Study of the Behavior of Secondary Raw Materials in Self- Compacting Cementitious System



Submitted By

Umar Maqsood	NUST-BE-CE-2010-188
Waleed Ali	NUST-BE-CE-2010-199
M. Faisal Sarwar	NUST-BE-CE-2010-100
Iftiaz Hussain	NUST-BE-CE-2010-58
Farjad Hassan	NUST-BE-CE-2010-42

BACHELORS IN CIVIL ENGINEERING

Year 2014

Project Advisor:

Prof. Dr. Syed Ali Rizwan

NUST Institute of Civil Engineering (NICE) School of Civil & Environmental Engineering (SCEE) National University of Sciences & Technology (NUST) This is to certify that

Thesis titled

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Umar Maqsood	NUST-BE-CE-2010-188
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Iftiaz Hussain	NUST-BE-CE-2010-58
Farjad Hassan	NUST-BE-CE-2010-42

Has been accepted towards the partial fulfillment

of

the requirements

for the award of degree of

Bachelor of Engineering in Civil Engineering

(Prof. Dr. Syed Ali Rizwan)

Head of Structural Engineering Department NUST Institute of Civil Engineering School of Civil Engineering and Environment

ACKNOWLEDGEMENTS

First and foremost, we would like to thank Almighty ALLAH, the most beneficent, the most merciful, for showering His countless blessings upon us and giving us the enough courage, strength, ability and dedication to complete this research project. Without His grace and blessings, we would not have achieved the desired results.

We express our profound gratitude to our project supervisor Professor Dr.-Ing Syed Ali Rizwan for always providing us with his generous support, kind encouragement and thorough guidance during the whole course of this project work. We are thankful to him for providing us with this opportunity to work with him on this project. We would also like to thank Associate Dean NICE Dr. Liaquat Ali khan for helping us with our materials testing.

We are extremely thankful to Principal SCME Dr. Muhammad Mujahid and Principal IESE Dr. Ishtiaq Ahmed Qazi for helping us obtain the results of Particle size analysis and X-Ray Fluorescence analysis. We also express our gratefulness to laboratory staff at SCEE, NUST for their full cooperation and helping us in carrying out our project work.

Finally, we are thankful to our parents and family members who encouraged us and prayed for our project success.

ABSTRACT

Self-compacting cementitious system (SCCS) is a modern concept introduced in the construction industry and is being used throughout the world. Self-compacting cementitious Systems have major applications in placements involving heavy and congested reinforcements such as tunnel linings, rafts and pile foundations, bridge piers, high rise buildings, pre-stressed concrete members, transportation structures and aircraft runways etc. SCCS has higher workability, provide uniform compaction throughout the placement area and hence higher durability and strength. These systems contain higher powder content and lower water to cement (w/c) ratio as compared to conventional concrete and thus provide a better packing density, ease of placement in heavily reinforced sections, less heat of hydration, volume stability, higher strength and enhanced durability. Secondary raw materials (SRM's) are used to replace cement contents to economize the manufacturing of concrete and to make an environment friendly concrete with improved microstructure.

This research project was undertaken to assess the feasibility of using various secondary raw materials including Marble Powder (MP), Bagasse Ash (BA), Hemi-hydrate (HH), Fly Ash (FA) and Brick Powder (BP). These materials are industrial by-products and were easily acquired. Various parameters including particle size distribution, chemical analysis using XRF, flow behavior, strength development and micro-structure were studied.

Flexural and compressive tests were performed on the paste samples using 10% replacements of all the secondary raw materials. All samples showed high strength development except brick powder. SRM, s were also used in blends, these blends also showed appreciable results. Fineness of SRM, s increases their surface area and thus increasing water demand to some extent. However, due to their smaller size they increase the packing density of the structure by adjusting themselves in the pores and acting as filler materials. Addition of super-plasticizers improved the workability by using their cement dispersion mechanism. They also ameliorated the durability and ease strength development. XRF results of few Secondary raw materials showed their pozzolanic nature because they contained CaO, SiO₂ and Fe₂O₃. Setting times and rate of strength development varied depending upon the type of material used in the sample. Mortar samples containing cement, SRM and sand in the proportion of 1:1:2 respectively were also tested using w/c ratio of 0.4. These mortar systems also exhibited high compressive strength.

Therefore, after carrying out careful study and detailed analysis of results it can be concluded that addition of secondary raw materials enhances both fresh and hardened state properties of cement based systems. In addition, use of blends further optimize the properties of self-compacting cementitious systems.

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CHAPTER 1 - INTRODUCTION

1.1 GENERAL:

Concrete is a composite material obtained by mixing binder, water, aggregates and chemical or mineral admixtures. Concrete is the most widely used construction material in the world and over the years concrete has become the material of choice for construction because of its economy, durability and ability to cast into any shape. The process of concreting involves heavy compaction by skilled labor. Inability to compact the concrete or lack of skill of labor may reduce the quality of concrete and harm its durability. One solution for the achievement of durable concrete structures independent of the quality of construction work is the use of self-compacting concrete.

1.2 Self-Compacting Concrete:

Self-compacting concrete (SCC) as defined by ACI 237R – 07 as "a highly flow able, non-segregating concrete that can spread into place, fill the formwork, and encapsulate the reinforcement without any mechanical consolidation". Self-compacting concrete (SCC) is a flowing concrete mixture which can be compacted into every corner of a formwork, purely by means of its own weight and without the need for vibrating compaction, filling forms completely and producing a void free mass. Another advantage of SCC is that time required for concreting is incredibly reduced as compared to normal concrete. On disadvantage of self-compacting concrete is the increased cost as compared to normal concrete but for larger projects like multistory buildings, roads, dams etc. The cost of self-compacting concrete is reduced due to reduced required time and less manpower required. Mechanical or by hand vibration is required for simple concrete, which may result in different settlement and different durability. The necessity of this type of concrete 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 [1]. Self-compacting concrete is presently used due to its obvious advantage and is described as a milestone in the modern concrete technology. There are a lot of site conditions and working limitations in construction industry that makes self-compacting concrete a better alternative to conventional high slump concrete. Most general are cost savings or performance enhancement of concrete. SSC has also gained popularity in recent years because of its incorporation of secondary raw materials.

1.2.1 Chemical Admixtures:

In self-compacting concrete, super-plasticizers are used as chemical admixtures. Two super-plasticizers used in project were Melflux 2651 F powder and Glenium 51 liquid of BASF. **Melflux** is a free flowing, spray dried powder of modified poly-carboxlic ether. Melflux is specially optimized for plasticization and water reduction of cementitious construction materials. It provides an excellent development of early strength. **GLENIUM** is poly-carboxylic ether based,

high range water reducing new generation super-plasticizer concrete admixture working with zero energy system developed for precast industry that needs high early and final strengths and durability.

1.3 Secondary Raw Materials:

Secondary Raw Materials are introduced in SCC systems in order to balance economy Secondary raw materials are industrial waste materials that are very fine and their size is usually lesser than that of cement.. Secondary raw materials are used in as a replacement of cement in self-compacting cementitious systems. SRMs are mostly inorganic materials that may also have pozzolanic. They are usually very fine-grained materials, which are added to the concrete mix to improve the fresh & hardened properties of concrete (mineral admixtures), [2], or as a partial replacement for Portland cement as in case of blended cements. As per ASTM C 125, a Pozzolanic material may be defined as "A siliceous or siliceous and aluminous material, which in itself possesses little or no cementitious value but will, in finely divided form and in the presence of moisture, chemically react with calcium hydroxide (CH) produced by cement hydration at ordinary temperatures to form compounds possessing cementitious properties" [3].Secondary raw materials are much smaller in size than cement used in concrete. They fill the voids left by cement increasing density of concrete thus increasing its strength. Moreover SRM's also show some late pozzolanic activity. SRM's used are Bagasse ash **BA**, Hemihydrate **HH**, Marble powder **MP**, Brick powder **BP** and Fly ash **FA**.

1.3.1 Bagasse Ash (BA):

Sugar cane bagasse is a fibrous material that remains after sugarcane are crushed to extract their juice in sugarcane industry. It is used worldwide as fuel at industrial scale. Its combustion yields high amount of burnt material (bagasse ash) that is rich in oxides of silicon. Particle structure of BA indicates it to be a porous material due to which it is considered to be a difficult material to be used as SRM in cement based materials.

1.3.2 Fly Ash (FA):

Fly ash is one of the residues produced during the combustion of coal in industry. Fly ash comprises of the particles that rise with the flue gases during industrial combustion of coal. Ash which does not rise is termed as bottom ash. It generally contains higher quantity of CaO and Fe₂O₃.

1.3.3 Marble powder (MP):

Marble powder or marble dust is crushed or ground marble particles. Characterized by its fine powdery texture can be used as a SRM in concrete. Very small size of marble powder particles increases the surface area thus increases the water requirement, so its addition as secondary raw material in SCP and SCM would increase strength and durability due to increased packing density of the system.

1.3.4 Hemihydrate (HH):

Powder gypsum (CaSO₄.2H₂O) or dehydrate is one of the earliest building materials. It owes its popularity to its ease of fabrication, environmental friendliness, fire resistance and low price. The CaSO₄.H₂O system is composed of five solid phases: dehydrate, hemihydrates, anhydrate I, anhydrate II and anhydrate III. Among them hemihydrates is usually used to produce gypsum plasterboard [R]. The hemihydrate initially increases the setting time of the system and plays role in delayed strength development.

1.3.5 Brick Powder (BP):

Brick powder or calcite clay is a very fine crush of brick aggregate. It is a waste material obtained from brick kilns. Brick is produced in a very large amount throughout the country which results in tons of waste material like brick dust and flakes of bricks. Waste bricks (calcite clay) are an artificial pozzolanic which can be hydrated in the presence of Ca(OH)2[4].

1.4 Experimental Techniques:

Some of the experimental techniques and methods to study of the behavior of SRMs in SCP are as follow:

1.4.1 Calorimetery:

Cement hydration is an exothermic reaction taking place in a series of stages. Calorimetery is the best procedure for measuring heat of hydration in all the stages. Determination of heat of hydration is very important because it affects almost all the properties of concrete including workability, setting times, strength development rates and pore structure development. Calorimetery is the technique used to measure the heat production of the cement paste with time and evaluate the hydration kinetics.

1.4.2 Scanning Electron Microscopy (SEM):

Scanning Electron Microscopy (SEM) is used for the investigation of microstructure morphology and the chemical composition. Scanning electron microscopy provides several advantages for characterization of concrete, cement, aggregate and secondary raw materials microstructure. SEM gives a very clear and fine image of concrete at very large magnifications.

1.4.3 Mercury Intrusion Porosimetry (MIP):

It is an experimental technique used for assessing the distribution of pore sizes in cementitious systems. Measurement of porosity in cement paste systems as well as concrete is done through MIP. Results obtained from MIP are affected by various factors which include the method adopted for sampling, condition of sample, sample mass and dimension used, pressure application rate.

1.4.4 Particle Size Distribution:

A distribution of particle sizes or particle size distribution (PSD) is a fundamental characteristic of cement powder and secondary raw materials. Accurate and precise results of particle size distribution are needed in computational modelling of hydration process and predict the performance of cementitious materials. Laser Diffraction Particle Size Distribution Analyzer is used to measure the particle size of cement and secondary raw materials.

1.4.5 X-Ray Fluorescence (XRF):

Chemical analysis of cement and secondary raw materials is done using X-ray fluorescence (XRF). XRF is an experimental technique in which emission of characteristic X-rays from a material takes place. XRF gives the complete chemical analysis of a sample giving the quantities of all the chemical compounds or elements present in a sample.

1.4.6 X-Ray Diffraction (XRD):

X-Ray Diffraction is an experimental technique used for detection and quantification of crystalline materials. The working procedure of X-Ray Diffraction involves a scattering process in which X-Rays are dispersed by electrons present in the materials without changing wavelength of X-Rays. This phenomenon produces a diffraction pattern that is a fundamental physical property of every substance. It gives quick identification and comprehensive explanation of micro-structure.

