdi et al 1998).

2.2.3 Fly ash and Limestone Powder Synergy:

Early strength of the cement is improved by the limestone's acceleration effect on hydration process while fly ash retards the initial hydration. However the combined effect of both limestone and fly ash tend to nullify the effects of each other. When we replace OPC by fly ash and limestone collectively, there is higher amount of aluminate content incorporated in the system due to fly ash. These aluminates react with limestone powder and enhance its effects on the cement paste system [25]. Studies reveal that blends of LSP and FA improve the strength and overall response of the system [ACTA 2012 pg. 91]. With the blend of LSP and FA more water was bound and the hydration products formed were proven to contain calcium carboaluminate hydrate.

According to De Weerd K., Justnes H., Kjellsen K.O. & Sellevold E.J. Nordic Concrete Research, 2010, Vol.42 (2) pp. 51-70 the 65% OPC + 30% FA + 5% limestone blend had a slightly higher strength than the 70% OPC + 30% FA blend, illustrating that due to the synergetic effect between fly ash and limestone powder, 5% additional OPC can be replaced.

2.3 High Range Water Reducers / Superplasticizers

A super plasticizer is an artificially prepared water soluble organic substances material, used as an ingredient of concrete along with water cement and aggregates added to the batch immediately before or during the mixing to decrease the amount of water considerably, needed to achieve a certain consistency of concrete which means higher compressive strength workability, durability and hence less porosity. The earliest known published reference to the use of small amounts of organic material to increase the fluidity of cement containing composition was made in 1932 [31] where polymerized naphthalene formaldehyde sulphonate salts were claimed as useful in this role. Requirement of high degree of workability and the retention are basic warrants that require Super Plasticizers (IS 9103 – 1999) to be used. The super plasticizer and mineral admixture hold the aggregates in suspension, and the combination of powder materials is also used to control the hardened properties, such as strength [32]. They may also be employed in the situations like in high temperatures to counter rapid slump loss or when there is delay between mixing and placing of the concrete. Studies showed that with SP water can be reduced up to 32-33% [33].

Superplasticizers can be used in concrete mixtures for three different purposes:

To increase workability, at a given mix composition, in order to enhance placing characteristics of concrete;

To reduce the mixing water, at given cement content and workability, in order to reduce the water-cement ratio (w/c) and therefore to increase strength and improve durability;

To reduce both water and cement, at given workability and strength, in order to save cement and reduce creep, shrinkage, and thermal strains caused by heat of cement hydration.

2.3.1 Mel-flux 2651 F:

Mel-flux 2651 F is one of the super-plasticizer used in the production of self-compacting concrete. It is a free-flowing spray dried powder of modified poly-carboxylic ether. High performance super-plasticizer for cement based construction materials is produced using Mel-flux 2651 F. It is optimized for reducing water of concrete and to provide excellent early strength.

Mel-flux is based on latest polymer technology. It contains side chains which are based on polyethylene glycol. Due to raw material production of the polyethylene glycol, the nature of radical polymerization and the composition, it is known that this technical product has some natural color variations from nearly colorless to yellowish up to slightly brownish. But these color variations don't affect the performance of the super-plasticizer.

It has following applications:

- Self-leveling underlayment (SLU)
- Feather edge products
- Cementitious fast-setting floor screeds
- Cementitious self-leveling floor screeds
- Injection mortars
- Repair mortars
- Non-shrink grouts
- Cementitious floor screeds
- Tile adhesives and joint fillers
- Dry mix concrete

2.4 Properties of self-compacting concrete

SCC is able to flow under its own weight. Making the concrete more affordable along with the ease of placement in congested reinforcement by the elimination of manual vibrators and high flow ability [26]

As use of SCC removes compaction, this evades the internal segregation between solid particles and the adjoining liquid which results in less porous transition zones between paste and aggregate and color of concrete is enhanced [26]. Moreover, increased strength and high durability can be achieved.

SCC are environmentally sustainable because their production includes less energy consumption, CO₂ foot print is minimum as compared to the Conventional Concrete Systems and more secondary raw materials are used [27].

Self-compacting reduces the noise pollution by removing the use of mechanical vibrators. Also SCC is energy efficient [28].

SRMs in the SCC fill up the voids and the mix formed is denser and shows a lower permeability and absorption by capillary action, due to less porous zone and refinement of pore structure [29].

Super plasticizer demand also decrease for the SCC as compared to the conventional one as per FA's lubricating effect. [30].

2.5 Experimental Techniques

2.5.1 Particle Size Distribution (PSD)

Particle size distribution" is an index (means of expression) that indicates the sizes (particle size) of particles which are present and in what proportions that is relative particle amount as a percentage where the total amount of particles in the respective sample particle group to be measured. To predict the actual performance of the cementitious materials and their computational modeling requires the accurate PSD results. ASTM115-96 defines the method for the determination of the fineness. This stubudimetic method has a lower detection limit of 7.5 μm [37]. Different techniques are also being employed that can even give more precision like the laser diffraction measurement and Blaine fineness test. However the laser technique is more efficient due to automation and lesser time consumption. The laser technique obtains the information about the particle size distribution measurement by assuming a specific gravity for the particles but still this technique have not been opted as a standard in the industry [38].

2.5.2 Calorimetry

Calorimetry is the science of measuring the heat flow that accompanies either a physical or chemical change. The device used to measure the heat flow is called Calorimeter. It consists of a wooden vessel that is isolated thermally from surrounding. Thermometer and heat sensors are used to measure the temperature of the contents, different mixes when mixed in the calorimeter they exchange heat with each other. Higher temperature mixes loose heat while lower temperature gains it. The principle of the calorimeter states that total heat loss is equal to total heat gained.

Hydration of the cement is extremely exothermic process that take place in a series of steps. These steps have been identified by Young, 1985 as:

- Rapid Initial Process
- Dormant Period
- Acceleration Period
- Retardation Period
- Long-term reactions

Properties like workability, strength development rates, setting times, pore structure development, early and long term performances of cement concrete all depends on the heat of hydration. Study of the hydration kinetics requires the measurement of heat production that is done by Calorimetry. Initially the heat of hydration is very high due to the hydration of the aluminate phase, C₃A, and Aft as the main product. Then follows the induction or dormant period in which the heat of hydration decreases to minimum within 2-3 hours. The pozzolanic reaction and the precipitation of the hydration products especially CSH Gel follow the dormant period after 15-18 hours in the case of SCC. Later on the process slows down and there are smaller peaks. Amount of heat liberated depend upon the cement type, the water to cement ratio, the blends and the percentage replacement of OPC. Rizwan et al [34] stated that super-plasticizer's presence in the cementitious systems delays the heap peaks produced during Calorimetry of cement process.

2.5.3 X-Ray Diffraction (XRD)

X-ray diffraction phenomenon results from the scattering process in which these rays are scattered by the electrons of atoms at constant wavelength. The pattern of the diffraction not only gives information about the pattern of the crystal consisting both position and physical property but also elucidation of its structure [35]

It is a strong and efficient experimental technique used for detection and quantification of crystalline materials in crystalline and amorphous substances. The microstructures identification and the comprehensive explanation can be studied by the results of XRD. Two types of XRD are performed on cementitious materials the quantitative and the qualitative. Although the quantitative analysis is more accurate but it is not usually used as being more time consuming, expensive and requiring special expertise.

XRD is also done to determine calcium sulphate that is unreacted in the cement and this is done by study of the amount of formed hydrates with time [36].

2.5.4 X-Ray Fluorescence (XRF)

X-ray Fluorescence is an experimental technique that is used for non-destructive chemical analysis of minerals, rocks and construction materials. In this technique, the emission of a characteristic X-ray takes place from a material when it is excited by bombarding with high energy rays. XRF gives the complete chemical analysis of the material giving the percentages of the elements present in the material.

Chapter - 3

EXPERIMENTAL PROGRAM

3.1 General:

The study of fresh and hardened properties of Self-Compacting Paste Systems (SCPS) requires first the determination of parameters that may influence the Paste characteristics; followed by studying the outcomes of changing these parameters.

Analyzing the influence of blends of Supplementary Cementitious Materials (SCMs), commonly known as Secondary Raw Materials (SRMs), with variable mixing water temperature encompassed a three step process:

- Study of physical and chemical properties of materials.
- Study of formulations with cement partially replaced with the blend of SRMs.
- Study of formulations' response to variable mixing water temperature.

The response was studied by keeping all external parameters maintained within a specific range of variation and noting down the values for each process like mixing and curing. This gave the consistent set of results for the involved variables i.e. OPC Replacement Percentage and Mixing Water Temperature.

The first phase in the project was the acquisition of materials and standard equipment.

3.2 Materials:

3.2.1 Cement:

The basic component in the Paste is the Cement. Hydration of cement results in the formation of the hard porous solid.

Lafarge Stallion is used; it is Ordinary Portland Cement, EN 197-1 CEM 1 42.5 grade, with clinker of low C3A content and grinded with reduced gypsum proportion than other available cements. It has a Blaine fineness of 3100-3200 cm²/g and a sieve residue greater than 45µm of 9-11 % by mass.

Eight bags were purchased from the primary distributor so that variation in different batches don't affect the research. Bags were stored in air-tight conditions.

3.2.2 Secondary Raw Materials:

3.2.2.1 Fly Ash:

A dark grey colored powdered fly ash; processed and refined at the Heyden Power Plant of E.ON Kraftwerke GmbH was used. It is a research grade, fine-grained pozzolanic binder, classified as class F fly ash because silica, alumina and ferric oxides exceed 70% of the total composition. The EFA-Fuller® HP fly ash offers cement replacement, with reduction of system's water demand, improved workability, higher compactability and resistance to chemical attacks. As moisture affects the pozzolanic natured powder, so fly ash was also stored in air-tight buckets.

Table 3.1 Properties of Fly Ash

Fly Ash, EFA-Fuller® HP Properties	
Physical Shape	Powder
Color	Dark grey
Loss on Ignition	5.0%
Bulk Density	1200 kg/m ³
Particle Density	2300 kg/m ³
Sieve Residue > 45 µm	(20 ± 10) % by mass

3.2.2.2 Limestone Powder:

Limestone Powder was bought from a Crusher in Taxila, Rawalpindi. Limestone was washed and dried, after which milled to reduce its size and then passed through ASTM #350 sieve. It had a uniform grey color and contained high percentage of calcium oxide.

Table 3.2 Oxides Content in Powders through XRF

Oxides	OPC	Fly Ash	Limestone Powder
Al₂O₃	ND	ND	ND
SiO₂	49.81	62.14	22.09
CaO	43.33	10.26	74.21
Fe₂O₃	5.61	22.44	2.23

3.2.3 Super-plasticizer:

A third generation super-plasticizer, a spray dried powder having poly-carboxylic ether was used. It is essentially for the flowability, plastification and reducing water needed to achieve the target flow of (30 ± 1) cm. Melflux 2651-F is manufactured at Germany by BASF and exported in 1 Kg air-tight jars.

Table 3.3 Properties of Super-plasticizer

Melflux 2651-F Properties	
Physical Shape	Powder
Color	Yellow to Brown
Drying Loss	Max 2.0%
Bulk Density	300-600 kg/m ³
Recommended Dosage	0.05-1.00% of cementitious mass
pH value at 20°C	6.5-8.5

3.2.4 Water:

Tap water from Structure Lab, NICE was stored in the container so that to achieve uniformity in the temperature and properties of water. The variations from the water source are also minimized. The characteristics of water are studied in the IESE lab.

3.3 Physical Analysis of Materials:

It included the determination of physical properties of dry powders of OPC and SRMs. Among the properties were: particle sizes, densities, surface area, shape and porosity.

3.3.1 Particle Size Distribution:

The test was performed on the OPC and SRMs with the objective to find the distribution of particle sizes. The apparatus used was Horiba Particle Size Analyzer in the Surface Engineering Lab at SCME. The dispersing agent used was ethanol. The samples were prepared in the 500 ml beakers by stirring 5g sample in the ethanol. The suspension was poured in the apparatus which gave quantitative and graphical measure of each particle size.

The well-graded powders ensure better packing and high density, thus a durable and less porous pastes. With smaller size particles, the surface area increases and the hydration undergoes faster and yields better strength.

3.3.2 BET Area:

Brunauer–Emmett–Teller (BET) gives the specific surface area of the materials. The test was performed in Surface Engineering Lab at SCME. The de-gassing conditions were set at 4 hours and the specific surface area of powders was found by physical adsorption of a gas on the surface and calculating the adsorbate gas quantity corresponding to a monomolecular layer on the surface.

