A STUDY OF SELF-COMPACTING PASTE SYSTEMS USING ACACIA MODESTA GUM AS AN ADMIXTURE

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All praise be to Allah Almighty alone

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Last but not the least; I would like to thank my parents for their prayers, unceasing support and substantial encouragement through the venture. I could not have imagined finishing the work without their affection.

Dedication

I dedicate this research to

Prof. Dr. Syed Ali Rizwan, my mentor

And

To my parents

A STUDY OF SELF-COMPACTING PASTE SYSTEMS (SCPs) CONTAINING ACACIA MODESTA GUM AS AN ADMIXTURE

ABSTRACT

This study presents response of self-compacting paste systems containing botanical Acacia Modesta (AM) gum, a natural organic ooze-out of an indigenous tree called *Phulai*, as an admixture.

AM gum's crystallography was performed through X-ray Diffraction (XRD). It was chemically characterized through X-ray Fluorescence (XRF), Nuclear Magnetic Resonance (NMR) and Fourier Transform Infrared Spectroscopy (FTIR). The behavior of AM gum in water and varying temperatures was also tested. These characterization tests showed that AM gum is composed mainly of hydrophobic components and therefore it behaves as a *nearly* inert material in self-compacting cementitious systems.

The Self-compacting paste systems (SCPs) containing 0.25 %, 0.5 %, 0.75 % and 1 % powdered gum of total dry cement weight were tested for super-plasticizer demand, flow, viscosity and air voids in fresh state and for density, total shrinkage, calorimetry and 90-day compressive strength. The results showed a decrease in total shrinkage by 41 %, a reduction in density by 21 %, an increase in viscosity and a progressive delay in setting time of the respective gum based SCPs. Moreover, the gum seemed to behave as an internal curing agent. The incorporation of AM gum therefore exhibited several potential fields of application in self-compacting cementitious systems. This research offers potential applications in thermally efficient freeze-thaw resistant light weight concrete constructions.

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LIST OF NOTATIONS\ABBREVIATIONS

ACI	American Concrete Institution
AH	Anhydrite
AM	Acacia Modesta
ASTM	American Society of Testing Of Materials
SCP	Self-Compacting Paste
SCM	Self-Compacting Mortar
SCC	Self-Compacting Concrete
XRD	X-ray diffraction
XRF	X-ray Fluorescence
SP	Super plasticizer
SRM	Secondary Raw Material
W/C	Water to Cement Ratio
SCCS	Self-Compacting Cementitious Systems
PC	Portland Cement

CHAPTER 1

INTRODUCTION

1.1 Problem Statement

The objective of current research was to explore response of self-compacting paste systems (SCPs) containing Acacia Modesta gum, a species of genus Acacia, as an admixture and present its potential fields of applications in concrete.

1.2 Research Significance

Little published work on the use of various Acacia gum types in self-compacting cementitious systems exists in literature. While the response of gums emanating from Acacia Senegal and Acacia Seyal in conventional cementitious systems has been reported in a few papers, these nascent works have not shown the differences in response of these gums or their use in modern self-compacting paste (SCP) systems with various characterization techniques. This novel research reports the effects of Acacia Modesta gum's incorporation on the response of SCP systems. It also provides a small comparison between responses of Acacia Modesta and Acacia Nilotica gums when used as admixtures in SCPs. This research is likely to find applications in thermally efficient freeze-thaw resistant light weight concrete constructions.

1.3 Research Methodology

The research was started with the conduction of relevant literature review. Having gained a deeper insight into the topic in question, some elementary tests were conducted on AM gum to see how this gum behaves in moisture and varying temperature conditions. Next characterization tests such as XRD, XRF, NMR and FTIR were conducted to find out the type of organic compounds that are present in the Acacia Modesta gum. These tests were conducted to investigate the likely response of Acacia Modesta gum in self-compacting paste (SCP) systems. After these tests, the main laboratory tests were carried out. These included tests for flow, setting times, density, viscosity, total shrinkage, calorimetry, air voids in fresh formulations and the compressive strengths. Finally detailed discussions with logical reasoning in light of relevant literature review and supervisor's and researcher's own knowledge and understanding about the subject are presented to understand the behavior of AM gum in SCPs.

1.4 Research scope

This research was conducted on self-compacting paste (SCPs) systems. The purpose of current research was to establish an approach into the analysis of AM gum based cementitious systems. The results produced in this study can be used as basis for future researches on concrete.

1.5 Thesis organization

The first chapter gives an introduction to the thesis topic. The next chapter presents the literature review. The third chapter presents various standard methods and apparatuses employed for conduction of laboratory tests. The fourth chapter presents discussions and reasoning. The fifth chapter gives conclusions and recommendations.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction to Self-Compacting Cementitious Systems (SCCS)

Self-compacting concrete is a kind of concrete that deforms easily and has a high resistance to segregation. As per ACI committee 237R-07 [1]

Self-compacting concrete (SCC) is a highly flowable, non-segregating concrete that can spread into place, fill formwork and encapsulate the reinforcement without any mechanical consolidation

According to the European Guidelines for Self-Compacting Concrete, this kind of concrete has the same durability and engineering characteristics as conventional vibrated concrete. The concept was first introduced in the late 1980s in Japan and has since then rapidly spread in almost all European countries. Self-compacting cementitious systems may be broadly classified into the following systems

- Self-compacting paste systems
- Self-compacting mortar systems
- Self-compacting concrete systems

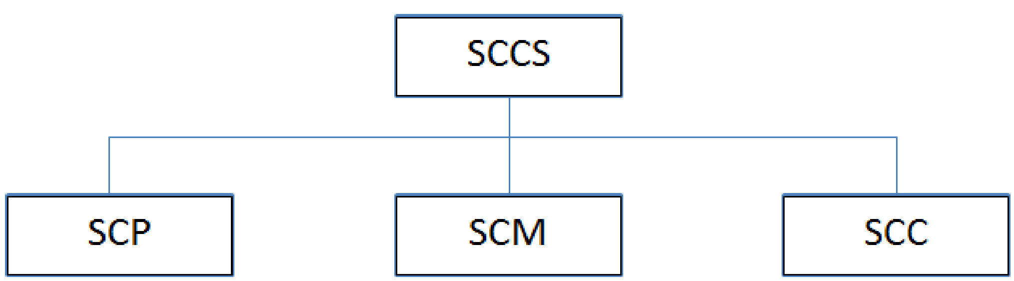


Fig 1.1 Schematic of SCCS

2.2 Self-compacting Paste Systems

Self-compacting paste systems serve as a fixative between the self-compacting mortar (SCM) and self-compacting concrete (SCC) systems and play a vital role in determining the over-all properties of a system. Therefore the addition of different admixtures to a paste could greatly improve the durability and service life of that system through reduction of maximum pore diameter [5] and formation of chemical bonds.

2.3 Admixtures

An admixture can be defined as a material added in small quantities to improve the properties of concrete, mortar or paste systems [2]. Acacia Modesta Gum has been used as an admixture for modifying certain properties of self-compacted paste systems in current research especially to reduce early-age shrinkage and to minimize the need for curing. The mineral admixtures ensure the following properties in self-compacting cementitious systems: Increased workability, reduced water-cement ratio (w/c ratio), increased durability of the system through effective particle packing and decreased permeability and economical solution to the mix design through minimum use of cement [3]. Chemical admixtures on the other hand ensure the following properties; prolonged initial and final setting times, increased segregation resistance during transportation and placing of concrete and enhanced fluidity of a SCC system for the same w/c ratio. The basic criteria for achieving self-compaction are high deformability, high passing ability and high resistance to segregation. A super-plasticizer may be used for achieving high flow and a viscosity enhancing agent may be used in the mix with low or moderate water-powder ratio to obtain higher resistance to segregation [4]

2.4 Role of Admixtures in SCCS

Secondary Raw Materials (SRMs) and various admixtures are often used by many researchers in self-compacting cementitious systems to achieve enhanced strength, economy, durability and environment friendliness [6].

2.5 Acacia Modesta (AM) Gum

Acacia Modesta is found mainly in the tropical countries [9]. In Pakistan it is found in the northern areas. Its gum emanates from the mature trees of Acacia Modesta that are injured and exists in the form of non-viscous liquid which is rich in soluble fibers and composed mainly of carbohydrates and proteins [6].