1.5 Objectives of Research:

The objective of the study was "to study the comparative response of various secondary raw materials **SRMs** in self-compacting paste **SCP** and self-compacting mortar **SCM**". The SRMs used in the study was **Fly ash (FA)**, **Bagasse Ash (BA)**, **Hemihydrate (HH)**, **Marble Powder (MP)** and **Brick Powder (BP)**. The aim of the research was to study the response of self-compacting cementitious systems using 10% replacement of secondary raw materials (SRMs) and using the blends of materials for water demand (WD), super plasticizer **(SP)** demand, setting time, flow measurements, Calorimetery and strength development.

CHAPTER 2: LITERATURE REVIEW

2.1 Self Compacting Cementitious systems:

This research thesis focuses on Self-compacting paste systems (SCP) and self-compacting mortar (SCM). These two are important components of self-compacting cementitious systems and they lead to self-compacting concrete (SCC) which is the primary objective of the research because self-compacting concrete (SCC) is the need of the today's construction industry and it is only possible once self-compacting paste (SCP) and self-compacting mortar (SCM) are optimized.

2.1.1 Development History:

Development of Self compacting cementitious systems (SCCS) originated in 1980's in Japan. In mid-1980's Japanese construction industry was facing some serious challenges related to durability and serviceability requirements of the concrete structures. To make durable concrete structures proper vibration of concrete is required to place it at placements where heavy and overcrowded reinforcement is present. These places include heavily reinforced columns, deep foundations, tunnel linings, piers of bridge etc. Skilled labor is needed to compact the concrete and to place it in such highly reinforced zones. But Japanese construction industry faced a severe challenge of shortage of skilled labor force capable of operating mechanical devices to place and compact concrete [5]. Lack of uniform compaction resulted in differential compaction and hence loss in durability. Thus, there was an immediate need for a type of concrete that would not require human efforts for its compaction and placement.

This whole scenario led to the development of self-compacting concrete and Professor Hajime Okamura was the first to give concept about it in 1986. Later on, in 1988 the first prototype was developed by Professor Ozawa at university of Tokyo [6]. Professor Okamura studied its properties in detail and named it "High Performance Concrete" but Professor Aticin had already used this term for concrete with low water to cement ratio and high durability [7]. So then Okamura adopted "Self-compacting high performance concrete" for this type of concrete [6]. Through invention of self-compacting concrete, the need for skilled labor was considerably reduced and concrete could be placed at any area without the need of any mechanical device. By early 1990, Japan became successful in implementing this type of concrete in tis construction industry that didn't require any manual vibration; instead it flowed under its own weight. This new technology presented economic and environmental benefits over traditional methods of placement of concrete. SCC provided better travel rates of concrete placement, smooth and easy flow of concrete around heavy and dense reinforcement, faster construction and less noise due to the absence of mechanical devices.

Self-compacting concrete has been successfully used in various construction projects since its invention. It was first used in the construction of a building in Japan in June 1990. Later in 1991, it was then used in the construction of the towers of Shin-kiba Ohashi Bridge (Shown in Fig. 2.1). SCC was also used to build the anchorages of Akashi Kaikyo Bridge in 1998 (Shown in Fig. 2.2).

This bridge had the longest span of 1991 meters in the world [5]. However, very less literature was available on self-compacting concrete in in early 1990's in Japan as this technology was not shared by Japanese companies to keep a commercial and economic advantage over their competitors.

Recognizing the importance of self-compacting concrete ACI formed a committee to further research purposes and study of SCC systems. This committee was named as "ACI 237R–07 Self Consolidating Concrete" [8]. Research was conducted on SCC in Europe in early 1990's and Sweden was the first country to study the properties of SCC and work on it. With the introduction of third generation polycarboxylate ether based super plasticizers, SCC systems became the center of attraction of European researchers and they started to analyze their physical and chemical properties. The idea of SCC spread further to United States and production of self-consolidating concrete touched one million cubic meter landmark in 2002 in North America [9]. Many professional organizations like American society for testing and materials (ASTM), American concrete Institute (ACI) and Precast/ Prestressed Concrete Institute (PCI) started to use SCC systems. Many seminar and conferences were held to spread awareness for this modern and efficient technology. American society for testing and materials (ASTM) formed a sub-committee (C09.47) to standardize nationwide practices and procedure for testing properties of Self-compacting concrete [5]. ACI also published a report on self-compacting concrete in April 2007.

Due to its vast advantages Self-compacting concrete became very popular throughout the world and many countries conducted including Germany, USA, Sweden, Japan etc. did extraordinary research on it and applied it in their construction industries for the sake of achieving high performance concrete. However; research work on self-compacting concrete in Pakistan is in its initial stages and it has been used at a very limited scale in construction industry due to the high prices of chemical admixtures in the market and lack of basic knowledge about SCC systems.



Figure 2-1:Shin-Kiba Ohashi Bridge [5]



Figure 2.2: Akashi Kaikyo Bridge

2.1.2 Definition of SCCS through literature:

Every author and researcher has defined SCC in a unique and different way due to its flexible nature. Khayat et al [10] defines SCC as "Self Compacting Concrete is a special type of concrete that should flow into place and around obstacles under its own weight without segregation and flow blockage and with no significant separation of material constituents thereafter until the setting". This definition clearly states that SCC must have adequate flow to reach into those places where normal and conventional concrete can't reach without the help of manual vibration and it should be able to resist segregation. Europe guidelines [11] for self-compacting concrete define it as ""the concrete that is able to flow and consolidate under its own weight, completely fill the formwork even in the presence of dense reinforcement, whilst maintaining homogeneity and without the need for additional compaction". This definition also pretty much supports the same idea mentioned above.

Miao liu [12] states that concrete must fulfill the 3 basic criteria to be called as selfcompacting concrete, it should have filing ability, passing ability and resistance to segregation. Filling ability means that concrete should flow under its own weight without using any mechanical vibration and should fill the formwork completely. Passing ability is to pass through narrow and congested places and around obstacles such as reinforcement without being blocked or clogged. And finally SCC should offer resistance to segregation which means that it should stay uniform and homogeneous during transportation and after placement. Thus, The most common definition of SCC is "a concrete that is able to flow under its own weight and completely fill the formwork, and then consolidating without the need for vibrating compaction" [13]. ACI formed a committee "ACI 237" to standardize the properties of Self-compacting concrete and make a standard definition for SCC because there was no standard definition for SCC. In April 2007, "ACI 237" submitted its findings and a standard definition was determined through voting. Now the definition given in "ACI 237R-07" is the only valid and lawful definition while the rest of the definitions are considered invalid. Self-compacting cementitious systems (SCCS) are categorized into three major categories.

- 1. Self-Compacting Paste Systems (SCPS) or Single Component Systems.
- 2. Self-Compacting Mortar Systems (SCMS) or Double Component Systems.
- 3. Self-Compacting Concrete Systems (SCCS) or Triple Component Systems.

The main goal to develop all these systems is to get only one product and that is Self-Compacting Concrete Systems but to achieve this goal self-compacting paste (SCP) and self-compacting mortar (SCM) have to be optimized first.

2.1.3 Further Studies on SCC:

Various studies and researches have been conducted on self-compacting cementitious systems to improve it and enhance its properties. Few important and basic studies are discussed below:

Rizwan et al [14] states that "as paste is the vehicle of aggregate phase, good workability can be achieved by reducing the aggregate content with an increased paste volume thus resulting in reduced internal friction". This statement suggests that by increasing the quantity of fines in the mixture, higher flow is achievable. It is also suggested that sand content can be increased to enhance the cohesiveness and strength of concrete mixture. But this might result in higher demand of super-plasticizer to meet the flow requirements. Rizwan et al [15] also reported that "shape, size, surface morphology and porosity of secondary raw materials play a very significant role in determining the water and super plasticizer demands of the system, but besides the physical properties, the chemical composition of the secondary raw materials is also very important". It is true because few secondary raw materials have much higher water demands as compared to others and this phenomenon is because of their texture and other physical properties. For example, Bagasse Ash due to its hygroscopic behavior absorbs much more water than marble powder.

Rizwan et al [14] also suggested that no more than 15% coarse aggregate particles shall be elongated and irregular as they tend to increase internal friction, cause internal voids and bleeding and require a higher paste volume. The study also describes the effect of water temperature on the flow response of self-compacting cementitious systems and suggests that any addition of even small quantity of water after the chemical admixtures have been added could significantly decrease the mix cohesion and could produce slightly inaccurate results. Brouwers [19] suggest that sand of 50% by weight of total mixture can be used for the production of self-compacting concrete.

Self-compacting concrete shows a great degree of thixotropic behavior due to the presence of accelerating admixtures or higher quantities of binder content which promote a faster progress in cohesion resulting from hydration of cement. Furthermore, flow of concrete through reinforcement bars can be increased by increasing w/c ratio or increasing paste volume that will result in lesser volume of coarse aggregate and thus reducing the internal friction. But increased amount of water can reduce the cohesiveness and viscosity of the mix [20]. Thus, it can be easily summed up that self-compacting concrete is categorized by low w/c ratio, usage of super plasticizer, high amount of fines to produce more paste, use of smaller size as well as lesser content of aggregate [16].

2.1.4 Advantages of SCC:

Self-compacting concrete is a unique product; it offers a large number of advantages.

- SCC is able to flow under its own weight. Thus, placing the concrete has become affordable due to the elimination of manual vibrators. Now, concrete can be easily placed in areas of high and congested reinforcement. [21]
- Self-compacting concrete possesses better qualities as compared to conventional or normal concrete and its use in construction industry improves productivity and working conditions.[17]
- As use of SCC removes compaction, this evades the internal segregation between solid particles and the adjoining liquid which results in less porous transition zones between paste and aggregate and color of concrete in enhanced. [18]. Moreover, increased strength and high durability can be achieved.
- Self-compacting concrete also helps to reduce the noise pollution by removing the use of mechanical vibrators. It has also eliminated the health related problems of workers such as "white fingers" and "deafness" which used to arise due the use of vibrating equipment [18].
- SCC gives a uniform and homogeneous mix in the hardened state as mechanical vibrator has not been applied on it. Additionally, effects of bad workmanship are greatly reduced [21].
- SCC gives high quality surface finish. Its good flow produces smooth surfaces and minimizes the need for additional finish like plaster. [21]
- The formwork doesn't need to be tighter due to the enhanced cohesiveness of SCC as compared to the conventional vibration requiring concrete. [21]

2.2 Secondary Raw Materials (SRMs):

2.2.1 General Information:

The basic role of secondary raw materials in self-compacting concrete is to ameliorate the certain properties like increased flow, higher strength and evolution of heat of hydration, increased durability and shrinkage reduction. Using variations in amount of secondary raw materials and changing their combinations can produce drastically different results [11]. Secondary raw materials can have both inert and pozzolanic nature. Production of Portland

cement is highly energy consuming and environment unfriendly process. This process consumes almost 4 G J of energy per ton of cement powder. Plus, it also releases 0.8-1.3 ton of carbon dioxide per ton of cement produced. Energy shortage is an acute problem of modern world, so considerable efforts are being made to conserve energy. For this purpose, secondary raw materials also known as SRM's are being introduced to replace the cement contents. These SRM's are also known as supplementary cementitious materials or waste materials in literature. Use of SRM's in place of cement results in reduced shrinkage and heat of hydration. SRM's are recognized are energy efficient materials as they are industrial by-products and are readily available. Additionally, they need very little or no pyroprocessing [22].