3.4 Chemical Analysis of Materials:

3.4.1 XRF:

Determination of chemical composition by bombarding the material with high-energy X-rays. This causes elements to emit secondary X-rays which are characteristic of elements and therefore used for identification.

The XRF is performed at the IESE lab and the results are annexed.

3.4.2 XRD:

Diffraction of incident X-rays in specific directions allows the identification of atomic and molecular structures. OPC, Flyash and Limestone powder are tested at SCME XRD Lab to get the the mineral composition and crystal formation. Results are annexed.

3.5 Mix Proportions:

As per the literature, the synergy effect of blends of Secondary Raw Materials enhance the fresh and hardened state properties. The blends having a mix of particle size give a better filler effect, reduces pores and increases density and strength.

The formulations initially considered are the cement replacements at 10, 20, 30 and 40 percent with equal percentages of FA and LSP in each. One Control formulation is also undertaken which is a neat cement paste having no replacement.

- CI-00 pure OPC
- CI-FA5-LSP5 5% of OPC replaced with each FA and LSP
- CI-FA10-LSP10 10% of OPC replaced with each FA and LSP
- CI-FA15-LSP15 15% of OPC replaced with each FA and LSP
- CI-FA20-LSP20 20% of OPC replaced with each FA and LSP

Afterwards the formulations giving the better results for strengths are undertaken for the effect of Variable Water Mixing Temperature. The temperatures considered are 10, 20 and 30 °C.

3.6 Testing Procedures:

3.6.1 Determination of Standard Consistency:

Commonly referred to as Water Demand of the mix. It is the minimum amount of water required for the complete hydration of the cement. Determined through the Vicat Apparatus when 10 mm dia plunger penetrates in the conical mold of 40 cm height such that distance between plunger and base-plate is (6 ± 2) mm.

3.6.2 Determination of Super-Plasticizer Demand:

The key feature of Self-Consolidating Pastes is their flowability and resistance to segregation, and it results due to the addition of Super-plasticizer. The trials were

performed with the formulations mixed at their Water Demand to achieve the target flow of (30 ± 1) cm. A glass surface with markings of 25 cm and 30 cm and a Hagerman's Mini-Slump flow cone (top dia 6 cm, bottom dia 10 cm and height 7 cm) were used.

3.6.3 Flow Times:

The times T25 and T30 which are the time taken to achieve 25cm and 30cm flow targets respectively. They are measured for the formulations at their water demand and superplasticizer demand. Stopwatch with a least count of 0.01 seconds is used.

The flow times give the speed of flow and are indicative of the rate of deformation.

3.6.4 Setting Times:

Initial and Final setting times for the formulations are measured using Vicat Apparatus. Initial setting time is given by the time elapsed after mixing when the 1 cm dia Vicat needle cannot reach the distance (6 ± 2) cm from bottom of cone. Final setting time is when the needle fails to leave the circular print of 3 cm dia circle.

To ascertain the effect of SP, the setting times with and without SP are determined.

3.6.5 Casting:

The mixes are cast in the rectangular moulds of 40*40*160 mm. conforming to the European Standards EN 196-1. Prisms are de-moulded after one day and put in Curing tank till the time of testing.

3.6.6 Strength Testing:

The prisms cast are tested for 1, 3, 7 and 28th day flexure and compressive strengths. One-day testing is done soon after de-moulding while prisms are put in curing tanks upto the date for 3, 7 and 28th day strengths and then tested in Saturated Surface Dry (SSD) condition.

The loading for flexure @ .02 kN/sec and for compression @ 0.2 kN/sec. A prism is placed in Dual Strength Chamber with Flexural arrangement, which breaks the sample in half. Each half then tested for compressive strength with Compression arrangement in Dual Strength Chamber.

3.6.7 Water Absorption:

Casting is done for the Water Absorption test, where a prism is weighed in SSD on the 1, 3, 7 and 28th day to measure the water absorbed by the mix proportion during the curing. This is an indicative of porosity.

3.6.8 Calorimetry:

Hydration is an exothermic reaction i.e. hydration proceeds with the heat release from the system. The calorimeter F-Cal 8000 logs the thermal data and indicates the rise in temperature over time during the reaction.

Mixtures are prepared and 800 grams poured in the calorimeter cylinders. A two-day data and curves are obtained for each mix.

3.6.9 Shrinkage:

Shrinkage apparatus gives the expansion and contraction that occurs during the hydration reaction. The apparatus is imported from Germany with 2-chambers, each fitted with a sensor that monitors the change in length with the least count of 0.1 micrometer.

3.6.10 Variation of Mixing Water Temperature:

The effect of water temperature is studied by adding water at three different temperatures i.e. 10, 20 and 30 degrees centigrade. The mix proportions considered are those which give better results of strength at the room temperature conditions.

Water temperature is varied by using Ice for cooling down the temperature and using the Water Dispenser for hot water. The temperatures are maintained in a separate container. Prior to mixing, the water quantity is weighed and temperature maintained in the Hobart Mixer Bowl so that the heat exchange from the bowl is neglected during mixing.

The water demand, SP demand, flow times, initial and final setting times, calorimetry and casting are performed with the varying water temperatures and compared with the room temperature conditions.

Chapter – 4

RESULTS

4.1 General

The formulations have been studied by replacing a percentage of OPC in the Self-Compacting Paste system by a blend of Fly Ash and Limestone Powder. First it is done at the controlled water mixing temperature of (20 ± 1) °C. After the determination of 28-Day strengths, the two replacement percentages giving better results are undertaken for their response to variable water mixing temperature; one temperature depicting hotter climates (30 ± 1) °C and the other temperature for cooler environments (10 ± 1) °C.

4.2 Self-Compacting Pastes with SRM Blends

This section gives the formulations tested to find the optimum replacement percentage. As discussed in the literature that SCCS often involves the incorporation of high volume of cementitious materials and fillers to bind some of free water [28], therefore high replacement levels of upto 40% are assessed.

The results produced in this chapter are the brief of several trials and verifications on each mix formulation. The testing is done at the mixing water temperature of (20 ± 1) °C. The lab temperature and relative humidity are constantly monitored. On average, lab temperature is at (20 ± 2) °C and relative humidity greater than 50% which are according to EN 196-3 Standards.

Mix Proportions are denoted as following:

Table 4.1 Mix Proportions

Mix Proportion	OPC (%)	FA (%)	LSP (%)	Denoted by
CI-00-WD	100	0	0	CI
CI-FA5-LSP5-WD	90	5	5	FL5
CI-FA10-LSP10-WD	80	10	10	FL10
CI-FA15-LSP15-WD	70	15	15	FL15
CI-FA20-LSP20-WD	60	20	20	FL20

Particle Size Distribution and Gradation Curve

As stated in the literature that incorporation of powder materials with different particle size distribution and morphology improves the packing density, reduces inter-particle friction and density; thus improving the flow, stability and self-compactability. Therefore, the foremost test is to analyze the distribution of particle sizes and the gradation curve of each of the powders i.e. OPC, FA and LSP. [29, 30, 31 and 32]

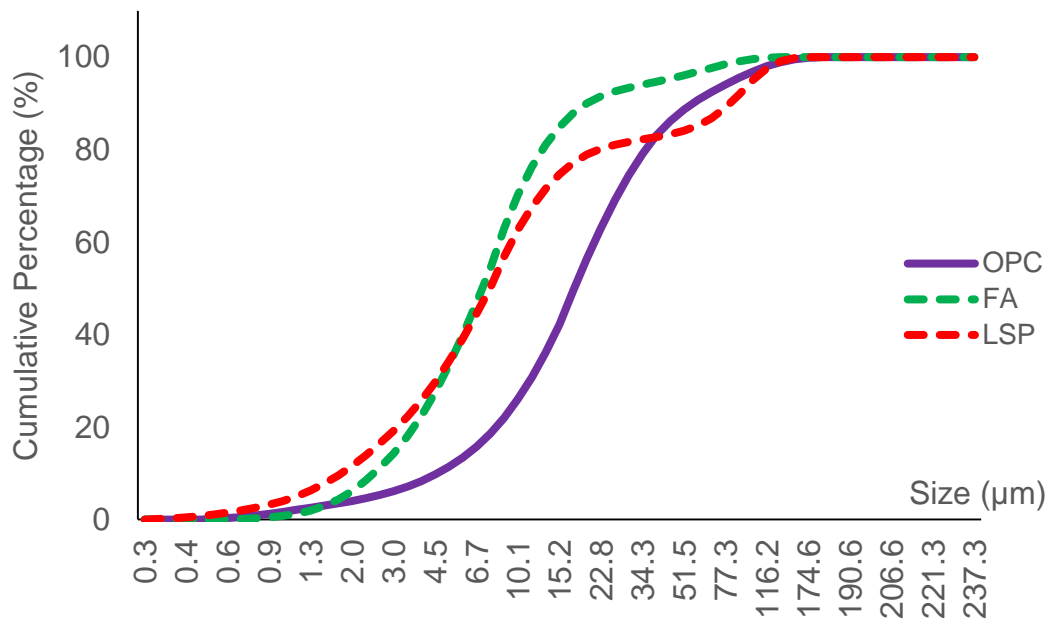


Fig. 4.1 Particle Size Distribution Curve

4.2.1 Water Demand:

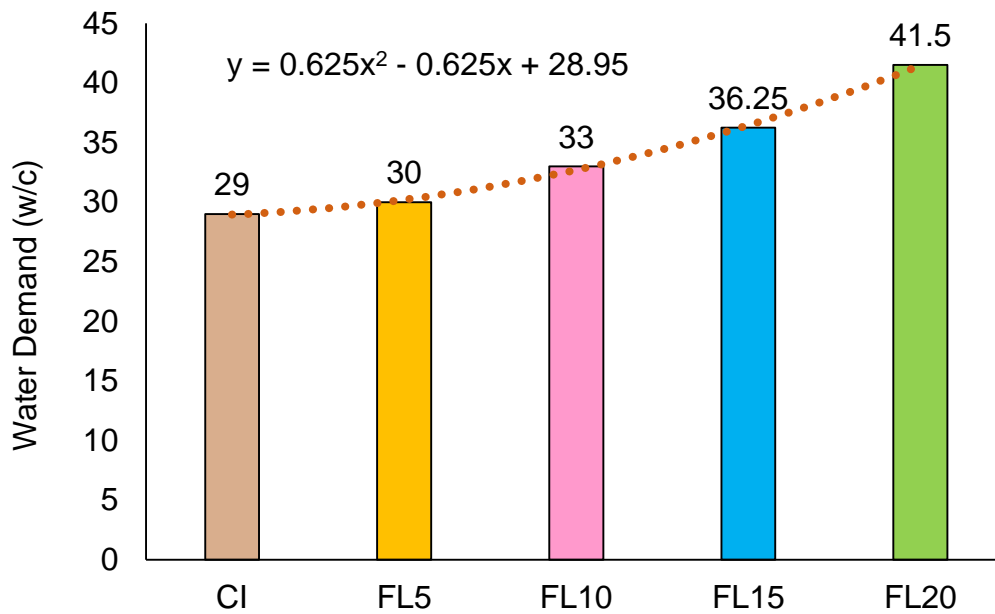


Figure 4.2.1 (a) Water Demand of Mixes w.r.t Cement Mass

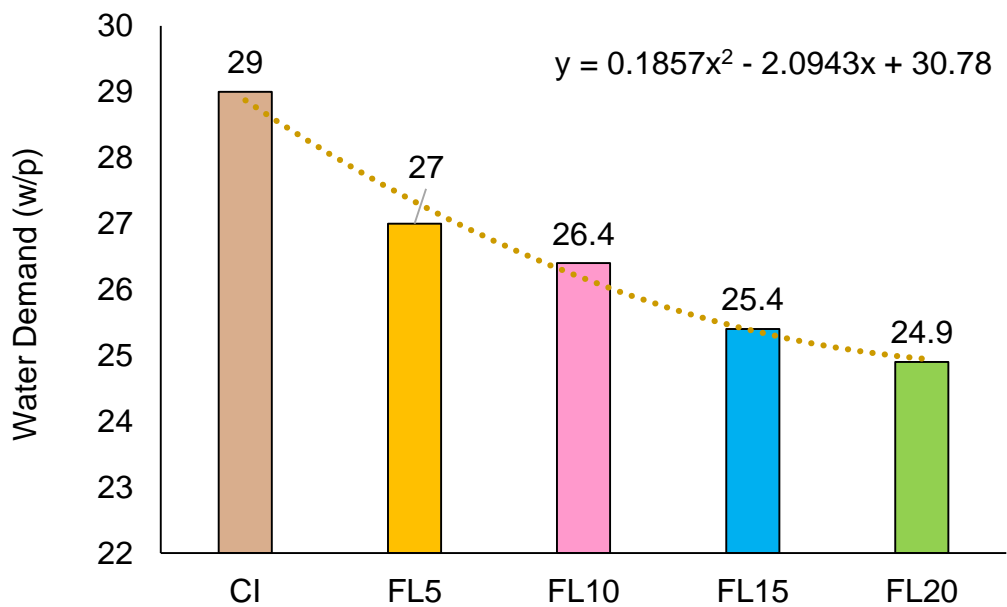


Figure 4.2.1 (b) Water Demand of Mixes w.r.t Powder Mass

The graphs show that with the cement replaced in the system, the total volume of water requirement decreases. But due to the finer SRMs, the percentage of water w.r.t cement

mass increases. This occurs due to the greater density and resistance offered to the Vicat apparatus plunger. There are less air pores and better packing of the paste.

