STUDY OF SELF LEVELLING PASTE SYSTEMS USING BLENDS OF CALCIUM ALUMINATE CEMENT AND PORTLAND CEMENT AT VARIABLE MIXING WATER TEMPERATURE

FINAL YEAR PROJECT UG 2011

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Final Year Project Titled

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for the undergraduate degree

in

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ABSTRACT

Rapid Hardening Self levelling Cementitious Systems are known for their applications in emergency repair work on runways, bridges and tunnel linings. They also provide excellent solutions for Self levelling underlayments. The aim of our research was to investigate effect of mixing water temperature on multi-binder systems. CAC was used to help accelerate the setting times of OPC. However, the drawback of using CAC is its strength degradation over time, a process known as Conversion. Our objective was to study this Conversion process under variable mixing water temperature. The water temperatures tested were **5°C, 23°C and 35°C**. Binary formulations were studied with varying percentage of CAC as a replacement to OPC. A ternary blend of CAC-OPC-Fly Ash was also studied under variable water temperature to study the behavior of Fly-Ash in binary Self Levelling Paste (SLP) Systems.

Tests conducted for SLP formulations were water demand, setting times, compressive strengths, calorimetry and shrinkage. Charts were plotted against each test and the effect of mixing water temperature was observed. Other tests performed were BET surface area, Particle Size Analysis (PSD), XRD and XRF. It was found that:

Binary SLPS of CAC-OPC cause faster setting in comparison with their parent formulations (CAC, OPC). This leads to higher water demands and plasticizer dosage. However, it was seen that for Cold Mixing Water, the water demands and SP demands decreased with increasing CAC replacement in OPC. At higher mixing water temperature, the trend reverses: Water demands and SP demands increase with increasing CAC replacement in OPC. Setting Times increased with increasing CAC replacement at cold mixing water temperatures and decreased at elevated temperatures. The replacement of OPC with Fly Ash retarded setting times at Cold water temperatures but this effect was not observed at elevated temperature. Compressive strengths were also plotted for 1 day, 7 day and 28 days of water curing against varying mixing water temperatures. Contrary to what literature suggested, the final 28 day strength of binary formulations was higher than that of 100% OPC and 100%CAC. Also, the conversion phenomenon was not observed at colder mixing water temperature but at elevated temperature (35◦C), 28 days compressive strength of CAC decreased compared to 7 day strength. Calorimetry was also performed for SLP formulations and temperature graphs were plotted for 1 day Hydration. It was seen that CAC gave the highest temperature peak followed by OPC, and their binary blends. XRD tests confirmed presence of di-calcium Aluminum Hydrates in pure CAC samples hydrated at 56 days which proved that conversion phenomenon requires higher mixing water temperatures above 35°C as well as more curing time. Presence of fast setting Ettringite was also confirmed in binary CAC-OPC blends

In short it can be concluded that elevated mixing water temperature greatly reduces setting times for binary blends of CAC-OPC but increases water demand and SP demand and reduces workability. Also strength is greatly reduced at higher temperatures owing to Conversion phenomenon as well as increased water demand which creates more voids.

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CHAPTER 1

INTRODUCTION

1.1 General:

Concrete is a major element used in the construction industry due to its high compressive strength and durability. However, working with concrete in its fresh state is difficult and requires a large labor force to properly level it and achieve a uniform degree of compaction. A more modern technology in the field of concrete materials is the Self-levelling and Compacting Systems. Self-compacting concrete (SCC) as defined by ACI 237R – 07, is a highly flow able, non-segregating concrete that can spread into place, fill the formwork, and encapsulate the reinforcement without any mechanical consolidation **[1]**.

Self levelling concrete is a kind of high performance concrete that is fluid and able to consolidate under its own weight. It is stronger and more durable and is easy to place in congested reinforcement. It has high degree of deformability and resistance to segregation.

Self levelling and compacting systems contain high powder content with low w/p ratio than conventional concrete. As a result these systems provide a better overall response in terms of packing density, heat of hydration, volume stability, ease of placement, higher durability and strength development.

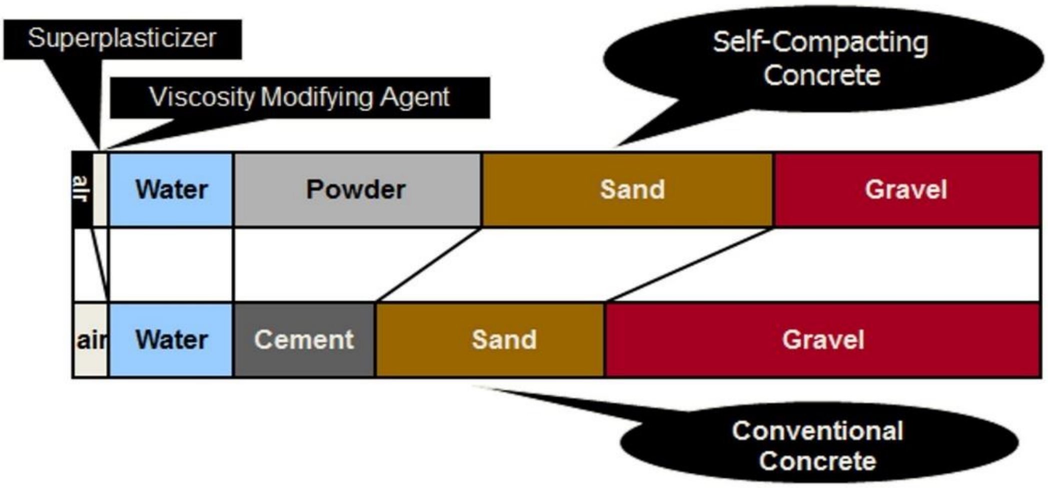

Figure 1.1: Comparison of Conventional and Self-Compacting Concrete

Self levelling and compacting systems are now being used extensively throughout the world. Its applications include repairs or massive placements in heavily congested and reinforced sections like bridge piers, tunnel linings, rafts, pile foundations, aircraft runways, heavily reinforced columns and pre-stressed concrete members. This technology provides us with a uniform degree of compaction throughout the concrete placement and results in a uniform durability of the structure.

In Pakistan, the application of Self levelling paste systems is very limited due to unavailability of chemical and mineral admixtures, costly prices of cement and a lack of understanding of the basic concepts of self-compacting concrete and an over-dependence on outdated construction techniques in our construction industry.

1.2 Historical Development:

Humans have been using concrete for around 7000 years and the modern Portland cement was introduced around 200 years ago. Fluid concrete is a much recent invention. Ancient experimenters tried thinning concrete with organic materials like milk, blood and lard with little success. It wasn't until the late 1950s and early 1960s that engineers in Germany and Japan first developed super plasticizers and was first made available in the United States in the mid-1970s.

The necessity of self levelling paste systems was felt first in Japan due to their inadequate labor force. The necessity of the type of concrete which can be compacted purely by means of its own weight and without the need for vibrating compaction was proposed by Okamura in 1986. Studies to develop self-compacting concrete, including a fundamental study on the workability of concrete, were carried out by Ozawa and Maekawa at the University of Tokyo. **[2]**

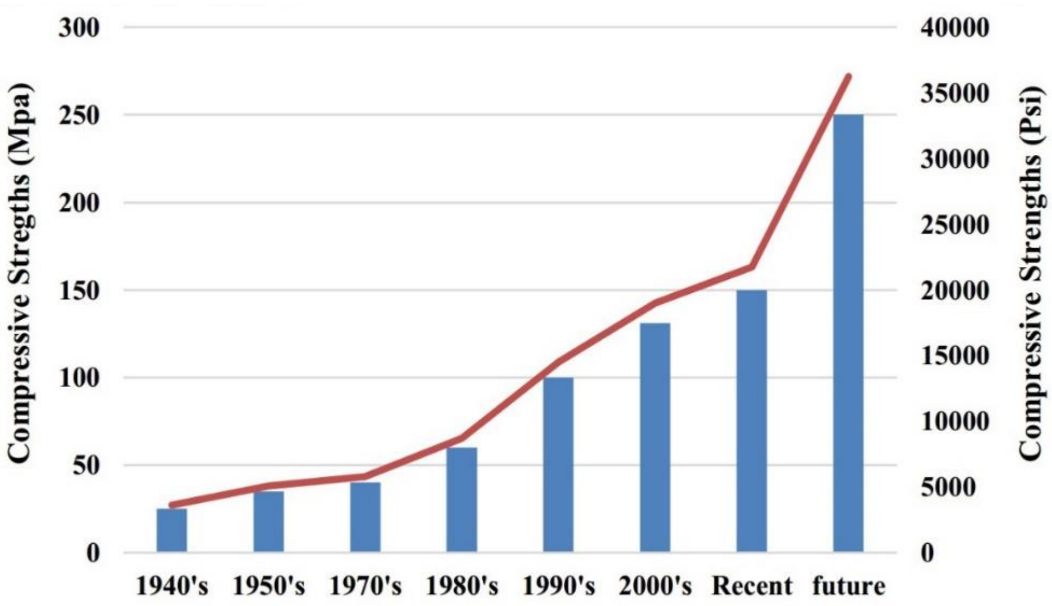

Figure 1.2: Development of Compressive Strengths of Concrete over time

Today its use is widespread. Contractors love the fact that it pumps easily, fills in narrow spaces, and requires little or no compaction to remove air bubbles, all while retaining its high strength.

The prototype of self-compacting concrete was first completed in 1988 using materials already on the market. The prototype performed satisfactorily with regard to drying and hardening shrinkage, heat of hydration, denseness after hardening, and other properties.

Self-compacting concrete was used for the wall of a large LNG tank belonging to the Osaka Gas Company, whose concrete casting was completed in June 1998. The volume of the self-compacting concrete used in the tank amounted to 12,000 m^3. The adoption of self-compacting concrete means that:

- The number of lots decreases from 14 to 10, as the height of one lot of concrete casting was increased.
- The number of concrete workers was reduced from 150 to 50.
- The construction period of the structure decreased from 22 months to18 months.

Self-compacting concrete is often employed in concrete products to eliminate the noise of vibration **[3]**.

1.3 Fly Ash:

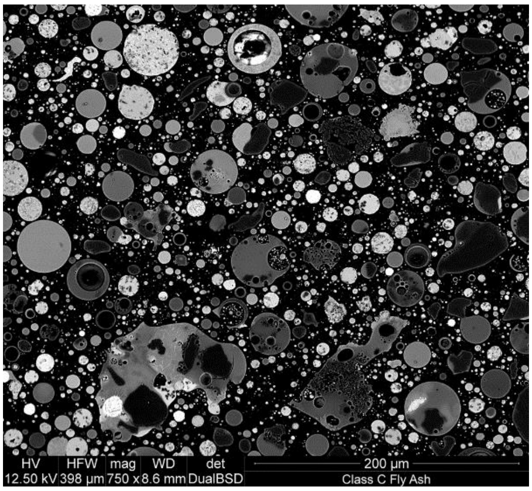

Figure 1.3: Cross Section of Fly-Ash Particle

Fly Ash is obtained from thermal power plants during combustion of coal. It is collected by particle filtration equipment like electrostatic precipitators. Particles of Fly Ash are spherical shaped and are Hydrophilic in nature. The spherical shape of particles make a ball-bearing effect that creates a lubricating action when paste is in fresh state, which then results in increased workability, reduced bleeding, reduced segregation, reduced slump loss, improved finishing and increased ease of pumping. The density of Fly Ash is 2.3gms/cm^3.

There are two classifications of Fly Ash, depending on the type of coal used; Class F and Class C.

Class F is produced from Anthracite and bituminous coal whereas Class C is produced by combusting lignite or subbituminous coal. **[4]**

Fly Ash is used as a secondary raw material to enhance the fresh and hardened properties of Self levelling Paste systems. It reduces the water demand and reduces the overall temperature of the system. It provides better packing of both cement and fine sand.

1.4 Super plasticizers:

Super Plasticizers are chemical admixtures that are added in mix so as to increase workability while keeping the mixture's composition unchanged, reduce water demand and increase strength and durability.

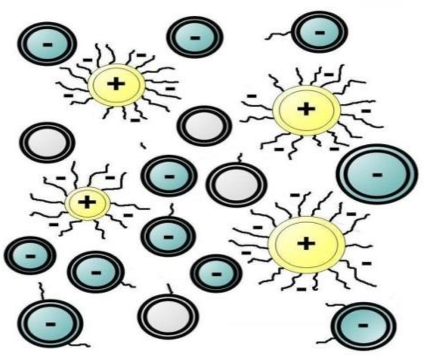

Figure 1.4: PCE displacement on a multi-phase suspension

Mechanism of the super plasticizers is known as adsorption. In this mechanism, agglomeration is broken down by repulsion of same charges and entrapped water is released. PCE's backbone, which is negatively charged, permits the adsorption on the positively charged colloidal particles. As a consequence of PCE adsorption, the zeta potential of the suspended particles changes, due to the adsorption of the COO- groups on the colloid surface. This displacement of the polymer on the particle surface ensures to the side chains the possibility to exert repulsion forces, which disperse the particles of the suspension and avoid friction. These forces can be directly detected by the use of the atomic force microscopy (AFM), working with model substances in liquid environment. **[5]**

1.5 Calcium Aluminate Cement:

Calcium aluminate cements are cements consisting predominantly of hydraulic calcium aluminates **[6]**. The method of making cement from limestone and low-silica bauxite was patented in France in 1908 by Bied of the Pavin de Lafarge Company. The initial development was as a result of the search for cement offering sulfate resistance. Calcium aluminate cements are the most important type of special cements. It is expensive and uneconomical to use it to substitute Portland cement but it is used for special applications and used in proportions to enhance properties of concrete such as:

- Rapid hardening e.g. Bridge widening.
- Abrasion resistance e.g. Dam Flushing gate.
- Abrasion and Thermal shock resistance e.g. Foundry floor.
- Thermal shock, freeze-thaw e.g. cryogenic facility.

- Resistance to bacteriogenic acid corrosion e.g. Sewer.
- Resistance to thermal shock e.g. fire training building.

Even though CAC has many benefits, it has a drawback that it reduces in strength over a long period of time. This reduction of strength is caused by a process known as conversion process in which metastable states of mono-calcium and di-calcium aluminates convert into a stable state of tri-calcium aluminate due to temperature and the time passed. This stable state of tri-calcium aluminate is denser, due to which the concrete shrinks and affects the strength of the structure.

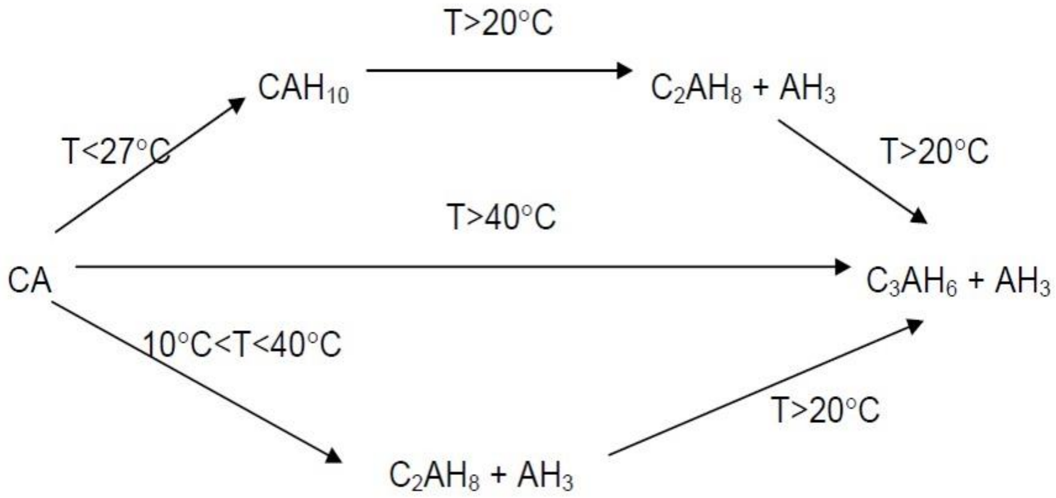

Figure 1.5: Conversion Reactions [60]

1.6 Research Objectives:

The objectives of this research are to:

- Investigate effect of Mixing Water Temperature on Self Levelling Paste System (SLPS).
- Investigate the acceleration effect of CAC on OPC in multi-binder SLPS.
- Investigate role of mixing water temperature in Conversion Phenomenon of CAC hydration.
- To increase open time of Rapid Setting Formulations by variating mixing water temperature.

CHAPTER 2

LITERATURE REVIEW

2.1 General:

In the world, Self-Compacting Cementitious Systems has been widely researched using mixed binary blends of Ordinary Portland Cements (OPC) and Calcium Aluminates Cements (CAC). It has been observed that CAC acts as an accelerator in the presence of OPC. But little study has been done on the behavior of CAC/OPC with Secondary Raw Materials (SRMS) in self -compacting paste systems at **Variable Mixing Water temperatures**.

Self-Compacting Cementitious Systems:

Self-Compacting Cementitious Systems (SCCS) can be divided into three phases:

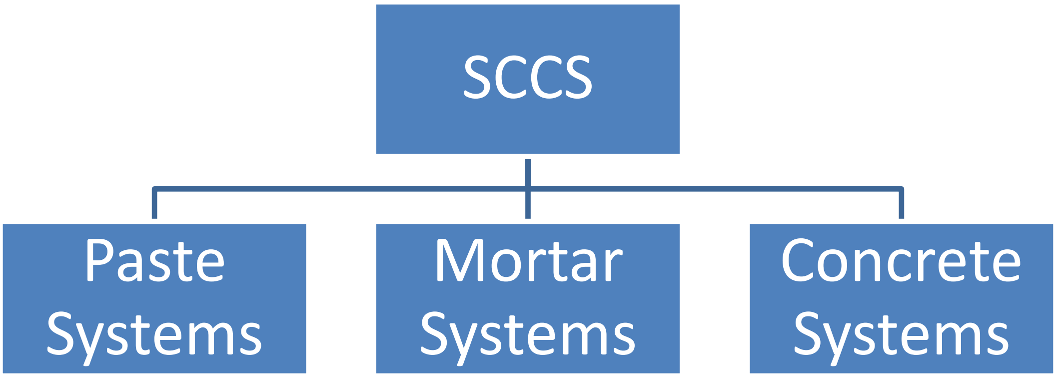

Figure 1.6: SCCS phases [58]

The sequential development of these systems is important and initially research should be carried out with paste systems to understand the variables involved. Generally what trend has been observed in the paste systems is also reflected in the mortar and concrete systems with little variation because of the inert nature of the aggregates involved.

Self -Compacting Concrete possess three essential fresh properties: [7]

- It should flow under its own weight and completely fill the formwork.
- It should flow through and around obstacles including reinforcements and narrow spaces without being blocked.
- It should remain homogeneous during and after transportation and placement against segregation.

