A STUDY OF SELF COMPACTING CEMENTITIOUS SYSTEMS USING BAGASSE ASH AND BENTONITE

A STUDY OF SELF COMPACTING CEMENTITIOUS SYSTEM USING BAGASSE ASH AND BENTONITE

By

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ABSTRACT

Self Compacting Cementitious Systems (SCCS) fall in the domain of modern concrete technology which has been used successfully through out the world. The major applications of such systems involve massive placements with heavily reinforced sections such as tunnel linings, bridges piers, transportation structures, high rise buildings, rafts, prestressed concrete members and repairs of structures. Such systems offer uniform degree of compaction as well as have uniform durability and contain higher powder content with lower w/p ratio compared with the conventional concrete. These environmental friendly systems have better overall response in terms of volume stability, better packing, placement ease, economy, lesser heat of hydration, strength development and higher durability. In high performance concrete (HPC) systems, all cement particles do not get hydrated, therefore secondary raw materials (SRM) are used to replace a part of cement in order to economize, to make an environmental friendly concrete with improved microstructure and to get better overall response in terms fresh and hardened properties of concrete.

Extensive successful research work has been carried out through out the world on use of secondary raw materials in self compacting cementitious systems and now this practice is quite popular. However, in Pakistan, despite the availability of various pozzolanic materials in abundance, both in natural as well as artificial form, the coordinated and well planned research work on these resources is still in the elementary stage due to non availability of response data. Accordingly, their usage in the cementations systems is very limited. This study was made to evaluate the feasibility of using Bentonite (BN), a naturally occurring pozzolan and Bagasse Ash (BA), an artificial pozzolanic material, in self compacting paste systems with ultimate aim to produce self compacting concrete with desired properties. The parameters studied include secondary raw material’s particle characterization, flow behavior, strength development, calorimetric investigations, volume stability, X – ray diffraction (XRD) study and microstructural analysis by scanning electron microscopy (SEM) and mercury intrusion porosimetry (MIP) of self compacting paste systems.

The results showed that the internal porosity of secondary raw material particles adversely affects the water demand and super plasticizer demand of self compacting paste system. While the shape, size and surface texture of secondary raw materials along with their chemical and physical properties all have vital effects on the flow behavior, strength and the durability. The rate of strength gain of self compacting cementitious system is also an important aspect besides the strength at a specified age as it varies with secondary raw material being used. Furthermore, the addition of secondary raw materials in replacement mode causes an overall reduction in the total amount of heat released and has a better effect on the volume stability of the mix. After the detailed analysis of all the results, it can also be stated that both secondary raw materials investigated, can be successfully used in self compacting cementitious systems as each contributes positively towards enhancement of certain properties of concrete and the use of their blends can further optimize the response of self compacting concrete.

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TABLE OF CONTENTS

Chapter 1 Introduction

Chapter 2 Literature Review

Chapter 3 Experimental Program

Chapter 4 Results

Chapter 5 Discussion

LIST OF NOTATIONS

AASHTO	American Association of State Highway and Transportation Officials
AFm	Aluminate Ferrite monosulfate or Alumina, Ferric oxide, mono-sulfate or $Al_2O_3 - Fe_2O_3$ – mono
Aft	Aluminate Ferrite Trisulfate or Alumina, Ferric oxide, tri-sulfate or $Al_2O_3 - Fe_2O_3$ – tri
ASTM	American Society for Testing and Materials
BA	Bagasse Ash
BN	Bentonite
C_2S	Di – Calcium Silicate
C_3A	Tri – Calcium Aluminate
C_3S	Tri – Calcium Silicate
C_4AF	Tetra – Calcium Alumino Ferrite
CH	Calcium Hydroxide
CSH	Calcium Silicate Hydrate
EDAX	Energy Dispersive X – ray Analysis
FHWA	Federal Highway Administration
HPC	High Performance Concrete
ITZ	Interfacial Transition Zone
MIP	Mercury Intrusion Porosimetry
OPC	Ordinary Portland Cement
PCE	Polycarboxylate ether
SCC	Self Compacting Concrete
SCCS	Self Compacting Cementitious Systems
SCM	Self Compacting Mortar
SCP	Self Compacting Paste
SP	Super Plasticizer
SRM	Secondary Raw Material

VMA	Viscosity Modifying Agent
w/c ratio	Water Cement Ratio
w/p ratio	Water Powder Ratio
XRD	X – Ray Diffraction

LIST OF TABLES

LIST OF FIGURES

CHAPTER 1 – INTRODUCTION

1.1 General

Modern concreting technology includes self compacting cementitious systems (SCCS) used at places involving massive placements with congested and heavily reinforced sections. These places include tunnel linings, bridge piers, heavily reinforced columns, rafts, transportation structures and pre – stressed concrete members. Due to its large and variety of applications, it is necessary that self compacting concrete should flow into the formwork and around reinforcements under its own weight, filling the formwork completely and getting self compacted without segregation or bleeding. This wonderful technology is rapidly replacing the conventional concreting techniques world wide due to the gradual reduction in the number of skilled workers, differential compaction of conventional concretes resulting into differential durability, ease of placement and better performance. In addition, with the ever increasing demand and consumption of cement, nowadays scientists and researchers through out the world are in a quest for improving the existing cement products, developing new binders, reusing the various industrial wastes and explore naturally available materials that are environmental friendly and contributing towards sustainable development. Thus the self compacting cementitious systems containing high powder content (cement as well as secondary raw materials (SRM)), low water cement ratio, suitable chemical admixtures and smaller size and content of coarse aggregate are rapidly finding their way in today's construction industry. These modified ingredients of the concrete mix affect the microstructure of the matrix and optimize the transition zone by decreasing the porosity and refining the pores; yet having a desirable level of flow. This finally results in durability and strength of concrete.

Self Compacting Concrete (SCC), a kind of high performance concrete (HPC) with excellent deformation and segregation resistance, was first developed in Japan in late 1980's [1, 2]. Due to its versatile nature and wide range of applications, the use of self compacting concrete spread through out the world very rapidly; however, in Pakistan still its application and use is very limited due to non availability of chemical and mineral admixtures at cheap rates and un – clear understanding of basic concepts underlying the manufacture of self compacting concrete. This study is being made to evaluate the use of local secondary raw materials in self compacting cementitious systems and is focused on the self compacting paste (SCP) systems. The paste phase of self compacting cementitious system plays the most important role in service life performance of the structure and acts as the vehicle for the transport of aggregate phase during flow. This phase mainly consists of cement, secondary raw materials and admixtures and the study on paste phase is the first essential step towards successful production of self compacting concrete using local secondary raw materials.

1.2 Self Compacting Cementitious Systems (SCCS)

The European Guidelines for Self Compacting Concrete [3] defines SCC as "the concrete that is able to flow and consolidate under its own weight, completely fill the formwork even in the presence of dense reinforcement, whilst maintaining homogeneity and without the need for additional compaction." Various other definitions are also found in literature. Khayat et al [4] states that "Self compacting concrete is a special type of concrete that should flow into place and around obstructions under its own weight without segregation and flow blockage and with no significant separation of material constituents thereafter until the setting". Ulucan [5] defines "Self compacting concrete as a concrete that has excellent deformability and high resistance to segregation and can be used to fill a heavily reinforced area without applying vibrations".

Three basic types of self compacting concrete systems are known which include powder type, viscosity modifying agent type and combination type. These types basically differ from each other mainly in the way the segregation resistance is achieved. In powder type self compacting cementitious system, a lower w/p ratio or higher powder content (cement as well as secondary raw materials) guarantees adequate segregation resistance while the same goal is achieved by viscosity enhancement agent in the viscosity agent type self compacting cementitious system. The combination type SCC allows production of a robust self compacting cementitious system and is believed to have excellent segregation resistance [6]. However, these systems must meet the other special performance requirement i.e.

high deformation in addition to high segregation resistance. This implies that the concrete produced should have low yield stress as well as adequate viscosity. Both these are contrasting requirements to be met. High deformation is obtained by using an efficient super plasticizer, whereas segregation resistance is achieved by using any of the earlier mentioned approaches. In the present study, the self compacting paste systems used, contained higher powder content along with PCE based powder type super plasticizer. The powders used include cement (OPC – ASTM type 1) and secondary raw materials including Bentonite or Bagasse Ash.

1.3 Secondary Raw Materials (SRM)

Secondary raw materials are pozzolanic or inert powders which usually replace a part of cement and are used to modify / improve the properties of concrete which may include the control of early heat evolution, reduction in shrinkage, better packing, optimization of flow, enhancement of strength, improvement of microstructure, economy and the durability of cement based systems. In the literature secondary raw materials have been mentioned in a variety of ways like supplementary cementitious materials, waste materials and fillers depending upon the properties they exhibit.

Secondary raw materials are basically used to replace a part of cement in concrete mix as during hydration process, all cement particles do not get hydrated and remain as fillers in voids through out the life of structure in SCC / HPC. Hence, the secondary raw materials reduces the cement content and further reacts with calcium hydroxide, released during cement hydration, to forms compounds possessing cementitious properties. This, in turns, refines the micro – structure of the matrix, reduces its porosity, gives better packing of the binder phase and improves strength of cement based composite [7]. Refinement of micro – structure and reduction in porosity help to control the negative impact of various deleterious mechanisms as the harmful agents cannot get penetrated into the interior of concrete. Thus, replacement of a part of cement by secondary raw materials improves the service life performance of the structure as well as its durability characteristics. The use of secondary raw materials also indirectly reduces the negative environmental effect of cement production as use of lesser cement due to secondary raw material replacement will result in lesser CO_2 emission by cement industry.

1.3.1 Pozzolan

As per ASTM C 125 [8], Pozzolan may be defined as “A siliceous or siliceous and aluminous material, which in itself possesses little or no cementitious value but will, in finely divided form and in the presence of moisture, chemically react with calcium hydroxide (CH) produced by cement hydration at ordinary temperatures to form compounds possessing cementitious properties”.

1.3.2 Mechanism of Pozzolanic Reaction

Ordinary Portland cement when mixed with water forms calcium silicate hydrate (CSH) gel along with calcium hydroxide and ettringite. However, when a pozzolan is present, its silica component reacts with liberated calcium hydroxide in hydrated cement paste in the presence of water to forms another form of calcium silicate hydrate. The reaction is generally slow, resulting in a slow rate of heat liberation and strength development. As the reaction consumes calcium hydroxide, thus reducing the weaker crystals, it has an important bearing on the durability of concretes especially in an acidic environment. Moreover, pore size distribution studies of such systems have shown that the reaction products are very efficient in filling up the large capillary pore, thus improving the strength and impermeability of the system [9]. In addition to reactive silica, pozzolans also contributes reactive alumina, which in presence of calcium hydroxide present in the system, forms cementitious products such as tetra calcium aluminate hydrate (of the form C_4AH_x, in which x varies from 9 to 13) , tricalcium aluminate hydrate (C_3AH_6), dicalcium aluminate silicate hydrate (C_2ASH_8), calcium carboaluminate ($C_3A.CaCO_3.H_{12}O$), high sulfoaluminate ettringite ($C_3A.3CaSO_3.32H_{12}O$) and low sulfoaluminate ($C_3A.CaSO_3.12H_{12}O$) [10, 11, 12].

1.3.3 Bentonite

Bentonite is naturally occurring absorbent pozzolanic clay and is commonly divided into sodium bentonite (high swelling), calcium bentonite (low swelling), aluminum bentonite and potassium bentonite. Typically high swelling bentonites have a frothy texture caused by its alternate swelling and drying, whereas low swelling types have a cracked appearance. Therefore the properties of pastes, mortars and concrete vary accordingly, depending upon the type of bentonite used [9, 13]. The bentonite used in the present study is Potassium or K – Bentonite. The potassium bentonite is potassium rich illitic clay formed from alternation of volcanic ash [14], where illite is a nonexpanding mineral and is either a phyllosilicate (silicate mineral) or layered alumino silicate [15]. The X – ray diffraction analysis showed that bentonite, under study, contains a dominant portion of illite (39%). The bentonite was obtained from Jahangira deposits, in NWFP, Pakistan. These deposits are spread over an area of 26 square kilometers with an average depth of 3.5 meters.

1.3.4 Bagasse ash

Bagasse Ash occurs in abundance in sugar cane producing countries like Pakistan, India and Brazil and is obtained from the sugar mills as industrial waste where sugar cane bagasse is burnt in boilers for steam generation. Bagasse Ash consists mainly of silica (SiO_2) and small portions of oxides of Calcium, Aluminum, Magnesium and Iron etc. Bagasse Ash used in the study was obtained from Premier Sugar Mill, Mardan, Pakistan, where the sugar cane bagasse is burnt for stream generation at temperatures varying between 800°C to 1200°C for duration of 4 – 6 hours, depending upon the moisture content in bagasse, in an oxidizing environment. This ash so obtained mainly contained silica in addition to few other oxides and the XRD analysis has shown that Bagasse Ash had large amorphous portion.

1.4 Investigational Techniques

A wide range of properties of self compacting paste systems with each secondary raw material was analysed using various experimental techniques. The details of these techniques are as under:-

1.4.1 Calorimetry

The heat of hydration of concrete influence the workability, setting behavior, strength gain rates and pore structure development, thus affecting early age behavior as well as long term performance of concrete. Calorimetry is the technique used to measure the heat evolution of cement paste with time. Better monitoring of concrete heat flow leads to an improved understanding of characteristics of concrete materials and mix proportions. With calorimetry, forecasting of setting time and required curing regime, prediction of strength gain, evaluation of thermal cracking risk and identification of materials incompatibility etc can be done.

1.4.2 Scanning Electron Microscopy (SEM)

SEM is used for imaging the microstructure of concrete at very large magnifications. It is an excellent multipurpose instrument to study the presence of various phases and their interconnectivity, micro structure, interfacial transition zone (ITZ) and hydration products. It also provides a quantitative chemical analysis in terms of Energy Dispersive X – ray Spectroscopy (EDAX). Application of scanning electron microscopy enhances our ability to characterize cement and concrete microstructure, evaluate the influence of secondary raw materials, estimate the concrete durability and indirectly predict the service life [7, 16]. With scanning electron microscopy, morphology and shape of micro particles can be analyzed as each material / compound has a specific individual appearance e.g., calcium hydroxide crystals are plates / blocks of hexagons. Ettringite has elongated rod like crystals appearance. Calcium silicate hydrate crystals have needles like appearance.

1.4.3 Mercury Intrusion Porosimetry (MIP)

MIP is used to get an idea about the porosity of powders or of cementitious material samples. In a standard procedure, a small specimen is first dried to empty the pores of any existing fluid. It is then weighed, transferred to chamber and the chamber is evacuated. Sample is subsequently surrounded by mercury and pressure on mercury is gradually increased. As the pressure increases, mercury is forced into the pores on surface of the sample. If the pore system is continuous and extends to surface, mercury penetrates the smallest pore necks with increase in pressure (penetrates the bulk sample volume). If the pore system is not continuous, mercury may penetrate the sample volume only by breaking through pore walls. Through tracking pressures and intruded volume during the experiment, it is possible to get a measure of the connecting pore neck diameter of a continuous system or a break through pressure in a discontinuous system. The pore width corresponding to the highest rate of mercury intrusion per change in pressure is known as “threshold”, “critical” or “percolation” pore width [17]. After achieving this pressure, mercury has been shown to penetrate interior of the sample. Using the technique, one also obtains the measure of total porosity in the sample as that corresponding to volume of mercury intruded at the maximum experimental pressure divided by the bulk volume of the un - intruded sample.

1.4.4 Volume Stability of Self Compacting Cementitious Systems

Volume stability of cement based system is the change in volume of final hydrated products compared with initial volume of constituent materials of the mix. Mostly the change in volume is shrinkage of the cementitious system, however in few rare cases it may be the expansion especially during the early hours of concrete placement if measured by linear technique. In general, shrinkage is caused by consumption / loss of water which includes the water consumed during hydration process and pozzolanic reaction, water suction by secondary raw materials and the external loss to the environment i.e. water evaporation. The expansion occurs mainly due to the growth of expansive species like calcium hydroxide and ettringite and may also be due to re – absorption of bleed water in the system.

Shrinkage is basically the sum of various parallel and over lapping simultaneously operating mechanisms which includes plastic shrinkage, chemical shrinkage, autogenous shrinkage and drying shrinkage. Out of all these, the rate of autogenous shrinkage is highest up till an age of 1 day and is quite significant for self compacting cementitious system using low w/c ratio. The measurement technique of shrinkage can be divided into two broad categories which are volumetric method and linear method. A careful analysis shows that both gives almost same results and choice of technique and interpretation of results require competence. The volumetric method gives shrinkage measurement as per its true definition of volume change whereas, its disadvantage includes sucking in of any entrapped air, thus falsely showing an increased shrinkage as well as bleeding resulting in loss of contact of sample with plastic condom membrane. With linear method, exact linear change in volume per unit length can be calculated; however significant shrinkage is registered after the onset of hardening only [7]. The technique used in the present study measured total shrinkage during first 24 hours.

1.4.5 X – Ray Diffraction (XRD)

X – Ray Diffraction is a unique and powerful scientific tool basically applied for detection and quantification of crystalline materials. It also yields fundamental data when applied to amorphous solids and liquids. The phenomenon of x – ray diffraction includes a scattering process in which x – rays are scattered by electrons of the material without any change in wavelength. The resulting diffraction pattern is a fundamental physical property of each individual substance which not only gives speedy identification but also the complete elucidation of micro structure. The x – ray diffraction analysis is fundamentally based upon Bragg's law, given below [7]:-

$N \lambda = 2 d \sin\theta$, where

N = integer denoting the order of reflection

λ = wavelength of X – ray

d = inter planar spacing between successive atomic planes in the crystals

θ = angle between atomic plane and both the incident and reflected beams

For cement based systems both qualitative and quantitative XRD analyses are performed. While quantitative analysis is more precise, it is certainly time consuming, more expansive and demands expertise. Therefore, qualitative analysis is preferred. XRD analysis is also used to follow the process of hydration by determining the amount of un – reacted clinker minerals and calcium sulfate and / or by determining the amount of formed hydrates as function of time. For determining the amount of un – reacted cement constituents, quantitative XRD appears to be more suitable and widely used method [18].

1.5 Design Office Practice in Pakistan

Design offices in our country carry out the structural analysis and design of infrastructural facilities using state of art softwares; however structural engineers are concerned with compressive strength and slump of concrete only, while giving no attention to other important material performance aspects such as shrinkage, creep and microstructural development etc. In addition, it is concrete that carries and transmits the loads to steel and is responsible for performance at service loads. Yet it is grossly ignored while laying emphasis on steel and its detailing. This approach results in problems related to durability and performance characteristics of the structure as a whole. Furthermore, in our country very limited varieties of cements are available and mostly "Ordinary Portland Cement" is used for all environments and areas. On the other hand, in today's developed world, a wide variety of cement types are available and accordingly these are utilized to suit the environment as well as the placement. So with these limitations, it is quite necessary that a careful application of use of locally available ingredients of concrete should be made. Moreover, volume stability, placement problems, micro structure, heat of hydration and pumping should also be considered and professional engineers be educated so that they can thereafter disseminate the information to field staff.

