

Use of Lime, Marble Powder and Bagasse Ash as SRMs in SCP Systems.

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Submitted By

Wajahat Ali	NUST-BE-CE-2010-198
Ibtsam ur Rehman Kiani	NUST-BE-CE-2010-57
Muneeb Khurshid	NUST-BE-CE-2010-132
Muhammad Shoaib	NUST-BE-CE-2010-116
Tariq Rehman	NUST-BE-CE-2010-186

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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)

Dedication

We dedicate this research work to our families

This is to certify that

Thesis titled

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Wajahat Ali	NUST-BE-CE-2010-198
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Muneeb Khurshid	NUST-BE-CE-2010-132
Muhammad Shoaib	NUST-BE-CE-2010-116
Tariq Rehman	NUST-BE-CE-2010-186

Has been accepted towards the partial fulfillment

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(Prof. Dr. Syed Ali Rizwan, PhD)

Head of Structural Engineering Department

NUST Institute of Civil Engineering

School of Civil Engineering and Environment

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Abstract

This study is focused on analyzing the effect of some of the local SRMs which include Lime, Bagasse Ash (BA) and Marble Powder (MP) on the self compacting paste systems (SCP). These materials, if left un-utilized pose detrimental effects to the environment, since general practice is to dump these materials in landfills and drains, which end up clogging the drains. Their use in concrete systems as SRMs helps in their effective disposal and also brings in economy to the system. Cement production is one of the major cause of environmental pollution in the modern era, researches are being carried out worldwide to search for alternate materials to the cement to reduce the environmental pollution, SRMs play an important role in this regard as partial replacements of cements. These SRMs possess in themselves little or no cementitious properties, but in their finely grounded form and in the presence of water they combine with calcium-hydroxide of cement based systems to produce additional Calcium Silicate Hydrate (CSH) products.

The parameters studied include secondary raw material's particle characterization, flow behavior, strength development, X-Ray Florescence (XRF) and micro structural analysis by scanning electron microscopy (SEM) of self compacting paste systems.

The maximum strength at 28 & 56 days is obtained for the formulation containing 10% replacement of marble powder. The SP demand to achieve the target spread was least for SCP formulation containing 10% replacement of marble powder. Lime at 10% replacement exhibited similar response as of neat cement. Bagasse Ash at 10% replacement showed lowest strength of all of other formulations used. Particle size of SRMs plays an important role in determining packing density, strength, workability and setting times.

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List of Notations and Abbreviations

ACI	American Concrete Institute
ASTM	American Society for Testing of materials
SCCS	Self-Compacting Cementitious systems
SCP	Self Compacting Paste
MP	Marble Powder
LI	Lime
SCBA	Sugarcane Bagasse Ash
SRM	Secondary Raw Materials
SEM	Scanning Electron Microscopy
SP	Superplasticizer
W/C	Water to Cement Ratio
WD	Water Demand
CSH	Calcium Silicate Hydrate
CH	Calcium Hydroxide
XRF	X-Ray Florescence

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Chapter 1- Introduction and Literature Review

1.1. General

Different problems like honeycombing, bleeding, segregation, pouring of concrete in heavily reinforced columns and beams of moment resisting frames that are used in seismic areas occur mainly due to some properties of ordinary concrete like fluidity etc. Also to make concrete flow through the reinforcement, proper vibration for the critical sections is required. Standard vibration techniques are not properly implemented which make structure unsafe. So the solution is, a concrete that is easily compactible with its own weight, is flow-able and can provide high deformations without segregation and bleeding[14]. A concrete that could flow and also could fill the formwork completely with reinforcement present without any needed any vibrations. These properties are only manifested by the Self Compacting Concrete (SCC).

Self Compacting Cementitious Systems (SCCS) systems provide uniform compaction and durability are characterized by their high w/p ratio, thus encouraging the use of locally available secondary raw materials SRMs. In high performance concrete (HPC) systems, all cement particles do not get hydrated, therefore secondary raw materials SRMs are used to replace some percentage of cement in order to economize and make an environmentally compatible concrete with improved micro-structure which would manifest an improved overall response in terms of fresh and hardened properties .These systems have wide and varied applications such as tunnel linings, bridge piers, transportation structures, densely reinforced sections of concrete and repairs of structures.

Self compacting concrete SCC, a kind of high performance concrete HPC with excellent deformation and segregation resistance, was first developed in Japan in late 1980's. Due to its versatile nature and wide range of applications, the use of self compacting concrete spread throughout the world very rapidly. However, due to non availability of chemicals and mineral admixtures at cheap rates and unclear understanding of basic concepts underlying the manufacture of self compacting concrete, its application is very limited. The paste phase of self compacting cementitious systems plays the most important role in service life performance of the structure and acts as a vehicle for transport of aggregate phase during flow. This phase mainly consists of cement, secondary raw materials and

admixtures and the study of paste phase is the first essential step towards successful production of Self Compacting Concrete using local Secondary Raw Materials.

ACI defines SCC as High Performance Concrete that meets special performance and uniformity requirements that cannot be achieved otherwise using conventional materials and practices.[18]

SCC includes powder and super-plasticizers as main ingredients along with the conventional ingredients of concrete. Addition of SP ensures high deformation without bleeding and segregation. SP also has a beneficial effect on the packing density of the system [16]. In modern concrete due to low w/c ratio all grains of cement may not be fully hydrated, so cement is replaced partially by secondary raw materials typically in the range of 5 μ m-10 μ m for improving packing density and for increased pozzolanic activity. [19]

1.3. Self Compacting Cementitious Systems SCCS:

According to a definition for SCC currently being considered by ACI committee 237R-07 “Self-consolidating concrete is the kind of concrete exhibiting high deformability while maintain resistance to segregation”.

ACI defines SCC as high performance concrete that meets special performance and uniformity requirements that cannot be achieved otherwise using conventional materials and practices. It is a highly flow-able, non-segregating concrete that can spread into places, fill the formwork and encapsulate the reinforcement without any mechanical consolidation.

SCC can be characterized by three properties, as follows: [20]

- **Passing Ability:** The ability of SCC to flow through tight openings such as spaces between steel reinforcing bars without segregation or blocking.
- **Flow Ability:** The ability of SCC to flow into and fill completely all spaces within the formwork, under its own weight
- **Segregation Resistance:** The ability of SCC to remain homogeneous in composition during transport and placing.

Along with cement aggregate and water, there are certain other materials that must be used in the mix to provide for the self-compaction of the system under the lone force of gravity. Those materials are the Mineral and Chemical admixtures which provide for the increased workability while

maintaining the reduced w/c ratio as well as increase the durability of the system by increasing the particle bonding and reducing the permeability of the system.

The replacement of cement by mineral admixture as secondary raw material account for bringing in economy to the system by reducing the quantity of cement. The SRMs typically lie in the size range of 5 μ m to 10 μ m for SCCS [19], thereby improving the pozzolanic activity and packing density, hence accounting for improved micro structure.

Chemical Admixtures prolong the setting times of the SCP in addition to providing for the increased resistance to segregation during transportation & placement and enhance the fluidity of the SCC for the same w/c ratio. [14,15]

1.4. Secondary Raw Materials (SRMs)

Secondary Raw Materials (SRMs) or Supplementary Cementitious Materials are waste or by products of industries that require no or less processing prior to their usage as cement replacements. There are basically two reasons in using the SRM's. One of them is related to the improvement in properties of concrete and second is related to the cost involved in the process of production of cement. Nearly 7% of total Carbon dioxide (CO₂) emission is due to cement industries and these consume 2-3% of the global energy. Efforts are made nowadays for energy efficient building, energy efficient materials so as to reduce the consumption of energy as much as possible. So addition of SRM's serves the purpose of energy efficiency because they require less processing and also are environment friendly.

Addition of SRMs doesn't affect the properties of cement but improves certain properties like workability, durability, strength and setting time etc. These SRMs enhance the response in fresh and hardened state. SRMs follow two mechanisms to enhance the response of an SCC system and those are "the filler effect" and "the pozzolanic activity". Even inert materials blended with cement may have a significant effect on the hydration of the clinker phases. This is usually referred to as a filler effect. The reactivity of most SCMs is highly dependent on the alkalinity of the pore solution, which builds up over the first few days. Consequently the extent of reaction of SCMs in the first day or so is usually negligible and changes in hydration kinetics are dominated by the filler effect. There seem to be two principal mechanisms contributing to the filler effect: [20]

- **Extra space:** As the filler does not produce hydrates, at the same water to solids ratio, the water to clinker ratio is higher and there is more space for the hydration products of the clinker phases.
- **Enhanced nucleation:** Particularly for fine materials, the extra surface provided by the SCMs acts as nucleation sites for the hydration products of the clinker phases.

Hydration process is believed to be followed by three mechanisms. Those are as follow:

- **Nucleation and Growth Phase (NG):**
- **Interaction Boundary Phase (I):**
- **Diffusion Mechanism (D):**

According to numerous data obtained by several investigators it is presupposed that the processes proceed one after other according to the following scheme: [54]



Marble powder (MP), Bagasse ash (BA) and lime (LI) were used as SRMs in this study. MP is an inert material so it provides filler effect. Whereas, BA enhances the response by pozzolanic activity. LI also incorporates its pozzolanic effect and enhances the response of the system.

1.5. Superplasticizers

These are chemical admixtures which improve the workability of SCC systems at lower WC ratios and also improve the microstructure and the durability of the concrete. They can reduce the water requirement by 30%. It improves the durability as workability requirement is fulfilled at lower WC ratio so lesser will be the water and lesser empty spaces and capillary pores would be there. This will resist other harmful substances like Carbon dioxide etc to enter into the structure and hence durability is improved.

SPs had effects on both fresh and hardened properties of system. It effect the setting time of system. That is why it is advised to use it with great care.

1.5.1. Action of Superplasticizers

Cement particles act as a colloid in suspension and entraps the water particle. Basic clinker phases in cement are C₂S, C₃S, C₃A and C₄AF. C₂S and C₃S have negative zeta potential while C₃A and C₄AF have positive zeta potential. This accelerates the coagulation process of the cement grain. So SPs retard that coagulation and flocculation process because they have very high negative zeta potential which promotes the dispersion of silicate phases and this dispersion also have negative zeta potential that hinders flocculation process. [20, 21, 23]

1.6. Cement Chemistry

“ASTM C-150 defines Portland cement as "hydraulic cement (cement that not only hardens by reacting with water but also forms a water-resistant product) produced by pulverizing clinkers consisting essentially of hydraulic calcium silicates, usually containing one or more of the forms of calcium sulphate as an intern ground addition". The setting and hardening properties of concrete are determined by the chemical reaction caused after the contact that arises between water and cement. The rate of heat of hydration of cement plays an important role in determining concrete strength and durability. On coming in contact with water hydration of cement starts. Hydration of cement is defined as “dissolution – precipitation process between binder grains and water” [25]. It is a process by which cement powder plus water transform from a fluid suspension to a porous solid within hours and strength gains over days and months [26]. It is generally believed that two types of reaction take place in hydration process known as through-solution hydration and solid-state hydration. The earlier process involves the dissolution of anhydrous compounds to their ionic constituents, formation of hydrates in the solution, and eventual precipitation of hydrates whereas the later deals with the hydration that takes place directly at the surface of the anhydrous cement compounds without the compounds going into solution.[27]”

The major constituents of the Ordinary Portland Cement (OPC) are tricalcium silicate (C₃S), dicalcium silicate (C₂S), tricalcium aluminate (C₃A) and tetracalcium aluminoferrite (C₄AF). These

constituents react with water to form various hydration products such as main hydration product of C-S-H (Calcium Silicate Hydrate), portlandite (CaOH_2), ettringite (C-A-S-H, calcium sulfoaluminate hydrate) and calcium monosulphoaluminate ($\text{C}_4\text{ASH}_{12}$) [28]. C_3S is responsible for early high strength of hydrated Portland cement gained through its early age upto 7 days. C_2S is responsible for the later developing strength of Portland cement paste and gains little strength until even 28days but its final strength is the same as that of C_3S . C_3A exhibits the flash set when hydrated, responsible for initial set after addition of gypsum in small amount (5%) and releases large amount of heat upon hydration. It shows little strength increase after 1day. Increase in amount of C_3A results in faster set. C_4AF behaves like C_3A in which it hydrates rapidly and gains low strength but does not exhibit flash set.

