EFFECT OF PARTICLE SIZE OF LOCAL SUGAR CANE BAGASSE ASH ON THE RESPONSE OF SELF COMPACTING MORTAR SYSTEMS

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EFFECT OF PARTICLE SIZE OF LOCAL SUGAR CANE BAGASSE ASH ON THE RESPONSE OF SELF COMPACTING MORTAR SYSTEMS

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ABSTRACT

This study was aimed at arriving at an optimal particle size of bagasse ash as filler material for use in SCM systems. The schematics of this research included firstly the preparation of bagasse ash. This was done by burning it at 500ºC for one hour in a semi oxidizing environment. The attained ash was then divided in four batches and put to grinding in Los Angeles abrasion machine for varying grinding times. The grinding times chosen were 5, 10, 15 and 20 min. PSDs of the ground bagasse ash batches were acquired and a comprehensive particle characterization was performed using SEM, XRD and XRF. The four bagasse ash batches prepared were then studied for their behavior in various fresh and hardened properties in SCM systems. Water demand, SP demand and flow time required for 30±1 cm flow spread were determined for the control mix as well as that for the mortar formulations containing 20 % cement replacement of bagasse ash. Setting times were also worked out for all the mortar formulations. Strength samples were casted for 3, 7 and 28 day flexural and compressional strength. 72 hour calorimeric and shrinkage studies were also performed on the fresh mortar formulations. The strength results, calorimetry and shrinkage findings were then verified using TGA and SEM along with EDS analyses. The study concluded that amongst all the bagasse ash formulations, BA28 yielded best overall response in strength attainment, volume stability and heat evolution during hydration and hence offers enhanced durability along with improved strength as compared to the control mix. So, we can say that the particle size of 28 microns is the optimal size of bagasse ash for use in SCC systems as filler material with 20% replacement level.

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SCC	Self-Compacting Concrete
SRMs	Secondary Raw Materials
ACI	American Concrete Institute
ASTM	American Society of Testing Materials
WD	Water Demand
OPC	Ordinary Portland Cement
SSD	Saturated Surface Dry
HPC	High Performance Cement
SCCS	Self-Compacting Cementitious Systems
JSCE	Japan Society of Civil Engineers
HRWRA	High Range Water Reducing Admixtures
SCBA	Sugar Cane Bagasse Ash
GGBFS	Ground Granulated Blast Furnance Slag
HSC	High Strength Concrete
SP	Super Plasticizer
RHA	Rice Husk Ash
BA	Bagasse Ash
CH	Calcium Hydrate
CSH	Calcium Silicate Hydrate
SAIs	Strength Activity Indices
ITZ	Interfacial Transition Zone
XRD	X-Ray Diffraction
TGA	Thermogravimetric Analysis
MPS	Mean Particle Size
PSD	Particle Size Distribution
XRF	X-Ray Fluorescence
SCMs	Self-Compacting Mortars
PCSIR	Pakistan Center for Scientific and Industrial Research
GCRC	Glass and Ceramics Research Center
SEM	Scanning Electron Microscopy
GT	Grinding Time
CM	Control Mix
EDS	Energy Dispersive Spectroscopy

CHAPTER 1: INTRODUCTION

1.1 General

In the rapidly changing world of technology, advancement is being carried out in all the areas of life. The world is shifting its concern from the conventional to innovative ideas. In this race of advancement, concrete technology is not lagging behind. Everyday research unravels new horizons of construction building, resolving the inadequacies of the past practices.

For centuries, the human race spent a good amount of effort into the old exhausting exercise of manual construction. Mega constructions in the past took the lives of hundreds of people. For then, transportation and placement of building materials was very difficult.

Then finally, the era of slow and arduous construction was overcome by the discovery of a truly remarkable material, which we all know today as self-compacting concrete (SCC). This material, through its astonishing properties, has managed to take the realm of construction years forward and way easy.

The introduction of self-compacting concrete made mass construction much easier and way faster. More architectural variations could be added to the structures with much better accuracy and finish. The problems of improved strength and durability were also better addressed. In other words, it redefined the domain of building construction.

Now, with the mass construction made this much easier, the cement demand was drastically rising up and so was its carbon footprint. So, in order to counter this problem, researchers came up with the idea of introducing secondary raw materials (SRMs) into the cement mix. They provide a relatively cheap and environment friendly replacement of cement. In addition, they impart useful properties to the cement blend as well.

The use of secondary raw materials (SRMs) enhances durability by improving microstructure of concrete. It also improves strength by improving density via better packing and offering of extra nucleation sites.

Thus, the modern concrete is just not a mixture of cement, sand and water; it has additional constituents as its basics too. The discovery of these materials has opened a new doorway to performance based design.

First put to test in 1980's [1], self-compacting concrete (SCC) already has and is still evolving with the extensive research being carried out in the whole world. This particular study is also a humble addition to this set of knowledge.

1.2 Self-Compacting Cementitious Systems (SCCS)

Different standards define self-compacting concrete in different ways, but pronounce the same maxim. Some of these are stated as under;

American Concrete Institute (ACI) defines self-compacting concrete as **"*Fresh concrete that can flow around reinforcement and consolidate within formwork under its own weight without vibration*"** [2]

American Society of Testing Material (ASTM) states it to be **"*concrete that can flow around reinforcement and consolidate under its own weight without additional effort and without exceeding specified limits of segregation*"** [3]

According to ENFARC SCC is **"*concrete that is able to flow and consolidate under its own weight, completely fill the formwork even in the presence of dense reinforcement, whist maintaining homogeneity and without the need for additional compaction.*"** [4]

Though SCC is a breakthrough in the practical application of architectural diversity, yet the need of costly chemical admixtures and high volumes of cement had limited its use in the everyday construction. So the researchers came up with the idea of replacing chemical admixtures with the mineral ones. This opened a new doorway to the self-compacting technology, where the use of SRMs not only reduced the carbon footprint of the cement in the environment but lowered the SCC production cost as well. Their use also added durability to the systems and put industrial waste to a constructive disposal [5-7]. The SRMs, their origin, pozzolanic activity and advantages will be discussed in detail in the forthcoming chapter of this report.

1.3 Secondary Raw Materials (SRM)

ACI defines supplementary cementitous materials (SCMs) or secondary raw materials (SRMs) as **"**inorganic material such as fly ash, silica fumes, meta-kaolin, or slag cement that reacts pozzolanically or hydraulically**"** [2]

ASTM defines SRMs in specification C1697-16 as **"**a slag cement or pozzolan that contributes to the properties of concrete or mortar through hydraulic or pozzolanic activity, or both**"** [8]

SCMs or SRMs are also known as mineral admixtures, as they are added during mixing phase of the cement formulation. Hence, ENFARC defines them as, **"**material added during the mixing process of concrete in small quantities related to the mass of cementitous binder to modify the properties of fresh or hardened concrete**"** [4]

The collective efforts to reduce the carbon foot print of cement and proposal of green construction solutions gave introduction to the use of SRMs in the world of construction, offering single solution to multi-dimensional problems.

1.4 Bagasse Ash

Bagasse is the residue left after the extraction of juice from cane sugar. It is very abundant in agro-based countries like Pakistan, India and Bangladesh. According to an estimate the worldwide sugar production is around 1500 million tons per year, producing approximate 600 to 675 million tons of bagasse per annum. [9, 10]. Pakistan is the world's sixth largest producer of sugarcane in terms of acreage, and the 12th largest producer of sugar. In Pakistan, sugarcane is grown on approximately one million hectares and provides the raw material for 84 sugar mills. The sugar industry is the country's second largest agro-industry after textiles. According to USDA Foreign Agricultural Service, In MY 2012/13 Pakistan's sugarcane production is estimated at 61 million tons, up 4 percent over previous year's harvest of 58.6 million tons. [11]

Hence, this industry produces enormous mass of bagasse which is fed to the boilers for steam generation. The furnaces run at high temperatures ranging from 800 ºC to 1200 ºC thus, converting raw bagasse into bagasse ash. This ash is further treated at customized conditions to attain a chemically active ash of required physical and chemical specifications. Henceforth, this modified bagasse ash is used as a cement replacement technique to bring down the cost of cement productions [7] and to make it eco-friendly. [12]

1.5 Research Objectives and Application

This research was intended with the purpose to

- Study the fresh and hardened properties of self-compacting mortars using varying particle size of bagasse ash, and hence arrive at an optimal particle size of bagasse ash that may give the augmented properties both, in the fresh as well as in hardened state.

The lateral advantages of this study included an energy conservative alternative to concrete constructions, giving a cost efficient solution while providing with an efficient environment friendly waste disposal of agro-industrial waste.

1.6 Research Significance

Until now, the research available covers a rather very narrow scope of the parameters studied using bagasse ash in the self-compacting formulations. This research work presents a very systematic and augmented study to arrive at an optimum particle size, right from the preparation of ash from raw form, which shows the best response in all the short-term as well as long-term properties, for the production of a cost effective, eco-friendly and durable concrete system.

1.7 Scope and Limitations

This study aims at evaluating the properties of different particle sizes of controlled burnt and milled bagasse ash, in self-compacting mortar systems. So, the parameters chosen to tract this research work are grouped as under;

The core variables include:

- Particle Sizes of Bagasse Ash
- Milling Time
- Water / Powder (w/p) ratio
- Quantity of Water (WD of the respective system)
- SP content was so as to achieve the target flow

The key constants of the study include:

- Burning Regime of Ash i.e. Semi-Oxidizing Environment
- Burning Temperature i.e. 500ºC for 1 hour
- Cement : Sand Ratio 1:2
- Sand Gradation having Fineness Modulus of 2.09 and size<2mm
- Replacement Level of Bagasse Ash i.e. 20% by weight of cement
- Target Flow i.e. 30$\pm$1 cm
- Mixing Regime

The term “Powder” refers to Ordinary Portland Cement (OPC) and SRM collectively. Each of the formulations made had the water content equal to water demand of the system. The target flow was achieved by using Melflux 2651F. The sand used was in saturated surface dry (SSD) condition.

CHAPTER 2: LITERATURE REVIEW

2.1 General

Literature review presented here will first cover a brief overview of self-compacting concrete (SCC) and its development, the manufacturing mechanism of SCC, its advantages and effect on the environment. Later, some light will be thrown on its sub group i.e. self-compacting mortars (SCMs). Following this, high performance concrete (HPC) will be introduced briefly. Then, secondary raw materials (SRMs) will be discussed in detail. This will include introduction of pozzolan, the mechanism of their reactive behavior and their advantages. Afterwards, studies already available on bagasse ash will be jotted down. Lastly, the investigational techniques will be discussed for SCC systems. These will include volume stability, SEM, XRD, XRF and TG analysis and calorimetric technique.

2.2 Self-Compacting Cementitious System (SCCS)

Self-compacting cementitious systems (SCCS) are a broad category of cementitious systems comprising of self-consolidating concrete, mortars and pastes. All these systems share the same fundamental principle of self-consolidation. One component systems i.e. pastes can be converted to mortars-the two component systems- by the addition of fine aggregate and successively to concrete, being the three component system, by incorporating coarse aggregate. Yet, the evolution of these phases must be carefully augmented for the development of an effective SCC system.

Japan Society of Civil Engineers (JSCE) [13] laid down the definitions of different types of SCC systems in their section 1.2 of their recommendations clause 13, 14 and 15 which are stated as follows;

I. **Powder-type Self-Compacting Concrete**: Self-Compacting Concrete proportioned to provide the required self-compactability not by using a viscosity agent but primarily by reducing the water-powder ratio (in effect increasing the powder content) to impart adequate segregation resistance and using an air-entraining and high-range water-reducing admixture or super plasticizer to impart high deformability.

II. **Viscosity agent-type Self -Compacting Concrete**: Self-Compacting Concrete proportioned to provide the required self-compactability by the use of a viscosity agent to impart segregation resistance and air-entraining and high-range water-reducing admixture or super plasticizer to impart high deformability to the fresh concrete.

III. **Combination-type Self-Compacting Concrete**: Self-Compacting Concrete proportioned to provide the required self-compactability primarily by reducing the water-powder ratio (in effect increasing the powder content) to impart adequate segregation resistance and using an air-entraining and high-range water-reducing admixture to impart high deformability. A viscosity agent is also added to reduce the quality fluctuation of fresh concrete, so as to facilitate the quality control of concrete.

All these definitions sanction the same basic properties of self-compacting concrete i.e. high deformation ability and segregation resistance.

2.2.1 Background/ Development of SCC

The concept of self-compacting concrete was conceived by Okamura in 1986, so as to cater for the durability issues that aroused due to the differential compaction of concrete. Its first prototype was developed in 1988. Research to study the rheological properties of self-compacting concrete were first carried out in 1989 by Ozawa and Maekawa [1] . By the start of 1990s, Japan was able to successfully develop fully functional self-compacting concrete. This concrete, as per the definition of SCC, was able to flow under its own weight and completely fill the formwork without any compaction.

Since then, extensive research has been carried out on this remarkable material to further enhance and customize its properties for custom use.

2.2.2 Effect of w/p Ratio and Super Plasticizer Content

Abraham's law [14] ascertains an inverse relation between the w/p ratio and strength of the cementitous system. Therefore, one of the primary requirements of the SCC development is to use a lower w/p ratio, as high w/p ratios invite segregation and yield low strengths. Since lower w/p ratio cannot yield sufficient workability, high range water reducing admixtures (HRWRA) or super plasticizers (SPs) are added to serve this purpose.

The use of low w/p ratios and super plasticizers, besides imparting adequate segregation resistance and deformability, give the added advantages of low porosity, improved microstructure, strength and durability [13].

A study suggests that SP content of around 1% (by weight of binder) is required for Self Compacting Paste systems and that equal to 2% for Self Compacting Mortars for optimal flow [5].

2.2.3 Environmental Effect

Sharif et al. states that production of cement is energy extensive process which consumes natural resources and causes global warming [15-17] . This notion has been endorsed by other researchers as well [18].