Cordiero et al [23] reports that SRM's influence the physical as well chemical properties of the concrete to increase its strength. Physical effects incorporate the higher packing density of concrete because SRM particles fill the open spaces present between the cement particles. This property depends on the size, shape and texture and morphology of the SRM particles. They produce chemical changes by producing siliceous and aluminous compounds that are produced as a result of chemical reaction with calcium hydroxide in the presence of water. Calcium hydroxide is produced through cement hydration. Rizwan and Bier [24] reported that three significant effects can be observed by adding mineral admixtures to concrete. These effects include dilution, heterogeneous nucleation and pozzolanic reaction. Heterogeneous nucleation is a physical process but it leads to chemical process which involves the chemical activation of cement hydrates. Rizwan and Bier [24] also stated that addition of SRM to concrete increases the demand for water and superplasticizer to achieve the required flow parameters because SRM particles absorb water molecules through their internal pores.

Tangpagasit et al [25] reported that during initial 28 days, physical effects of SRM's dominate and they contribute towards the strength development. However, the chemical reactions or pozzolanic activities contribute towards strength development at later stages. Very small size of SRM's provides concrete with great advantages as their small particles fit in the cement grains and improves the packing density. Hence, it reduces the bleeding and increases the compressive strength. More dense packing at the aggregate/paste interface produces a thinner transition zone with a denser, more consistent microstructure [26]. Cyr et al [27] reported that an optimum amount of Secondary raw materials should be used to achieve the required properties. Using above this optimal value, benefits of using Secondary raw materials start to diminish. Therefore, it is generally recommended to use 10% replacements of cements contents.

Kronnlof et al [28] reported that small size particles were considered to increase the water demand due to enhancement in surface area. Increase in water demand meant harmful effects to concrete. However, mathematical particle packing theories show that secondary raw materials particles fill the empty spaces present between aggregate particles. Those spaces which are left in between fine particles are filled with water molecules and to some extent air also. But to improve workability excess water is needed which can result in strength loss. So here comes the role of plasticizer and super-plasticizer, which is to disperse the particles into spaces within their size ranges. In those mixes where super-plasticizer is not used, SRM particles get flocculated and more water is required to enhance workability. But if super-plasticizers are used, they break the flocculation and hence dense packing is achieved.

In lean concrete, mineral powders should be used to fill the aggregate spacing instead of air or water as far as workability is maintained because high packing density and high compressive is the basic requirement of achieving good quality concrete. While using rock powders, water reduction is considerable in super-plasticizer mixes for lean concrete [28]. Inclusion of secondary raw materials in lean concrete with the addition of super plasticizer not only improves the strength of the concrete but also enhances the interaction of paste and aggregate. This interaction depends on following factors:

- SRMs interfere with the positioning of large crystals at aggregate and paste interface.
- Large quantities of SRMs may change the rheology and lessen the internal bleeding at paste aggregate interfaces.
- > Both the components paste and aggregate form a homogeneous mixture.

In this research work to prepare self-compacting paste and self-compacting mortar, we will be using marble powder, fly ash, gypsum, bagasse ash and brick powder. Firstly, these secondary raw materials will be used in preparation of paste with 10% by weight of total powder and then they these materials will be used in combinations with equal percentages.

2.2.2 Bagasse Ash (BA):

Sugarcane today plays an important role in the global economy. Sugar and alcohol are produced by it. Countries are earning impressive profits through the imports of these two products. Production process of sugar and alcohol through sugarcane produces bagasse as a waste, which is then burnt as a fuel to stoke boilers that generate steam for electricity production. The final product of this burning is residual sugarcane bagasse ash (SBA), which is normally used as fertilizer in sugarcane plantations. Ash stands out among agro industrial wastes because it results from energy generating processes. Many types of ash do not have hydraulic or pozzolanic reactivity, but can be used in civil construction as inert materials [29]. It has been known that the worldwide total production of sugarcane is over 1500 million tons [30].



Sugarcane bagasse ash has recently been tested and tried in some parts of the world for its use as a cement replacement material. It was found out by results that bagasse ash certainly enhances some properties of the paste, mortar and concrete. These properties included high packing density, compressive strength and water tightness, but these advantages were only achieved if bagasse was used at certain replacement percentages and small size. The higher silica content in the bagasse ash was considered to be the main reason for high strength and all other improvements in the quality of concrete. Although the silicate contents can vary from ash to ash depending upon the burning conditions and other properties of the raw materials including the soil on which the sugarcane is grown, it has been reported that the silicate undergoes a pozzolanic reaction with the hydration products of the cement and results in a reduction of the free lime in the concrete [31].

Cordeiro et al [32] stated that bagasse ash shows pozzolanic behavior so it can be considered as pozzolanic material but its pozzolanic activity heavily depends upon its particle size. Smaller the particle size of bagasse ash more will be the compressive strength of concrete. Cordeiro et al [33] also reported that plastic viscosity increases slightly due to integration of bagasse ash in high performance concrete. However, the yield stress always reduces when bagasse ash is used, which reflects the positive effect of the ash on concrete. The negligible carbon content (LOI of 0.42%) of bagasse ash contributes to better rheological behavior as the concrete with high carbon content mineral admixtures requires more amount of super plasticizer for same workability. However, no significant effect was noted on the compressive strength of high performance concrete with small addition of bagasse ash (10%), but a negative trend was observed with increase in bagasse ash content beyond 10% increase.

2.2.3 Fly Ash (FA):

Fly ash also known as pulverized fuel ash is a residue that is obtained as by-product of combustion of pulverized coal in furnaces of thermal power plants. Fly ash is the principal product of transformation of mineral impurities present in coal after its combustion in

pulverized fuel furnaces. The properties of fly ash heavily depend upon the type of combustion system and also on the type of coal being used. During the thermal process coals melts at the high temperatures of around 1400°C-1500°C and after burning at such high temperatures a small part of it falls in the bottom of the boiler producing the so-called bottom ash. However, the main part is carried by the exhausted gases, cooling, and is then cooled and solidified in the form of small sphere-shaped particles, which are basically fly ash particles. Fly ash is separated by the fumes through use of electrostatic or mechanical precipitators and then collected in the hoppers. After collecting fly ash, it is stocked dried in silos is then transported by trucks or shipped to various destinations for its use. Generally, the diameter of the fly ash particles is less than 100 microns. Obtained fly ash is grey in color. Chemically, it comprises of alumina, ferrous oxides and high contents of silica [35]. Now-a-days dry method is used to produce fly ash and it gives much better and homogeneous fly ash as compared to that produced through wet method. To reduce air pollution, LSP is now being introduced into the combustion chamber to absorb sulfur contents released by burning high sulfur coals.

High volume Fly ash incorporated concrete for structural use was first developed by the Canadian Centre for Mineral and Energy Technology (CANMET) in the late 1980s [34]. Glass contents present in fly ash react with lime in the presence of water and in this way its pozzolanic activity is enhanced. According to ASTM C 618, fly ash will be considered as class F it contains oxides of alumina, silica and iron more than 70% of total composition. If oxides are greater than 50% then it will be class C fly ash. Class C is produced from lignite or sub-bituminous coal. Moreover, Class C has some cementitious properties in addition to pozzolonic properties. Fly ash becomes more pozzolonic as its particle size reduces [36].

Generally, fly ash contents used in concrete are limited to 15% to 25% of cement replacement [37]. Feldman et al. [38] reported that in high-volume fly ash/cement (FC) pastes, the fly ash starts reaction with Calcium hydroxide between 3 and 7 days, but significant quantities of Calcium hydroxide and fly ash still don't react even after 91 days of hydration. Calcium silicate hydrates (CSH) that are the reaction products have lower calcium-to-silica ratios. Berry et al. [39] stated that fly ash acts as filler in the early stages and its physical properties are dominant in this period. Additionally, it is involved in the formation of ettringite. However, at the later stages they are involved in hydration reaction mainly as silico-aluminate binders. Studies have showed that use of fly ash can increase the slump of concrete mix without adding up extra cost. It reduces the dosage of superplasticizer to obtain similar slump compared to concrete made by Portland cement [40]. Use of fly ash also improves the rheological properties of concrete and it reduces cracking in concrete by lowering heat of hydration of cement [41].

Kim et al. [42] reported that replacement of cement by 30% of fly ash resulted in super flowing concrete and addition of fly ash ameliorated the flow and workability of concrete.

High-strength self-compacting concrete production using fly ash has been a very encouraging impact to the sustainability of concrete technology. Use of fly ash decreases the demand for cement, fine fillers and sand [43]. The low water to cement ratio of the concrete imparts high durability to concrete, in addition to better mechanical integrity of the structure. More importantly, flaws due to wrong or inadequate vibration for compacting the concrete which often reduce the durability of the structure are completely avoided [44].

2.2.4 Gypsum (Hemihydrate) (HH):

Gypsum plaster or hemihydrate is among the earliest building materials used for construction. It has been being utilized for 4000 years [45]. Gypsum plasterboard has got excellent resistance to fire. It is being widely used for interior walls and ceilings due to its easy fabrication, environment friendly nature, aesthetics and considerably low price [46]. Hemihydrate's chemical formula is CaSO₄.0.5H₂O and it is also commonly known as plaster of Paris.

Calcium sulfate alpha-hemihydrate (CaSO₄.0.5H₂O) is generally prepared by heating natural or synthetically produced calcium sulfate di-hydrate (CaSO₄.2H₂O) to temperatures of over 100°C in an autoclave. Industrially, hemihydrate is manufactured through this process, and the first industries that used this method to produce hemihydrate were built in Germany in 1962 and Japan in 1973 [47]. Alpha-hemihydrate can also be produced by alternative hydrothermal method. This method involves di-hydrate boiling in aqueous solutions of concentrated salts or inorganic acids under atmospheric pressure. This is a new technique to produce alpha-hemihydrate but it hasn't been used in industry yet [47]. Literature states that this method is quite efficient for waste gypsum beneficiations as the impurities present in it can dissolve and remain in the liquid phase. Dehydration of calcium sulfate can be elaborated by following reaction:

$CaSO_4 \cdot 2H_2O + heat \rightarrow CaSO_4 \cdot 0.5H_2O + 1.5H_2O$ (steam)

Considerable amount of water is needed to produce plaster of Paris by calcium sulfate due to large surface area of calcium sulfate. Due to excess water elimination during the curing period poses some serious problems to gypsum, such as more heat consumption which is not good and a high porosity [48].

Hemihydrate is now being used in producing self-compacting concrete to replace cement contents. Calcium sulfates are much more environment friendly as compared to cement and thus their use in concrete is on increase in place of cement as a binding material. Temperatures of up to 1480°C are required for calcination of ordinary Portland cement. However, calcination of hemihydrate requires temperature of 170°C. Thus, this process saves a lot of energy as compared to production of cement. Moreover, cement industry produces 6% of total carbon dioxide produced in the world which is approximately 1.4 billion tons per year and it has a considerable effect on global warming. So using calcium sulfate binders instead of cement would help in decreasing the global carbon dioxide (CO₂)

production [49]. Using gypsum for the preparation of self-compacting concrete can produce concrete with a compressive strength up to 4350 psi. And it would possess properties of self-compacting cementitious systems like flow ability and segregation resistance. Calcium sulfate based self-compacting concrete that has compressive strength of around 4350 psi can be used for variety of purposes. It can replace cement contents where low to medium strength is required. Another important application of gypsum can be in gypsum boards. These gypsum boards can be used in place of dry wall and these boards can be self-supporting [50]. Selecting the right particle size can enhance the particle packing and hence packing density. This can be achieved by adding fillers and aggregates in right proportions [51]. An improvement in particle packing would result in better physical and mechanical properties of concrete mix. The flow ability of fresh concrete mix improves and segregation resistance also increases. Water content of a concrete mix reduces and thus capillary pores formed will also be removed. This would result in higher compressive strength and better durability [52].

2.2.4 Marble Powder (MP):

Marble has been normally used as a building and construction material since the ancient ages. However, it was mainly used to increase the aesthetics of the buildings. It was used as stone bricks in walls, arches, or as lining slabs in walls, roofs or floors. Marble blocks were cut into smaller pieces for its efficient utilization but it left its wastage at quarry or at the sizing place. This cutting and resizing of marble resulted into millions of tons of marble powder or dust which constituted the 40% of total marble mass [53]. During the cutting process, about 25% marble results in marble dust [55]. This huge marble powder mass consisted of very fine size particles and it produced some serious environmental issues causing air pollution. To effectively utilize this huge amount of waste, various efforts have been done and its utilization in construction industry has been tried out. Few actions have been taken to evaluate the prospects of using waste marble powder in mortars and concretes and the findings about strength and flow ability have been compared with the samples made of conventional concrete or mortar [53].