The passing ability distinguishes SCC from other high consistence concrete. [8]

2.2Understanding the Cements:

2.2.1 Ordinary Portland Cement (OPC):

Cement is defined as the binding material having cohesive and adhesive properties which make it capable to unite the different construction materials. [9]. The Portland cement consists of lime containing components (CaO), clayey materials (Al_2O_3), silica (SiO_2). The main constituents of OPC include (C_2S), (C_3S), (C_3A) and (C_4AF). Other minor constituents include as Calcium Sulphate, Calcite, Calcium Oxide, and Magnesium Oxide. These constituents react with water to form various hydration products such as C-S-H, ($CaOH_2$), (C-A-S-H, calcium sulfo-aluminate hydrate) and (C_4ASH_{12}) [6]. High early strength is provided by C_3S allowing cement to achieve most of its strength in 7days. C_2S is responsible for the later developing strength of Portland cement paste and gains little strength until even 28days but its final strength is the same as that of C_3S. C_3A exhibits the flash set when hydrated, responsible for initial set after addition of gypsum in small amount (5%) and releases large amount of heat upon hydration. It shows little strength increase after 1day. Increase in amount of C_3A results in faster set. C_4AF behaves like C_3A in which it hydrates rapidly and gains low strength but does not exhibit flash set. The weight compound composition in OPC is shown in Table 2.1 [10].

Chemical Formula	Shorthand Notation	Chemical Name	Weight Percent
$3CaO.SiO_2$	C_3S	Tricalcium Silicate	50
$2CaO.SiO_2$	C_2S	Dicalcium Silicate	25
$3CaO.Al_2O_3$	C_3A	Tricalcium Aluminate	12
$4CaO.Al_2O_3.Fe_2O_3$	C_4AF	Tetracalcium Aluminoferrite	8
$CaSO_4.2H_2O$	CSH_2	Calcium Sulfate dihydrate (gypsum)	3.5

Table 2.1 Compound Composition of OPC [Nawy, Edwards G.2nd Edition]

A “Phase” is a name given to distinct materials with a specific combination of elements with specific arrangement. In all the phases of the cement, elements like Calcium, Silicon, Aluminum and iron are always present with some quantity of Oxygen. These combinations of elements with oxygen (known as Oxides) are further presented with notations such as [11]:-

C = CaO (Lime)

S = SiO_2 (Silica)

A = Al_2O_3 (Alumina)

F = Fe_2O_3 (Ferric Oxide)

S = SO_3 (Sulfate) generally referred as such

H = H_2O (Water)

When there is no water (Hydrogen Oxide) mixed with the cement, all the phases present in the cement are known as “anhydrous phases”. On mixing water in the cement combinations form between oxides and water, thus the phases are known as “hydrates”.

2.3 Basic Mechanism of OPC Hydration:

Hydration of cement is defined as “dissolution – precipitation process between binder grains and water” [12] (Figure 1.7). It is a process by which cement powder plus water transform from a fluid suspension to a porous solid within hours and strength gains over days and months [13]. Mainly hydration kinetics of cement involves two types of phases which are Silicate Phases (C_2S & C_3S) and Aluminate Phases (C_3A & C_4AF). Silicate phases are negatively zeta potentials and aluminate phases are positively zeta potentials. In impure state these cement phases are known as [12]:-

C_3S - Alite

C_2S - Belite

C_3A - Portlandite

C_4AF - Ferite

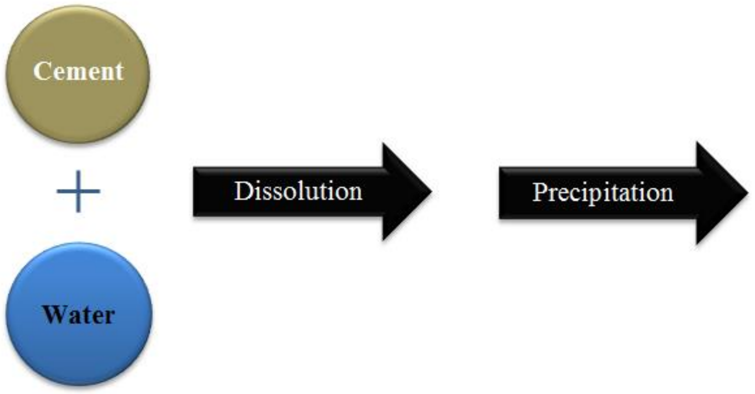

Figure 1.7: Schematic process of Cement Hydration

2.3.3 Hydration Products of OPC.

As a result of dissolution/precipitation process a growth is observed with the help of Scanning Electron Microscope (SEM). These growths are termed as Hydration Products and can be mainly classified as:-

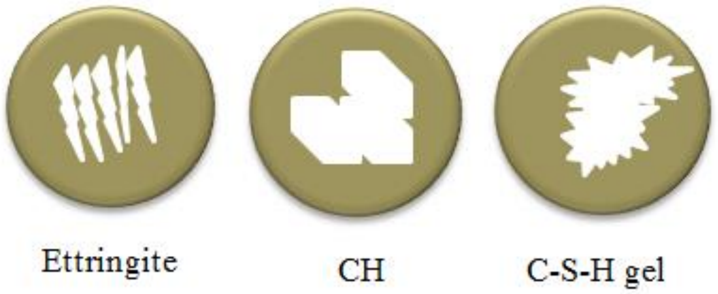

Figure 1.8: Products of Hydration

Ettringite forms at the earliest and is detectable within 8-9 minutes. It’s a needle shape product and is the result of C_3A phase reaction. It is also known as Calcium Sulfo-aluminate Hydrate (C-A-S-H). CH and C-S-H gel are the products which are associated with C_3S phase. Calcium Hydroxide (CH) is a well-defined crystal while Calcium Silicate Hydrate (C-S-H) is a poorly crystalline material. C-S-H gel can be further classified as High Density C-S-H Gel and Low Density C-S-H Gel. Former one is desirable whereas as latter one is the cause of creep and shrinkage. Only high density C-S-H gel can be seen from SEM [14].

2.3.4 Hydration Kinetics of OPC:

The kinetics of cement hydration is concerned with the relation between the degree of hydration "α" and the age "t" and with the factors that influence them. [15]

General pattern of hydration of cement is shown in Figure 2.4. This pattern involves certain stages which explain the sequential process of heat release, dormant period, and acceleration and deceleration stage under the influence of different phases of cement. Different stages are as under [12]:-

Stage-I:	Period of rapid heat of evolution by C_3A. This normally lasts for few minutes.
Stage-II.	Period of relative inactivity or induction, also known as Dormant Period.
Stage-III.	This is the acceleration stage which involves maximum rate of heat evolution. This is the beginning of C_3S phase at the end of the induction or dormant period.
Stage-IV.	This is deceleration stage attributed to C_4AF phase. This stage is diffusion control stage.
Stage-V.	This is a steady state stage under the influence of C_2S phase.

Table 2.2: Stages of OPC Hydration

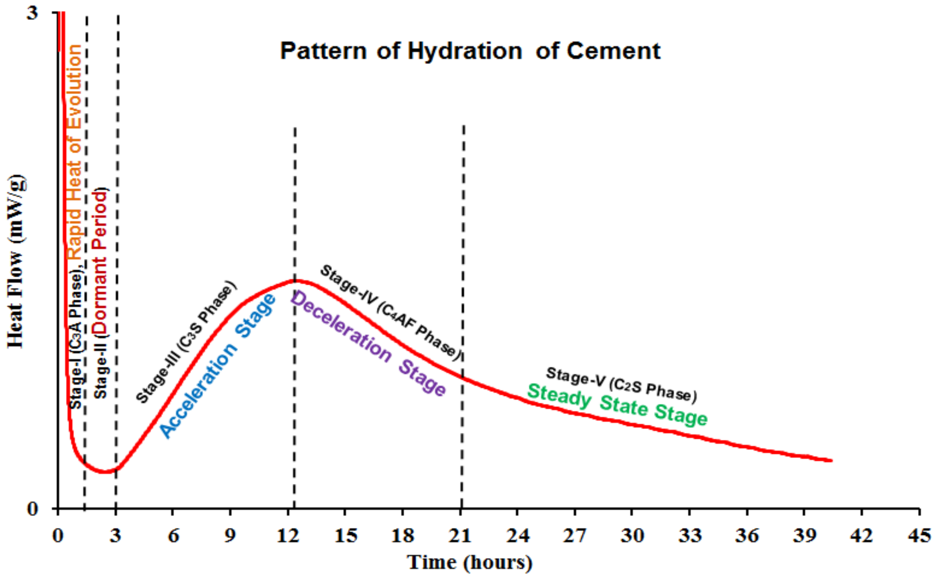

Figure 1.9: Hydration Kinetics of OPC [58]

2.3.5 Reasons for Induction/Dormant Period.

The actual reason for this dormant period still remains unclear. However, two theories exist to explain the process. According to the first theory, a protective layer forms around the reacting grains just after the initial high heat release which prevents the further reaction till the onset of acceleration period. The main problem with this theory is that no evidence of formation of such layer has yet been obtained and there is no satisfactory explanation for disappearance of the layer with commencement of acceleration period.

The second hypothesis denies the presence of any real induction period and specifies it with slow development of hydrates by nucleation and growth. Drawbacks of this theory includes that neither it explains the drastic slowdown of the rate of reaction just after the initial very high heat release nor there is any clarification of the reason of slow reaction of highly soluble Alite [16,17].

The dormant period ends after few hours due to the onset of growth of two main hydration products, calcium hydroxide (CH) and calcium hydrate silicate (CSH). This marks the start of acceleration period where the calorimetric curve again ascends and reaches a second peak. This acceleration can be related to C_3S hydration which continues for 15 to 20 hours. The start of the acceleration period can also be related to the initial setting time by Vicat. The hydration of C_2S is much slower usually starts after 15 hours hence after reaching the second peak then again there is deceleration, but the hydration process of C_2S continues with a steady rate and decreases gradually [12].

The early stage of cement hydration plays a decisive role in workability. Concrete is a material that undergoes a change from plastic to elastic state due to chemical reactions taking place in the first few days after mixing. Being in the plastic state concrete properties is described by rheology (yield stress and viscosity). Evolution from plastic to elastic state is due to chemical reactions taking place on the surface of cement particles. The C-S-H growing from the cement grains are responsible for setting of the cement and strength development [9].

2.4 Calcium Aluminate Cement (CAC):

High-Alumina Cement (HAC) with 32-45% Al_2O_3 was introduced in United Kingdom after World War I. This type of cement is different from Ordinary Portland Cement which contains much less share of alumina as shown in Figure 2.5. With the passage of time and new development trends in the cement field many types of aluminous cements have been introduced with alumina contents varying from 50-90%. Common trait of all these cements is that the hydrates produced are calcium aluminate due to which they are best known as Calcium Aluminate Cements (CACs). As compared to OPC these cements are much costly, less produced and are used for special purposes and in routine applications they do not compete with OPC [18]. Calcium Aluminate Cement (CAC) is special cement which is considered as the most important type of non-Portland cements. It is very expensive and its use is only about one thousandth of that of commonly used Portland cement. CAC is only used at places where certain special properties to mortar or concrete are required; therefore it is not economical to use CAC just as a simple replacement of Portland cement [8].

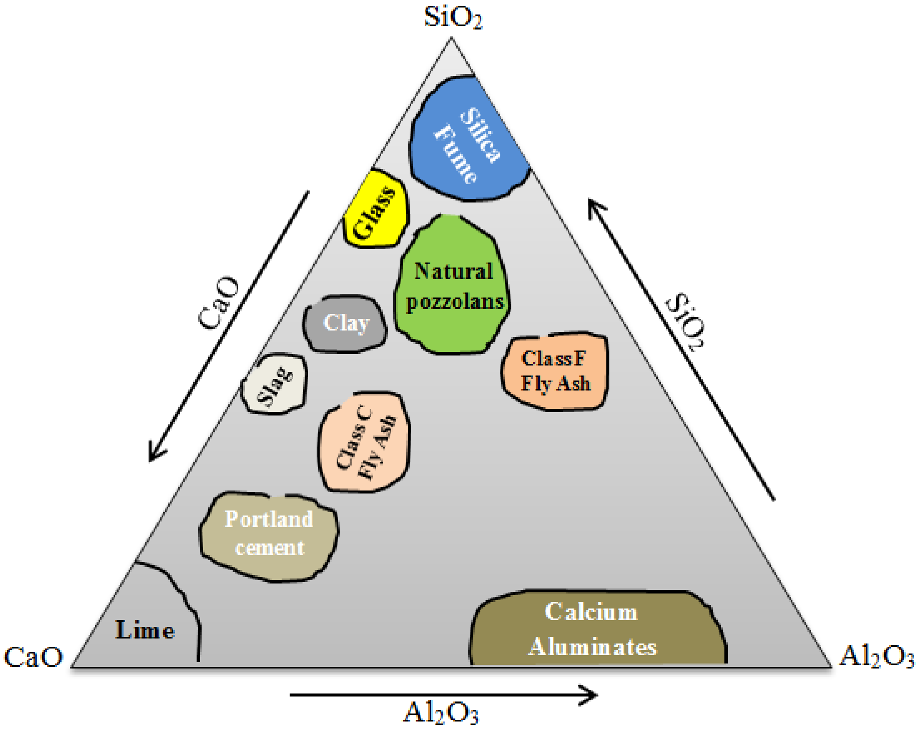

Figure 2.0: Phase Diagram of the CaO-Al2O3-SiO2 Systems [Nawy, Edward G.]

2.4.1 Origin of CAC:

Calcium Aluminates Cements were developed and commercialized in the late 19th and early 20th - century, as an alternative to Ordinary Portland cements based on calcium silicates based Cements. [22]

The reason for its development was the decomposition problem associated with Ordinary Portland Cement (OPC) under Sulphate attack and sea water because of the presence of Calcium Silicate phases.[23,25]

The industrial development of CACs is linked with the work of J.Bied in the laboratories of Pavin de Lafrage Company based in the South of France. Although the name of Bied is inexorably linked to Ciment Fondu Lafrage (CFL) which was patented in 1908, the development of cements resistant to aggressive environments was initiated by L.J. Vicat [26] from the 1840s. The latter proposed a link between the chemical composition and sulfate resistance. Vicat classified cements according to an index, expressed as the ratio between the fraction silica and alumina, originating from clay, and the fraction calcium + magnesium, from limestone. Based on several experiments, he proposed that an index greater than the unity should provide a cement highly resistant to sulfate attack. However when this ratio is increased by increasing the silica content, the reactivity of the material decreases. Simply, increasing the proportion of alumina in the normal process led to flash setting. Bied focused in the investigations on lower lime to alumina compounds and experimented for many years to produce cement with low content of silica and high content of alumina. The fusion process of bauxite or other aluminous and ferruginous materials with limestone had finally been mastered in 1916.

The first centenary of French patent was celebrated in 2008, with the organization of the latest in a series of International Conference on Calcium Aluminates Cements. This event clearly demonstrated that CACs continue to generate high scientific interest in the field of construction materials. However the use of CACs has gone through several up and downs since the patents from Bied in France, Spackman in USA, Roth in Germany and Snelus in UK. The manufacturing methods required several years of improvement before obtaining satisfactory properties on an industrial scale. The reconstruction of civil infrastructure following World War 1, participated in the development of the material in reinforced concrete structures.

Although CAC has not been recommended for structural concrete since the 1970s, this material develops unique properties and technical solutions, involving specific fields of construction such as self-leveling compounds in floor finishing, rapid hardening materials in the structures repair, refractory and heat resistance or abrasion resistance concretes.

2.4.2 Composition of CAC:

Like OPC, CAC clinker also comprise of the same basic oxides (SiO_2, Al_2O_3 & CaO) However, their proportion varies. OPC is defined as Calcium Silicate cement because of high lime and silica content, whereas, CAC is based on high alumina and lime content.

The main Alumina (Al_2O_3) content can range between 40-80%. The main phase of Calcium Aluminates cements is mono calcium Aluminates (CA). Other minor phases may include dodecacalcium heptaaluminate ($C_{12}A_7$), tetracalcium aluminoferrite (C_4AF), dicalcium silicate (C_2S) and dicalcium alumniosilicate (C_2AS).

Mono calcium aluminate ($CA = CaO.Al_2O_3$): CA is the main constituent. It is responsible for the particular cementitious behavior in CAC. Its setting is slow, but it hardens very rapidly. [28]

Dodeca calcium heptaaluminate ($C_{12}A_7=12CaO.7Al_2O_3$): It is present in cement in small percentages. It sets rapidly but it makes no contribution to the strength, so not desirable constituent in CAC except in minor amounts.[28]

Tetra calcium aluminoferrite ($C_4AF=4CaO.Al_2O_3.Fe_2O_3$): C_4AF is the second most abundant component of CAC. Nevertheless, it makes very little contribution to the setting and the strength development.[23] It helps triggering the setting process. However its presence is strictly controlled by the manufactures since too much would lead to early stiffenening.

Dicalcium silicate (C_2S=2CaO.SiO2): C_2S behaves similar to OPC . Its hydration is slow and its contribution to the strength is at later stages rather than at early ages.

Dicalcium aluminosilicate (Gehlenite) ($C_2AS = 2\ CaO.Al_2O_3.SiO_2$): Like C2S , C2AS sets slowly and contributes to the strength after a considerable period.[28]

2.4.3 Hydration reaction in CAC:

Chemical process of hydration in CAC is somewhat quite similar to that of OPC. However, the products of CAC are quite different from those formed in OPC. Hydration process in OPC occurs due to “dissolution and precipitation”. Anhydrous phases in cement dissolve in water to give ions in the solution just like sugar or salt mixing in water. Unlike sugar or salt the ions of cement phases come out of solution because of their lower solubility as hydrate phases. On the other hand when CAC is placed in water Calcium ions and Aluminum ions dissolve in the water to give a solution. Results are the combinations of several different types of hydrates along with poorly crystallized gel-like phases; which are not stable and generally termed as “Meta stable hydrates” [14].