As a result of dissolution/precipitation process a growth of hydration products occurs, Ettringite forms at the earliest and is detectable with 8-9 mins. It's a needle shape product and is the result of C_3A phase reaction. It is also known as Calcium Sulfoaluminate Hydrate (C-A-S-H). CH and C-S-H gel are the products which are associated with C_3S phase. Calcium Hydroxide (CH) is a well-defined crystal while Calcium Silicate Hydrate (C-S-H) is a poorly crystalline material. C-S-H gel can be further classified as High Density C-S-H Gel and Low Density C-S-H Gel. Former one is desirable whereas as later one is the cause of creep and shrinkage. Only high density C-S-H gel can be seen from SEM [29].

1.7. Research Objectives

The objectives of this research conducted on SCP systems were

1. To see the response of SCCS systems using following SRMs
 - a. Marble Powder (MP).
 - b. Lime (LI).
 - c. Bagasse Ash (BA).
2. To study the fresh and hardened properties of the SCP system by incorporating the above mentioned SRMs.
3. To look for the effective SRMs in scenario of the local conditions in terms of availability of SRMs.

Chapter No 2 - Secondary Raw Materials.

2.1. Marble Powder:

Stone industry is a vital factor in the worldwide economy of today. While processing the stone, huge amount of residues is produced with different dimension particle size and texture. Marble powder is a product of the Marble Industry which is produced in abundance all over the world. It is basically the waste of the marble industry and studies are being carried out on how to use this waste effectively as it poses damages to the environment if dumped in such huge amounts.[31]

There are two types of stone processing waste i.e. solid waste and semi liquid or slurry (Almeida et al.,2007) [31]. During the processing of marble gang saws are used along with water that acts as a coolant, so the powder flows along with it as waste marble slurry. The sludge generated is almost equal to 20-30% of the weight of the stone worked (Bertolini et al., 1990) [32] depending upon the process. These residues are then deposited in land fills hence rendered useless and harmful to the environment.

Marble powder from different marble industries and markets was bought and analyzed through visual inspection. It was observed that most of the MP from industrial waste had indigenous impurities and showed textured appearance, i.e. traces of black and copper colored impurities characteristic to the iron oxides and many other. After thorough inspection the best sample with the lowest content of impurities as analyzed by visual inspection was selected and has been used in the study. However the particle size of the selected sample was much coarser than the one intended to be used in the project, so the MP sample was properly milled to a size of 1 Micron. Milling services were sought from a local vendor near Islamabad.

Marble powder acts as a non-pozzolanic inert filler in the SCC paste, and its properties resemble to those of limestone, as obvious from the fact that geologists also call 'metamorphised limestone'. So due to such characteristic properties it contributes to the early hydration of the paste and subject to its very fine size acts as an excellent filler material providing better packing density.

2.2. Bagasse Ash:

Bagasse is an abundant waste produced in the sugar industries, when all the economical sugar is extracted from sugar cane it leaves 40-45% fibrous residues. These residues are then burnt in

the incinerators to generate energy due to their high calorific value, after burning of this residue the ash remains which is often called a bagasse ash. [35]

Bagasse ash has recently been accepted as a pozzolanic material, however very limited research data is available on the effects of using bagasse ash as a partial replacement of cement. A pozzolana is a material, occurring either naturally or artificially, and which contains silica, iron and aluminium ions. According to ASTM C618 (1992), a pozzolana is defined as a siliceous or siliceous and aluminous material which in itself possess little or no cementitious value, but one which in finely divided form and in the presence of moisture, will chemically react with calcium hydroxide at ordinary temperatures to form compounds possessing cementitious properties.[36]

Amorphous silica that is found in some pozzolanic materials (Habeeb et al., 2009), React with lime more readily than those of crystalline form. The most essential asset of SCBA that identifies its pozzolanic activity is the amorphous phase substance. The production of SCBA can lead to the formation of approximately 60% to 75% by weight of amorphous silica (Srinivasan et al., 2010).

According to Ernesto et al [3] BA can become pozzolanically active when burnt in temperature range of 800-1000C and products formed during pozzolanic reaction are not crystalline compounds.

Bagasse ash can be characterized as a pozzolanic material but its activity depends significantly on its particle size, its fineness and compressive strength of cementitious system containing bagasse ash is inversely proportional to its particle size. {2}

Plastic viscosity increases with addition of bagasse ash. However yield strength always decreased when bagasse ash was used which suggests positive effect of BA on concrete rheology. Then negligible of BA contributes to better rheology as concrete with higher carbon content requires more amount of SP for same workability. However no significant effect was noted on compressive strength of concrete with small addition of bagasse ash(10%) but a negative trend was observed with increase in BA content(beyond 10%)

Bagasse ash used for the study was bought from the local sugar factories, which was then oven dried at 100oC for one day to remove moisture. The dry bagasse then was grounded to a size of 10 microns so that its pozzolanic behavior may become obvious. Bagasse used in this study has been kept under controlled conditions in oven so that it may not absorb any moisture from the environment. The chemical composition of SCBA was determined using X-Ray fluorescence technique, XRF, the results

are given in Table 1. The major oxide observed in SCBA is silica (SiO₂). The X-Ray Diffraction (XRF) analysis of the SCBA shows the amorphous silica formation with traces of low quartz as shown in Fig. 1.

Based on the various studies conducted, it can be succinctly concluded that SCBA is a good pozzolana for concrete cementation and partial blends of it with OPC could give good strength development and other engineering properties in concrete. An optimum of 10% SCBA blend with OPC could be used for reinforced concrete with dense aggregate.

2.3. Lime:

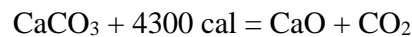
Lime is a general term that includes various forms of lime which include hydrated lime, quick lime and hydraulic lime. It may be high-calcium, magnesium or dolomite. In recent times cement has replaced use of lime to a great extent. Though cement is a remarkable material of construction yet lime has some advantageous properties like good workability, plasticity, less shrinkage on drying, durability and above all is cheap.

2.3.1 Physical forms of Lime:

Lime is available in two physical form;

Quick lime:

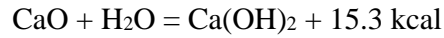
This is the substance left immediately after heating of limestone to redness in air, a process known as calcination of limestone. It is also defined as calcined limestone, the major parts of which are calcium oxide or calcium oxide in association with magnesium oxide, capable of slaking with water.



Hydrated lime.

This is the substance left after pouring of water on quicklime a process known as slaking. It is usually ground to the powder form. It is also defines as a dry powder obtained by treating quicklime with water enough to satisfy its chemical affinity for water under the conditions of its hydration. It

consists essentially of calcium hydroxide or a mixture of calcium hydroxide and magnesium oxide or magnesium hydroxide or both.



2.3.2. Lime Classification:

Lime is usually divided into three classes according to chemical composition. In many books only two have been mentioned i.e. fat lime and hydraulic lime ignoring lean lime [47].

- a. Fat lime. It is obtained by calcination of nearly pure limestone, chalk or seashells. Addition of water to quick lime produces heat and increase volume two to three times. It is slow setting, takes long to harden and contain less than 5% impurities like silica and alumina. It is also termed as rich, pure or high calcium lime.
- b. Lean lime. It contains more than 5% of clayey impurities because of which it takes longer to slake than the fat lime. It sets and hardens slowly. It is used both for plaster and mortar.
- c. Hydraulic lime. It is obtained by calcination of limestone possessing appreciable quantity of silica and alumina in the form of clay. On calcination carbon dioxide escapes leaving behind quicklime which reacts with silica and alumina forming silicates and aluminates of lime. On addition of water to finally ground lime, chemical action starts among constituents resulting in setting and hardening. It possesses less tendency to shrink.

2.3.3. Lime Specification:

ASTM-C-821-78 (Reapproved 1995)[49] specifies requirement of Lime for use with pozzolans. This covers all types of commercial hydrated lime such as high calcium, magnesium, or dolomite-hydrated lime. By-product limes and slaked quick limes in dry, wet, or slurried forms are also included.

- a. Chemical and Physical Requirements. The lime shall conform to the requirements listed in Table 2.1.
- b. Performance Requirements. If the chemical factor, Blaine fineness, or pozzolanic receptivity index is below the limits give in Table 2-1; the lime shall be required to meet the applicable performance requirements listed in Table 2-2. Where such lime complies with the limits listed in Table 2.2, it shall be considered acceptable for use with pozzolans.

However, it may be necessary to increase the lime content in the lime-pozzolan mixtures in order to meet minimum design criteria.

Chemical Factor	50
Blaine Fineness, min, cm ² /g	10,000
Pozzolanic Receptivity Index, min	100

Table 2. 1 Chemical and Physical Requirements

Plastic Composition	
Lime Pozzolan Strength, min, psi (Mpa)	600 (4.13)
Non-plastic Composition	
Lime Pozzolan Agregate Strength, min, psi (Mpa)	400 (2.76)
Vacuum Saturation Strength, min, psi (Mpa)	400 (2.8)

Table 2. 2 Performance Requirements

CHAPTER 3 - Experimental Program.

3.1 GENERAL:

This chapter deals with the results of experimental program that were conducted on the materials to study their behavior. Secondary raw materials which are used in the experimentation are first milled to required size. Consistency of system at 10% replacement and also at 5% replacement, particle characterization and super plasticizer content is also found. Standard molds are casted and tested at specified age of 1, 3, 7, 14, 21, 28, 56 days. Scanning Electron Microscopy (SEM), X-ray fluorescence (XRF), particle size analysis are also being conducted on the materials that are used in the project.

3.2 Materials:

3.2.1 Cement:

'Bestway' Ordinary Portland Cement OPC, grade 43, type 1, manufactured in Pakistan, conforming to ASTM standard C150-04 having average particle size $15.41\mu\text{m}$ was used.