Literature continuously states that use of SRMs largely decrease the CO_2 emission by reducing the amount of cement utilized in the formulations.[19]

Sustainable concretes can be made by employing industrial solid waste as mineral admixtures, which could preserve the non-renewable energy resources and the environment. Also, this practice can put wastes such as SCBA i.e. sugar cane bagasse ash to a productive use [20].

2.2.4 Advantages

As discussed above, the advantages of using SCC can be summed up in the bullets as under;

- Little or no labor and compaction required for the placement of SCC
- Finished level of SCC requires less or at time no screeding at all
- Construction speed increased due to easy placement of the concrete
- Effective against congested placements or high reinforced members such as tunnels, bridges and columns of high rise buildings
- Architectural variation opportunities made easy through SCC
- Excellent surface finish and durability
- Extensive use in precast member industry

2.3 Self Compacting Mortars (SCMs)

Mortar phase plays a vital role in the successful development of self-compacting concrete, as it contributes positively to the active suspension phase of aggregates. High powder content and optimum fine aggregate is required for the desired flowability and stability of mortar.

Basheerudeen et al. used the particle packing approach for designing the mortar phase of self-compacting concrete. Puntke test (optimization of powder and sand), marsh cone studies (Optimization of SP dosage), and mini slump studies (Optimization of glass fibers) were used to optimize various constituents of self-compacting mortar. The study showed the production of optimized mortar at powder to sand ratio of 40:60, with powder having 60% cement and 40% GGBFS.

This was achieved at 0.1% volume fraction of glass fibers with 2% super plasticizer [5].

2.4 High Strength Concrete (HSC)

Initially used in 1970s, high strength concrete (HSC) is actually the concrete made to exhibit high strength by carefully controlling and selecting its raw materials. The basic technique adopted for its manufacture is the same as that used for ordinary concrete. These concretes were typically used in columns of high rise buildings back then. They differ greatly from the modern concretes in terms of proportions, ingredients, technology and their performance.[21]

2.5 High Performance Concrete (HPC)

High performance concrete (HPC) are the modern high strength concrete. High performance concretes are always high strength but many high strength concretes may not be high performance concretes. They actually are high durability concretes [1].

As per Pierre-Claude Aïtcin et al. [21], the distinguishing parameter for concrete to be either high strength or high performance is the water to binder ratio (w/b). According to him a concrete having low w/b ratio is called high performance concrete. He defines this lower limit of w/b ratio as suggested by Powers et al. (1968) for complete hydration of Portland cement i.e. 0.4. He states various reasons for selection of this figure. First of all, this would be completely irrational to use water less than that require for complete hydration of cement. Secondly, use of lower w/b ratios will invite autogenous shrinkage. Lastly, it would be absolutely impractical to use and place concretes with extremely low w/b ratios.

Apart from using low w/b ratios, the inherent components of HPC also includes super plasticizers (SP). This imparts workability to the system and yields better modulus of elasticity (E) and mechanical strength, reduces permeability and increases abrasion resistance. Thus, overall durability of concrete is improved.

2.6 Secondary Raw Materials (SRMs)

Immense industrialization during the past few decades has led our environment pollute treacherously and has posed massive heads of wastes. But thanks to the introduction of contemporary research, that is resolving the research boundaries to cater for accompanying aspects alongside. Now-a-days apart from the area of specialization, the scientists are trying to address the lateral consequential aspects and probable supplementary benefits of the research as well. Such studies are being carried out in the concrete world likewise. One of the milestones achieved in this context is the successful accommodation of the industrial waste in the production of durable concrete technology. These are typically termed as "Secondary Raw Materials" or "Supplementary Cementitous Materials" in vocabulary of the construction world.

2.6.1 Pozzolan

Pozzolan are mineral admixtures that may be naturally occurring or are artificially made. Naturally occurring pozzolan mostly include volcanic ashes and pumices. Artificial pozzolan are basically industrial by-products for example; meta-kaolin is made from thermal activation of kaolin clay, fly ash from coal fired electricity production, and silica fume is obtained by smelting of silicon. Artificial pozzolan sometimes include organic materials as well, such as rice husk ash (RHA) and bagasse ash (BA). Pozzolan are widely used as secondary raw materials (SRMs) across the world for optimizing cement and concrete properties.

Given below is a brief account of the remarks and findings of different researchers upon the use and behavior of pozzolan in self-compacting cementitous systems.

Sharif et al. states that the pozzolan have little or no cementitious value by itself however they react chemically with alkalis present in presence of water thus contributing to improvement in strength and other properties of concrete [16].

According to Johari et al., different types of materials used as partial cement replacement materials or as mineral additives can have different effects on the properties of cementitious matrices due their different chemical and mineralogical compositions, as well as different particle characteristics, which determine their water requirement, packing ability, and their reactivity when used as part of a binder for concrete. In general, the use of these materials in concrete has been associated with the refinement of the concrete pore structure [22]. Ribeirio et al. presented similar findings [20].

2.6.2 Mechanism of Pozzolanic Behaviour

The pozzolanic activity of a material primarily depends on two factors: the amount of calcium hydroxide available for the reaction with the pozzolan, and the reaction rate that this combination occurs. The amount of calcium hydroxide depends on the chemical properties of the pozzolan used, the nature of its active phase, the content of SiO_2 in the active pozzolan and the $Ca(OH)_2$/pozzolan ratio in the mixture. The reaction rate depends on the physical factors, such as the surface area of the pozzolan, the solid to water ratio of the mixture and the temperature [20].

In simple words, the decisive property for a material to be used as an SRM is either its fineness or the amorphous silica content. For every specific material, only one of these governs to contribute largely to its pozzolanic activity. For some materials, the net pozzolanic activity may be a combination of both these parameters.

Various researchers come up with their own findings of their experimental experiences which are described below.

2.6.2.1 Physical Packing Effect

Improved properties of cementious systems due to the filler effect of the SRM or pozzolan is known as *Physical Filler Effect.* This is sometimes referred to as *fineness of the pozzolan* in literature.

SRMs show improved performance of cement formulations due to physical filler effect when its silica content is in crystalline state. [16]

This is largely due to the fact that pozzolan normally have particle sizes lesser than that of cement, resulting in enhanced overall packing density. This radically increases strength of the cement formulation.

Besides denser packing, density also accounts for reduced porosity- number of pores, their size and connectivity- ensuring lesser water permeability [23]. Reduced permeability ensures lesser bleeding and evaporation, as well as lesser risk of corrosion, thereby augmenting overall durability of the system. [5]

The use of pozzolan in the fine and ultrafine size range along with Portland cement can also allow reaching greater packing density of the mortar or cement mixture, due to the so-called micro-filler effect [24] . The other physical effect that becomes potentially important with the reduction in particle size is the heterogeneous nucleation. In this case, pozzolan fine particles settle in between the clinker crystals, allowing a nucleation of hydrates on foreign fine particles by reducing the energy barrier [25].

2.6.2.2 Chemical Pozzolanic Effect

Sheikh et al. states that silica is the main constituent imparting the pozzolanic activity in all pozzolanic materials [16]

The chemical activity of pozzolan is explained extensively in literature. Initially cement reacts with water to form calcium hydroxide (CH) and calcium hydrate silicate (CSH) gel. This formed calcium hydroxide (CH) reacts with SiO_2 from pozzolan to form additional CSH gel. This supplementary CSH is responsible for additional strength imparted due to the inclusion of pozzolan in the cement mix. [19, 26-30]

Paula et al.[31] found that the composition of SCBA was approximately 84% silica. Similar values were also found by, Lima et al.[32] and Frías et al.[33] who found silica composition percentages of 75% and 70%, respectively in SCBA. SCBA therefore has high levels of silica under normal conditions, and if suitable calcination parameters are used, such as controlled calcination temperatures,

heating rate and burn time, it is possible to keep the silica in the amorphous state [28].

2.6.3 Advantages

Sharif et al. reports that introduction of pozzolanic agro-industrial waste materials not only improves the strength and durability of concrete but are also helpful in reducing global warming through productive disposal of waste material.[16, 34]

Incorporation of finer materials as replacement of cement have shown tremendous improvement in packing density, enhancement of particle distribution, reduction of thermal cracks and in improving mechanical properties [35].

Materials with pozzolanic characteristics may be used to partially replace cement in mortars or concrete, and has been shown to increase the durability of products [36, 37]

Hence, the advantages of using SRMs in concrete systems can be summed up as follows;

- Use of SRM can lead to achievement of desired properties in concrete with lesser amounts of cement in both fresh and hardened state.
- Use of SRM will reduce cement content which is expensive constituent of concrete.
- Filler and chemical effect of SRM give better mechanical properties like compressive and flexural strengths with improved microstructure.
- SRMs are finer than cement hence they give better packing and better volume stability.
- Use of SRMs leads to an environmental friendly concrete systems

2.6.4 Bagasse Ash

Extensive studies have been carried out on the use of bagasse ash in cement formulations. Literature portrays utilization of bagasse ash in SCC systems for

various purposes. Some researchers use its filler affect to augment the cement properties, others use its chemical pozzolanic property to investigate the exhibited behavior in fresh and hardened states. Few authors give attention to the thermal calcination of the ash to enhance its chemical reactivity. A brief account of some of these studies is summarized as under;

Literature extensively asserts that inclusion of bagasse ash as cement replacement helps in modifying the overall behavior of self-compacting cementitous systems by increasing the setting time and water demand, lowering the heat evolved during hydration and improving the ductility of concrete [16, 19, 29, 38-40]. Sharif et al. states that the use of sugarcane bagasse ash as partial replacement of cement not only improves the compressive strength of concrete but also improves the durability of concrete by reducing shrinkage and permeability [16]. This is endorsed by Frías et al. as well [17].

Muangtong et al presented his findings about the pozzolanic behavior of bagasse ash by stating that pozzolanic behaviour of bagasse ash can be attributed to its high silica SiO_2 content which is the main phase of pozzolanic reaction in cement [19].

Different authors describe their findings about the calcination temperatures of bagasse ash. Sharif et al. reports highest strength activity indices (SAIs) of bagasse ash samples calcined at 500ºC for one hour, both at 7 and 28 days than that at higher calcination temperatures. He supports this finding by adding the XRD patterns of his samples, which show rounded amorphous peaks of silica in 500ºC calcination temperature only. Frías et al. [33] notes that even with cross-contamination by soil particles, ash obtained under controlled calcination conditions have higher pozzolanic activity than waste obtained directly from the production line of the sugar cane ethanol industry. Sharif et al. also states that production of bagasse ash for its maximum pozzolanic activity depends on calcination temperature and heating duration, as endorsed by other authors as well [16, 41].

Cordeiro et al. investigated the role of mill type and grinding circuit configuration in laboratory. It was observed that, although different size distributions were produced by the different mills and milling configurations, the

pozzolanic activity of the ground ash was directly correlated to its fineness, characterized by its 80% passing size or Blaine specific surface area. From a low pozzolanic activity of less than 50% of the as-received ash, values above 100% could be reached after prolonged grinding times. Incorporation of an ultrafinely-ground ash in a high-performance concrete in partial replacement of Portland cement (10, 15 and 20% by mass) resulted in no measurable change in mechanical behavior, but improved rheology and resistance to penetration of chloride ions [27].

Preliminary investigations with sugar cane bagasse ash (SCBA) demonstrate that it presents appropriate chemical composition for application as a pozzolan, mainly in regard to its high amorphous silica content [42-45].

One study proposes a kinetic-diffusive model that allows for determination of pozzolanic activity as related to optimum calcining temperature on the activation of wastes from sugar cane industries and their use as pozzolanic material. Research based on the reaction kinetics between lime (calcium hydroxide [CH]), sugar cane straw ash (SCSA), and sugar cane bagasse ash (SCBA) calcined at 800 and 1000 °C (1472 and 1842 °F) is described. The SCBA has less pozzolanic activity than the SCSA, and its pozzolanic reactivity varies substantially with the calcining temperature [46].

Grinding sugar cane bagasse ash (SCBA) with D_{80} values less than 60 μm and Blain fineness above 300 m^2/kg can yield products that can be classified as pozzolan [26, 27].

In a study made by Cordeiro et al., SCBA gave maximum reactivity at burning temperature of 500ºC using Chapelle test. Quantitative XRD analysis based on the Rietveld method was performed using Bruker's Topas v.3 software to determine the amorphous content of this SCBA. A content of amorphous material of 24% was found with an estimated error of ±4%. Besides that, the proportions of cristobalite and quartz were estimated as 16% and 59% (in mass), respectively.[26]

Another evidence of presence of reactive material in SCBA is the result of ^{29}Si nuclear magnetic resonance spectroscopy of hardened cement pastes which indicated that the intensity of the Q^4 peak, characteristic of amorphous silica (chemical shift of-110 ppm), decreased with the hydration development.[26]

A direct relationship exists between the compressive strength of mortar containing SCBA and the Blaine fineness of the ash. On the other hand, the compressive strength of mortar containing SCBA is inversely proportional to SCBA's particle size. According to the investigation of the SCBAs produced by vibratory grinding, the finest SCBA provided the highest packing density of mortar, which generated a higher compressive strength and pozzolanic activity. Moreover, a clear correlation was observed between Chapelle reactivity and fineness of SCBA.[26]

The pozzolanic activity of SCBA was established from the comparison with an insoluble material at the same packing density. In that case, a different behavior was verified in relation to compressive strength of mortars produced with the mineral admixtures, SCBA and quartz. After 28 days of curing, the compressive strength of SCBA mortar was 31% higher than the strength of CQ-T mixture. This discrepancy was also observed in pozzolanic activity, mechanical response, as well as in results from the modified Chapelle method.[26]

Thus, the results suggested that the SCBA presents physico-chemical properties appropriate for its use as mineral admixture and its reactivity was mainly dependent on particle size and fineness.[26]

2.7 Investigational Techniques

Researchers have used various investigational techniques to access different physical and chemical properties of self-compacting cementitious systems. A brief account of these is given below;

2.7.1 Calorimetry

The progress of cement hydration can be mapped out by measuring heat evolved during the reaction. This sole parameter can be an indicative of several short-term and long-term properties of cement formulations. Calorimetric technique can help us track them at an early age.