Water is required for the hydration of cement, and with cement replaced with SRMs, less water is required in the system. This is beneficial in preventing bleeding and segregation.

4.2.2 Super-Plasticizer Demand:

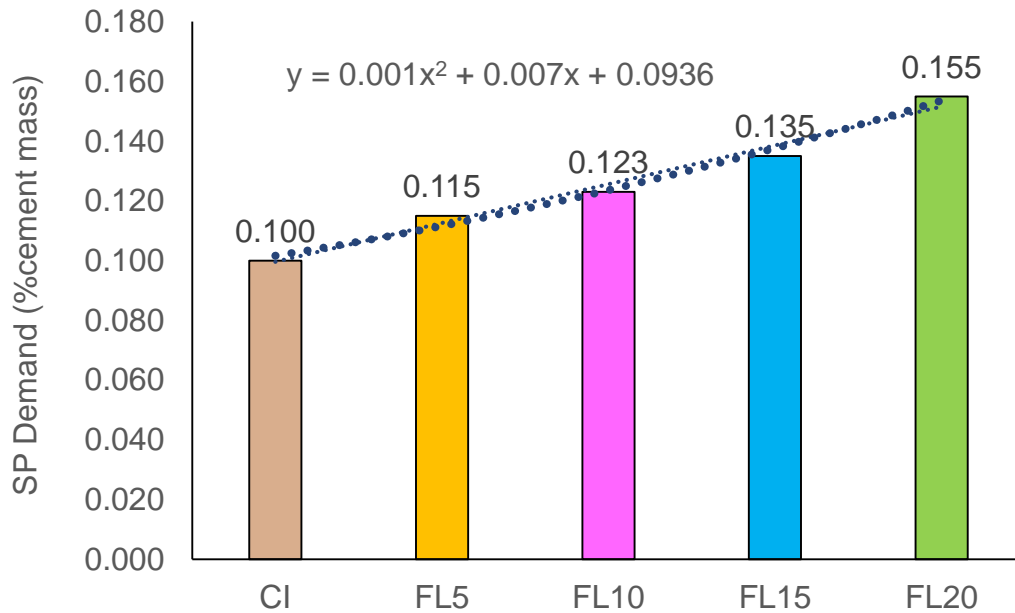


Figure 4.2.2 Superplasticizer Demand of Mixes

SRMs have fine particle size, this increases the surface area, causing higher viscosity and resistance to flow. Therefore, the target flow is achieved with greater quantity of superplasticizer when quantity of SRMs in the system is increased.

4.2.3 Flow Times

With higher SP content, the flow times reduce. However at 40% OPC replacement, distance between cement particles is high, requiring considerably more time than at 30% OPC replacement.

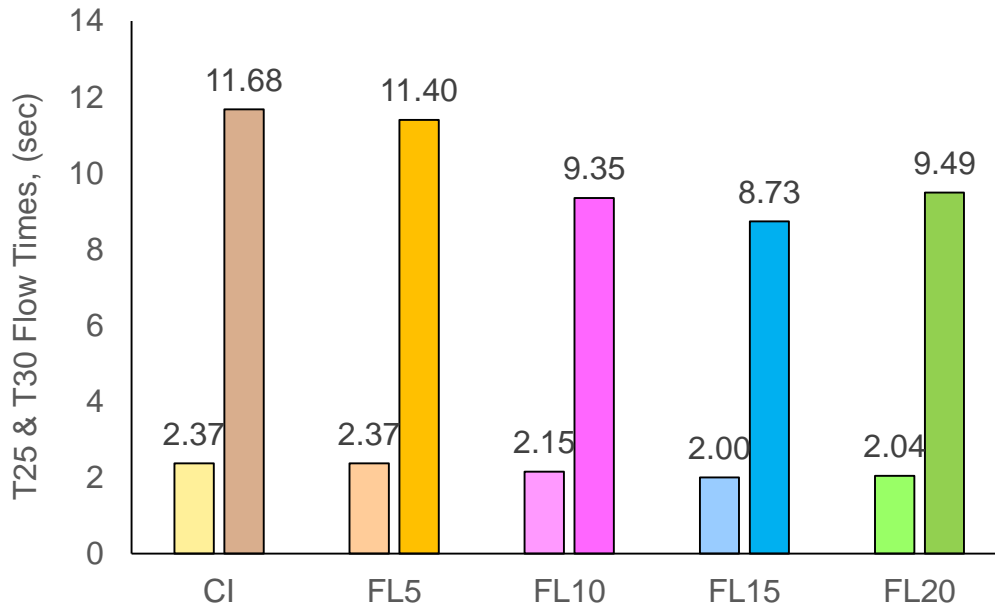


Figure 4.2.3 Flow Times of Mixes at WD & SPD

4.2.4 Setting Times:

Hydration is a “dissolution-precipitation Process”. During this process cement plus water transform from a fluid suspension to a porous solid over a matter of hours, a process called setting.

Thereafter “Hardening stage” appears during which strength develops over days and months.

Hydration of OPC and of SRM’s hydraulic reaction starts simultaneously but the reaction of most SCMs is slower than the reaction of the clinker phases.

SRMs effect the reactivity of each other. The increased setting times of blends can be explained by the retarding attributes of fly ash and the porous LSP. The delayed setting times allows continuous mass concreting and better bonding in poured concrete.

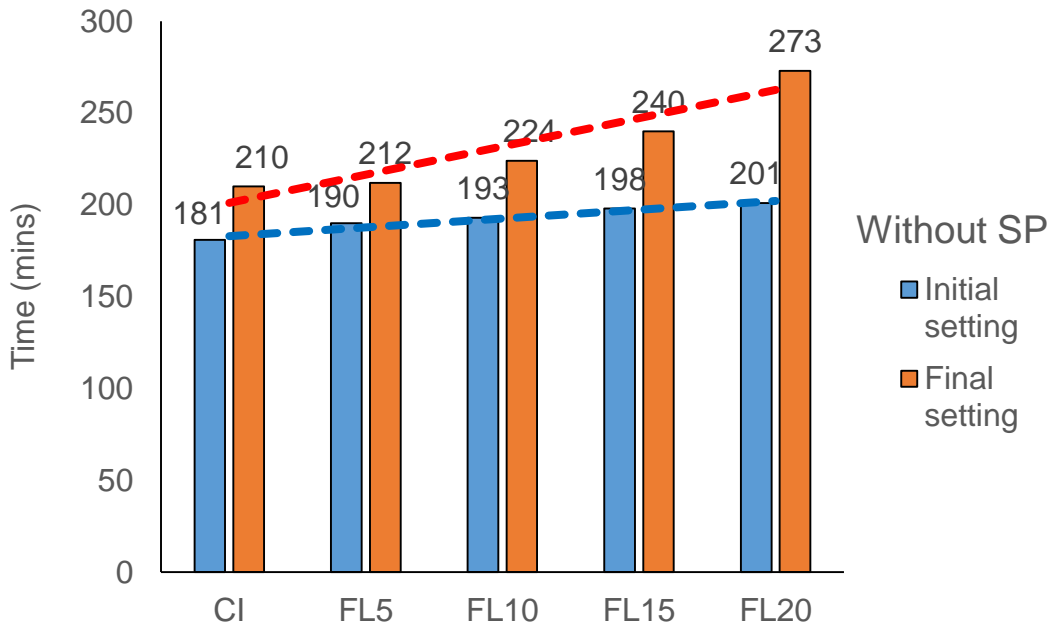


Figure 4.2.4 Initial and Final Setting Times of Mixes without SP

4.2.5 Flexural Strengths:

Flexure strength is a measure of tension taken by the concrete paste systems. While replacing the OPC content with blends of fly ash and limestone powder, the flexure strength tends to decrease for all 1,3,7 and 28 days. This because of the less flexural properties of SRMs.

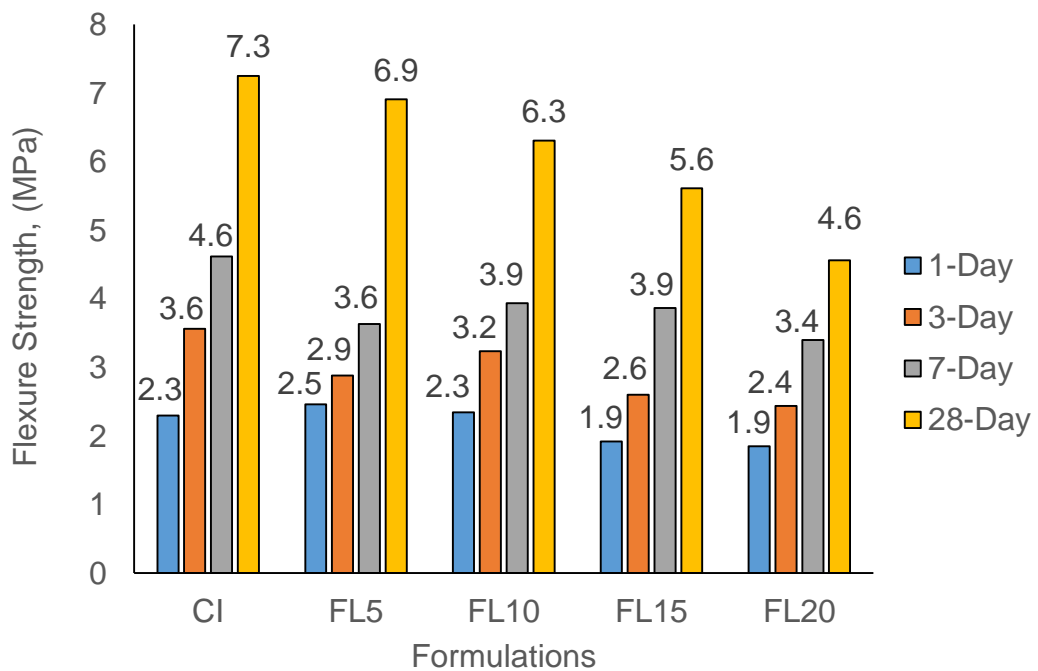


Figure 4.2.5 Flexural Strengths of Mixes at 1, 3, 7 and 28 Day

4.2.6 Compressive Strengths:

Margin of Compressive strength increase depends on the pozzolanic activity of SRM. Better packing decreases the porosity of hardened paste in the vicinity of the walls of aggregates. Improved and finer microstructure results in increased strength and offers Higher Resistance to fracture under loads.

Although Limestone particles can act as nucleation sites for reactions in the first stages of the hydration, however, at later time it acts only as inert filler and play no role in strength development of the hardened concrete at ambient or high temperatures. FA reacts between 3 and 7 days with CH to form crystals and enhances the strength. The optimum formulation is given by the highest strength which happens at 30% replacement of OPC by equal mass blend of FA and LSP.