Valeria et al (2005) reported that Blaine fineness value of marble powder is very high. Its value is about 1.5 m2/g with 90% of particles passing through 50 μ m sieves and 50% particles passing through 7 μ m. Very small size of marble powder particles increases the surface area of marble powder, so its addition as secondary raw material in mortars and concretes would increase cohesiveness. Numerous studies have been conducted in literature to check the performance of concrete using marble powder or waste marble aggregate especially its use in self-compacting concrete, its application in the mixture of asphaltic concrete and its utilization as an additive in production of cement [54]. Hanifi Binici et al (2007) reported that concrete containing marble powder has higher compressive strength as compared to the lime stone dust concrete having equal water to cement ratio and mix proportion. His research findings also showed that marble powder concrete has

lower water permeability than the concrete made using lime stone [53]. Ali Ergun (2011) conducted research to investigate the mechanical properties of the concrete in which he used 5% replacements of cement with diatomite and marble powder in one case. And in another case, he replaced 10% cement with waste marble powder and he found better mechanical properties and compressive strength as compared to 1st case [53].

Self-compacting cementitious systems require high water and large amount of cement due to finesses of aggregates. Excess water imparts harmful effects like bleeding, segregation of particles and weak interfaces between granular materials [56]. In SCCS, addition of fine contents makes an important influence on the quality of fresh and hardened concrete. Bedrina et al. [57] in his study on "reuse of local sand in sand concretes" states the importance of marble powder on improving the mechanical and rheological properties. By optimizing the percentage of marble powder in self-compacting concrete that can be in the range of 200kg/m³, researchers have been able to make more segregation resistant and workable concrete. Jiang and Mei [57] showed that the key to make self-compacting concrete is to use large volumes of fine material. They reported that workability increases by increasing quantity of fines in the mixture. Bhattacharya et al [58] reported that addition of fine materials and marble powder fillers have resulted in consistent slump flow values. However, these marble powder fillers have increased the slump flow values as compared to other materials. Therefore, by adding powder materials to cement improves the rheological properties of pastes and as a result the workability of the concrete mixture becomes better. Brain (2004) studied the effect of adding marble powder with Portland cement. He stated that it ameliorates the relative content in hydrates as well as microstructure. Marble powder reacts with calcium hydroxide to form calcium silicate hydrate. So volume of binders is increased, which enhances the strength and reduces the permeability [59].

2.2.5 Brick Powder (BP):

Brick powder is a waste material obtained from different brick kilns and tile manufacturing factories. Many brick kilns have developed over decades whole across the country. They are producing large volumes of bricks which result in tons of waste products like brick dust, broken pieces or flakes of bricks. These waste materials have been used to fill low lying areas or they are just dumped as waste material [60]. Annual demolition waste production in United States of America is estimated to be 136 million tons by Environmental Protection Agency [61]. It was calculated to be 180 million tons per year in the first 15 countries of the European Union [62]. Major portion of this demolition waste comes from masonry debris consisting of clay bricks. Brick and tile manufacturing industry yields a huge amount of rejected bricks and tiles due to being off-standard. These products may be broken, distorted, weak, underburned or over burned. Sometimes, this wasted material is used for landscaping purposes but generally it is dumped into sites around the brick factories or used in concrete as aggregate or filler material [61].

Various studies and researches have been conducted in 20th century to evaluate the potential use of brick powder or crushed bricks in concrete. Investigations on the use of crushed bricks in concrete have been carried out since 1928. In Germany, it became necessary to remove the debris left from the World War II and supply it to construction industry to use it as aggregate in concrete. To optimize the debris, rubble recycling plants were built. This provided two major advantages; elimination of debris from the environment and supply of aggregate for the new concrete. These recycling plants produced 11.5 million m³ crushed brick aggregate by the end of 1955 [63]. Continuous research on using brick aggregate in concrete proved that crushed bricks can be used for producing concrete that can be used for a wide range of applications [61].

Akhtaruzzaman and hasnat [64] conducted research using well-burned bricks as coarse aggregate in concrete; they found out that high strength concrete can be made using crushed bricks as coarse aggregate. Brito J et al [65] stated that strength of concrete ranging from 22% to 45% respectively for 33% and 100% replacement of natural aggregate. Debieb and Kenai [66] did research on using crushed bricks in concrete and concluded that using both coarse and fine crushed bricks show a decrease in strength ranging from 20% to 30% depending upon the range of replacement. Khatib [67] and poon and chan [68] also did some work on the use of crushed brick as aggregate in concrete and reported a decrease of concrete strength with usage of crushed bricks as fine aggregate substitute. However, Khaloo [69] and Hensen [70] reported that use of crushed brick aggregates in concrete increases the tensile strength of concrete as compared to the concrete made with natural aggregates. Bektas et al [61] did study on use of brick aggregate in mortar. Experimental study disclosed that brick aggregate negatively affected the mortar flow. It didn't affect compressive strength much when added in 10% and 20%. Adding 20% crushed brick aggregate reduced free shrinkage of the mortar. While adding 10% increased the free shrinkage as compared to control mix. Moreover, freeze-thaw resistance of mortar enhanced by increasing the amount of crushed brick replacement. Bricks used by Bektas were highly alkali reactive with a pessimum content of 30%. ASR susceptibility of the clay brick aggregate calls for further study and extent of its effects on concrete should be assessed in detail to avoid any future problems.

2.3 Super Plasticizers (SP):

The high water reducing admixtures generally known as super-plasticizers are artificially prepared water soluble organic substances that are used to significantly decrease the amount of water needed to achieve a certain consistency of concrete which means higher compressive strength and higher workability. Super-plasticizers can be used to serve following purposes:

1. To reduce the water content, this would result in increased strength, better permeability and higher durability.

2. It acts as cement dispersant for the same water content to increase consistency and workability.

Super-plasticizers are a comparatively new category and better version of plasticizer. Super-plasticizers use was started in Japan and Germany during 1960 and 1970 respectively. The chemical composition of super plasticizers is different from that of normal plasticizers. Using Super-plasticizers can reduce water up to 30% percent without any reduction in workability as compared to normal plasticizers which can only reduce water up to 15%. Super-plasticizers are generally used for the production of high flowing, self-leveling and self-compacting concrete. In American literature, they are termed as High Range Water Reducers (HRWR). Use of Super-Plasticizer has allowed water cement ratio as low as 0.25 or in some cases even lower than that and yet they are able to make concrete of around 120 N/mm² strength or more [69]. Super-Plasticizers are considered to be the important part of high performance concrete. They are not only used to lower the water demand but also regulate the open time and setting time. Their basic structure resembles a "comb type" molecule. The Poly-carboxylate group forms the main trunk or major chain of the molecule while the polyether groups are connected to poly-carboxylate trunk by chemical bonding. These polyether groups are also knows as "side chains" or "grafts". Their name suggests a likely connection between poly-carboxylate and ether groups. There are basically three types of "grafts" or "side chains" which are ether, ester and amide. Ester is common group among them. Super-plasticizers are responsible for lowering the porosity of cementitious systems in hardened state [70].

2.3.1 Mechanism of Super Plasticizers:

Now-a-days super-plasticizers are used at large scale in concrete industry. Two major super-plasticizers that are being used are polynaphthalene sulphonate and polymelamine sulphonate formaldehyde condensates. These are polymers and both follow the same dispersing mechanism. The main polymers chains (sulphonate groups $-SO_3^-$) get themselves absorbed onto the surface of cement particles; as a result of it cement particles get negative charge on them. Therefore, electrostatic repulsion takes place between the cement particles and they start repelling each other. These repulsion forces between the cement particles offset the inter-particle attractive forces. Poly-carboxylate super-plasticizers increase dispersion because of their better performance in dispersing cement particles. They easily disperse cement particles even at smaller dosages and maintain the concrete slump without prolonging setting times of concrete [70]. Electrostatic repulsion plays an important role in the dispersion mechanism of super-plasticizers. Poly-carboxylate polymers contain -COO- groups instead of $-SO_3^-$ groups in sulphonated condensate. Two super-plasticizers were used in this research study, which are:

- 1. Mel-flux 2651 F
- 2. Glenium 51

2.3.2 Mel-flux 2651 F:

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

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

2.3.2.1 Applications:

It has following applications:

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

2.3.3.2 Properties:

-	
Physical Shape	Powder
Appearance Characteristic	Yellowish to Brownish
Drying Loss	Max 2.0%
Bulk Density	300-600 kg/m ²
Dosage Recommendation	0.05-1.00% by weight of cementitious
	material
pH value at 20°C, 20% solution	6.5 - 8.5
r	

2.3.3 GLENIUM 51:

GLENIUM 51 has been generally used for applications in the ready mixed concrete and precast concrete industries where high durability and better performance is needed. It does not contain chlorides and obeys ASTM C494 types A and F. It can be used in all Portland cements that meet international standards. It has been used in the production of self-compacting mortar in this study.

2.3.3.1 Working Mechanism:

Their working mechanism of GLENIUM 51 is different from that of conventional superplasticizers. It has distinctive carboxylic ether polymer with log lateral chains. This pattern enhances the cement dispersion. At the initial stages of mixing process, electrostatic dispersion process is same as that of other conventional super-plasticizers but the lateral chains which are linked to polymer backbone produce a steric hindrance and it stabilizes the cement particles capacity to separate and disperse. So this mechanism produces concrete that is highly flow able and required very little water.

2.3.3.2 Applications:

Its excellent dispersion properties make it very suitable for the precast and ready mixed concrete where low water to cement ratio is needed. Very high early strength concrete with minimum voids can be produced using it. GLENIUM 51 can be used to produce high early strength floor screeds.

GLENIUM 51 offers following advantages:

- High workability.
- Resistance to segregation.
- Vibration is not required.
- > Concrete produced can be placed and compacted in congested reinforcement.
- Less dependency on labor.
- ➢ Better surface finish.

2.3.3.3 Properties:

Form	Viscous liquid
Color	Light Brown
Relative Density	1.1 @ 20°C
рН	6.6
Viscosity	128 +/- 30cps @ 20°C
Transport	Not classified as dangerous
Labelling	No hazard label required

2.4 Experimental Techniques:

2.4.1 Calorimetery:

Cement hydration is extremely exothermic reaction taking place in a series of steps. It has following stages (Young 1985):

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

Calorimetery is the best procedure to find all the stages of hydration process of cement (Bensled 1987). Determination of heat of hydration is very important because it affects almost all the properties of concrete including workability, setting times, strength development rates and pore structure development. Heat of hydration affects the early age of concrete as well as the long term performance of concrete. Basically, Calorimetery is the technique used to measure the heat production of the cement paste with time and evaluate the hydration kinetics. Calorimetric curves produced during a specific interval of time form various peaks. At initial stages, heat of hydration is high due to the fast hydration of aluminate phase, C₃A, in this phase the main product formed is AFt. After the initial period, heat of hydration decreases to minimum within 2-3 hours. This phase of hydration reaction is called induction period or dormant period. This dormant period is then followed by pozzolanic reaction and mass precipitation of hydration products, mainly Calcium silicate hydrate (CSH) gel. Then the calorimetric curve rises to a second highest value of hydration process and this happens after 15-18 hours for self-compacting paste systems. After this the hydration process slows down and smaller peaks are formed in the process [71]. Rizwan et al [72] stated that super-plasticizer's presence in the cementitious systems delays the heap peaks produced during Calorimetery of cement process. Moreover, amount of heat evolved depends upon a variety of factors which include cement type, its content and water to cement ratio because super-plasticizer acts as retarder.