Heat evolved is directly related to the rate of reaction of cement formulations, and consequently strength development. This corresponds to the setting times of the system as well. Heat liberated also influences the rate of water

evaporation, thereby affecting workability and early shrinkage of cementitious systems. Hence, calorimetry can help us speculate setting times and required curing regimes, strength development patterns and evaluation of thermal cracking.

Better monitoring of concrete heat flow leads to an improved understanding of characteristics of concrete materials and mix proportions.

Calorimetry is performed by recording heat evolved by the system against time. The obtained data is plotted into graphs to attain calorimetric curves. These curves are composed of various peaks, depicting different stages of cement hydration. The first stage consists of a very fast deceleration rate, due to the active consumption of C_3A phase. This is followed by dormant period of cement hydration. In dormant period, the heat evolved drops down to a minimum. Hence, an extended delay is observed in the curve. After this, the curve then attains its thermal peak showing the maximum precipitation of hydration products, calcium hydroxide Ca $(OH)_2$ and CSH gel . This is termed as "acceleration stage" in the literature, which continues until sufficient water and finer cement grains are available for the hydration reaction. Then again, a drop is observed in the heat evolved which becomes almost constant after 72 hours or so.

Literature states that the added SRMs give recurring peaks in the calorimetry, showing the pozzolanic reaction. These added thermal peaks illustrate the additional strength due to the added SRM.[47]

The active calorimetry may consists of the first few days, but the process goes on for months until all the cement grains available are consumed by water.

Rizwan et al. reports that various factors affect rate of calorimetry. These include super plasticizer, w/b ratio, cement type and its content type. The author reports that SP acts as slight retarder for cementitious systems.[48]

2.7.2 Volume Stability of Self Compacting Cementitious Systems

The term "volume stability" basically refers to the expansion or shrinkage of the cementitious systems. This is an important prerequisite of the cementitious systems as large volume instabilities can render the basic purpose of the structure impractical for use.

In general, expansion is caused by the production of expansive materials like calcium hydroxide and ettringite during the hydration reaction, and may be due to the reabsorption of the bled water. However, shrinkage occurs as a result of water consumed during the hydration reaction, uptake of water by the added supplementary cementitious material (SCM) or by water loss due to evaporation. In both cases, the overall deflections cannot be allowed to cross the specified limits. Hence, it is important to keep an eye on the volume instabilities of the cementitious systems.

There are various methods to measure early shrinkage using linear and volumetric techniques. Both the type of methods has their respective pros and cons. But since they yield similar results for the early shrinkage, they are equally good for measuring the early volume stability of cementitious systems. [49]

Literature reports that pozzolanic powders are incorporated in cementitious systems to control heat of hydration and early shrinkage. This improves the microstructure, offering enhanced durability. [48]

Cook et al. [50] reported a direct relation between the rate of degree of hydration and w/c ratio. Literature reports that 35% degree of hydration is achieved during first 24 hours. Hence, the initial 24 hours are very crucial for shrinkage measurement of SCC. [51]

2.7.3 Scanning Electron Microscopy (SEM)

SEM is a technique used for imaging the microstructure of concrete at very large magnifications. This is a very helpful tool to study particle morphology, its porosity and the connectivity of pores. Presence of various phases of cement and the products of hydration can also be detected with the help of this. The interfacial transition zone (ITZ) can also be spotted using SEM.

Such information can help us understand the physical characteristics of the raw materials and the hydration products. This will yield to better understanding of mechanics of water absorption and pore development, and the progress rate of hydration kinetics and strength development. Hence, the compatibility of various materials can be evaluated.

Moreover, the modern SEMs now offer real time ColorScan, ultra fast mapping and Electron imaging, making its efficiency even better. The new P/B-ZAF formalism is extraordinarily tolerant of varying measuring conditions and sample states, yielding reasonable quantitative results even with completely unprocessed samples. The self-calibrating P/B-ZAF analysis provides absolute (none normalized) results without standardizing or reference measurement and is optimally suited for remote spectrum analysis.

SEM analysis can yield significant information about progress of hydration, its products and their packing density. Muangtong et al. studied these parameters of his hardened samples and reported that at low age maturity, sample exhibited more porosity and needlelike ettringite structures. Whereas at high age maturity, sample showed more dense structure of resultant cement which contains less pores and ettringite needles due to the supplementary cementing phase of CSH. CSH gel of the pozzolanic reaction is a principal coordination phase to increase compressive strength of resultant cement. [19]

2.7.4 X-Ray Diffraction (XRD)

X – Ray Diffraction (XRD) is a scientific tool employed for the detection and quantification of crystalline materials. It also yields fundamental data when applied to amorphous solids and liquids.

The seclusion of crystalline and amorphous materials is important in cement based formulations so as to assess the reactivity of the materials. In crystalline materials, elements are struck at specified atomic distances and therefore cannot take part in chemical reaction. However for cement hydration, the basic qualification is to be reactive. The amorphous substances pass this criterion and therefore are preferred for cement formulations

XRD is a useful technique to identify the various amorphous and crystalline structures in any material. Sharif et al. performed XRD of his calcined powder bagasse ash samples and reported that silica SiO_2 is the main constituent imparting the pozzolanic activity in all pozzolanic materials. He reported that round peaks of silica were observed for samples calcined at 500°C thus representing the amorphous state of bagasse ash; however, sharp peak of quartz were observed for

bagasse ash samples calcined at 600°C & 700°C indicating crystallization of bagasse ash. [16]

Muangtong et al. performed XRD of his hardened mortar samples and reported that XRD patterns are detected as tricalcium silicate (Ca_3SiO_5, C_3S), dicalcium silicate (Ca_2SiO_4, C_2S), calcium silicate hydrate (Ca1.5 SiO3.5.×H_2O, C-S-H), calcium hydroxide ($Ca(OH)_2$, CH), quartz (SiO_2), and ettringite ($Ca_6Al_2(SO_4)_3(OH)12.26H_2O$), respectively. At high age maturity, the amount of CH phase decreased while that of CSH phase increased, in the cement replaced with SCBA as compared to that without SCBA. It can be deduced that CH phase reacts with SiO_2 phase to transform into CSH phase in the pozzolanic reaction.[19]

2.7.5 X-Ray Fluorescence (XRF)

XRF technique is used to perform chemical analysis of a substance. It is important to carry out the chemical analysis as this can give us vital information regarding the chemicals present in the raw materials and their most probable products. Essentially, specific raw materials should yield their characteristic products during hydration. The deviations from trends can be investigated to find out the underlying reasons.

This is also done so as to check suitability of the materials to be used in the research work. Satisfying standards makes the research work more worldwide applicable and easy adaptable.

2.7.6 Thermogravimetric Analysis (TGA)

Thermo-gravimetric analysis is a thermal technique used to determine the change in mass of a material as a function of time at a pre-determined temperature, or over a specified temperature range using a pre-determined heating rate. This technique is highly suitable for assessing heat stability and loss of components over time for evaluating durability of materials spanning their service life.

TG analysis yields vital information on the chemical activity of a material. It can be very helpful in predicting the reactivity of pozzolan. Literature widely uses TG analysis technique to predict and verify the pozzolanic activity of SRMs.

Riberio et al. used TG analysis to access the pozzolanic activity of bagasse ash calcined at 500°, 600° and 700° C. He suggested the temperature range of 500° to 700° C for achieving high pozzolanic activity. He also proposed a direct relation between the calcinations temperature and particle size of the resulting ash and an inverse relation with that of the specific gravity due to loss of the organic matter. [28]

CHAPTER 3: EXPERIMENTAL PROGRAM

3.1 General Methodology

The experimental program designed was aimed to check the effect of particle size of controlled burnt and milled bagasse ash on the response of self-compacting mortar systems. The parameters set up for burning and replacement level were taken up from literature, for optimized properties of bagasse ash in SCC systems.

In order to standardize the ash preparation, Los Angeles abrasion machine was employed for milling purpose. The burnt ash was divided in four batches and milling time was fixed for each batch, starting from 5 min, with the increasing interval also to be 5 min. This approach gave us our four testing ash batches having varying mean particle sizes (MPS). The representative samples of these batches were put to Particle Size Distribution (PSD) analysis to obtain their respective D_{50}.

Later, comprehensive particle characterization, of the powders used in the experimental study, was carried out. This included Scanning Electron Microscopy (SEM), X-Ray Diffraction Analysis (XRD) and X-Ray Fluorescence Technique (XRF) of the fines used.

Afterwards, the established formulations were studied for self-compacting mortar properties of flow, calorimetry and shrinkage after determining the Water Demand and Setting Times. The target flow was chosen to be 30 ± 1 cm as given in literature to be called as self-compacting [51], and was achieved using PCE based HRWRA keeping w/p ratio at minimum.

Furthermore, strength tests along with SEM and TGA analyses of the hardened samples was done at the specified age maturities, to have better idea of the hydration products and the hydration kinetics to access microstructure and durability of the samples made.

3.2 Materials /Storage of Materials

The study was made on self-compacting mortar systems (SCMs) [5] having a target flow of 30 ± 1cm [51] and cement: sand ratio equal to 1:2. The replacement

level of the SRM chosen was 20% by weight of cement as mentioned extensively in literature for optimized properties [9, 19, 27, 29, 43, 52]

All the materials used in this experimental study were kept at room temperature. This was ensured by storing the materials in lab at least 24 hours before carrying out the practical work.

3.2.1 Cement

The BestWay Cement, grade 53, conforming to ASTM 150C [53] was employed for the proposed study. Different physical and chemical properties of the cement used are mentioned in Table 4.1 of section 4.3.3 of the subsequent chapter.

3.2.2 Fine Aggregate

Fine aggregate used in the present study was Lawrencepur sand. The selected sand size was <2mm, so the sand was first sieved through ASTM sieve no. 10 (ASTM C807) using a mechanical shaker. The passing sand was collected and oven dried at 100°C for 24 hours. It was then cooled at room temperature for one day. This treated sand was stored in dry air-tight container to avoid contact with moisture.

Well-graded sand was used to ensure better packing of the formulations, leading to improved micro-structure and enhanced durability. Fineness Modulus of the sand used is 2.09.

3.2.3 Super Plasticizer (SP)

Melflux 2651F, manufactured by BASF, was used to carry out the intended experimental program. It is a powder type third generation PCE based SP, produced by spray-drying of a modified poly-carboxylic ether, having side-chains of polyethylene glycol. Its basic properties are listed below in Table 3.1.

Table 3.1: Basic Properties of Melflux 2651F

Appearance	Powder
Color	Yellowish
Bulk density, [g/100cm^3]	30-60
Drying loss, [w/w %]*	Max. 2.0
pH value at 20ºC, 20% solution	6.5-8.5

*by weight of cementitious material

3.2.4 Water

Firstly, the Water Demand of each system was worked out carefully. Then the w/p ratio for each formulation was kept equal to water demand of the system, to ensure complete hydration and allowing least porosity due to excess water.

3.2.5 Bagasse Ash

The Bagasse Ash used was obtained from Premier Sugar Mill, Mardan, Pakistan. It was first cleaned manually to remove any organic matter like hair or stones, and then was treated at controlled conditions to study their response on the chosen self-compacting mortar systems. The treatment of the ash included burning and milling, details of which are summarized under the following heads;

3.2.5.1 Burning of Bagasse Ash

The burning regime chosen was taken up from literature to get rid of all the un-burnt carbon[20]. It was accomplished by utilizing the facility present at Glass and Ceramics Research Centre (GCRC), PCSIR Lahore. Coolyman furnace was used for this purpose. Maximum achievable temperature through this furnace is 1000°C. Intended temperature is maintained by controlling the amount of air allowed, to fire the furnace, with the help of a blower and is monitored consistently via temperature sensors.

The burning regime followed in attaining the required finished product, for this specific study, is tabulated as under;

Table 3.2: Burning Regime of Bagasse Ash

Burning temperature	500ºC
Retention time	1 hour
Burning environment	Semi-oxidizing

Figure 3.1: Coolyman Furnace in Operation

3.2.5.2 Milling of Bagasse Ash

The milling operation of the bagasse ash was carried out by employing the Los Angeles abrasion machine. A comparison of four different particle sizes of bagasse ash was designed for this research work. Physical and chemical properties of various particle sizes used in this study are mentioned in section 4.3.3 of the forthcoming chapter.

Table 3.3: Milling Regimes of Bagasse Ash

Sample Designation	Grinding time (min)	Particle Size (µm)
BA95	5	95.2
BA30	10	30.8
BA28	15	28.2
BA25	20	25.5

Figure 3.2: Los Angeles Abrasion Machine

3.3 Mixing Regime and Mix Proportions of SCM Formulations

Mixing of the mortar systems was carried out using Hobart Mixer of 5L capacity. The mixing regime adopted was picked up from literature for optimized values of flow and SP dosage [48]. Mixing water was added in two sections, 80% and 20% by weight, to boost activity of the super plasticizer (SP). Slow mixing was carried out for 1 min (60 sec) at 145 rpm and fast mixing for 4 min (260 sec) at 285 rpm. Hence, the formulations received a total mixing time of 5 min (300 sec). The detailed mixing regime is summarized in Table 3.4 as follows;

Table 3.4: Analyzed Mixing Regime

Mixing Speed	Mixing Time	Constituents Added
Slow Speed i.e. 145 rpm	00-30 sec (0.5 min)	Dry mixing containing Cement, Sand and SRM (quantities as per the mix design)
Slow Speed i.e. 145 rpm Fast Speed i.e. 285 rpm	30-60 sec (0.5 min) 60-120 sec (1.0 min)	Addition of 80% water
Walls of the container cleaned via hand mixing		
Fast Speed i.e. 285 rpm	120-300 sec (3.0 min)	Addition of 20% water and SP

The designed experimental investigation consisted of five different SCM formulations. One being the control mix, while the others having varying particle sizes of bagasse ash used. As mentioned earlier too, all the formulations used equal replacement percentage of bagasse ash i.e. 20%. The remaining parameters were kept constant too, except for particle size of the bagasse ash used. The detailed mix proportions of various SCM formulations used are given in Table B1 of Annexure B.