1.6 Research Objectives

At national level, we have a vibrant construction industry, but now the time has come to incorporate modern ideas into our conventional construction materials and practices to make structures economical, durable and environment friendly. Use of self compacting cementitious systems with locally available secondary raw materials is a step in right direction which will also render a cost effective solution. The primary objective of this research was to assess the possibility of using Bagasse Ash and Bentonite as pozzolans in self compacting cementitious systems. The specific objectives were:-

- To study the behavior of fresh concrete including its flow characteristics, segregation resistance and deformability.
- To study hydration kinetics of the system using calorimetry and the volume stability by linear shrinkage measurements.
- To study the strength characteristics and water absorption of the system.
- To study the micro structure of secondary raw materials particles as well as the hydration products of self compacting paste systems containing these secondary raw materials using scanning electron microscopy, mercury

intrusion porosimetry and X – ray Diffraction analysis.

Application of the study is threefold; first is introduction of use of self compacting cementitious system in the country while utilizing locally available secondary raw materials and to provide information to engineers about the field of modern concrete engineering, secondly, energy saving in cement industry required for the production of cement and to address the related environmental aspects in carbon dioxide emission reduction, thirdly concrete produced through partial replacement of cement will be a cost effective solution as over all self compacting cementitious system in an expensive technology, but in certain structures, it may be the only possible solution, therefore cost dimension is ignored.

CHAPTER 2 – LITERATURE REVIEW

2.1 General

Although, some work has been reported on the use bagasse ash as secondary raw material [19 – 24], but little research has been documented for the use of bentonite in concrete. Moreover, most of the research is being carried out worldwide on self compacting cementitious systems using other pozzolans like rice husk ash, fly ash, metakioline, lime stone powder etc. But little research has been done on the self compacting cementitious system using the already mentioned secondary raw materials available in Pakistan. Furthermore, the research on modern concrete systems is at the initial stage in Pakistan and needs to be coordinated, focused and updated.

2.2 Self Compacting Cementitious System

Self compacting cementitious system (SCCS) may be the single component system such as self compacting paste (SCP) system or two components system such as self compacting mortars (SCM) system or three components system such as self compacting concrete (SCC) system. However, the sequential development of these systems lead to the final goal i.e. successful production of self compacting concrete, for which the paste has to be optimized first, followed by mortar while understanding various parameters step by step.

Self compacting concrete was develop in Japan and Okamura was the first to propose the necessity of development of SCC after considering problems in concreting such as the lack of uniformity of compaction resulting in differential / inadequate compaction of concrete and hence in differential durability. Studies to develop self compacting concrete were carried out by the researchers at university of Tokyo, Japan, in 1980's and by early 1990's Japan had developed as well as used the concrete that did not require any vibration to achieve full compaction and flowed under its own weight [25, 26].

Self compacting cementitious systems offers many advantages for cast in place construction as well as for pre cast pre stressed concrete industry. These includes rapid rate of concrete placement with faster construction times, easy of flow of concrete around congested reinforcement, elimination of differential compaction to achieve high level of homogeneity with uniform concrete strength and durability of structure, providing potentials for a superior level of finish, easy demoulding and faster use of elements and structures, reduced exposure of workers to noise and vibration at batch plant as well as construction site and finally the involvement of lesser labour [3, 26]. In addition, its uses also include SLU's, SIFCON, light weight reinforced concrete, pre – placed aggregate concrete and for rapid repairs and constructions.

Numerous studies have been made on self compacting cementitious systems as the technology is constantly being analyzed and developed. Few significant, fundamental and imperative studies can be summarized as follows:-

Rizwan et al [6] reports that "as paste is the vehicle of aggregate phase, good workability can be achieved by reducing the aggregate content with an increased paste volume thus resulting in reduced internal friction." Further it is suggested that the sand content may be increased to augment the cohesiveness and stability of the concrete mix. This may also result in higher requirement of super plasticizer content to meet the selected flow target and a slightly lower modulus of composite. Regarding the aggregate requirement, the study specifies that no more than 15% of aggregate should be elongated as these can cause internal friction, bigger voids and bleeding and require a higher paste volume. Then for the mixing water, the study describes the effect of water temperature while mixing, on the flow response of self compacting cementitious systems and suggests that any addition of even small quantity of water after the chemical admixtures have been added could significantly reduce the mix cohesion and / or could yield slightly inaccurate results which appears contradictory to the literature [3].

Brouwers [27] reports that a sand of about 50% of the total aggregate mass has been found satisfactory for self compacting concrete especially from stability point of view. Khayat et al [28] reports that self compacting concrete can exhibit a greater degree of thixotropy arising from the incorporation of a set accelerating admixture and / or higher binder content which promote a faster development of cohesion resulting from cement hydration. In addition, an increase in w/c ratio or paste volume, resulting in lower coarse aggregate volume, can reduce the degree of internal friction, thus increasing the ability of concrete to flow through restricted bars, but increased water content can reduce the cohesiveness and viscosity of mix.

Rizwan at el [29] reported that shape, size, surface morphology and porosity of secondary raw materials play a very significant role in determining the water and super plasticizer demands of the system, but besides the physical properties, the chemical composition of the secondary raw materials is also very important e.g. rice husk ash with high porosity, high specific surface area and high water vapour adsorption, does show a significantly low plasticizer demand when 20% of it is added to fly ash in self compacting mortar system compared with similar mass addition of silica fume in fly ash based self compacting mortar formulation. Thus suitable blends of secondary raw materials can provide an improved overall response of self compacting mortar systems using well graded aggregates. The study specifies that T25 cms time is thought to be a function of viscosity of a system, whereas the total spread of mini slump cone is a function of the yield stress of such systems and for high performance self compacting cementitious system, the first 24 hours shrinkage is very important.

Hence, self compacting concrete is characterized with low w/p ratio, use of super plasticizer, higher powder content to produce adequate paste to act as lubricant for the aggregate phase, use of viscosity modifying agents and use of smaller size as well as lesser content of aggregate possibly with continuous grading [2]. In addition, self compacting concrete also shows good placement and esthetic performance, but is different from of high performance concrete (HPC).

2.3 High Performance Concrete

Various definitions of high performance concrete are available in literature but these define HPC in a qualitative manner. In 1996, Federal Highway Administration (FHWA), USA published the definition which quantified a variety of characteristics and provided criteria for high performance concrete. Russell et al [30] further worked on the definition and proposed certain amendments. The Federal Highway Administration, USA considered eight performance characteristics while defining high performance concrete. These encompassed both durability and structural design. The four performance characteristics related to durability are freeze and thawing durability, scaling resistance, abrasion resistance and chloride ion penetration. The four structural design characteristics are compressive strength, modulus of elasticity, shrinkage and creep. Each performance characteristics was categorized and graded basing on a standard test method published by American Society for Testing and Materials (ASTM) or by American Association of State Highway and Transportation Officials (AASHTO). The details of grading are given in Table 2.1 whereas Table 2.2 gives the details of the test methods to be used.

Table 2.1 – Grades of Performance Characteristics for High Performance Structural Concrete

Performance Characteristics	**Standard Test Method**	**FHWA HPC Performance Grade**			
		1	**2**	**3**	**4**
Freeze and Thaw Durability (x = relative dynamic modulus of elasticity after 300 cycles)	AASHTO T161 ASTM C 666 Procedure A	60%≤x≤ 80%	80%≤x	-	-
Scaling Resistance (x = visual rating of surface after 50 cycles)	ASTM C 672	X=4,5	x=2,3	x=0,1	-
Abrasion Resistance (x = average depth of wear)	ASTM C 944	2.0>x>1.0 mm	1.0>x>0.5 mm	0.5 mm >x	-
Chloride Penetration (x = coulombs)	AASHTO T 277 ASTM C 1202	3000≥x> 2000	2000≥x> 800	800≥x	-
Strength (x = compressive strength)	AASHTO T 22 ASTM C 39	41≤x≤55 MPa (6≤x≤8 ksi)	55≤x≤69 MPa (8≤x<10 ksi)	69≤x≤97 MPa (10≤x<14 ksi)	x≥97 MPa (x≥14 ksi)
Elasticity (x = Modulus of Elasticity)	ASTM C 469	28≤x≤40 GPa (4≤x<6x10^{6} psi)	40≤x≤50 GPa (6≤x<7.5x10^{6} psi)	x≥50 GPa (x≥7.5x10^{6} psi)	-
Shrinkage (x = microstrain)	ASTM C 157	800>x≥600	600>x≥400	400>x	-
Creep (x = microstrain / pressure unit)	ASTM C512	75≥x> 60/MPa (0.52≥x> 0.41/psi)	60≥x> 45/MPa (0.41≥x> 0.31/psi)	45≥x> 30/MPa (0.31≥x> 0.21/psi)	30/MPa≥ x (0.21/psi ≥x

Table 2.2 – Details of Test Methods for Determining High Performance Concrete Performance Grades

Performance Characteristics	**Standard Test Method**	**Notes**
Freeze and Thaw Durability	AASHTO T161 ASTM C 666 Procedure A	1. Test specimen 76.2x76.2x279.4 mm (3x3x11 in) as cast or cut from 152.4x304.8mm (6x12 in) cylinder. 2. Acoustically measure dynamic modulus until 300 cycles.
Scaling Resistance	ASTM C 672	1. Test specimen to have a surface area of 46,451 mm^2 (72 in^2). 2. Perform visual inspection after 50 cycles.
Abrasion Resistance	ASTM C 944	1. Concrete shall be tested at three different locations. 2. At each location, 98 N, for three 2 minute abrasion periods shall be applied for a total of 6 minutes of abrasion time per location. 3. Depth of abrasion shall be determined per ASTM C 779, Procedure B.
Chloride Ion Penetration	AASHTO T277 ASTM C 1202	1. Test per standard test method.
Strength	AASHTO T 22 ASTM C 39	1. Molds shall be rigid metal or one time use rigid plastic. 2. Cylinders shall be 100 mm in diameter by 200 mm long (3.9x7.8 in) or 150 mm in diameter by 300 mm long (5.9x11.2). 3. Ends shall be capped with high strength capping compound, ground parallel, or placed onto neoprene pads per AASTO “Specifications for Concretes”. 4. Use of neoprene pads on early age testing of concrete exceeding 70 MPa at 56 days should use neoprene pads on the 56 day tests. 5. The 56 day strength is recommended.
Elasticity	ASTM C 469	1. Test per standard test method.
Shrinkage	ASTM C 157	1. Use 76.2x76.2x285 mm (3x3x11.25 in) specimen. 2. Shrinkage measurements are to start 28 days after moist curing and be taken for a drying period of 180 days.
Creep	ASTM C512	1. Use 152x305mm (6x12 in) specimen. 2. Cure specimens at 73°F and 50% relative humidity after 7 days until loading at 28 days. 3. Creep measurements to be taken for a creep loading period of 180 days.

Russell et al [30] studied the existing discrepancies / inconsistencies in the FHWA definition of high performance concrete and recommended that each characteristic rather then having different numbers for different characteristic. Three new characteristics were proposed to be incorporated in the definition of high performance concrete which include Alkali silica reactivity, sulfate resistance and workability. The details are as given in Table 2.3

Table 2.3 – Proposed Performance Characteristic Grades for High Performance Structural Concrete

Performance Characteristics	Standard Test Method	FHWA HPC Performance Grade		
		1	2	3
Freeze and Thaw Durability (F/T = relative dynamic modulus of elasticity after 300 cycles)	AASHTO T161 ASTM C 666 Procedure A	$70\% \leq F/T \leq 80\%$	$80\% \leq F/T \leq 90\%$	$90\% \leq F/T$
Scaling Resistance (SR = visual rating of surface after 50 cycles)	ASTM C 672	SR=4,5	SR=2,3	SR=0,1
Abrasion Resistance (AR = average depth of wear)	ASTM C 944	$2.0 > AR > 1.0$ mm	$1.0 > AR > 0.5$ mm	0.5 mm $> AR$
Chloride Penetration (CP = coulombs)	AASHTO T 277 ASTM C 1202	$2500 \geq CP > 1500$	$1500 \geq CP > 500$	$500 \geq CP$
Alkali-silica reactivity (ASR = expansion at 56 days, %)	ASTM C 441	$0.20 \geq ASR \geq 0.15$	$0.15 \geq ASR \geq 0.10$	$0.10 \geq ASR$
Sulfate Resistance (SR = expansion, %)	ASTM C 1012	$SR \leq 0.10$ at 6 months	$SR \leq 0.10$ at 12 months	$SR \leq 0.10$ at 18 months
Workability (SL = slump, SF = slump flow	AASHTO T 119 ASTM C 143 ASTM C 1611	$SL > 190$ mm (7-1/2 in) $SF < 500$ mm (20 in)	$500 \leq SF < 600$ mm ($20 \leq SF < 24$ in)	500 mm$<SF$ (24 in$<SF$)
Strength (f'_c = compressive strength)	AASHTO T 22 ASTM C 39	$55 \leq f'_c \leq 69$ MPa ($8 \leq f'_c \leq 10$ ksi)	$69 \leq f'_c \leq 97$ MPa ($10 \leq f'_c \leq 14$ ksi)	97 MPa $\leq f'_c$ (14ksi$\leq f'_c$)
Elasticity (E_c = Modulus of Elasticity)	ASTM C 469	$34 \leq E_c \leq 41$ GPa ($5 \leq E_c < 6x10^6$ psi)	$41 \leq E_c \leq 48$ GPa ($6 \leq E_c < 7x10^6$ psi)	48GPa$\leq E_c$ ($7x\ 10^6$ psi $\leq E_c$)
Shrinkage (S = microstrain)	ASTM C 157	$800 > S \geq 600$	$600 > S \geq 400$	$400 > S$
Creep (C = microstrain / pressure unit)	ASTM C512	$75 \geq C > 55$/MPa ($0.52 \geq C > 0.38$/psi)	$55 \geq C > 30$/MPa ($0.38 \geq C > 0.21$/psi)	30/MPa $\geq C$ (0.21/psi$\geq C$)

2.4 Secondary Raw Materials

According to Cordeiro et al [21], capability of pozzolanic materials to enhance the strength of concrete includes the physical as well as chemical effects. The physical effects are primarily associated with their influence on the packing characteristics of the mixture, which depend on size, shape and texture of the particles. The chemical effects are associated with their capability of providing siliceous and / or aluminous cementitious compounds that are produced after chemical reaction with calcium hydroxide in presence of water. Few other studies [31, 32] suggest that during initials days (up to 28 days), the physical effects of fly ash and silica fume contributes towards the strength development while beyond that chemical effects (pozzolanic reaction) becomes significant. Tangpagsit et al [31] reports that the smaller particle size of pozzolanic materials tends to produce higher compressive strength than the coarse one.

Rizwan et al [33] reported that when mineral admixtures are added, three effects can be quantified including dilution, heterogeneous nucleation (physical) and pozzolanic reaction (chemical) depending on the amount and solubility of amorphous silica, where heterogeneous nucleation is a physical process leading to a chemical activation of hydration of cement such that mineral admixture particles act as nucleation centers for the hydrates thus enhancing cement hydration. Further, Rizwan et al reported some where else [34] that in addition to cement grains, secondary raw material particles also adsorb super plasticizer and water. The internal powder pores of bottle neck type usually result in higher super plasticizer demand and finally Cyr et al [35] reported that a smaller amount of powder has an optimum efficiency and results in a large increase in compressive strength, whereas the use of a large amount of powder has smaller effects. This was the reason of using secondary raw materials at the rate of 10 % of cement mass in self compacting systems in the present study.

2.4.1 Bentonite

Katsioti [36] reported that bentonite presents strong colloidal properties and its volume increases several times when in contact with water, forming a gelatinous and viscous liquid. However, the special properties of bentonite such as hydration, swelling, water absorption, viscosity and thixotropy, render it as a valuable material for a wide range of possible usage and applications in concrete.

Neville [10] describes that natural pozzolans when heated can improve their reactive ability in cement paste and resultantly can improve certain properties of concrete. Parger et al [37] also stated that the heated clay minerals provide sulfate resistance greater than the high sulfate resistance cement. The same was also studied by Mirza et al [8] while heating bentonite from 150°C to 950°C and reports that there is a decrease in the concrete compressive strength initially with addition of bentonite, but then samples with bentonite gains strength remarkably at later ages. The main reason for this being the pozzolanic reaction that takes place at slower rate than the hydration of cement, coupled with the micro filler effect caused by bentonite. The compressive strength of concrete containing 20% bentonite was 0.68 times that of control mix (concrete without bentonite) at the age of 7 days, but it increased to 0.79 time of the control mix at the age of 56 days. Then the maximum strength activity index (according to ASTM C 618 [38]) was shown by the samples with heated bentonite up till 150°C, even higher than the control mix and there is an improvement in resistance against sulfate attack to a degree of around 30%. This may be due to lower calcium hydroxide concentration in the system because of its consumption in pozzolanic reaction which is highly prone to sulfate attack. The study also specifies that for same w/b ratio, concrete made with cement containing bentonite is less workable than the control mixture.

Grishin et al [39] reports that for bentonite based concrete the average value of strength gain from 28th day to 90th day, is 1.4 times the strength at 28 days and after 365 days the average strength gain was 2.31 time of the strength at 28 days.

2.4.2 Bagasse Ash

Cordeiro et al [21] reports that bagasse ash can be classified as a pozzolanic material, but its activity depends significantly on its particle size and fineness and the compressive strength of mortar containing bagasse is inversely proportional to bagasse ash's particle size. At another instant Cordeiro et al [19] reports that plastic viscosity increases marginally due to incorporation of bagasse ash in high performance concrete. However, the yield stress always decreased when bagasse ash was used, which suggests the positive effect of the ash on concrete rheology. Then the negligible carbon content (LOI of 0.42%) of bagasse ash contributes to better rheological behavior as the concrete with high carbon content mineral admixtures requires more amount of super plasticizer for same workability. However, no significant effect was noted on the compressive strength of high performance concrete with small addition of bagasse ash (10%), but a negative trend was observed with increase in bagasse ash content beyond 10% increase.

According to Ernesto et al [20] bagasse ash can become pozzolanically active when burnt in the temperature range of 800°C to 1000°C and the reaction product formed during pozzolanic reaction are not crystalline compounds base on the interpretation of XRD data.

2.5 Calorimetry

Calorimetry is the technique used to measure the heat evolution of the cement paste with time and determine the hydration kinetics. Calorimetric curves obtained during the specified duration of test are composed of various peaks. The first peak represents the very high amount of heat released due to initial fast hydration of aluminate phase, C_3A, in which the main product is AFt. The heat – flow then drops down to minimum within initial 2 – 3 hours. This phase is called induction period or dormant period which is followed by acceleration of hydration / pozzolanic reaction and mass precipitation of hydration products, primarily CSH gel. The system sets down during this process and the calorimetric curve rises to a second peak which occurs at around a time of 15 – 18 hours for secondary raw material added self compacting paste systems. It is followed by the slowing down of the reaction and may encounter smaller peaks of heat flow in the process [40].

Rizwan et al [33] reported that the presence of super plasticizer delays the heat peaks observed in the calorimetry of cement pastes and the amount of heat liberated depends upon the cement type, its content and w/c ratio because super plasticizer mainly acts as slight retarder.

2.6 Scanning Electron Microscopy (SEM)

Rizwan [7, 18] reports that scanning electron microscopy is a versatile tool for study of hydration products including calcium silicate hydrate, calcium hydroxide and ettringite. Calcium hydroxide has influence both on strength and durability of cementitious systems. The quantity of calcium hydroxide produced depends upon the water content, available space, cement clinker type and its content. Higher mixing water and clinker content give higher calcium hydroxide content. The addition of pozzolanic secondary raw material in the system reduces the calcium hydroxide content compared with plain cement formulation due to reduction in clinker content (dilution) as well as due to pozzolanic reaction of secondary raw materials which goes side by side with hydration reaction. Calcium hydroxide being a weaker crystal generally reduces the strength and durability of the concrete composite. It is important that its content should be reduced to improve the performance of cement based systems. Moreover, ettringite formation is faster (after about 10 – 20 minutes of mixing water, ettringite formation starts) and its content is more significant with blended Portland cement and calcium aluminate cement. The calcium hydroxide crystals can be detected after about 8 – 10 hours of water mixing which is approximately the Vicat final set of self compacting concrete and about 90% of total calcium hydroxide is in place after the end of first 24 hours.