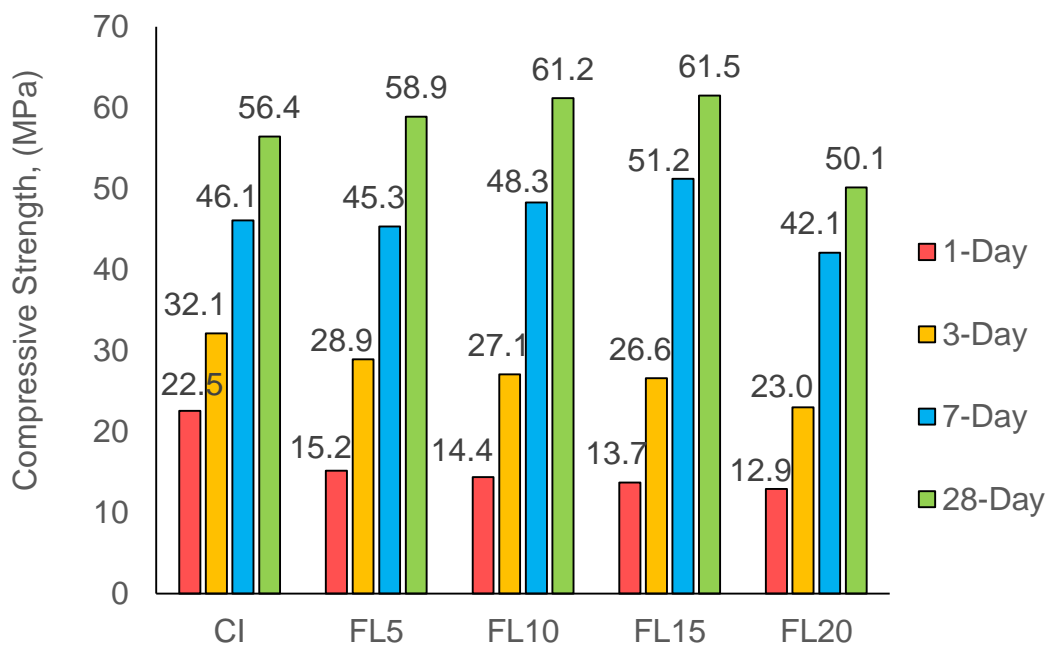


Figure 4.2.6 Compressive Strength of Mixes at 1, 3, 7 and 28 Day

4.2.7 Calorimetry:

When SRMs are added to cement, the hydration kinetics of Cement are modified depending upon the shape, size and morphology of the admixture. FA reduces early heat of hydration while LSP faster hydration due to nucleation. Blends have decreased the rate and total release of heat of hydration. Calorimetry peaks show that blends give lowest shrinkage and higher early volume stability

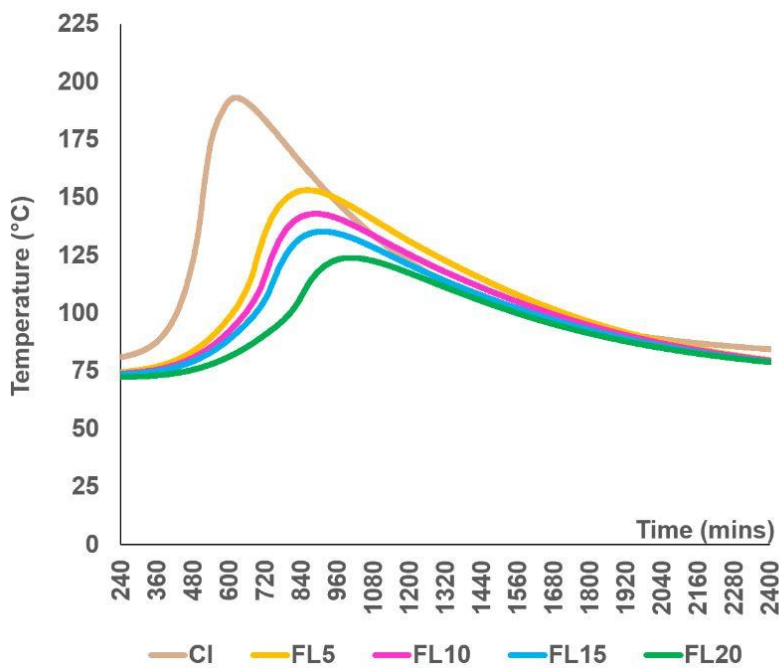


Figure 4.2.7 Calorimetry Curves for Mix Formulations

4.3 Self-Compacting Pastes with Variable Mixing Water Temperature

4.3.1 Water Demand

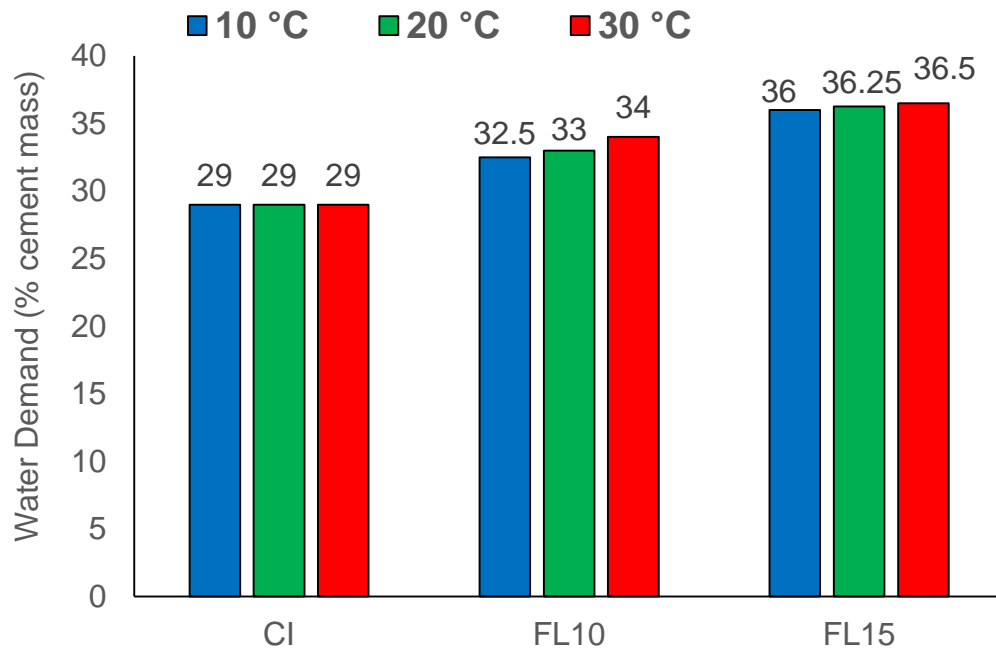


Figure 4.3.1 Water Demand of Mixes at Variable Mixing Water Temperature

It can be seen that the Water Demand of neat paste system is not affected by the varying water temperature. However, with 20 percent replacement the water demand increased for both higher and lower water temperatures. The linear response is displayed by the OPC replacement with 15% FA and 15% LSP.

4.3.2 Super-Plasticizer Demand

SP acts as dispersant for hydrated cement particles by modifying inter-particle potentials which results in higher fluidity. With the increase in mixing water temperature, it is observed that SP demand is decreasing. Surface of the particles gets lubricated, there is less viscosity and hence relatively less SP is required to achieve the same slump flow value

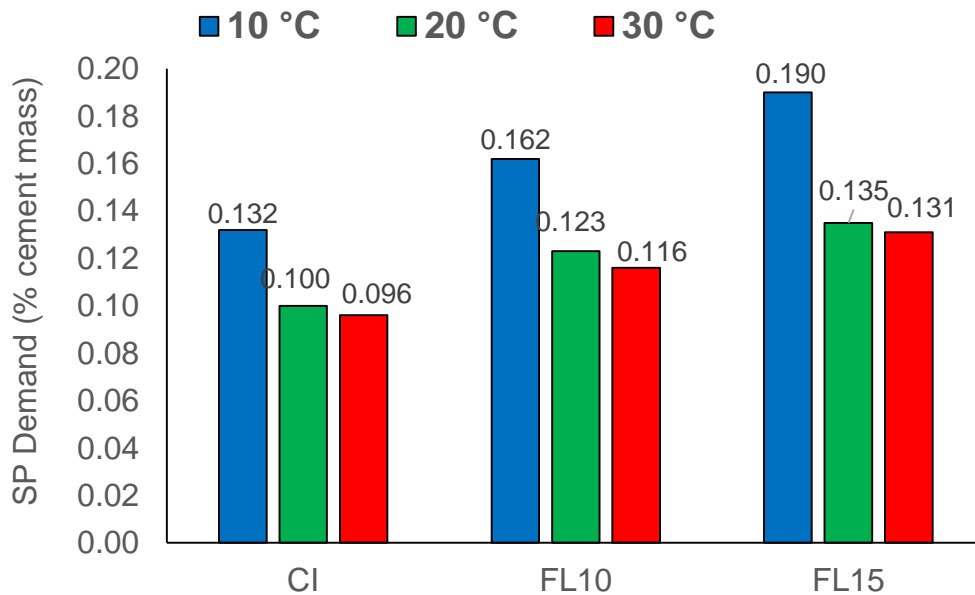


Figure 4.3.2 Superplasticizer Demands at Variable Mixing Water Temperatures

4.3.3 Flow Times

With the increase in mixing water temperature, it is observed that flow times is decreasing. Surface of the particles get lubricated, there is less viscosity and hence relatively less time is required to achieve the same slump flow value. It has the same trend as for SP demand.

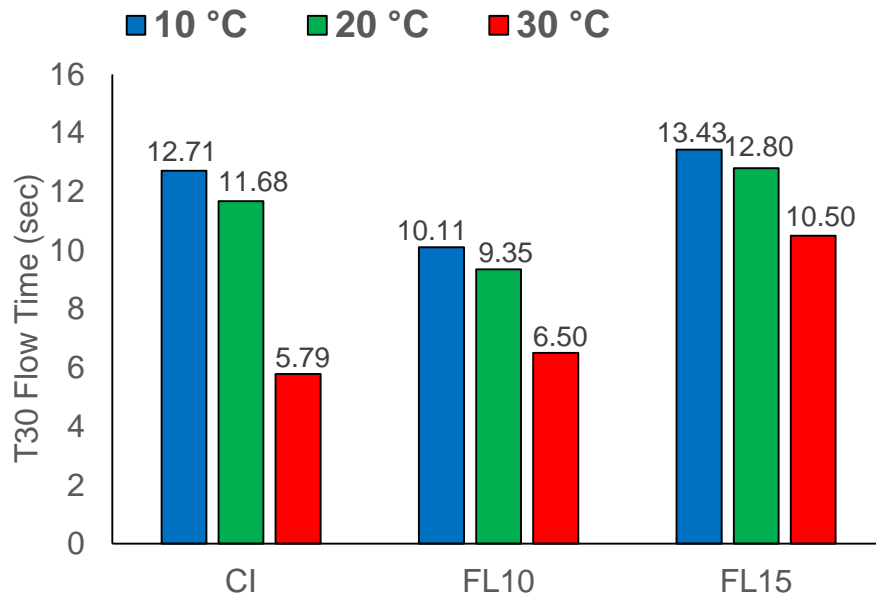


Figure 4.3.3 Time required for Target Spread at Variable Mixing Water Temperatures

4.3.4 Setting Times (without SP)

Due to fast hydration of cement at higher mixing water temperatures, skeleton formation takes place at an early age compared to the formulations with low mixing water temperature.

These results are further verified by the calorimetry study of mixes.