3.3.1 Sample Preparation

The moulds casted for strength tests were covered with polythene sheet, to minimize loss of moisture. They were then de-moulded and weighed after 24 hours, and kept in the curing tank till the specified maturity was achieved. The samples were weighed again in SSD condition before strength testing was carried out.

3.4 Investigational Techniques and Methodology

3.4.1 X-Ray Diffraction (XRD)

The four bagasse ash batches were investigated for their crystallography by X-Ray Diffraction (XRD) Analysis. Around 5-10 grams of powder sample was prepared. The measurement parameters provided were:-

- Angular range: 5° to 70° (2 Theta)
- Step size: 0.013°
- Counting time per step: 29 seconds
- X-ray tube power: 40kV/40mA

- X-ray tube anode: Cu
- Radiation Cu Kα

3.4.2 X-Ray Fluorescence (XRF)

Powders including cement and the four bagasse ash samples were analyzed for their chemical composition using JEOL JSX 3202 M (Na-U) Element Analyzer.

XRF is a powerful technique to study the elemental or chemical analysis of any material. It uses X-rays (or gamma rays) to chemically decompose and find out the constituents of any substance. The process involves making palettes of the sample using a Hydraulic Press. Hydraulic press is a simple mechanism that utilizes air pressure to densify the materials. The palettes made are then put into the Element Analyzer to find out the constituents. The typical output is a computer generated report, specifying the components along with their percentages.

3.4.3 Water Demand, Super Plasticizer Demand and Setting Times

The first step in the experimental process included determination of the water demand (WD) and setting times of various self-compacting mortar formulations, using Vicat Apparatus. Firstly, WD of mortar systems was determined as a sum of the water demand of paste and that of the aggregates, in accordance with ASTM standards of C187, C127 and C128 respectively. Later, setting times were worked out in accordance with ASTM C807 Standards.

Powder type third generation PCE based HRWRA was used as super plasticizer (SP) to enhance workability of the SCM systems. Trial and Error approach was employed to determine the SP demand of the formulations to reach the target flow of 30$\pm$1 cm, using Hagerman's Mini Slump Cone Apparatus.

All formulations had mixing water equal to water demand (WD) of the respective formulation. This was done to ensure complete hydration without compromising on strength by allowing porosity via excess water. The Super Plasticizer (SP) content used was as required to obtain the target flow of 30$\pm$1 cm for each formulation.

3.4.4 Flow Times of Self Compacting Mortar Systems

Hageman's Mini Slump Cone Apparatus was employed to measure flow times of the formulations under study. It had a height of 60 mm, with upper and lower diameter equal to 70 mm and 100 mm respectively. The mini slump cone was filled with the mortar formulations having the amount of SP as required to attain the target flow of 30±1 cm. The cone was lifted and the time taken by the formulation to reach the T30 mark was noted down.

3.4.5 Calorimetry

F-Cal 8000 Field Calorimeter was used to trace the heat evolution of the SCM formulations. It is a simple apparatus with 8 calorimetric compartments. Each formulation requires a sample of 600 grams.

Like the apparatus, sample preparation is also quite simple. Formulation just needs to be prepared in the Hobart Mixer and poured into the calorimetric tank. Computer software is then used to digitally record the readings. It has to be logged in 60 min before keeping the sample, to neutralize the inner temperature of the apparatus. The logging in of the calorimeter is started when water is poured into the mix. After that the sample is placed in the calorimetric compartment. Data can be extracted from the logger after the specified time of interest. In addition to the raw data, it also provides the auto-generated graphs.

A calorimetry of five said formulations was performed for 72 hours by the procedure explained above.

3.4.6 Early Shrinkage

Early volume stability of the formulations was assessed using a modified version of German Shrinkage Channel Apparatus. It is a linear shrinkage recording device measuring 4x 6x 25 cm in dimensions. It is capable of sensing of 0.31 µm/m displacement. Its assembly consist two channels: Channel 1 and Channel 2, each having two gates. One of which is fixed while the other is moveable. The latter is attached to the displacement sensing device. The data logger records the readings at an interval of 60 sec through the displacement sensor. It also has an attachment for humidity sensitivity. Temperature variation can also be monitored using this

assembly. Other details of the measuring device can be seen in the supporting literature [54].

Figure 3.3: German Shrinkage Channel

Picture Credits: Niazi MS Thesis, 2010

Preparation of the apparatus consisted of greasing the channel and then placing a neoprene sheet inside it. After that, the formulation was prepared in the Hobart Mixer using the specified mixing regime and poured into the channel. The channel was then covered carefully using a polythene sheet to avoid the moisture loss. Computer Software was used to initiate the start up, then Data Logger digitally recorded the readings. The formulations were kept in this assembly until 72 hours. Afterwards, the data was extracted and imported into Excel for processing. The uaed assembly can be seen in the accompanying picture.

3.4.7 Strength

Standard prisms measuring 4x4x16 cm^3 were casted for determining strengths of the said self-compacting mortar formulations. After being de-moulded and cured, these were tested for flexural and compression strengths as per EN 196-1 for 3, 7 and 28 days respectively. Special care was taken while centering the samples for testing, especially for flexure, otherwise stress concentration could taper the results. The samples were first tested for flexure, which broke the samples into two smaller parts. These broken samples were then used for compression test. Hence, an average of three samples was used to evaluate flexural strength while that of six samples gave the strength in compression.

Figure 3.4: Strength Testing Machine

3.4.8 Scanning Electron Microscopy (SEM)

Particle morphology, microstructure and qualitative characterization were investigated with the aid of Scanning Electron Microscopy (SEM). Powders as well as selected hardened samples of SCM formulations at the ages of 28 days were examined using SEM technique. Scanning Electron Microscope with Field Emission Gun was employed for this purpose. One micro-spatula of powdered sample was required for each set of SEM images. For the hardened samples, hydration was first stopped using Acetone at the specified age and then was grounded after being oven dried for one day. After that the hardened samples were put for sonication 60 min before carrying out SEM, whereas the powders were directly adhered to the examination stub. Afterwards, they were coated with carbon for SEM analysis.

3.4.9 Thermo Gravimetric Analysis (TGA)

Thermo Gravimetric Analysis (TGA) serves as a powerful tool in accessing the heat stability and loss of components over time. For TGA analysis, the sample is first dried and weighed. It is then heated up to the required temperature in an oxidizing environment at a pre-set heating rate. The sample is then allowed to cool down at the same rate. The built in data logger records the data digitally. This data is then imported to excel where it is processed to plot the TGA curves.

All the hardened samples were tested for identification of compounds by TG analysis. It was carried out by using samples obtained from the prisms, tested for flexure and then for compression, at specified ages. Test sample was prepared by grinding the hardened sample into powder and feeding it to the heating chamber. Only a few milligrams of the sample are required. The measurement parameters provided were:-

- Temperature Range: 0-600 °C
- Heating Rate: 10 °C/min
- Atmosphere: Nitrogen
- Sample Coating: Gold

3.5 Specimens Designation

The specimens were designated specifying the SRM used and its respective particle size, followed by age maturity of the formulation. Hence, a typical formulation used in the experimental setup may be written as; BA95-3D. Here, the first two letters signify bagasse ash whereas 95 stand for particle size in microns. The subsequent hyphenated term depicts the age in days of the sample. Complete specimen designation of the experimental samples is summed up in the Table 3.5 that follows;

Table 3.5: Specimen Designation

Sample / Age[Days]	**CM**	**BA95**	**BA30**	**BA28**	**BA25**
3	CM-03	BA95-03	BA30-03	BA28-03	BA25-03
7	CM-07	BA95-07	BA30-07	BA28-07	BA25-07
28	CM-28	BA95-28	BA30-28	BA28-28	BA25-28

CHAPTER 4: RESULTS AND DISCUSSION

General

This chapter will present the results obtained from the experimental program explained in the previous chapter. Discussion on the acquired results will be done alongside with the results presented.

4.1 Particle Size Distribution (PSD) of SRM

Bagasse ash used in the study was prepared using a controlled burning regime, as explained in the previous chapter in detail. It was then divided into four batches and was put to milling for specified time using a commercial grinding machine. The difference in the grinding time gave four different particle sizes of bagasse ash that were used in the study. The Particle Size Distributions of these batches, along with their respective D_{50}, are shown in figure 4.1

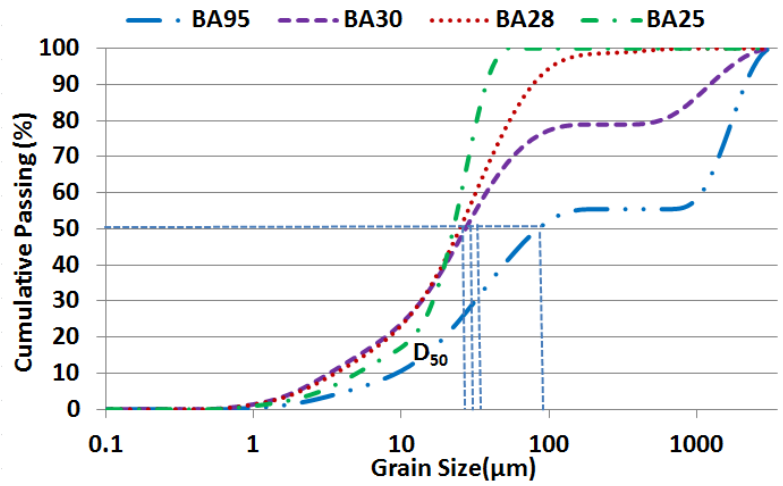


Figure 4.1: Particle Size Distribution of the Four Bagasse Ash Batches

It can be seen from the PSD plot that the SRM used contains two population of particle sizes; one is BA95, having a mean particle size of around 95µm and the other in the range of 25 to 30µm. This difference can help us interpolate the behavior of the particle sizes lying within as well, whilst letting us have an idea of a wide range of particle sizes.

It can also be seen from the PSD plot that the spread of the curves is shrinking with the increasing grinding times. This shows that the intensity of the similar particle sizes is increasing with the increase in the grinding times.

The relation of grinding time (GT) versus mean particle size (MPS) is also shown in figure 4.2, which indicates that with the increasing grinding time, the reduction in the mean particle size (MPS) becomes less prominent, whilst the morphology of the particles starts becoming similar. This is the reason we have got a very similar SEM imaging of BA28 and that of BA25.

4.2 Relation of Grinding Time (GT), Mean Particle Size (MPS) and Specific Surface Area (SSA)

The relation of grinding time (GT), mean particle size (MPS) and specific surface area (SSA) is shown in the figures 4.2 and 4.3. It can be seen from the GT versus MPS plot that MPS decreases drastically in the first 10 min of grinding and after that there is no considerable change in the particle size of the ash being ground. Rather the further grinding works more on the morphology and intensity of the particles. We can see the curves of the PSDs getting steeper with the increased grinding times and morphology of BA28 and BA25 becoming very much alike, as seen in the SEM imaging of the ground ash batches.

The relation of MPS with SSA is rather interesting. As seen in the plot, the SSA increases till 15 min of grinding and after that it got reduced. This is due to the flocculation effect induced in the materials prone to prolonged grinding times. Prolonged grinding induces electrostatic forces of attraction in between the particles which result in formation of floccs, thus decreasing the overall surface area. Moreover, later these floccs act as one unit, as the electrostatic forces are not easy to overcome.

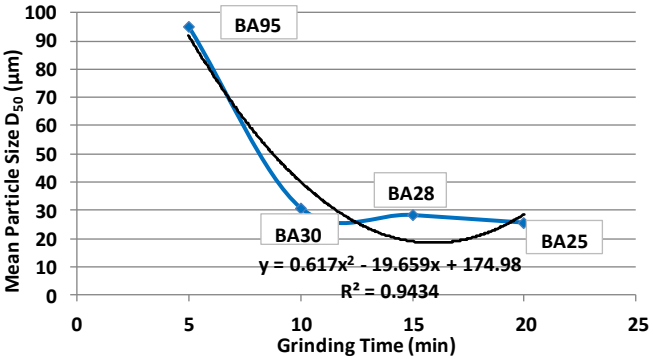


Figure 4.2: Relation of Grinding Time (GT) against Mean Particle Size (MPS)

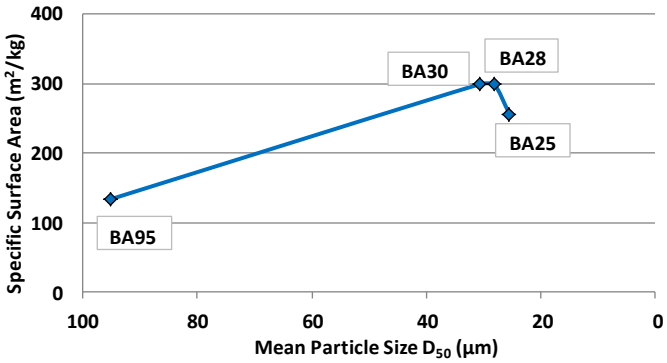


Figure 4.3: Relation of Mean Particle Size (MPS) against Specific Surface Area

4.3 Characterization Results

The four bagasse ash batches prepared through careful burning and milling process were put to a comprehensive particle characterization using Scanning Electron Microscopy (SEM), X-Ray Diffraction Analysis (XRD) and X-Ray Fluorescence technique (XRF). Theses characterization results are presented in separate heads as under;

4.3.1 Morphology of Bagasse Ash by Scanning Electron Microscopy (SEM)

SEM was performed to study the shape and particle morphology of the SRM used in the study. The images, at magnification values of 2 and 10 microns, of the four ash samples are presented in set of figures 4.4 as under.