Hydration reaction occurs by the process of dissolution and precipitation. The anhydrous phases of cement dissolve to give ions in solution; these ions recombine in different proportions with the ions of water to form precipitates. When CAC is dissolved in water, the main phase is CA, which ionizes in water to liberate calcium ions (Ca^{2+}) and aluminates ions ($Al(OH)_4^-$) , which continues till the saturation level. Meanwhile, the PH of liquid phase increases up to 12, particularly 11.2. [30-32]. It is followed by nucleation and precipitation of calcium aluminates hydrate crystals and gibbsite (AH_3). These hydrate crystals start to precipitate close to the unreacted clinker surface by forming a gel layer. [36, 30-33] Fujii et al [30] reported that this layer is permeable to water. Therefore, further hydration of the underlying clinker grains take place easily as a continuous process as long as enough water for dissolution and hydration is available. [36, 30-35]

The hydration and strength development are illustrated in the figure 2.1 [36]

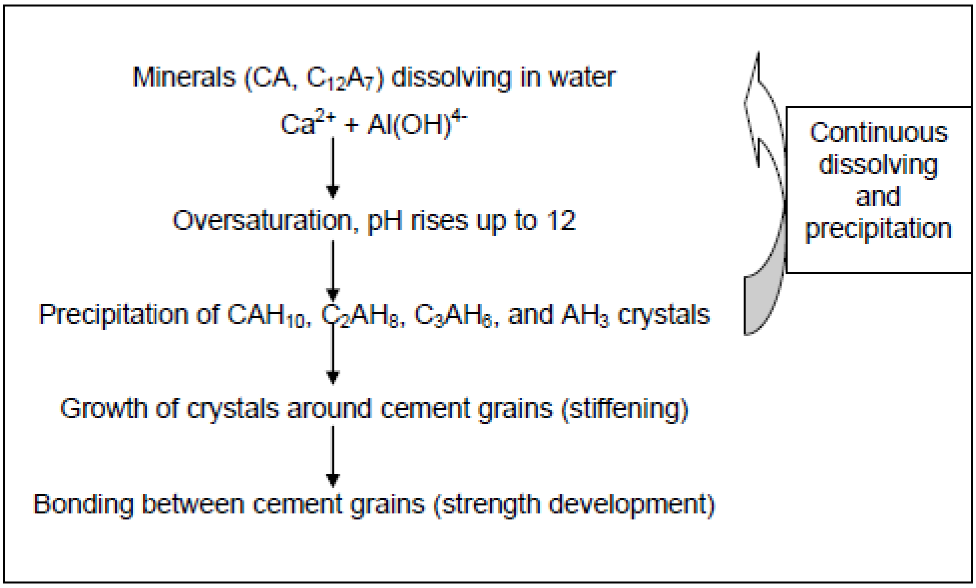

Figure 2.1: Summary of Hydration Mechanism of CAC.[30]

As seen in the figure above, at initial stages, hydration crystals form a gel layer coating the unreacted grains. The mix is still plastic. Depending on the progress in crystal formation as a result of continuous dissolving / precipitation, some bonds take place among clinker grains, resulting in stiffening of the mix. As hydration proceeds further, the mix gets stiffer by gaining strength. Finally, the final stable structure is formed when the water is used up and no further hydration crystal grows. [36]

Different hydration products are formed but CA and $C_{12}A_7$ dominates the main phases of the cement. The different calcium aluminate hydrates include CAH_{10}, C_2AH_8, C_3AH_6, C_4AH_{19}, $C_4A_3H_3$. Nevertheless, principle hydrate forms are CAH_{10}, C_2AH_8 and C_3AH_6, together with crystalline AH_3 and/or amorphous AH_3 gel [30,31,37,38]. Since unlike OPC, there is no portlandite (CH) in hydration products, a good resistance of CAC to many aggressive agents is assured. [23,25]

2.4.4 Conversion reaction in CAC:

Formation of meta stable phases in CAC are the temporary phases which have low density, less porosity, high energy and almost occupy all the available space. Because of this fact CAC exhibit high early strength as compared to OPC. Depending upon water content available and ambient temperature these temporary phases tend to mix together thus forming new phases which are denser and with less energy causing reduction in overall volume owned by these phases. These converted phases are called "Stable phases" which enhance the porosity within the matrix, resulting reduction in overall strength in some cases up to 50% [39].

As a fact conversion process is affected by water-cement (w/c) ratio and ambient temperature. In case when w/c ratio is ≥ 0.7 at low temperature, conversion process accelerates. Results are decrease in the volume (previously occupied by metastable hydrates), leaving high porosity and consequent decrease in the strength. This schematic volume relationship is shown in Figure 2.2[39].

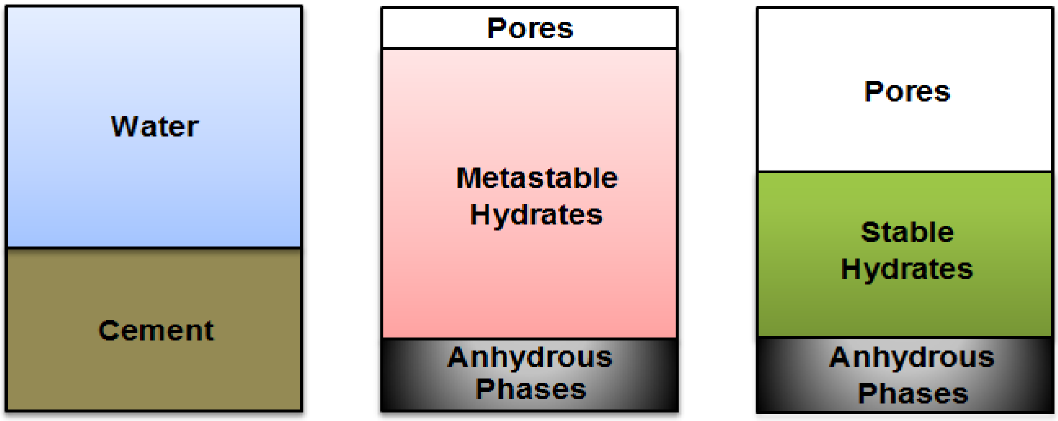

Figure 2.2: Schematic Volume changes due to Conversion Process at w/c > 0.7 [Newman, J., 2nd Edition]

a. Water + Cement b. Before Conversion c. After Conversion

In a case where w/c ratio is kept ≤ 0.4, less water and space is available for all the cement grains to react, resultantly less of the unstable hydrates are formed. Water released due to conversion process is again utilized by more cement grains and again unstable hydrates form and again conversion takes place. In this way process of conversion becomes systematic and develops slowly. Moreover reduction in solid volume is less and matrix proves to be less porous.

2.4.5 Properties of Fresh CAC Concrete.

2.4.5.1 Workability:

For the conventional concretes and without admixtures ease in placing CAC concretes is the same as that of OPC concretes for the same w/c ratio. Apparent look of the CAC concrete is dryer but does not mean that it demands more water and flows better under vibration. Workability of concrete retards and suffers when worked with blend of CAC and OPC in considerable proportion (keeping main proportion of OPC) due to faster setting. These effects of less flow ability and faster setting are controlled with judicial dosage of modern generation Super plasticizers. With w/c range of 0.35-0.40 it is possible to produce flowing concrete for 2 hours, with strength of around 30 MPa (4500 Psi) at 8 hours and with ambient temperature of 5°C. [40]

2.4.5.2 Rate of Reaction and Heat Evolution.

In Portland cements hydration products are deposited around cement grains due to nucleation effect where as in calcium aluminates cement hydration products develop throughout the space. A schematic process is shown in Figure 2.9. Rate of reaction slows down due to either consumption of water or due to lack of space for the hydrates to precipitate. In CAC concretes heat of hydration is evolved for the short time. This heat generation is rapid as compared to rate of heat dissipation, due to which self-heating gears up in the concrete body. Due to this phenomenon of self-heating, temperature within the body rises in certain cases from 50°C to 80°C. [41]

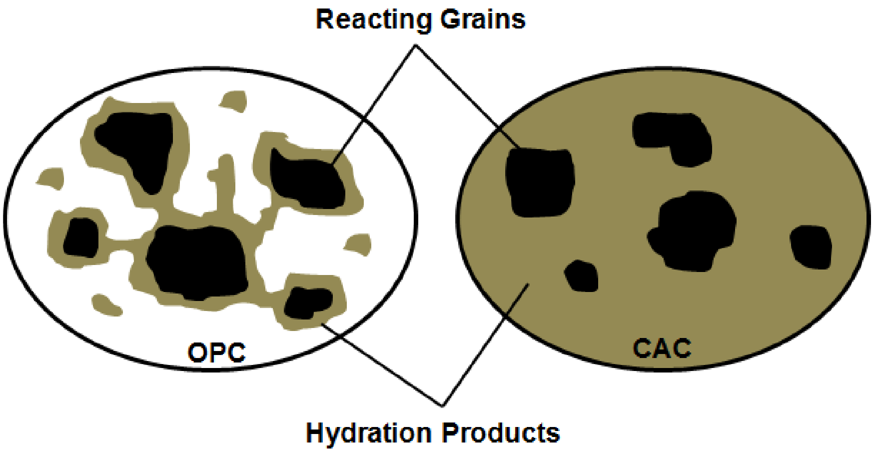

Figure 2.3: Schematic representation of the Nucleation of hydration products in OPC and CAC [Lamour, V. H. R. et al]

Due to pattern of reactions and high heat emission CAC concrete hardens rapidly and obtains strength of more than 20MPa (3000Psi) at 6hours and 40MPa (6000Psi) at 24 hours. CAC concrete needs to be kept wet in order to avoid drying and surface damage due to dehydration. For the same reason it is recommended that formwork should be removed after 6hours and surface should be sprayed with water. The biggest advantage of self-heating is that concreting can continue in very cold weather even at sub-zero temperatures provided concrete is protected against freezing [14].

2.4.6 Strength Development of CAC Concrete.

Due to the high heat of hydration, successive formation of Meta stable and stable hydrates, the strength development of CAC concretes is more complicated than OPC concretes. Effects of various temperatures and w/c ratios on the strength development profile are shown in Figure 2.11.[14]

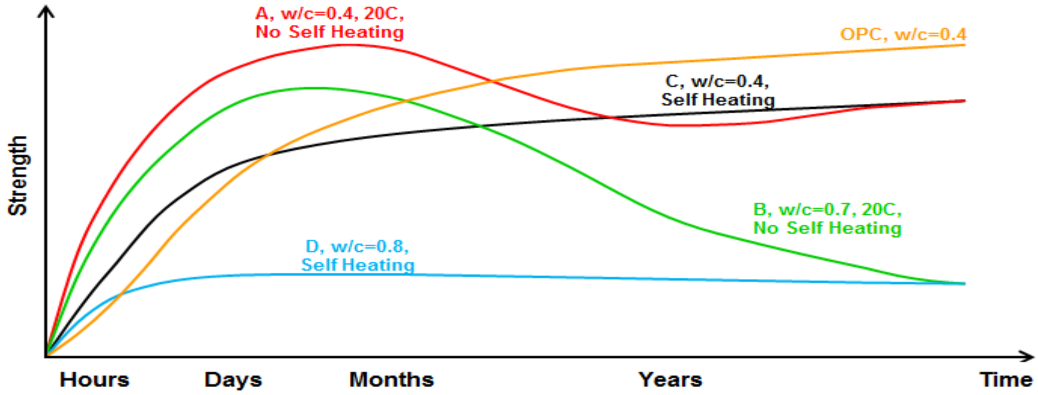

Figure 2.4: Strength development of CAC concrete at low and high w/c ratios, compared with Portland cement concrete [Newman, J., 2nd Edition].

Curve-A with w/c ratio of 0.4 at temperature of 20^{o}C shows a loss in strength due to the conversion phenomenon in CAC concrete. This type of case is very rare to find as no CAC concrete is without self-heating, same is the situation with Curve-B. Substantial strength reduction in case of Curve-B is due to w/c ratio, reason for which is already explained and shown in Figure 2.8. Strength gain is seen in case of Curve-A only because of low w/c ratio as already explained in Figure 2.9. In case of self-heating with different w/c ratios and curing at the initial stage no strength drop is observed in Curves C &D [14]. Strength reduction in both the cases is considerable but important point to note is that it does not approach zero. Strength drops due to conversion but again concrete at ambient temperatures of 10^{o}C-20^{o}C might take many years to perform conversion. [39]

With increases in w/c the converted strength of CAC concrete decreases. For this purpose w/c ratio of 0.4 is recommended which keeps the allowance of water absorption by aggregates as CAC reacts much faster than OPC and aggregates have less time to absorb water .[40]

2.4.7 Factors Affecting Conversion and Strength Development.

2.4.7.1 Curing.

Conversion is a response of a chemical reaction in the presence of self-heating environment which cannot be avoided as concrete cannot be kept in dry environment for its complete life. To avoid the after effects of rapid hardening, solution is curing and to avoid the substantial loss in strength due to conversion. Solution is low w/c ratio at low ambient temperatures. Investigations have proved that concretes with w/c of 0.4 first stored in the air but later cured in water has resulted in good strength [41].

2.4.7.2 Addition of Silica.

Researchers [43- 45] on the basis of their investigations have recommended the use of Supplementary Cementitious Materials which contain Silica e.g., Silica Fume, Ground Granulated Blast Furnace Slag (GGBFS), Metakaolin etc for the better control of conversion. Investigations proved that at temperatures below 50^{o}C unstable hydrates react with silica and form stable hydrates and if the silica addition is high there would almost be no strength retardation due to conversion. Care must be exercised as very high addition of silica affects the early age strength development.

2.4.7.3 Type of Aggregate Used.

Reportedly [45] higher strengths are achieved by using calcareous aggregates as compared to siliceous aggregates.

2.4.7.4 Use of Supplementary Cementitious Materials (SCMs) in CAC.

Common SCMs like Fly Ash, Silica Fume, GGBFS, Metkaolin and Limestone powder are used in OPC for better flow ability, stability and durability .[46] All those SCMs which are rich in silica if used in replacement mode with CAC in appreciable quantity can contribute towards stable and durable CAC concrete. [43-45] But as a matter of fact additions of such materials with CAC have not been studied extensively. Very little work is done which registers some improvements in rheology of Self-Compacting Concretes. [44]

2.4.8 Use of CAC in Mixed Binder Systems.

Knowing well the advantages and disadvantages of CAC and OPC, need arose to have such Cementitious arrangements which fulfill the requirements of special applications. This technical desire gave birth to a system – known as “Mixed Binder Systems” in the world of concrete technology. Ingredients of the interest are blended dry with fixed proportions of each on the basis of their chemistry knowledge. Typical applications of these arrangements are purely non-structural. [14]

Researchers [47, 48] have worked out these blends on the basis of CAC, OPC and CS systems as shown in Figure 2.5.

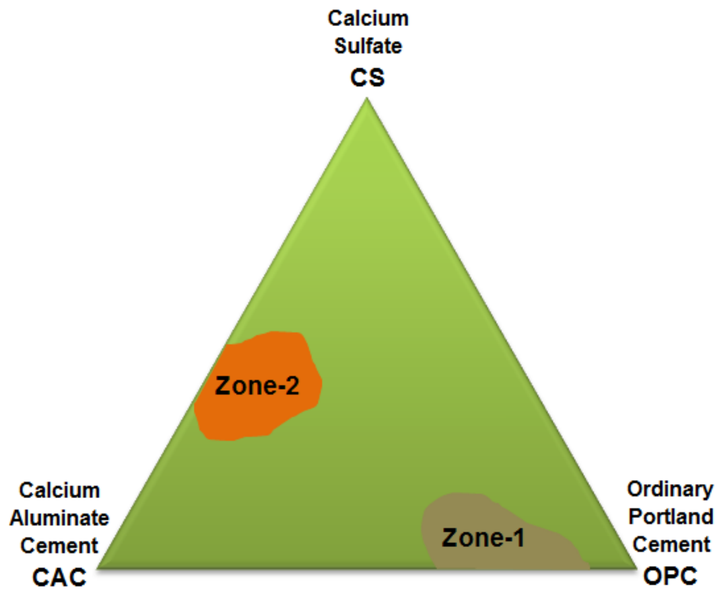

Figure 2.5: Zones of Formulations in Cement and Building Chemistry

These special binder systems can also contain admixtures both chemical (super plasticizers, accelerators, retarders, VEA etc) as well as minerals (fillers and pozzolanic SCMs). In addition to all above mentioned fine aggregates can also be the part of these formulations. Zone-1 indicates the blends based on binary mixtures of OPC and CAC keeping more percentage of OPC and smaller of CAC. Effect of this system is rapid setting and hardening. Flow ability, stability and durability parameters of this mix in self-compacting paste (SCP) systems and self-compacting mortar (SCM) systems are achieved with the help of mixing suitable proportions of SCMs, super plasticizers, VEA etc. [48]

Basic parameters which control the traditional/conventional concrete are w/c ratio, low temperatures and air-water curing. Still a lot is required to study and investigate the effects of pozzolans in the binary or ternary mixes in Self-Compacting Cementitious Systems.

2.5 Admixtures.

Admixtures are generally divided into two categories i.e., Physical Admixtures and Chemical Admixtures. Both the classes can further be sub-divided as shown in Figure 2.12 [15] Chemical Admixtures are used to accelerate hydration whereas Mineral admixtures are used to act as a filler or pozzolans basing upon their chemical and physical characteristics. When SRMs/SCMs are added with clinker during grinding then these minerals are termed as *Additives* but when they are added in a replacement mode during mixing of different ingredients, they are termed as *Admixtures.* [15]

2.5.1 Super plasticizers.

Properties of hardened cement paste and concrete are affected by their flow behavior which is controlled by dispersing cement grains into the mixing water. Super plasticizers have been used for the purpose. Selection of adequate chemical admixture and evaluation of their effect on early age properties like workability retention and initial setting time are of significant importance [49].

2.5.2 Need for Super plasticizers.

All powders particles which have been subjected to grinding carry some electrical charges which might get activated on addition of water. With the previous knowledge we know that cement powder carry two phases named as Aluminate Phases (Positively Charged) and Silicate Phases (Negatively Charged). Upon addition of water these charges would get activated and resulting in observable flocculation in the cement paste

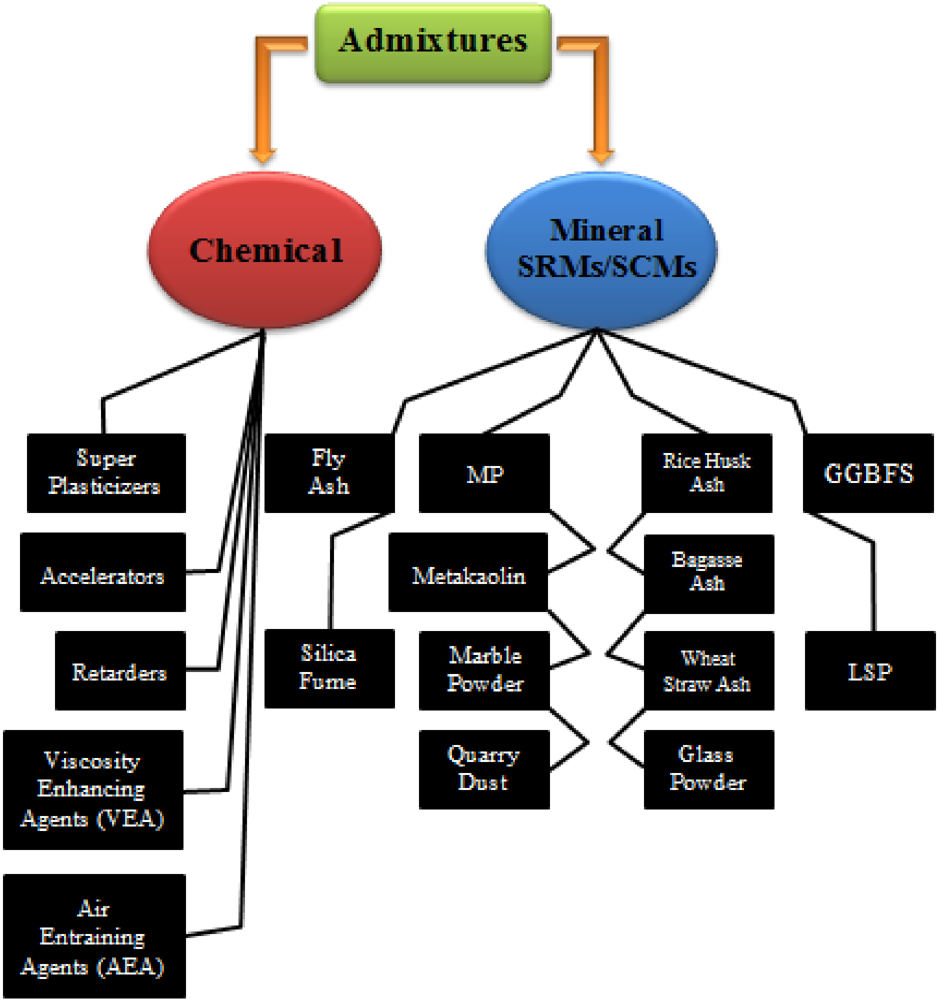

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Figure 2.6: Classification of Admixtures

This flocculation is due to attraction of opposite charges. There are two ways to break this flocculation which are:-

- Add more water
- Add some Super plasticizers

Adding water would increase the distance between cement grains and flocculation would break but this process is coupled with porosity, the only foe of durability. There will be substantial loss in strength which at any cost is undesirable. Considering the validity, Super plasticizers (SP) are added which get grafted on Aluminates Phases and these phases start acting as negatively charged phases. SP also increase the charge of Silicate phases. In the matrix now all the phases are negatively charged so they repel each other. Flocculation is broken even with the bare minimum requirement of water to get the desired or targeted flow ability. This bare minimum but essential water required to get the desired flow ability by adding SP is termed as *Water Demand (WD)* of a system. Therefore SP may always be considered not only the workability enhancer but durability enhancer also [15]. Mechanism of SP can also be seen in Figure 2.7

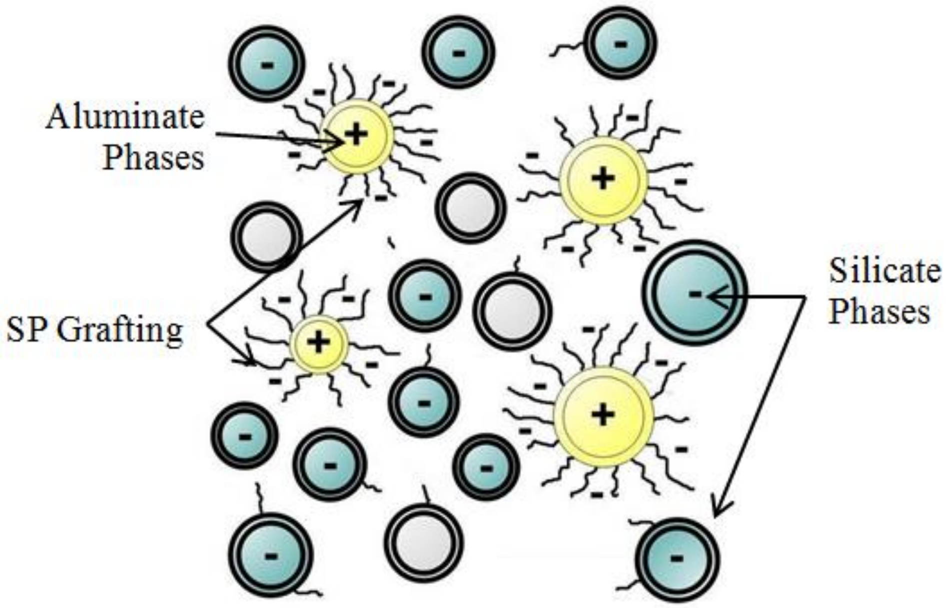


Figure 2.7: Mechanism of Super plasticizers [Wikipedia]

Certain SP are retarders which lengthen the dormant period during hydration whereas those SP as accelerators shortens the dormant period. Almost 90% SP act as retarders also.