2.7 Mercury Intrusion Porosimetry (MIP)

Rizwan [7] reported that that after three days there is only a very little improvement in the pore structure refinement hence for the study of pore structure refinement of Portland cement based formulations by mercury intrusion porosimetry the samples up till 7 days can give sufficient details. It was also reported that most of the porosity of cement-based systems relates to the paste phase and the aggregate phase possesses little porosity and offers internal resistance to shrinkage.

Cook [17] stated that increased curing time and decreased w/c ratio, results in lower total porosities and smaller values of threshold pore widths for all pastes; as it is widely accepted that as hydration proceeds, hydration products grow into the pore spaces of a hardened cement paste. In addition the rate of degree of hydration decreases with decrease in w/c ratio, however for all w/c ratios around 35% degree of hydration is achieved during initial 24 hours and thereafter the rate varies according to w/c ratio.

However diamond [41] suggests that the mercury intrusion porosimetry cannot provide a true pore size distribution as the pore system in concrete is not cylindrical (as assumed by Washburn equation model) as well as continuous. It also has the problem of accessibility of interior pores from the outer surface. Then there is contribution of air voids which magnifies the results and finally, the mercury must pass through the narrowest pores connecting the pore network, to get an accurate data. The threshold pore width, however, may provide a better indicator of material durability as it has an important influence on the permeability and diffusion characteristics of cement paste.

Laskar et al [42] recommends the minimum number of samples / tests to be conducted in order to estimate the values of mercury intrusion porosimetry parameters for an accuracy of 95% basing on certain statistical formulas supported by the experimental data. The study further recommends that the mortar adhered with aggregate should be tested by mercury intrusion porosimetry for measuring the accurate properties of concrete rather than testing mortar devoid of aggregate.

2.8 Volume Stability of Self Compacting Cementitious Systems

Rizwan et al [33] reported that the pozzolanic powders are sometimes incorporated in self compacting cementitious systems to control the early shrinkage and to improve the microstructure for enhanced durability. At another place Rizwan et al [43] reports that though time-zero concepts have been suggested in the literature, but it was found that zeroing the shrinkage measurement data for self compacting cementitious systems does not deviate significantly from non-zeroed data due to the fact that only first 24 hours shrinkage has been measured and a little volume change occurred in linear measurement method before Vicat set thus making time-zero concepts insignificant for the systems investigated.

Several investigators have used linear or volume methods for determining the early volume stability of the cementitious systems and they suggest that both methods have their own plus and minus points and it had been established that both the linear and volumetric methods give almost the same results of early shrinkage which is more significant for self compacting cementitious systems [44].

Cook [17] reported that the rate of degree of hydration decreases with decrease in w/c ratio, however for all w/c ratios, around 35% degree of hydration is achieved during initial 24 hours and the rate varies according to w/c ratio, hence initial 24 hour are very much important for shrinkage measurement of SCC as also reported by Rizwan [29]. It has also been documented in the literature [45] that certain calcium sulfo aluminate based admixtures are used for shrinkage compensation. These admixtures help in growth of ettringite crystals. These expansive ettringite crystals grow in voids which in turn reduce / compensate shrinkage of gel and drying shrinkage. Growth of ettringite in the void is shown in Fig 2.1.

Fig 2.1. Growth of Ettringite in Void – Shrinkage Compensation

CHAPTER 3 – EXPERIMENTAL PROGRAM

3.1 General

The secondary raw materials used were first milled in Los Angeles Abrasion Machine and then sieved through number 200 sieve. A systematic experimental approach was adopted by first determining the particle characterization by using Beckmann Coulter LS 230 Laser Granulometer. Scanning electron microscopy (SEM) by FEI XL 30 Environmental Scanning Electron Microscope with field emission gun (ESEM FEG) was done to obtain the shape and morphology of powder particles. The chemical composition of secondary raw materials and cements was obtained and the densities were determined. The secondary raw material particles were also characterized using X –Ray Diffraction. For all self compacting paste formulations, the secondary raw materials were used in replacement mode only. The water demand / super plasticizer demand of the self compacting paste formulations were calculated along with setting times using Vicat apparatus. The flow level was achieved by using PCE (Polycarboxylate ether) based super-plasticizer. Segregation resistance was achieved by using low w/p ratio. The flow and flow times were noted for each system using Hagerman's mini-slump cone and V funnel. Water absorption and strength testing was done. Besides calorimetric analysis, shrinkage measurements, microstructural study by scanning electron microscopy and mercury intrusion porosimetry and X –Ray Diffraction analysis of hydrated sampled at specified ages were also carried out.

3.2 Materials

CEM 1 42.5 R (Schwenk, Germany) and Faugi brand Pakistani Ordinary Portland Cement (OPC), both conforming to ASTM C150 [46], were used in the experimental program carried out at NUST, Islamabad and at TU Freiberg, Germany. Bagasse Ash was obtained from Premier Sugar Mill, Mardan, Pakistan. Bentonite was obtained from Jehangira deposits, in NWFP, Pakistan. The super plasticizer used was powder type third generation PCE based, Melflux 2651 F. The mixing water in all the reported formulations was as per the water demand (WD) of the systems which is well below 0.44 as mentioned for complete hydration [47].

3.3 Powder Particle Characterization

The results by Laser Granulometer, BET method and chemical analysis of CEM 1, Faugi Cement and both secondary raw materials are given in Table 3.1 and calculations of Bogue's Limits for CEM 1 are given at Table 1 of Annexure A.

Table 3.1 – Physical and Chemical Properties of Powders

Properties / Oxides	OPC		Secondary Raw Materials	
	Faugi Cement (Pakistan)	CEM 1 42.5 R (Germany)	Bagasse Ash	Bentonite
SiO_2 (%)	17.15	19.17	66.23	51.38
Al_2O_3 (%)	5.60	5.21	2.35	18.24
CaO (%)	64.09	61.12	2.59	3.52
Fe_2O_3 (%)	3.21	1.44	8.01	7.81
P_2O_5 (%)	0.16	-	2.27	0.17
K_2O (%)	1.19	2.63	5.47	4.63
MgO (%)	1.74	2.51	1.97	3.71
Na_2O (%)	1.86	1.23	0.51	1.83
TiO_2 (%)	0.32	-	0.12	0.68
SO_3	2.66	1.89	0.15	Nil
Loss on Ignition (%)	1.77	2.0	16.65	7.76
Particle size d50 (um)	24.8	18.15	22.36	4.326
Density (g/cm^3)	3.19	3.15	2.198	2.7931
Specific Surface, BET (m^2/g)	0.585	1.6	16.993	25.348
Moisture content (%)	-		2.89	3.12

3.4 Mixing Regime and Mix Proportions of SCP Formulations

The mixing was done using Hobart Mixer of 5 L capacity. The constituents including cement, secondary raw material and powder super plasticizer were first manually mixed in closed plastic container in dry state. These were then fed into the bowl of mixer containing requisite mixing water. A slow mixing (145 rpm) was carried out for 30 seconds and then interior of bowl was cleaned. Thereafter, again slow mixing was done for 30 seconds and finally, the formulation received 120 seconds of fast mixing (285 rpm). The total mixing time was thus 3 minutes (180 seconds).

The mix proportions of various self compacting formulations used are given in Table 1 of Annexure A.

3.5 Water Demand, Super Plasticizer Demand and Setting Times

As per the European Guidelines for Self Compacting Concrete [3], the calculation of a system's water demand (WD) is often the first step required in self compacting cementitious system design. The WD of the system is the sum of water demand of powders as well as the aggregate phase. The water demand of powder component can be determined by taking cement and other selected fillers in the desired pre – selected proportions by mass and testing with Vicat apparatus, whereas for aggregate phase water demand can be found by procedures outlined in ASTM C127 and 128 standards [48, 49]. For the production of a durable self compacting concrete mix, it is important that the water content should not significantly exceed the water demand of the system.

The water demand and setting times of various self compacting paste systems were determined by Vicat apparatus at 20±1^{o}C and 20±5% relative humidity. The super plasticizer amount required for a target flow of 30±1 cms was obtained for all formulations using Hagerman's mini slump cone. It had an upper diameter equal to 70 mm, a lower diameter equal to 100 mm with a height of 60 mm. All formulations had mixing water equal to water demand of the respective system during the entire experimental program.

3.6 Flow of Self Compacting Paste Systems

The flow of self compacting paste system is usually characterized by Hagerman's mini slump cone spread and its time and V Funnel time. Hence, after determining the water demand and amount of super plasticizer, the total spread and time required to reach a spread of 25 cms (T25) was noted [7] using Hagerman's mini slump cone (spread divided by slump cone's lower diameter, 25/10 = 2.5) on the analogy of measuring T50 cms time for SCC using slump cone of 20 cms lower diameter (spread divided by slump cone's lower diameter, 50/20 = 2.5). The flow target was 30±1 cms. V funnel time was also measured for all the formulations. The funnel consisted of a rectangular cross section with top dimension as 270 x 30 mm and bottom opening as 30 x 30 mm. The total height of funnel was 315 mm with a 75 mm long straight bottom section with a gate. For the test measurement, the V funnel is completely filled with self compacting paste, then the bottom gate is opened and the time for the paste to flow out of funnel is precisely measured. The test results are given in Chapter 4.

3.7 Estimation of Thixotropy

The total spread of self compacting paste system without any secondary raw material as well as those containing 10% secondary raw materials was measured using Hagerman's mini slump cone. After mixing the formulations, these were kept in the slump cone for certain time duration (0, 3, 5, 10, 15, 30 and 60 minutes). The slump cone was then lifted after the specified time to allow the formulation deform under it own weight and the total spread was measured. It was done to estimate the relative thixotropy imparted by secondary raw materials and to find the open time available for working with different formulations. The test results are given in Chapter 4.

3.8 Strength

Prisms for flexural and compressive strength measurement having 4 x 4 x 16cms dimensions were cast for different self compacting paste formulations. The samples were cured in sealed plastic bags for initial 24 hours at 90% relative humidity and 20±1°C. Then these were demoulded, weighed and put underwater till the age of testing. The samples were weighed in SSD condition at test ages to get the relative water absorption with reference to the internal water in specimen at the age of 24 hours. These were then tested for flexural and compression strength as per EN 196-1.

3.9 Calorimetry

72 hours conduction calorimetry was done on the cement pastes with super plasticizer and with as well as without secondary raw materials. As only a small sample mass, say 5 grams is required. The samples were taken from the dry mixes of formulations prepared for other tests e.g. strength / shrinkage tests etc. The calorimetry was performed in parallel to these tests. The calorimetric test requires a very careful handling of sample as it involves a very small quantity of dry mix and even smaller quantity of water which is to be hand mixed with the dry constituents. Then placing of small crucibles along with their lids in the calorimeter again asks for special care to prevent any unnecessary heat conduction to or from the calorimeter. However, the suitable software allowed the measurement of total cumulative heat generated over the time to a fair degree of accuracy.

3.10 Scanning Electron Microscopy (SEM)

Scanning electron microscopy was done using FEI XL 30 Environmental Scanning Electron Microscope. The specimens of powdered secondary raw materials as well as the self compacting paste samples were studied at specified ages after stopping hydration with Iso-Propanol and Acetone. The purpose was to study the microstructure, morphology and the hydration products at various ages. In addition to the SEM images, qualitative chemical composition and X – ray micro analysis by Energy Dispersive X – ray Spectroscopy (EDAX) was also obtained for each sample.

3.11 Mercury Intrusion Porosimetry (MIP)

The pore size distribution study was performed using Pascal 440 mercury intrusion porosimeter. The samples were prepared from the previously cast prisms for flexure and compression tests. These prisms after subjected to compression were broken to a size of 5mm by chisel and hammer. The hydration process was stopped by dipping the samples in a mixture of Iso-Propanol and Acetone. After drying the samples, these were subjected to a controlled incremental pressure till 400 MPa. All the measurement of pressure, volume of mercury intruded into sample and radii of pores etc were recorded by Pascal 440 Porosimeter. The contact angle between mercy and the pore wall was 140^o and mercury surface tension was 480 dyne / cm.

3.12 Early Shrinkage

For the measurement of shrinkage, a modified version of German shrinkage channel apparatus, measuring 4x 6x 25 cm interfaced with the computer was used for non-zeroed, linear early shrinkage measurements at 20 ± 1 ^oC with a relative humidity of 50 ± 5%. Fig 3.1 shows details of the apparatus.

Fig 3.1 German modified shrinkage channel apparatus

The samples were tested in both uncovered and covered conditions thus approximating the field conditions wherein evaporation is either possible or not possible respectively. The covering of the apparatus was done by using adhesive polythene sheets. The measurements started ten minutes after the water addition to the constituents of self-compacting cementitious systems and these measurements lasted for the first 24 hours only. The frontal end of the apparatus is fixed while the rear end is flexible and is therefore, capable of a recording of 0.31 microns displacement due to shrinkage or expansion of cement based systems. The computer software recorded the shrinkage readings automatically with one minute interval. At the end of 24 hours, the shrinkage strains were picked up and imported into Excel sheets. The details of the measuring device can be seen in literature [7].

3.13 X – ray Diffraction (XRD)

The specimen pieces of about 2 – 4 mm size were obtained from 4 x 4 x 16 cm prism samples broken in flexure. The hydration was stopped using Acetone and Iso-Propanol. Thereafter the specimen pieces were ground to about 5 – 30 micron size. 5 – 10 grams of the powder was used for XRD analysis. The measurement parameters were:-

- Angular range: 5…90° (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

3.14 Specimens Designation

A typical formulation used in the experimental program may be written as; C + 10 BA, 1D where the first letter donates cement, followed by a numeral which indicates mass of secondary raw material in percent mass of cement. The next letters denote secondary raw material type, followed by the age of sample in days. The designation does not specify water and super plasticizer content, as for all formulations, the mixing water used was equal to water demand of the system and the super plasticizer added was equal to amount required for the target flow of 30±1 cms using Hagerman’s mini slum cone which is given in Table 2 of Annexure A.

CHAPTER 4 – RESULTS

4.1 Particle Characterization by Scanning Electron Microscopy

Secondary raw material's particle shape and morphology are quite important parameters for understanding their role in flow, strength, shrinkage and microstructure of self compacting paste systems. The scanning electron microscopic pictures of secondary raw material particles used in the study are shown in Fig 4.1. More or less both secondary raw materials exhibited different morphologies and shapes. Average particle size of these secondary raw materials conforms to that given in Table 3.1.

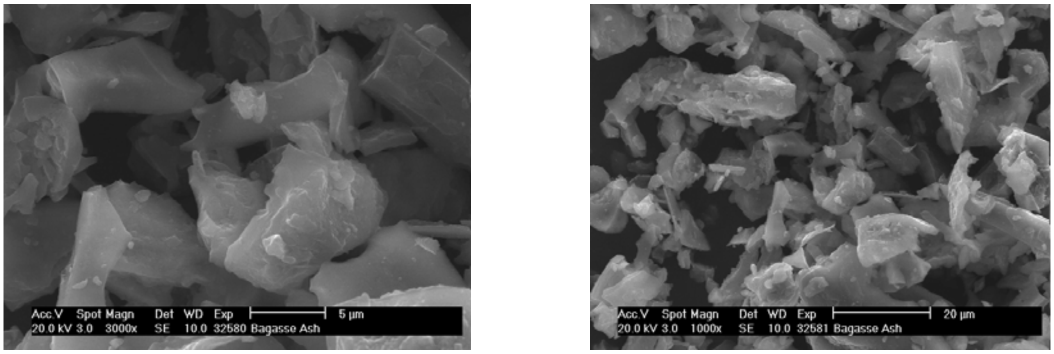


Fig 4.1(a) SEM characterization of Bagasse Ash particles

Fig 4.1(a) shows broken, elongated and flaky bagasse ash particles of varying sizes. The surface of these particles seems comparatively smoother and less porous. Fig 4.1(b) shows bentonite particles, which possess irregular surfaces and seems to have internal porosity.

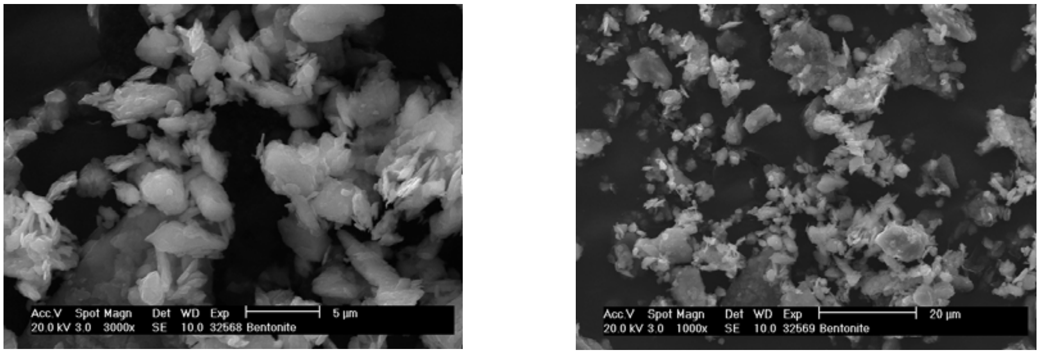


Fig 4.1(b) SEM characterization of Bentonite particles

Fig 4.1 Scanning Electron Microscopic Images of Secondary Raw Materials

4.2 Water Demand, Super Plasticizer Demand, Setting Times and Mix Proportions

Fig 4.2 shows the water demand, super plasticizer demand and setting times of self compacting paste formulations. The water demand and super plasticizer demand for each secondary raw material increases with increase in the amount being used. However, the change in the setting time of formulations varies according to the individual characteristics of each secondary raw material used.

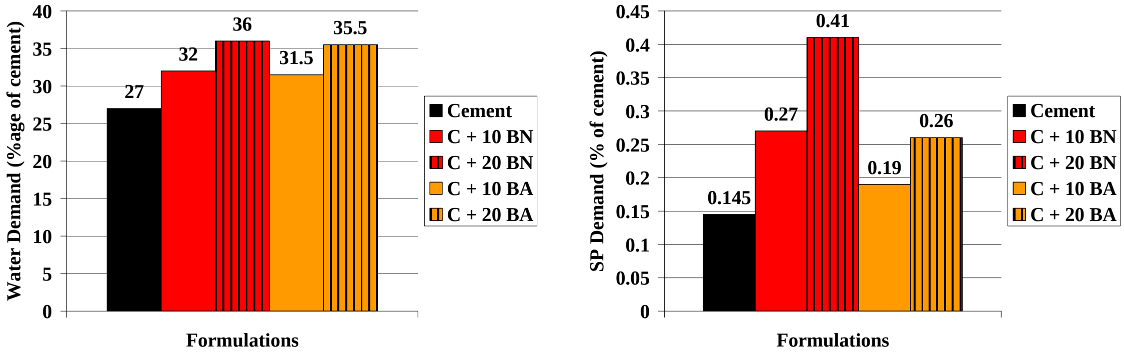


Fig 4.2(a) Water Demand   Fig 4.2(b) Super Plasticizer Demand

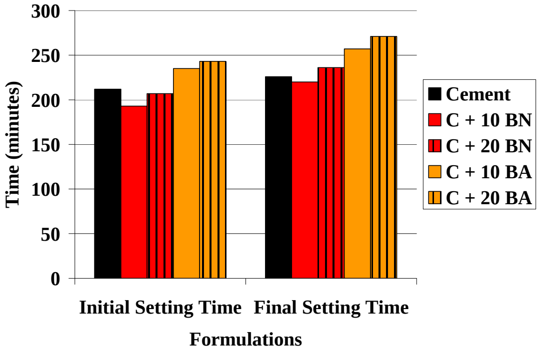


Fig 4.2(c) Setting Times

Fig 4.2 Water Demand, Super Plasticizer Demand and Setting Times of SCP formulations

The results of Water Demand, Super Plasticizer Demand and Setting Times of various self compacting paste formulations are also given in tabulated form in Table 2 of Annexure A.