3.2.2 Secondary Raw Materials (SRMs)

Secondary raw materials used for this experimentation consist of:

- Mable Powder (MP) which is being bought from a factory located at Sanjhani, Taxila
- Bagasse Ash (BA) which is taken from Structure lab, NICE, NUST.
- Lime (LI) which is bought from local shop located in sector G-11

3.2.3 Super Plasticizer Used:

Super plasticizer used is a third generation SP, named as Melflux2651-F.

Water demand and setting time were determined using standard VICAT apparatus in accordance with ASTM C-187. C191 and BS 12:1978 for setting time.

Different trial specimens with varying specimens of water were prepared. When plunger penetrated to point 5 ± 1 mm from bottom of VICAT mold on scale, the percentage of water used to prepare the mold was noted. Water was expressed as percentage by weight of dry cement. Similarly

for setting time, periodic penetration tests were performed by allowing VICAT needle to settle into paste. The initial setting time was noted when penetration was 5 ± 1 mm deep. Final setting time was noted when needle could not leave a complete circular impression in paste surface.

3.2.4. Grinding/Milling of SRMs:

The SRMs in their original sizes were too coarse to be used as an SRM in the paste and to assume the properties of a pozzolan, so all the SRMs i.e. Marble Powder, Lime and Baggasse Ash were subjected to milling and grinding process.

Marble powder's grinding was outsourced from a grinding mill in the vicinity of Islamabad, however the other materials were ground by Los Angeles Abrasion Machine was employed and for further reduction in the particle size, grinder was used to achieve the required particle size i.e. of 8 to 9 microns.

3.3. Formulation:

7 Formulations were studied with different SRMs described earlier and with different percentages of them. These formulations were as follows;

1. C-O-WD
2. C-MP-10%-WD
3. C-MP-10%-WD
4. C-MP-10%-WD
5. C-MP-5%-BA-5%-WD
6. C-LI-5%-BA-5%-WD
7. C-MP-5%-LI-5%-WD

The formulation code can be comprehended as, C represents Cement i.e OPC, SRM represents the Secondary Raw Material and the %age represents the Replacement content. i.e.

Type 1: C-SRM-10%--WD

Type 2: C-SRM1 (5%)-SRM2 (5%)--WD

3.4. Mixing Regime:

Materials i.e. Cement SRM and the superplasticiser are first dry mixed by hand in a bowl until the mix appeared to be uniform, then this mix was poured into the bowl of Hobart Mixer where weighed quantity of water depending upon the water demand of each mix was added. Materials were mixed in two steps in the Hobart Mixer, i.e., firstly material was mixed at slow speed at 145 rpm for 30 seconds and then for the next 30 seconds the walls of the bowl were cleaned with the help of spatula and after that it was mixed for 90 seconds at High speed of 285 RPM. Thus the total time of mixing was 3 minutes for each of the formulation.

3.5. Physical and Chemical Analysis:

3.5.1. X-ray fluorescence (XRF).

X-ray fluorescence (XRF) is the emission of characteristic "secondary" (or fluorescent) X-rays from a material that has been excited by bombarding with high-energy X-rays or gamma rays. The phenomenon is widely used for elemental analysis and chemical analysis, particularly in the investigation of metals, glass, ceramics and building materials, and for research in geochemistry, forensic science and archaeology.

XRF tests services were taken from NUST IESE where XRF tests were conducted on each material to analyze their physical and chemical composition. Particle Size analysis services were taken from NUST SCME.

<u>Parameters</u>	<u>Cement</u>	<u>Marble Powder</u>	<u>Bagasse Ash</u>	<u>Lime</u>
Particle Size	16.0253	1.1654	17.2050	46.5
<u>Chemical Analysis</u>				
Silicon Di-oxide	--	--	66.2992	--
Ferric Oxide	9.5187	0.2166	6.7046	0.1344
Calcium Oxide	83.8	99.6787	11.2766	99.1125
Sulphur Trioxide	3.7278	--	--	--
Pottassium Oxide	1.8069	--	14.7627	--
Manganese Oxide	0.0979	--	0.3064	0.0688
Stontium Oxide	0.2333	0.1047	0.1158	0.6843
Zinc Oxide	--	--	0.5346	--

3.5.2. Scanning Electron Microscopy (SEM):

Scanning electron microscopy is a very useful method to analyze the micro-structure and the hydration progress at different stages. In concrete it can reveal some of the very important information about degree of hydration of cement, formation and distribution of hydration products, adhesion to aggregates and homogeneity of cement. On the whole it is very useful tool in determining and characterizing the bonding arrangement in which different material particles bond with each other. SEM analysis was performed on jed-2300 Scanning Electron Microscope where samples of all the materials used i.e. lime, marble powder, bagasse ash and cement were put under test and were analyzed at different magnifications, revealing their micro-structure. The results of the SEM are documented along the report.

3.6. Water Demand and Setting Time:

Setting time and water demand of neat cement paste and all other formulations i.e. with replacement with SRM was determined using standard VICAT apparatus in accordance with ASTM-C-187. C191 and BS-12:1978 at temperature and humidity in accordance with EN-196-3. Different trial specimens with varying specimens of water were prepared. When plunger penetrated to point 5 ± 1 mm from bottom of VICAT mold on scale, the percentage of water used to prepare the mold was noted. Water was expressed as percentage by weight of dry cement. Similarly for setting time, periodic penetration tests were performed by allowing VICAT needle to settle into paste. The initial setting time was noted when penetration was 5 ± 1 mm deep. Final setting time was noted when needle could not leave a complete circular impression in paste surface.



Figure 1: Vicat Needle Apparatus

3.7. Flow Test/Super-plasticizer Demand:

Flow tests were performed using the mini slump cone with its bottom diameter to be 10 cm, and minimum diameter of 7 cm and the height of the cone 6 cm and a glass plate as shown in fig. The required target flow was kept at 30 \pm 1 cm. Flow was measured using the hit and trial method by using different quantities of super plasticizer and was measured for all the formulations, the water demand of all these formulations was kept the same as determined earlier by the Vicat's Needle apparatus. Firstly the mini slump cone is placed on a properly leveled glass surface with markings on it at 10cm, 25cm and 30 cm respectively. Then the self-compacting paste from the Hobart mixer is poured into the cone. Then the cone is lifted, the self-compacting mixture flows outward in a circular fashion. Then two orthogonal diameters are measured and taken average of, to measure the flow. The quantity of super plasticizer added, at which the flow stopped at 30 \pm 1 cm mark was taken to be as the super plasticizer demand of that specific formulation. It should be kept in mind that the total flow must not be any more or less than the 30 \pm 1 cm.

$$D=(D1+D2)/2$$

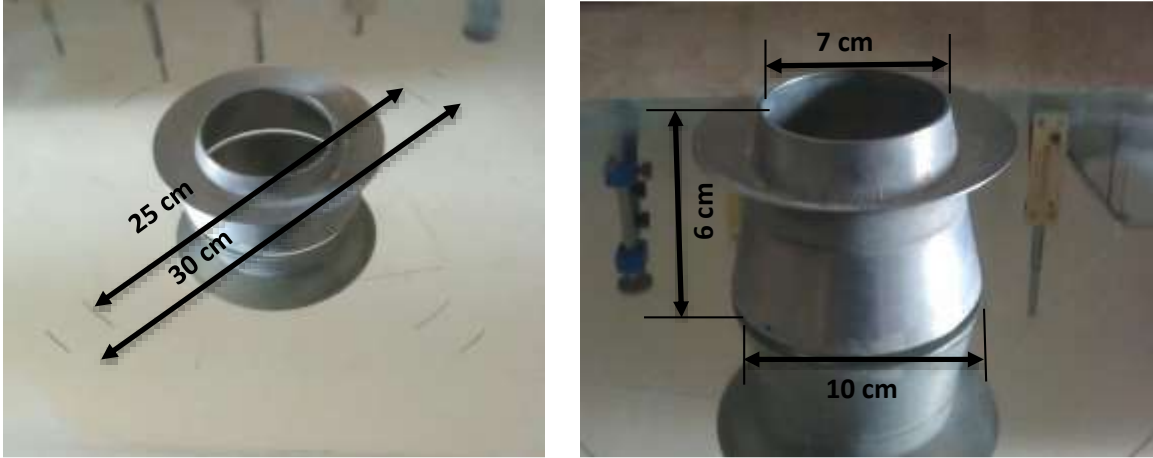


Figure 2: Hagerman's mini slump cone apparatus

3.8. Casting curing and Strength of Samples:

Casting curing and testing of the formulation is performed as per EN 196-1 of 1994. The materials are first mixed in the Hobart Mixer and then poured into the prisms of standard sizes of 40x40x160mm size. The prisms are then covered with sheet to prevent moisture evaporation from the surface of samples. Prisms are unmolded after 24 hours and kept for moist curing in water tub at room temperature. Then these prisms were taken out of tub at specified ages of 1, 3, 7, 14, 21 and 28 days to test for their strengths.

The specimens of size 40x40x160mm are then tested both in flexure and compression. Flexural strength is taken as the average of the flexural strength results of three samples at that specified age. The broken samples after the flexural strength test are then subjected to the compressive strength test. Cross sectional area of samples tested in compression was 40x40mm and these are tested between two steel plates of size 40x40mm, one at the top and the other at the bottom.



Figure 3: Casting molds and final shape of sample

3.9. Calorimetry:

Calorimeter is an instrument or apparatus which monitor the quantity of heat released during a chemical reaction and capable of plotting graph with time. This process of measuring heat is called calorimetry. The model of calorimeter which we used in our lab is F-Cal 8000. It has eight pockets in it to serve eight sample at a time for heat testing which evolved during reaction. The purpose of calorimetry is that by calorimeter we can be able to compare the effects or changings occur in heat evolved and ultimately to track reaction progress due to addition of SRMs in cement paste in SCCS. We prepare the samples of each seven formulations at their calculated water demand. No super



Figure 4: Mcal 500 Calorimeter Apparatus

plasticizer was added. Mixing is simple hand mixing. Each dry sample weight is 100g. Out of eight pockets seven pockets were used, one sample in each pocket, and tested for 90 hours. After that data was taken and graphs were plotted.

Chapter 4 - Results

The results of the experimental program conducted are presented in this chapter.