2.6 Secondary Raw Materials (SRMs).

Secondary raw materials are either naturally occurring or the industrial by-products or waste materials. In today's modern concrete world they are best known as Secondary Raw Materials (SRMs) or Supplementary Cementitious Materials (SCMs) which can be either inert or pozzolanic in nature. They are also known as fillers or powders. These SRMs/SCMs are pozzolanic (with Cementitious value) or inert powders (crystalline in nature) which are usually added as a replacement for a part of the cement and are used to modify the properties of modern concrete systems in both fresh and hardened state. SRMs are made finer than cement and are always added to improve the response of concrete in fresh and hardened state. These responses may include the control of early heat evolution, reduction in shrinkage, dense packing, better flow, increase in strength, and improvement in microstructure, economy of the system and durability of the concrete.

During process of cement hydration in modern concrete mix, all cement grains do not get hydrated and act as fillers in voids. SRMs at first place reduces cement content due to cement replacement, secondly they react with CH (Calcium Hydroxide) – produced as a result of cement hydration; further forms products of hydration which possess Cementitious properties. Net results of this hydration kinetics are the refinement of microstructure, reduction in porosity and improved strength of the system. Refined microstructure and less porosity are the strong defensive mechanisms against certain deleterious attacks and harmful agents. Hence SRMs improve performance of structures including durability characteristics [50].

2.6.1 Advantages of Using SRMs.

Uses of SRMs can bring the following advantages [50]:-

- SRMs improve the transport properties of the system,
- Reduction in the water demand,
- Increased flow ability and strength,
- Reduced shrinkage.

2.6.2 Mechanism of Action of SRMs.

Initially it was believed that fine particles would increase the water demand which would affect the durability of the concrete. Later on investigations proved that these fine particles comprising of binders and mineral powders fill the spaces between aggregate particles. Space left within the fine particles is then filled with water and air. Mixing of these fine particles in the systems enhanced the problem of their flocculation further resulting in more demand of water for better workability. Super plasticizers were introduced to break the flocculation in order to achieve uniform packing and flow [50]. SRMs alone or their blends usually result in binary or ternary binder systems and it is very difficult to describe their hydration mechanism. SRMs are normally included in replacement mode which produces Dilution (of clinker content), physical packing with or without nucleation effect and pozzolanic effects [51]. It must be noted that the self-compacting paste (SCP) systems act as a transport medium for aggregates within self-compacting concrete (SCC) systems and SRMs forms the essential part of SCP systems. They also improve or modify the properties of self-compacting mortar (SCM) systems, thus producing necessary cohesive and viscous paste/mortar systems and resulting in workable, stable and durable concrete [52].

2.6.2.1 Fly Ash (FA).

Fly Ash (Figure – 2.14) is a byproduct of combination of coal pulverized coal in the furnaces designed for thermal power plants. Characteristics of FA are dependent upon coal chemistry and combustion process. By burning Lignite type coal, Class-C Fly Ash is obtained while Class-F Fly Ash is a result of Anthracite type coal. The temperature during combustion is as high around 1200^{o}C. Final product ash in fine powdered shape (<100um) is carried along in the air stream and collected by thermal/mechanical precipitators and during the process it cools down quickly. With heavy particles of around 15% are called Bottom Ash. Under high temperatures inorganic compounds in coal melt and liquid assumes a spherical shape forming fly ash glass particles containing various minerals as well. SCC with ASTM Class-F Fly Ash would delay the setting, will have low heat of hydration, will have less shrinkage. Its early strength development is less but around 28days its strength will start rising [51]. Fly Ash has usually hollow circular particles and they are Hydrophilic in nature. Under varying power demands ignition efficiency changes and peak temperature vary resulting in varying amounts of un-burnt Carbon in Ash which is undesirable. High Calcium Fly Ash is usually obtained by combustion of Lignite Coal and contains up to 30% Calcium Oxide and 11% Magnesium Oxide. ASTM-168 states that for Class-F Fly Ash the sum of SiO_2, Al_2O_3 and Fe_2O_3 should be greater than or equal to 70 (%). While for Class-C Fly Ash the above Oxides content should be at least 50 (%) [50].

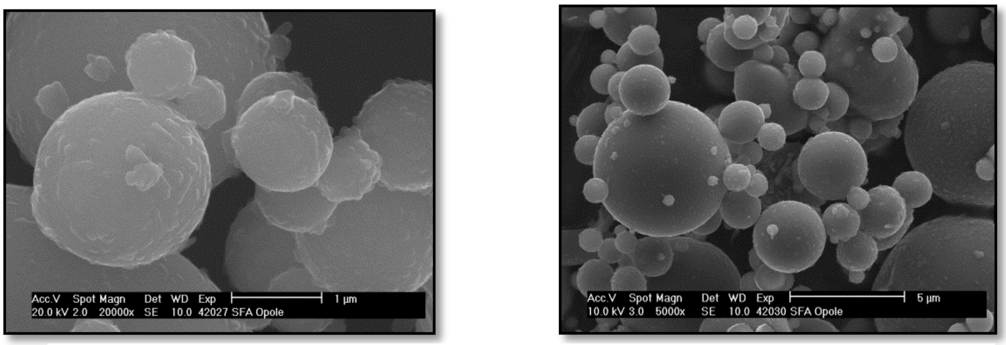


Figure 2.8: Scanning Electron Microscopic Images of Fly Ash particle shape and surface morphology

Few characteristics of the Fly Ash are listed below [17]:

- Like cement Fly Ash also acts as a binder in the presence of cement and H_2O.
- Particle shape of the Fly Ash is spherical / circular.
- Fly Ash is hydrophilic in nature resultantly less water is demanded.
- The density of Fly Ash is 2.3gms/cm^3 as compared to that of cement which is 3.15gms/cm^3.
- Fly Ash provides Nucleation sites where products of hydration precipitate preferentially.
- Fly Ash reduces temperature of the system due to dilution process and act as a heat sink.
- Fly Ash provides better packing of both cement and fine sand.

In the presence of Gypsum, Fly Ash particles show enhanced dissociation resulting in the formation of more hydration products and enhanced strength of the system.

Lignite based Fly Ash (Class-C) in High Strength Concrete offers generally good freeze-thaw resistance. Higher is the Fly Ash content in a system; lower is the freeze-thaw resistance. Replacement of cement with Class-F Fly Ash leads to lower freeze-thaw resistance.

2.6.2.2 Hydration Mechanism of FA:

Fly Ash has been widely used in concrete since it reduces cost of concrete materials, conserves energy and reduces environmental problems. Certain problems are also linked with this material, as FA has relatively low surface area and slow pozzolanic activity. At normal temperatures, the pozzolanic reaction is slow to start and it does not progress to any significant degree until several weeks after the hydration starts [53].

The effect of FA on the hydration kinetics of cement depends on FA replacement ratios and curing temperatures. At temperatures from 20°C to 35°C, FA retards cement hydration in the early period but accelerates in the later period. On increasing the replacement ratio at normal temperatures, stronger effects are observed. At temperatures above 50°C and with higher replacement ratios, hydration of cement is impeded and effect is retarded. [54] With regards to the influence of curing temperatures in water on FA-cement paste, it is reported [54] that with the w/c ratio of 0.50, FA enhances cement hydration at lower curing temperatures but shows a retarding effect at high curing temperatures. At low w/c ratios and at higher curing temperatures with high replacement of FA, hydration of cement gets retarded because of insufficient water or space available for the C-S-H gel to grow.

FA is believed to be inert at very early stage. During initial 24 hours of hydration effect of FA is dependent upon the amount of water available and the different mechanisms at the different stages of hydration. In initial minutes heat of hydration increases with addition of FA as more water is available. In the dormant period, the increase in water reduces the calcium concentration in the pore solution. As the next stage after dormant only starts when super saturation in the dormant period is achieved. This reduction of Ca^{2+} is attributed to the presence and condition of FA. This action of FA as a “Ca sink” agent depresses the calcium concentration in the initial hours thus delays the CH and C-S-H nucleation and crystallization, resulting in retardation of hydration [48]. Researchers [55] observed that this retardation effect by FA is very intense with C_3A and C_3S. By following Figure 2.3, it is evident that retardation effect in hydration remains in dormant (Stage-II) as well as in acceleration (Stage-III) stage also. But it accelerates hydration after the acceleration stage as increase in water at this stage helps the hydration process.

CHAPTER 3

EXPERIMENTAL PROGRAM

3.1 General:

Before this investigation a lot of research was done on self-compacting and self-leveling concrete systems. In recent construction practices these types of concrete systems are being widely used. However investigation of variable water temperature on self-leveling concrete is a pioneering concept, have significant effects on concrete properties. A well-planned systematic investigation approach was adopted comprises on chemical composition and densities of SRMs and cement was determined. A variable ratio of SRM was blend with OPC and CAC while forming different formulations. Approach also includes determination of particle size with the help of XRD of powder particles. With help of Vicat apparatus water demand, setting times along with super plasticizer demand of self-leveling paste were obtained. All the paste formulations were blended using the Hobart Mixer, shrinkage value from shrinkage apparatus, Flow is achieved using Mel-flux (Polycarboxylate ether based plasticizers), to avoid segregation low w/p ratio was used. Flow rate was noted for each system using Hagerman’s mini-slump cone. Curing and strength testing was done as per DIN EN-196. Besides calorimetric analysis, shrinkage measurements, microstructural study by scanning electron microscopy and mercury intrusion porosimetry and XRD analysis of hydrated sampled at specified ages was also the part of research.

3.2 Research Materials:

OPC grade 40 and Calcium Aluminate Cement type ISTRA-40 were used in the experimental program, secondary raw material i.e. Fly Ash (BauMineral KraftWerkstoffe) was used along with the CACs in replacement mode. The super plasticizer used was third generation PCE based, Mel-flux Powder 2651 F for paste system. The mixing water in all the paste formulations was as per the water demand of the SLP Systems.

3.3 Powder Particle Characterization:

The results for particle size and surface area were carried out with help of Beckmann Coulter LS 230 laser granulometer. Chemical analysis of both the cements i.e. OPC and Calcium Aluminate Cement (CAC ISTRA-40) and SRM i.e. Fly-Ash (Bau Mineral KraftWerkstoffe) were also carried out to know the composition of powders.

Chemical Analysis	OPC	CAC	FA
SiO_2	19.61	2.58	54.94
Na_2O	0.00	0.00	0.00
MgO	2.03	0.72	1.03
Al_2O_3	4.89	40.03	27.18
K_2O	0.85	0.22	2.02
Fe_2O_3	2.62	15.93	9.26
SO_3	2.92	0.00	0.32
CaO	63.07	37.31	2.33
Particle size, d_{50} (μm)	13.09	17.85	18.59
Specific Surface Area, (cm^2/g)	3470	3290	9527

Table 2.3: Physical and Chemical Properties of Powders

3.4 Formulations:

Variant formulations were formed, with varying percentage of powder cement and SRMs, to avoid confusion a code is assigned to each formulation that indicate percentage of cement and any other SRM used, followed by type of cement and SRM used. A typical formulation is shown below:

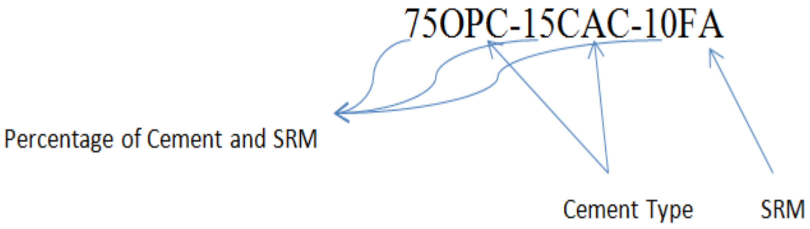


Figure 2.9: Formulation Syntax

3.4.1 Formulations used in our project:

1. A= 100OPC
2. B= 100CAC
3. C= 90OPC-10CAC
4. D= 85OPC-10CAC
5. E= 75OPC-15CAC-10FA

3.5 Blending Regime and Proportion of SL Paste Formulations.

The mixing was done using Hobart Mixer (2.9.1) of 5 L capacity. The constituents including cement, secondary raw material and powder super plasticizer were first manually mixed in closed plastic container in dry state. These were then fed into the bowl of mixer containing requisite mixing water. A slow mixing (145 rpm) was carried out for 15 seconds and then interior of bowl was cleaned. Thereafter, fast mixing (285 rpm) was done for 2minutes and 45seconds. The total mixing time was thus 3 minutes (180 seconds).

Figure 2.9.1: Hobart Mixer

Mixing was done to get target flow spread of 36±1 cms using SP. Mixing water was equal to the demand of the system.

3.6 Water Demand, Super Plasticizer Demand and Setting Times.

The estimation of water demand for different formulations is often initial step required in self-leveling paste system. WD is calculated as per European guidelines for self-leveling concrete [56]. Water demand of powder component is determined by taking cement and other SRMs with fixed ratios by mass as per formulation, and testing with Vicat apparatus.

As per thesis we investigate water demand and setting times at variable water temperature i.e. at 5, 23, 35°C. During testing ambient temperature and humidity were controlled at 20±1°C and 60±5% respectively. The super plasticizer amount required for a target flow of 36±1 [56] cms was obtained for all paste formulations using

Hagerman's mini slump cone. It had an upper diameter equal to 70 mm, a lower diameter equal to 100 mm with a height of 60 mm.

3.7 Self-Leveling Paste Flow:

The flow of self-leveling paste system was carried out by Hagerman's mini slump cone with the maximum dia of 10 cm, minimum of 7 cm and height of 6 cm. Super-plasticizer demands for paste obtained according to target spread of paste to 36±1 cm. T36 (Time required paste to flow 36cm dia) was noted. Through flow test one can get good idea about viscosity, segregation resistance and bleeding etc. in addition to deformability [57].

Figure 2.9.2: Hagerman Slump Diameter Measurement

3.8 Early Shrinkage.

To obtain the early shrinkage values, a modified version of German Classical shrinkage channel apparatus, dimensioned 4x6x25 cms integrated with computer was used for non-zeroed, with controlled ambient laboratory conditions at 20±1°C and relative humidity of 60%. Figure depicts the detail of the apparatus.

The tested samples were in covered conditions using adhesive polythene sheets. Reading started ten minutes after the addition of water to the constituents of SLP. The rear end is flexible while the front end was fixed, sensitive of a measuring of 0.30 microns displacement due to shrinkage or expansion of paste.

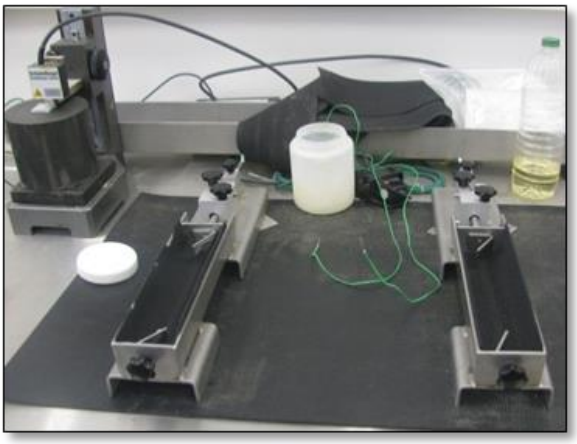


Figure 2.9.3: German modified shrinkage channel apparatus

3.9 Prism Casting &Strength Measurements.

Prisms for curing and strength measurement with 4x4x16cms dimensions were cast for different self-leveling paste as per DIN EN-196. The samples were placed and cured in an enclosed humid chamber for initial 24 hours at 90% relative humidity and 20±1°C. Then these were demoulded, weighed and put underwater in the curing tank after weighing and marking till specified ages of testing.

The samples were weighed in SSD condition at test ages to get the relative water absorption with reference to the internal water in specimen at the age of 24 hours. These were then tested for flexural and compression strength as per DIN EN 196-1. Samples were tested on displacement controlled machine (Figure 3.4) with following rates of loading:

(1) Rate of loading for Compression = 0.2 kN/sec

(2) Rate of loading for Flexure = 0.02kN/sec

For flexural strength, the average of 3 samples broken in flexure was noted at ADR Auto 250 machine and recorded as the strength of that sample at that prescribed age. For compressive strength, average of 6 samples was taken as strength of the sample.

Figure 2.9.4: (left) Prism Molds, (right) Samples tested in Dual Chamber

3.9.1 Calorimetry:

Calorimetry, together with fluidity and compressive strength measurements, has been exploited to investigate the hydration kinetics of several types of cement and SRMs in the presence of Mel-flux (super-plasticizers) for periods up to 24 hours.