The mix proportions of various self compacting formulations used are given in Table 1 of Annexure A.

4.3 Flow of SCP Formulations

Fig 4.3 shows the Hagerman's mini slump cone time for a spread of 25 cms and Fig 4.4 shows the V Funnel time of formulations. Fig 4.5 shows the reduction of total spread of the formulations with time using Hagerman's mini slump cone.

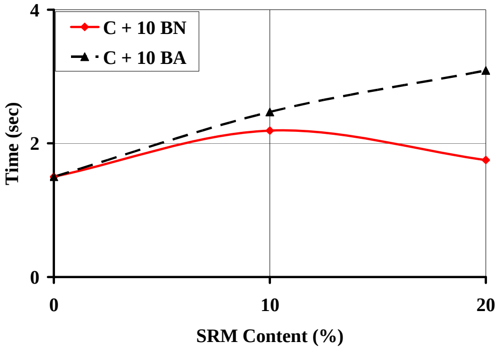


Fig 4.3 Variation of T25 cms time of SCP formulations

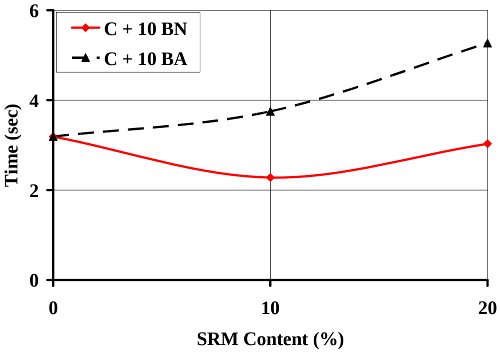


Fig 4.4 Variation of V Funnel time of SCP formulations

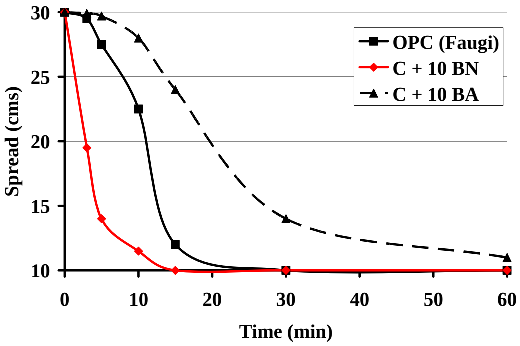


Fig 4.5 Estimation of Thixotropy of Various SCP formulations

4.4 Strength

Fig 4.6 and Fig 4.7 show the compression as well as flexure strength development with time of specimen pertaining to different formulations.

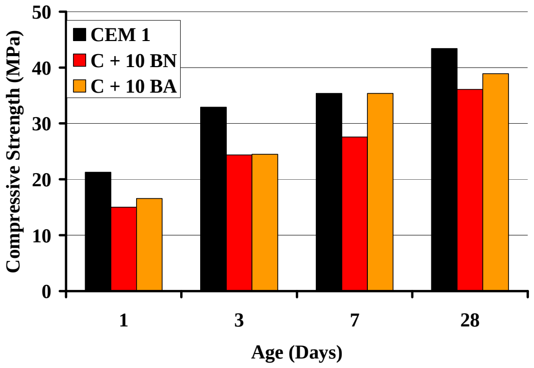


Fig 4.6 Compressive Strength of SCP with various secondary raw materials

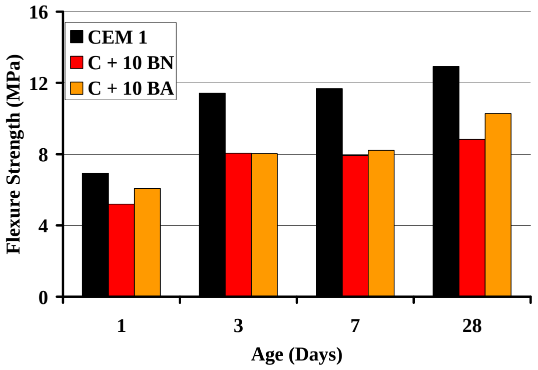


Fig 4.7 Flexural Strength of SCP with various secondary raw materials

Fig 4.8 shows the relative water absorption at of 3, 7, 15 and 28 days of age of different formulations with reference to internal moisture present in samples at the time of demoulding.

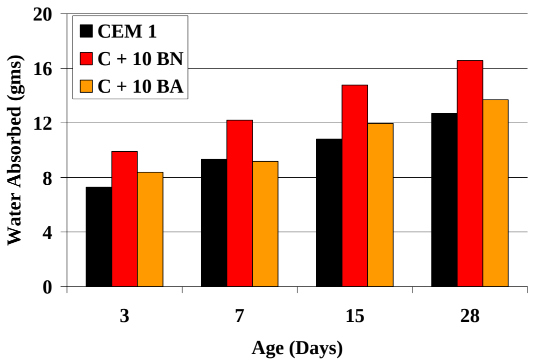


Fig 4.8 Relative water absorption of SCP systems using various secondary raw materials

The strength results of various self compacting paste formulations are also given in tabulated form in Table 1 of Annexure B.

4.5 Calorimetry

The objective of the Calorimetry was to analyze the effects of addition of secondary raw materials on the heat released and on the hydration kinetics of the self compacting paste systems. Fig 4.9 shows the graphic representation of heat flow of self compacting paste systems for duration of 72 hour.

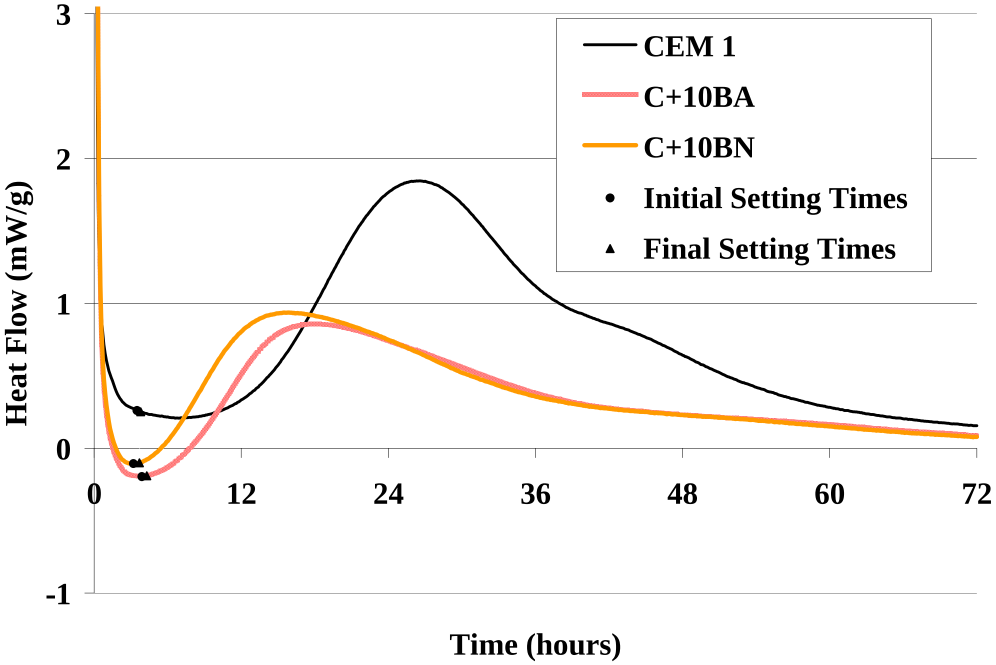
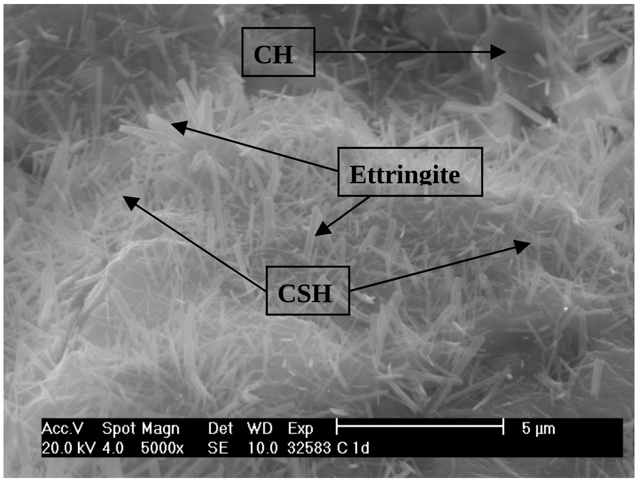


Fig 4.9 Heat Flow Representation by Calorimetry of various SCP Systems

4.6 Study of Microstructure by Scanning Electron Microscopy

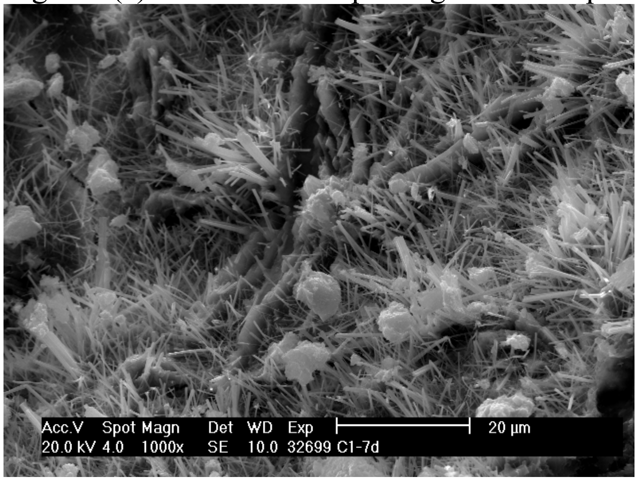
The scanning electron microscopic pictures of various formulations at the age of 1 and 7 days along with Dispersive X – ray Spectroscopy (EDAX) analysis are shown in Fig 4.10. All formulations exhibit the growth of ettringite in the form of needle like cubic crystals and growth of calcium hydroxide’s large hexagonal crystals. Calcium silicate hydrate gel which does not possess a well defined crystalline structure is also visible at various places. The graphic representation by EDAX of self compacting paste formulations is also shown in Fig 1 to Fig 6 of Annexure C.



EDAX

Elements	Weight (%)
MgO	02.48
Al_2O_3	07.92
SiO_2	23.16
SO_3	07.34
K_2O	01.20
CaO	54.33
Fe_2O_3	03.57

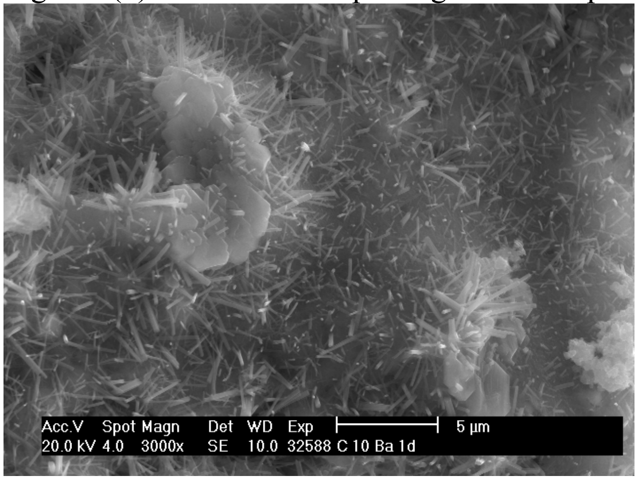
Fig 4.10(a) Pure Self Compacting Paste Sample at an Age of 1 Day along with EDAX



EDAX

Elements	Weight (%)
MgO	00.40
Al_2O_3	13.43
SiO_2	08.78
SO_3	17.44
K_2O	01.06
CaO	57.62
Fe_2O_3	01.28

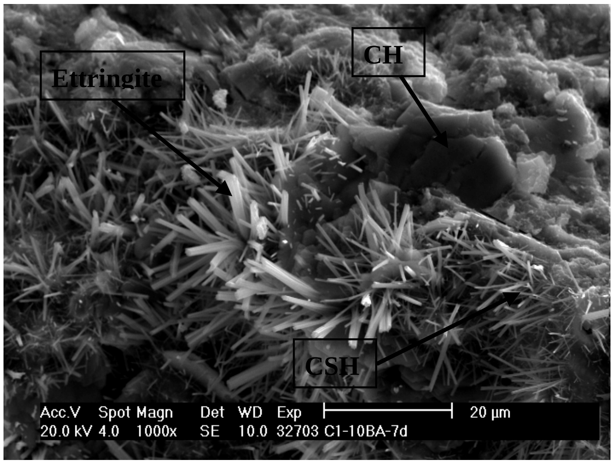
Fig 4.10(b) Pure Self Compacting Paste Sample at an Age of 7 Day along with EDAX



EDAX

Elements	Weight (%)
MgO	01.18
Al_2O_3	07.45
SiO_2	17.22
SO_3	06.51
K_2O	00.64
CaO	61.08
Fe_2O_3	05.92

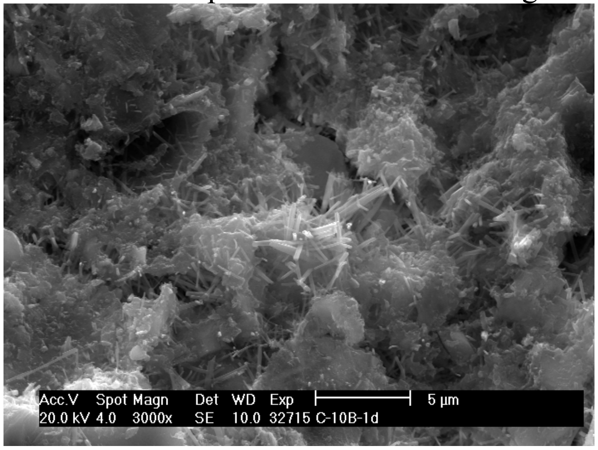
Fig 4.10(c) Self Compacting Paste Sample containing 10% Bagasse Ash in Replacement Mode at an Age of 1 Day along with EDAX



EDAX

Elements	**Weight (%)**
MgO	00.66
Al_2O_3	14.08
SiO_2	08.87
SO_3	24.04
K_2O	00.67
CaO	50.59
Fe_2O_3	01.09

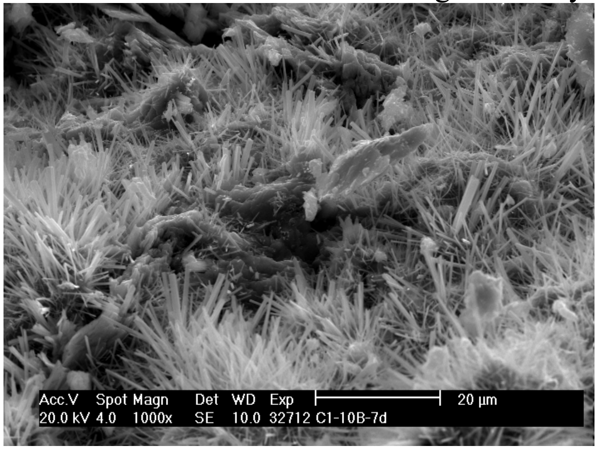
Fig 4.10(d) Self Compacting Paste Sample containing 10% Bagasse Ash in Replacement Mode at an Age of 7 Day along with EDAX



EDAX

Elements	**Weight (%)**
Na_2O	00.36
MgO	01.22
Al_2O_3	07.58
SiO_2	29.24
SO_3	08.43
K_2O	01.31
CaO	49.05
Fe_2O_3	02.80

Fig 4.10(e) Self Compacting Paste Sample containing 10% Bentonite in Replacement Mode at an Age of 1 Day along with EDAX



EDAX

Elements	**Weight (%)**
Na_2O	00.27
MgO	00.62
Al_2O_3	15.70
SiO_2	09.40
SO_3	19.74
K_2O	01.21
CaO	51.48
Fe_2O_3	01.58

Fig 4.10(f) Self Compacting Paste Sample containing 10% Bentonite in Replacement Mode at an Age of 7 Day along with EDAX

Fig 4.10 Scanning Electron Microscope Presentation of Products of Hydration along with EDAX

4.7 Mercury Intrusion Porosimetry (MIP)

In order to get knowledge of pore structure and various other parameters of secondary raw material particles, mercury intrusion porosimetry was done with the help of Pascal 440 Porosimeter. Mercury intrusion porosimetry was done up to only 7 days as after three days there is only a very little improvement in the pore structure refinement [7]. Fig 4.11 shows the average pore size radius, total porosity and bulk density of the samples. These values are directly obtained from Pascal 440 mercury intrusion porosimeter result output data sheet. A typical Pascal 440 mercury intrusion porosimeter result output data sheet is given in Table 1 to Table 4 of Annexure D.

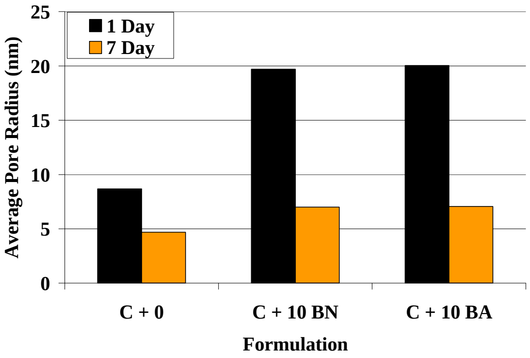


Fig 4.11(a) Average pore width of SCP formulations

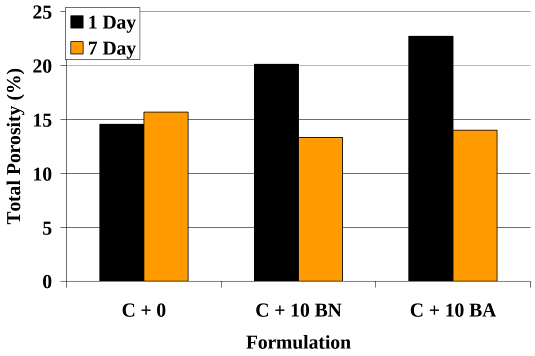


Fig 4.11(b) Total Porosity of SCP formulations

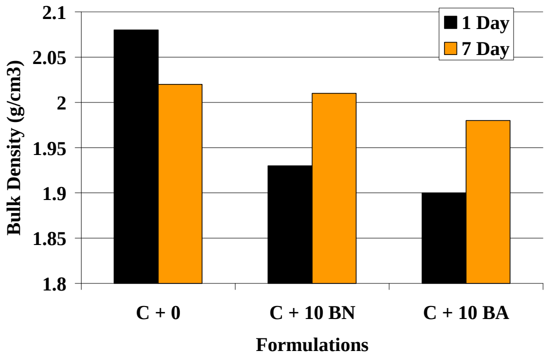


Fig 4.11(c) Bulk Density of SCP Systems

Fig 4.11 Various Properties of Hydrated Samples Measured by MIP

Fig 4.12 shows the relationship between pore size and volume intruded of intruded of self compacting paste system. The threshold diameters were approximately obtained by visual inspection of curves plotted in Microsoft Office Excel Sheets as shown and marked with arrows in Fig 4.12. These are also tabulated in Table 5 of Annexure D along with the maximum pore radius of self compacting paste formulations. Fig 1 and Table 6 of Annexure D shows the pore radius range distribution with relative volume intruded.