Its use dates back to the second millennium BC when Egyptians used it as an ink and adhesive. Today, owing to its widely known uses, the gum has found its way to textile, ceramics, cosmetics and food industry etc. It is used as an emulsifying agent in foods such as soft drink syrup, creams and gummy candies etc. Only very recently researchers have discovered about the antioxidants properties of the Acacia Gums and its positive role in the digestion of lipids and treatment for various degenerative diseases [7].

2.6 Composition of Acacia Modesta Gum

Different types of Acacia gums are found all over the world. While they bear some differences, the compounds present in these gums are more or less the same though in different percentages [9]. Acacia gum mainly consists of sugars such as rhamnose, arabinose and galactose [8]. Studies have shown that arabinose, glucose and galactose are polyhydroxy aldehydes [10]. Polyhydroxy compounds (sugars) readily form complexes with silicates (also present in the cement paste) in a basic medium of having pH greater than or equal to 10.5 and at a room temperature [11], a few polyhydroxy compounds (sugars) are unable to react with silicates owing to their specific stereo-composition. In 2004 Lambert and co-researchers examined sugar silicates in detail and reported how a few monosaccharides such as arabinose, glucose, mannose and galactose could not form sugar silicate complexes [8]. Since AM gum is likely to contain of monosaccharides as primary constituents, it may be reasonably assumed that these sugars present in the gum render it unreactive in the cement paste and may cause it to behave as a nearly inert material in cementitious systems. This could be one of the reasons for increase in setting times when AM gum is incorporated in SCP systems.

2.7 Super Plasticizers

A super plasticizer may decrease the water demand by as much as 30 % without causing excessive retardation in setting times. Its uses are summarized as follows:

- Establishes a concrete of desirable workability at a lower water-cementitious material (w/cm) ratio
- Produces highly flowable concrete with self-leveling properties at a lower water-cementitious material (w/cm) ratio. This ensures easy placement and consolidation of concrete.

Depending on their properties, HRWR admixtures may be classified as Type F or Type G high-range water reducing agents or they may fall in the Type 1 or Type 2 category of ASTM C 1017 when used to enhance flow-ability of concrete. Their classification is done based on the following three ingredients; sulfonated melamine-formaldehyde condensate, sulfonated naphthalene-formaldehyde condensate and polyether poly-carboxylates

These admixtures are more efficient than conventional water-reducing admixtures since these ensure more effective dispersion of fine materials such as cement and various SRMs [12]

2.4 Melflux

Meflux is a third generation high-performance super-plasticizer used in cement based mixes. For current research, Meflux 2651F manufactured by BASF Germany has been used. It is poly-carboxylate ether (PCE) powder optimized for use as a plasticizer and water reducing agent and ensures development of high early strength. When used in apposite proportions, it helps in prevention of bleeding and segregation.

2.5 Bingham Model for Cement Paste

In scientific parlance, rheology is defined as the “the distortion and flow of matter when shear stress is applied”.

In literature every material is studied on the basis of some model. Likewise for cement paste, Bingham model has been established to explain its rheological properties. The model is based on the following two rheological parameters

- Yield Stress
- Viscosity

Bingham’s model effectively explains the inter-particle forces that are present in the concentrated suspensions of cement paste and concrete.

Cement paste is a non-Newtonian fluid. Its viscosity decreases on application of shear stress and increases when the stress is removed. The reason could be attributed to Brownian motion which occurs once the paste is stationary. The particles collide and move to more favorable positions. This results in their attachment to various parts and causes temporary increase in the viscosity [15].This phenomenon is known as "thixotropy" and is a time-dependent process [14]. The process is both reversible and isothermal [16]. Once shear stress is applied, the fluid regains its flowing ability. This process is known as shear thinning and the temporary loss in viscosity is known as pseudo-plasticity [17].

Measurement of thixotropy in case of cement based mix however is not so simple. The pseudo-plasticity is dependent on time as well. This is due to the hydration process which starts as soon as the water comes in contact with the cement. The process of shear thinning remains dominant till a certain time period after which the process of gaining viscosity becomes irreversible [18, 19].

Thixotropic behavior of paste systems may be explained in terms of coagulation, dispersion and re-coagulation of the particles. Coagulation happens when two or more cement particles stick to each other due to the potential energy between them and are separated only when work is done on them [20].

2.6 Workability

Workability, which is the property of a mix in its fresh state, may be defined as the ease of placement. It is determined through slump cone test using Hagerman's cone. The procedure is described in detail in Chapter 3.

2.7 Shrinkage

All types of concrete shrink. Shrinkage begins as soon as the water comes in contact with the cement [21]. The most common types of shrinkage include; plastic shrinkage, drying shrinkage, autogenous shrinkage and carbonation shrinkage. Plastic shrinkage occurs while the concrete is still in its plastic form, drying shrinkage occurs due to the loss of water contained in the gel pores. Autogenous shrinkage has been defined as "the bulk strain of a closed, isothermal, cementitious material system not subjected to external forces" [21]. Carbonation shrinkage results from the reaction of CO_2 with the hydrated cement in the presence of moisture. Another type of shrinkage, known as chemical shrinkage, has also been defined in the literature. It is “the volume reduction associated with the hydration reactions in a cementitious material” [21].

2.8 Calorimetry

Calorimetry refers to the process of measuring the heat of chemical reactions or physical changes associated with a certain phenomenon. In this case, it measures the heat changes occurring due to the hydration process.

2.9 Curing

Curing is the process of maintaining sufficient moisture content, which is not part of the mixing water, in a concrete mix at early ages. This helps in achievement of the desirable cementitious properties and formation of quality products through a process called hydration [22]. Curing helps in strength gain, durability, serviceability and resistance to wear and abrasion [22].

2.9.1 Internal Curing

Conventional curing methods ensure that the mixture stays moist and warm at the surface. This makes possible continued and complete hydration of a typical cement mix having w/c ratio more than 0.42 [23]. With the advent of high rise buildings and long spanned bridges, production of high strength concrete with limited w/c ratio is in high demand. Such mixtures have insufficient free water content which might result in self-desiccation of cement matrix. Therefore it is recommended to have a material that plays the role of internal reservoirs inside the matrix. The internal reservoirs may be inducted in the form of saturated lightweight fine aggregates, superabsorbent polymers, or saturated wood fibers. These ensure completion of the hydration process and prevent early age cracks. ‘Internal curing’ is often also referred as ‘Self–curing’.

CHAPTER 3

EXPERIMENTAL PROGRAM

This chapter discusses the materials used and the procedures adopted to carry out the experiments.

3.1 Research Materials

3.1.1 Cement Type

Bestway Ordinary Portland Cement (OPC), grade 53, type I conforming to ASTM standard C150-04, was used. It is manufactured in Pakistan and is locally available in Islamabad.

3.1.2 Acacia Modesta Gum

Acacia Modesta gum, found in the Northern Areas of Pakistan, was obtained from a local source in Rawalpindi in the form shown in fig. 3.1(a) and then grinded to form a fine powder.

Fig. 3.1 (a) ***AM gum in un-grinded form***

Fig 3.1 (b) ***Grinding AM gum***

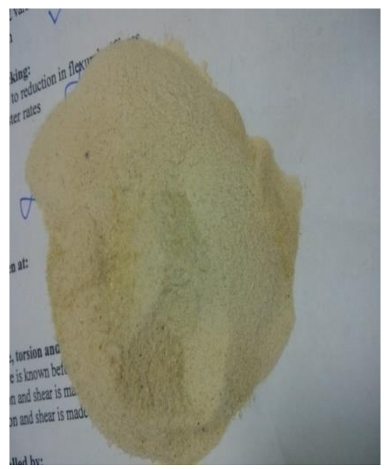


Fig 3.1 (c) ***AM gum in powdered form***

3.2 Formulation Types

Five SCP formulations containing varying amount of AM gum were prepared at their respective super plasticizer demands and constant water content i.e. 27 % by weight of total cement. The 27

% was taken since it is the water demand of neat cement paste. The formulations were prepared at their respective super plasticizer demands for the target flow of 30 ± 1 cm measured by Hagerman's cone measuring 6 x 7 x 10 cm^3. The formulations tested were as follows:

1. C1-SP0.175-AM0.0-27
2. C1-SP0.28-AM0.25-27
3. C1-SP0.35-AM0.50-27
4. C1-SP0.42-AM0.75-27
5. C1-SP0.52-AM1.00-27

A typical formulation of, for instance, C1-SP0.28-AG0.25-27 should be considered as having CEM 1 type cement, 0.28 % Super-plasticizer (SP) dose needed for target flow, 0.25 % Acacia Modesta (AM) gum and 27 % mixing water content at 25 ºC and 55 % relative humidity (RH) in the laboratory. All other formulations can be understood accordingly. All percentages in formulation nomenclature are with respect to the weight of cement.