For our research calorimetry was conducted parallel with prism casting for each paste formulation. After well blending with Hobart mixer, resulting paste diligently pour into crucibles covered by lids, account for special care to prevent unnecessary heat conduction. Each filled crucibles carried equal amount of paste by mass. Place those samples into calorimetric chamber for 24 hours, followed by sensitive computer integrated device obtain the heat values resulting from hydration reaction. Finally heat values plotted as calorimetric curve.

Figure 2.9.5: F-CAL 8000 Calorimeter

CHAPTER 4

RESULTS

4.1 Water Demand for SLP Formulations

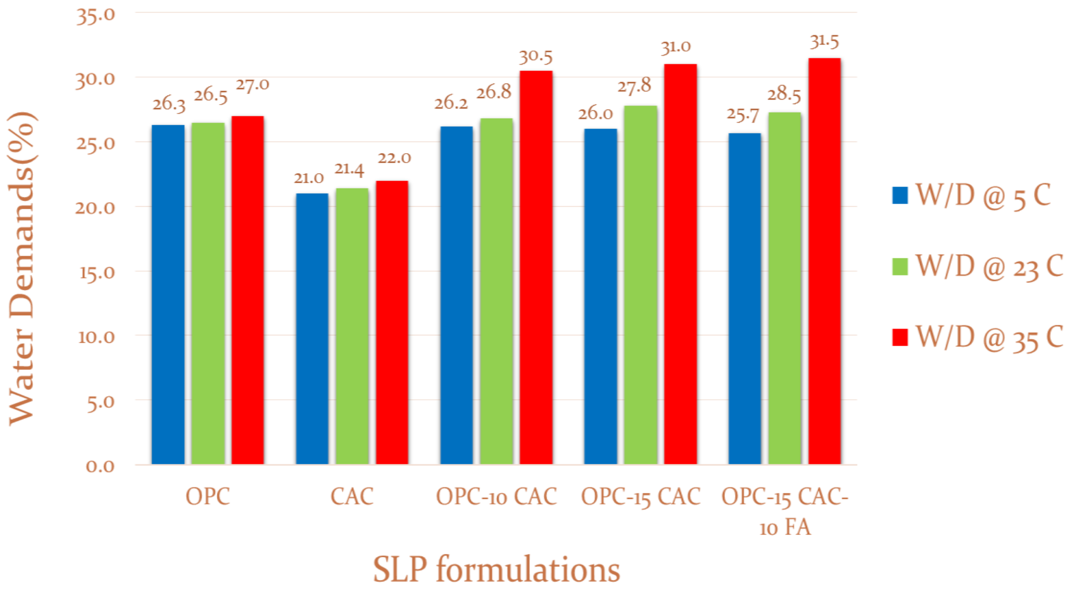


Figure 3.1: Water Demands

Water Demands have been measured on Standard Vicat Apparatus using EN-196-3. We saw that at 5 C, the water demand remained fairly constant on average_ 26% by mass of binder. At 23 C and onwards, the CAC started acting as an accelerator for OPC: As a result, more water was needed to allow the Vicat Rod to penetrate the paste. With increase in CAC replacement%, the acceleration effect increases and hence Water Demand increases. 100 CAC showed extremely low water demand compared to its counterparts. Like 100 OPC, 100 CAC wasn't sensitive to mixing water temperature and the change in water demand is only marginal.

4.2 Setting times:

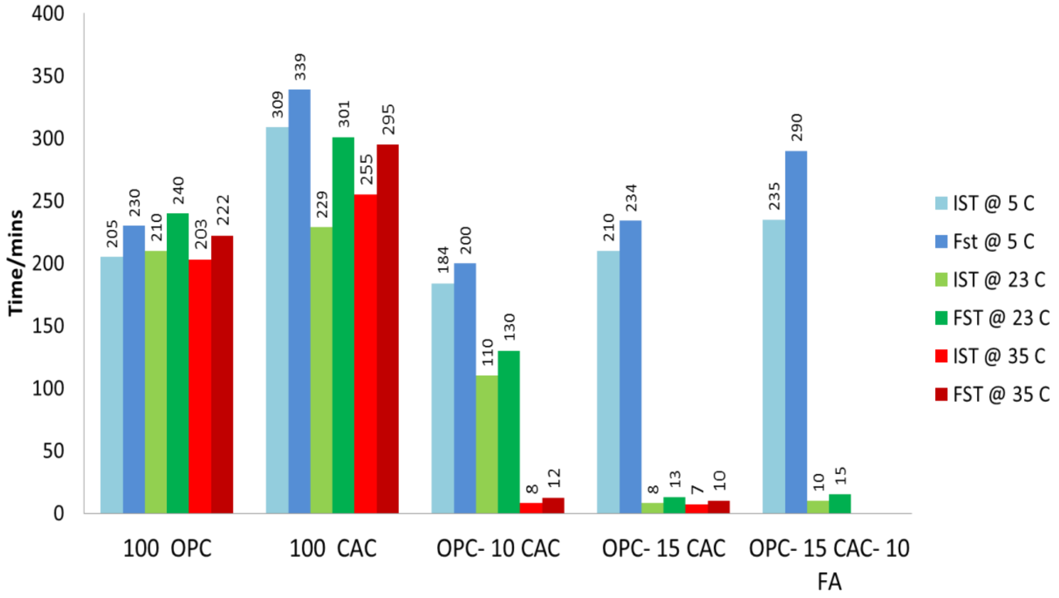


Figure 3.2: Setting times

Setting Times were measured according to EN-196-3. For 100 CAC/OPC, setting times weren't sensitive to temperature change and the difference was only marginal. For binary formulations, there was a distinct decrease in setting times with increased mixing water temperature. We were able to achieve setting under 10 minutes with relative ease simply my increasing mixing water temperature. The downside was a decrease in open time and workability of the paste which suffered at elevated temperatures. Rapid setting phenomenon can help explain such shortened Setting times: The gypsum from OPC reacts with aluminates from CAC to produce Ettringite which is known for its fast setting properties. At colder temperature, the fast setting phenomenon disappears completely and instead CAC hinders the precipitation of CSH gel. Setting times were delayed as the replacement % of CAC increased.

4.3 Super-Plasticizer Demand:

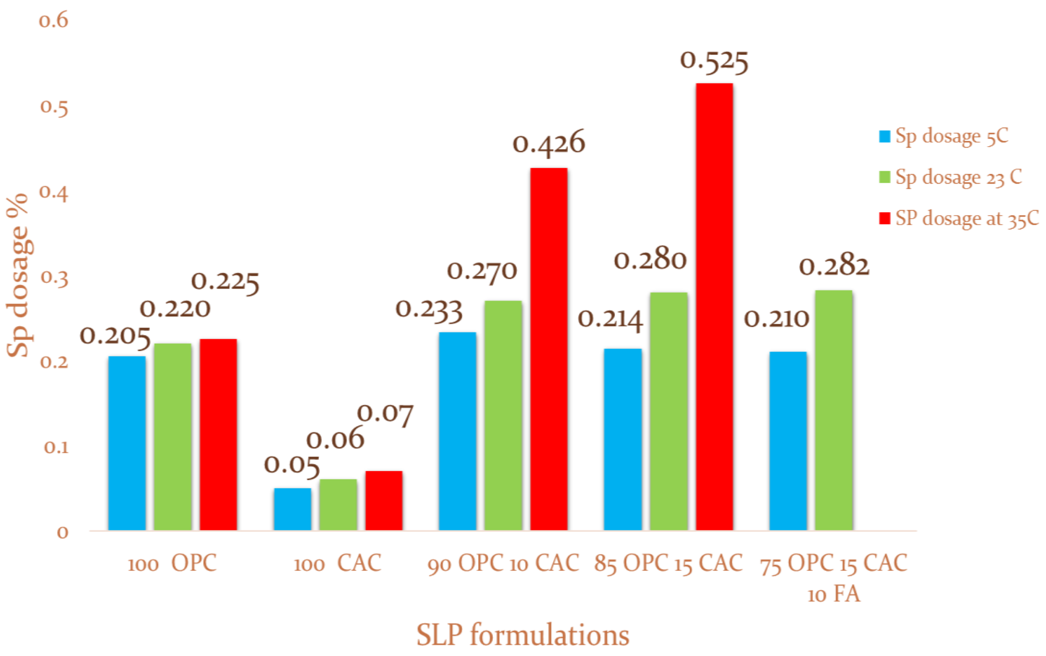


Figure 3.3: SP demands

SP dosage was measured for achieving 36cm spread on Hagerman Cone. 36 cm mark is considered adequate for self-levelling underlayments and hence all formulations were tested for SP dosage to achieve that value. It's been reported that SP dosage follows the trend of water demand. [58] A fairly consistent trend was seen with respect to SP demand. As temperature increased, SP demand increased owing to rapid stiffening of the paste. For binary formulations, this increase was more pronounced due to rapid setting effect caused by CAC. For the ternary formulation, the setting process was instant at 35◦C making it unsuitable for use as Self-Levelling formulation. At 23◦C and onwards, the SP dosage increased with increasing replacement % of CAC. However, at 5◦C, the rapid setting disappears and SP demand, in fact, decreases with increasing CAC replacement.

This is consistent with the fact that Melflux disperses CAC particles better owing to its better adsorption with PCE chains. This fact is further realized by looking at the SP dosage for 100 CAC which is abnormally low compared to its counterparts.

4.4 T36 for SLP formulations:

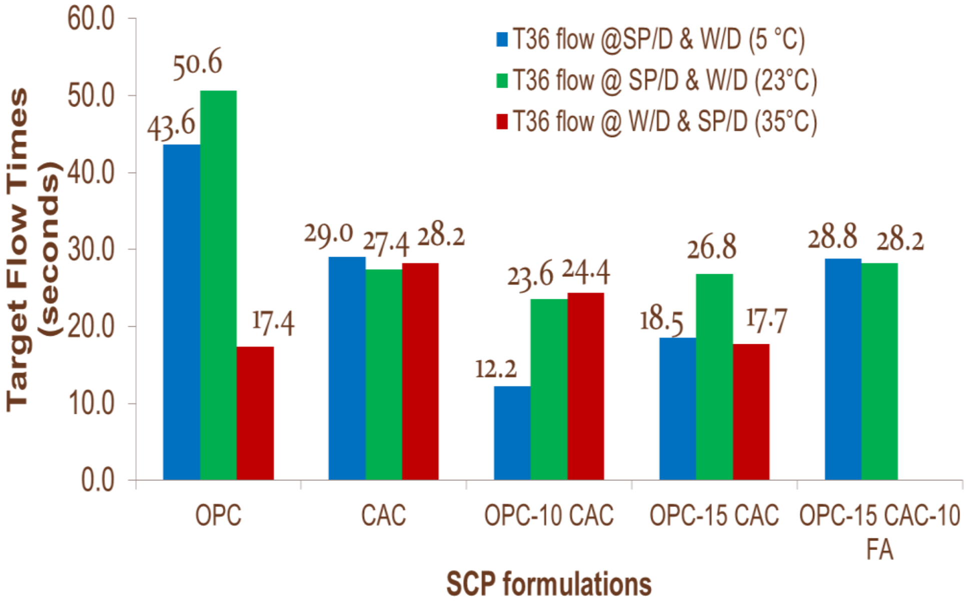


Figure 3.4: T36 times

The time to achieve 36 cm flow was recorded for all the formulations and plotted as shown. It can be seen that at elevated temperatures, Melflux performs better for 100 OPC but for colder temperatures, Melflux performance decreases significantly. Amongst Binary Formulations, 10 % replacement with CAC gave on average shortest times.

4.5 Compressive Strengths for SLP formulations:

Compressive Strengths were tested according to EN-196-1. Wet Curing regime was adopted and the tests performed at 1, 7, 28 days respectively. Mixing water temperatures adopted were 5˚C, 23˚C, 35˚C. The results are as under:

4.5.1 Compressive Strength for 5˚C:

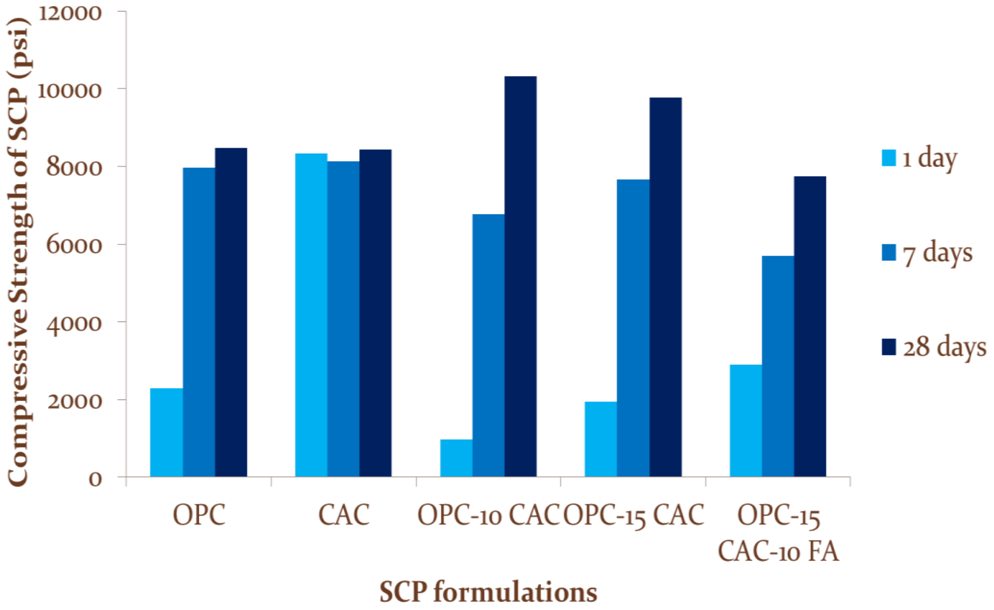


Figure 3.5: fc' @ 5˚C

4.5.2 Compressive Strengths for 23˚C:

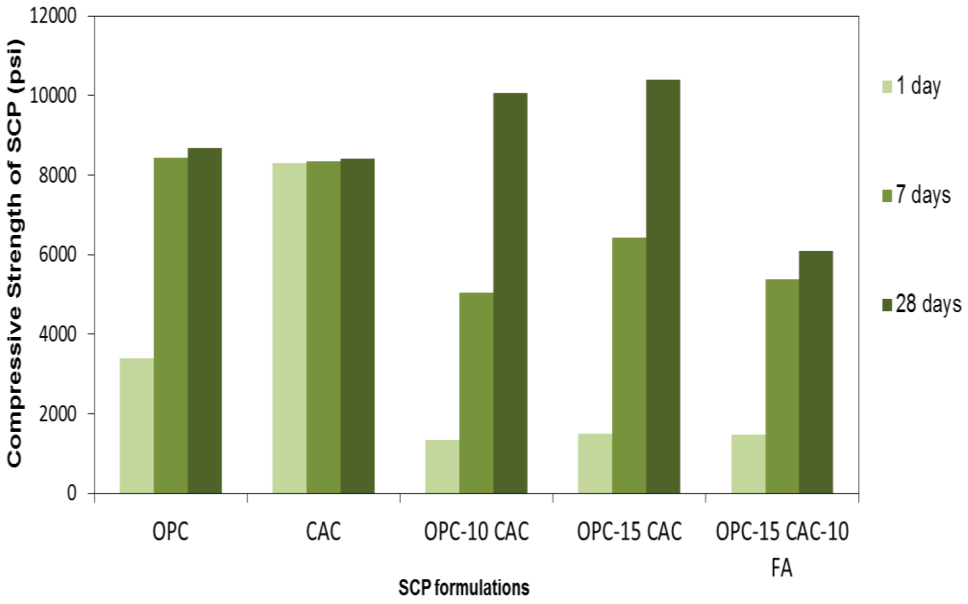


Figure 3.6: fc' @ 23˚C

4.5.3 Compressive Strengths at 35°C:

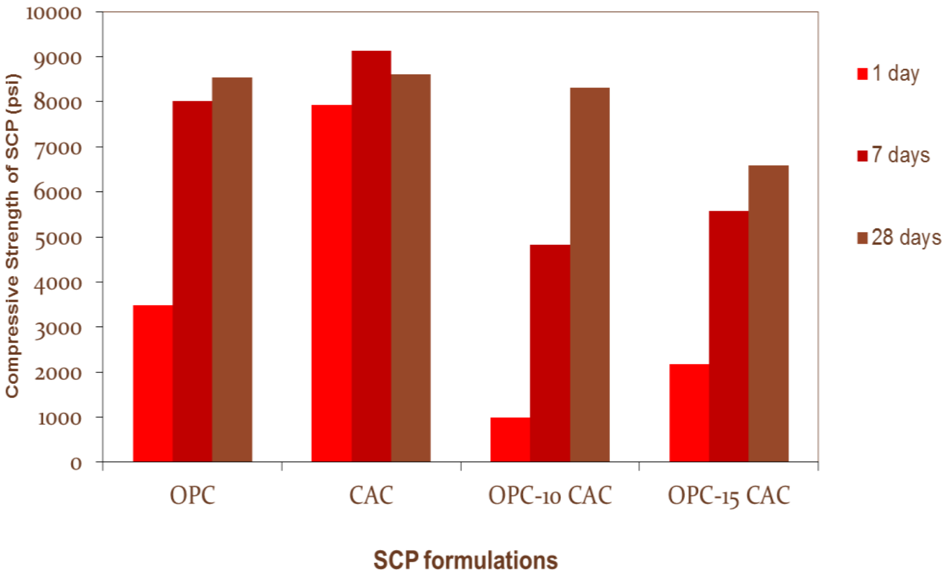


Figure 3.7: fc' @ 35°C

4.5.4 Comparison of 1 day Compressive Strengths at variable temperature:

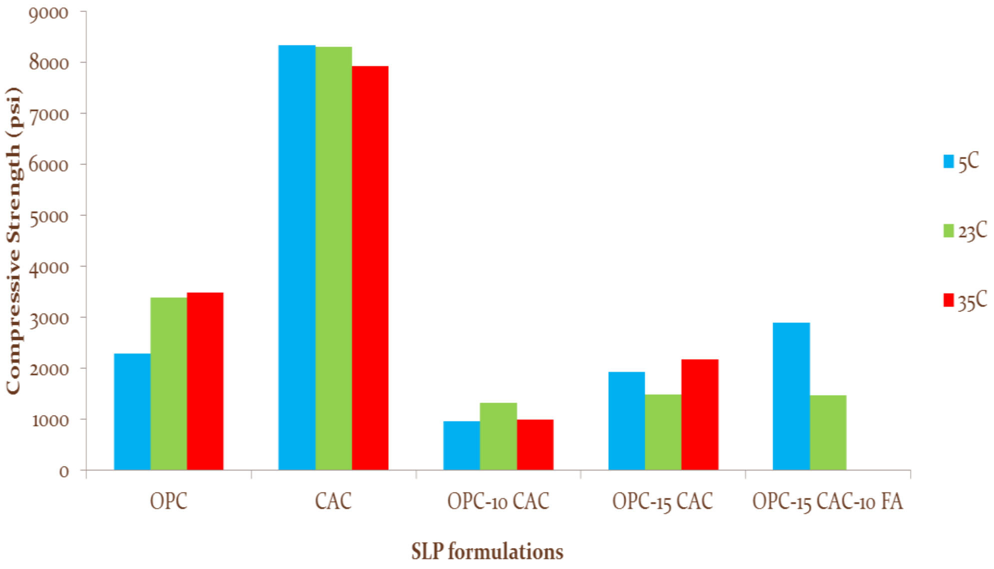


Figure 3.8: fc' 1 day @ variable temp

4.5.5 Comparison of 28 day Compressive Strengths at variable temperature:

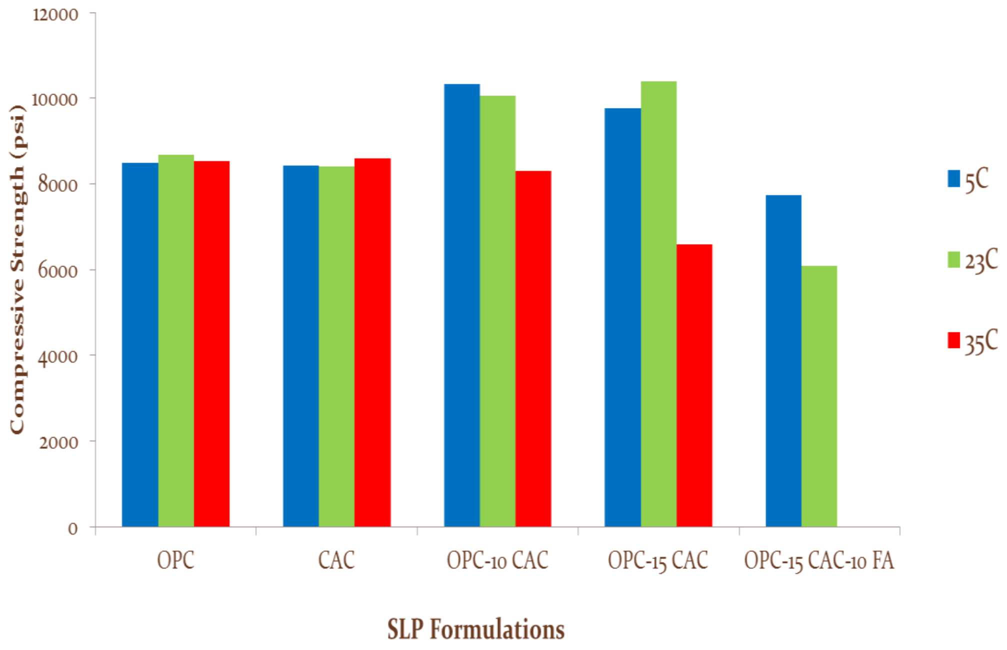


Figure 3.9: fc’ 28 day @ variable temp

4.5.6: Effect of w/c ratio on Compressive Strength @ 23˚C mixing water temperature:

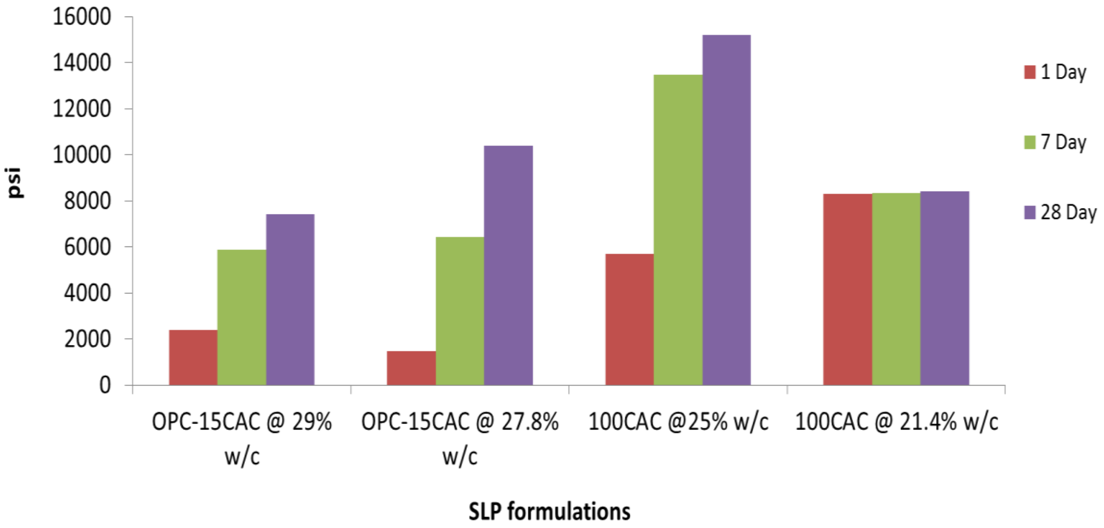


Figure 4.0: effect of w/c on fc’ @ 23˚C

4.6 Calorimetry:

Calorimetry was performed on F-Cal 8000 and software used for data logging was CalCommander. A total paste of 700 g was weighed prior to pouring in the calorimetric container and time noted from start of mixing. The mixing water temperatures were 5C, 23C, 35C. Logging time was under 30 hrs. The results are as under:

4.6.1 Calorimetry @ 5°C:

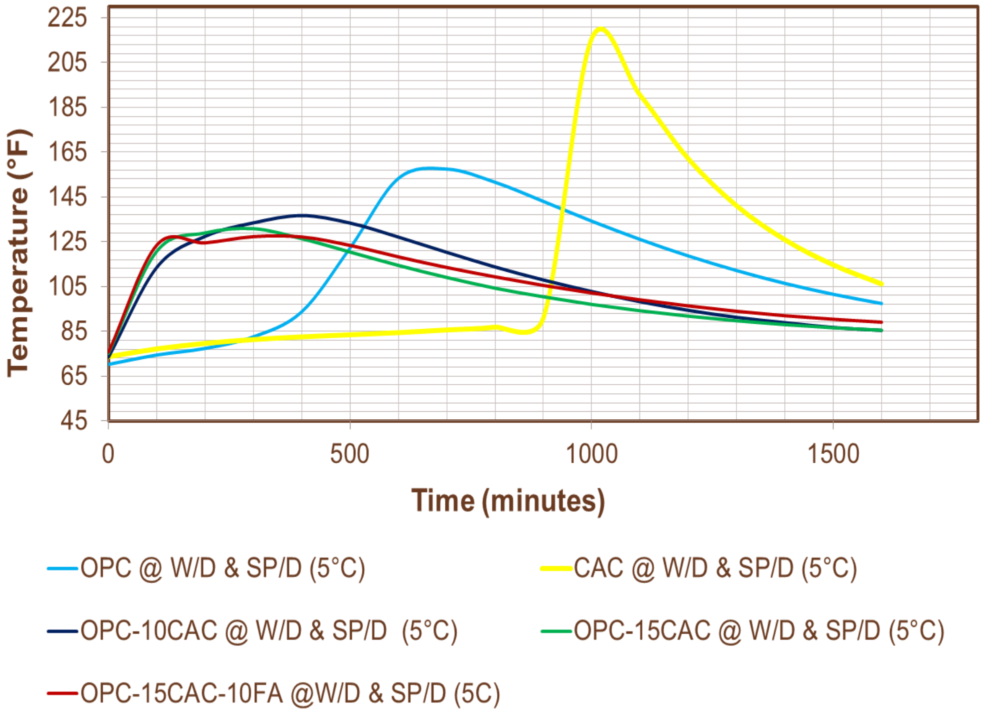


Figure 4.1: calorimetry @ 5°C

4.6.2 Calorimetry @ 23°C:

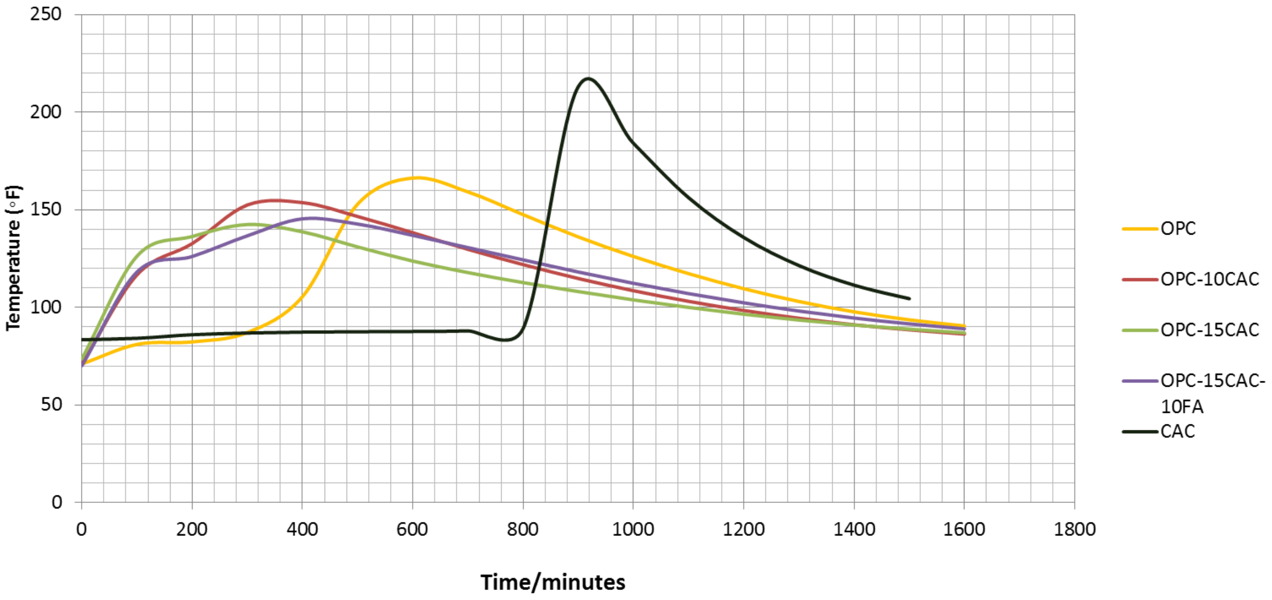


Figure 4.2: Calorimetry @ 23°C

4.6.3 Calorimetry @ 35°C:

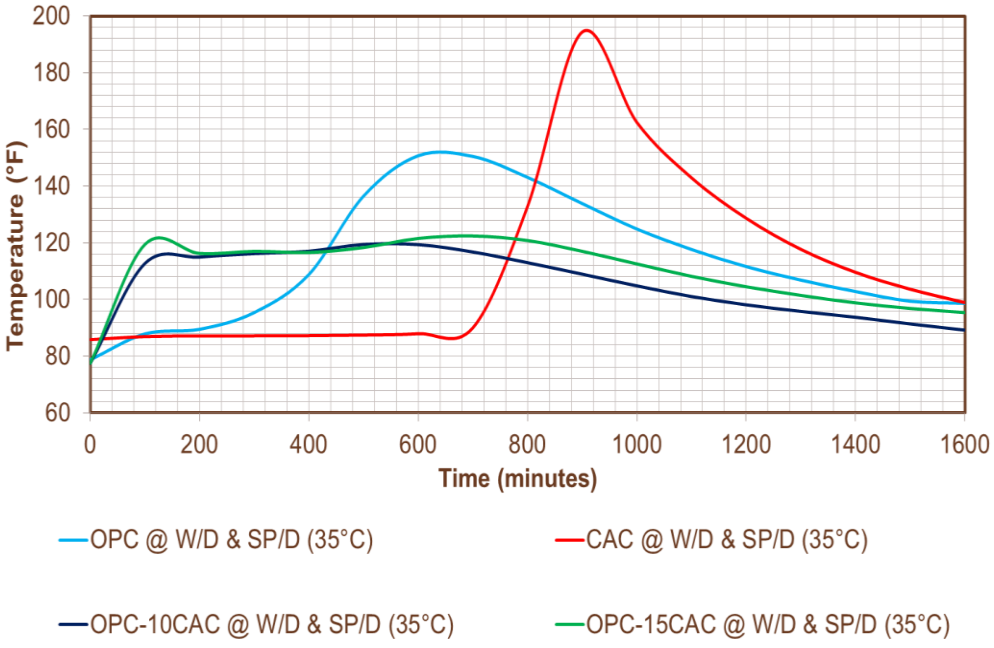


Figure 4.3: Calorimetry @ 35°C

4.6.4 Effect of mixing water temperature on Calorimetry:

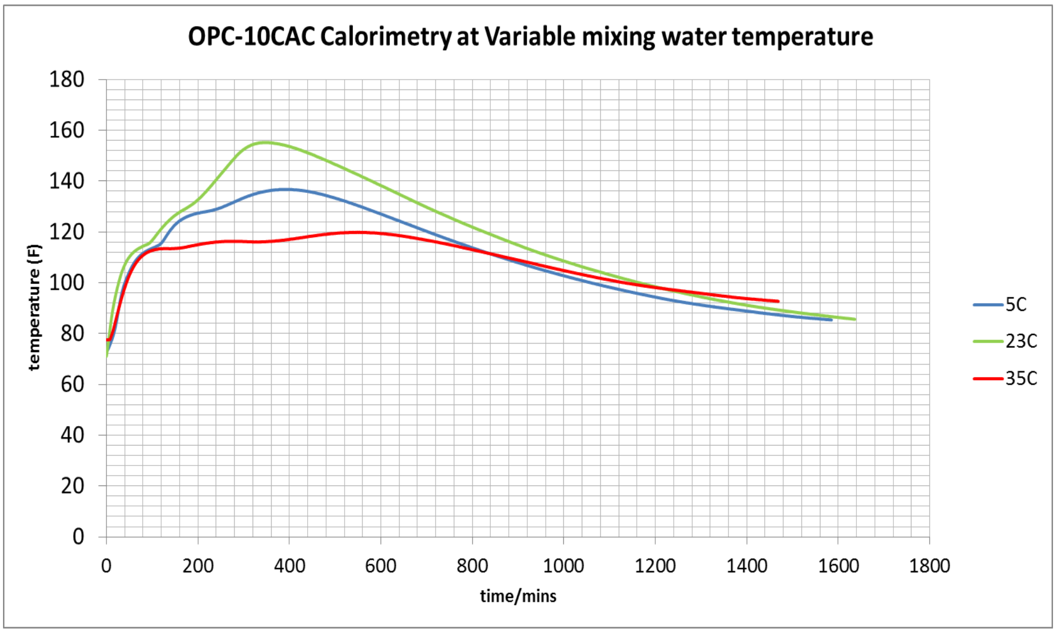


Figure 4.3.1: OPC-10CAC calorimetry at variable temp

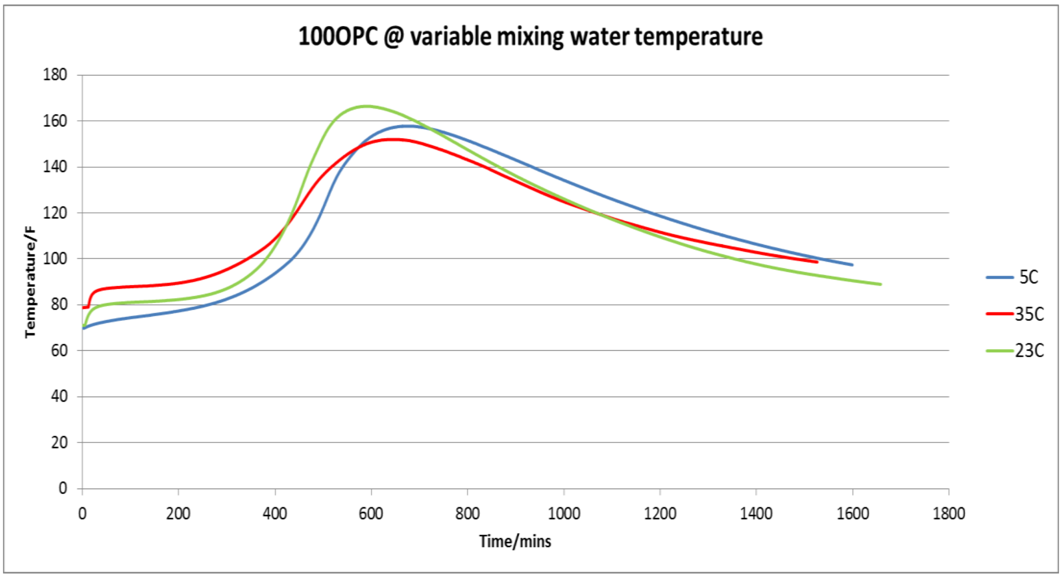


Figure 4.3.2: 100OPC calorimetry at variable temperature

4.7 Shrinkage Test Comparison with Calorimetry

Shrinkage test was performed on Shwindrinne Apparatus. Approximately 1200g of SLP was prepared according to its Water/SP demand and poured in the channels with plastic wrapped on top to reduce evaporation of surface moisture. Data was logged for a day and the results are shown as under: (in the plot, the shrinkage has been plotted on positive axis for easy comparison with calorimetric Graph.)