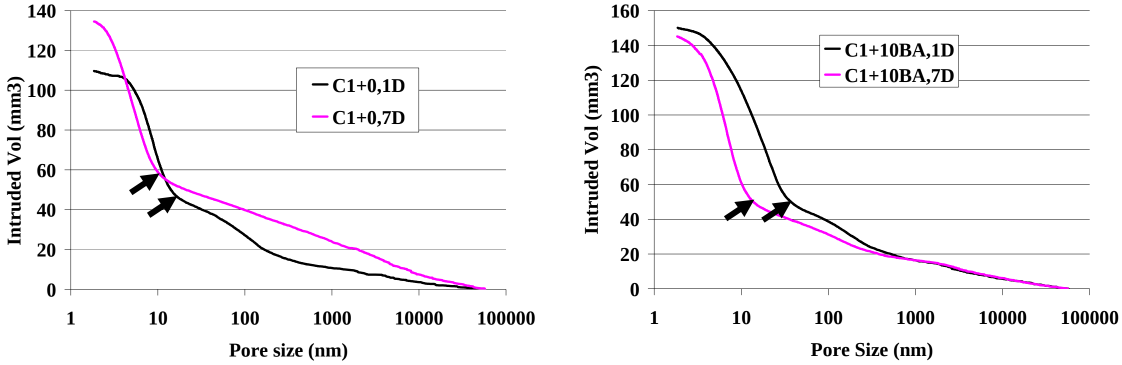


Fig 4.12(a) Pure Self Compacting Paste Sample at specified ages

Fig 4.12(b) SCP samples containing 10% Bagasse Ash in replacement mode at specified ages

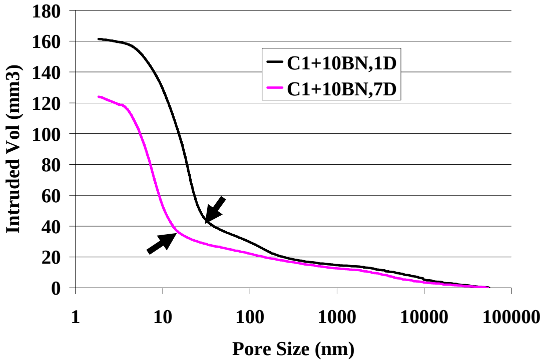


Fig 4.12(c) SCP samples containing 10% Bentonite in replacement mode at specified ages

Fig 4.12 Mercury Intrusion porosimetry study of various SCP Systems

4.8 Early Shrinkage

Fig 4.13 shows the arrangement of early shrinkage measurement of self-compacting cementitious systems studied in this research. The shrinkage plots presented in figure show the effect of secondary raw material on the amount of early shrinkage of self-compacting paste systems measured in two exposure conditions i.e. covered and uncovered conditions.

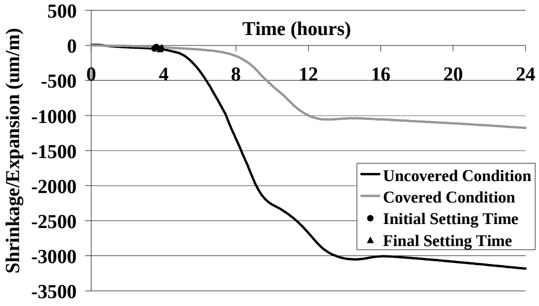


Fig 4.13(a) Pure Self Compacting Paste Sample

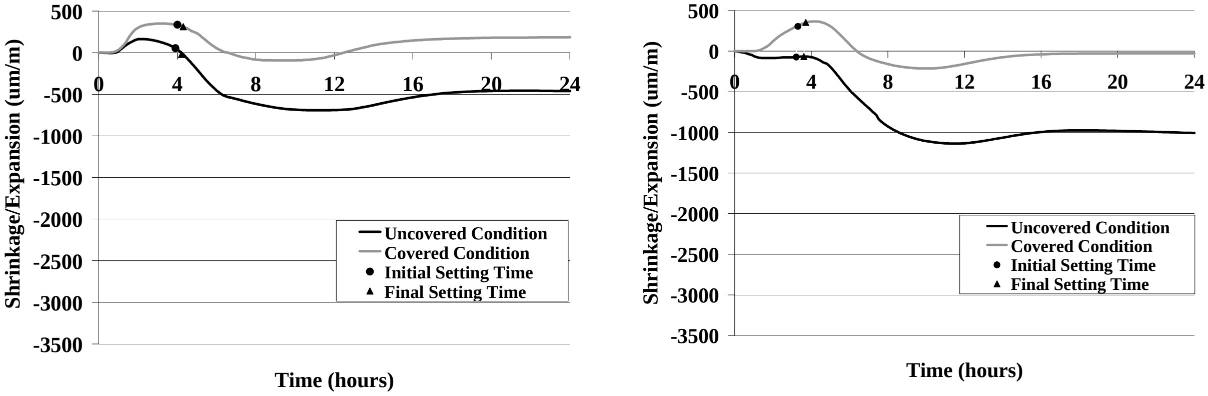


Fig 4.13(b) SCP samples containing 10% Bagasse Ash in replacement mode

Fig 4.13(c) SCP samples containing 10% Bentonite in replacement mode

Fig 4.13 Volume Stability study of various SCP Systems

4.9 X – Ray Diffraction

Fig 1 to Fig 9 of Annexure E show the X – ray Diffraction analysis of the cement particles, secondary raw material particles and self compacting paste formulations at specified ages. Quantitative phase analysis was possible for the samples of Faugi Brand Cement and Bentonite particles only. However, for Bagasse Ash and all SCP formulations qualitative analysis was carried out. The results show that Faugi cement sample contains typical OPC phases, in particular Alite (60%), Belite (17%), Ferrite (11%), Aluminate (7%), and Anhydrite (4%). The bagasse ash sample has a large amorphous portion accompanied by minor amounts of Quartz and Cristobalite. Furthermore, there is a trace of a phase that is possibly feldspar (Albite). Bentonite sample contains Quartz (25%), Calcite (5%), clay minerals (Clinochlore, Illite;50%), and feldspar (Albite, Microcline; 18%). C + 0, 1D sample contains typical phases of hydrating OPC based mortar, in particular Alite, Belite, Ferrite, Quartz, Portlandite, and Ettringite. C + 10BA, 1D sample contains Alite, Belite, Ferrite, Quartz, Portlandite, and Ettringite phases and additional Cristobalite. C + 10BA, 1D sample contains Alite, Belite, Ferrite, Quartz, Portlandite, and Ettringite phases. In all samples of self compacting paste formulations at an age of 7 days, the same phase assemblage can be found. Alite, Belite, and Quarz are non-hydrated remainders and Ettringite and Portlandite are hydrated part of the formulations.

CHAPTER 5 – DISCUSSION

5.1 ASTM Requirements for Pozzolanic Secondary Raw Material

Table 5.1 compares the chemical and physical requirements of ASTM C 618 – 01 [38] for the secondary raw materials to be classified as pozzolans. Bagasse Ash satisfactorily fulfils the requirements to be classified as pozzolan except having a little higher loss on ignition which indicates the higher water and carbon contents. Bagasse Ash also has a little higher water requirement. Bentonite also qualifies ASTM requirements except for the moisture content and a small excess requirement of water. The greater moisture content may be due to the reason that Bentonite was collected from the quarry site close to a stream bed.

Table 5.1 Comparison of Chemical and Physical Properties of secondary raw materials with selected ASTM C 618–1 Requirements to Classify a Powder as Pozzolanic

Parameters	Bagasse Ash	Bentonite	Requirements of ASTM C 618 – 01 (Class N)
$SiO_2 + Al_2O_3 + Fe_2O_3$ (%)	70.02	77.63	> min 70.0%
SO_3 (%)	0.15	Nil	< 4.0%
Moisture content (%)	2.89	3.12*	< 3.0%
Loss on Ignition (%)	16.65	7.76	< 10.0%
Strength Activity Index			
• 7 days strength (% of control)	100**	77.26**	> min 75.0%
• 28 days strength (% of control)	89.18**	83.18**	> min 75.0%
Water requirement (% of control)	116.7**	118.5**	< 115.0%

* collected near a stream bed.

** formulations containing 10% secondary raw materials as cement mass replacement.

5.2 Particles Characterization, Water and Super Plasticizer Demand and Setting Times

Particle shape, size and morphology of secondary raw materials are quite important parameters for understanding their role in terms of water and super plasticizer demands, flow, strength, shrinkage and microstructure of self compacting paste systems. The scanning electron microscopic pictures of secondary raw material particles used in the study are shown in Fig 4.1. Both the secondary raw materials exhibited different morphologies and shapes. The average particle size of these secondary raw materials, as visible in the scanning electron microscopic images (Fig 4.1), conforms to that given in Table 3.1. Fig 4.1(a) shows the broken, elongated and flaky bagasse ash particles of varying sizes. This variation in the size makes it suitable for better packing, however due to their shape (broken and elongated), these offer higher internal resistance during flow (Fig 4.4) and result in the longer flow times. However, the surface texture of these particles seems smooth and less porous as compared to bentonite particles. Fig 4.1(b) shows bentonite particles, which possess irregular surfaces and seems to have internal porosity. The bentonite has a smaller particle size than bagasse ash particles and its surface seems to have numerous minor pores. Due to such characteristics, bentonite particles show higher water demand and super plasticizer demand for the target flow (Fig 4.2(a) and Fig 4.2(b)) and higher relative water absorption by the SCP sample prisms made for strength test (Fig 4.8).

Water demand and super plasticizer demand for both bagasse ash and bentonite increased with their increase in percentage used as cement replacement. This is due to the fact that particles of bagasse ash as well as bentonite both show porosity though bagasse ash particles seem less porous. Bentonite particles have greater specific surface as compared to bagasse ash particle. The greater specific surface shows greater surface pozzolanic reaction capability of the particles and accordingly the greater is the water demand as well as super plasticizer demand of bentonite (Fig 4.2). Specific surface may also indicate porosity. Greater specific surface with smaller particle size would mean that the particles have more internal porosity. This can be seen in the scanning electron microscopic images of bentonite particles as shown in Fig 4.1.

Furthermore, it has already been documented [34, 50, 51] that certain amount of super plasticizer is also adsorbed by secondary raw materials depending upon the morphology and surface texture of the particles. Again same can be confirmed by higher super plasticizer demand of Bentonite (Fig 4.2(b)).

Retardation in setting time of self compacting formulation is also recorded with bagasse ash replacement and is more pronounced as the content of bagasse ash increases (Fig 4.2(c)). This is due to dilution effect and the higher carbon content (LOI equal to 16.65 %) of bagasse ash [10]. In addition, the greater particle size of bagasse ash also contributes towards the retardation. On the other hand, there is an overall acceleration in the setting time for the self compacting formulation with bentonite which can be attributed to the smaller particle size of bentonite with more specific surface. Furthermore, the setting time for bentonite self compacting formulation decreases with 10 percent replacement and then there is a slight increase with 20 percent replacement. With 10 percent replacement, the voids between the cement grains are effectively filled with the smaller particles of bentonite and these particles acts as the centre of nucleation for deposition of products of hydration, hence accelerates the setting. With further increase, these smaller particles, now being greater in number, increase the particle to particle distance between the cement grains, thus resulting in slight increase of setting time.

5.3 Flow of Self Compacting Paste Systems

After establishing the water demand, super plasticizer demand and the setting times, the next parameter to be investigated for self compacting cementitious system is the flowability of the cementitious system; for which slump flow and V funnel tests are usually prescribed. The slump flow test using mini slump cone aims at investigating yield stress, deformability and the unrestricted filling ability of self compacting cementitious systems. It basically measures two parameters; the total flow spread and a suggested flow time T25 cms. The former indicates the free unrestricted deformability or the yield stress and the later indicates the rate of deformation within a defined flow distance. The smaller value of flow time T25 cms indicates lesser internal friction offered, during the flow, by the powder particles and translates into higher deformation. The V funnel time, on the other hand, is the time required by a defined volume of self compacting cementitious system to pass through a constantly narrowing cross section. It indicates the viscosity of self compacting cementitious system while overcoming internal as well as external friction encountered during the flow [3, 7]. With higher w/p ratio, the cementitious system flows faster out of the V funnel. In addition, the flow through the changing cross section of V funnel also depends heavily upon the shape of secondary raw material particles.

The trend of the T25 cms time curves (Fig 4.3) and V Funnel time curves (Fig 4.4) is opposite to each other i.e. T25 cms time initially increases with 10% secondary raw material replacement and then either decreases or its rate of increase reduces with further increase in the secondary raw material content. On the other hand, it is vice versa for V Funnel time or the viscosity. In general, this specifies the positive effect of secondary raw material’s replacement on the characteristic requirements of self compacting cementitious systems that is low yield stress and higher viscosity. While in particular, the results indicate that bagasse ash particles, due to their flaky and elongated nature, offer more resistance to flow hence these formulations have higher yield stress but are also more viscous then the formulation with bentonite.

Another important aspect that can be noted in Fig 4.3 and Fig 4.4 is that time T25 cms for bentonite self compacting paste formulation increases with 10% replacement and then it decreases with 20% replacement at the similar flow spread. Similarly, the V Funnel time decreases with 10% replacement and then increases with 20% replacement. While, bagasse ash self compacting formulation shows increase in time for both the V Funnel as well as T25 cms spread, with increase in secondary raw materials percentage replacement. This can be explained as the average particle size of Bentonite is of much smaller than size of cement particle and at 10% replacement these smaller particles fill the voids between the cement grains. With further increase in secondary raw materials content, these smaller particles being greater in number, increase the particle to particle distance between the cement grains thus minimizing the yield stress but increasing the viscosity. While the increase in the time for bagasse ash formulation may be attributed to its elongated and irregular shaped particles. Moreover, the V funnel time (Fig 4.4) shows that these formulations do not behave in the same way in filling ability of the formwork and have different degrees of placement ease which again depends upon the shape and morphology. Fig 4.5 gives an estimate about the degree of thixotropy and the open working time available while using a particular secondary raw material. It also gives an idea about start of hydration process and setting times. It showed that Bentonite is quite thixotropic with minimum open working time.

5.4 Strength of Self Compacting Paste Systems

Self compacting paste formulations containing 10% bagasse ash in replacement mode gave better strength results (Fig 4.6 and Fig 4.7) at all ages up to 28 days than self compacting paste formulations containing 10% bentonite in replacement mode. This occurs as bagasse ash particles are less porous then that of bentonite particles; hence results in availability of more effective water in the mix for hydration process. The lower porosity also is reflected in terms of lesser water demand (Fig 4.2(a)) as well as lesser relative water absorption of self compacting paste formulations (Fig 4.8). However, bentonite may give better strength result at later ages as also mentioned in the previous studies [9, 10 and 37]. This aspect can also be seen in mercury intrusion porosimetry result of Fig 4.12 in which the pore refinement of bentonite self compacting paste formulation is more than bagasse ash self compacting paste formulations at an age of 7 days. The argument can be further strengthened while analyzing the rate of strength gain by bentonite as shown in Fig 5.1. While keeping the strength at 1 day of each formulation as the baseline or 100 percent, the strength gain by bentonite self compacting paste formulation is maximum at 28 days i.e. 2.41 times of the strength at 1 day, followed by bagasse ash self compacting paste formulation (2.35 times) and then pure CEM 1 self compacting paste formulation (2.04 times). It can also be inferred from Fig 5.1 that at 7 days bagasse ash formulation seen to have connected porosity which is exhibited in higher rate of strength gain but becomes disconnected at later ages.

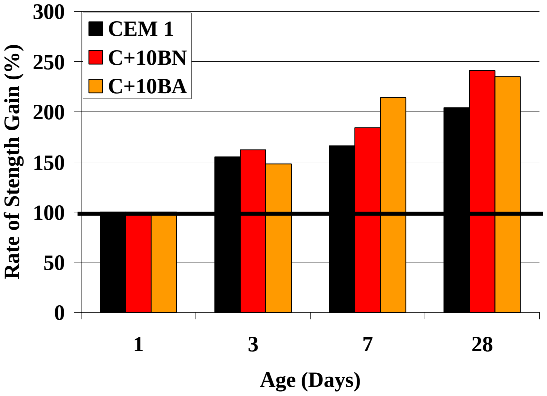


Fig 5.1. Rate of Compressive Strength Gain by Various SCP Formulations

Secondary raw material's particle size in combination with its chemical composition plays a very important role in the packing of the mix (physical effects) and pore refinement (chemical effects). If the size is two orders less than the size of cement particles it can pack the binder phase effectively and hence reduce porosity. Porosity, being the function of degree of packing, always controls the strength. The secondary raw materials which have smaller particle size than the cement particles can pack efficiently in the smaller voids between the cement grains and hence result in better packing and consequently better strength. The average particle size of bagasse ash was somewhat equal to the average particles size of CEM 1 and that of local Faugi brand Pakistani OPC, whereas bentonite had much smaller average particle sizes and gave better packing efficiency of binder phase and lower porosities in specimen due to physical, chemical and site nucleation effects. This aspect can also be seen in mercury intrusion porosimetry results (Fig 4.11, Fig 4.12 and Table 5 of Annexure D).

In general it can be stated that 10 percent replacement of cement by bagasse ash and bentonite results in reduced strength at all ages due to dilution effect as also verified by calorimetry but at later ages a better gain in strength is observed.

5.5 Calorimetric Study of Self Compacting Paste Systems

The hydration of cement phases and consequently the heat flow in the process is a complex phenomenon and depends upon various factors. It can influence concrete workability, setting behavior, strength gain rate and pore refinement; hence affect the early age behavior as well as long term performance of concrete. The knowledge and measurement of heat flow is also necessary to get an estimate of quantity of formed hydrates [52] and to get an idea about the curing regime [7].

Calorimetry is one of the techniques used to measure this heat flow with time. The Calorimetry curves shown in Fig 4.9 represent the heat flow of various formulations. To understand the heat flow during hydration process and the effect of secondary raw materials on heat evolution, it is necessary to have the knowledge of the pattern followed by the curves. These curves show various phases or peaks. The initial phase or the first peak represents the initial reaction of the aluminate phase. Here almost all the clinker phases especially aluminate phase throw their ionic species into the solution. It is followed by the dormant or induction period where the hydration reaction slows down at a rapid rate and allows the concrete to be placed before the onset of main reaction leading to setting, hardening and the strength gain by the concrete. The actual reason for this dormant period still remains unclear. However, two theories exist to explain the process. According to the first theory, a protective layer forms around the reacting grains just after the initial high heat release which prevents the further reaction till the onset of acceleration period. The main problem with this theory is that no evidence of formation of such layer has yet been obtained and there is no satisfactory explanation for disappearance of the layer with commencement of acceleration period. The second hypothesis denies the presence of any real induction period and specifies it with slow development of hydrates by nucleation and growth. Drawbacks of this theory includes that neither it explains the drastic slow down of the rate of reaction just after the initial very high heat release nor there is any clarification of the reason of slow reaction of highly soluble Alite [52, 53].