2.4.2 Scanning Electron Microscopy (SEM):

Scanning electron microscope (SEM) is one of the most multipurpose instruments available for the investigation of microstructure morphology and the chemical composition. Electron microscopy has been developed by using high energy electron beam instead of normal light source [73]. Scanning electron microscopy provides several advantages for characterization of concrete, cement, aggregate and secondary raw materials microstructure. Scanning electron microscope gives better identification of hardened cement paste constituents with higher contrast and better spatial resolution as compared to optical methods and gives a better element analysis and imaging. Quantitative information such as composition of mixture, phase abundance and distribution can be gathered using electron microscopy [74].

SEM gives a very clear and fine image of concrete at very large magnifications. Scanning Electron Microscopy (SEM) offers a quantitative chemical analysis in terms of energy Dispersive X-ray spectroscopy (EDAX) [75]. Shape and morphology of micro particles can be easily assessed using scanning electron microscopy because each material has its own specific individual form. For example, calcium hydroxide crystals are plate like or blocks of hexagon, ettringtie has elongated rod like crystals appearance, Calcium silicate hydrate crystals have needles like appearance.



Figure 2.4 SEM imaging of cement structure [75]

Secondary electron images showing the hexagonal habit of calcium hydroxide, needle-like habit of ettringite, and the sheet-like habit of calcium silicate hydrate. In the upper image, crystal growth into a void, where space restrictions are minimal, allowing development of euhedral forms. The lower image, being a more mature paste with limited space, exhibits crystal forms are more subhedral to anhedral. Here one may identify the plate-like CH morphology and fine bundles of Type-I C-S-H, platy Type-II C-S-H, and ettringite needles.

2.4.3 Mercury Intrusion Porosimetry (MIP):

Mercury Intrusion Porosimetry (MIP) is an experimental technique that is extensively used for assessing the distribution of pore sizes in cementitious systems. It is very simple and rapid technique but it doesn't work well when it is used for materials of irregular pore geometry [72]. Mercury Intrusion Porosimetry (MIP) has been successfully used for the study of cement paste systems and cement mortar systems. But now it is being used for the determination of porosity in concrete systems as well [77, 78]. However, it must be kept in mind while using MIP on concrete that its results are affected by the method adopted for sampling, condition of sample, sample mass and dimension used, pressure application rate, assumed pore shape, values of contact angle and surface tension of mercury. Mercury Intrusion Porosimetry (MIP) results can also be affected by the expansion of under pressure sample cell, simple compression, hydrostatic head of mercury and differential mercury compression.

The standard procedure of conducting MIP involves a small specimen that is first dried to empty the pores of any existing fluid. Then it is weighted, moved to chamber and the chamber is emptied. Sample is afterwards surrounded by mercury and the pressure is gradually amplified. With increase in pressure, mercury goes into the pores which are present on the surface of sample. For continuous pore system that extends to surface, mercury penetrates the smallest pore necks with increased pressure. However, if the pore system is not continuous, mercury penetrates the sample volume by going through pore walls. Tracking pressures and intruded volume make is possible to determine the connecting pore neck diameter of a continuous system or a break through pressure in a discontinuous system. The pore width conforming to the highest rate of mercury intrusion per change in pressure in called as "threshold", "critical" or "percolation" pore width [79]. Once this pressure has been achieved, mercury penetrates into the interior of the sample. Total porosity of the sample corresponding to volume of mercury intruded at the maximum pressure divided by the bulk volume of the un-intruded sample, can be measured using this technique.

2.4.4 Particle Size Distribution:

Particle size distribution (PSD) is an experimental technique used to measure the size of the particles present in a sample and their distribution in relation to size. A distribution of particle size or particle size distribution (PSD) is a fundamental characteristic of cement

powder and secondary raw materials. Accurate and precise results of particle size distribution are needed in computational modelling of hydration process and predict the performance of cementitious materials. ASTM115-96 is the standard method currently in use; it is a tubudimetric method for the determination of fineness. This method can only determine size with a lower detection limit of 7.5 μ m [80]. However, new techniques are being introduced now that can measure the size even lower than 7.5 μ m. The laser diffraction measurement is also being currently used for cement particle size distribution. Many cement producers use PSD for the quality control of their cements, in association with the measurement of Blaine fineness. The laser diffraction measurement of blaine fineness. The laser diffraction measurement provides an estimation of powder surface area by assuming a specific gravity for the particles. Although laser diffraction is widely used by the cement industry, it is still not a standard test to measure the particle sizes and their distribution [81].

2.4.4 X-Ray Fluorescence (XRF):

X-ray fluorescence (XRF) is an experimental technique in which emission of characteristic X-rays from a material takes place. This emission is result of excitation of materials done by bombarding them with high energy X-rays or Gemma rays. XRF is extensively use for elemental analysis and chemical analysis of metals, ceramics, glass and building materials. XRF gives the complete chemical analysis of a sample giving the quantities of all the chemical compounds or elements present in a sample. Chemical analysis of cement and secondary raw materials can be done using variety of experimental techniques including wet chemical analysis and instrumental measurements. X-ray fluorescence (XRF) spectrometer is often used for chemical analysis of cement, despite the high costs and additional equipment-handling operations. X-ray fluorescence spectrometer offers various advantages including ease of sample preparation, suitability for determining a range of elements occurring in cements, ease of calibration as well as linearity of calibration curves over extended concentration ranges for most elements [82].

2.4.5 X-Ray Diffraction (XRD):

X-Ray Diffraction is a strong and efficient experimental technique used for detection and quantification of crystalline materials. It also produces experimental results when applied to amorphous substances. Amorphous substances are those substances that do not show crystalline nature. Mostly naturally occurring minerals are present in crystalline form. The working procedure of X-Ray Diffraction involves a scattering process in which X-Rays are dispersed by electrons present in the materials without changing wavelength of X-Rays. This phenomenon produces a diffraction pattern that is a fundamental physical property of every substance. It gives quick identification and comprehensive explanation of microstructure. Both quantitative and qualitative X-Ray Diffraction analysis can be performed

for cementitious systems. Quantitative analysis performed using XRD gives precise results but it consumes time, requires special expertise and is highly expensive. So generally qualitative analysis is performed and is preferred. X-Ray diffraction is also used to keep track of hydration process by determining the amount of un-reacted clinker materials and calcium sulfate. It can also be done by determining the amount of formed hydrates as function of time. Quantitative analysis is more helpful in an attempt to find the amount of un-reacted cement constituents [83].

Chapter 3: EXPERIMENTAL PROCEDURE

3.1 General:

The SRMs used in self-compacting systems were finely grinded powder passed from sieve #350. Materials were stored in air tight containers to control the changes in moisture content. Lab temperature and relative humidity was constantly noted to observe the variations. A sequential experimental approach was followed to study the behavior of different SRMs in self-compacting systems. Initially physical and chemical properties were determined using particle size analysis and XRF respectively. Several formulations of self-compacting paste were formulated with 10% replacement of cement with single SRM and with the blend of SRMs also. Water demand and setting time were determined for each formulation using Vicat apparatus. SP demand and flow times were determined using Hagerman's mini slump cone. Calorimetric analysis was done using field calorimeter and finally flexural and compressive strength were determined using dual chamber assemblies.

3.2 Materials:

3.2.1 Cement (OPC):

Ordinary Portland cement (OPC) Grade 43 of Bestway (local manufacturer) following EN 196-1 /ASTM C150 was selected for the following research purpose. Fresh OPC free form any lumps was obtained and stored in air tight containers to keep it away from moisture. Physical and chemical properties of OPC is shown in table

3.2.2 Secondary Raw Materials (SRMs):

Secondary raw materials (SRMs) used in this research are as follow

3.2.2.1 Fly ash (FA):

Fly ash for this research purpose was imported from a German company **Bau Mineral KraftWerkstoffe**. The purpose to use this materials is to ensure the quality and consistency in the material. The fly ash used is finally grinded and a homogeneous material. The chemical and physical properties of the fly ash is shown in **Table 1 Annexure A**.

3.2.2.2 Marble Powder (MP):

Marble Powder is a finely grounded marble obtained from a local manufacturer. The powder obtained in a white powder and chemical analysis shows a larger amount of CaO which ensures the quality of marble powder. The marble powder was stored in air tight containers to ensure the consistency. The physical and chemical properties of MP is shown in **Table 1 Annexure A**.
3.2.2.3 Bagasse Ash (BA):

Bagasse ash is a byproduct of sugar industry and is actually an ash produced by the burning of sugarcane husk at elevated temperature. The ash obtained from the industry is finely grinded in Los Angles abrasion machine then oven dried and stored in air tight containers. The physical and chemical properties of BA is shown in **Table 1 Annexure A**.

3.2.2.4 Hemihydrate (HH):

Hemihydrate is CaSO₄. 0.5 H_2O is obtained from a local distributor. It is prepared by heating gypsum CaSO₄. 2 H_2O at elevated temperature. The sample may include anhydrous and di-hydrated Calcium Sulfate. Physical and chemical properties are shown in the **Table 1 Annexure A**.

3.2.2.5 Brick Powder (BP):

Brick powder is a waste material available at brick kiln produced by the wear and tear of bricks. The mass includes fine powder, broken parts of bricks and parts of burnt bricks. The powder was obtained from a local kiln at Lahore Pakistan and was grinded to fine powder. The powder contain high % of iron contents due to which the color of it is bright red. Physical and chemical properties are shown in **Table 1 Annexure A**.

3.2.3 Sand:

In contrast with conventional concrete, fine aggregate are major constituent of selfcompacting concrete. Different properties of concrete are defined by their types and amount of aggregate used. For example flow ability and segregation resistance is increased when used in suitable amount. Also they change concrete strength when used in different proportions with cement and coarse aggregate compressive strength and mixing water requirement are affected by particle shape, surface texture, surface area and void concentration. Well graded fine aggregates are used as they increase the packing density and durability and enhance the workability of mortar and hence improve the flowing of SCC. Also the amount of coarse aggregate depends upon the fineness modulus of fine aggregates.



Figure 3.1 Gradation curve of sand

3.2.4 Chemical Admixtures:

Super plasticizers are used as chemical admixtures in this project. **Melflux** 2651 F is the super plasticizer use with self-compacting paste system and Glenium 51 is the super plasticizer used in self compacting mortar systems. Both admixtures are produced by BASF and were imported from Germany.

3.3 Particle Size Analysis:

Different SRMs used in the project had different particle sizes/ grain sizes. So particle size analysis was carried out to check the particle size of each SRM. Packing density of the material is a function of particle size. Smaller the particle size higher will be the packing density. The smaller particles fills the pores/gaps thus as a result increases the density of the material. For the analysis of particle size of the materials particle size analysis was carried out.

Samples were initially sieved and then particle size analysis was carried out using Particle Size Analyzer. The results obtained from the particle size analyzer was the particle size against its relative abundance. So the result was loaded in MATLAB and using the built in curve fitting tool best fit curve was selected. Now by using the equation and coefficients of the newly fitted curve obtained from the curve fitting tool D50 was calculated using a code for D50 to make the results more accurate.

Method for D50:

Following steps were adopted to calculate the average particle size/D50.