4.1 Water Demand.

Water demand of seven formulations was found. Results showed that with the addition of SRMs in OPC tend to increase the water demand. The formulation containing both SRMs at 10, 5% replacement of cement in equal proportions. Bagasse Ash showed highest increase in the water demand compared to other SRMs that are used in this project. Also lime showed intermediate water demand. Marble powder showed very less variation in water demand with water demand of cement as compared to other SRMs but still its value was higher than that of OPC. Percentage of water demand by weight of cement is shown in **Fig 4.1**

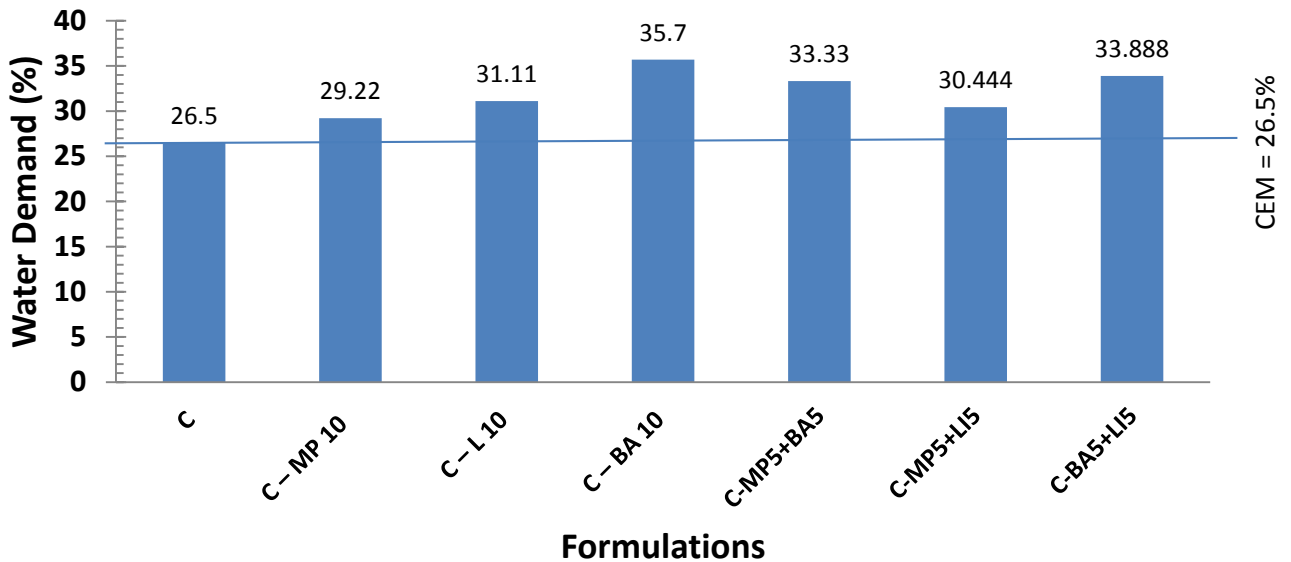


Figure 5: Water Demand of SC Paste Systems

4.2 Super plasticizer Demand.

The Hagerman's mini slump cone was used to find superplasticiser demand for a target flow of 30 ± 1 . Percentage of SP demand by weight of cement is shown in Figure 4-2. Replacement of lime 10% by weight of cement showed highest superplasticizer demand. Marble powder showed lowest of all values.

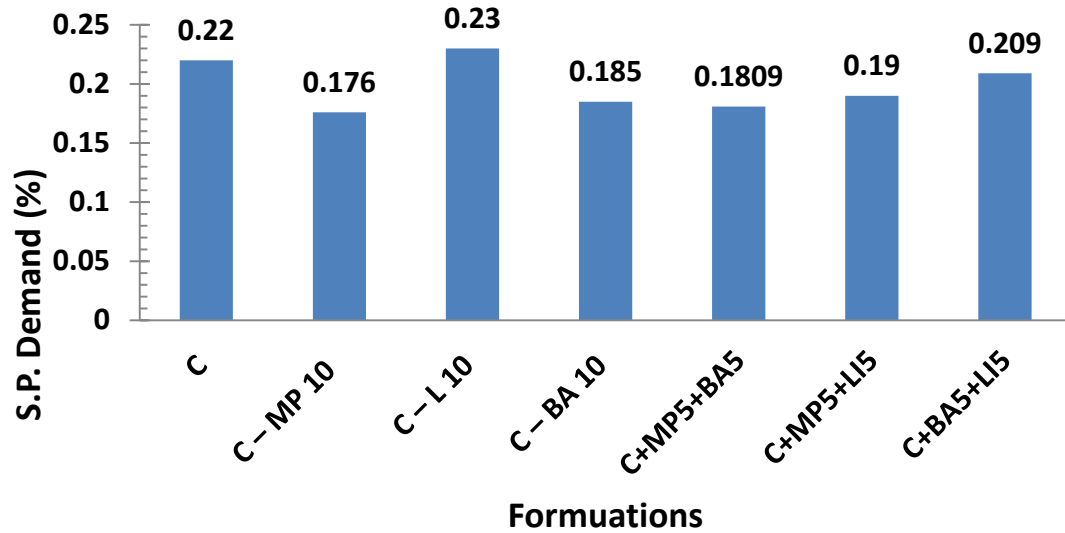


Figure 6: Superplasticizer Demand of formulations

4.3 Setting Time.

Individual characteristics of each secondary raw material along with the amount of that SRM incorporated in that system are the reason of variation in the setting times of formulations. The initial & final setting time found by using Vicat's apparatus. The initial & final setting time

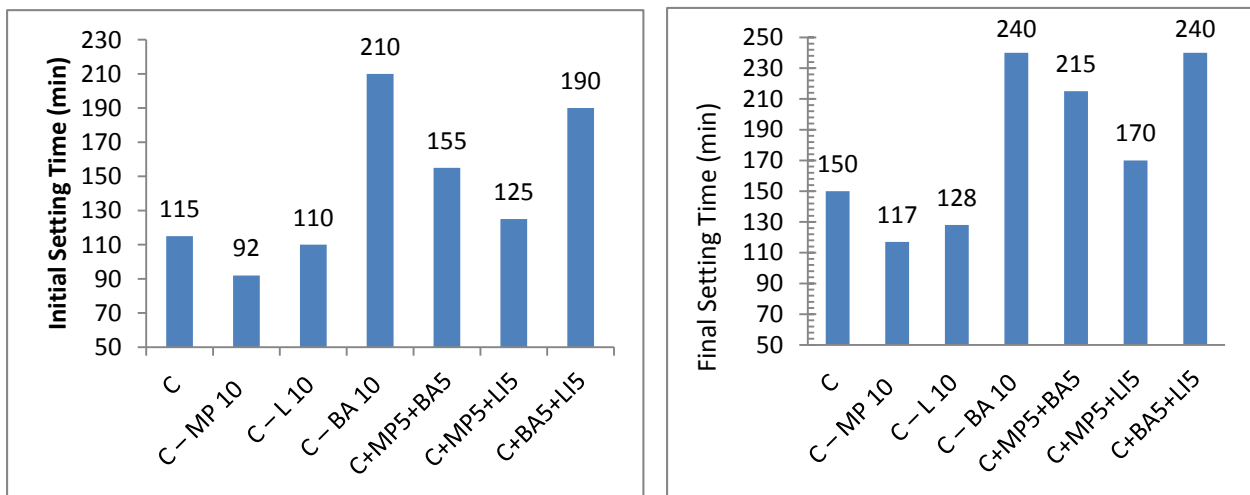


Figure 7: Initial and Final Setting times of formulations

of neat cement formulations were 115 min & 150 min respectively at 22 °C. Values of initial and final setting times are shown in **Figure.4-3**.

4.4 Strengths.

Standard Prisms of 4x4x16 cm of Self Compacting paste system (SCPS) incorporating varying amounts of LI, MP and BA were tested for strength evaluation at ages of 1, 3, 7, 14, 28 and 56 days. **Figure 8** show compressive strength development of self-compacting paste system incorporating different SRMs used.

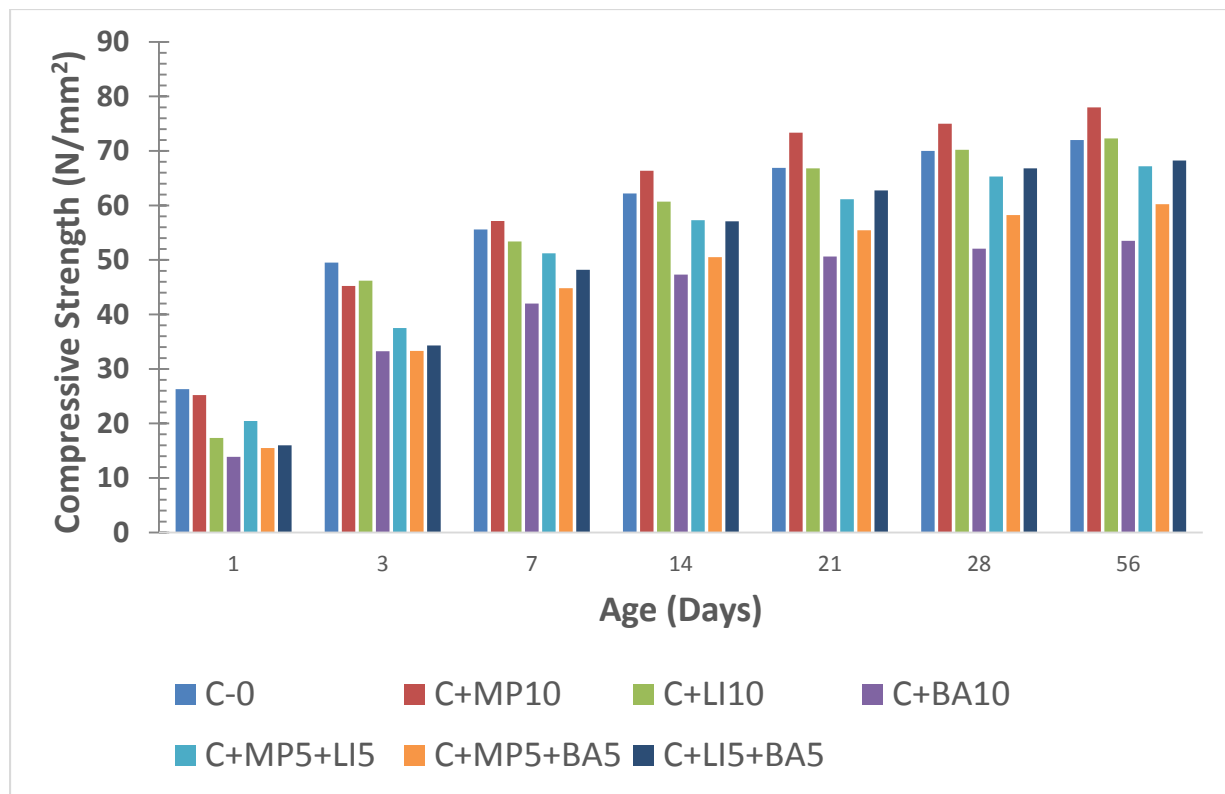


Figure 8: Compressive Strengths of formulations used.

Figure 9 shows Flexural strength development of self-compacting paste system incorporating different formulation used.