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

Fig 4.9 shows that the heat liberated at second peak is reduced after the use of both secondary raw materials in replacement mode. This occurs due to the dilution effect caused by secondary raw materials replacement resulting in reduction of cement content in the mix. However, the hydration kinetics of both formulations with secondary raw materials get increased as secondary raw material particles fill the space between the cement grains and act as centre of nucleation for deposition of hydrates. While comparing these self compacting paste systems with each other (Fig 4.9), though both bagasse ash as well as bentonite formulation of self compacting systems give almost identical hydration kinematics, but the magnitude of heat released by bentonite formulation is more than the bagasse ash formulation at the second peak as well as it occurs earlier indicating a possibly stronger pozzolanic reaction capability of bentonite. More heat liberated by bentonite self compacting formulation as compared to bagasse ash formulation can be attributed to the smaller size of bentonite particles with more surface area, chemical composition (higher content of $SiO_2 + Al_2O_3 + Fe_2O_3$ as specified by ASTM C 618) and low loss on ignition. Similar trend is also observed in other experimental results for bentonite as its particles seem more porous (Fig 4.1), its formulation sets earlier (Fig 4.2(c)), is more thixotropic (Fig 4.5) and has higher rate of strength gain (Fig 5.1) than bagasse ash formulations.

The bentonite particles also have the capability to adsorb the super plasticizer which results in minimizing retarding effect of super plasticizer by reducing its effective quantity in the mix. Hence an early heat flow is observed in the calorimetric analysis for bentonite self compacting formulation. However, the overall setting time as well as the hydration process also depends upon other factors as discussed earlier.

5.6 Microstructure of Self Compacting Paste Systems

Microstructure of cement based materials is an extremely important parameter which affects both the strength and durability characteristics. Various tools such as scanning electron microscopy, mercury intrusion porosimetry and X ray diffraction analysis are available to study the microstructure of cement based composites. For scanning electron microscopy a very small space of a specially prepared representative sample is viewed at high magnifications with the help of special instruments to see the hydration products, presence of different phases and their interconnectivity. An expert study of images obtained can give very useful and accurate information about microstructure of cement based composites.

The scanning electron microscopic images of various self compacting formulations at the age of 1 and 7 days are shown in Fig 4.10 to study the products of hydration. All formulations exhibit the growth of ettringite in the form of needle like cubic crystals, calcium hydroxide's large hexagonal crystals and the calcium silicate hydrate gel which does not possesses a well defined crystalline structure. The growth of crystals corresponds to the specified ages in the images.

The microstructure study of self compacting paste formulations by mercury intrusion porosimetry showed that by incorporating the secondary raw materials in cement pastes, the size of maximum pore reduces rapidly with the age compared with pure cement self compacting formulations. This can be attributed to better pore refinement effect of secondary raw material by pozzolanic mechanism in addition to the hydration reaction and the better packing of the binder phase. The mercury intrusion porosimetry studies shows that the maximum reduction in pore size was recorded for self compacting paste system containing bentonite (Table 1 of Annexure D). As Cook [17] reports that threshold pore width may provide a better indicator of material durability and has an important influence on the permeability characteristics of cement paste, so basing on the argument, it can be said that bentonite formulation being having smaller threshold pore radius (Table 1 of Annexure D) may also exhibit durability characteristics at later stages. The mercury intrusion porosimetry for CEM 1 self compacting formulation samples shows an increase in porosity at an age of 7 days. This phenomenon can be explained by better pore connectivity within the matrix of pure cement paste sample at an age of 7 days than that of bagasse ash and bentonite self compacting formulations.

Fig 4.1 shows various parameters of self compacting paste formulation obtained from mercury intrusion porosimetry results and Fig 4.2 shows the intruded volume of mercury in the sample of self compacting paste formulations. The intruded volume of mercury is maximum for bentonite self compacting formulation at the age of 1 days. This supplements the argument that bentonite has got the internal porosity. However, at an age of 7 days bentonite formulation shows least total porosity (Fig 4.11) and the least intruded volume of mercury (Fig 4.12). This also confirms the better pozzolanic reaction capability of bentonite.

The X – ray diffraction analysis also furnishes important information about secondary raw materials and the hydration products. A hump or a wide band in XRD pattern suggests the presence of amorphousness of materials (vitreous matter) and absence of such band shows crystallinity [20, 40]. For bagasse ash this band is observed between 18^o and 32^o (Fig 4 of Annexure E) and for bentonite it is from 22^o to 32^o (Fig 7 of Annexure E). With the age, a decrease in vitreous wide band for hydrated samples is also observed which suggest formation of non crystalline compounds during hydration process. Compounds formed by pozzolanic reaction are likely to be calcium silicate hydrate belonging to tobermorite family whose poor crystallinity is well known and contributes to difficulty in their identification.

It is also important to note from XRD results that bagasse ash has a large amorphous portion accompanied by minor, but predominant presence of crystalline phases of Quartz and Cristobalite and there is a trace of a phase that is possibly feldspar (Albite) (Fig 4 of Annexure E). Bentonite contains 25% Quartz, 5% Calcite, 50% clay minerals (39% illite, 11% Clinochlore) and 18% feldspar (16% Albile, 2% Microcline) (Fig 7 of Annexure E). The XRD results for Faugi Cement sample (Fig 1 of Annexure E) showed that it contains typical OPC phases, in particular Alite (60%), Belite (17%), Ferrite (11%), Aluminate (7%), and Anhydrite (4%). The pure self compacting paste sample (Fig 2 of Annexure E) and SCP sample containing 10 % bentonite in replacement mode (Fig 8 of Annexure E) both at the age of 1 day contains typical phases of hydrating OPC based paste, in particular Alite, Belite, Ferrite, Quartz, Portlandite and Ettringite; whereas the SCP sample containing 10 % bagasse ash in replacement mode at the age of 1 day (Fig 5 of Annexure E) contains the same phases along with the additional Cristobalite. In all samples at the age of 7 days (Fig 3, Fig 6 and Fig 9 of Annexure E), the same phase assemblage can be found. Alite, Belite, and Quartz are non-hydrated remainders and Ettringite and Portlandite are hydrated part of the material.

5.7 Dimensional Stability of Self Compacting Paste Systems

Total shrinkage of self compacting paste formulations is the sum of various parallel and over lapping simultaneously operating mechanisms including plastic shrinkage, chemical shrinkage, autogenous shrinkage and drying shrinkage. In general, shrinkage is caused by internal consumption of water (during hydration process or suction by porous secondary raw materials) / external loss of water to environment. The expansion occurs mainly due to the growth of expansive species, thermal gradient and may also be due to re – absorption of bleed water in the system. The measurement technique of shrinkage includes volumetric and linear method.

The paste phase is mainly responsible for the shrinkage / expansion of the cementitious systems. Greater the cement content in the paste phase greater would be the shrinkage due to higher consumption of water during hydration process. As the amount of cement is reduced by secondary raw material replacement; lesser shrinkage is noted in bagasse ash as well as bentonite self compacting paste formulations. By addition of both these pozzolans in replacement mode, independently in self compacting paste systems, expansion occurs in the covered condition for initial 6 hours for both formulations. Later, the bagasse ash formulation again expands after 12 hours whereas; the bentonite formulation undergoes the shrinkage recovery after 10 hours. While in uncovered condition, there is a great deal of reduction in shrinkage for both self compacting formulations with secondary raw materials.

For CEM 1 self compacting paste formulation in both covered and uncovered conditions, the shrinkage starts after around four hours (final set) while for formulation with both secondary raw materials, there is an early start of shrinkage / expansion and later a plateau in the curve is observed (Fig 4.13). This confirms that the pozzolanic reaction capability of both secondary raw materials which goes side by side with the hydration process. This fact is also evident in the calorimetric results as both self compacting paste formulations of bagasse ash as well as bentonite show early heat flow than CEM 1 self compacting formulation. The early hydration as well as pozzolanic reaction probably results in the production of ettringite which is expansive specie and usually is formed at early ages (earlier than CSH gel) as a result of reaction of alumina with calcium sulfate.

In Fig 4.13(b), for uncovered condition of bagasse ash self compacting formulation, the initial expansion may by attributed to bleed water and after the final set, the shrinkage starts. Whereas, for covered condition, though the pattern followed by the curve is generally the same, but more expansion is observed in the form of a plateau especially after 12 hours which specifies the growth and precipitation of expansive calcium hydroxide. In addition, the thermal effects also contributes towards the expansion as there is less exchange of temperature with the environment in covered condition hence thermal effects also contributes towards the expansion.

For the uncovered condition of bentonite self compacting formulation, no expansion is observed due to the water suction by the porous secondary raw material and in covered conditions though the pattern followed is the same as that of bagasse ash formulation curve but only shrinkage recovery is observed and there is no expansion (Fig 4.13(c)). This may again be attributed to the water suction by bentonite, thus leaving lesser water for growth of expansive calcium hydroxide.

While summarizing the shrinkage effects of bagasse ash and bentonite replacements in self compacting paste systems, it can be stated that water absorption by secondary raw material, its pozzolanic activity, heat liberated during hydration / pozzolanic reaction and the thermal effects all contribute towards the shrinkage / expansion of the self compacting paste formulations and their combination gives the final response of total shrinkage or expansion.

CHAPTER 6 – CONCLUDING REMARKS

This research work provides useful information and data about the use of local secondary raw materials in self compacting cementitious systems. Based on test results, it can be stated that a wide range of properties of each secondary raw material is required to be carefully analysed before commenting its overall response in self compacting cementitious systems. The shape, size, surface texture, morphology, internal porosity and chemical composition of secondary raw material's particles all have vital effect on water demand, super plasticizer demand, flow behavior, volume stability, micro – structure refinement, strength development and the durability of self compacting paste system. The important conclusions drawn from the study are as under:-

- Use of bagasse ash in self compacting paste systems in comparison with bentonite gives better strength results, shows lesser heat release at second peak in calorimetric investigations due to delayed setting and improves volume stability of the system. Thus, it can be utilized in variety of applications of self compacting cementitious systems. However, it also confers higher flow timing of the formulations which is reflective of increased internal resistance. This drawback is mainly due to the particle size and its shape. Hence in order to optimize the flow response, the fineness of bagasse ash must be increased by milling process to get an optimum particle size which should results in better flow characteristics of the formulation as well as packing efficiency of the binder phase.
- Bentonite based self compacting paste formulations exhibit least flow timings with lesser yield stress and adequate viscosity, but is more thixotropic. Though, more shrinkage is observed as compared to bagasse ash formulation at given time, but it gives much lesser shrinkage than pure cement self compacting paste formulation probably

due to dilution effect. Its formulation also shows better micro – structure development and pore refinement with age, thus exhibiting the durability characteristics. Hence, bentonite may also find its way in advanced self compacting concrete technology but a more careful application is required due its porous and expansive nature.

- Maximum amount of secondary raw material to be used in self compacting paste systems may not exceed 10% of the cement content in replacement mode as any further increase would not only affect the water and super plasticizer demand as well as the strength characteristics but would also violate the ASTM C618 requirements outlined in the thesis.
- A particular secondary raw material, depending upon its individual morphology, shape, size and chemical composition, exhibits improvement in certain properties of modern concretes while the other may enhance certain other properties of the self compacting cementitious systems. Hence, in order to get a better overall response of self compacting cementitious systems, the blends of bagasse ash and bentonite may be investigated.

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ANNEXURE A – BOGUE'S LIMIT CALCULATIONS, MIX PROPORTIONS, WATER DEMAND, SUPER PLASTICIZER DEMAND AND SETTING TIMES OF SCP FORMULATIONS

Table 1 – Bogue's Limit Calculations [11, 47]

Clinker Phase	**Formula** **(Oxides* in percentage)**	**Calculated Value of CEM 1 (%)**
Tetra – calcium Aluminoferrite (C_4AF)	3.043 (Fe_2O_3)	4.38
Tri – calcium Aluminate (C_3A)	2.65 (Al_2O_3) – 1.692 (Fe_2O_3)	11.37
Tri – calcium Silicate (C_3S)	4.071 (CaO) – 7.6 (SiO_2) – 6.718 (Al_2O_3) – 1.43 (Fe_2O_3) – 2.852 (SO_3)	60.68
Di – calcium Silicate (β –C_2S)	2.867 (SiO_2) – 0.7544 (C_3S)	9.18

* Values of Oxides in percentage are taken from Table 3.1.

Table 2 – Mix Proportions of SCP formulations

Formulations	**Cement (kg/m^3)**	**SRM Content (kg/m^3)**	**Water Content (kg/m^3)**	**Paste Density (kg/m^3)**
Pure Cement SCP Formulation	1679.58	-	453.49	2133.07
SCP Formulation containing 10% Bagasse Ash	1454.07	145.41	458.03	2057.51
SCP Formulation containing 20% Bagasse Ash mode	1290.38	258.08	458.09	2006.55
SCP Formulation containing 10% Bentonite	1463.60	146.36	468.35	2078.31
SCP Formulation containing 20% Bentonite	1314.43	262.89	473.20	2050.52

Calculations for mixed proportions were based on following formula:-

Total Volume (take 1 m^3) = Mass of Cement / Density of Cement + Mass of Water in terms of percent of Cement / Density of Water + Mass of SRM in terms of percent of Cement / Density of SRM + Volume of Air (say 2 % of total volume)

The units of mass are in kgs and density is in kg/m^3.

For example, formula for SCP Formulation containing 10% Bagasse Ash will be:-

$1 = C / 3190 + 0.315C / 1000 + 0.1C / 2198 + 0.02$

Table 3 – Water Demand, Super Plasticizer Demand for Target Flow and Setting Times of SCP formulations

	Pure Cement SCP Formulation	SCP Formulation containing 10% Bentonite	SCP Formulation containing 20% Bentonite	SCP Formulation containing 10% Bagasse Ash	SCP Formulation containing 20% Bagasse Ash
Water Demand (%)	27	32	36	31.5	35.5
Super Plasticizer (%)	0.145	0.27	0.41	0.19	0.26
Initial Setting Time (min)	212	193	207	235	243
Final Setting Time (min)	226	220	236	257	271

ANNEXURE B – STRENGTH RESULTS

Table 1. Flexure and Compression Strength of SCP formulations

Age	Flexure Strength			Compression Strength		
	CEM 1	C+10BN	C+10BA	CEM 1	C+10BN	C+10BA
	Stress (N/mm^2)					
1 Day	6.92	5.2	6.07	21.25	15.01	16.56
3 Days	11.42	8.06	8.03	32.92	24.37	24.46
7 Days	11.68	7.92	8.22	35.37	27.56	35.37
28 Days	12.92	8.83	10.28	43.4	36.1	38.9

ANNEXURE C – ENERGY DISPERSIVE X – RAY SPECTROSCOPY (EDAX)

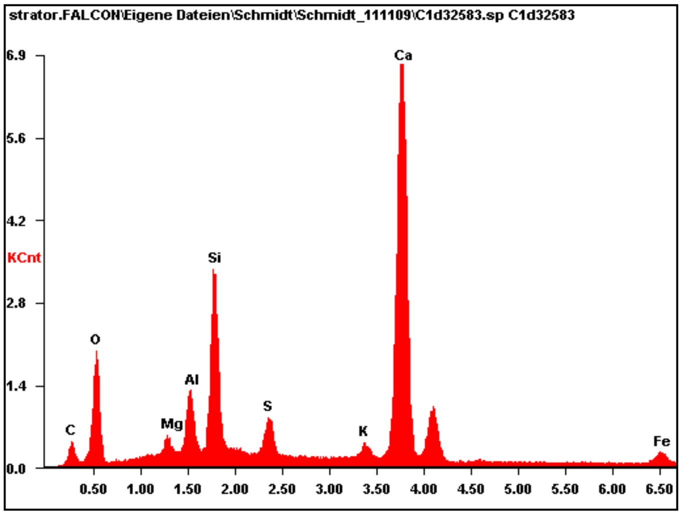

Fig 1. EDAX of Pure Self Compacting Paste Sample at an age of 1 Day

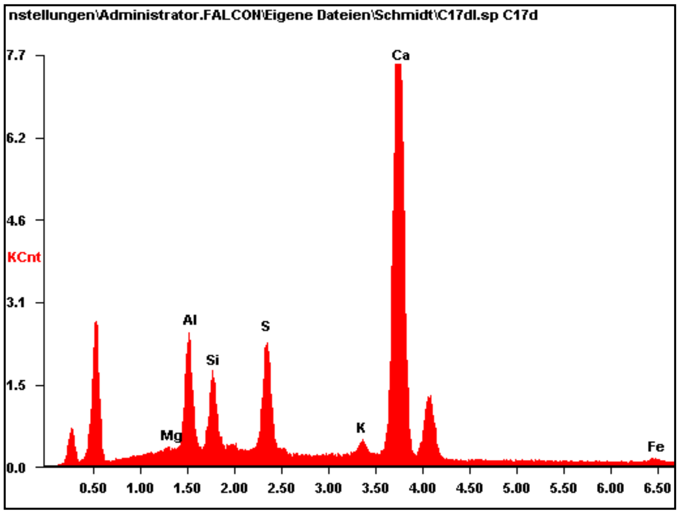

Fig 2. EDAX of Pure Self Compacting Paste Sample at an age of 7 Day

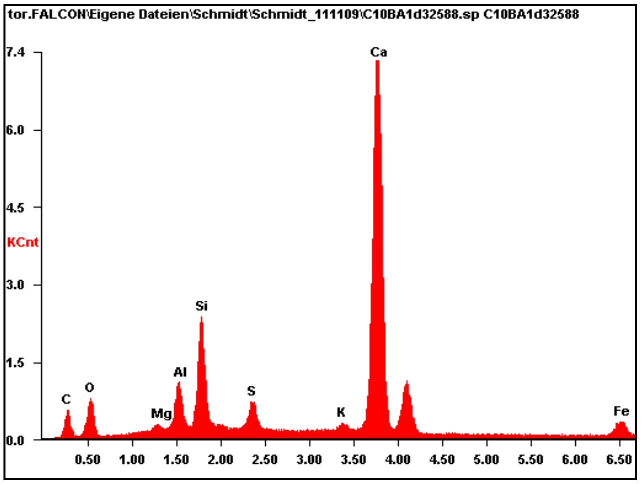

Fig 3. EDAX of Self Compacting Paste sample containing 10% Bagasse Ash in replacement mode at an age of 1 Day

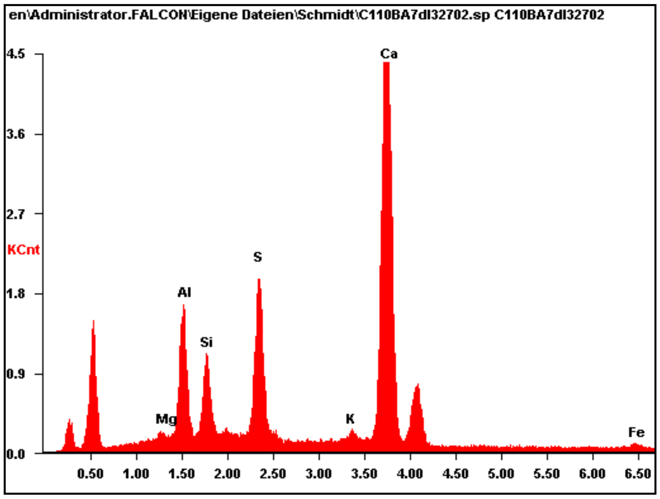

Fig 4. EDAX of Self Compacting Paste sample containing 10% Bagasse Ash in replacement mode at an age of 7 Day