The mixing was done in the Hobart mixer as per DIN-196-3. The dry contents were first mixed manually and then fed into the mixer bowl containing the mixing water. Slow mixing at 145 rpm was done for 30 seconds. Thereafter the interior of bowl as well as paddle was cleaned followed by fast mixing at 285 rpm for 150 seconds. Total mixing time was 180 seconds. Fifteen 4 x 4 x 16 cm^3 specimens were cast. Immediately after casting, the samples were covered with plastic sheet and de-molded after 5 days. Some of these were cured in air in the laboratory whereas others were water cured in the water tank. These were tested as per DIN 196 standards and different curing regimes gave different results.

3.3 Flow Measurements

The flow measurements were determined using Hagerman's mini-slump cone of 6 x 7 x 10 cm^3. The target flow was 30 ± 1 cm. The SP demand for each formulation was achieved through trials.

$$\text{Flow} = (D_1 + D_2)/2$$

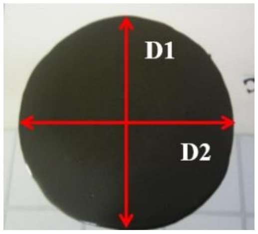


Fig 3.2 (a) Diameters in directions orthogonal to each other

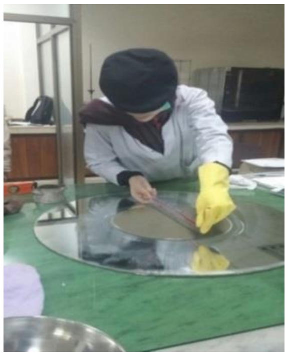


Fig 3.2 (b) D_1 measurement

Fig 3.2 (c) D_2 measurement

3.4 Rheometric Investigation apparatus

Brookfield DV-III Ultra Programmable Rheometer with SC4-27 spindle was used to measure viscosity of the control sample and the SCP samples containing 0.25 %, 0.5 %, 0.75 % and 1.0 % gum at 50 rpm (17 s^{-1}), 62 rpm (21 s^{-1}), 80 rpm (27 s^{-1}) and 100 rpm (34 s^{-1}) respectively. The highly viscous nature of formulations containing AM gum made it difficult for the spindle to rotate. Therefore the w/c ratio was increased to 0.40 with the corresponding SP content for all SCP formulations to facilitate the movement of spindle

3.5 Strength Tests

The formulations were tested for flexure and compression as per EN 196-1:1994 on the 7th, 14th and 28th day. Ten prisms of 4 x 4 x 16 cm dimensions were cast and the average values of the flexure and compressive strength results were taken to determine the strength of five different formulations.

3.6 Shrinkage Tests

German Schwindrine apparatus measuring 4 x 6 x 25 cm^3 was used to know the total shrinkage response of one control SCP sample and four AM gum based SCP formulations for the first 90 hours. The average laboratory temperature was 32 °C and the relative humidity was 55 %.

Fig. 3.3: German Schwindrine apparatus

3.7 Calorimetry

Calorimetry was conducted on F-CAL 8000 Calorimeter. The purpose was to analyze the effect of paste formulations with Acacia Modesta Gum on the heat released and on the reaction kinetics. Each formulation was prepared at constant water content and at its respective super plasticizer demand. All formulations were placed in the Calorimeter for 180 hours. Six out of eight channels were used. Having taken out the samples on 8th day, graphs were plotted on the software.

3.8 XRF Process

The test was conducted on 6490 (LA). The sample of Acacia Modesta Gum was prepared and fed into the machine. Fig. 3.4 (a) shows the XRF machine used and fig. 3.4 (b) illustrates the sample after the test was conducted.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Physical and Chemical Characterization of AM gum

The average particle size (D50) and particle size distribution (PSD) of Acacia Modesta gum was found using sieve analysis technique. Its specific gravity was determined using the approximate water displacement method. Fig 4 shows the PSD of Acacia gum and calculation of average particle size (D50).

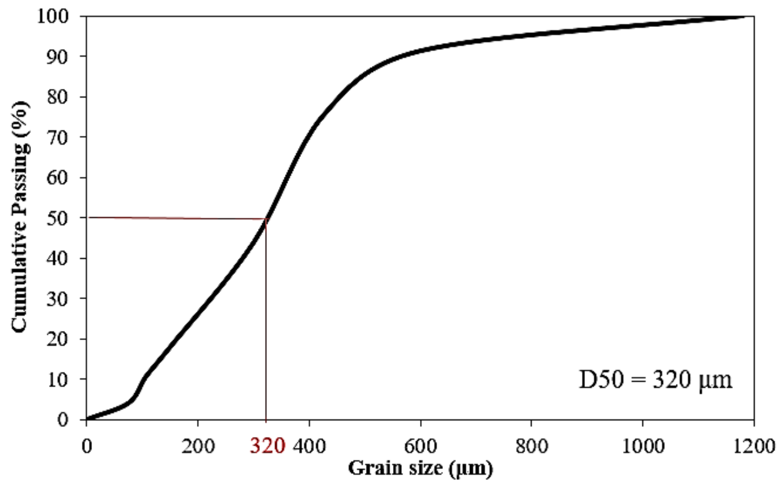


Fig 4: Particle Size Distribution of Powdered AM Gum

The physical properties of the AM gum are shown in Table 1

Table 1: Physical Properties of Powdered AM Gum

Physical Properties	
Average Particle Size	320 µm
pH (30 % AM gum soln.)	5.5
Specific gravity	1.5

XRD was performed on the powder form of AM gum using PANalytical - X'Pert³ Powder. The qualitative XRD of gum powder in Fig 4.1 shows that it is poorly crystalline. The values of 2θ ranged from 10 to 60 degrees in XRD test.