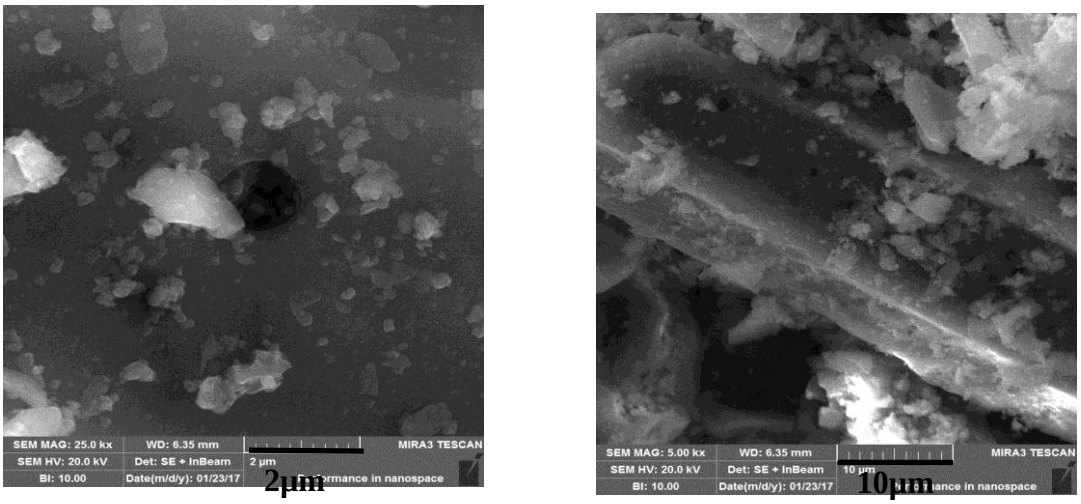


Figure 4.4(a): SEM Characterization of BA95 Particles

Particle size, shape and morphology of the bagasse ash batches are well studied using the scanning electron microscopy (SEM), as shown in the set of figures 4.4. It can be seen that BA95 possesses elongated particles having regular cylindrical shape. The texture seems smooth with minimal surface pores. The BA30 particles holds rather rounded texture with least surface porosity, in all the bagasse ash batches prepared. BA28 and BA25 both possess elongated particles with high visible surface and internal porosity.

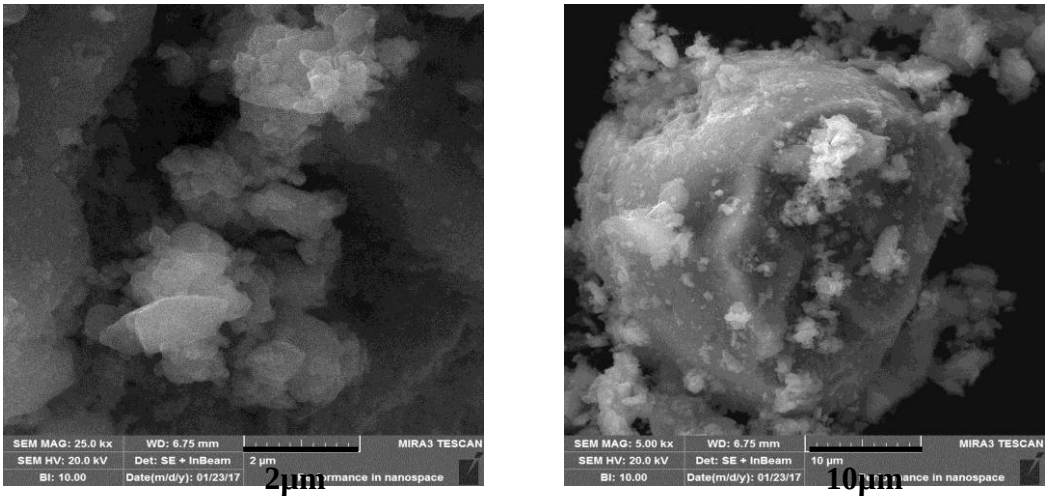


Figure 4.4(b): SEM Characterization of BA30 Particles

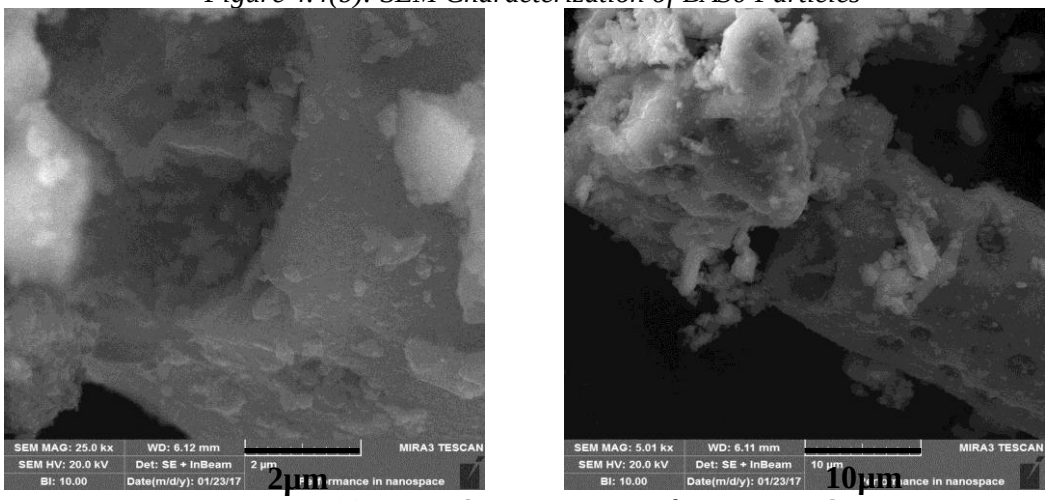


Figure 4.4(c): SEM Characterization of BA28 Particles

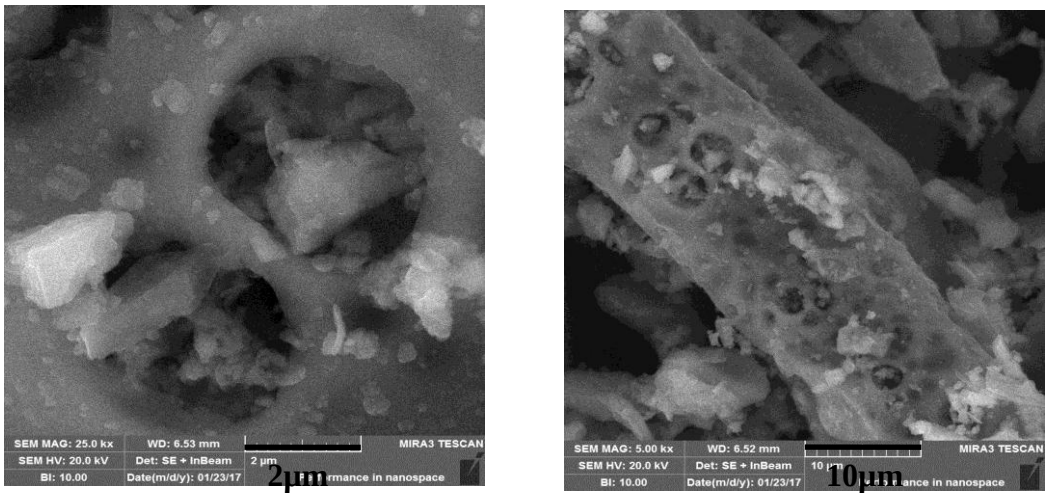


Figure 4.4(d) SEM Characterization of BA25 Particles

Figure 4.4: SEM Characterizations of the Four Bagasse Ash Batches

4.3.2 Crystallography of Bagasse Ash by X-Ray Diffraction (XRD)

XRD was performed to check the crystallographic state of the bagasse ash batches prepared. The XRD patterns of the bagasse ash batches are shown in figure 4.5. The plot shows several peaks of quartz and crystabolite [20]. The sharp peak patterns indicate that the material is partially crystalline. Yet all the bagasse ash batches share the same state of crystallography as shown in the XRD plots.

Since, the objective of this study was to analyze the behavior of particle size in the self-compacting mortar systems; hence, we can say that the material is well prepared for the objective to be met.

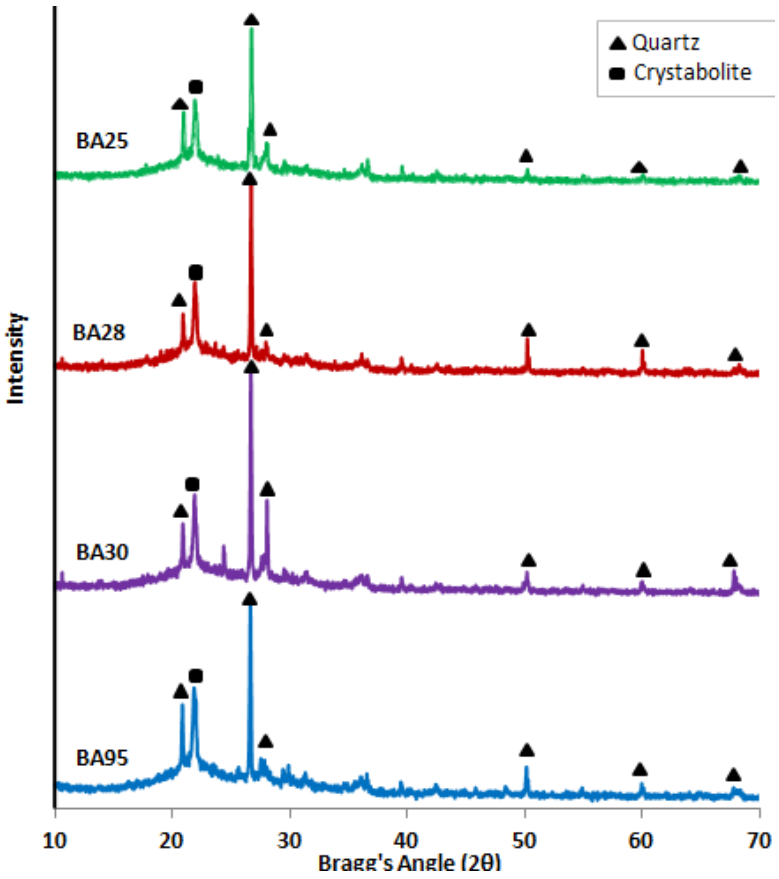


Figure 4.5: XRD Characterization of Bagasse Ash Batches

4.3.3 Physical and Chemical Properties of Powders by X-ray Fluorescence (XRF)

The detailed physical and chemical analyses of powders, bagasse ash batches as well as cement used, using PSD, BET Method and XRF are summed up in the Table 4.1. It can be seen that all the bagasse ash batches contain a similar silica (SiO_2) content of around 60%.It must be noted that this silica content is partially crystalline as verified by the XRD of the ground ashes. Hence, XRD and XRF characterization suggests that ash samples are well prepared for the proposed objective of the study.

Table 4.1: Physical and Chemical Properties of Powders

Properties/Oxides	**OPC**	**Secondary Raw Materials Bagasse Ash**			
	BestWay Grade 53	**BA95**	**BA30**	**BA28**	**BA25**
SiO_2 (%)	19.19	63.66	66.93	65.30	65.52
Al_2O_3 (%)	4.79	2.27	2.34	2.46	2.22
CaO (%)	65	5.78	5.44	4.79	5.01
Fe_2O_3 (%)	3.27	22.74	20.29	18.76	19.24
K_2O (%)	0.51	3.08	2.36	4.79	2.22
TiO_2 (%)	0.29	0.87	0.69	0.74	0.64
SO_3 (%)	1.79	0.23	0.19	0.15	0.15
Specific Surface Area, BET (m^2/kg)	1100	133.5	299	299.2	256.4
Mean Particle size D_{50} (μm)	16.4	95.2	30.8	28.2	25.5
Loss on Ignition (%)	3.84	18.78			
Moisture content (%)	-	5.21			
Density (g/cm^3) (Before milling)	3.19	2.32			

4.4 Fresh Properties of Self Compacting Formulations

4.4.1 Water Demand of SCP Formulations

The results of the water demand determined using Vicat apparatus are presented in the figure 4.6. The SRM used seems to be a hydrophilic material, hence an increase in the water demand can be observed. Also, the WD shares an inverse relation with the particle size of the system. This data is also presented in a tabulated form in Table B2 of Annexure B.

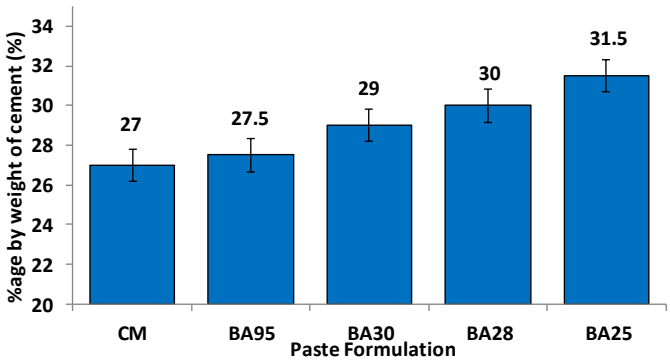


Figure 4.6: Water Demands of SCP Formulations

Literature documents that inclusion of bagasse ash tends to increase water demand of the system [16]. The bagasse ash formulations used in the study also showed an increase in the water demand, as compared to that of the control mix (CM). This increase is attributed to the porous nature of the bagasse ash particles, as observed in the SEM imaging.

The general trend of water demand shares a direct relation with the surface area and an inverse relation with the particle size. Figure 4.6 seems to follow this trend except for BA25, which despite having comparable particle size and lesser surface area than BA28 and BA30, shows higher water demand. This deviation can be easily understood if we look at the SEM imaging of BA25, which shows a very high internal porosity and surface roughness.

4.4.2 Super Plasticizer Demand of SCM Formulations for Target Flow

Figure 4.7 presents super plasticizer demand of the self-compacting mortar formulations under study. As mentioned earlier, the SP demand was determined for the target flow of 30±1cm.