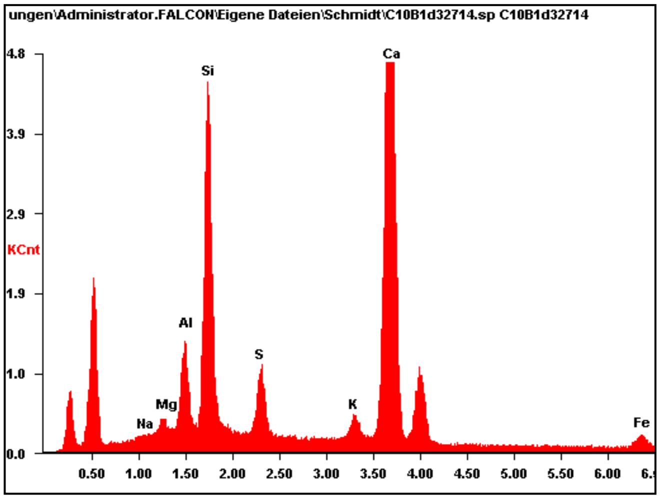

Fig 5. EDAX of Self Compacting Paste sample containing 10% Bentonite in replacement mode at an age of 1 Day

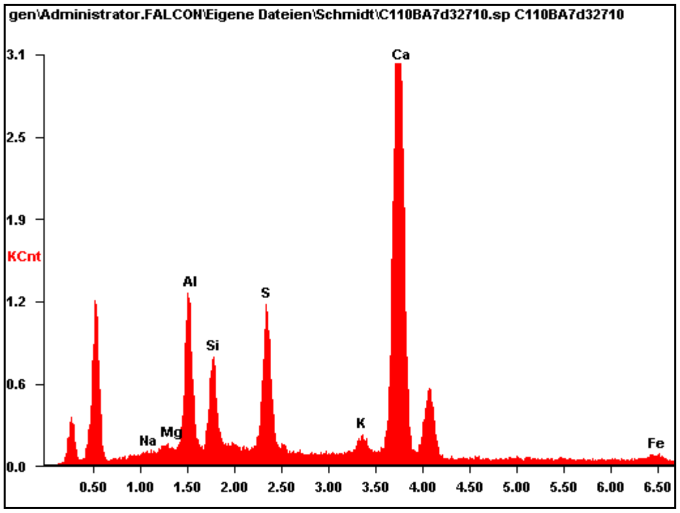

Fig 6. EDAX of Self Compacting Paste sample containing 10% Bentonite in replacement mode at an age of 7 Day

ANNEXURE D – MERCURY INTRUSION POROSIMETRY RESULTS

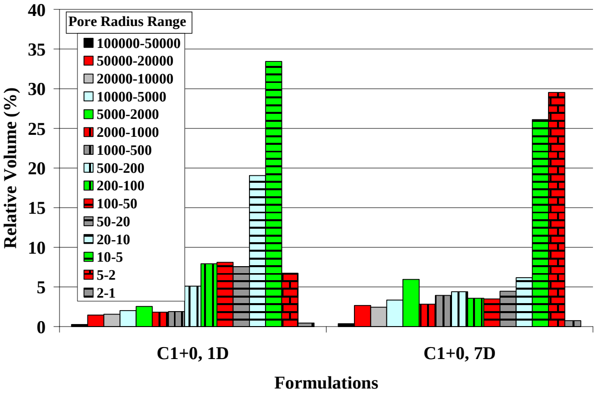

Fig 1(a). Pure Self Compacting Paste Sample

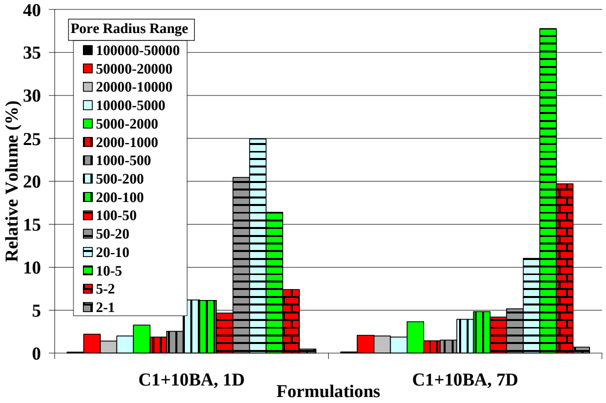

Fig 1(b). Self Compacting Paste sample containing 10% Bagasse Ash in replacement mode

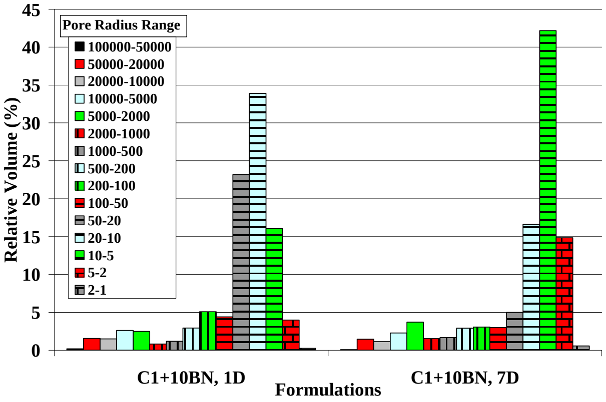

Fig 1(c). Self Compacting Paste sample containing 10% Bentonite in replacement mode

Fig 1. Pore Size Distribution (nM) of SCP formulations by MIP

Table 1. Typical Output Data Sheet of Pascal 440 Mercury Intrusion Porosimeter – Part 1

Instrument type : Pascal 440	
Sample name : CEM I-1 Day	
Filename : C:\PASCAL Data\Proben\CEM I-1Tag-kombi.440	
Date : 15.01.2010	
Sample information	
Company Name	TU Bergakademie Freiberg
Operator	Querner
Date	16.12.2009
Sample name	CEM I-1Day
Preparation	Bilal
Sample mass (g)	1,565
Sample density (g)	0
Mercury height (mm)	93,218
Vol. at run (mm3)	461
Weight dilatometer+mercury+sample (g)	166,98
Weight dil.+mercury+sample correct(g)	167,305
Capillary mercury height (mm)	65,218
Acquisition parameters	
Max pressure	400
Pump speed increase	7
Pump speed decrease	9
Pump speed inc. compensation	Pascal
Pump speed dec. compensation	Pascal
Dilatometer number	1
Dilatometer type	Normal
Calculation parameters	
Contact angle (Deg)	140
Hg surface tension (Dyne/cm)	480
Hg density (g/cm3)	13,5463
Temperature Hg (°C)	20
Pores model	Cylindrical
Sample volume correction	Yes
Region start	0
Region stop	100000
Region number of intervals	50
Pores average mode	Range
Particle size distribution	Yes
Kp factor	11,53

Cut off radius	1-10000
Blank parameters	
Blank data file path	C:\PASCAL Data\Blank\94-1-kombi.440
Blank name instrument	Pascal 440
Num. of blank data points increase	181
Num. of blank data points decrease	149
Filling volume (mm3)	485 (from blank file)
Weight dilatometer (g)	59,62 (from blank file)
Weight dilatometer+Hg (g)	175,95 (from blank file)
Capillary radius (mm)	1,5 (from blank file)
Dilatometer cone height (mm)	28 (from blank file)
Hg density blank (g/cm3)	13,5463 (from blank file)
Temperature Hg (°C)	20
Report options	
Report unit pores radius	Nanometer
Report unit Spec./Rel. volume	cc/g
Line factor	1
RESULTS	
Total cumulative volume (cc/g)	0.070100002
Total specific surface area (m²/g)	12.06900024
Average pore radius (Nanometer)	8.692527771
Total porosity (%)	14.55459976
Bulk density (g/cm3)	2.076390028
Apparent density (g/cm3)	2.430079937
Cut-Off radius range (Nanometer)	1-10000
Total pore volume (cc/g)	0
Pore specific surface area (m²/g)	0
Cumulative volume [Cut-Off] (cc/g)	0.070100002
Surface area [Cut-Off] (m²/g)	12.06900024
Sample volume correction	0.912232399

Table 2. Typical Output Data Sheet of Pascal 440 Mercury Intrusion Porosimeter – Part 2

PORE SIZE DISTRIBUTION (custom ranges)					
Pore radius ranges	Specific Volume	Relative Volume	Relative Volume	Specific Surface	Relative Surface
(nM)	(cc/g)	(cc/g)	(%)	(m²/g)	(m²/g)
100000-50000	0.00019	0.00019	0.27	0.0	0.0
50000-20000	0.00121	0.00102	1.46	0.0001	0.0001
20000-10000	0.00230	0.00109	1.55	0.0002	0.0002
10000-5000	0.00371	0.00141	2.01	0.0007	0.0004
5000-2000	0.00550	0.00179	2.55	0.0019	0.0012

2000-1000	0.00677	0.00128	1.82	0.0036	0.0017
1000-500	0.00812	0.00134	1.91	0.0075	0.004
500-200	0.01169	0.00358	5.1	0.0325	0.025
200-100	0.01725	0.00556	7.93	0.1151	0.0825
100-50	0.02294	0.00569	8.11	0.2761	0.161
50-20	0.02824	0.00530	7.57	0.6209	0.3448
20-10	0.04160	0.01335	19.05	2.7421	2.1212
10-5	0.06505	0.02345	33.45	9.1398	6.3977
5-2	0.06978	0.00473	6.75	11.738	2.5982
2-1	0.07010	0.00032	0.46	12.0687	0.3307

Table 3. Typical Output Data Sheet of Pascal 440 Mercury Intrusion Porosimeter – Part 3

Particle Size Distribution				
Particle diameter range	Cum. Distr. Vol.	Rel. Distr. Vol.	Cumulative Area	Relative Area
(nM)	(%)	(%)	(m²/g)	(m²/g)
10000-1000	16.78	16.78	0.1405	0.1405
1000-100	59.94	43.17	4.0983	3.9578
100-10	92.65	32.7	12.0639	7.9657
10-0	92.65	0.0	12.0639	0.0

Table 4. Typical Output Data Sheet of Pascal 440 Mercury Intrusion Porosimeter – Part 4

Data Report							
Pressure	Volume	Radius	Spec.Vol.	Rel.Vol.		Spec.Surf.	Rel.Surf.
(MPa)	(mm3)	(nM)	(cc/g)	(cc/g)	(%)	(m²/g)	(m²/g)
0.0130	0.0	56724.461	0.00000	0.00000	0.00	0.0000	0.0000
0.0142	0.3	51939.980	0.00019	0.00019	0.27	0.0000	0.0000
0.0160	0.4	46087.164	0.00026	0.00006	0.09	0.0000	0.0000
0.0166	0.5	44422.176	0.00032	0.00006	0.09	0.0000	0.0000
0.0183	0.6	40289.570	0.00038	0.00006	0.09	0.0000	0.0000
0.0202	0.7	36494.586	0.00045	0.00006	0.09	0.0000	0.0000
0.0208	0.8	35272.660	0.00051	0.00006	0.09	0.0000	0.0000
0.0216	0.9	33972.250	0.00058	0.00006	0.09	0.0000	0.0000
0.0228	1.0	32190.637	0.00064	0.00006	0.09	0.0000	0.0000
0.0259	1.1	28346.523	0.00070	0.00006	0.09	0.0000	0.0000
0.0267	1.4	27504.498	0.00089	0.00019	0.27	0.0000	0.0000
0.0279	1.5	26324.910	0.00096	0.00006	0.09	0.0001	0.0000
Continued	continued	continued	continued	continued	continued	continued	continued

Table 5. Threshold Radius and Maximum Pore Radius from Cumulative MIP Diagram

Properties	**Self Compacting Paste Formulations**					
	C1+0, 1D	C1+0, 7D	C1+10BA, 1D	C1+10BA, 7D	C1+10BN, 1D	C1+10BN, 7D
Threshold Radius (nM)	15.346	10.641	38.6	14.47	29.18	13.01
Maximum Pore Radius (nM)	56724.5	57353.7	57012.3	56166.3	55570.5	53849.0

Table 6. Pore Size Distribution by MIP

Pore Size Distribution of Self Compacting Paste Formulations						
Pore Radius Ranges (nm)	**Relative Volume Intruded (%)**					
	C1+0, 1D	C1+0, 7D	C1+10BA, 1D	C1+10BA, 7D	C1+10BN, 1D	C1+10BN, 7D
100000-50000	0.27	0.37	0.13	0.14	0.19	0.08
50000-20000	1.46	2.68	2.2	2.07	1.55	1.45
20000-10000	1.55	2.45	1.4	2.0	1.49	1.13
10000-5000	2.01	3.35	2.0	1.86	2.6	2.26
5000-2000	2.55	5.95	3.26	3.65	2.48	3.71
2000-1000	1.82	2.83	1.87	1.45	0.81	1.53
1000-500	1.91	3.94	2.53	1.52	1.18	1.69
500-200	5.1	4.39	6.2	3.93	2.91	2.9
200-100	7.93	3.57	6.13	4.82	5.08	3.06
100-50	8.11	3.49	4.66	4.2	4.4	2.98
50-20	7.57	4.46	20.45	5.17	23.17	5.0
20-10	19.05	6.17	24.92	11.03	33.89	16.61
10-5	33.45	26.1	16.39	37.77	16.05	42.18
5-2	6.75	29.52	7.4	19.71	3.97	14.84
2-1	0.46	0.74	0.47	0.69	0.25	0.56

ANNEXURE E – X RAY DIFFRACTION RESULTS

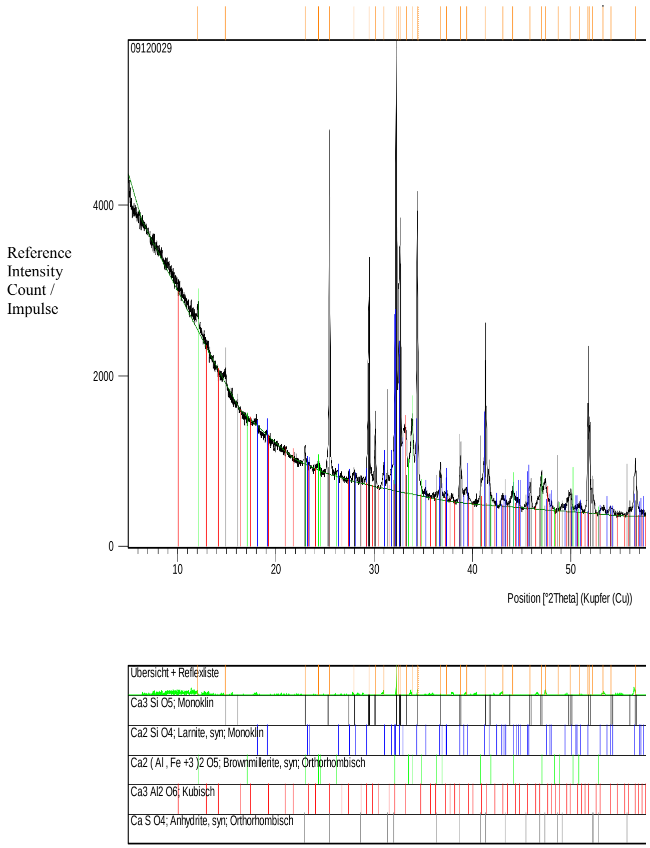

Fig 1. XRD analysis of Faugi Cement Particles

Main Phases – Alite (60%), Belite (17%), Ferrite (11%), Aluminate (7%), and Anhydrite (4%)