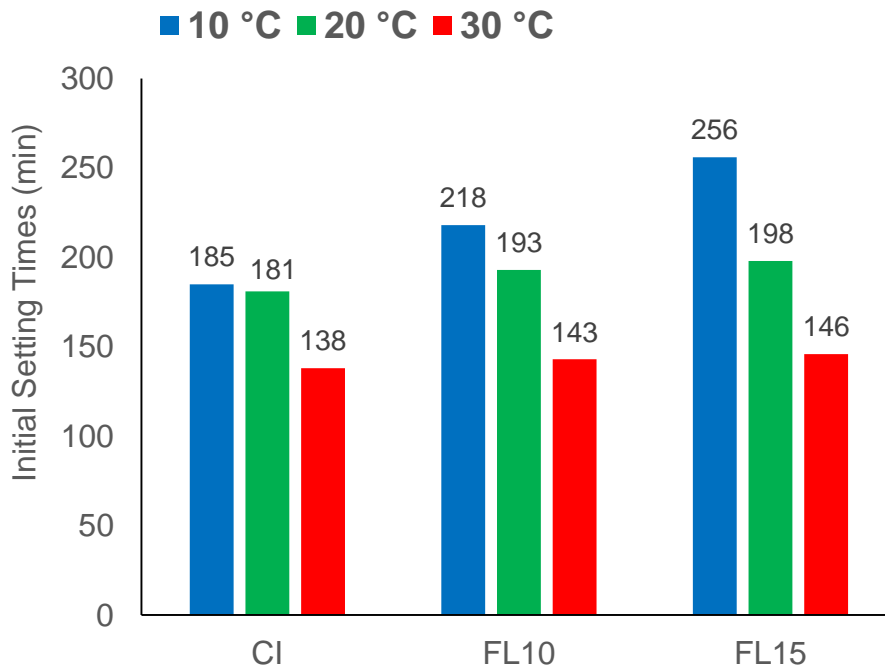


Figure 4.3.4 (a) Initial Setting Times of Mixes at Variable Mixing Water Temperatures

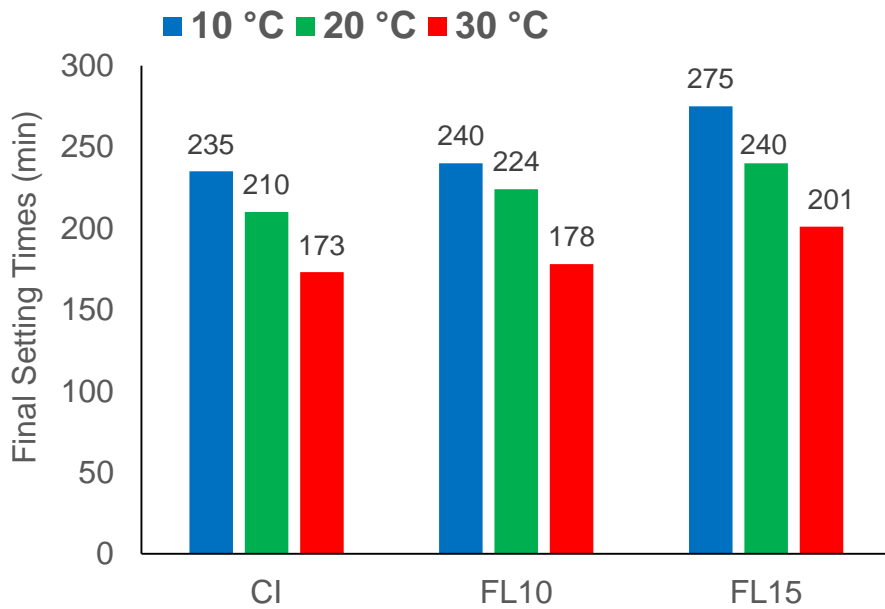


Figure 4.3.4 (b) Final Setting Times of Mixes at Variable Mixing Water Temperatures

4.3.5 Flexural Strengths

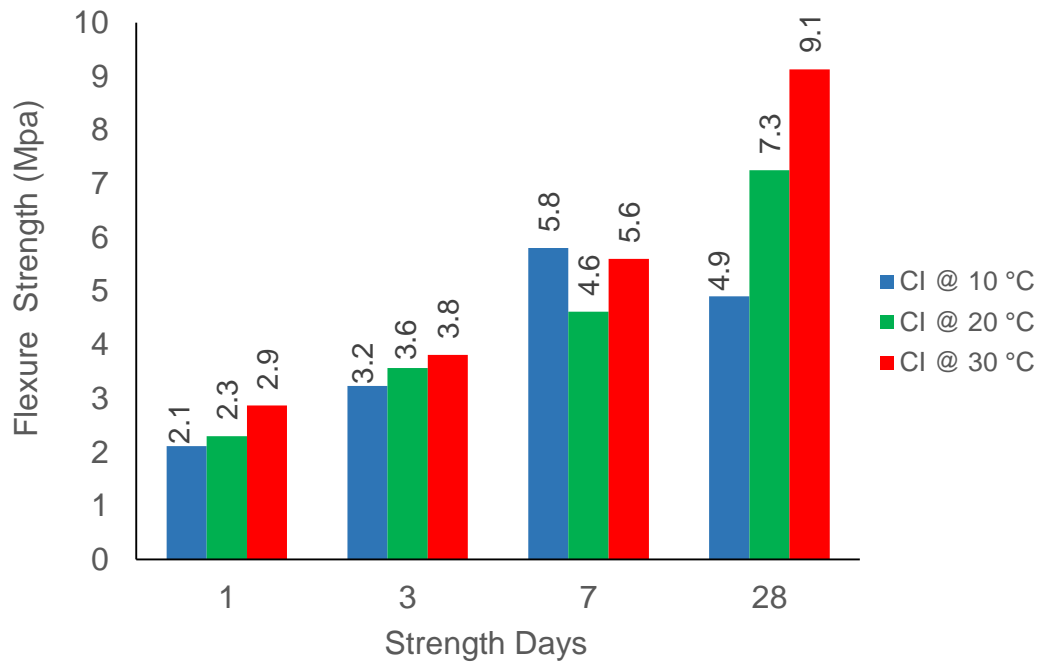


Figure 4.3.5 (a) Flexural Strength of CI at Variable Mixing Water Temperature

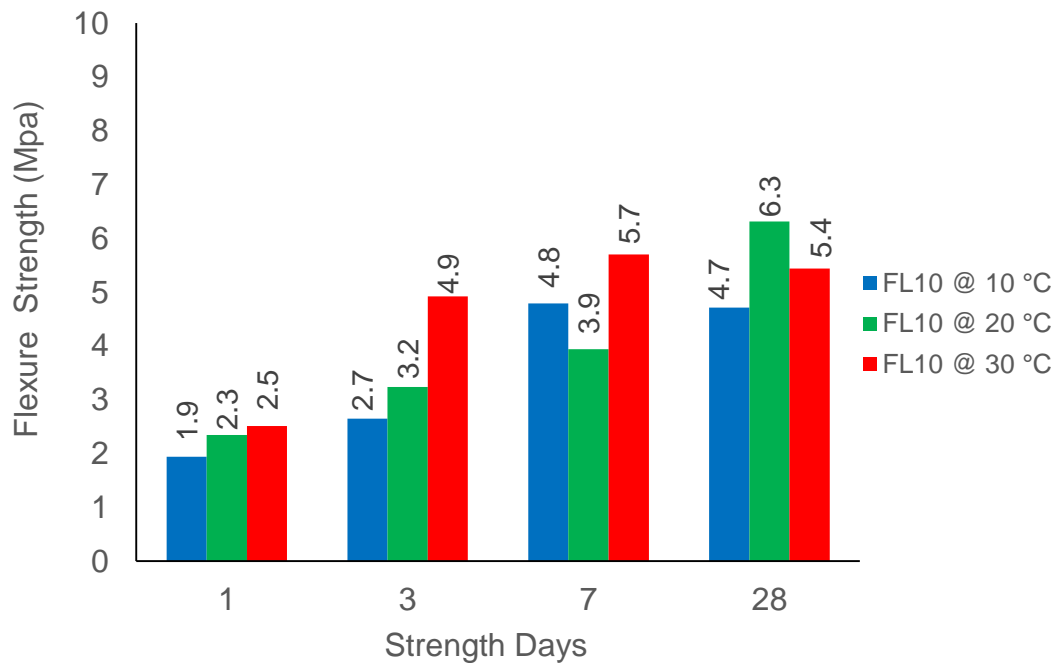


Figure 4.3.5 (b) Flexural Strength of FL-10 at Variable Mixing Water Temperature

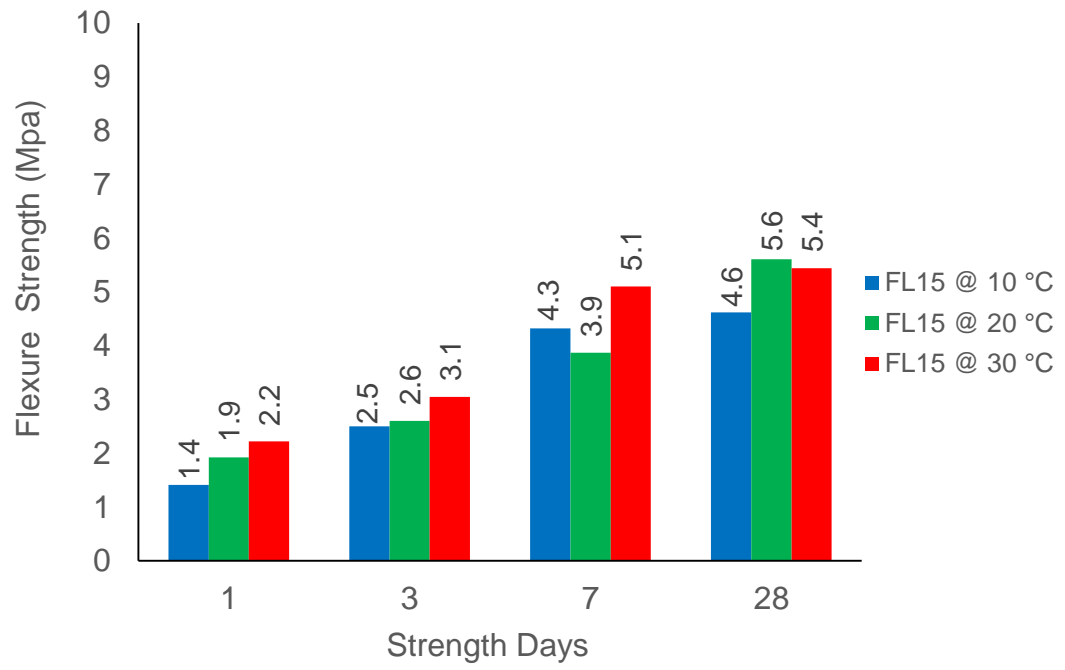


Figure 4.3.5 (c) Flexural Strength of FL-15 at Variable Mixing Water Temperature

4.3.6 Compression Strengths

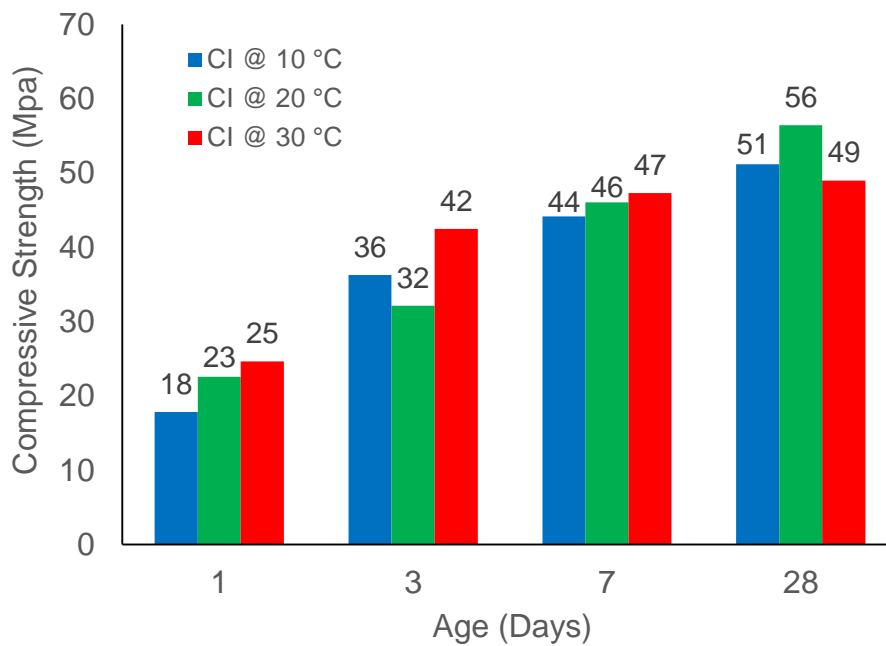


Figure 4.3.6 (a) Compressive Strength of CI-00 at Variable Mixing Water Temperature

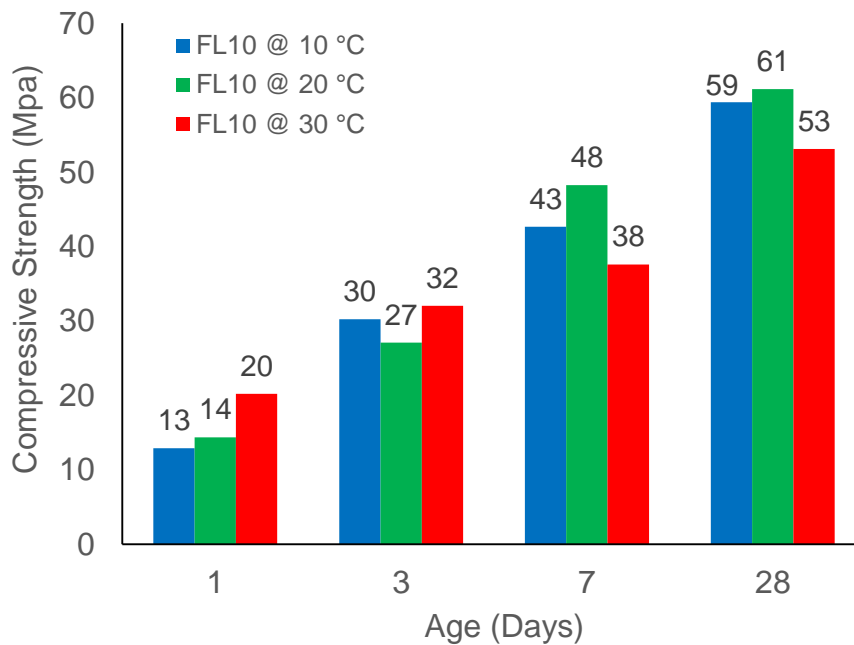


Figure 4.3.6 (b) Compressive Strength of FL-10 at Variable Mixing Water Temperature