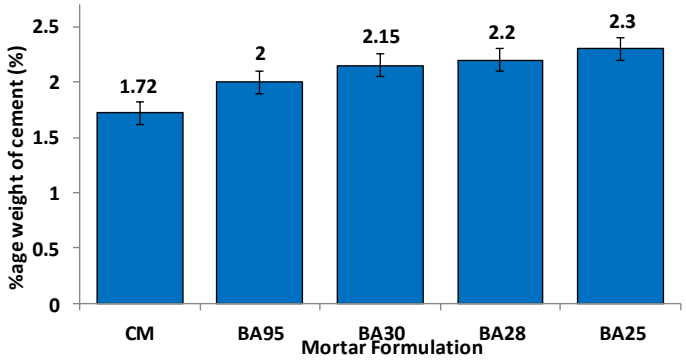


Figure 4.7: Super Plasticizer Demands of SCM Formulations

It can be seen from the plot that SP demand follows similar trend as that of WD. This again is directly related to the particle size and morphology. Regular shaped particles require less SP to overcome the internal resistance, while angular particles require more SP. Surface roughness also accounts for higher SP requirements. Furthermore, it has already been documented that certain amount of super plasticizer is also adsorbed by secondary raw materials depending upon the morphology and surface texture of the particles [55-57]. Hence, SP demand is highest for BA25 and lowest for BA95 after the control mix.

4.4.3 Flow Times of SCM Formulations

The flow values of the SCM formulations are shown in figure 4.8. Data in tabulated form is also given in Table B2 of Annexure B.

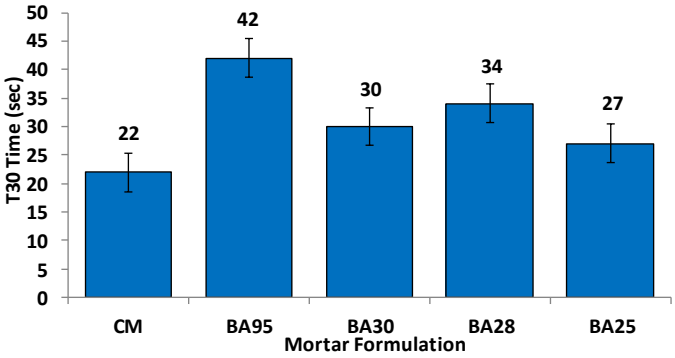


Figure 4.8: Flow Values of SCM Formulations for the target flow

Flow is the principle property of the self-compacting concrete systems indicating its free unrestricted passing-ability. It signifies the yield stress and deformability of the SCC system. The parameters governing flow are the particle shape, size and morphology. The key factor controlling the flow is particle size. Larger particles offer more hindrance to flow while the smaller ones ease the deformability. Particles' shape also plays a vital role in determining the flow times. Regular and rounded particles offer less resistance and hence present lower flow times than the irregular ones. Surface roughness also accounts for affecting the flow times, higher roughness results in higher flow times.

We can see from the plots that the control mix presents least flow times, as the bagasse ash formulations contain the large rough particles of the SRM which offer more resistance to flow. Also, amongst the bagasse ash formulations, we can see that BA95 has maximum flow times due to its large particle size. Now, we can see that the particle size of BA30, BA28 and BA25 are comparable so, here the determining factor for flow time is the particle shape and morphology. Since, BA30 has rather rounded particles with a very smooth surface texture; it presents flow times less than that of BA28. Again, we can see that the flow times have increased for BA28. This is due to the surface roughness of the BA28 particles, as seen in the SEM imagery also. We have already discussed that BA28 and BA25 have a very similar morphology so again particle size governs and BA25 gives lesser flow times due to smaller particle size of its ash particles.

4.4.4 Setting Times of SCP Formulations

The vicat setting times of the SCP formulations are shown in Figure 4.9. Data in tabulated form is also given in Table B2 of Annexure B.

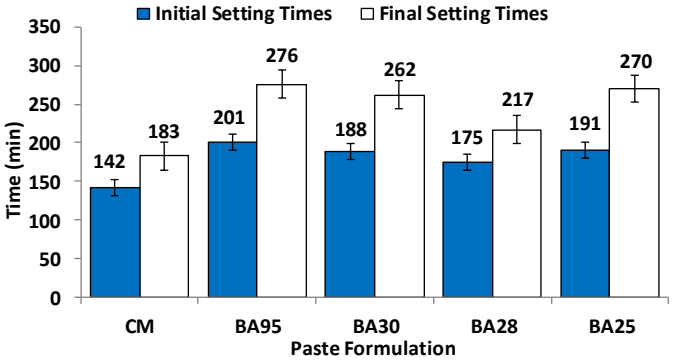


Figure 4.9: Vicat Setting Times of SCP Formulations

In general, setting times of the SCM formulations are retarded by the addition of bagasse ash as cement replacement, as seen in the setting times plot of the SCM formulations. This is due to the dilution effect of the bagasse ash in the mortar matrix. The added bagasse ash increases inter cement distance, due to its larger particle sizes as compared to the cement grains, and consequently the distance between the hydration products resulting in longer setting times.

The trend of the setting times in the bagasse ash formulations is principally governed by the relative inter-cement distance. We can see that BA95 gave the longest setting times owing to its largest particle size amongst the bagasse ash formulations. Now, we can see that despite having least MPS BA25 surpassed BA28 and BA30 and gave the second highest setting times. This is due to the flocculation effect imparted due to prolong grinding times, inducing electrostatic forces of attraction between the particles. This has also been established earlier that these floccs act as one unit in any medium as these electrostatic forces are not easy to overcome. So, these floccs provide with the larger effective inter cement distance, as compared to that provided by BA28 and BA30 particles, in the mortar matrix.

4.4.5 Calorimetry of SCM Formulations

Calorimetry of the mortar formulations was performed to analyze effect of addition of SRM on the heat released in the hydration reaction. Figure 4.11 shows graphic representation of heat flow of the self-compacting mortar systems for duration of 72 hours.

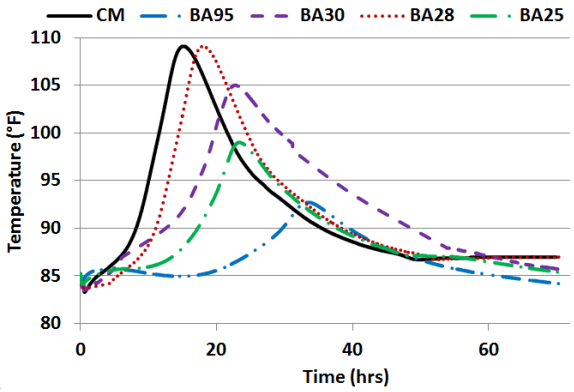


Figure 4.10: Calorimetric Plots of SCM Formulations

Calorimetry presents the same trend as that of the strength. The underlying reason also appears to be the same. Control mix presents the highest heat evolved during hydration due to closely placed cement particles. The heat evolved are lowered and(or) the peaks are being delayed with the increase in the effective inter cement distance in the bagasse ash formulations [16]. Hence, order of the highest to lowest heat flow, recorded in the mortar formulations is control mix CM, BA28, BA30, BA25 and BA95 respectively.

4.4.6 Shrinkage of SCM Formulations

Shrinkage studies were performed to see early volumetric flux due to the addition of SRM of varying particle size. A comparative plot of the shrinkage measurements is presented in Figure 4.

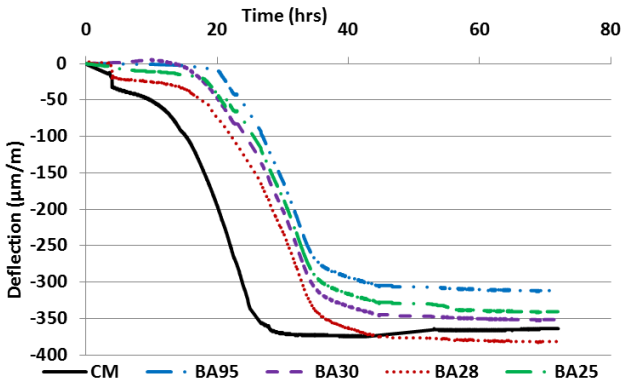


Figure 4.11: Shrinkage Plots of SCM Formulations

Shrinakage also follows the trend of strength and calorimetry. Increased volumetric stability in the bagasse ash formulation is due to the dilution of the cement concentrations. Later again, the effective inter cement distance governs the shrinkage pattern amongst the

bagasse ash formulations. Hence, the order of volumetric stability of the mortar formulations is CM, BA28, BA30, BA25 and BA95 respectively.

4.5 Hardened Properties of Self Compacting Mortar Formulations

4.5.1 Strength Data

Strength tests of flexure and compression were performed on the hardened samples at 3, 7 and 28 days. Their comparative plots are presented in set of figures 4.10.

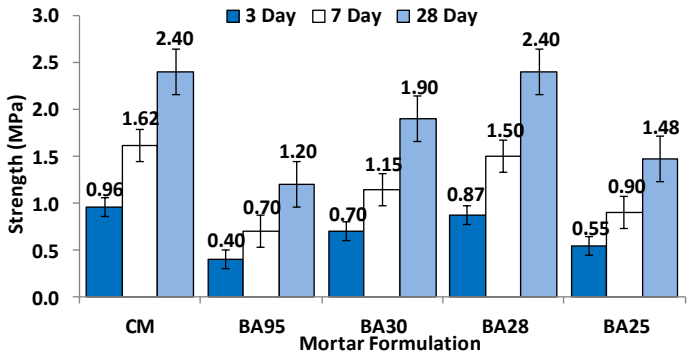


Figure 4.12(a): Flexural Strengths of Hardened Samples

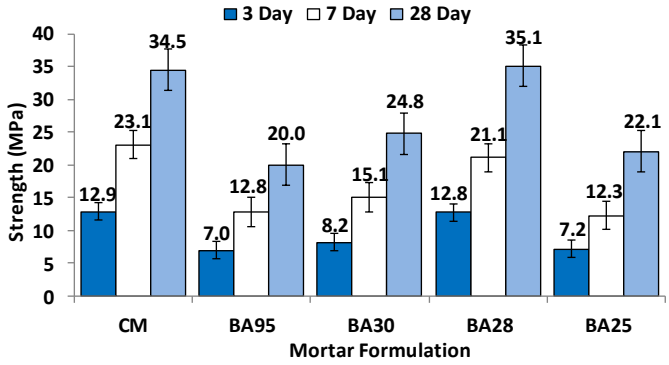


Figure 4.12(b): Compressional Strengths of Hardened Samples

Figure 4.12: Mechanical Strengths of Hardened Samples

We can see from strength plots that control mix presented the highest strength in the mortar formulations at all ages, both in flexure as well as compression. This is due to the dilution effect of bagasse ash in the mortar matrix which increased the inter cement distance, thereby moving the hydration products apart and consequently decreasing the strength. Larger is particle size of the SRM added, larger will be the inter cement distance and lower will be the strength developed. This can quite explain the strength trend obtained for the mortar samples.

One thing is to be noticed in the strength plots that, the flexural strength of BA28 is the same as that of the control mix at 28 days and the compressional strength of BA28 even surpasses that of CM. This is due to the fact that the development of well-formed hydration products depends highly on the space available. In the pure cement mortar formulation the cement grains are closer together, so the CH crystals do not get enough space; hence their formation is compromised. Whereas in BA28 formulation, the adequate

inter cement distance leads to development of well-formed CH crystals presenting better strength.

Also, the similar strengths of BA28 and that of the control mix at 28 days implies that 20 percent replacement level of bagasse ash is the optimum level of cement replacement for ground bagasse ash as a filler material in SCM systems, as reported earlier in literature too.

4.5.2 Thermo gravimetric Analysis of Hardened Samples

TGA analysis was performed as a confirmatory test to verify the findings of the strength trend at all ages. The results were found coordinating and are presented in set of figures 4.13.