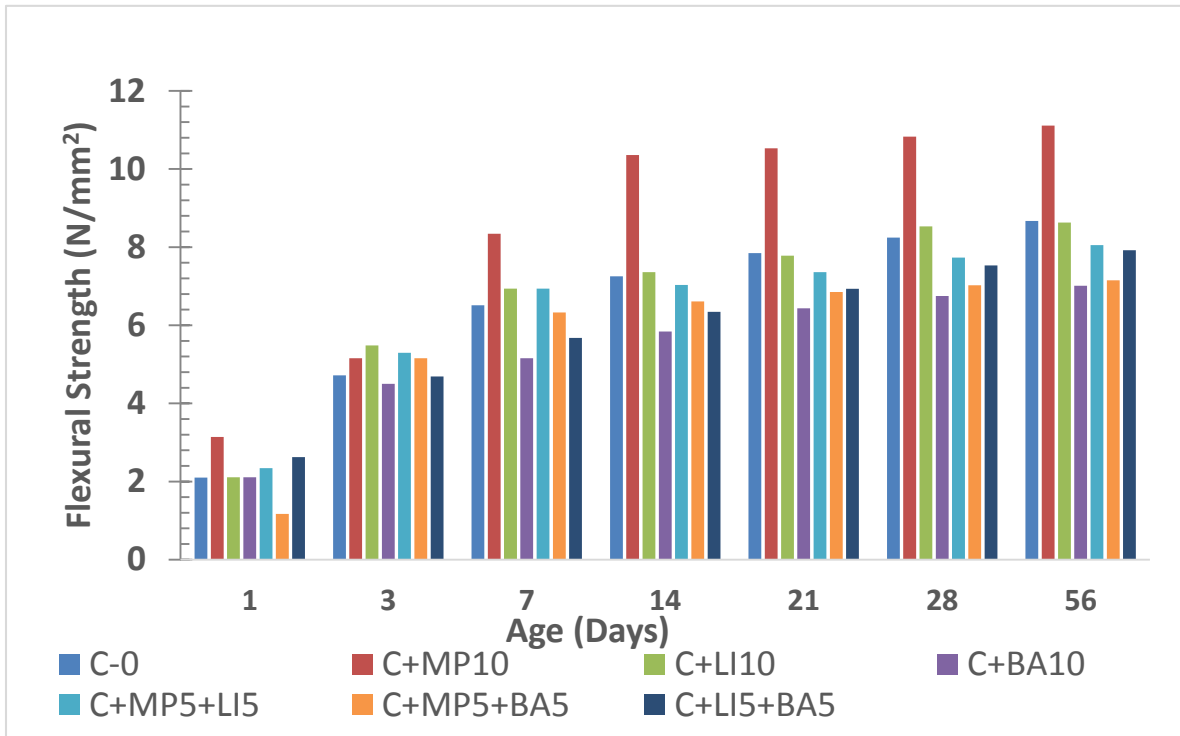


Figure 9 Flexural Strengths of formulations used.

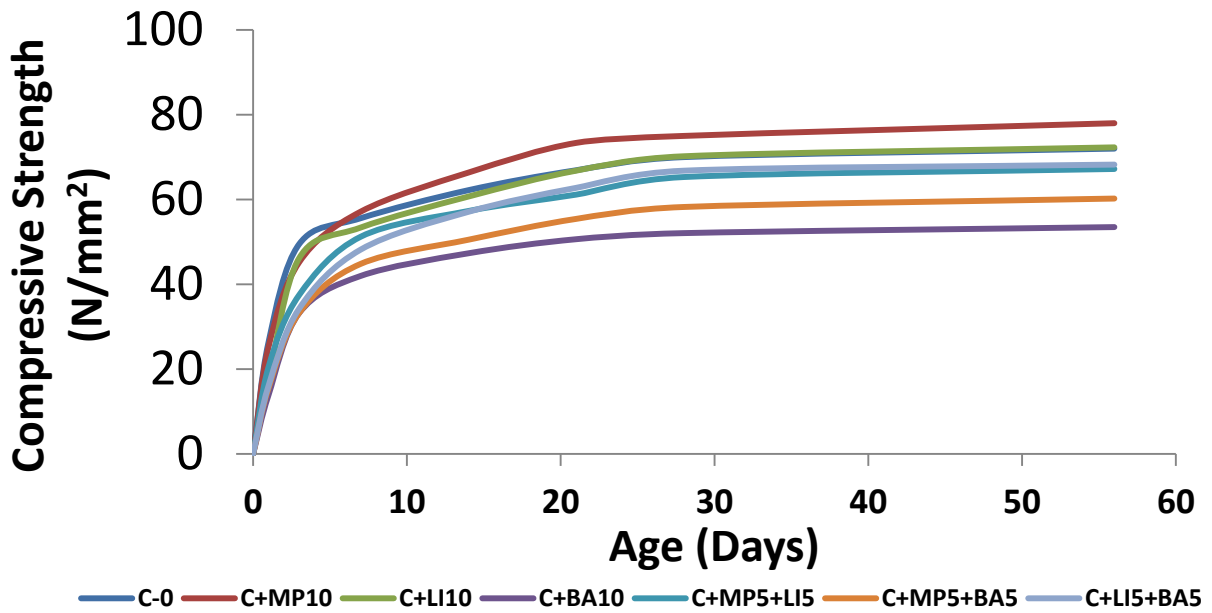


Figure 10 Compressive Strength of formulations

Figure 10 shows Compressive strength development of self-compacting paste system incorporating different SRMs used (graphical view).

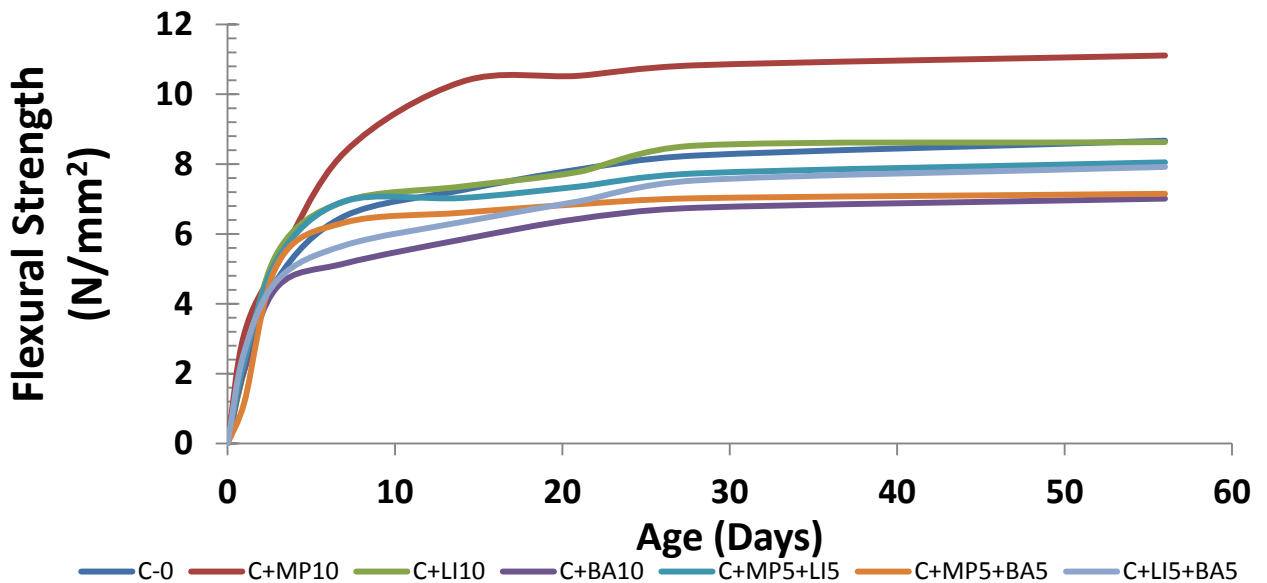


Figure 11 Flexural Strength of formulations

Figure 10 shows Flexural strength development of self-compacting paste system incorporating different SRMs used. (Graphical view).

4.5. SEM Results

The images of the SEM results are very important to have an insight about the microstructure of the particles. The SEM tests results of the materials are as follows:

Cement :

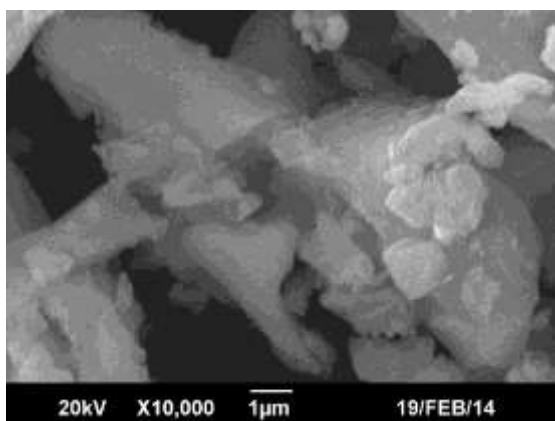


Figure 11: SEM CEMENT X10000

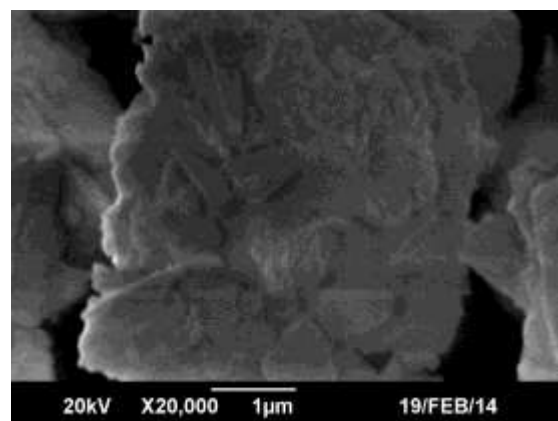


Figure 12: SEM CEMENT X20000

Bagasse Ash:

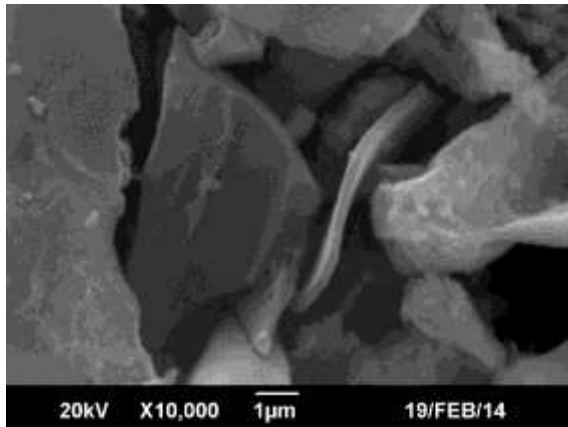


Figure 13: SEM Bagasse Ash x10000

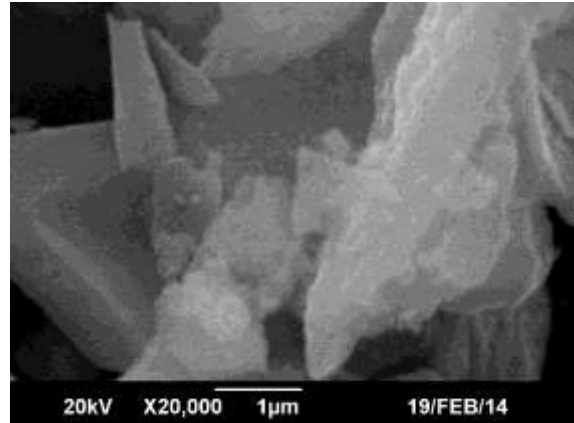


Figure 14: SEM Bagasse Ash x20000

Lime:

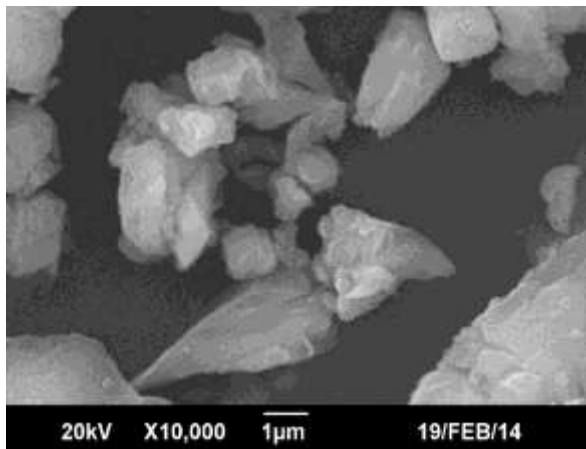


Figure 15: SEM Lime x10000

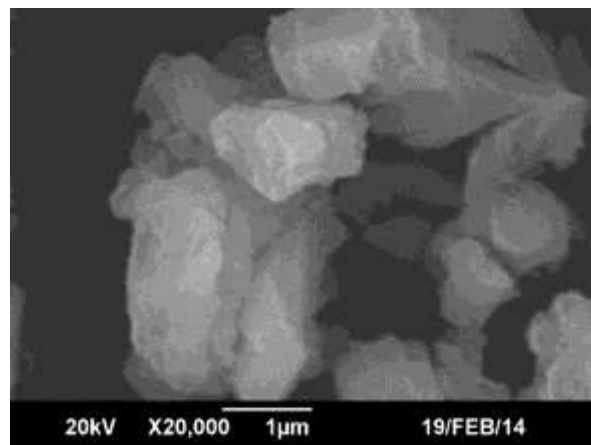


Figure 16: SEM Lime x20000

Marble Powder:

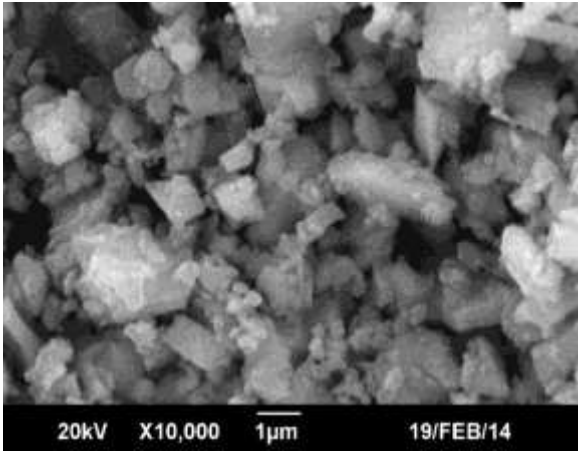


Figure 17 SEM Marble Powder x10000

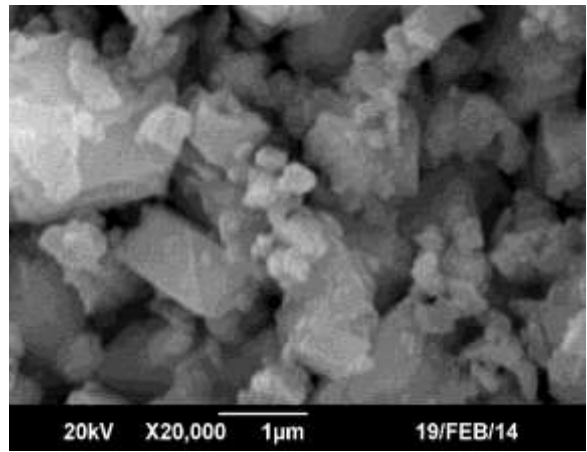


Figure 18: SEM Marble Powder x20000

Chapter-5 Discussion

This chapter focusses on the possible phenomenon that take place behind the results observed in chapter 4. When SRMs are added to a Self Compacting Paste system (SCP) they alter the properties of the system in one or many ways, the variations in results, with respect to the SRMs are discussed in this chapter with a focus to look for the probable causes that could be the reason behind such variation and alteration of the properties.

SRMs play a vital role in altering the properties of the SCP system either by filler effect, dilution effect, chemical effect or physical effect. SRMs added into the SCP paste are in very fine forms with particle sizes almost half of that of the cement hence providing for excellent packing of the system, change in water demand and alteration in the flow of the system that in turn accounts for higher achieved strengths of the SRM replaced formulation than the control mix. The addition of the powder might be due to the improved nucleation of the particles, the physical effect. The alteration in properties can be attributed to dilution of the cement particles in the mix, that influence the average particle to particle distance of the system and in turn affecting the availability of the water for hydration. The addition of fines also influences the porosity of the system by filling up the voids in the system, depending upon the particle size of the cement and the SRM. Generally SRMs act as inert fillers but some SRMs play a vital roles in the chemistry of the system depending upon their composition hence influencing the hydration products of the system.

The addition of Fine SRMs affect the system's water demand in certain ways, either the Water demand of system is increased due to the increased surface area of the system as characterized by the abundance of finer particles or the water demand can be decreased due to the higher packing density of the system by the elimination of the voids by filling of the pore spaces by finer particles, leaving lesser space for the water particles to occupy. In over view of such effects, certain properties of the system concerning flow, water demand, setting times and strength development are studied and discussed in this chapter.

5.1. Water demand of the SCP

The results, as indicated in the chapter 4 show that there was an increase in water demand of the SCP system with addition of SRMs as compared with the control mix of cement 'C-0'. This can be attributed to the particle size of the SRMs. The lesser the particle size of the SRMs the greater the surface area of the system and hence greater was the water demand of the system. The water demand of BA was observed to be the highest as compared to the MP and LI for equal percentage replacement. Where as blended formation showed intermediate results as anticipated.

BA formulation manifested highest water demand which can be attributed to its porous behavior and its high silica content, so water is adsorbed on its surface, it is also observed that the grain size of the BA is lesser than that of the cement, which results in increased surface area of the system and hence increased water demand. Also high silica content poisons the nucleation sites, thereby requiring more water for the hydration of the paste. The water demand of LI was observed to be intermediate between BA and MP, which can be related to its lower particle size and higher surface area of the system. MP showed lesser water demand than other SRMs used, this can be because of its lowest particle size among other SRMs accounting for increased packing density and hence reduction of pores, so providing lesser void spaces for water to enter, and hence lower water demand than other SRMs.

Blended formulations however showed intermediate response, where BA was used with LI and MP it manifested almost the same water demand for each of the SRM but was still higher than when MP or LI were used individually but lesser than when BA was used individually. This can be justified by the porosity of the BA and reduced particle size of the MP and LI. However when blend of LI + MP was used its water demand was noticed to be in between the water demand of the formulations when LI and MP were used individually. Which can easily be related to their individual behavior, i.e. their water demand is influenced by their particle sizes.

It can be summarized as, the water demand of formulations including BA is controlled by the porosity of the BA whereas for LI and MP formulations, particle size is the major factor.

5.2. Super-Plasticizer Demand of SCP

Certain amount of the Super-plasticizer is absorbed by the SRMs depending upon their morphology and surface texture. SEM images of cement and SRMs can help us a good deal in understanding the morphology and surface texture of the materials, as shown in Figure.4.8 to (Figure 4.15).

It has been observed that the formulation containing LI i.e. C+LI10 as SRM exhibited the highest superplasticizer demand for the target flow. SEM images of the LI show that it is a highly porous material having relatively large voids between its particles as compared to the other SRMs (Figure 4.12, 4.13), adding to that, its particle size is also very small, so both porosity and its particle size play a role in its SP demand. It has a large surface area due to its particle size and lesser density due to its porosity, so some of the SP is absorbed by LI rendering it, the material with the highest SP demand. BA on the other hand showed lesser SP demand than LI, though it also appears to be porous but its particle size is quite large as compared to that of LI, also its texture as shown in SEM images (Figure.4.10, 4.11) is broken and flakey, rendering better interconnectivity of the particles, so it has

lesser surface area to absorb the SP and hence has the lower SP demand than the LI. Marble powder MP on the other hand has the lowest SP demand among all single replacement formulations, it can be argued that it has the lowest particle size thus it must exhibit highest SP demand, but the justification is that it has the lowest porosity and has high density (Figure 4.14, 4.15), thus with particle size so small, it increases the density of the paste, rendering lesser surface area for the SP absorption and hence the lowest SP demand.

Blended formulations, as anticipated showed the similar response, i.e. those containing lime had greater SP demand than the one without lime. Namely C+BA5+LI5 had the highest SP demand among blended formulations, this can be attributed to their porous nature, i.e. both BA and LI are porous hence greater is the absorption of SP, so greater the SP demand for the target flow. C+MP5+LI5 had lower SP demand as compared to the C+BA5+LI5, this is because of the high porosity and large surface area of LI but due to the higher packing density of the system attributed to MP, it had relatively lower SP demand. The formulation C+MP5+BA5 had the lowest SP demand as compared to the other blended formulations, this is because, this formulation is less porous than the one having LI so it has the lowest SP demand, however its demand is still higher than the one where MP is used individually i.e. C+MP10, which can be attributed to the BA morphology.

5.3. Setting times:

Setting times were calculated using Vicat needle apparatus for single and double replacements.

Setting of cementitious system is due to dissolution of cement grains which is followed by growing ionic concentration in water. This leads to formation of compounds (Hydration products) in the solution. After reaching a specific concentration compound precipitate out. Densification of these products cause setting of cement.

Amongst single replacements bagasse ash had highest initial and final setting time which can be attributed to its organic nature. Sugar itself is a retarder for cementitious system and so are cellulose derivatives. Also, BA contains high silica content, which being a vital component of CSH gel, contributes greatly to the hydration of the system, thereby increasing the setting time of the paste (the dormant period) [50]. Lime and Marble powder however when used as single replacements for cement reduced initial and final setting times. Both marble and lime provide Ca^{+} ions to the solution which speeds up the setting time by increasing nucleation and growth of CSH molecules.[50]

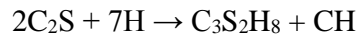
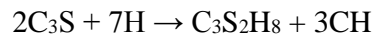
Results of setting times for double replacements are in accordance with results of single replacements. For instance setting times for C+MP5+BA5 are greater than those observed for C+MP10 but they are lesser than those seen for C+BA10. Among the double replacements C+BA5+Li5 gave largest setting times as shown by graph.

Both marble powder and lime when used as single replacement reduce setting times but when used together as double replacement give setting time higher than that of C+MP10 AND C+Li10. This can be explained by taking into account their particle sizes, due to their smaller particle sizes they fill the gaps between cement grains and a little voids are left for water to fill in. This slows the hydration and thus delays setting of cementitious paste.

5.4. Strength characteristics

Compressive Strength and flexure strength tests were performed on specimens at the age of 1, 3,7,28 and 56 days.

Strength of cement is due to hydration of calcium silicates. Both C3S and C2S react to produce C-S-H and C-H. [53]



As densification of hydration products precedes the porosity decreases cementitious system gains strength. Following figure shows contribution of different products at different stages for densification and strength gain. It is evident that role of ettringite in providing strength is paramount in first three days. However C-S-H and C-H continue to densify strengthen the system as time passes.