• Particle size analyzer gives results in excel sheet having particle size in a column and other having the abundance in %

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Figure 3.2 Results of PSA in Excel Sheet

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- Write "clear;" then "clc;" to remove the previous variables
- Select a variable "x=[];"
- Copy the column of diameters from excel and paste it in **x**
- Similarly select a variable y and paste the column of abundance in it i.e. "q"
- Then write **"cftool;"** then run the function from run key
- select "change folder" and a new window of curve fit will appear

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- Select x matrix in x data and y matrix in y data and select and also select the method of method of curve fitting and degree of fitting curve so that the new curve must be optimized
- The result part of the window indicate the equation and coefficients of equation we will use this to calculate the particle size



Figure 3.3 Curve fitting tool window

- Now open another blank m file and generate a variable x with the diameters as done before
- Then copy all the coefficients from the curve fitting window as shown and paste it in the new file
- Then initialize variables as **ps=0**; **product=0**; **and y=0**;
- Then generate a for loop for the number of iteration equal to the data points of matrix x
- In that loop, paste the function as "y= " as shown in fig with x changes to x(i)
- Multiply y with the same ordinate of x in variable product and add it in a variable of **ps** as shown then close the loop
- Out of loop divide the variable **ps** by **100** and print the variable **ps**. This will give the particle size of material

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1 -	x=[0.022,0.026,0.029,0.034,0.039,0.044,0.051,0.058,0.067,0.076,0	0.087,0.1,0.115,0.131,0.15,0.172,0.197,0.226,0.259,0.296,0.339,0.389,0.445,0.51,0.584,0.669,0.7
2 -	a1 = 4.618;	
3 -	bl = 15.97;	
4 -	c1 = 5.804;	
5 -	a2 = 3.892;	
6 -	b2 = 23.16;	
7 -	c2 = 8.735;	
8 -	a3 = 3.823;	
9 -	b3 = 9.951;	
10 -	c3 = 4.14;	
11 -	ai - 1.818; Carffinington 6	
12 -	b4 = 38.75; Coefficients of	Equation
13 -	c4 = 15.25;	
14 -	a5 = 1.946;	
15 -	b5 = 64.75;	
16 -	c5 = 26.74;	
17 -	a6 = 0;	
18 -	b6 = 47.05;	
19 -	c6 = 0.1964;	
20 -	a7 = 1.302;	
21 -	b7 = 110.3;	
22 -	c7 = 51.31;	
23 -	ps=0;product=0;y=0;	
24 -	for 1=1:85	
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Figure 3.4 Code for D50

3.4 Test Procedure:

3.4.1 Mixing Regime:

It is mixing scheme that affects the shear rate. The mixing regime we followed was: For paste systems:

- \checkmark Dry mixing for 60 seconds
- \checkmark Addition of water
- ✓ Initial slow mixing for 30 seconds
- ✓ Sides clearing
- ✓ Slow mixing for 30 seconds
- ✓ Fast mixing for 60 seconds

For mortar systems:

- \checkmark Dry mixing for 60 seconds
- ✓ Addition of water 80%
- ✓ Initial slow mixing for 30 seconds
- ✓ Fast mixing 60 seconds
- ✓ Sides clearing
- \checkmark Addition of 20% water with SP dissolved in it
- ✓ Fast mixing 3 minutes

3.4.2 Mixing Proportions:

In SCP systems: 10% of cement was replaced with SRMs. In single SRM replacement one SRM was added as 10% of cement and in blends of SRM 5% of two SRMs each was added. Brick powder was used as SRM in 5, 10 and 15% of cement.

In SCM systems: 1:1:2(Cement: SRM: Sand) was used to ensure higher performance of the system.

3.4.3 Water Demand (WD):

Water Demand or standard consistency is the minimum amount of water required by cement for complete hydration reaction. Water demand was measured using Vicat apparatus:



Figure 3.5 Vicat Apparatus

3.4.4 Setting Time:

Initial setting occurs when paste begins to stiffen considerably. Final setting occurs when the cement has hardened to the point at which it can sustain some load. Setting time of cement is measured by ASTM C191-01 using Vicat needle.

3.4.5 SP demand:

Super plasticizer demand is measured for both paste and mortar systems using flow test apparatus. Apparatus consists of a glass tray and a mini slump cone. SP amount is optimum in paste and mortar when diameter of flow on tray in 25 to 30 cm.

3.4.6 T25 and T30:

Time required for flow to reach diameter on glass tray of 25 and 30 cm are measured and noted as T25 and T30 respectively.



Figure 3.6 Hagerman's Mini slump cone

3.4.7 Casting and Curing:

The casting and curing was done according to EN 196-1. SCP samples were cast into prisms having dimensions 40x40x160 mm. After de-molding samples were placed in a closed water tank till the time of testing i.e. 3 days, 7 days, 21 days and 28 days.





Figure 3.7 Molds and Samples

3.4.8 Strength Evaluation:

Flexural and Compressive strength were determined using Dual chamber. Strengths were measured for 1, 3, 7, 21 and 28 days. Initially the prisms were testes for the flexural strength at the loading rate of **0.02 kN/sec**. The two samples obtained after the flexural test were passed through compression test at a higher loading rate of **0.2 kN/sec**. The results obtained from those two samples were then average out to get the more accurate results.





Figure 3.8 Dual Chamber for flexural and Compression test

3.4.9 Calorimetery:

Hydration is a very exothermic reaction. Calorimetric analysis was carried out to study the hydration kinetics of cement with respect to different SRMs. F-CAL 8000 field calorimeter was used for the purpose. Samples were prepared at standard consistency and with the calculated super plasticizer demand. Containers were filled to 3/4 nearly and placed in the respective slots. Calorimetric analysis was carried out for 48 hours.



Figure 3.9 F-Cal 8000 Field Calorimeter

CHAPTER 4: TEST RESULTS

4.1 Test on Self Compacting Paste:

4.1.1 Self Compacting Paste with 10% SRM

4.1.1.1 Water Demand (WD) and Setting Time of SCP:

The water demand generally increases with the addition of secondary raw material (SRM) in OPC, however it depends on the particle size and nature of SRM. Water demand of pure cement (C1-0) is considered to be the reference for all other formulations. Water Demand of marble powder (MP) and fly ash (FA) increases slightly. Bagasse ash (BA), brick powder (BP) and hemihydrate (HH) shows higher increment in the water demand.



The setting times of the SCP with SRMs depends on the characteristics of the particles used. The initial and final setting times of reference formulation is used to compare the behavior of each SRM used. The setting times of different formulations are shown in **fig 4.2**



4.1.1.2 SP Demand and Flow Time of SCP:

The SP demand was calculated by Hagerman's Apparatus to achieve the target flow of 30 ± 1 cm. The SP demand of any SRM depends on its micro structure and chemical behavior. SP demand and flow time for 25cm (T₂₅) and 30cm (T₃₀) is shown in Fig 4.3 &4.4



4.1.1.3 Strength test of SCP:

Prisms of size **40x40x160cm** (as defined by EN196-1) were tested having 10% SRM at standard consistency (WD) and calculated SP demand. Tests were conducted at 1, 3, 7, 21 and 28 days to study the behavior of SRM in the strength development of SCP. As described before SCP of pure cement is considered as a reference to study the behavior of different SRMs. **Fig 4.5&4.6** shows the flexural and compressive strength of different formulations.



4.1.1.4 Calorimetery of SCP

Calorimetric study was carried out to study the comparative response of SRMs on hydration kinetics. Control formulation was selected as a reference to compare the hydration kinetics of different SRMs. **Fig 4.7** shows the Calorimetery of different formulations up to 48 hours.



4.1.2 Self Compacting Paste with SRM blends

4.1.2.1 Water Demand (WD) and Setting Time of SCP:

In the case of blends of SRMs, cement was substituted 10% by two different SRMs, 5% each in every formulation. The water demand of blends increased nearly equally from reference control formulation except the blend of HH and BA whose water demand increased to much higher value. Fig shows the water demand of different blends.



As described earlier, setting time depends on the characteristic of particles of SRM used in SCP. **Fig 4.9** shows the initial and final setting time of blends used in SRMs.





4.1.2.2 SP Demand and Flow Time of SCP:

SP demand follows nearly the same trend as that of water demand for the blends. This is due the fact that blend of different SRMs complement each other. The flow times depend on the particle characteristics of SRMs used in SCP. SP demand and flow times of different blends in SCP is shown in **Fig 4.10&4.11**.



Figure 4.10 SP Demand of SCP System



Figure 4.11 Flow Measurement of SCP System

4.1.2.3 Strength test of SCP:

Prisms of size **40x40x160cm** (as defined by EN196-1) were tested having blend of SRMs 5% each at standard consistency (WD) and calculated SP demand. Tests were conducted at 1, 3, 7, 21 and 28 days to study the behavior of SRM in the strength development of SCP. Fig shows the flexural and compressive strength of different formulations.



Figure 4.12 Flexural strength of SCP System



Fig 4.13 Compression Strength of SCP System

4.1.2.4 Calorimetery of SCP

Calorimetric study was carried out to study the comparative response of blends of SRMs on hydration kinetics. Control formulation was selected as a reference to compare the hydration kinetics of blends of SRMs. **Fig 4.14** shows the Calorimetery of blended formulations up to 48 hours.



Figure 4.14 Calorimetery of SCP System

4.1.3 Self Compacting Paste with varying % of Brick Powder (BP):

A separate study with the different proportion of brick powder (BP) was conducted to study the behavior of BP as SRM in SCP. For this study cement is being replaced by 5%, 10% and 15% with BP. Water demand, setting times, SP demand and strength was evaluated for each formulation to check the behavior as SRM in SCP.

4.1.3.1 Water Demand and Setting Times

Water demand of BP in SCP increases nearly linearly with the increase in the % of BP. Similarly setting times also shows nearly the same trend as that of water demand. **Fig 4.15 &4.16** shows the water demand and setting times of various % of BP in SCP.



Figure 4.15 Water Demand of various % BP in SCP



Figure 4.16 Setting Times of various % BP in SCP

4.1.3.2 SP Demand and Flow Measurements:

SP demand for the SCP with various % of BP increases with the increase in the amount of BP in SCP, similarly flow times increases as the % of BP increase in SCP. Fig show the SP demand and flow times of the formulations of BP in SCP.



Figure 4.18 SP demand of various % BP in SCP

4.1.3.3 Strength test of SCP:

Prisms of size **40x40x160cm** (as defined by EN196-1) were tested having varying % of BP at standard consistency (WD) and calculated SP demand. Tests were conducted at 1, 3, 7, 21 and 28 days to study the behavior of different % of BP in the strength development of SCP. Fig shows the flexural and compressive strength of different formulations.



Figure 4.19 Flexural Strength of various % BP in SCP



4.1.3.4 Accelerating Hydration:

BP 15% shows exceptional delay in the setting which results in lesser early strength. So to accelerate the rate of hydration accelerators were used to reduce the setting time of BP 15% in SCP. Accelerators used for the current study is lithium carbonate (Li_2CO_3) and sodium carbonate (Na_2CO_3). Fig 4.21&4.22 shows the reduction in the setting time of 15% BP in SCP for different amounts of accelerators.



Figure 4.22 Setting Time of %15 BP in SCP with Na₂CO₃

4.2 Test on Self Compacting Mortar:

Mix proportion used for the Self-Compacting Mortar is **1:1:2** (Cement: SRM: Sand). The water cement ratio w/c used for the study was 0.4 and water to powder ratio w/p was 0.2. Super plasticizer used to achieve the desired flow was **Glenium 51**.

4.2.1 SP demand and Flow Measurement

Glenium 51 was used to achieve the desired flow of 30 ± 1 cm using Hagerman's cone apparatus. Fig 4.23&4.24 shows the SP demand and flow times of SCM having FA and MP as SRMs.



Figure 4.23 SP Demand of SCM having different SRMs



4.2.2 Strength Test on SCM:

Prisms of size **40x40x160cm** (as defined by EN196-1) were tested. Tests were conducted at 1, 3 and 7 days to study the behavior MP and FA in the strength development of SCM. Fig shows the flexural and compressive strength of different formulations



Figure 4.25 Flexural Strength of SCM having different SRMs



Figure 4.26 Compressive Strength of SCM having different SRMs

CHAPTER 5: DISCUSSION

On the basis of results shown in previous chapter, discussion will be made to get useful conclusions. The SRMs having particle size sufficiently smaller than cement particle fills the pores and increases the packing density as a result increases the strength and durability of SCP and SCM. SRMs modifies the properties of the system on the basis of its chemical effect, physical effect, dilution effect and filler effect. [] The physical effect is concerned with providing the nucleation sites for the hydration reaction to proceed. Dilution effect is related to the adsorption or absorption of water in system thus reduces the water cement ratio w/c in the system. Filler effect is related to actually fill the pores in the system by fine powder thus reduces the water requirement of the system because the space initially available for the water is now filled by the finer powders [1], along with this fine powder increase the dry surface area thus more water is required to lubricate the surface of powder. Chemical effect is related to SRMs actually taking part in chemical reaction in the presence of water and calcium hydroxide (Ca(OH)₂) often at later stages (pozzolanic activity). SRMs plays its role with respect to one of these effects or the combination of them. These effects influences the properties of cement paste thus ultimately influence concrete behavior. On the basis of above discussion some results are summarized that includes water demand, SP demand setting time and strength.