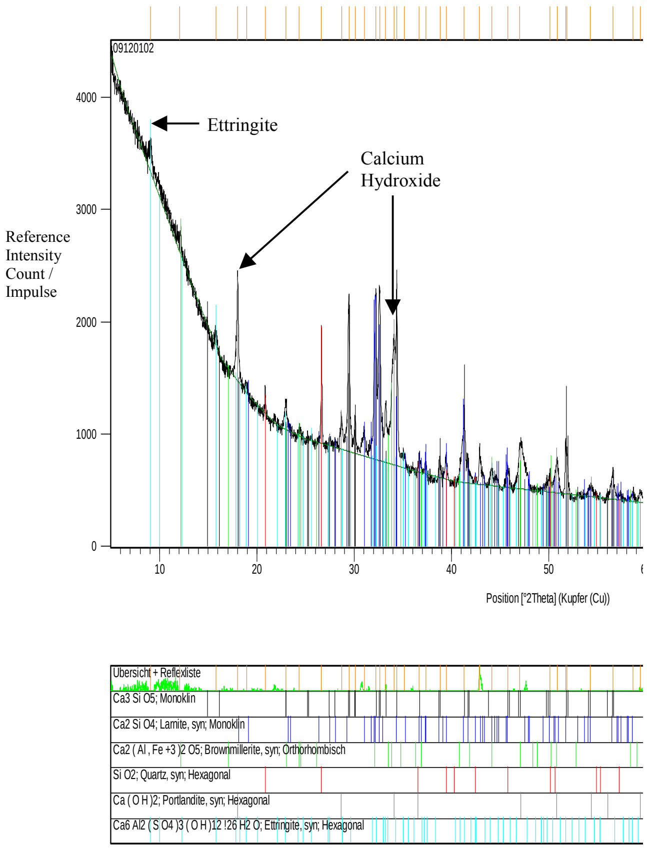

Fig 2. XRD analysis of Pure Self Compacting Paste Sample at an age of 1 Day

Main Phases – Alite, Belite, Ferrite, Quartz, Portlandite and Ettringite

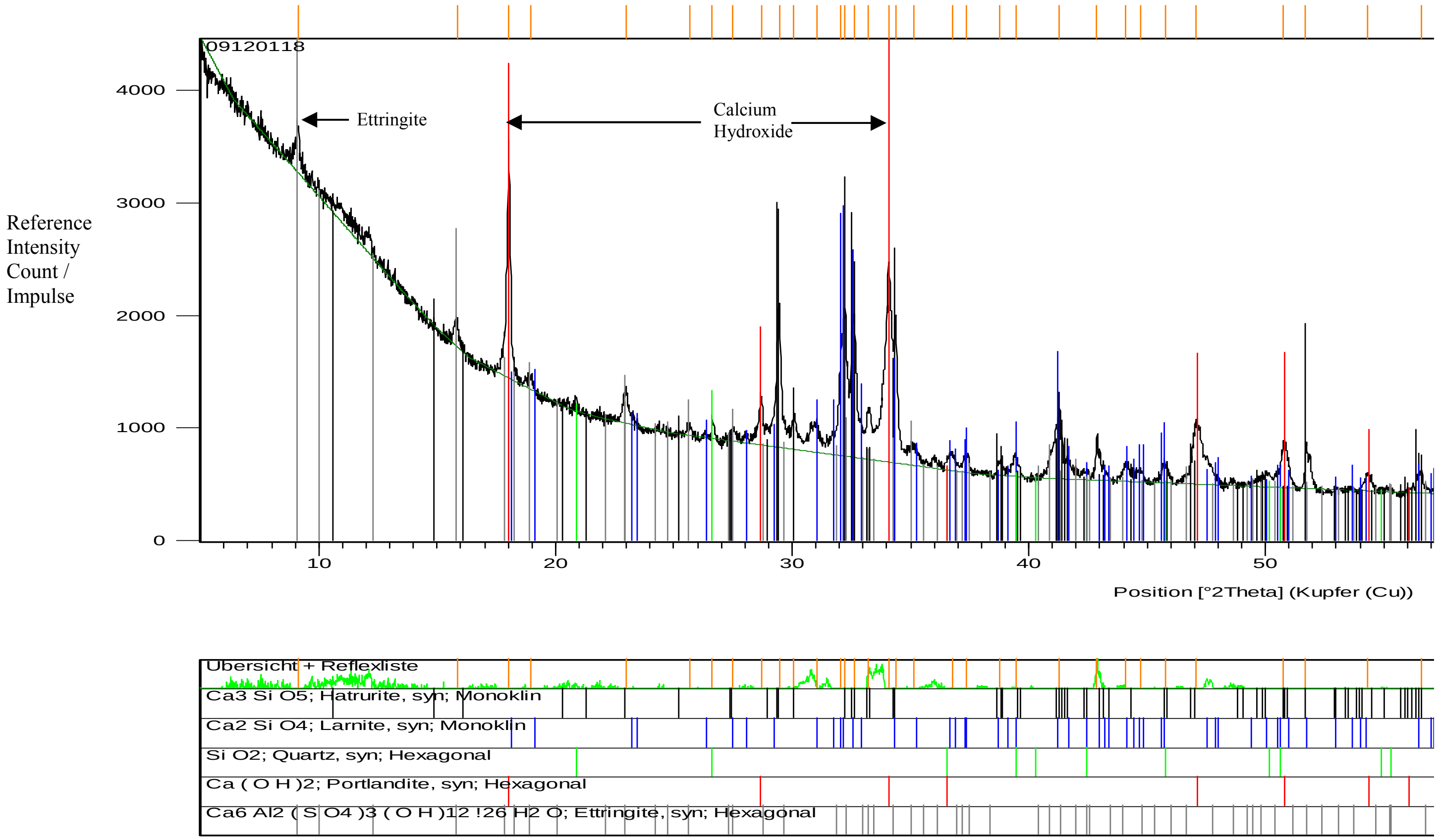

Fig 3. XRD analysis of Pure Self Compacting Paste Sample at an age of 7 Day

Main Phases – Alite, Belite, Ferrite, Quartz, Portlandite and Ettringite

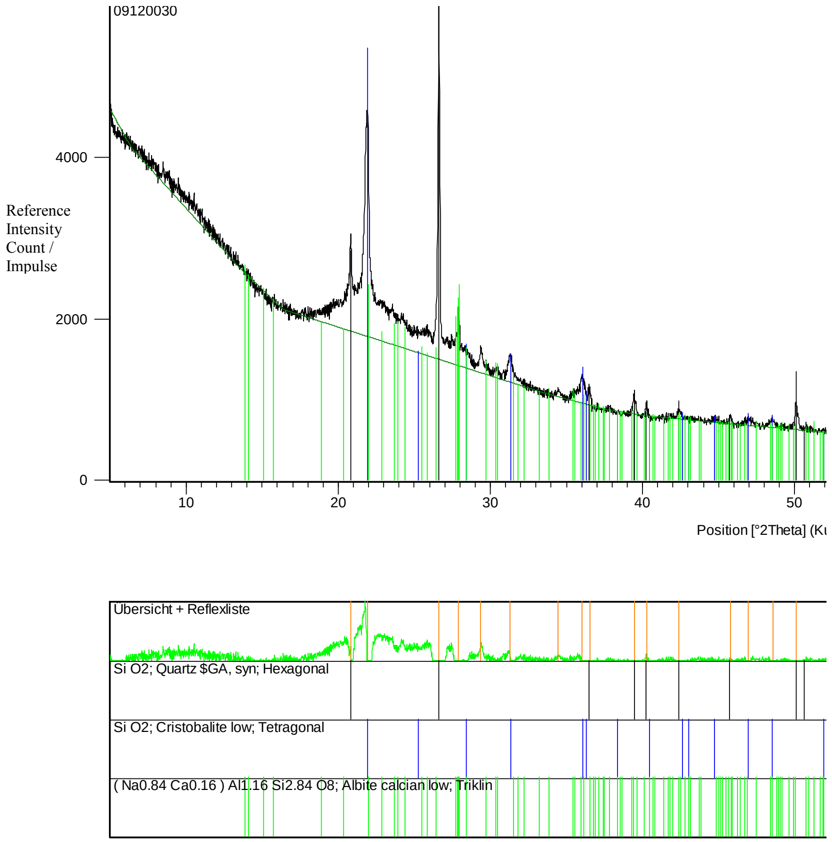

Fig 4. XRD analysis of Bagasse Ash Particles

Main Phases – Quartz, Cristobalite and feldspar (Albite)

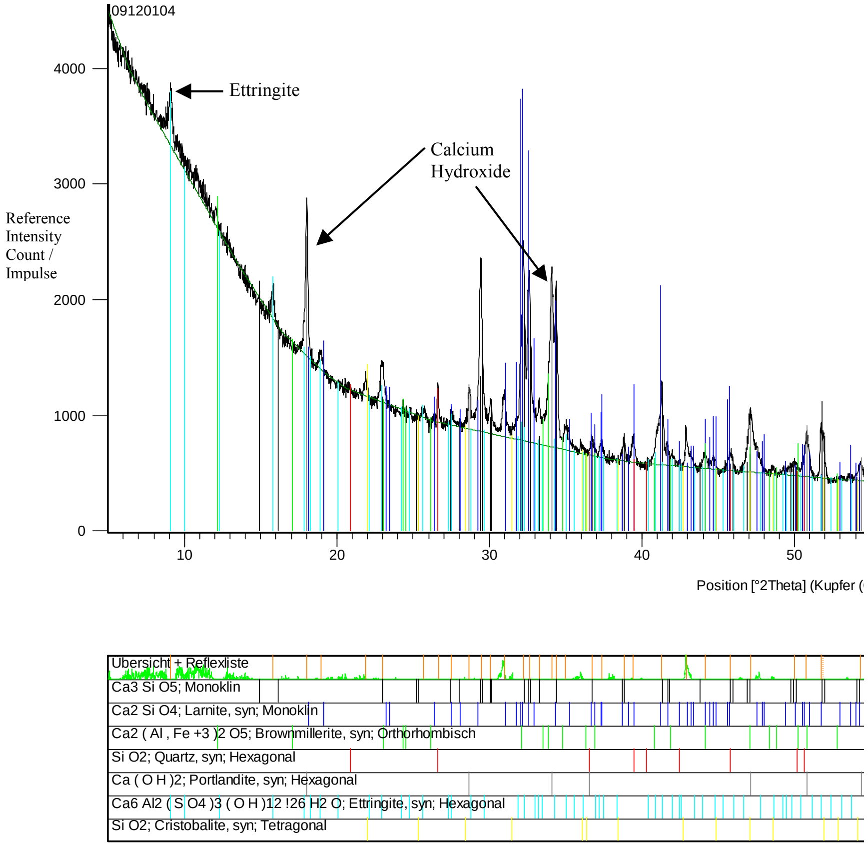

Fig 5. XRD analysis of SCP sample containing 10% Bagasse Ash in replacement mode at an age of 1 Day

Main Phases – Alite, Belite, Ferrite, Quartz, Portlandite, Ettringite and Cristobalite

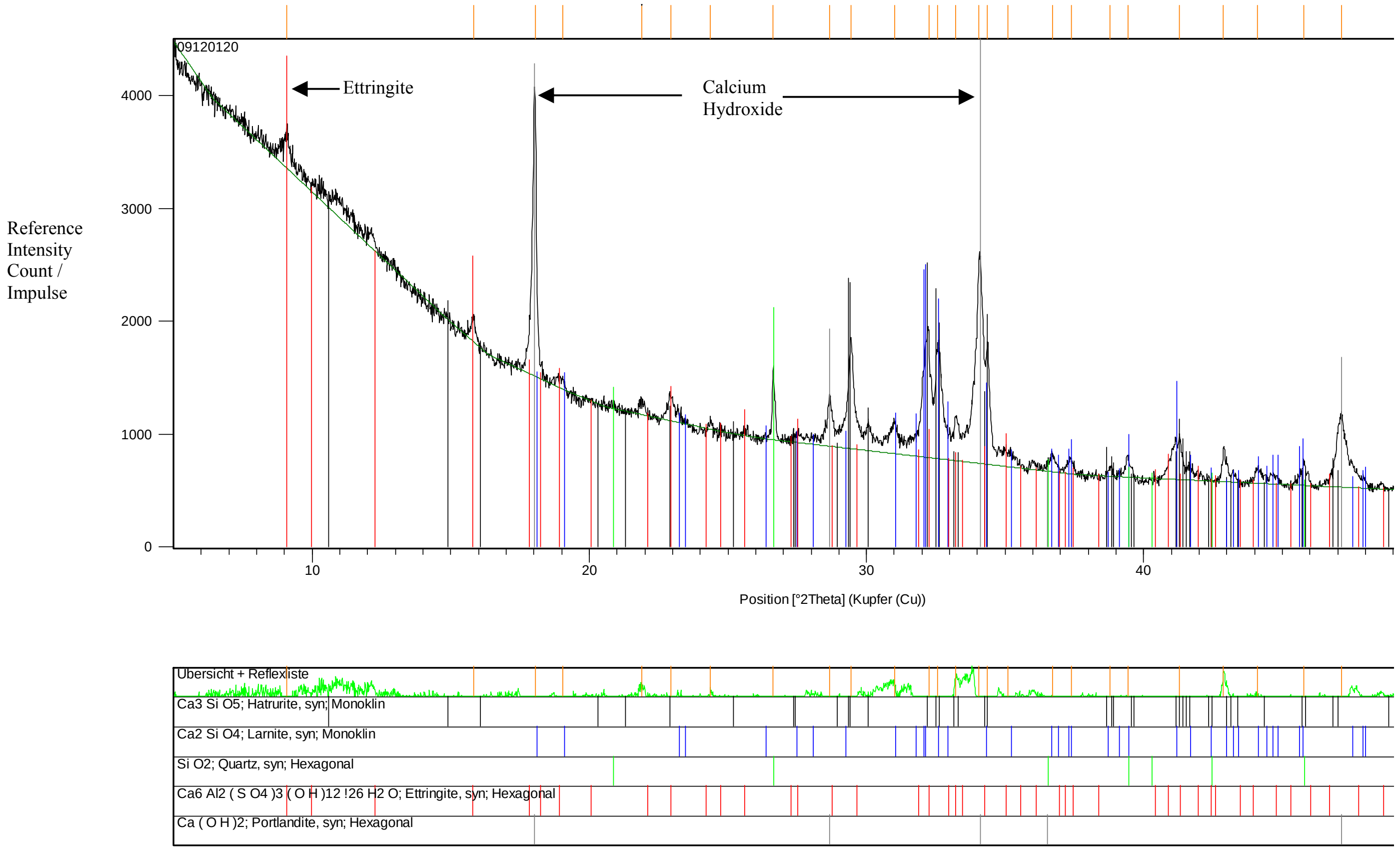

Fig 6. XRD analysis of SCP sample containing 10% Bagasse Ash in replacement mode at an age of 7 Day

Main Phases – Alite, Belite, Ferrite, Quartz, Portlandite, Ettringite and Cristobalite

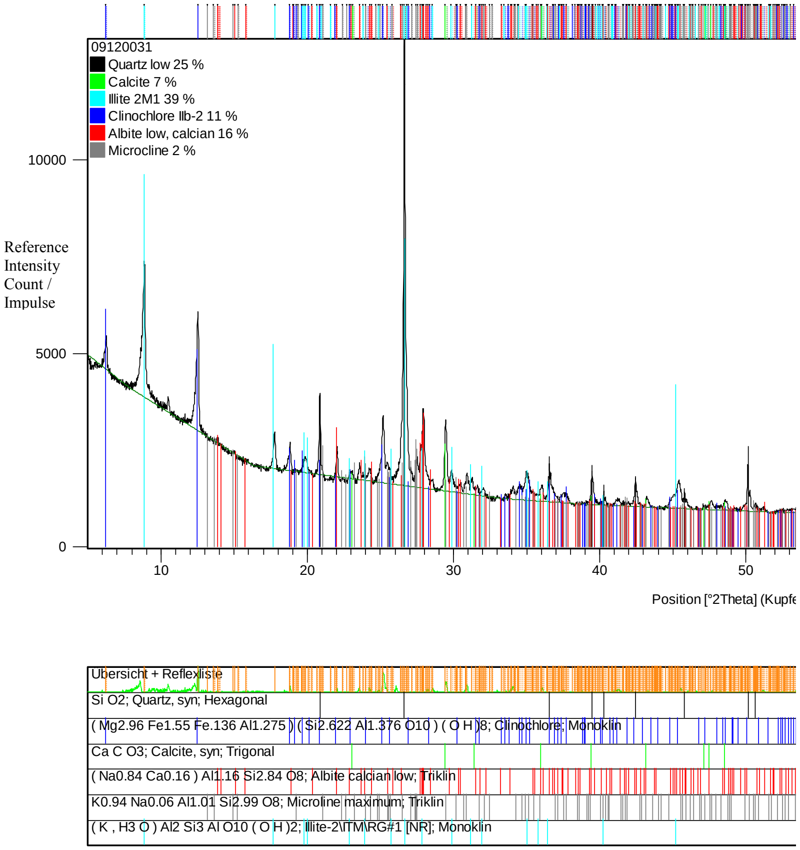

Fig 7. XRD analysis of Bentonite Particles

Main Phases – Quartz(25%), Calcite(5%), clay minerals(50%) (39% illite, 11% Clinochlore) and feldspar(18%) (16% Albile, 2% Microcline)

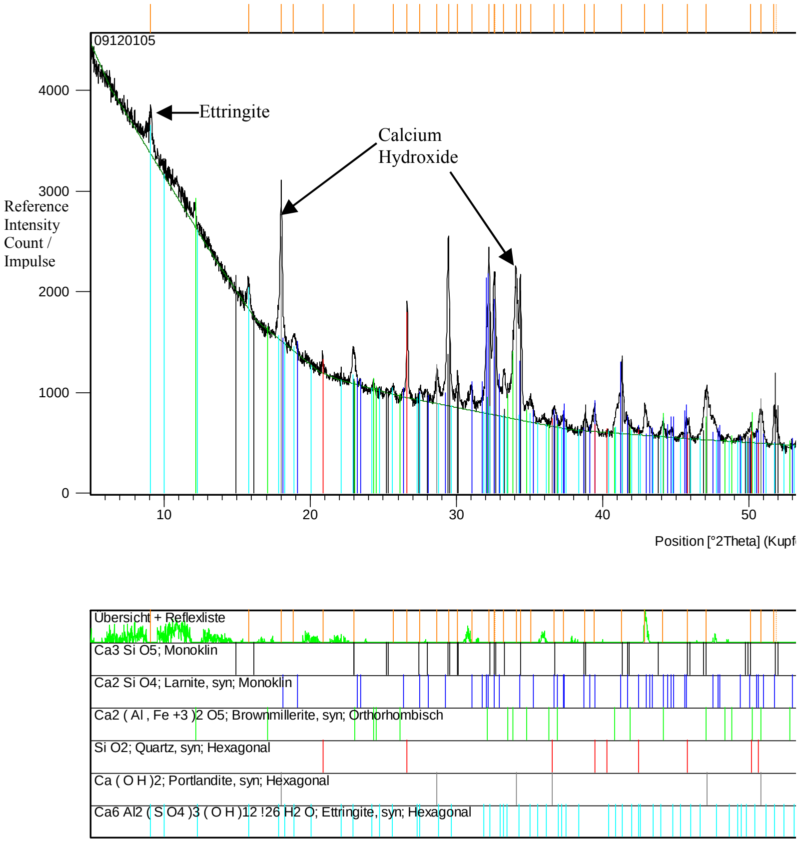

Fig 8. XRD analysis of SCP sample containing 10% Bentonite in replacement mode at an age of 1 Day

Main Phases – Alite, Belite, Ferrite, Quartz, Portlandite and Ettringite

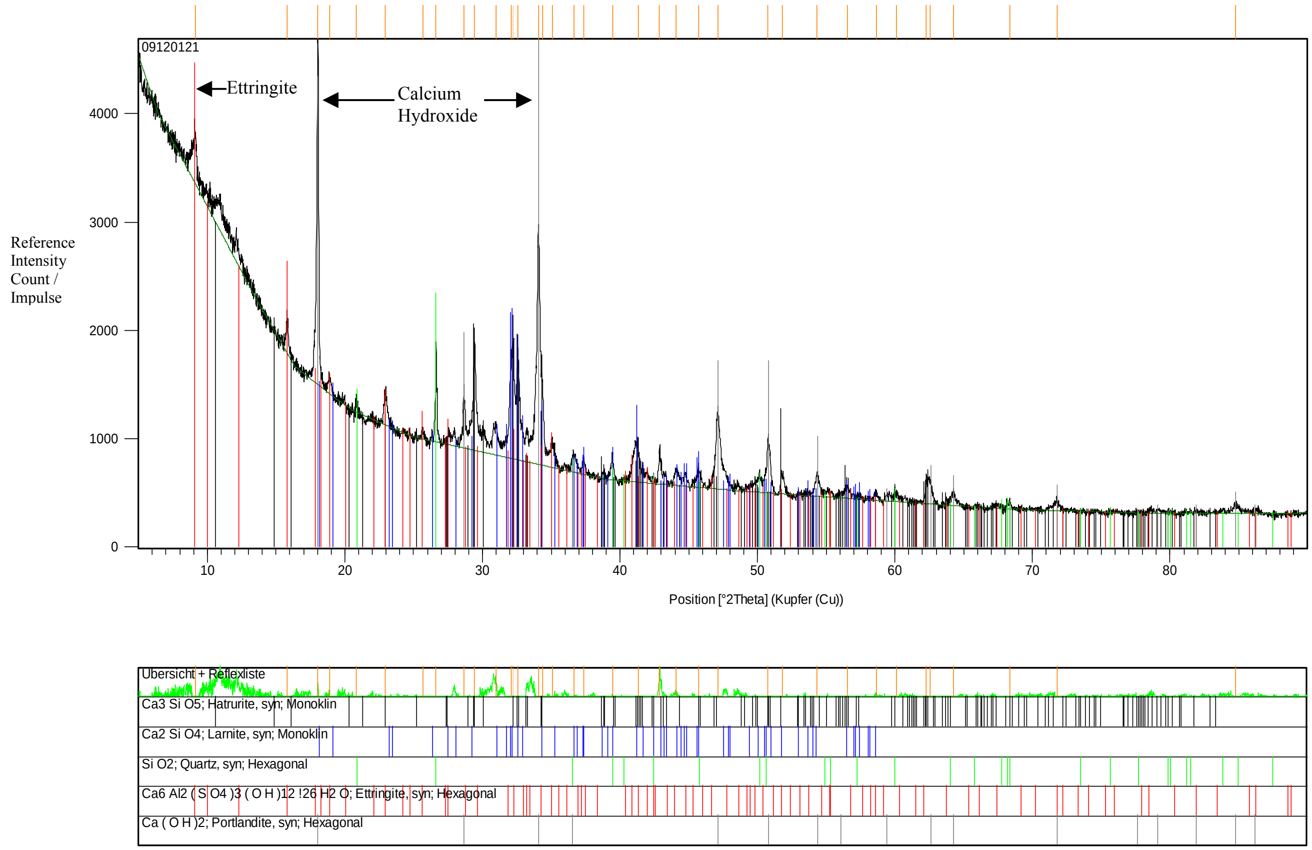

Fig 9. XRD analysis of SCP sample containing 10% Bentonite in replacement mode at an age of 7 Day

Main Phases – Alite, Belite, Ferrite, Quartz, Portlandite and Ettringite