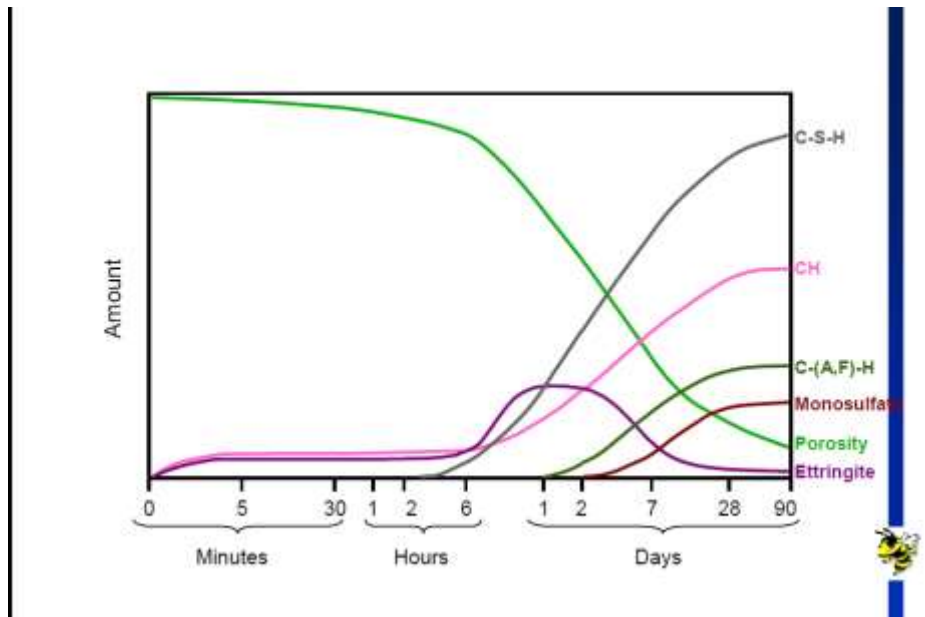


Figure 19: Hydration Products of OPC w.r.t. Time [53]

C+MP10 gave the highest strength among seven formulations .this is because MP is rich in calcium and it provides more Ca^+ ions to the cement slurry thus providing greater quantity of hydration products(C-S-H and C-H) and ettringite.

C+MP10 is followed by C+LI10 as it is also rich in calcium concentration. The difference in strengths provided by MP and LI can be accounted for by considering their particle sizes.MP being smaller in size has larger surface area thus it provides greater number of sites for reaction of water with the powder.

Formulations including BA yield lower strengths because of silica present in it and its porous nature. Silica present in BA poisons the nucleation sites thus inhibiting the development of hydration products. Porous surface of BA holds water in it and reduces the water available for hydration.

The difference in strength provided by BA replacements and replacements of MP and Li increase with time. There is significant difference in strengths of C-MP10 and C-BA10 at age of 28 and 56 days. This is due to the difference in amount of Ca^+ ions available for formation of C-S-H and C-H.

Similar trends are seen for compressive and flexure strength test results. It has also been observed that in double replacements the strength values are in accordance with those seen in single replacements. For example strengths for C+MP5+BA5 are lesser than those observed for C+MP10 but they are greater than those seen for C+BA10. the same is true for C+LI5+BA5.

But there is an anomaly to this trend just as there was in case of setting times: the strength results for C+MP10+LI10 are lesser than those observed for C-MP10 and C+Li10. This is due to packing effect of LI and MP, due to their smaller particle sizes they leave a little number of water filled voids which leads to reduced hydration. Both LI and MP contain excess amount of calcium, but there is need of energy for dissolution which is absent due to oversaturation of the solution. This is also a reason for reduced strength.

Chapter-6 Conclusions

We can conclude the following main points from our study, after experimentation, analysis of results through observation of trends and confirmation from literature:

- The maximum strength at 28 days & 56days is obtained for the formulation containing 10% replacement of marble powder because of small particle size which is major cause of filler effect and provision of nucleation sites.
- The SP demand to achieve the target spread was least for SCP formulation containing marble powder.
- Lime at 10% replacement exhibited similar response as of neat cement because it act as alkaline activator that's why its increase is very little.
- Bagasse Ash at 10% replacement showed lowest strength of all of other formulations used because of it highly porous morphology.
- Blends of all formulation used also showed intermediate values.
- Particle size of SRMs plays an important role in determining packing density, strength, workability and setting times.

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Chapter-8 Annexure

Annexure –A

Table 1: Water Demands

Formulations	C-0-WD	C-MP10-WD	C-LI10-WD	C-BA10-WD	C-MP5-LI5-WD	C-MP5-BA5-WD	C-LI5-BA5-WD
Water Demand (%)	26.5	29.22	31.11	35.7	30.44	33.33	33.88

Table 8. 1 : Water Demands of formulations studied

Table 2: Initial & Final Setting Time

Formulations	Initial Setting Time	Final Setting Time
C-0-WD	115	150
C-MP10-WD	92	117
C-LI10-WD	110	128
C-BA10-WD	210	240
C-MP5-LI5-WD	125	170
C-MP5-BA5-WD	155	215
C-LI5-BA5-WD	190	240

Table 8. 2: Initial and Final setting time of formulations studied

Table 3: Flow Measurement

Formulations	Superplasticizer Demand (%)
C-0-WD	0.22
C-MP10-WD	0.176
C-LI10-WD	0.23
C-BA10-WD	0.185
C-MP5-LI5-WD	0.19
C-MP5-BA5-WD	0.181
C-LI5-BA5-WD	0.209

Table 8. 3: Superplasticizer Demand of formulations studied.

Table 4: Compressive Strength

	1 day (MPa)	3 day (MPa)	7 day (MPa)	21 day (MPa)	28 day (MPa)	56 day (MPa)
C-0-WD	26.3	49.5	55.6	66.9	70	72
C-MP10-WD	25.2	45.23	57.15	73.35	75	78
C-BA10-WD	13.87	33.24	41.99	50.64	52.08	53.5
C-LI10-WD	17.35	46.2	53.4	66.8	70.2	72.3
C-MP5-BA5-WD	15.5	33.3	44.83	55.43	58.24	60.23
C-MP5-LI5-WD	20.45	37.5	51.23	61.15	65.3	67.2
C-LI5-BA5-WD	16	34.3	48.2	62.75	66.8	68.23

Table 8. 4: Compressive strength of formulations used

Table 5: Flexural Strength

Formulations	1 day (MPa)	3 day (MPa)	7 day (MPa)	21 day (MPa)	28 day (MPa)	56 day (MPa)
C-0-WD	2.1	4.7183	6.5123	7.848	8.246	8.67095
C-MP10-WD	3.140	5.1557	8.34286	10.53	10.83	11.11
C-BA10-WD	2.109	4.49952	5.1557	6.434	6.75	7.012
C-LI10-WD	2.109	5.48379	6.93676	7.78042	8.53034	8.63
C-MP5-BA5-WD	1.1717	5.1557	6.32745	6.85302	7.023	7.15
C-MP5-LI5-WD	2.3435	5.29631	6.93676	7.35859	7.73355	8.051
C-LI5-BA5-WD	2.625	4.687	5.6761	6.9355	7.53355	7.921