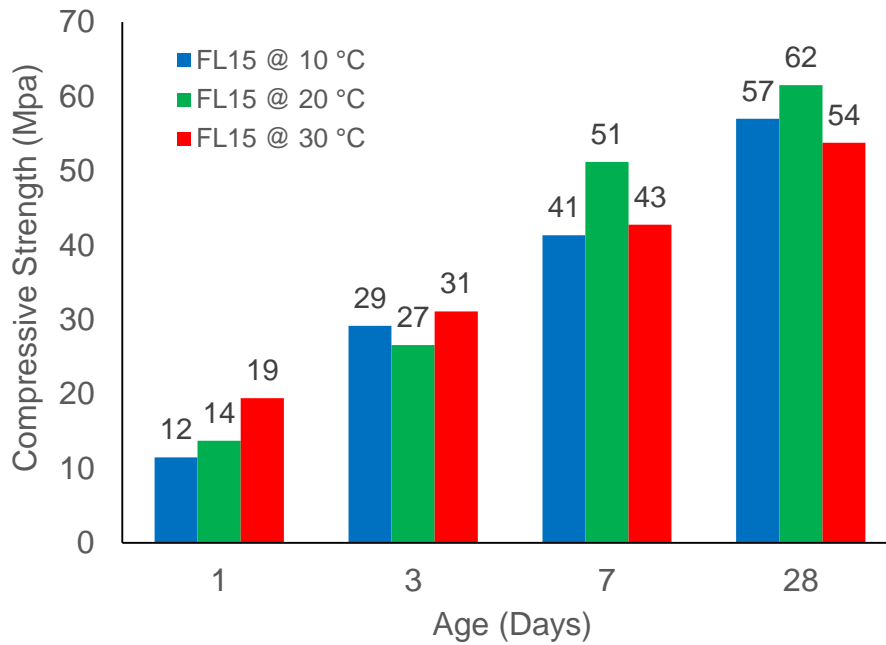


Figure 4.3.6 (c) Compressive Strength of FL-15 at Variable Mixing Water Temperature

Optimum results were obtained at temperature of 20°C. At lower mixing water temperature, the kinetic energy of water is low and hydration of SCP is slow. Also some of the reaction sites of the Powder particle may remain inactive due to poor interaction/collision between water and powder particles.

At higher temperature, the results are other way round. Due to high early hydration reaction, we have high early age strength of SCCS but at later ages reaction slows down. This may be associated to the poor skeleton formation at higher temperatures during the fast hydration process.

4.3.7 Calorimetry

Higher in-place concrete temperatures allow for faster rate of strength gain but later a slowdown in strength. The curves show that higher temperature mixing water for all mixes, the red lines, attain peaks much early and reach higher temperatures. The cooler water gives late peak. But with SRMs in the system, they enhance the hydration reaction for the 10 degree water temperatures. It proceeds slowly but release more energy for temperature rise. Strengths data for 3 day also show that 10 degree mixes have higher strength than 20 degree mixes.

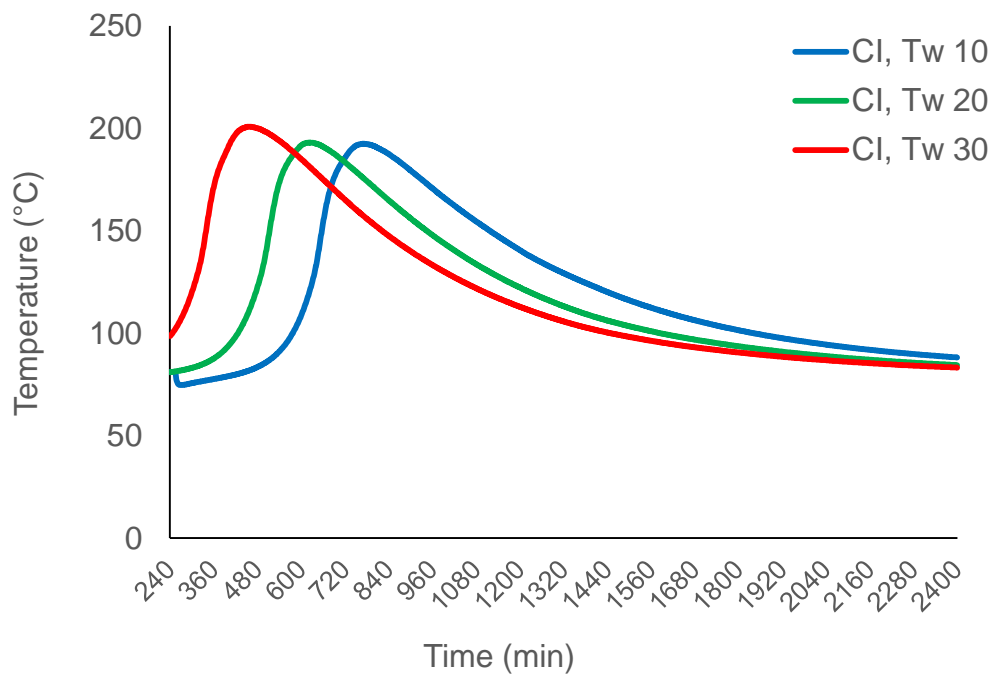


Figure 4.3.7 (a) Calorimetry Curves of CI-00 at Variable Mixing Water Temperature

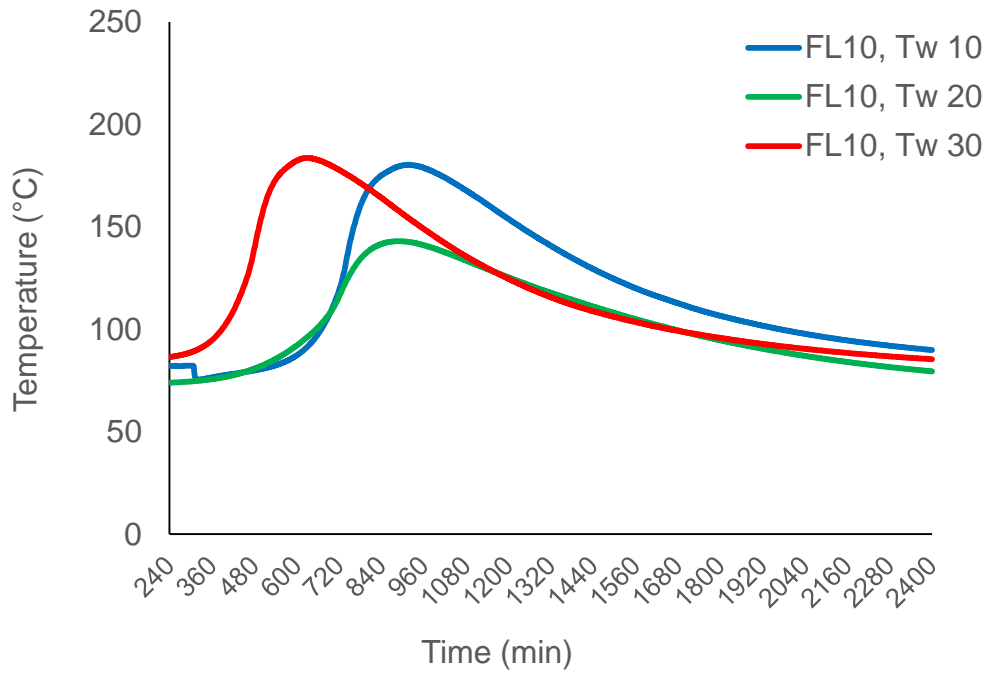


Figure 4.3.7 (b) Calorimetry Curves of FL-10 at Variable Mixing Water Temperature

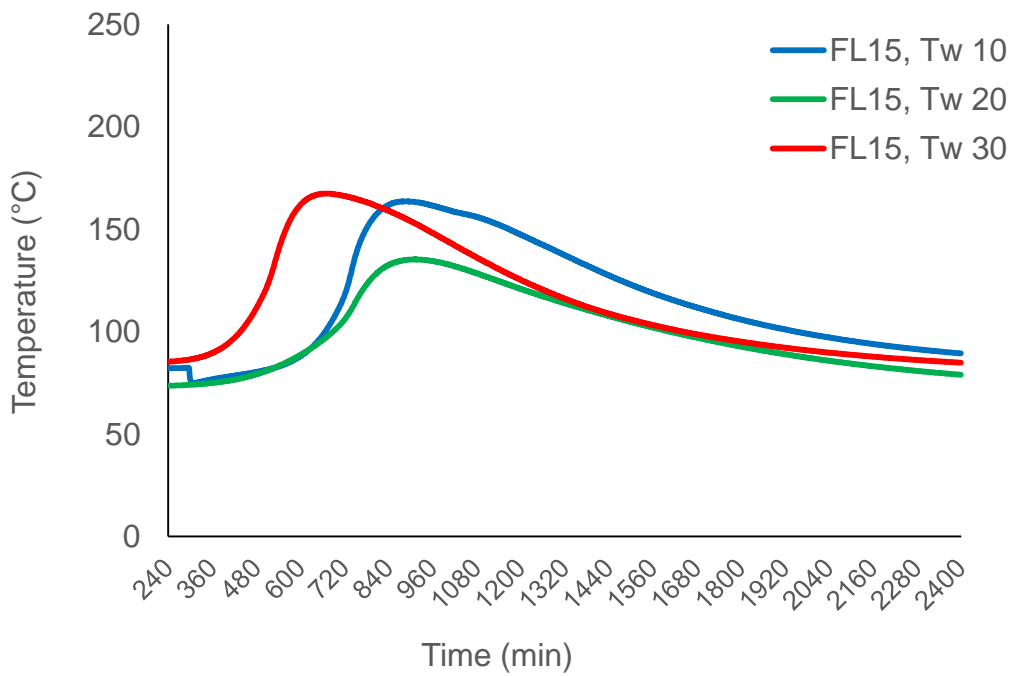


Figure 4.3.7 (c) Calorimetry Curves of FL-15 at Variable Mixing Water Temperature

Chapter – 5

DISCUSSION

This section provides the discussion on the results of testing on Self-Compacting Paste Systems in which Cement is partially replaced with four blends of Fly Ash and Limestone Powder at three different mixing water temperatures.

SRMs are known to have physical and chemical influence on the hydration reactions namely the Dilution effect, role as Filler, acting as nucleation sites, pozzolanic activity and providing calcium hydroxide. Dilution of powders occurs and water is adsorbed by the SRMs, reducing the free water in the system, and minimizing the bleeding. SRMs act as filler due to their finer size and fill the air pores with solid powders, enhancing the density and durability of system. SRMs also act as nucleation site in which the crystals precipitate from the hydration reaction on the fine powders. They provide $\text{Ca}(\text{OH})_2$ for the hydration reaction and increases the growth of Calcium Silicate Hydrate crystals which is the primary product of OPC hydration. The shape, size, morphology and surface characteristics of SRMs determine their role in the system.

The above stated SRMs' functions are discussed w.r.t results of water demand, superplasticizer demand, setting times, calorimetry and strength.

5.1 Water Demand of Systems

For the mixing water temperature at room temperature, the four OPC-replacement levels are studied which show an increase of w/c ratio in 10, 20, 30 and 40% respectively compared to the neat paste system i.e. pure OPC system. This is due to the finer size of FA and LSP, increasing the surface area of the powders. More water is required to lubricate and flow the paste system. On the other hand, w/p decreases; this happens because system has now less pores and voids available. The quantity of cement is reduced in the system and so is the hydration water requirement.

On varying the temperature of mixing water, the neat paste has no effect on the water demand. While for 20% replacement of cement i.e. FL10 mix, w/c is highest for 30°C. At cooler temperatures, less water is required for hydration and flow; there are less water losses at higher temperatures, the water. For FL15 mix which has 30% of OPC

replaced by blend, the response is linear. W/c increases as the mixing water temperature is increased.