5.1 Water Demand and SP demand:

Water demand and SP demand generally depends on the particle size, porosity and nature of SRM used in the system. Water demand and SP demand of Self-Compacting Cementitious Systems with SRMs is generally higher than the control system due the lager surface area. With the addition of finer material in the system the surface area of the dry powder increases thus to lubricate it more water is required. Water demand of MP and FA shows a slight increment from control system due to lager surface area (as describe above). Water demand of BA and HH is sufficiently high due to the nature of particles. BA is a porous material that have the capacity to absorb water and keep it in its structure thus lesser water is available for the hydration reaction, so to proceed the reaction more water is required which results in the increase in the water demand. HH is $CaSO_4.0.5$ H₂O, naturally it exist in the state of hydrated calcium sulfate di-hydrate CaSO₄.2H₂O, so as water is added a portion of water is being held chemically by calcium sulfate to change its state from hemihydrate to di-hydrated thus increases the water demand of the system. The BP due to its clayey and porous nature absorbs a lot of water thus increases the water demand significantly, so as the % of BP increases the water demand increases due to more surface area and more porosity in the system.

In case of blends of SRM, all formulations showed nearly same increase in the water demand from the standard self-compacting cement paste except blend of HH and BA. The reason for this sequence is that in blends different SRMs complement each other thus

showed the nearly equal water demand. Blend of BA and HH showed extra demand for water due to the similar absorption nature of both materials.

SP demand shows nearly the same trend as that of water demand. Certain amount of SP is being adsorbed by the secondary raw materials due to its morphology and surface texture of particles. [84]. SP demand for BA and HH is higher due to the particle size and nature of material as described above. The rest of materials showed slight increment due to increased surface area and surface adsorption of SP.

5.2 Initial and Final setting

Initial and final setting is generally increased with the addition of SRM in the system. Initial and final setting of MP decreases, **MP** contain very high amount of CaCO₃ which is an inert material, so it provide nucleation sites for the hydration reaction thus the setting time decreases. **BA** increases the setting time due to its porous structure it retains the moisture in it thus induce delay in the setting time. **HH** changes the hydration reaction and result in the delay in the setting time. **FA** slightly increase the water demand, it is due to increase in the surface area. When cement is replaced by BP by various % initial and final setting increased dramatically. At 15% final setting time reaches to 390min, this is due to higher porosity and clayey nature of BP. To reduce the setting time of Li₂CO₃ and Na₂CO₃ was used as an accelerator. Li₂CO₃ reduced the setting time appreciably but the texture was observed to be damaged which is not desirable.

5.3 Strength of SCP and SCM

Higher packing density is an attribute of self-compacting cementitious systems. Due to the presence of finer particles in the system, they act like a filler and fills the pores in the matrix which result in higher packing density and resultantly higher strength. At the point of failure, the cracks travels through the pores in the system but with the addition of finer powder in the system which is approximately four to five times smaller than particle of cement itself the pores get filled by these fine particles thus the porosity of the system decreases. As a result larger loads are required to fail the material. This ultimately increases the strength of the material. So by addition of SRMs whose particle size is sufficiently less than that of cement increases the strength of material significantly. Along with the filler action of SRMs pozzolanic activity also plays an important role in developing higher strength. At early age, the strength is mainly due to higher packing density but with the passage of time pozzolanic activity also started its role in strength development. FA gives the maximum strength among the SRMs used due to its highly pozzolanic nature, FA contains higher % of CaO and Fe₂O₃ along with the small amount of SiO₂ so initially FA particles provide the nucleation sites and then at later stages increase the strength by its pozzolanic activity. MP contain higher % of inert CaCO₃ thus mainly increases the strength by its filler effect and by providing the nucleation sites. BA and HH showed lesser strength then control system due to the nature of its particle. BA is highly porous and absorb larger amount of water from the system along with this due to its organic nature its reduced the strength of the SCP when used as 10% replacement. Similarly HH absorb water and delay the strength development due to its sulfate contents. For the BP the strength decreases as the % of BP increases in SCP due the clayey nature it resist the chemical bonding between the cement particles thus reduces the strength.

In SCP with blends of SRMs, all formulations showed higher strength then the control formulation due to the fact that blends complement each other. HH and BA individually did not develop much higher strength but in the case of blend exceptional strength was observed. BA have SiO₂ in excess and smaller amounts of CaO and Fe₂O₃ while HH have CaO in excess so these materials complement each other to produce a formulation that had better pozzolanic activity. Initial development of the strength was slow but at later days strength development was high because initially HH resulted in delayed strength development due to the reasons described above but at later stages pozzolanic activity dominated and resulted in higher strength development. Blend of MP and HH also showed higher strength development, MP initially provided the nucleation sites while strength development occurred at later stages due to filler effect of MP and activity of HH. Although formulations of FA-HH and MP-BA showed good results but lesser than other formulations because of lesser pozzolanic activity and lesser complementation of SRMs. FA and BA showed much higher strength development due to the same reason as that of HH-BA i.e. the formulations complement each other in its properties. FA-MP showed higher strength as initially MP provided nucleation sites and at later stages FA developed the strength due to its pozzolanic activity.

In SCM, both MP and FA showed good development of strength at early ages. The filler effect of these materials resulted in higher performance of mortar. The rate of strength development of MP in SCM started decreasing while for FA it kept increasing.

5.4 Comparison of Results

5.4.1 Comparison with Results of Bilal Sulaman Niazi

Mr. Bilal Sulaman Niazi published his work **"A Study of Self Compacting Cementitious Systems using bagasse ash and Bentonite"** through NUST as a thesis for the requirement of Master of Science in Civil Engineering. In this session his work on bagasse ash is being compared with the present study to deduce some useful conclusions.

5.4.1.1 Basic Comparison:

Mr. Bilal Sulaman Niazi worked on self-compacting cementitous systems using various % of Bagasse ash and Bentonite. For the present comparison the results for the 10% BA is being compared with the results obtained for 10% BA in the present study. **Table 5.1** shows the chemical constituents of OPC and BA used in both studies. **Table 5.2** shows the comparative properties and results of both studies.

	Study (OPC)	Bilal (OPC)	Study (BA)	Bilal (BA)
SO ₃	3.922	2.66	-	0.15
SiO ₂	-	17.15	61.4416	66.23
K ₂ O	1.2937	1.19	19.8403	5.47
CaO	85.6513	64.09	13.7741	2.59
TiO ₂	0.5884	0.32	0.4337	0.12
V2O5	0.1016	-	-	-
MnO	0.1094	-	0.3108	-
Fe ₂ O ₃	8.1545	3.21	4.1995	8.01
SrO	0.1784	-	-	-
Al ₂ O ₃	-	5.6	-	2.35
P2O5	_	0.16	_	2.27
MgO	_	1.74	-	1.97
Na ₂ O	_	1.86	_	0.51

Table 5.1 Basic Chemical Composition

	Present Study	Bilal Niazi	
Particle Size of BA	5 microns	22.36 microns	
Moisture Content	Oven Dried	2.89%	
	25 microns (Bestway Cements	24.8 microns	
Particle Size of C1	Grade 43)	(Fauji Cements Grade 43)	
Mixing Scheme	3min total mixing	3min total mixing	
Water Demand of C1	27%	27%	
Water Demand C1+BA10	33.5%	31.5%	
SP Demand C1+BA10	0.246%	0.19%	
Initial Setting	210 min	207 min	
Final Setting	252 min	236 min	

Table 5.2 Basic Properties and Results

The table 5.1 indicates the comparison between the chemical composition of OPC and BA used in both studies. The OPC used by Mr. Bilal contain verity of constituents it contain appreciable amounts of CaO, Fe_2O_3 and SiO_2 while the OPC used for the present study contain the higher percentage of CaO and Fe_2O_3 with lesser amount of SiO₂. Comparing BA, BA used in the present study contain a verity of materials. It contain the appreciable amounts of SiO₂, CaO and Fe_2O_3 which resulted in more pozzolanic activity of BA than the BA used by Mr. Bilal which contain only SiO₂ in higher quantity thus showed lesser pozzolanic activity. Considering the physical properties of the materials the particle size of OPC used in both studies is nearly same **Table 5.2** while the particle used by Mr. Bilal. Due to this smaller particle size the water requirement of the matrix increases due to the increased surface area as indicated in the **Table 5.2**. For the same reason the SP demand increased for the smaller particle sized material.



Figure 5.1 Compressive Strength of SCP (C1+BA10)

Fig 5.1 indicates the comparative response of SCP (C1+BA10) for the compressive strength. The compressive strength of the sample prepared in the present study had higher strength due to the fact that the BA used had smaller particle size as described earlier. The smaller particle size filled the gaps between the cement particles thus increase the packing density of the material. So due to this higher packing density the strength of the material increased. The chemical composition of the material also indicate that BA used in the present study would also show more pozzolanic activity which is also a reason of more compressive strength than the samples prepared by Mr. Bilal.

5.4.1 Comparison with Results of Ali Raza Khalid

Mr. Ali Raza Khalid published his work "Response of Self Compacting Cementitious Systems (SCCS) using Lime Stone Powder (LSP) and Marble Powder (MP)" through NUST as a thesis for the requirement of Master of Science in Civil Engineering. In this session his work on Marble Powder is being compared with the present study to deduce some useful conclusions.

5.4.1.1 Basic Comparison:

Mr. Ali Raza Khalid worked on self-compacting cementitous systems using various % of Lime Stone Powder (LSP) and Marble Powder (MP). For the present comparison the results for the 10% MP is being compared with the results obtained for 10% MP in the present study. **Table 5.3** shows the chemical constituents of OPC and MP used in both studies. **Table 5.4** shows the comparative properties and results of both studies.

	Study OPC	Ali Raza OPC	Study MP	Ali Raza MP
SO ₃	3.922	4.215	-	0.1
SiO ₂	-	15.437	-	2.033
K ₂ O	1.2937	-	-	-
CaO	85.6513	68.1	99.6262	90.489
TiO ₂	0.5884	-	-	-
V2O5	0.1016	-	-	-
MnO	0.1094	0.055	-	-
Fe ₂ O ₃	8.1545	4.525	0.3738	0.383
SrO	0.1784	0.2421	-	0.067
Al ₂ O ₃	_	3.56	_	0.589
MgO	-	1.929	-	6.233

Table 5.3 Basic Chemical Composition

	Present Study	Ali Raza
Particle Size of MP	6 microns	6 microns
	25 microns	24.8 microns
Particle Size of C1	(Bestway Cements G43)	(Bestway Cements G43)
Mixing Scheme	3min total mixing	3min total mixing
Water Demand of C1	27%	26.5%
SP Demand C1	0.2%	0.15%
Initial and Final Setting of		
C1 (min)	135/155	156/177
T25 and T30 of C1	4.2/15	2.18/14.09
Water Demand C1+MP10	29.75%	28%
SP Demand C1+MP10	0.206%	0.167%
Initial and Final Setting		
C1+MP10 (min)	121/142	167/270
T25 and T30 of C1+MP10	3.1/13.3	2.5/13.25

Table 5.4 Basic Properties and Results

Marble Powder used as a SRM acts as a filler in the SCCS. The chemical composition indicates that the marble powder is mainly CaCO₃. As CaCO₃ is an inert material so it did not take part in the chemical reaction i.e. it is not a pozzolanic material. It just provides the sites for the nucleation sites. Comparing the results the cement used in the present study and of Ali Raza, the cement used in the study contain higher amount of CaO and lack SiO₂ while the cement used by Ali Raza contains verity of components thus the cement used by Ali Raza would give more strength as compared to use in the present study. The particle size of the cement used is nearly same, grade used in both studies was also same so the water demand and SP demand is nearly same while the strength development was different due to difference in the chemical composition of the cement. The **Fig 5.2** shows the comparison of strength development of OPC of Ali Raza and present study.