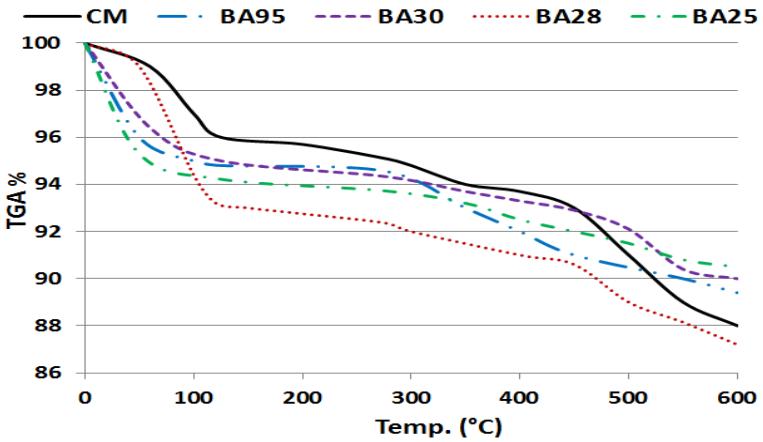


Figure 4.13(a): TGA of Hardened Samples at 3 Days

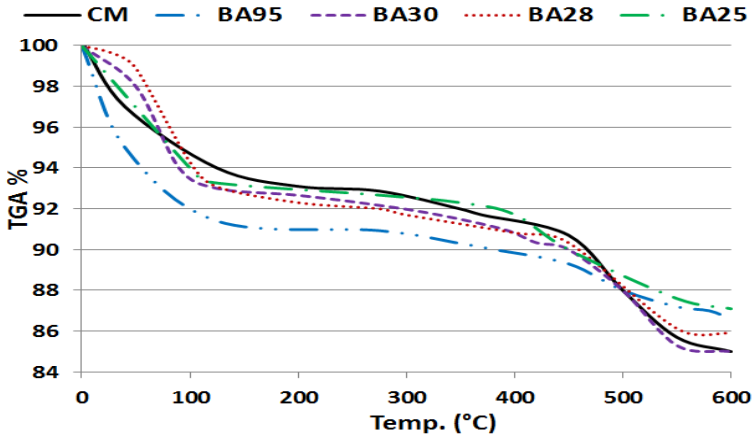


Figure 4.13(b): TGA of Hardened Samples at 7 Days

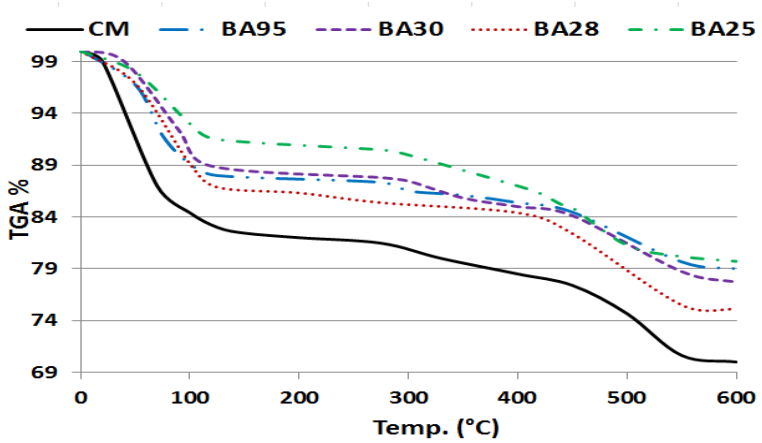


Figure 4.13(c): TGA of Hardened Samples at 28 Days

Figure 4.13: TGA of Hardened Samples at Various Ages

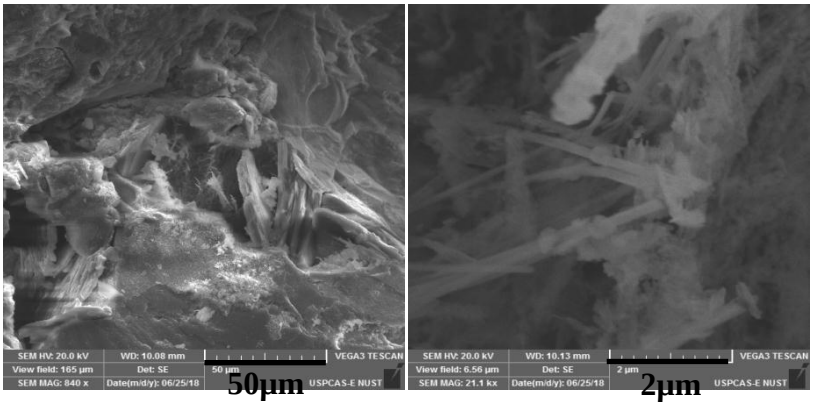
The decomposition range of calcium hydroxide (CH) is 450-550ºC [58]. Highest weight loss values of CM in this temperature range indicate highest CH concentrations and hence more strength at all ages. The subsequent samples in CH concentrations as found by TGA analysis are BA28, BA30, BA25 and BA95 at all ages. This data is presented in tabulated form in Table 4.2 as under;

Table 4.2: % weight loss in TGA from 450-550 ºC, corresponding to the decomposition of CH

Sample / Age	CM	BA95	BA30	BA28	BA25
3	4.0	1.0	2.5	2.45	1.2
7	5.0	2.1	3.9	4.2	2.4
28	6.77	4.8	5.4	6.9	4.72

4.5.3 SEM and EDS of Hardened Samples

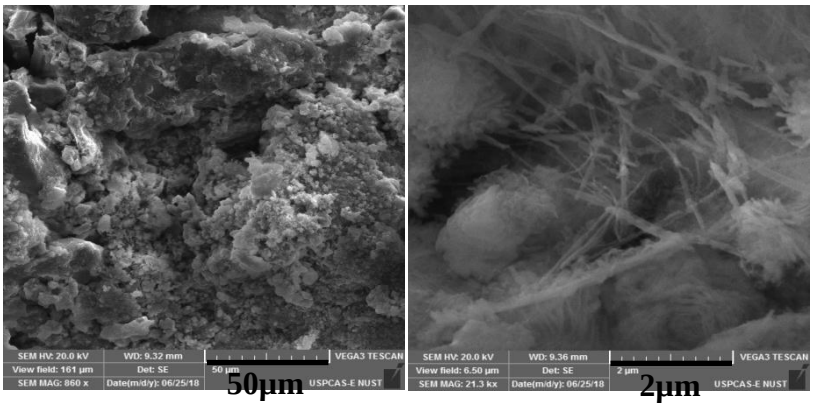
Scanning Electron Microscopy (SEM) along with Energy Dispersive Spectroscopy (EDS) analysis was performed again on the hardened samples to study the micro-structure. The imaging, performed on the selected 28 day hardened samples, is presented in the set of figures 4.14 as follows;



EDS

Element	Weight%
CaCO3	17.51
SiO2	55.54
Al2O3	0.48
SiO2	2.49
Wollastonite	23.98

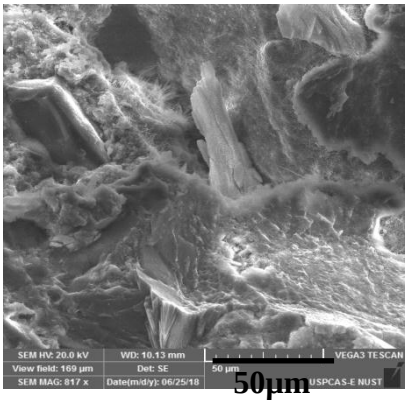
Figure 4.14(a): Pure Self-Compacting Mortar Sample



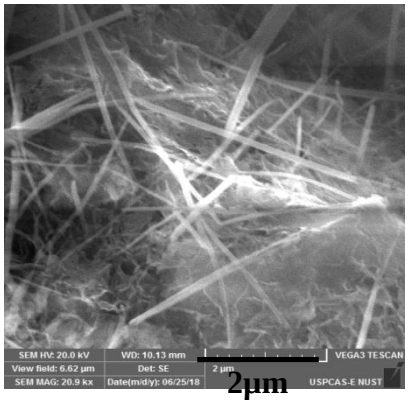
EDS

Element	Weight%
$CaCO_3$	9.41
SiO_2	60.82
MgO	0.74
Al_2O_3	10.03
SiO_2	12.94
Feldspar	4.02
Wollastonite	0.89
Fe	1.14

Figure 4.14(b): Self-Compacting Mortar Sample having Bagasse Ash in Replacement Mode with MPS of 30µm



50µm



2µm

EDS

Element	**Weight%**
$CaCO_3$	6.16
SiO_2	65.67
Albite	0.73
Al_2O_3	5.59
SiO_2	16.17
Feldspar	5.15
Wollastonite	0.53

Figure 4.14(c): Self-Compacting Mortar Sample having Bagasse Ash in Replacement Mode with MPS of 25µm

Fig 4.14: 28 Day Scanning Electron Microscope Presentations of Hydration Products along with EDS

The 28 day hardened samples of control mix, BA30 and BA25 were put to SEM and EDS analysis as a secondary confirmatory test .SEM and EDS analyses confirm the findings of the strength trend.

The low calcium oxide percentages in bagasse ash samples advocated the dilution effect of the cement concentrations than that in the sample of control mix. The order of decreasing calcium oxide percentages by EDS analysis are CM, BA30 and BA25. The SEM micrographs validate the findings of EDS analysis by showing more ettringite growth in BA25 sample, BA30 trails whereas CM presents least ettringite concentrations.

4.6 Summary of Results

- BA25 gave highest WD and SP demand due to least MPS and roughest surface morphology amongst all the bagasse ash formulations.
- BA95 gave longest flow times due to its elongated and large sized particles.
- Delayed setting times are observed due to the dilution effect of cement in the bagasse ash formulations. Higher is the effective inter cement distance offered by the bagasse ash batch, higher are the setting times.
- Bagasse ash batch providing the least effective inter cement distance in SCM formulations gave max strength and showed better volumetric stability and lower heat evolved during hydration.
- TGA analysis showed highest CH concentrations in BA28 after CM, reflecting the highest strength development of BA28 amongst the bagasse ash formulations, at all ages.
- SEM and EDS also exhibited highest Calcium oxide concentrations in CM, reflecting the dilution effect of SCM formulations due to bagasse ash.

CHAPTER 5 - CONCLUSIONS and RECOMMENDATIONS

5.1 Conclusions

- Grinding gave significant reduction in particle size in the first 10 minutes, after that there is no considerable change in the particle size.
- Flocculation effect was observed after 15 minutes of grinding that led to the reduction in SSA of BA25.
- In the light of the above stated findings, it can be said that BA28 is the optimal particle size for cement replacement in SCM systems presenting the best overall response in strength attainment, volume stability and heat evolution during hydration and hence offers enhanced durability along with improved strength as compared to the control mix.

5.2 Recommendations

- Other properties like viscosity, air content and porosity be studied through Rheometer, Air Content Meter and MIP respectively
- Additional confirmatory techniques like XRD, MIP and FTIR analysis be used to better understand the effect of various particle sizes
- Direct Pozzolanic Activity tests be employed to have a direct idea of the pozzolanic reactivity of the SRM used e.g. Chapelle test
- Bagasse Ash be used in concrete systems instead of mortars to study the effect in presence of coarse aggregate
- Bagasse Ash prepared using different burning regimes be used in replacement mode to draw comparison of the properties exhibited

References

1. Okamura, H. and M. Ouchi, *Self-compacting concrete.* Journal of advanced concrete technology, 2003. **1**(1): p. 5-15.
2. ACI, *ACI Concrete Terminology*. 2013.
3. ASTM, C., *125 Standard terminology relating to concrete and concrete aggregates.* Annual Book of ASTM Standards, 2003. **4**.
4. EFNARC, *European Guidelines for Self-Compacting Concrete.* 2005.
5. Basheerudeen, A. and S. Anandan, *Particle Packing Approach for Designing the Mortar Phase of Self Compacting Concrete.* Engineering Journal, 2014. **18**(2): p. 127-140.
6. Dinakar, P., K.P. Sethy, and U.C. Sahoo, *Design of self-compacting concrete with ground granulated blast furnace slag.* Materials & Design, 2013. **43**: p. 161-169.
7. Obaid, H., *PRODUCTION OF LOW COST SELF COMPACTING CONCRETE USING BAGASSE ASH* in *NICE*2006, NUST. p. 25.
8. C1697, A., *Standard Specification for Blended Supplementary Cementitious Materials.* Annual Book of ASTM Standards, 2016.
9. Hussein, A.A.E., et al., *Compressive Strength and Microstructure of Sugar Cane Bagasse Ash Concrete.* Research Journal of Applied Sciences, Engineering and Technology, 2014. **7**(12): p. 2569-2577.
10. Ajay Goyal, A., K. Hattori, and H. Ogata. *Properties of sugarcane bagasse ash and Its Potential as Cement-Pozzolana Binder*. in *Twelfth International Colloquium on Structural and Geotechnical Engineering, Ain Shams University, Faculty of Engineering, Department of Structural Engineering*. 2007.
11. Service, U.F.A., *Global Agricultural Information Network*, 2013.
12. Niazi, B.S., *A STUDY OF SELF COMPACTING CEMENTITIOUS SYSTEMS USING BAGASSE ASH AND BENTONITE*, in *NICE*2010, NUST. p. 3-4.
13. (JSCE), J.S.o.C.E., *Recommendation for Self-Compacting Concrete*.
14. Yeh, I.-C., *Modeling of strength of high-performance concrete using artificial neural networks.* Cement and Concrete Research, 1998. **28**(12): p. 1797-1808.
15. Ogbeide, S., *Developing an optimization model for CO2 reduction in cement production process.* Journal of Engineering Science and Technology Review, 2010. **3**(1): p. 85-88.
16. Sharif, B., R. Firdous, and M.A. Tahir, *Development of Local Bagasse Ash as Pozzolanic Material for Use in Concrete.* Pakistan Journal of Engineering and Applied Sciences, 2016.
17. Sujjavanich, S., W. Mairiang, and S. Sinthavorn, *Some effects on datum temperature for maturity application on fly ash concrete.* Editorial Board: Natural Sciences Social Sciences, 2004: p. 150.
18. Worrell, E., et al., *Carbon dioxide emissions from the global cement industry 1.* Annual Review of Energy and the Environment, 2001. **26**(1): p. 303-329.

19. Muangtong, P., et al., *Effects of fine bagasse ash on the workability and compressive strength of mortars.* Chiang Mai J. Sci, 2013. **40**(1): p. 126-134.
20. Ribeiro, D.V. and M.R. Morelli, *Effect of Calcination Temperature on the Pozzolanic Activity of Brazilian Sugar Cane Bagasse Ash (SCBA).* Materials Research, 2014. **17**(4): p. 974-981.
21. Aïtci, P.-C., *High Performance Concrete*, 1998. p. 5-6.
22. Johari, M.M., et al., *Influence of supplementary cementitious materials on engineering properties of high strength concrete.* Construction and Building Materials, 2011. **25**(5): p. 2639-2648.
23. Bentz, D.P., et al., *Effects of cement particle size distribution on performance properties of Portland cement-based materials.* Cement and Concrete Research, 1999. **29**(10): p. 1663-1671.
24. Goldman, A. and A. Bentur, *The influence of microfillers on enhancement of concrete strength.* Cement and Concrete Research, 1993. **23**(4): p. 962-972.
25. Lawrence, P., M. Cyr, and E. Ringot, *Mineral admixtures in mortars: Effect of inert materials on short-term hydration.* Cement and Concrete Research, 2003. **33**(12): p. 1939-1947.
26. Cordeiro, G., et al., *Pozzolanic activity and filler effect of sugar cane bagasse ash in Portland cement and lime mortars.* Cement and concrete composites, 2008. **30**(5): p. 410-418.
27. Cordeiro, G.C., et al., *Ultrafine grinding of sugar cane bagasse ash for application as pozzolanic admixture in concrete.* Cement and Concrete Research, 2009. **39**(2): p. 110-115.
28. Cordeiro, G.C., R.D. Toledo Filho, and E.M.R. Fairbairn, *Effect of calcination temperature on the pozzolanic activity of sugar cane bagasse ash.* Construction and Building Materials, 2009. **23**(10): p. 3301-3303.
29. Chusilp, N., C. Jaturapitakkul, and K. Kiattikomol, *Utilization of bagasse ash as a pozzolanic material in concrete.* Construction and Building Materials, 2009. **23**(11): p. 3352-3358.
30. Anderson, D., et al., *A preliminary assessment of the use of an amorphous silica residual as a supplementary cementing material.* Cement and Concrete Research, 2000. **30**(3): p. 437-445.
31. Paula, M.O.d., *Potencial da cinza do bagaço da cana-de-açúcar como material de substituição parcial de cimento Portland.* 2006.
32. Lima, S.A., et al., *Análise de argamassas confeccionadas com a cinza do bagaço da cana-de-açúcar em substituição ao agregado miúdo.* Revista Tecnológica, 2010: p. 87-97.
33. Frías, M., E. Villar-Cociña, and E. Valencia-Morales, *Characterisation of sugar cane straw waste as pozzolanic material for construction: calcining temperature and kinetic parameters.* Waste management, 2007. **27**(4): p. 533-538.
34. Sharif, M. and M. Tahir, *Development of Local metakaolin as a pozzolanic material.* Mehran University Research Journal of Engineering and Technology, 2010. **29**(1): p. 89-96.
35. Nanthagopalan, P. and M. Santhanam, *Experimental investigations on the influence of paste composition and content on the properties of self-*