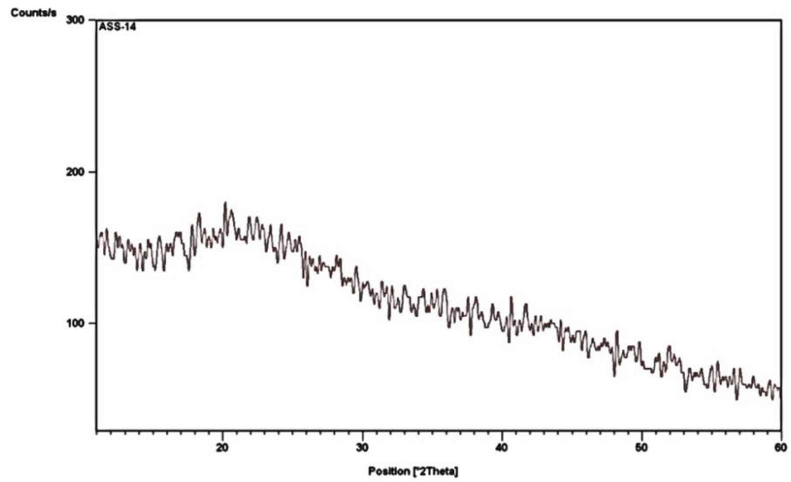
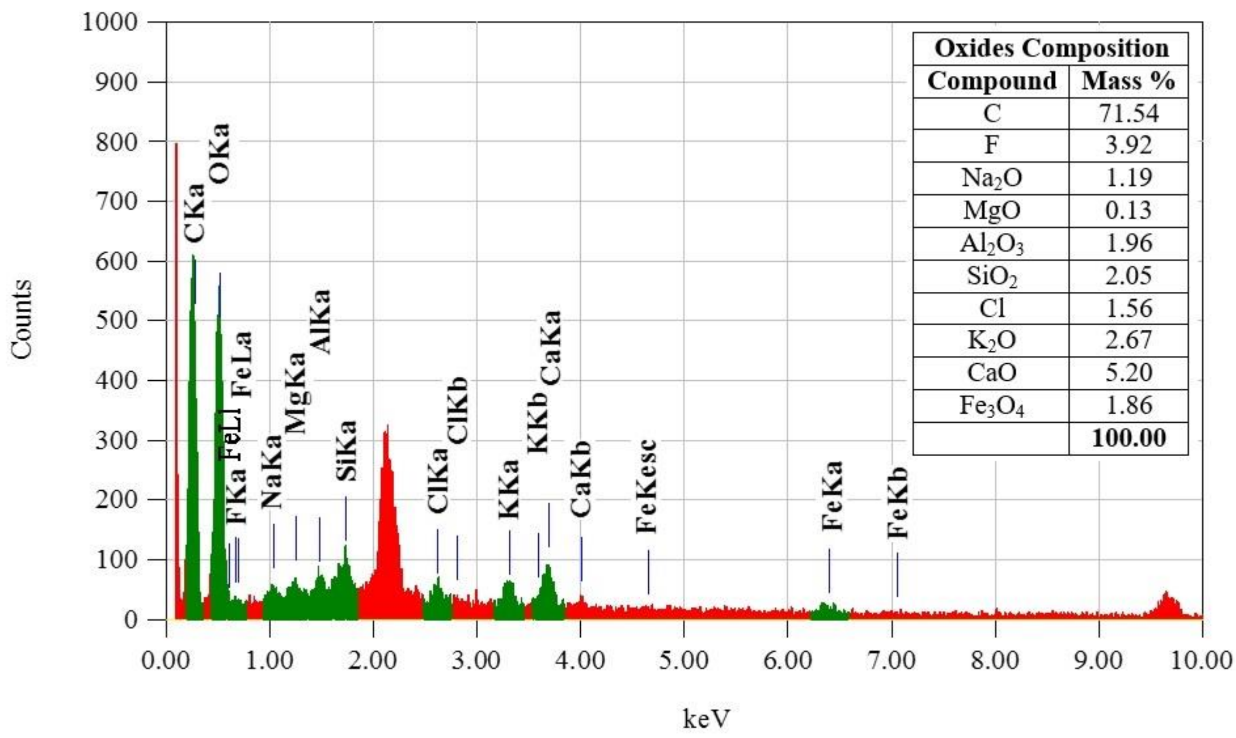


Fig 4.1 Qualitative XRD of AM gum Powder showing poorly defined crystals

The XRF test of powdered AM gum shown in Fig 4.2 indicates that the carbon is present in abundance and the metallic oxides (SiO_2, $Al2O_3$ and Fe_3O_4) are almost negligible (≈10.65 % of the total mass). Therefore, it may be reasonably assumed that the material is organic in nature and needs requisite tests for its characterization.



Oxides Composition	
Compound	**Mass %**
C	71.54
F	3.92
Na_2O	1.19
MgO	0.13
Al_2O_3	1.96
SiO_2	2.05
Cl	1.56
K_2O	2.67
CaO	5.20
Fe_3O_4	1.86
	100.00

Fig 4.2 XRF Test Results on a Powdered AM Gum

4.2 NMR and FTIR Characterization of AM Gum

Nuclear Magnetic Resonance (NMR) was conducted on 300 MHz AVANCE Series Swiss spectrometer. The peaks correspond to the relative amounts of compounds present in the Acacia Modesta (AM) gum as shown in Fig. 4.3. The highest peak at 4.699 ppm shows the presence of amide group which may be due to glycoproteins [24]. The amides tend to participate in hydrogen bonding with water and are considered to be more soluble as compared to the hydrocarbons [25]. Therefore these may be responsible for constituting the hydrophilic response of the gum. The series of peaks between 3.2 ppm and 4.2 ppm possibly represent halogenated hydrocarbons (CH_2F, CH_2Cl, CH_2Br or CH_2I) or nitro-methanide ($CH_2NO_2^-$). Halogenated hydrocarbons, being "non-polar compounds", are unable to form intermolecular interactions with water and other polar compounds [26] and result in flocculation. The third and fourth highest peaks at 1.166 and 1.152 ppm indicate the presence of long-chain hydrocarbons. These hydrocarbons are also hydrophobic in nature and may be responsible for the water repellent response of the Acacia Modesta (AM) gum [27].

It can be inferred that Acacia Modesta gum consists of both hydrophilic (glycoproteins) and hydrophobic (hydrocarbons) components. It has been confirmed that the gum when added to water forms lumps or 'micelles' (see section 4.3). In light of the above discussion, this formation of the lump-like structure (Fig. 7) could be attributed to the presence of hydrophobic hydrocarbon chains which are oriented towards the inner side of the micelle and cause the hydrophilic portion (possibly the amide groups) to come in contact with the surrounding water [28].

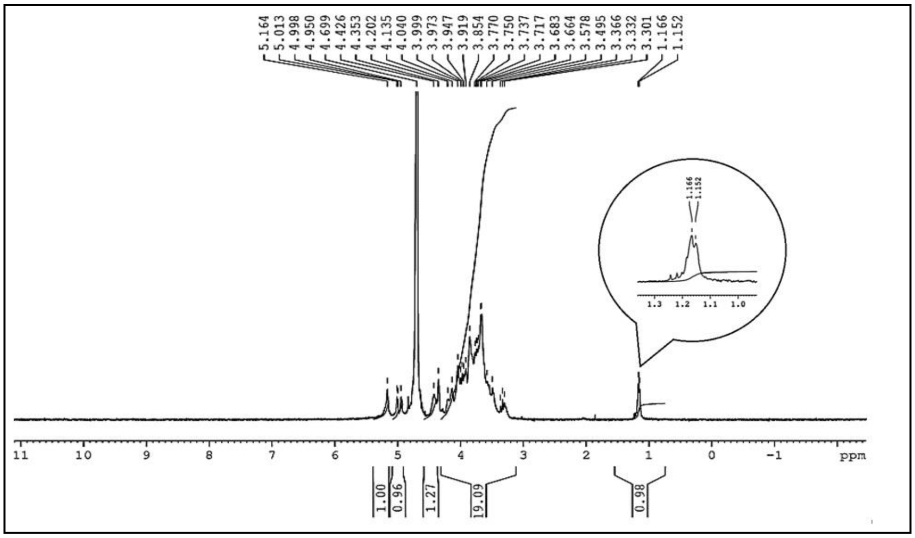


Fig 4.3 NMR Spectrum of AM gum between -1 to 11 ppm concentration

FTIR was conducted on Bruker Optics FT-IR Tensor 27 with standard KBr beam splitter technology in the scanning range of 500 – 4000 cm^{-1}. The lowest drop in Fig 4.4 at 506.82 shows the existence of alkyl halides in the gum. The second lowest drop at 1040.58 cm-1 may be due to the aliphatic amines and a rather gentle plateau at 1339.68 cm^{-1} indicates the presence of aromatic amines [29]. The plateaus at 1594.72 cm^{-1} and 3323.16 cm^{-1} are possibly due to hydrocarbons [29].