Figure 5.2 Compressive Strength of OPC

The MP used in the study contain higher amount of CaO while the MP used by Ali Raza contain smaller amounts of other materials but the materials did not supposed to play any role in strength development. MP just act as a filler material and increased the strength by the increasing the density of the material. **Fig 5.3** shows the comparison of the strength development of SCP with 10% MP as SRM. The difference in the strength development is basically due to the role of OPC as described before. SRM just act as a filler in this case and do not play role in strength development other than providing the site for the nucleation.



Figure 5.3 Compressive Strength of C1+MP10

CHAPTER 6: CONCLUSIONS

On the basis of the results and discussions in the previous chapters following conclusion could be drawn

- Fly Ash (FA) give better results than all other SRMs when used as 10% replacement in SCP due to its finer particle size and chemical composition which results in its pozzolanic activity.
- When use as 10% replacement in Self Compacting Paste Marble Powder (MP) give better results due to its finer particle it provide nucleation sites and increases the packing density thus increases the strength of the material.
- **Brick Powder (BP)** due to its porous particle and clayey nature result in higher water demand and prolonged setting time resultantly do not help in strength development.
- **Bagasse ash (BA)** and **Hemihydrate (HH)** individually did not play role in strength development in self-compacting paste (SCP) but as a blend showed appreciable strength development by complementing each other in physical and chemical properties.
- Blend of Marble Powder/Hemihydrate (MP-HH), Fly ash/Bagasse ash (FA-BA) and Fly ash/Marble Powder (FA-MP) showed appreciable results due to the complementing nature of SRMs.
- More strength development could be achieve using the **Bagasse ash (BA)** with smaller particle size (**Table 5.2**)

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ANNEXURE-A

MATERIALS

Oxides	Cement Oxide	Hemihydrate Oxide	Bagasse Ash Oxide	Marble Powder Oxide	Fly ash	Brick Powder
SO ₃	3.922	49.2482	-	-	-	12.9416
SiO ₂	-	-	61.4416	-	4.3111	-
K ₂ O	1.2937	-	19.8403	-	4.6617	3.2185
CaO	85.6513	50.3131	13.7741	99.6262	29.3679	17.2863
TiO ₂	0.5884	-	0.4337	-	4.3662	2.2630
V2O5	0.1016	-	-	-	0.1171	-
MnO	0.1094	-	0.3108	-	0.3212	0.2545
Fe ₂ O ₃	8.1545	0.3034	4.1995	0.3738	53.0545	60.7952
SrO	0.1784	0.1353	-	-	2.2060	0.9033
Cr ₂ O ₃	-	-	-	-	-	0.1656
ZnO	-	-	-	-	0.4647	1.9399
Rb ₂ O	-	-	-	-	0.1252	0.1340
MoO ₃	-	-	-	-	-	0.0980
CuO	-	-	-	-	0.1831	-
As ₂ O ₃	-	-	-	-	0.1141	-
NiO	-	-	-	-	0.2280	-
ZrO ₂	-	-	-	-	0.4792	-
Particle size microns	25	8	5	6	8	13
Physical Appearance	Greenish Grey Powder	Yellowish white Powder	Dark Black light weight Powder	Bright white silky fine Powder	Greyish fine Powder	Reddish Brown Powder

Table 1: Physical and Chemical Properties of Materials

ANNEXURE-B

SELF COMPACTING PASTE

• Self-Compacting Paste with 10% SRM

Formulations	Water Demand %
C1-0	27
C1-MP10	29.75
C1-BA10	33.5
С1-НН10	33.25
C1-FA10	29.75
C1 DD10	22.25

Table B-1 Water Demand of SCP with different SRMs

Formulation	Initial Setting	Final Setting
rormulation	(min)	(min)
CEM1-27	135	155
CEM1+10MP-29.75	121	142
CEM1+10BA-33.5	210	260
CEM1+10Hemi-33.25	105	170
CEM1+10FA-29.75	150	190
CEM1+10BP-33.25	210	300

Table B-2 Initial and Final Setting of SCP with different SRMs

Formulation	SP Demand%
C1-0-27	0.2
C1-MP10-29.75	0.206
C1-BA10-33.5	0.246
С1-НН10-33.25	0.256
C1-FA10-29.75	0.227
C1-BP10-33.25	0.225

Table B-3 SP Demand of SCP with different SRMs

Formulations	T25 (sec)	T30(sec)
C1-0	4.5	15
C1-MP10	3.1	13.3
C1-BA10	3.2	11.5
С1-НН10	3.5	12.7
C1-FA10	4.3	14.5
C1-BP10	4.6	16.3

Table B-4 Flow Times of SCP with different SRMs

Formulations	1Day	3Day	7Day	21Day	28Day
CEM1-27	1.406	13.828	15.703	15.9375	15.70313
CEM1+10MP-29.75	1.875	12.656	12.656	14.29688	15.46875
CEM1+10BA-33.5	2.109	3.281	7.734	11.95313	12.18975
CEM1+10Hemi-33.25	2.109	2.578	7.266	11.48438	12.1875
CEM1+10FA-29.75	11.25	12.656	13.593	15	21.09375
CEM1+10BP-33.25	6.796	8.437	10.781	12.1875	12.1875

Table B-5 Flexural Strength (N/mm²) of SCP with different SRMs

Formulations	1Day	3Day	7Day	21Day	28Day
CEM1-27	33.3	41.3	48.4	49.7	52.1
CEM1+10MP-29.75	30.9	37.8	45.9	50.8	54.5
CEM1+10BA-33.5	21.7	36	40.6	43.3	46.8
CEM1+10Hemi-33.25	24	29	38	40.3	45.4
CEM1+10FA-29.75	34.3	42.6	55.3	61	74.9
CEM1+10BP-33.25	21.1	25.6	36.3	37.6	52

Table B-6 Compressive Strength (N/mm²) of SCP with different SRMs

• Self-Compacting Paste with blends of SRM

Formulation	Water Demand %
C1-FA5-MP5	29.5
C1-FA5-BA5	31
C1-FA5-HH5	31
C1-MP5-BA5	31
C1-MP5-HH5	31
С1-НН5-ВА5	33

Table B-7 Water Demand of SCP with blend of SRMs

	Initial Setting	Final Setting
Formulation	(min)	(min)
C1-FA5-MP5	150	185
C1-FA5-BA5	185	220
С1-FA5-HH5	170	220
C1-MP5-BA5	160	200
C1-MP5-HH5	135	212
С1-НН5-ВА5	180	238

Table B-8 Setting Time of SCP with blend of SRMs

Formulation	SP Demand %
C1-FA5-MP5	0.159
C1-FA5-BA5	0.2016
C1-FA5-HH5	0.194
C1-MP5-BA5	0.2016
C1-MP5-HH5	0.1984
C1-HH5-BA5	0.1952

Table B-9 SP Demand of SCP with blend of SRMs

Formulation	T25(sec)	T30(sec)
C1-FA5-MP5	2.8	11.2
C1-FA5-BA5	2.2	10.3
C1-FA5-HH5	3.1	11.5
C1-MP5-BA5	5	14
С1-МР5-НН5	2.5	10.2
C1-HH5-BA5	3	13

Table B-10 Flow Times of SCP with blend of SRMs

Formulation	1Day	3Day	7Day	21Day	28Day
C1-FA5-MP5	1.968	3.75	3.211	3.56	3.703
C1-FA5-BA5	1.875	2.953	3.984	4.17	4.289
C1-FA5-HH5	1.148	2.016	2.484	3.07	3.492
C1-MP5-BA5	1.921	3.35	3.234	3.328	3.421
C1-MP5-HH5	1.757	2.461	2.766	3.797	4.031
C1-HH5-BA5	1.383	2.953	2.86	3.093	3.351

Table B-11 Flexural Strength (N/mm²) of SCP with blend of SRMs

Formulation	1Day	3Day	7Day	21Day	28Day
C1-FA5-MP5	24.29	38.28	58.48	67.5	72.05
C1-FA5-BA5	21.07	40.85	54.1	68.3	75.38
C1-FA5-HH5	15.61	38.93	42.3	53.6	60.42
C1-MP5-BA5	19.38	37.26	42.84	53.6	60.42
C1-MP5-HH5	18.18	35	43.9	70	81.16
C1-HH5-BA5	14.83	27.94	36.03	71.51	75.73

Table B-12 Compressive Strength (N/mm²) of SCP with blend of SRMs

• Self-Compacting Paste with Brick Powder as SRM

Formulation	Water Demand %
C1+BP5	31
C1+BP10	33.25
C1+BP15	35.7

Table B-13 Water Demand of SCP with BP as SRM

Formulation	Initial Setting (min)	Final Setting (min)
C1+5BP	182	262
C1+10BP	210	300
C1+15BP	298	391

Table B-14 Setting Time of SCP with BP as SRM

Formulation	SP Demand %
C1-BP5-31	0.21
C1-BP10-33.25	0.225
C1-BP15-35.7	0.245

Table B-15 SP Demand of SCP with BP as SRM

Formulation	T25 (sec)	T30 (sec)
C1-BP5	3.8	12.4
C1-BP10	4.6	16.3
C1-BP15	5.5	18.4

Table B-16 Flow Times of SCP with BP as SRM

Formulation	1Day	3Day	7Day	21Day	28Day
CEM1+5BP-31	5.625	12.18	13.36	16.41	15
CEM1+10BP-33.25	6.797	8.44	10.78	12.18	12.19
CEM1+15BP-35.7	4.22	8.91	10.3	11.25	11.25

Table B-17 Flexural Strength (N/mm²) of SCP with BP as SRM

Formulation	1Day	3Day	7Day	21Day	28Day
C1+5BP	21.9	29.8	35.2	59.6	63.8
C1+10BP	21.1	25.6	36.3	47.2	52
C1+15BP	14.9	20.5	31.8	37.4	39.5

Table B-18 Compressive Strength (N/mm²) of SCP with BP as SRM

• Comparison of SCP

Day	Study	Bilal
1	21.7	16.56
3	36	24.46
7	40.6	35.37
28	46.8	38.9

Table B-19 Compressive Strength (N/mm²) of SCP with BA as SRM

Day	Study	Ali Raza
1	33.3	41.55
3	41.3	45.88
7	48.4	50.57
28	52.1	79.84

Table B-20 Compressive Strength (N/mm²) of SCP (C1)

Day	Study	Ali Raza
1	30.9	44.12
3	37.8	51.68
7	45.9	63.7
28	54.5	74.11

Table B-21 Compressive Strength (N/mm²) of SCP with MP10
ANNEXURE-C

SELF COMPACTING MORTAR

• Self-Compacting Mortar with FA and MP as SRMs

	SP Demand	
Formulation	%	
C1+MP+Sand	3.6	
C1+FA+Sand	3.5	

Table C-1 SP Demand of SCM with SRMs

	T25	T30
Formulation	(sec)	(sec)
C1+MP+Sand	5.5	24
C1+FA+Sand	12	45

Table C-2 SP Demand of SCM with SRMs

Formulation	1Day	3Day	7Day
C1+MP+Sand	1.148	3.141	3.305
C1+FA+Sand	1.054	2.555	3.281

Table C-3 Flexural Strength (N/mm²) of SCM with SRMs

Formulation	1Day	3Day	7Day
C1+MP+Sand	24.62	50.06	53.13
C1+FA+Sand	19.99	44.31	54.85

Table C-4 Compressive Strength (N/mm²) of SCM with SRMs

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