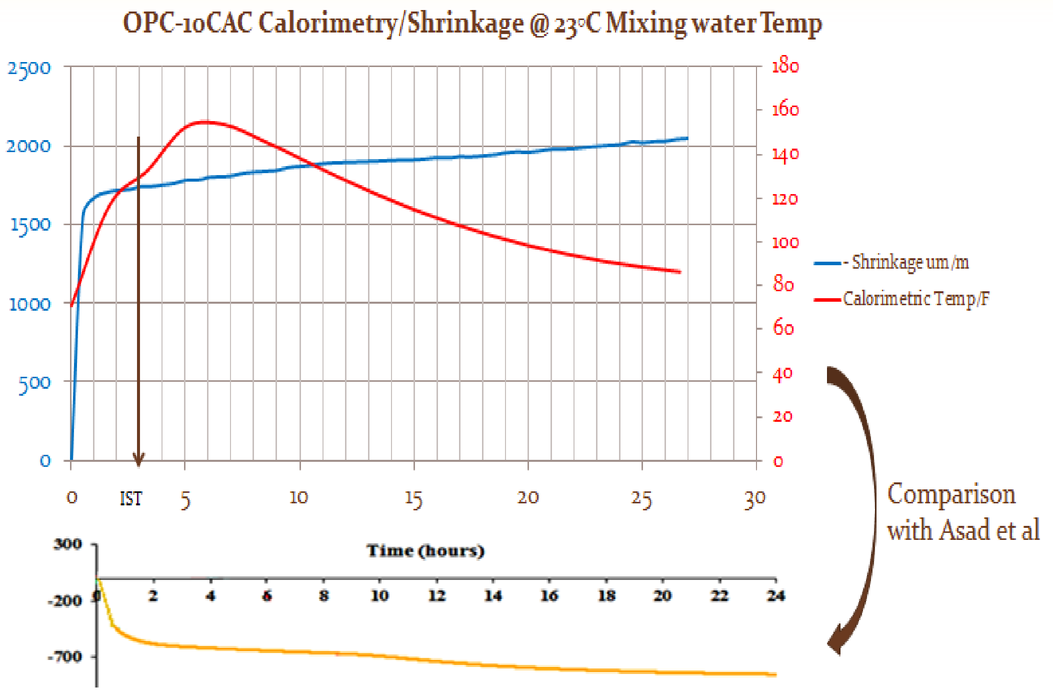


Figure 4.4: Shrinkage and calorimetry comparison

It can be clearly seen from above comparison that replacement of CAC with Fly Ash reduces early shrinkage considerably (Up to 65%). We also noticed that Initial Setting time coincides with roughly (1/3rd) of Peak Time. Peak time is the time from start of reaction to attainment of highest temperature. To put things in perspective:

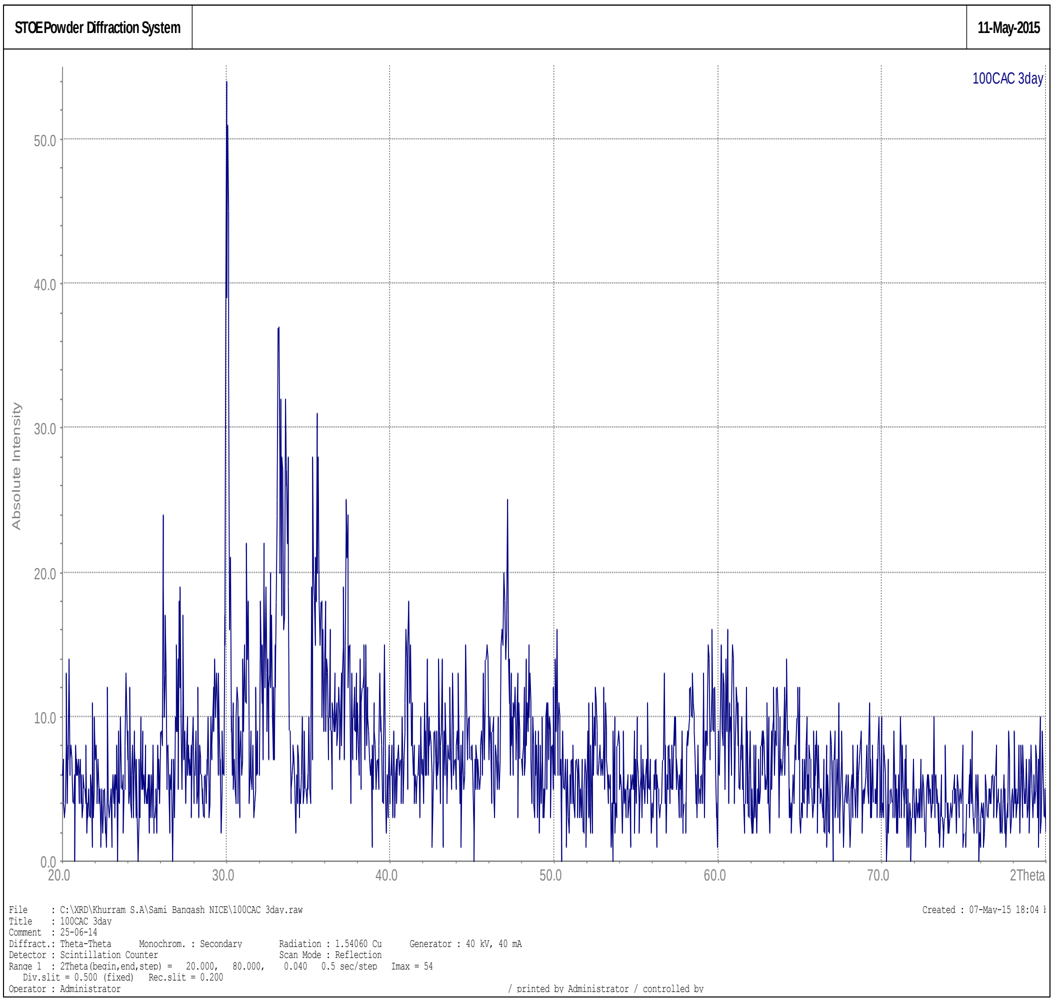
From Graph, peak time for Calorimetry is approximately = 6hrs.

6/3= 3 hrs= 180 mins. Our IST was 184 mins. So we can conclude that a shorter method of finding Initial Setting Time is to take peak time for calorimetry and take 1/3rd of it. That will roughly be the initial setting time.

4.8 XRD

XRD was also performed for OPC-10 CAC formulation. The samples were cured for 56 days and treated with Iso-Proponol and Acetone. Prior to testing, samples were grounded to fine powder. The results showed traces of Ettringite and C2AH6. However, there was a lot of noise in the samples and no definite pattern could be identified with utmost guarantee. Reasons for the noise need to be investigated. XRD diffractograms are shown as under:

4.8.1 100 CAC_3 day @ 23°C:



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File     : C:\XRD\Khurram S.A\Sami Bangash NICE\100CAC 3day.raw                                                              Created : 07-May-15 18:04
Title    : 100CAC 3day
Comment  : 25-06-14
Diffract.: Theta-Theta      Monochrom. : Secondary       Radiation : 1.54060 Cu      Generator : 40 kV, 40 mA
Detector : Scintillation Counter                         Scan Mode : Reflection
Range 1  : 2Theta(begin,end,step) =   20.000,   80.000,    0.040   0.5 sec/step   Imax = 54
   Div.slit = 0.500 (fixed)   Rec.slit = 0.200
Operator : Administrator                                                                  / printed by Administrator / controlled by
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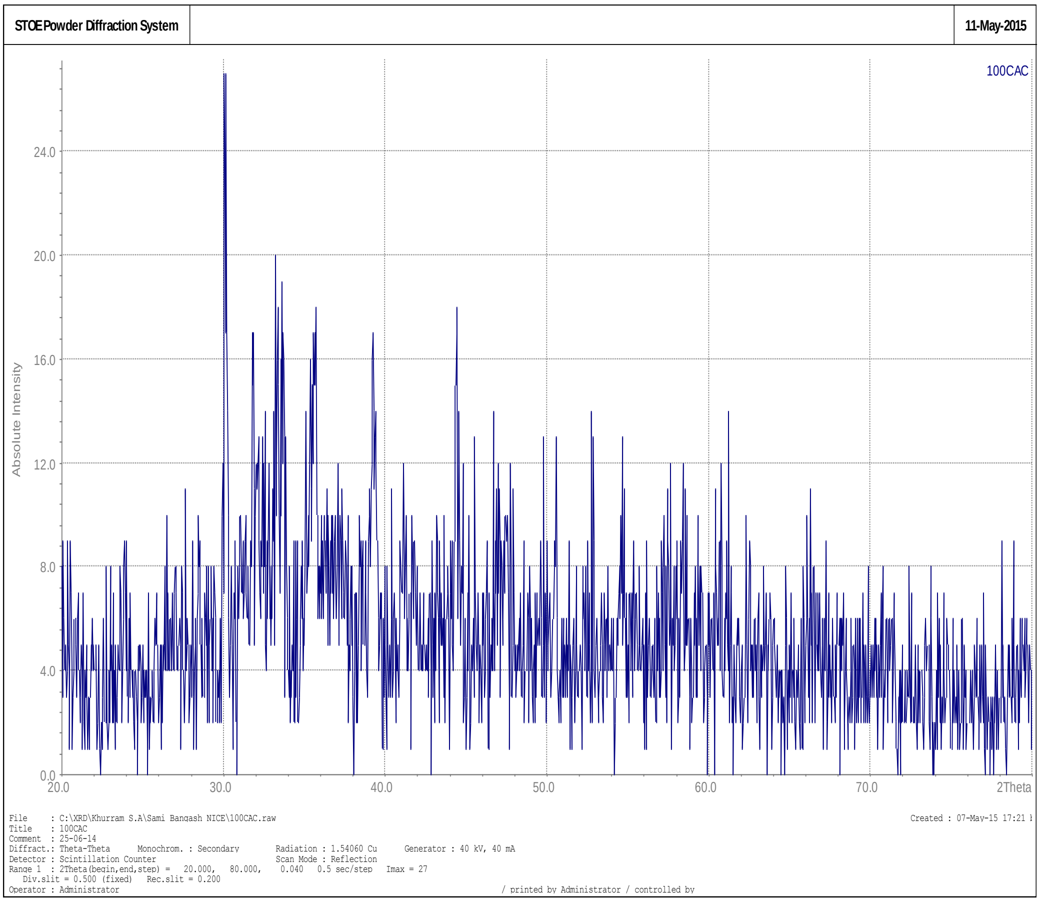
Figure 4.5: XRD 100CAC - 3 day @ 23°C

4.8.2 100 CAC_56 day @ 23°C:



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Comment  : 25-06-14
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Detector : Scintillation Counter                         Scan Mode : Reflection
Range 1  : 2Theta(begin,end,step) =   20.000,    80.000,    0.040    0.5 sec/step    Imax = 27
   Div.slit = 0.500 (fixed)    Rec.slit = 0.200
Operator : Administrator                                                    / printed by Administrator / controlled by
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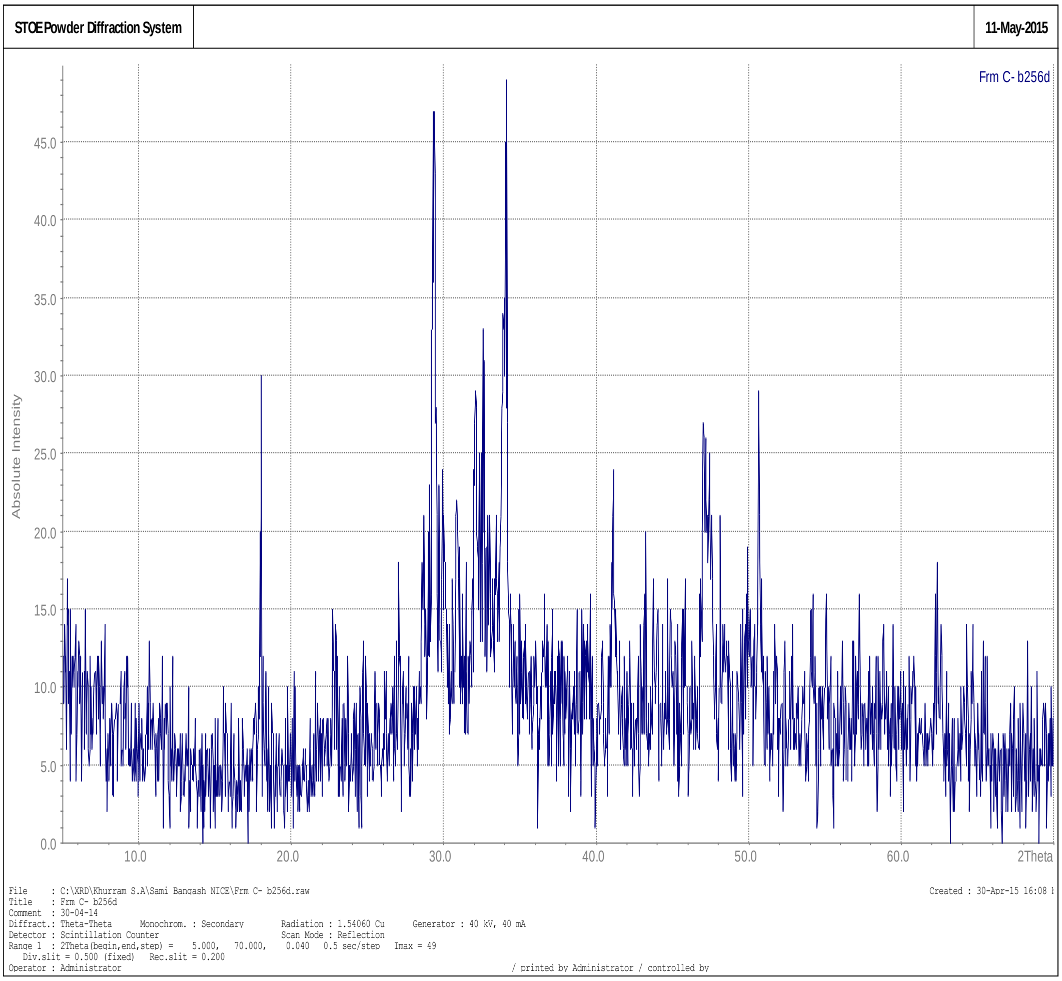
Figure 4.6: XRD 100 CAC - 56 day @ 23° C

4.8.3 OPC-10 CAC_56day @ 35°C:



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Comment  : 30-04-14
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Detector : Scintillation Counter                        Scan Mode : Reflection
Range 1  : 2Theta(begin,end,step) =    5.000,   70.000,    0.040   0.5 sec/step   Imax = 49
   Div.slit = 0.500 (fixed)   Rec.slit = 0.200
Operator : Administrator                                                           / printed by Administrator / controlled by
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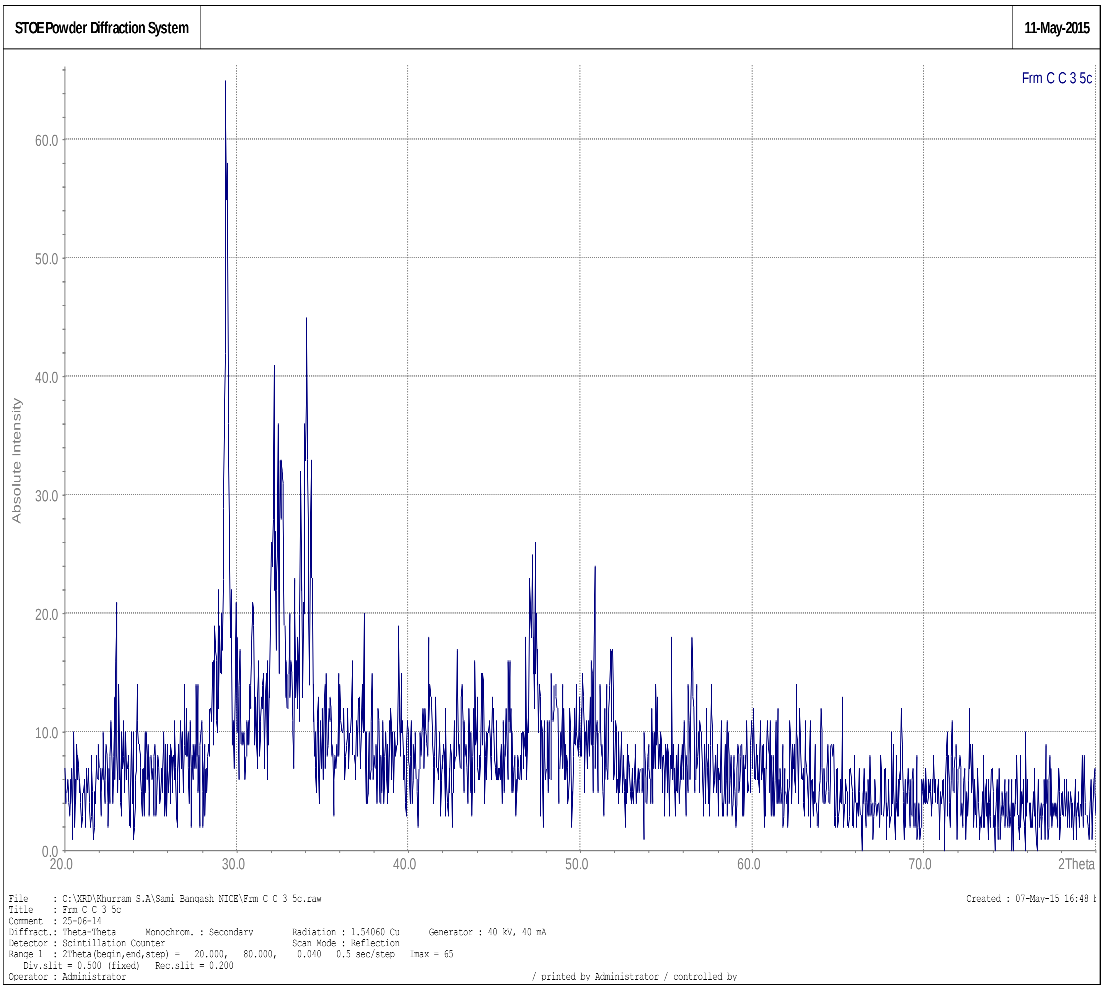
Figure 4.7: OPC-10CAC_56day @ 35°C

4.8.4 OPC-10 CAC_56day @ 5°C:



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Title    : Frm C C 3 5c
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Detector : Scintillation Counter                          Scan Mode : Reflection
Range 1  : 2Theta(begin,end,step) =   20.000,   80.000,    0.040   0.5 sec/step   Imax = 65
   Div.slit = 0.500 (fixed)    Rec.slit = 0.200
Operator : Administrator                                              / printed by Administrator / controlled by
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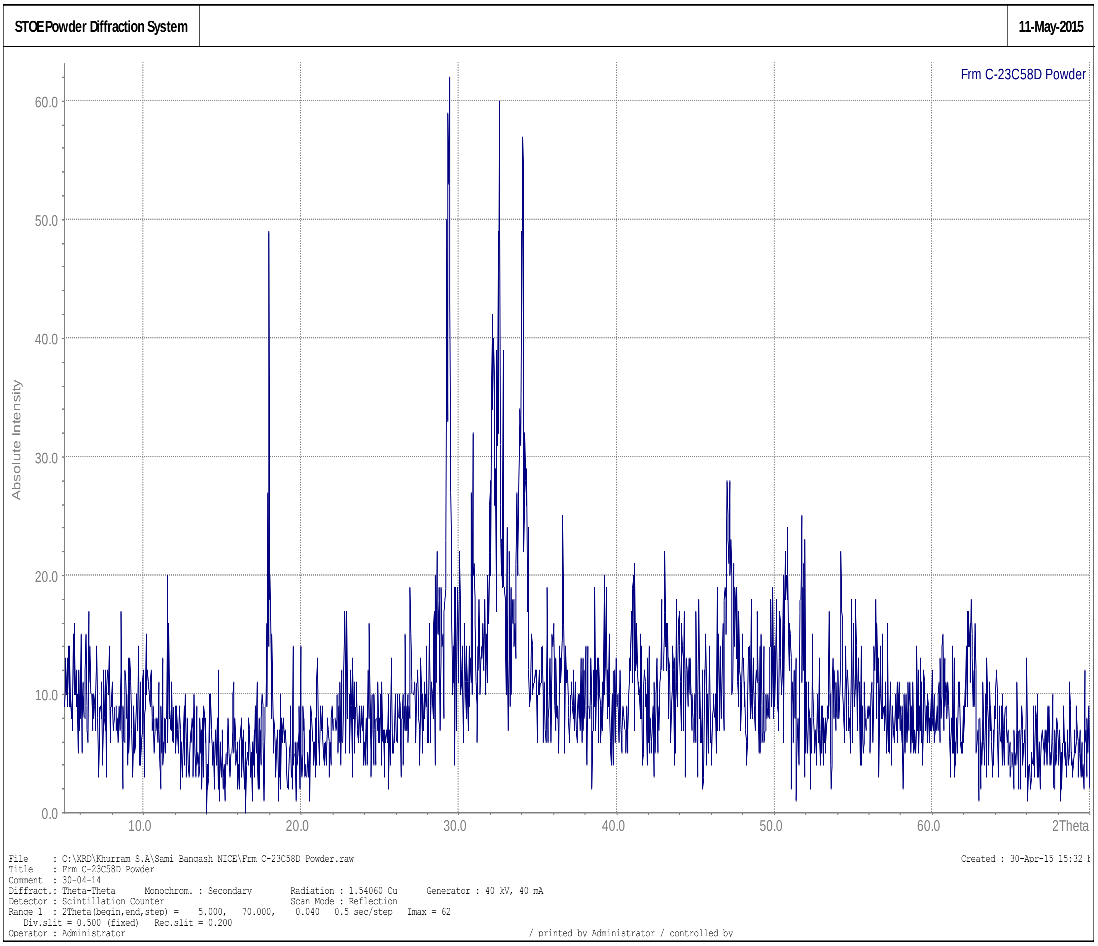
Figure 4.8: OPC-10CAC_56 day @ 5°C

4.8.5 OPC-10CAC _56 day @ 23°C:



STOE Powder Diffraction System

11-May-2015

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Detector : Scintillation Counter                          Scan Mode : Reflection
Range 1  : 2Theta(begin,end,step) =    5.000,   70.000,    0.040   0.5 sec/step   Imax = 62
   Div.slit = 0.500 (fixed)   Rec.slit = 0.200
Operator : Administrator                                                                   / printed by Administrator / controlled by
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Figure 4.9: OPC-10CAC_56 day @ 23°C

CHAPTER 5

DISCUSSION

5.1 Water Demands:

Water Demand for binary blends of CAC and OPC are considerably higher than that of OPC and CAC alone. This only applies for mixing water temperatures that are 23°C or higher. At colder temperatures, water demand doesn't change w.r.t to 100 CAC/100 OPC. At higher temperatures, more water is needed to prevent rapid flocculation of Ettringite and keep the paste workable enough for Vicat Apparatus. Also more water is required so as to compensate for water loss due to rapid heat evolution for binary blends. Water Demand for CAC is extremely low compared to its counterparts due to its larger particle size and hence smaller surface area. Also, the affinity for water for CAC appears to be much less compared to OPC which tends to flocculate more in water. This also explains other unusual characteristics that will be explained later. At 5°C mixing water temperature, rapid flocculation disappears and CAC particle helps reduce water demand. This is because CAC is thixotropic in nature and becomes very flow able under even the slightest agitation. This lends further support for CAC's low water demand. For the ternary blend, the addition of Fly-Ash increases the water demand at higher temperature compared to Control Formulation (100 CAC/100 OPC) but at colder temperature, Fly- Ash reduces water demand considerably. There is a great deal of ambiguity regarding Fly-Ash role in Water Demand: some literature suggests that it reduces water demand [59] owing to its glassy spherical shape and hydrophobic nature. While some suggest that it increases water demand due to its greater surface area. We observed both trends at varying temperatures. So we can conclude that Water Demand is highly temperature dependent for Formulations containing Fly-Ash.