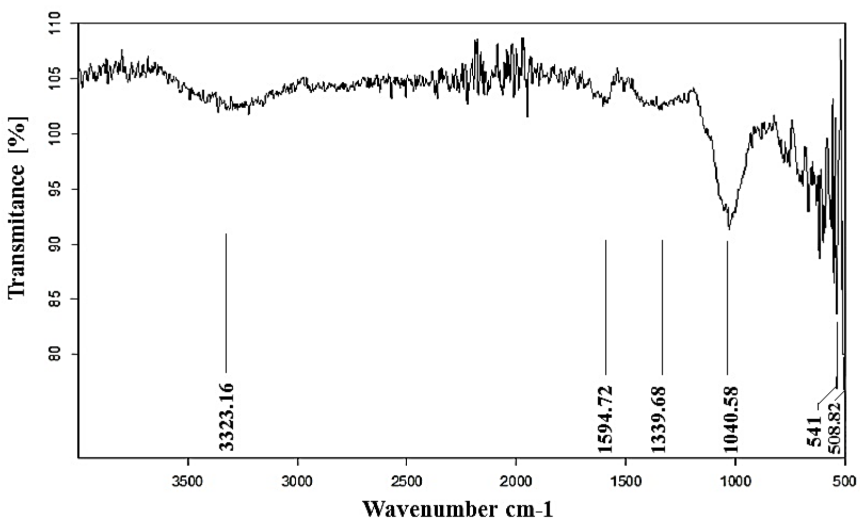


Fig 4.4 FTIR Characterization of AM Powder

4.3 Water-Entraining and Detraining Properties of Acacia Modesta Gum

Acacia Modesta gum is a complex polysaccharide consisting of branched chains [30]. Although polysaccharides react readily with water, molecular associations in case of Acacia gums do not change the basic structural components when these come in contact with water [31]. When AM gum was added to water, the following observations were made:-

- In solutions containing 5 % gum content, small lumps were formed at once. These lumps settled at the bottom of the beaker shown in Fig 4.5 (a).
- In solutions containing 10 % gum content, medium sized lumps were formed as shown in Fig 4.5 (b).
- In solutions containing 30 % gum content, even bigger sized lumps were formed as shown in Fig 4.5 (c). The solution became turbid possibly due to the presence of soluble impurities in the Acacia Modesta gum. The level of turbidity increased with increasing AM gum content. It can be inferred that the size of lumps depends on the amount of AM gum content in water. Higher the content of gum, more will be the adsorbed water.

Fig 4.5 (a) 12 g powdered AM gum in 240 g of water

Fig.4.5 (b) 24 g powdered AM gum in 240 g of water

Fig.4.5 (c) 72 g powdered AM gum in 240 g of water

From the above experiment it can be concluded that the Acacia Modesta gum is unable to form solution with water through complete dissolution process at room temperatures (25 ± 1o C). When added to water, the gum adsorbs specific moisture content according to its type and grain size, irrespective of the ambient water amount and forms lumps or ‘micelles’ (see Section 4.2). The lumps when taken out of water as shown in Fig 4.6 (a), Fig 4.6 (b) and Fig 4.6 (c) exhibited glue-like properties. These lumps were worked upon manually to bring them in shape and left to dry on a table surface for 5 days.

Fig 4.6 (a) Day 1- The gum adsorbs water and becomes gooey

Fig 4.6 (b) Day 1 – The lump exhibits sticky properties.

Figs 4.6 (c) Day 5 – Lumps retain their shape and become stiffer with time.

The lumps retained their shape and progressively became stiffer with time due to evaporation of water. It seems that the bigger sized micelles have larger pores which may be connected to surfaces on the inside. This property enables these micelles to readily engage and disengage water as per their size and pore-connectivity as explained in Section 4.2.

4.4 The Effect of Temperature and Moisture on AM lumps

On the 5th day, the lump of size 2 cm, acquired from a 30 % gum based solution, was placed in a steel cylinder surrounded by hot water and was subjected to varying temperatures to check the effect of temperature on the lump weight. The corresponding weights at different ambient water temperatures were measured and the graph, shown in Fig 4.7, was obtained.

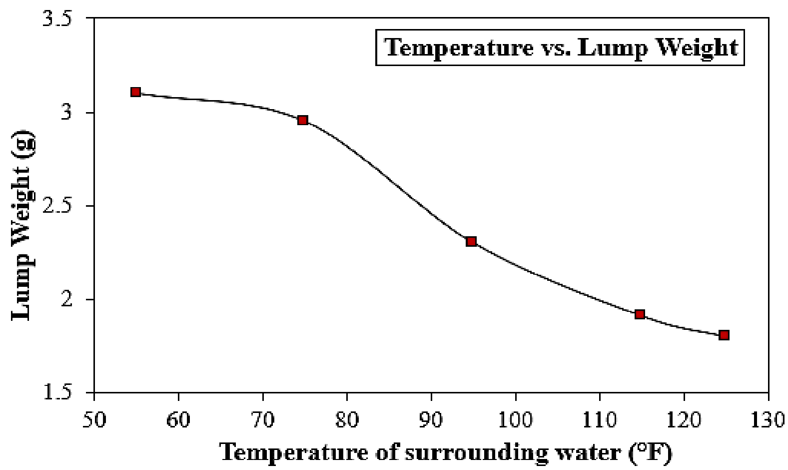


Fig 4.7 Variation of Temperature w.r.t AM lump weight

With increasing temperature, the weight decreased thereby indicating that the water was adsorbed physically which got released due to increased temperature. Appreciable weight loss occurs for temperatures between 80 °F and 115 °F. This may mean that in SCPs incorporating AM gum, if the internal temperature of SCPs increases beyond 80 °F due to hydration, the water is released back into the matrix making it available for hydration. This could be one of the reasons why calorimetric curves for the gum based systems begin to rise when the mix temperature increases beyond 80 °F (Fig 11). The other possibility is the creation of expansive crystals like CH within the matrix.

On the 5th day when the lumps were placed in the submerged conditions (50 g of lumps added to 75 g of water), these started to lose their stiffness in approximately 10 minutes and soon became plastic again. Therefore it can be reasonably assumed that the lumps, even in their stiffened form, are sensitive to the surrounding moisture content and the temperature value. This shows that the material is visco-plastic in nature.

4.5 Flow Measurements and Setting Times of SCP Systems

The flow of SCP systems incorporating AM was determined through Hagerman's mini-slump cone of 6 x 7 x 10 cm3. The target flow was 30 ± 1 cm. The super-plasticizer demand for each SCP formulation was achieved through trials. The mixing was done in the Hobart mixer.

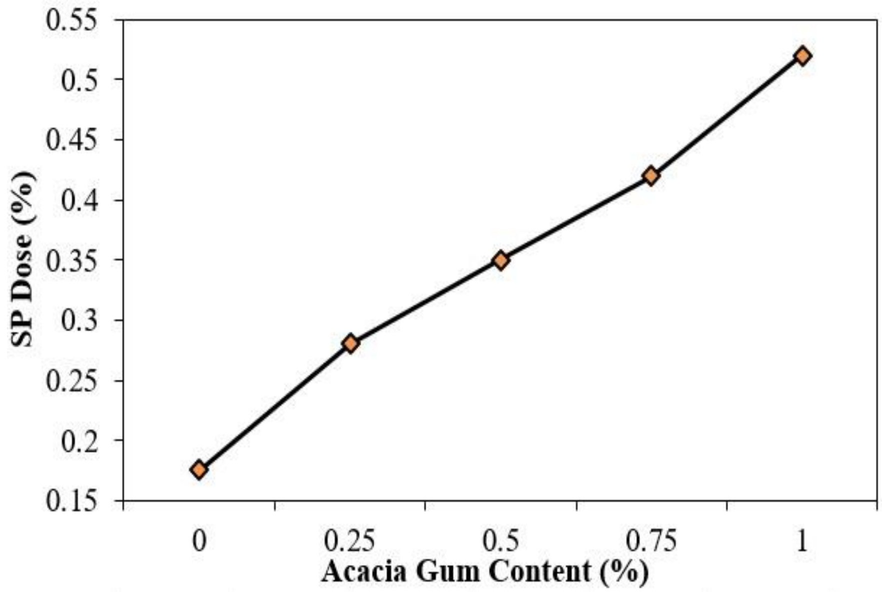


Fig 4.8 Determination of Super plasticizer demand of SCP formulations varying AM content

SP demand of SCP systems increased with increasing the AM gum content. This could be due to the viscous nature of gum which reduces the flow-ability of the respective cement paste system and seems to encourage flocculation. As already established in Section 4.4, AM gum adsorbs water physically and the amount of adsorption depends on the quantity of AM gum present in the cementitious system as well as the water content. Higher the quantity of both AM gum and water content, greater will be the water adsorption and more will be the degree of flocculation of AM gum. To counter this effect of flocculation, more super-plasticizer would be required to increase the flow of the matrix through de-flocculation of cement particles by electrostatic repulsion. It should be noted that although SP dosage increases, the time required (T30) to reach the specific flow value (30 ± 1 cm) also increases indicating that the viscosity of the respective SCP system becomes higher. This shows that the SP has more tendency of de-flocculating cement particles as compared to lumps of AM gum.

The initial and final Vicat setting times (Fig 4.9), found as per DIN-196-1, increased with increasing the AM gum content in SCP systems. The results were also compared with calorimetric response of similar SCP formulations.