compacting concrete. Construction and Building Materials, 2009. **23**(11): p. 3443-3449.
36. Cook, D.J., R.P. Pama, and B.K. Paul, *Rice husk ash-lime-cement mixes for use in masonry units.* Building and Environment, 1977. **12**(4): p. 281-288.
37. Yogananda, M. and K. Jagadish, *Pozzolanic properties of rice husk ash, burnt clay and red mud.* Building and Environment, 1988. **23**(4): p. 303-308.
38. Singh, N., V. Singh, and S. Rai, *Hydration of bagasse ash-blended portland cement.* Cement and Concrete Research, 2000. **30**(9): p. 1485-1488.
39. Srinivasan, R. and K. Sathiya, *Experimental study on bagasse ash in concrete.* International Journal for Service Learning in Engineering, Humanitarian Engineering and Social Entrepreneurship, 2010. **5**(2): p. 60-66.
40. Ali, K., N. Amin, and M.T. Shah, *Physicochemical study of bagasse and bagasse ash from the sugar industries of NWFP, Pakistan and its recycling in cement manufacturing.* Journal of the chemical society of Pakistan, 2009. **31**(3): p. 375-378.
41. Frías, M., E. Villar, and H. Savastano, *Brazilian sugar cane bagasse ashes from the cogeneration industry as active pozzolans for cement manufacture.* Cement and concrete composites, 2011. **33**(4): p. 490-496.
42. Payá, J., et al., *Sugar‐cane bagasse ash (SCBA): studies on its properties for reusing in concrete production.* Journal of Chemical technology and Biotechnology, 2002. **77**(3): p. 321-325.
43. Ganesan, K., K. Rajagopal, and K. Thangavel, *Evaluation of bagasse ash as supplementary cementitious material.* Cement and concrete composites, 2007. **29**(6): p. 515-524.
44. Martirena Hernández, J.F., et al., *Use of wastes of the sugar industry as pozzolana in lime-pozzolana binders: study of the reaction.* Cement and Concrete Research, 1998. **28**(11): p. 1525-1536.
45. Singh, N.B., V.D. Singh, and S. Rai, *Hydration of bagasse ash-blended portland cement.* Cement and Concrete Research, 2000. **30**(9): p. 1485-1488.
46. Villar-Cocina, E., M.F. Rojas, and E.V. Morales, *Sugar cane wastes as pozzolanic materials: application of mathematical model.* ACI Materials Journal, 2008. **105**(3): p. 258.
47. Rahhal, V. and R. Talero, *Calorimetry of Portland cement with silica fume, diatomite and quartz additions.* Construction and Building Materials, 2009. **23**(11): p. 3367-3374.
48. Rizwan, S.A., T.A. Bier, and M.S. Nizami, *High Performance Self-Compacting Mortars Containing Pozzolanic Powders*, in *Brittle Matrix Composites 8*2006, Woodhead Publishing. p. 175-186.
49. Lura, P., *Autogenous strain of cement pastes with superabsorbent polymers*2006. 57-65.
50. Cook, R.A. and K.C. Hover, *Mercury porosimetry of hardened cement pastes.* Cement and Concrete Research, 1999. **29**(6): p. 933-943.
51. Syed Ali, R. and A.B. Thomas, *Self-Consolidating Mortars Using Various Secondary Raw Materials.* Materials Journal. **106**(1).

52. Sua-iam, G. and N. Makul, *Use of increasing amounts of bagasse ash waste to produce self-compacting concrete by adding limestone powder waste.* Journal of Cleaner Production, 2013. **57**: p. 308-319.
53. C150-01, A., *"Standard Specification for Portland Cement"*, 2004, Annual Book for ASTM Standards, American Society of Testing & Materials.
54. Rizwan, S.A., *High – Performance Mortars and Concretes using Secondary Raw Materials*, October 2006, Technical University: Freiberg, Germany.
55. Bier, S.A.R.a.T. *Early Volume Changes of High-Performance Self-Compacting Cementitious Systems Containing Pozzolanic Powders*. in *Proceedings of Intl RIELM Conference on Volume Changes of Hardening Concrete: Testing and Mitigation*. 2006. Technical University of Denmark, Lyngby, Denmark.
56. Bier, S.A.R.a.T.A. *Role of Mineral Admixtures in High Performance Cementitious Systems*. in *2nd All Russian International Conference, Vol. 3, Concrete Technology*. 2005. Moscow, Russia.
57. Kada, H.B.W., E. and Duthoit, B., *Determination of Apparent Activation Energy of Concrete by Isothermal Calorimetry.* Cement and Concrete Research, Vol. 30, 2000: p. pp. 301-305.
58. Tantawy, M., A. M. El-Roudi, and A. Salem, *Utilization of Bagasse Ash as Supplementary Cementitious Material*. Vol. 3. 2014. 1342-1348.

ANNEXURE A -ASTM TABLES AND CHARTS

Table A1 ASTM C807 Sieve Sizes

Millimeters	Microns	Inches	ASTM Sieve	Tyler Sieve	*ANSI Table 2	*ANSI Table 3
5.60	5600	0.220	3 1/2	3 1/2	S-S	-
4.75	4750	0.187	4	4	4	-
4.00	4000	0.157	5	5	5	-
3.35	3350	0.132	6	6	6	-
2.80	2800	0.110	7	7	7	-
2.36	2360	0.093	8	8	8	-
2.00	2000	0.079	10	9	10	-
1.70	1700	0.067	12	10	12	-
1.40	1400	0.055	14	12	14	-
1.18	1180	0.046	16	14	16	16
1.00	1000	0.039	18	16	20	20
0.850	850	0.033	20	20	22	24
0.710	710	0.028	25	24	24	-
0.600	600	0.024	30	28	30	30
0.500	500	0.02	35	32	36	36
0.425	425	0.018	40	35	40	-
0.355	355	0.014	45	42	46	46
0.300	300	0.012	50	48	54	54
0.250	250	0.010	60	60	60	60
0.212	212	0.008	70	65	70	70
0.180	180	0.007	80	80	80	80
0.150	150	0.006	100	100	90	90
0.125	125	0.005	120	115	100	100
0.106	106	0.004	140	150	120	120
0.075	75	0.0030	200	200	150	150
0.063	63	0.0025	230	250	180	180
0.053	53	0.0021	270	270	220	220
0.045	45	0.0018	325	325	240	240

ANNEXURE B – MIX PROPORTIONS, WATER DEMAND, SUPER PLASTICIZER DEMAND, SETTING TIMES AND FLOW VALUES OF SCM FORMULATIONS

Table B1 *Mix Proportions of SCM formulations

Formulation Number	Mortar Formulation	w/b	Cement	SRM	Sand	Super Plasticizer
			(g)	(g)	(g)	(% by weight of Cement)
1	CM	0.270	200	-	400	1.72
2	BA95	0.275	160	40	400	2
3	BA30	0.290	160	40	400	2.15
4	BA28	0.300	160	40	400	2.2
5	BA25	0.315	160	40	400	2.3

*calculations for 600 g of mortar formulation.

Table B2 Water Demand, Super Plasticizer Demand for Target Flow, Setting Times and Flow Values of SCM formulations

System	Water Demand	Super Plasticizer Demand	Setting Times		Flow	
			Initial	Final	T25	T30
	(%)	(% by weight of cement)	(min)	(min)	(sec)	(sec)
CM	27	1.72	142	183	8	36
BA95	27.5	2	201	262	9	42
BA30	29	2.15	188	270	5	30
BA28	30	2.2	175	217	5	34
BA25	31.5	2.3	191	276	5	27

ANNEXURE C – STRENGTH RESULTS

Table C1 Flexure and Compression Strength of SCM formulations

SCM Formulation	Strength (MPa)					
	Flexure			Compression		
	3 Day	7 Day	28 Day	3 Day	7 Day	28 Day
CM	0.96	1.62	2.40	12.89	23.10	34.51
BA95	0.40	0.70	1.20	6.97	12.80	20.00
BA30	0.70	1.15	1.90	8.20	15.10	24.78
BA28	0.87	1.50	2.40	12.77	21.10	35.10
BA25	0.55	0.90	1.48	7.20	12.30	22.09

ANNEXURE D – PARTICLE SIZE DISTRIBUTION

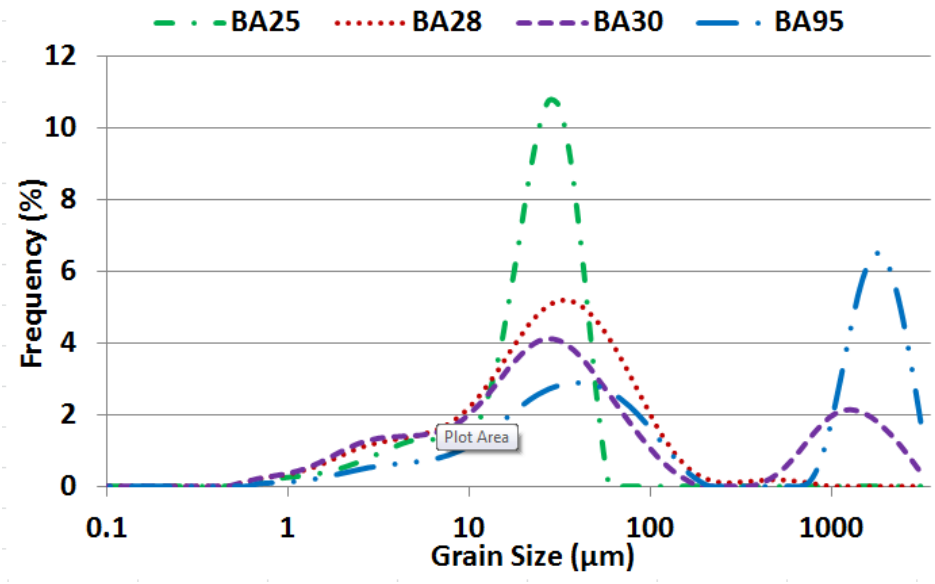


Figure D1 Particle Size Distribution

ANNEXURE E – EDS RESULTS

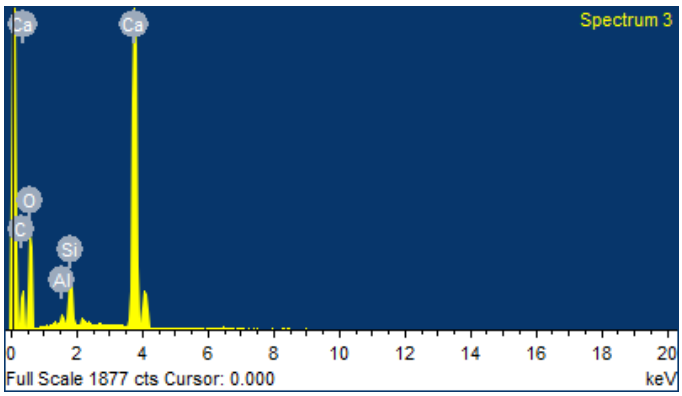


Figure E1 (a) EDS Spectrum of 28 Day Hardened CM

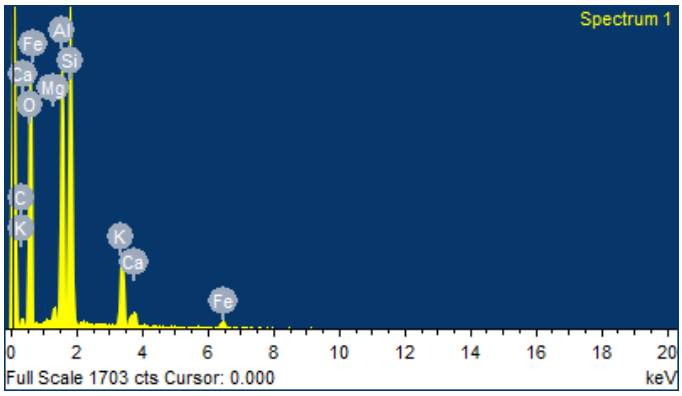


Figure E1 (b) EDS Spectrum of 28 Day Hardened BA30 Sample

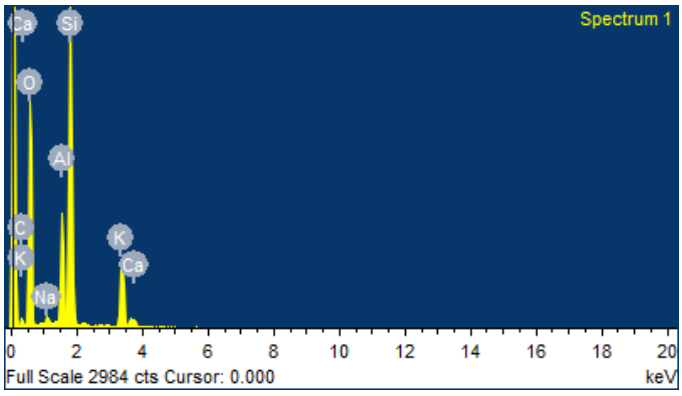


Figure E1 (c) EDS Spectrum of 28 Day Hardened BA25 Sample

Figure E1 EDS Spectrum of 28 Day Hardened Sample