Table 8. 5: Flexural strength of formulations used

Annexure – B

XRF Results

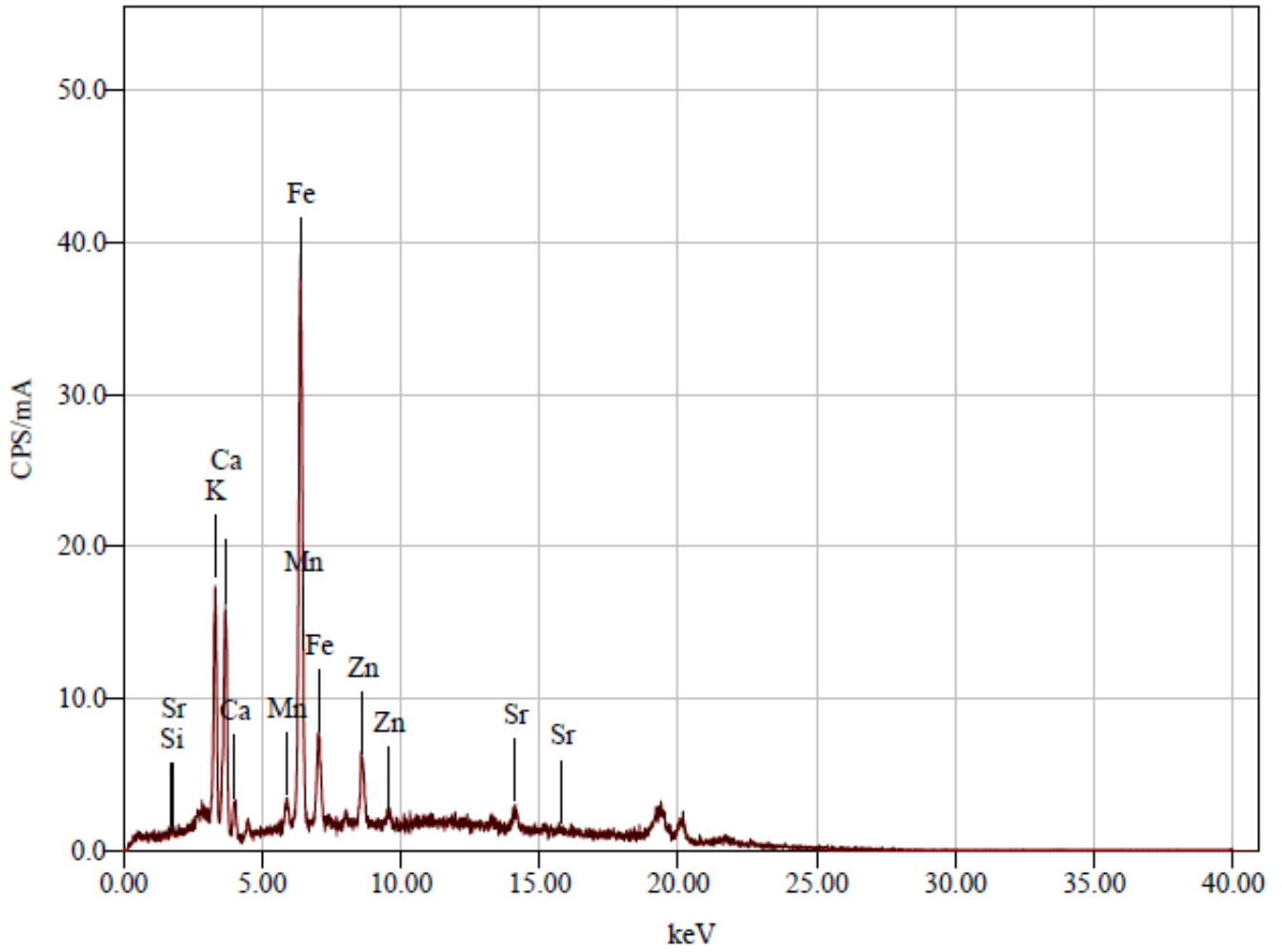


Figure 20: XRF Results of Bagasse Ash

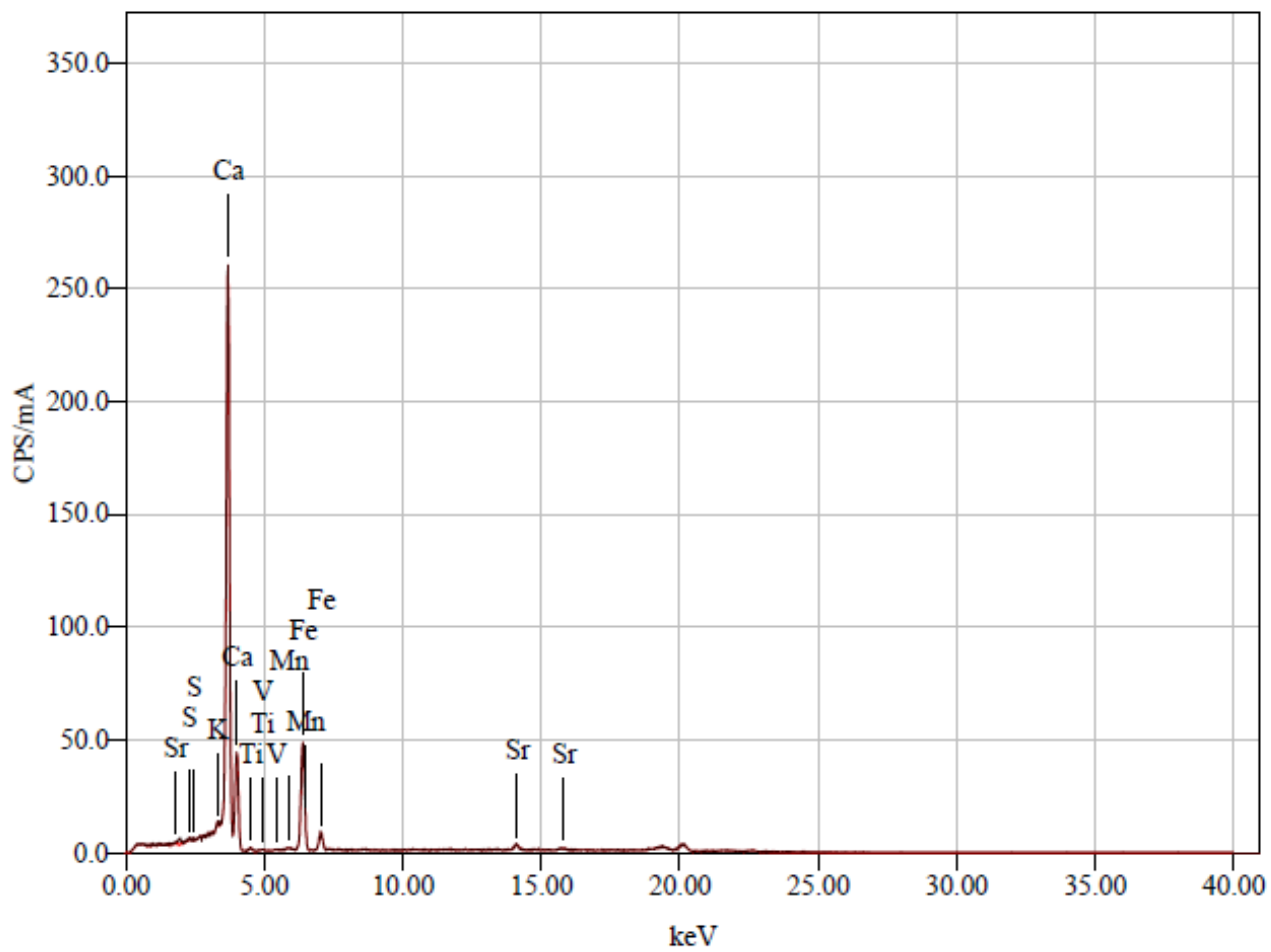


Figure 21: XRF Results of Cement

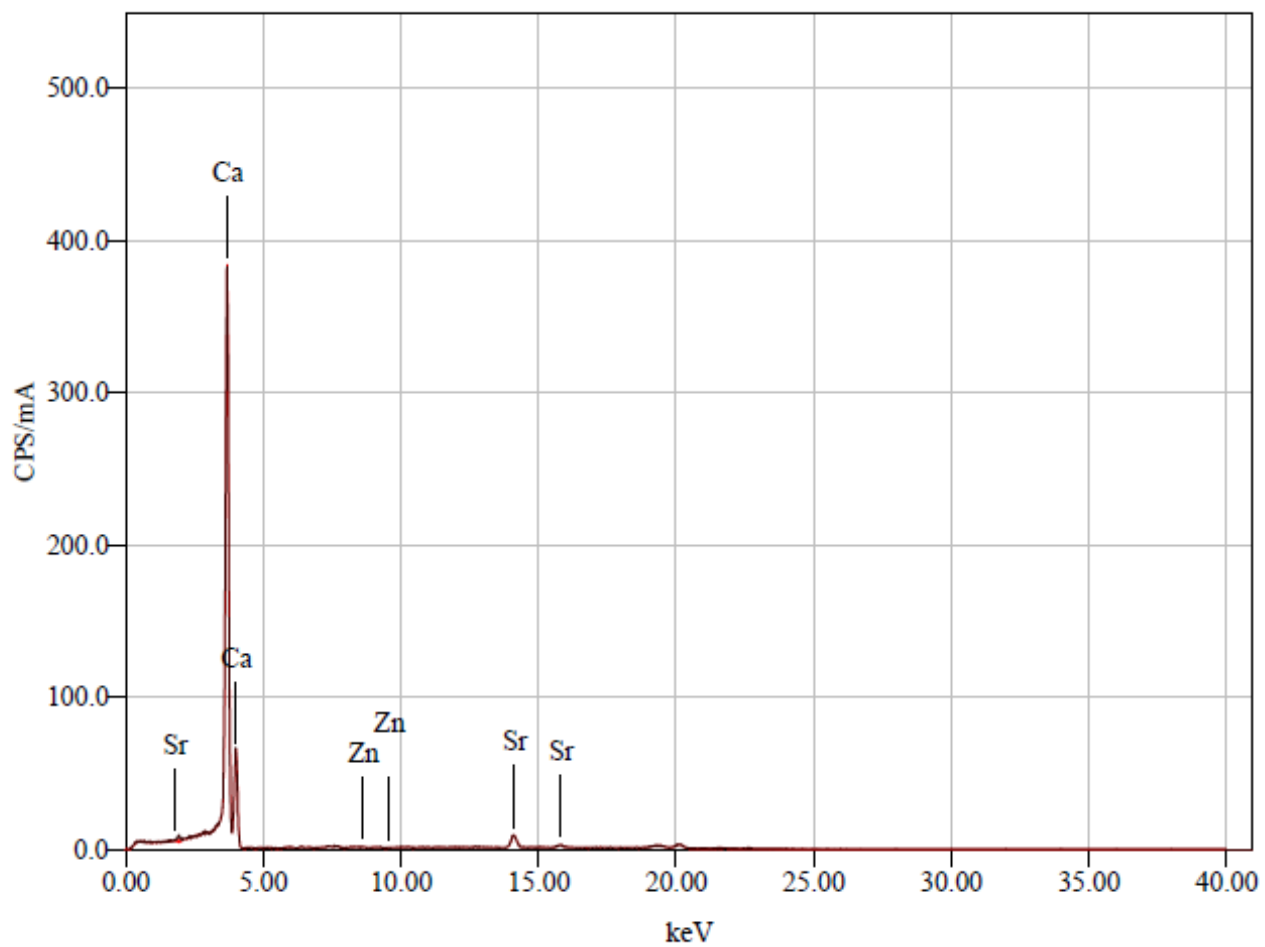


Figure 22: XRF results of Lime

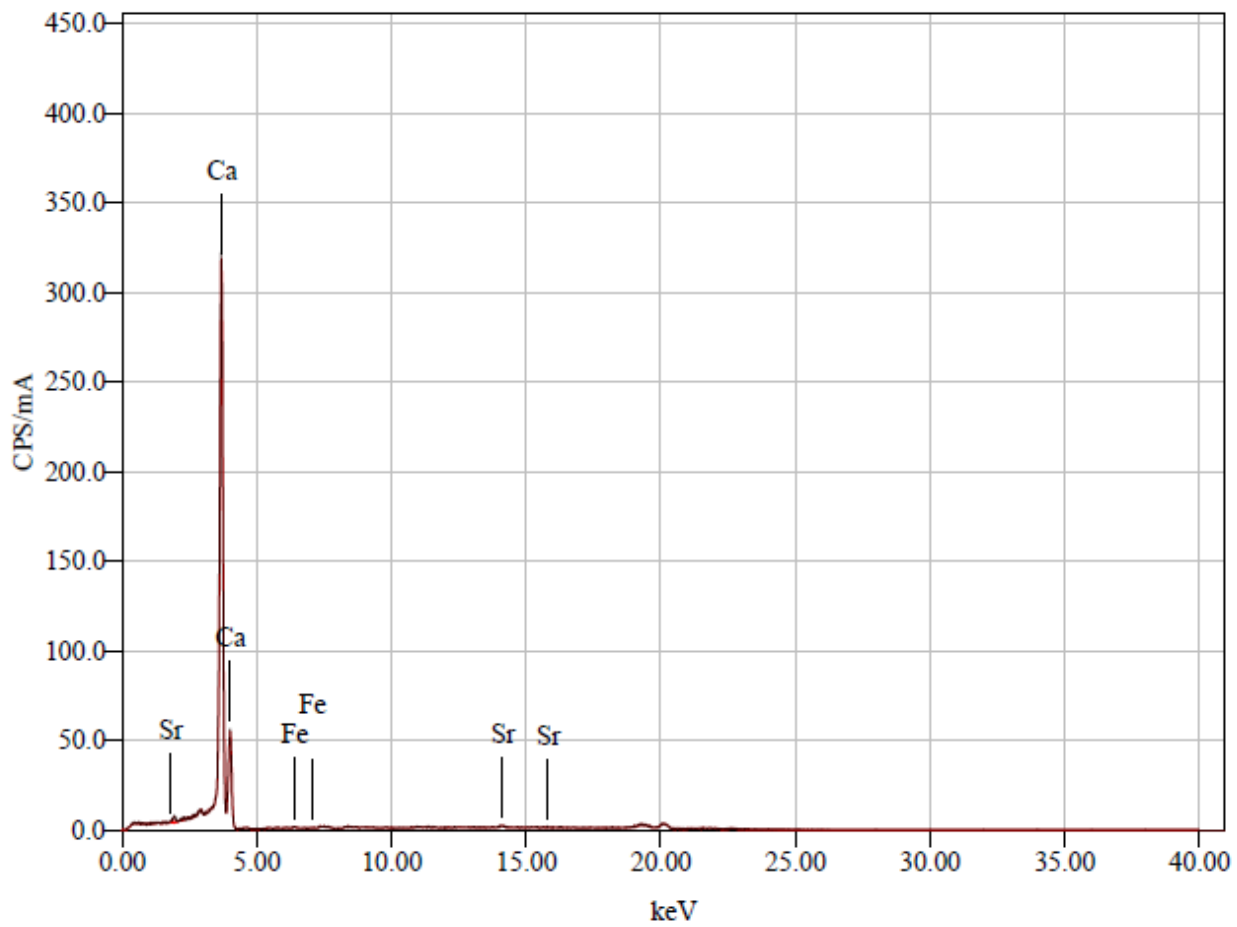


Figure 23: XRF results of Marble Powder