5.2 Setting Times:

Setting times were performed for 3 temperature tiers; namely 5˚C, 23˚C, 35˚C. The results showed that for binary formulations, rapid setting phenomenon is enhanced exponentially_ to the point of becoming un-workable and too stiff to be called self-levelling formulations. At colder temperature, this rapid setting phenomenon disappears and dormant time is extended. For control formulations, the effect of mixing water temperatures wasn’t significant and only a marginal change was observed in setting times. It was observed that factors like ambient air temperature and humidity play a more significant role in setting times when compared to mixing water temperature. However, for formulations containing CAC replacement up to 15 %, the effect of elevated temperature was acceleratory. This suggests that higher temperatures favor Ettringite growth and dissolution of cement grains. For ternary formulation, the effect of Fly-Ash at higher temperature further enhanced the flocculation process_ resulting in instant flash set. This is because Fly-Ash is much higher in pozzolanic Oxides compared to OPC and higher temperature favors Hydraulic reaction to proceed ahead. At colder temperature, there isn’t sufficient energy for dissolution process to proceed and setting times are delayed. This delay was further enhanced with increasing replacement of OPC with CAC and Fly-Ash. This suggests that at colder temperatures, CAC particles inhibit the setting process by extending the induction period. At 23˚C, the Fly-Ash particles retard the setting times slightly compared to other formulations not containing Fly-Ash. This is confirmed in literature as well.

5.3 SP dosage and T36 Times:

It was observed that SP demand roughly follows a similar trend as Water Demand. For binary blends of CAC and OPC, the SP demand increases with increasing temperature. At higher temperature, more SP is required to break the flocculation and disperse cement grains. The mechanism for PCE based plasticizer requires contact with the anhydrous cement particle for effective repulsion to take place. But when hydration products start accumulating on cement particles faster due to higher temperature, more SP is required to break through that flocculation_ hence higher the demand. At colder temperature, the trend is reversed and CAC actually lowers the SP demand: this was evident with increasing CAC replacement. Fly-Ash also helped lower the SP demand at 5°C further due to its glassy spherical shape. For 100 CAC, the SP demand was unusually low compared to its counterparts. Hence we can conclude that PCE based plasticizers work extremely well with CAC. It's also effective for binary blends of CAC and OPC albeit at lower temperatures.

The T36 times were recorded for all SLP formulations. We can conclude from the results that 10% replacement with CAC roughly gives the shortest T36 times. With increasing CAC replacement in OPC, rapid setting introduces friction which enhances yield stress for the paste and increases T36 times. 100CAC also gave excellent flow response to Melflux® and proved to be an excellent formulation for rapid hardening and self-levelling applications. For binary blends, however, we noticed that immediately after placing in the mold, the paste became stiff very early. This is not desirable from self-levelling perspective. We suggest that retarders should be used to control the rapid setting phenomenon and obtain workable pastes with sufficient open time.

5.4 Compressive Strength:

We noticed some remarkable patterns in compressive strength analysis for CAC based formulations. Contrary to established trends that suggest that 28 day compressive strength for CAC-OPC blends is generally lower than their parent formulations (100 CAC/100 OPC) [60], we noticed that strengths for binary blends were generally higher than OPC and CAC (**see fig 4.1)**. CAC gave rapid hardening after 1 day that was equivalent to OPC's 28 day strength but after 1 day, CAC's strength increase was only marginal and not significant. 1 day strengths for binary blends were lower than 100 OPC which didn't bode well for using CAC-OPC blends as a rapid hardening formulation. It's recommended that we use accelerators to achieve rapid strength for our binary formulations. One aspect of our research was to investigate Conversion Phenomenon famously associated with CAC hydration.

We noticed no appreciable strength loss (associated with conversion phenomenon) at 5°C and 23°C. However, we did observe strength loss at 35°C when we compare CAC 7 day strength with 28 day. (**See Fig: 3.7).** From (**Fig: 3.9)**, we can conclude that binary formulations are a lot more sensitive to mixing water temperature than 100 OPC and 100 CAC formulations: 28 day strengths for binary formulations decrease rapidly with increasing temperature. This might be due to higher w/c ratio required at higher temperature which in turn increases porosity and reduces strength. Also, we observed that 28 day strength for binary formulations (with CAC replacement of OPC) are lesser than OPC and CAC formulations at 35°C mixing water temperature. This trend is confirmed by previous research. We also concluded that with increasing CAC replacement of OPC, initial 1 day strength increases but final 28 day strength decreases. This is due to the fact that CAC grains inhibit the growth of CSH gel due to poisoning effect. However the question (Why 28 day strength for binary formulations at 5°C and 23 °C is higher than OPC/CAC?) remains unclear. We noticed no appreciable difference in strength due to addition of Fly-Ash. In-fact lower 28 day strength was observed compared to other formulations. We can conclude that either Fly-Ash starts playing its strength enhancing role at 56 day onwards or the high replacement % of CAC is interfering with Fly-Ash pozzolanic activity.

We also studied the effect of w/c ratio on Compressive Strength for 100CAC and OPC-15CAC formulations (**see Fig: 4.0).** A surprising thing to note was CAC’s behavior to w/c ratio: Its 28 day strength at 25% w/c ratio was much higher compared at 21.4% w/c ratio. We suspect that at lower w/c ratio of 21.4% (which is also its Vicat Water Demand), the samples undergo severe dehydration due to high heat of hydration as seen from (**Fig: 4.1-4.3).** At higher w/c ratio of 25%, sufficient water is available to compensate for water lost due to evaporation. This explanation still needs to be investigated further since it doesn’t explain why 21.4% w/c ratio isn’t adequate despite being Water Demand value for 100CAC. For OPC-15CAC, the trend was predictable and more typical of cement formulations: At higher w/c ratio, 28 day strength decreases: This is in line with the fact that higher water content increases ‘pore size’ which reduces strength.

5.5 Calorimetry and Shrinkage:

Calorimetry graphs provide an insight of pozzolanic reactivity, setting times, and effect of admixtures added to cement. From the graphs plotted against 3 temperature tiers, we can conclude that dormant times are shortened significantly for binary formulations of OPC-CAC when compared to 100% OPC and 100% CAC. This indicates rapid Ettringite growth which is associated with rapid heat release. However the final peak temperature is lower compared to OPC and CAC. This means that CAC grains interfere with C_3S hydration of OPC which is responsible for peak gain. Also the addition of Fly-Ash reduces peak gain which proves the fact that Fly-Ash addition reduces heat of hydration which is beneficial from volumetric stability point of view. (**From fig 4.1-4.3)**, we can observe that time to peak temperature reduces with increasing CAC replacement. This is compatible with our findings regarding setting time: as CAC replacement increases, setting times are reduced. CAC showed an unusual delay in its dormant period. We conducted calorimetry on CAC repeatedly to confirm this unusual delay but still got the same result. This delay was followed by an extremely sharp increase in temperature. This sharp increase in heat loss coupled with very low water demand (21.4%) confirms that severe dehydration causes low strength in 100 CAC samples: there just isn't enough water or space available for hydration products to grow. This is further supported by literature which suggests that initial CAC hydration product (CAH_{10}) requires sufficient moisture to precipitate in the void spaces [60]. The question as to why there is a significant delay in CAC hydration couldn't be ascertained. It was also observed that as CAC replacement in OPC increases, the peak temperature attained also reduces. This is counter-intuitive at first but it indicates that Ettringite precipitated only contributes to initial heat release and not to the final temperature gain. This is proven by SEM pictures [60, 61] from research that indicates that Ettringite volume is far less compared to CSH gel hydrated. So we can conclude that the bulk of heat value is associated with CSH hydration. Looking at (**Fig: 4.3.1-4.3.2)** we found an unusual development: peak temperature attained for both formulations (100OPC and OPC-10CAC) was maximum @ 23°C while the final temperature gain was minimum @ 35°C. This was in fact observed with all other formulations as well. Higher mixing water temperature tends to flatten the peak gained while 23°C temperature appears to produce maximum heat gain.

Looking at (**Fig 4.4)** we can see a meaningful comparison of Calorimetric curves with Shrinkage curves. Initially we were of the opinion that point of sharp gradient change in shrinkage curve would coincide with time of maximum temperature gain but it wasn't the case. The shrinkage cutoff occurred much sooner compared to time of peak temperature gain. Maximum shrinkage (90%) occurs well before the initial setting time for the formulation. Also we compared our shrinkage values with results of [62] that used 5% replacement of CAC with Fly-Ash. The incorporation of Fly-Ash showed significant decrease in shrinkage (65% reduction) compared to our formulation (OPC-10CAC). This supports literature which suggests that use of Fly-Ash helps reduce shrinkage by lowering heat of hydration.

Also we were able to confirm the (1/3rd) rule of setting times for our formulations. (1/3rd) rule suggests that we take a third of time to maximum peak in calorimetry and that will give us a rough approximation of setting times. A calculation has been shown below (**Fig: 4.4)** as an illustration.

5.6 XRD:

XRD was performed on formulations (100CAC, OPC-10CAC) at variable mixing water temperatures and cured for 3day, 56 day respectively. The diffractograms confirmed the presence of Ettringite, CH, C2AH8 and CAH10. The absence of C3AH6 confirms results found in literature that suggest that much higher temperature 40°C are required for full conversion to take place. It means that much longer hydration times are required (years) before full strength degradation of (100CAC can take place.) It’s suggested that for future XRD experiments following instructions be followed:

- Avoid grinding the hydrated sample by kneading action. This destroys the crystal matrix and doesn’t give proper results.
- Heat sample in oven up to 50°C to remove moisture and place the powdered sample in air tight container.
- XRD machine settings to be followed are as under:
    - Division Slit= 0.5 (fixed)
    - Receiving Slit= 0.3
    - Scan Speed= 2sec/step. Where step = 0.04°

CONCLUSION

- As mixing water temperature increases, W/D for binary and singular blends increases.
- W/D also increases with increasing CAC replacement at elevated Temperatures.
- W/D decreases with increasing CAC replacement at 5°C.
- Fly-Ash only reduces W/D and SP demand at 5°C. At elevated temperature, its beneficial properties are reduced due to CAC dominance in the formulation.
- Setting Times are drastically reduced with increasing temperature (for binary formulations.
- Setting times and Water demand aren’t sensitive to mixing water temperature for singular blends of CAC and OPC.
- Fly-Ash retards setting times significantly at cold temperatures while at elevated temperatures its effect reduces.
- CAC doesn’t exhibit significant Conversion Process at given temperature ranges.
- Strength for binary blends of CAC-OPC is generally higher compared to OPC and CAC (especially cold mixing water temperatures.)
- Addition of Fly-Ash gave lower 28 day strength on account of high CAC replacement which interferes with CSH development.
- Binary Blends of OPC and CAC only show lower 28 day strength at 35°C when compared to 100 OPC and 100CAC.
- CAC shows massive strength gain at 25% w/c ratio compared to its original W/D of 21.4% @ 23°C mixing water temperature.
- As CAC replacement increases for the formulation, 28 day strength decreases.
- Fly-Ash helps reduce net shrinkage in ternary formulations and reduces heat of hydration released.
- Optimum blend for achieving fastest T36 times is OPC-10CAC.
- 23°C mixing water temperature gave maximum temperature peak for all formulations as seen from Calorimetric Curves.
- 1/3rd rule can be employed to find setting times in a fast and efficient manner; as explained in Discussion section.

RECOMMENDATIONS

- Use Citric Acid retarders for binary formulations to control the flash set and achieve workable pastes with sufficient open time.
- Control lab ambient air temperature and humidity to investigate the role of mixing water temperature more efficiently.
- Investigate the effect of GGBFS on CAC-OPC blends to help improve the durability, flash setting and strength gain.
- Investigate the effect of OPC grade on binary blends of CAC-OPC formulations.
- Increase the maximum temperature of mixing water to 40°C and extend the curing time to 56 days onwards to investigate the Conversion Phenomenon more clearly.
- Perform extensive XRD and SEM testing of CAC-OPC blends to better understand the transformations leading to rapid set.
- Investigate effect of mixing time in Hobart mixer for binary blends of CAC-OPC.

APPENDIX-A

Key:

OPC = Best way grade 43 (Farooqia)

CAC = Istra 40

T1= room temperature in degree Celsius

H= relative humidity of lab %

T2= initial mixing water temperature

T3=Paste temperature after mixing

Water Demand @ 23°C mixing water temperature:

Serial No.	**Formulations**	**W/d %**	**T1/◦C**	**H/%**	**T3/◦C**
1	**100 OPC**	26.5	17.2	44	23.6
2	**100 CAC**	21.4	21.4	54	21.5
3	**90 OPC 10 CAC**	26.8	20.5	43	26.3
4	**85 OPC 15 CAC**	27.8	18.6	53	26.4
5	**75 OPC 15 CAC 10 FA**	28.5	18.6	33	27.7

Water Demand @ 5°C mixing water Temperature:

Serial No.	Formulations	W/d %	T1/◦C	H/%	T3/◦C
1	**100 OPC**	26.3	17.4	49	17.7
2	**100 CAC**	21	20.2	54	18.4
3	**90 OPC 10 CAC**	26.4	20.8	55	17.5
4	**85 OPC 15 CAC**	26	18.5	53	16.8
5	**75 OPC 15 CAC 10 FA**	25.7	21.2	52	17.5

Water Demand @ 35°C mixing water temperature:

Serial No.	Formulations	W/d %	T1/◦C	H/%	T3/◦C
1	**100 OPC**	27	18.1	44	23.6
2	**100 CAC**	22	20.2	54	25.3
3	**90 OPC 10 CAC**	30.5	20.8	55	28.3
4	**85 OPC 15 CAC**	31	23.8	55	36.5
5	**75 OPC 15 CAC 10 FA**	30.5	23.8	55	31.4

APPENDIX-B

Setting Times:

Key:

OPC = Best way grade 43 (Farooqia)

CAC = Istra 40

T1= room temperature in degree Celsius

H= relative humidity of lab %

T2= initial mixing water temperature

T3=Paste temperature after mixing

23 degree mixing water temperature:

Serial No.	**Formulations**	**IST**	**FST**	**T1/◦C**	**H/%**	**T3/◦C**
1	**100 OPC**	210	240	18	44	22.9
2	**100 CAC**	229	301	21.2	54	21.4
3	**90 OPC 10 CAC**	110	130	20.5	43	26.3
4	**85 OPC 15 CAC**	8	13	18.6	53	26. 4
5	**75 OPC 15 CAC 10 FA**	10	15	18.6	33	23.2

5 degree mixing water temperature:

Serial No.	**Formulations**	**IST**	**FST**	**T1/◦C**	**H/%**	**T3/◦C**
1	**100 OPC**	205	230	17.4	49	17.7
2	**100 CAC**	309	339	20.2	54	18.4
3	**90 OPC 10 CAC**	184	200	20.8	55	17
4	**85 OPC 15 CAC**	210	234	18.2	51	16.2
5	**75 OPC 15 CAC 10 FA**	235	290	15.8	81	17.3

35 degree mixing water temperature:

Serial No.	**Formulations**	**IST**	**FST**	**T1/◦C**	**H/%**	**T3/◦C**
1	**100 OPC**	203	222	18	44	27.7
2	**100 CAC**	255	295	20.2	54	25.3
3	**90 OPC 10 CAC**	8	12	20.8	55	28.3
4	**85 OPC 15 CAC**	7	10	21.2	52	29.4

APPENDIX-C

Plasticizer Demand:

Key:

OPC = Best way grade 43 (Farooqia)

CAC = Istra 40

T1= room temperature in degree Celsius

H= relative humidity of lab %

T2= initial mixing water temperature

T3=Paste temperature after mixing

23 degree mixing water temperature:

Serial No.	**Formulations**	**SP %**	**T25/s**	**T30/s**	**T36/s**	**T1/◦C**	**H/%**	**T3/◦C**
1	**100 OPC**	0.22	2.69	7.99	50.6	18.2	51	24.1
2	**100 CAC**	0.06	2.6	10	27.4	24.3	32	23.6
3	**90 OPC 10 CAC**	0.27	2.4	5.6	23.6	22.2	64	27.8
4	**85 OPC 15 CAC**	0.28	1.5	5.4	26.8	20.2	52	25.1
5	**75 OPC 15 CAC 10 FA**	0.282	2	6.1	28.2	17.2	44	25.4

5 C mixing water temperature:

Serial No.	Formulations	SP %	T25/s	T30/s	T36/s	T1/◦C	H/%	T3/◦C
1	**100 OPC**	0.205	3.05	7.58	43.63	18.1	51	17.1
2	**100 CAC**	0.05	3	11	29	23.9	63	18.7
3	**90 OPC 10 CAC**	0.233	1.59	3.84	12.24	20.2	59	18.7
4	**85 OPC 15 CAC**	0.214	2.00	4.90	18.50	20.2	52	17.2
5	**75 OPC 15 CAC 10 FA**	0.21	2.08	5.70	28.79	15.8	75	19

35 C mixing water temperature:

Serial No.	Formulations	SP %	T25/s	T30/s	T36/s	T1/◦C	H/%	T3/◦C
1	**100 OPC**	0.225	1.62	4.10	17.42	20.2	44	28.8
2	**100 CAC**	0.07	0.5	2.1	28.2	23.9	63	27.7
3	**90 OPC 10 CAC**	0.426	1.80	4.90	24.40	20.2	59	32.2
4	**85 OPC 15 CAC**	0.525	1.14	2.63	17.74	23.8	55	31.2

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