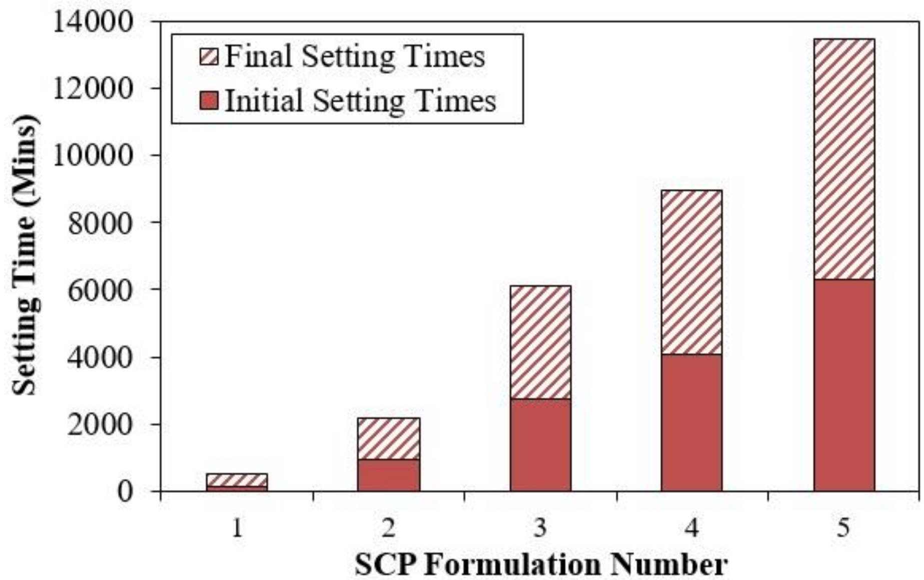


Fig 4.9 Determination of setting times of SCP systems using AM gum

The primary reason for delay in the setting times could be attributed to the significantly lower tendency of gum to chemically react with both the cement paste and the water content and the physical uptake of water (Section 4.4). It seems that the gum prolongs the setting time through adsorption of water molecules on its surface which occurs due to different reasons depending on the specific architectures and physiochemical interactions between the particles [32]. Also it must be kept in mind that water demand of the systems containing Acacia gum generally increases with increase in the gum content [24] whereas for this study mixing water content was kept constant (27 % of the dry cement weight). This shows that the effective water content reduces below the required water content level in the respective gum based cement paste systems. The lower amount of effective water content could be another reason for prolonged setting times. Lastly, as already established in the previous section the higher dose of SP could also be the reason for delay in setting times.

4.6 Calorimetric response of SCP systems using AM Gum

Calorimetry was done using F-CAL 8000 Calorimeter to know the hydration kinetics of SCP systems containing AM gum [33]. Each formulation was prepared at constant mixing water content (27 %) and at its respective super plasticizer demand required for the target flow. All six formulations were placed in the Calorimeter for 180 hours. The samples were taken out on the 8th day and the graphs were plotted and are shown in Fig 4.10.

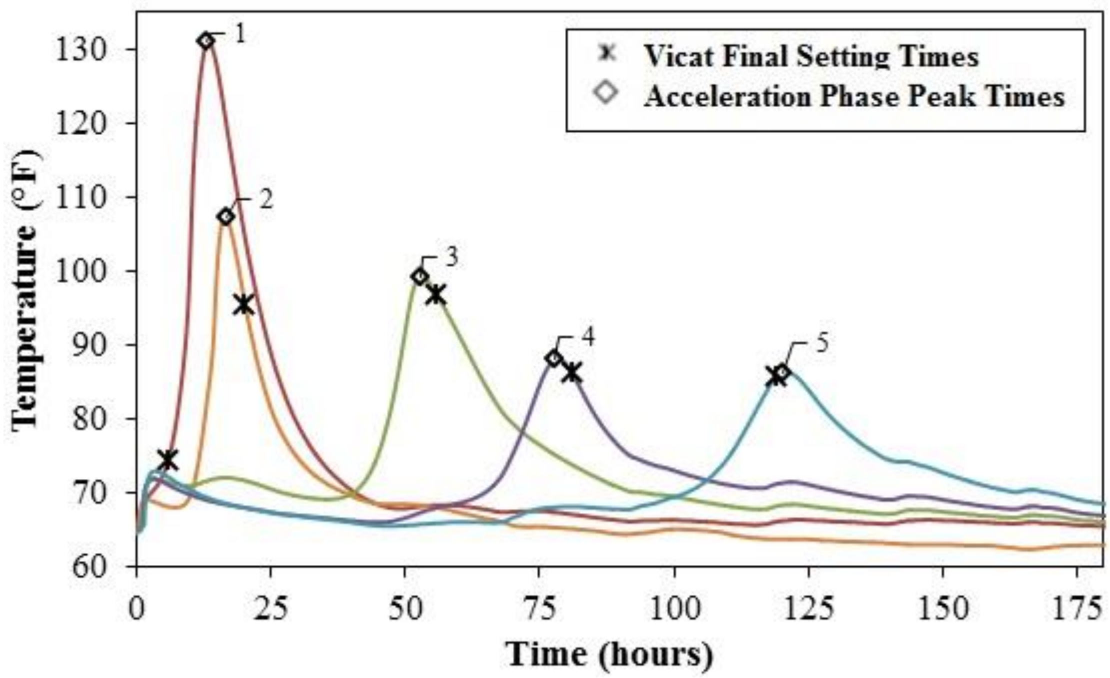


Fig 4.10 Calorimetry Response of SCP systems using AM

A progressive delay in calorimetric peaks and reduction in peak values is seen for formulations 1 to 5. Also it can be seen that as AM gum content in SCP systems increases, the final Vicat setting times and the Calorimetric peaks become closer. Final Vicat setting time is generally indicative of the end of the dormant period and start of the precipitation.

Lower peaks are indicative of the reduced hydration activity in the respective SCP systems which could be due to lower effective water in the mix containing AM gum and higher dosage of super-plasticizer. The delay in peaks shows that AM gum increases the dormant period. AM gum seems to behave as a nearly inert material in a cementitious system and adsorbs water physically. This leads to temporary reduction in available water content in the respective SCP system and formation of a coat (of lumps) nearby cement particles. This coat may form a barrier between water in mix and the surface of cement grains, thereby impeding the process of hydration for a specific period of time. The dormant time, as can be seen in Fig 4.10, seems to be approximately proportional to the amount of Acacia Modesta gum present in the SCP system.

As the hydration process gradually gains momentum, the temperature of the SCP matrix rises (as proposed by the theories on dormant period in literature). The lumps of AM gum, being vulnerable to heat energy which is now supplied due to acceleration process (Section 4.4), begin to disengage the temporarily held water content. As more water becomes available, the hydration activity increases and the temperature of the respective mix begins to rise, slowly at first and then rapidly with time. This is where the calorimetry curve for each formulation reaches peak of its acceleration stage.

4.7 Comparison between Calorimetry and Vicat Final Setting Times

Fig 4.11 compares the phase peak times of Calorimetry with Vicat final setting times. It can be seen that the final setting times occur only a few hours after the peak times of Calorimetry (Fig 4.10) and the gap gets reduced with increasing AM content in the SCP system. This shows that the precipitation time occurs at different times for different formulations. For the SCP formulation with AM gum, it lies near the end of acceleration period indicating maximum precipitation has already occurred.