5.2 Superplasticizer Demand

Addition of fines increases the internal friction and yield stress. To achieve the target of (30 ± 1) cm, the SPD increases in high replacement mixes. The results show a trend of high SP requirement as the quantity of fly ash and limestone powder increases because the deformability decreases as the microstructure densifies and has resistance to flow and spread.

Considering different mixing water temperatures, at cooler temperatures, there is a remarkable increase in the SPD. This occurs due to the increased surface friction and low water energy to cause flow. While the SPD also increases in all mixes for the 30°C mixing water, the rise is not prominent. It may be due to higher absorption on the surface of SRMs.

This increased SP content in mixes at variable mixing water temperature affects the flow times. As can be seen the time for 30 cm target spread is highest for 20°C,

5.3 Setting Times

Setting times are determined for 20°C first without addition of SP and then with SP. Without SP, initial setting followed a general trend of taking lesser time in higher replacement mixes. However, final setting times increased upto FL15 mix; afterwards FL20 has nearly taken the same time to final set as that with no SRM. With SP, retardation is prominent as it takes considerable more time to set than the CI mix. Initial set in this shows a decline in the trend at 40% replacement.

On varying the temperature of water, the CI mix has initial setting highest at 20°C than the 10 and 30 °C. Rest all give a pattern of increasing setting times, both initial and final setting, when there is higher temperature water added.

5.4 Calorimetry

Addition of SRMs cause the lower energy release. As the replacement levels are increased, the temperature peaks fall and occurs late as well. This is because of the less hydration occurring in the system due to reduced cement content.

With variation in temperature, it is seen that higher the temperature of any mix, the peaks are higher and occurs earlier. But one anomaly can be seen that for FL10 and FL15, the peak for 20°C is lower than the 10°C.

5.5 Compressive Strengths

The compressive strength enhancement is the prime objective. With replacement of cement with blends at 20°C, the strengths increase upto the 30% blend in the system, yielding highest strength of 61.5 MPa in comparison to CI having 56.4 MPa. The rate of strength development is though slow, especially the one day strength is decreasing with increasing SRMs. FL10 also gave a considerable high strength of 61.2 MPa. SRMs fill the pores, give better packing, aids in hydration and gives a denser system; this minimizes the free spaces and resists crack penetration during the load application.

The formulations undertaken for variation in water mixing are the control mix CI and the highest strength yielding mixes i.e. FL10 and FL15. Temperature inputs the energy in the system, it affects the rate of development of the system. Eventually, it is 20°C mixes that attain the maximum strengths.

Chapter - 6

CONCLUSIONS

One of the Measures of Effectiveness (MOE) is the strength-to-cement ratio. From the results of 28 day compressive strength, the strength-to-cement ratio at 20°C are:

Table 6.1 Strength-to-Cement Ratios

Mix	% OPC	Compressive Strength (MPa)	Strength-to-cement ratio
CI	100	56.43	56.43
FL5	90	58.88	65.42
FL10	80	61.17	76.46
FL15	70	61.50	87.86
FL20	60	50.14	83.57

The above table shows the effectiveness achieved in economy and sustainability. Cement being most expensive item in production of concrete, when partially replaced with FA and LSP yields the comparable strength results. At the same time, it minimizes environmental degradation of processing of virgin materials in production of cement, energy utilization and carbon emissions. Also, the waste management is incorporated as less dumping sites will be required.

Results also show that the SRMs in blend give a complementary response for the setting times and rate of strength development.

Calorimetry is a very useful technique to study hydration dynamics. It shows the dormant periods and acceleration periods. From the results, it can be inferred that the high temperature water cause faster reaction but gives lower strengths because of exothermic nature of hydration reaction.

More SP is needed to overcome internal friction and viscosity when SRM content is increased. Retardation also occurs as the SRM proportion in the mix is increased.

Highest strength results from 30% replacement of OPC and 20°C water temperature. Afterwards the strength response starts to decline but it can still be effectively used to attain economy, sustainability and environment protection.

ANNEXURE - A:

Physical and Chemical Characteristics of Materials

Table A-1 Physical Properties of Cementitious Materials

Oxides	OPC	Fly Ash	Limestone Powder
Physical State	Powder	Powder	Powder
Particle Size (D50)	17.82 μm	7.20 μm	7.70 μm
Colour	Dark grey	Light grey	Light grey

Table A-2 Chemical Composition of Cementitious Materials

Oxides	OPC	Fly Ash	Limestone Powder
Al ₂ O ₃	ND	ND	ND
SiO ₂	49.81	62.14	22.09
CaO	43.33	10.26	74.21
Fe ₂ O ₃	5.61	22.44	2.23
SrO	0.25	0.97	1.33
TiO ₂	0.35	1.73	-
ZnO	0.44	0.17	-
MnO	0.07	0.20	-
Cr ₂ O ₃	0.07	-	-
V ₂ O ₅	0.06	0.02	-
K ₂ O	-	1.75	-
ZrO ₂	-	0.21	-
Rb ₂ O ₃	-	0.06	-
Ga ₂ O ₃	-	0.05	-

ANNEXURE - B:

Self-Compacting Paste Systems with Mixing Water at Room Temperature

Table B-1 Blend Formulations

Formulations	% OPC	% Fly Ash	% Limestone Powder
CI-00	100	0	0
CI-FA5-LSP5	90	5	5
CI-FA10-LSP10	80	10	10
CI-FA15-LSP15	70	15	15
CI-FA20-LSP20	60	20	20

Table B-2 Water Demands

Formulations	Mixing Water Temperature (°C)	Water Demand (% cement mass)
CI-00	20.6	29.00
CI-FA5-LSP5	20.4	30.00
CI-FA10-LSP10	20.6	33.00
CI-FA15-LSP15	21.0	36.25
CI-FA20-LSP20	21.0	41.50

Table B-3 Super Plasticizer Demands

Formulations	Mixing Water Temperature (°C)	SP Demand (% cement mass)
CI-00	20.70	0.100
CI-FA5-LSP5	19.80	0.115
CI-FA10-LSP10	19.90	0.123
CI-FA15-LSP15	19.00	0.135
CI-FA20-LSP20	19.30	0.155

Table B-4 Flow and Flow Times

Formulations	Flow (cm)	T-25 (sec)	T-30 (sec)
CI-00	29.65	2.37	11.68
CI-FA5-LSP5	29.85	2.37	11.40
CI-FA10-LSP10	30.05	2.15	9.35
CI-FA15-LSP15	29.05	2.00	12.80
CI-FA20-LSP20	30.00	2.04	10.30

Table B-5 Setting Times without SP

Formulations	Initial Setting Time (min)	Final Setting Time (min)
CI-00	181	210
CI-FA5-LSP5	190	212
CI-FA10-LSP10	193	224
CI-FA15-LSP15	198	240
CI-FA20-LSP20	201	273

Table B-6 Flexural Strengths

Formulations	1-Day (MPa)	3-Day (MPa)	7-Day (MPa)	28-Day (MPa)
CI-00	2.30	3.56	4.62	7.48
CI-FA5-LSP5	2.46	2.88	3.63	7.21
CI-FA10-LSP10	2.34	3.23	3.94	7.04
CI-FA15-LSP15	1.92	2.91	3.87	6.82
CI-FA20-LSP20	1.85	2.44	3.40	5.40

Table B-7 Compressive Strengths

Formulations	1-Day (MPa)	3-Day (MPa)	7-Day (MPa)	28-Day (MPa)
CI-00	22.55	32.14	46.07	56.43
CI-FA5-LSP5	15.17	43.37	48.27	58.88
CI-FA10-LSP10	14.37	27.07	57.00	61.17
CI-FA15-LSP15	13.72	31.97	45.32	61.50
CI-FA20-LSP20	12.93	26.18	30.33	50.14

ANNEXURE - C:

Self-Compacting Paste Systems with Variable Mixing Water Temperature

Table C-1 Temperature Variation Blend Formulations

Formulations	% OPC	% Fly Ash	% Limestone Powder
CI-00	100	0	0
CI-FA10-LSP10	80	10	10
CI-FA15-LSP15	70	15	15

Table C-2 Water Demands

Formulations	T_w = (10 ± 1) °C Water Demand	T_w = (20 ± 1) °C Water Demand	T_w = (30 ± 1) °C Water Demand
CI-00	29	29	29
CI-FA10-LSP10	33.50	33	34
CI-FA15-LSP15	36	36.25	36.50

Table C-3 Super Plasticizer Demands

Formulations	T_w = (10 ± 1) °C SP Demand	T_w = (20 ± 1) °C SP Demand	T_w = (30 ± 1) °C SP Demand
CI-00	0.132	0.100	0.105
CI-FA10-LSP10	0.162	0.123	0.126
CI-FA15-LSP15	0.190	0.135	0.146

Table C-4 Flow Times:

Formulations	T_w = (10 ± 1) °C		T_w = (20 ± 1) °C		T_w = (30 ± 1) °C	
	T25 (sec)	T30 (sec)	T25 (sec)	T30 (sec)	T25 (sec)	T30 (sec)
CI-00	2.45	7.68	2.37	11.68	3.59	5.79
CI-FA10-LSP10	1.37	8.79	2.15	9.35	2.25	6.50
CI-FA15-LSP15	1.93	9.16	2.00	12.80	2.50	10.50

Table C-5 Setting Times without SP:

Formulations	T_w = (10 ± 1) °C		T_w = (20 ± 1) °C		T_w = (30 ± 1) °C	
	IST (min)	FST (min)	IST (min)	FST (min)	IST (min)	FST (min)
CI-00	143	173	193	210	185	235
CI-FA10-LSP10	138	178	164	224	218	240
CI-FA15-LSP15	146	201	157	273	256	275

Table C-6 Flexural Strengths for $T_w = (10 \pm 1) ^\circ\text{C}$:

Formulations	1-Day (MPa)	3-Day (MPa)	7-Day (MPa)	28-Day (MPa)
CI-00	2.11	3.23	6.19	7.25
CI-FA10-LSP10	1.41	2.65	4.79	6.31
CI-FA15-LSP15	2.11	2.60	5.39	5.61

Table C-7 Compressive Strengths for $T_w = (10 \pm 1) ^\circ\text{C}$:

Formulations	1-Day (MPa)	3-Day (MPa)	7-Day (MPa)	28-Day (MPa)
CI-00	32.00	36.28	50.70	51.20
CI-FA10-LSP10	21.70	30.23	55.60	59.40
CI-FA15-LSP15	20.85	29.18	41.35	57.00

Table C-8 Flexural Strengths for $T_w = (30 \pm 1) ^\circ\text{C}$:

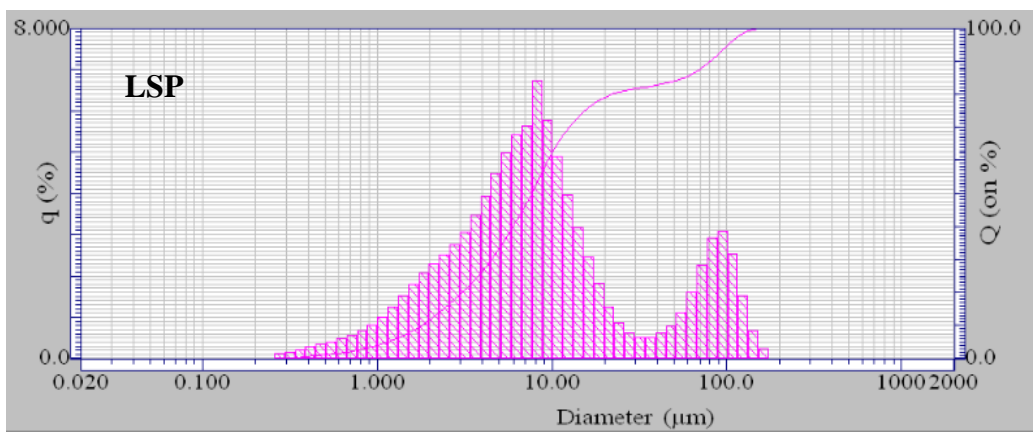
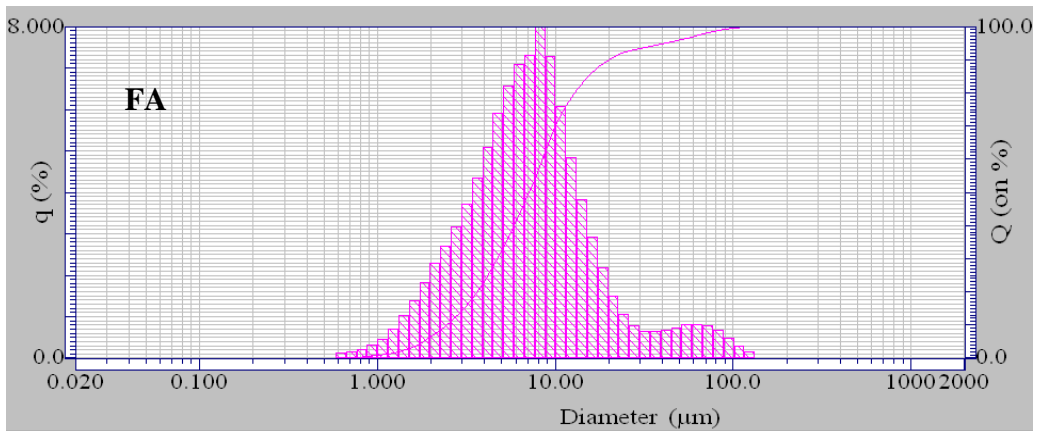
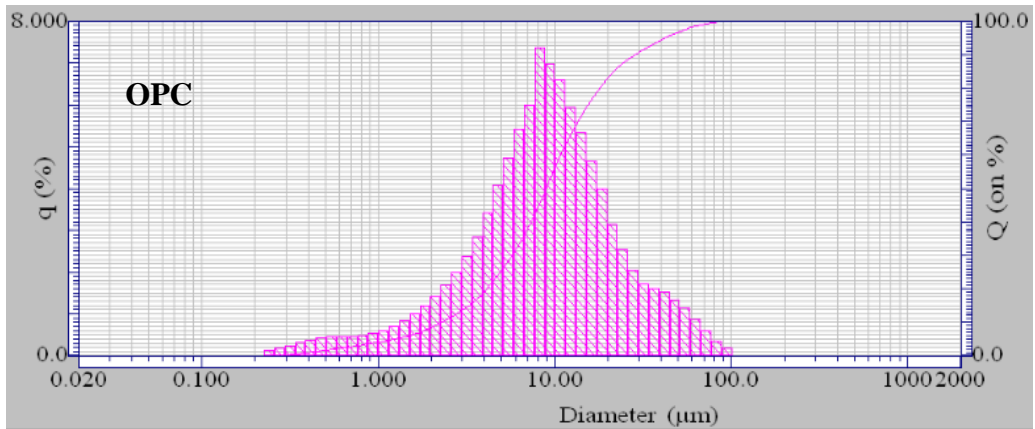
Formulations	1-Day (MPa)	3-Day (MPa)	7-Day (MPa)	28-Day (MPa)
CI-00	2.87	3.81	5.60	9.13
CI-FA10-LSP10	2.51	4.92	5.70	5.44
CI-FA15-LSP15	2.22	3.05	5.10	5.44

Table C-9 Compressive Strengths for $T_w = (30 \pm 1) ^\circ\text{C}$

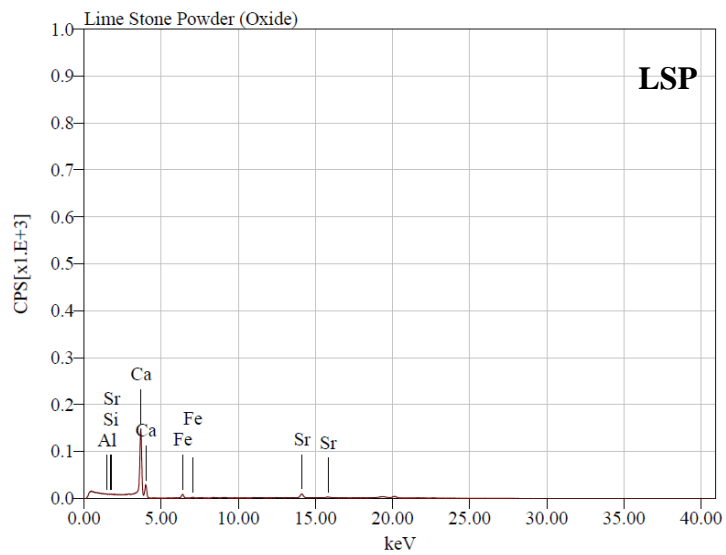
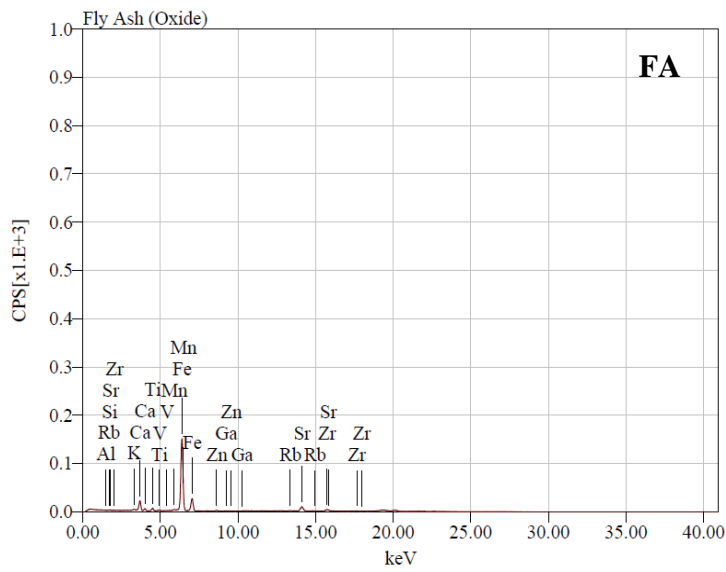
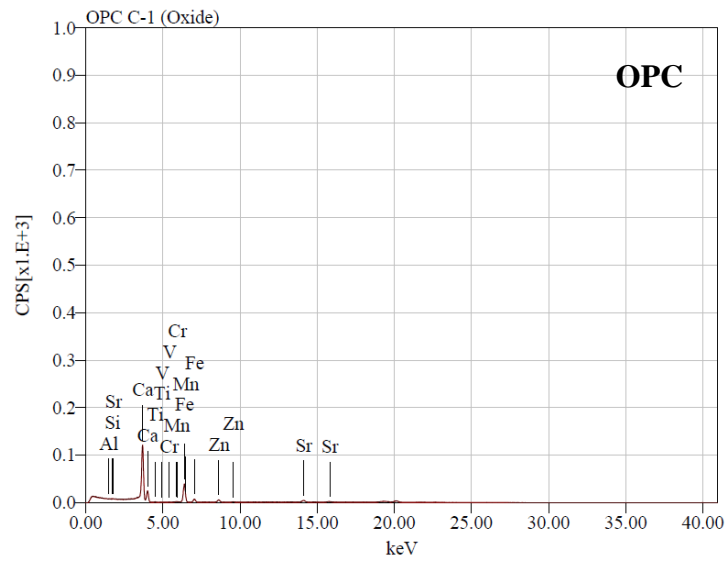
Formulations	1-Day (MPa)	3-Day (MPa)	7-Day (MPa)	28-Day (MPa)
CI-00	24.65	42.48	47.70	49.00
CI-FA10-LSP10	20.22	32.03	37.60	53.10
CI-FA15-LSP15	19.45	31.10	42.75	53.75

ANNEXURE – D

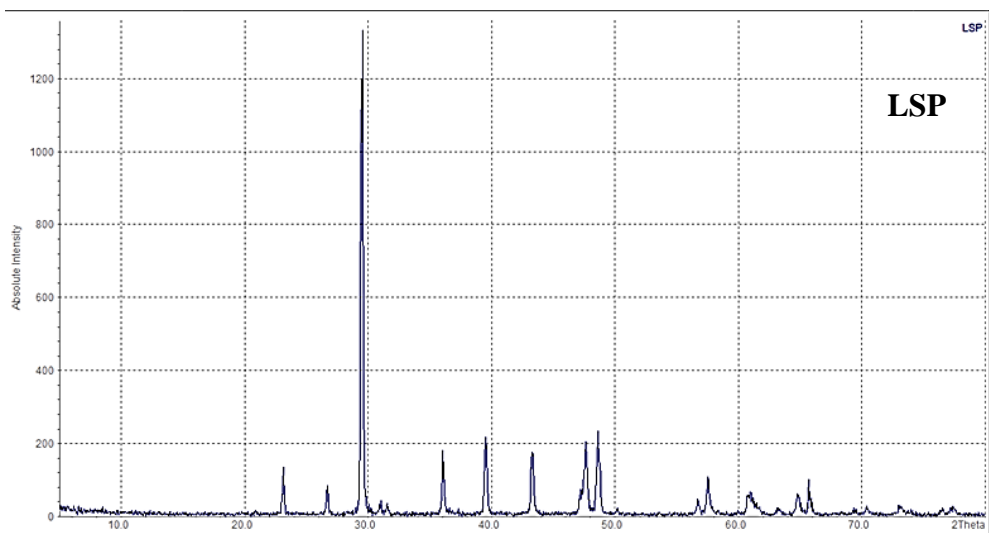
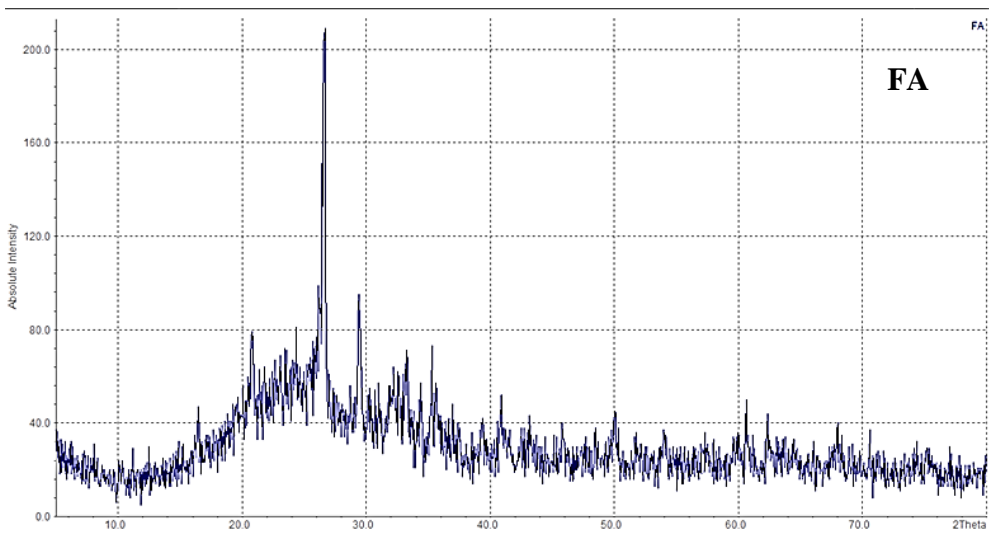
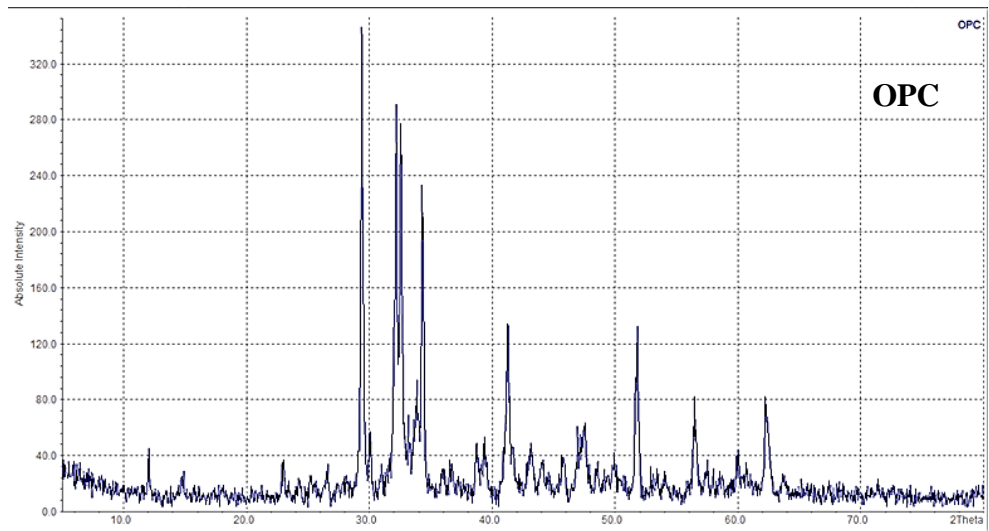
PARTICLE SIZE ANALYSIS:



X-RAY FLUORESCENCE (XRF):



X-RAY DIFFRACTION (XRD):



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