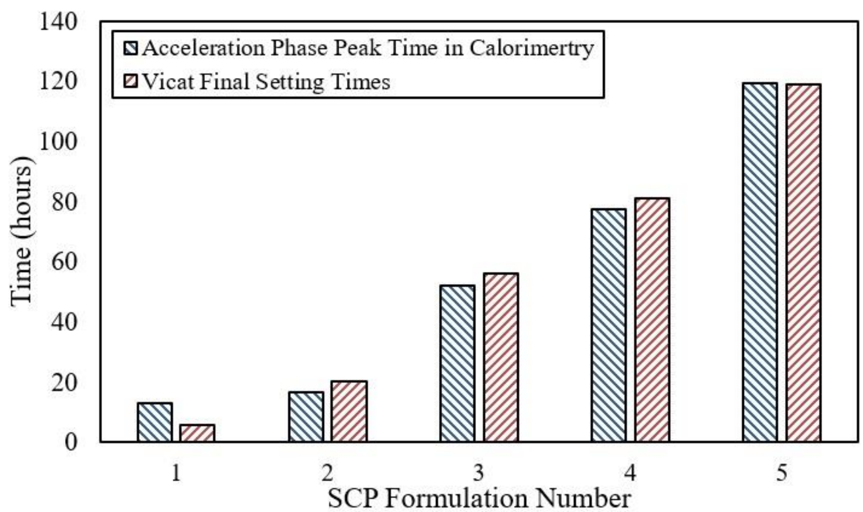


Fig 4.11 Comparison between peak values of Calorimetry and Vicat Final Setting times

4.8 Density and Viscosity Measurement of SCP systems

Actual dimensions of the cast samples were measured using Vernier Caliper and volume was calculated along with their weights at age of 7th, 14th and 28th day of casting. Three control samples (C1-SP0.175-AM0.0-27) were placed in the water curing tank whereas the remaining twelve SCP samples containing varying amounts of Acacia Modesta gum and super-plasticizer were air cured.

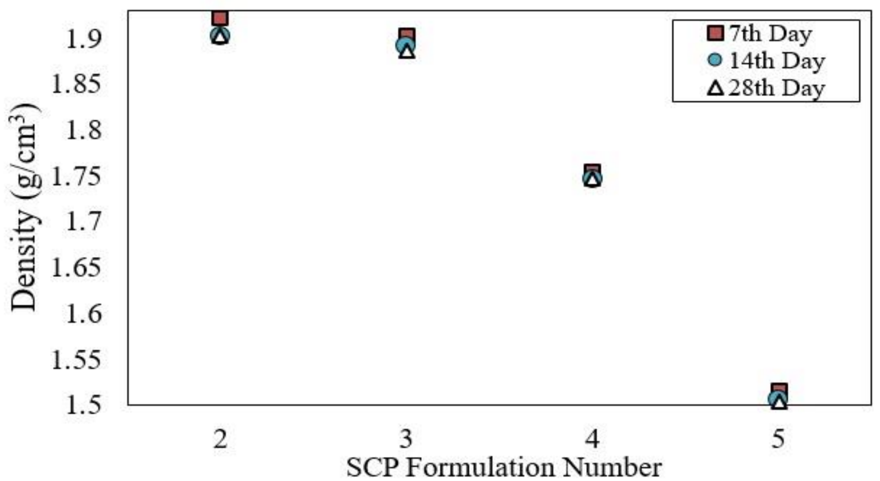


Fig 4.12 Bulk Density Measurements of SCP systems

The results showed a decrease in the density values with increase in the gum content of air cured samples (Fig 4.12). This might be due to increase in air content in SCP formulations containing SP and AM gum. The creation of air voids is a time dependent phenomenon and is particularly prominent when chemical and autogenous shrinkage relationships depart [34]. Increased air content leads to reduction in the density of the hardened SCP systems.

4.9 Rheometric Investigation results

The method for finding plastic viscosity has already been discussed in section 3.5 Plastic Viscosity and shear rate response is plotted in Fig. 16.

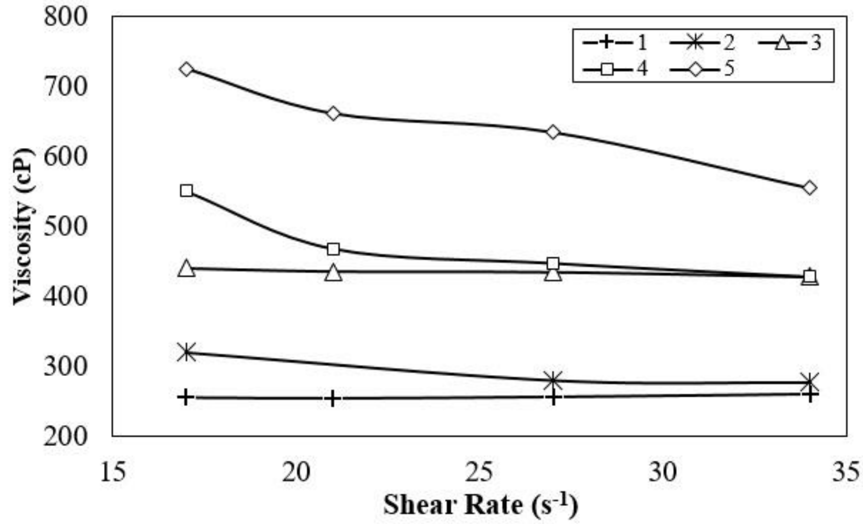


Fig 4.13 Viscosity Measurements of Typical SCP formulations

As the gum content increases in SCP systems, the viscosity also increases. The reason could be attributed to the highly branched structure of the AM gum which causes it to physically attach itself with the surrounding water molecules and cement particles at once [24]. On increasing the shear rate, these associations break and a decrease in the viscosity is observed.

A gum based self-consolidating paste system may be classified as a non-Newtonian fluid (Bingham plastic) whose viscosity decreases on application of shear stress and increases when stress is removed. When allowed to rest in stationary position, the particles move to more favorable positions. This results in their attachment to various parts and causes temporary increase in the viscosity [35]. This phenomenon is known as "thixotropy" and is a time-dependent reversible process [36, 37]. The process may be both reversible and isothermal [38]. Once shear stress is applied, the fluid regains its flowing ability [39]. This process is also known as shear thinning and the temporary loss in viscosity is known as pseudo-plasticity [40].

Viscosity was also measured based on flow time measurements (Fig. 4.14) and this procedure may be regarded as an indirect method of viscosity measurement [33]. Time taken to reach 25 cm (T25) and 30 cm diameter (T30) on the flow apparatus was recorded for each SCP formulation, the first time (T25) giving an idea about the viscosity of the system and the second one (T30) about the yield stress of the system. Use of AM gum increases the plastic viscosity and also the yield stress

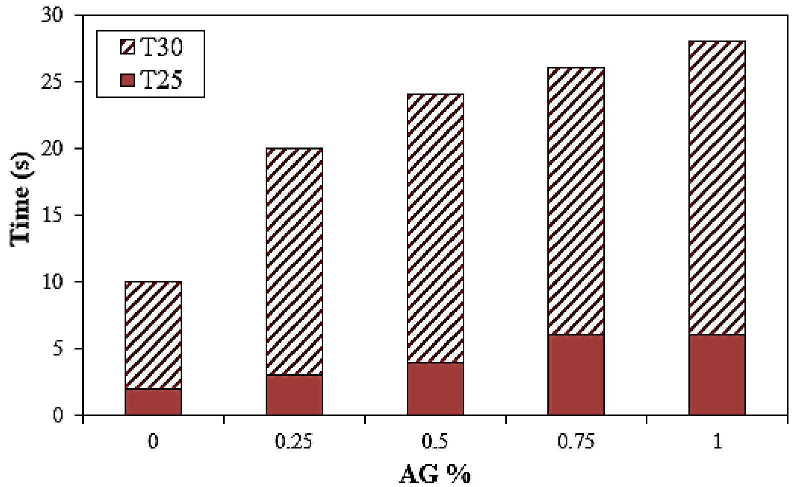


Fig 4.14 Indirect Viscosity Measurement time using Hagerman cone

4.10 Shrinkage Response of SCP

Shrinkage at early ages affects the volume stability of cement based systems [33]. The method for finding shrinkage of the respective SCP formulations has been discussed in Chapter 3. The results are shown in Fig.4.15

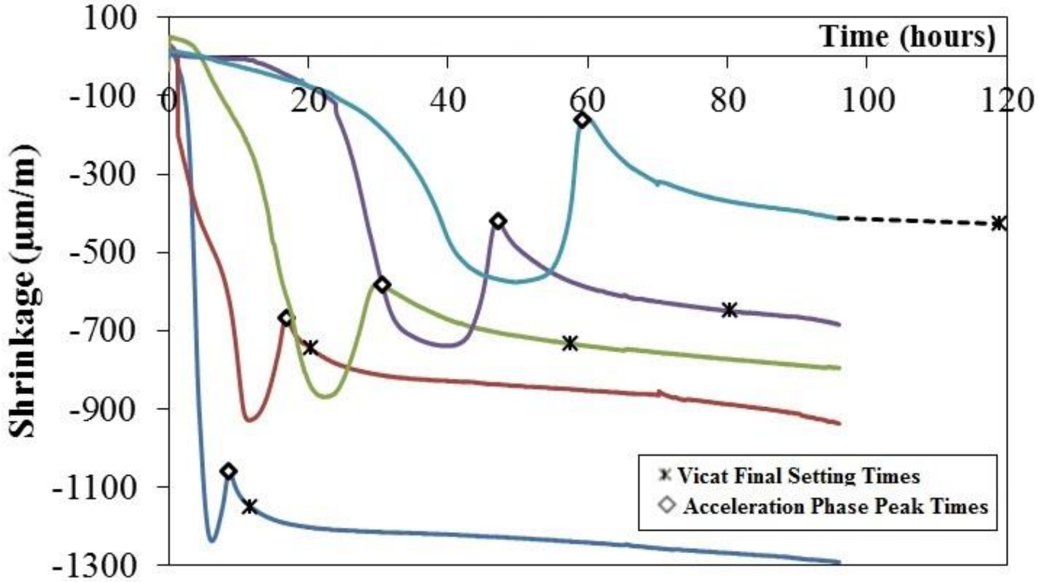


Fig 4.15 Shrinkage Response of SCP Formulation containing AM gum

The following observations can be made from the Fig 18 :

1. Samples 2, 3, 4 and 5 reduce maximum total shrinkage by 24 %, 29 %, 40 % and 53 % as compared to that of the control sample respectively. Increase of AM gum in SCP systems reduces total shrinkage.
2. The trend shows that setting process is delayed with increase of AM in SCP systems and the response becomes faltter. After showing maximum total shrinkage a comparative delay in time of shrinkage arrest is clearly visible which may be due to creation of some internal expansice species or release of entrained water by AM gum. If later is the case then Acacia Modesta gum seems to behave as an internal curing agent. However more work needs to be done on this aspect.
3. The final Vicat peak time moves away from the maximum arrested shrinkage time when AM gum content in SCP systems increase and shrinkage starts increasing again.

Table 2 gives the details obtained from total shrinkage curves obtained for SCP systems

Table 1: Time and Volume Change(s) in the selected formulations

SCP Formulation Number	Formulation(s)	Time to attain maximum overall shrinkage (hrs)	Initial Expansion (μm/m)	Maximum total Linear Shrinkage (μm/m)	% Reduction in Maximum Shrinkage (w.r.t the control specimen)
[1]	C1- SP0.175-WD27	6.15	15.185	-1235	0
[2]	C1- SP0.28-AM0.25-WD27	12.1	26.6	-929	24.77
[3]	C1- SP0.35-AM0.50-WD27	22.8	49.41	-870	29.55
[4]	C1- SP0.42-AM0.75-WD27	40	6.3	-739	40.16
[5]	C1- SP0.52-AM1.00-WD27	50	12.65	-576	53.36

It can be seen that the shrinkage in acacia gum based formulations has been reduced by almost 53 % as shown in Table 2.

4.11 Compressive Strength Test on SCP systems

Fifteen SCP samples (three samples for each formulation) were prepared in the molds of 4 x 4 x 16 cm^3 and tested in machine as per DIN-196 for compression. SCP Samples containing 0 %

gum were taken out on the next day of casting and placed in a water curing tank for 28 days till the time of testing in SSD condition. The rest of the SCP samples were taken out after their respective final VICAT setting times and were placed under the plastic sheet for air curing during the remaining days. Fig 4.16 shows the compressive strength of SCP systems.

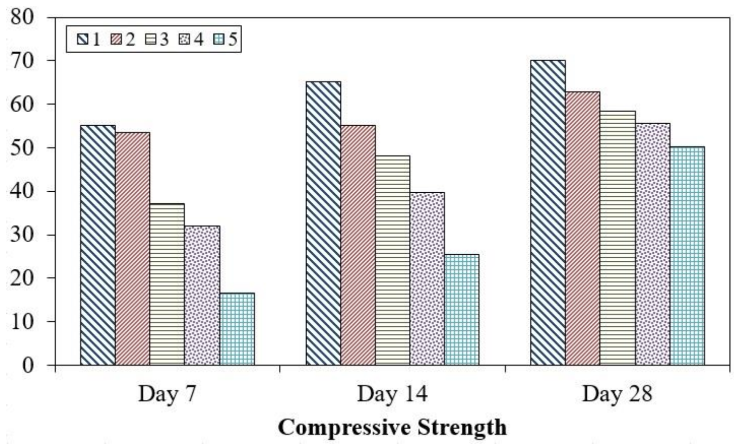


Fig 4.16 Compressive strengths of SCP samples subjected to air curing

It can be seen that the compressive strength decreases as the gum content increases. The overall average reduction in samples containing AM gum is around 15 %. The reduction could be due to increase in the total entrapped air content which leads to increase in porosity and weakens its properties. The reduction can be accounted for by adding suitable SRMs [24].

4.11Effect of Curing on SCP Systems containing AM gum

Acacia Modesta gum when used in cementitious systems seems to behave as an internal curing agent (Section 9). Fig 4.16 shows compressive strength results of SCP formulations cured in air while Fig 4.17 shows the compressive strength of SCP formulations cured in water.

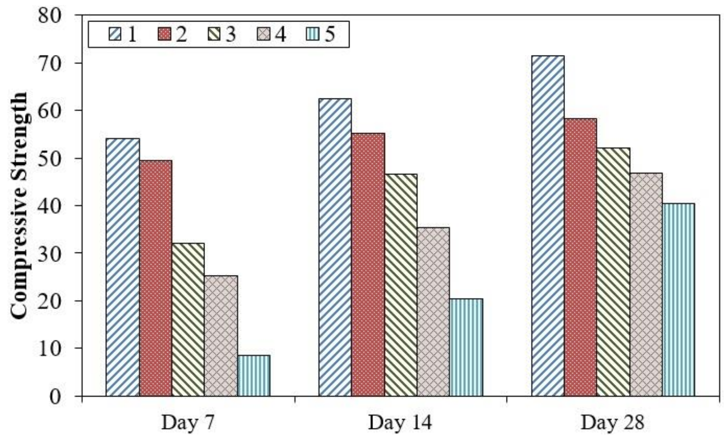


Fig 4.17 Compressive Strength of samples subjected to conventional water curing

Almost 30 % reduction in strength values (Fig 4.17) occurred as compared to the 15 % reduction which happened when air curing was done (Fig 4.16). These results are in agreement with earlier study [24].

4.13 Air Content Estimation in Fresh Formulation

The air content for fresh SCP formulations was determined using Luftgehaltspruefer Testing of 1 liter capacity. The SCP was prepared at constant water content (27 % of the total weight of dry cement) and at their respective super-plasticizer demands. Fig. 21 shows the internal air content in fresh SCP mixes.

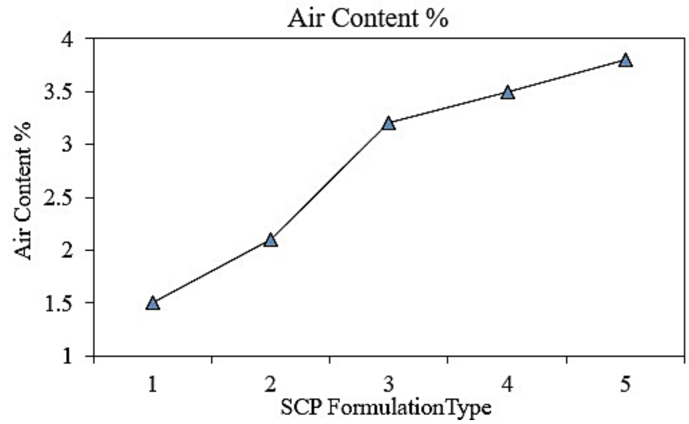


Fig 4.18 Effect of AM Gum on Air Content in fresh SCP Samples

It can be seen (Fig 4.18) that as the gum content increases air content also increases. This could be due to several reasons including that of air induction by SP, size and properties of AM gum and entrapment of air due to increased viscosity during initial mixing in the Hobart Mixer.

4.13.1 Comparison between SCP systems containing Acacia Modesta and Acacia Nilotica gums as admixtures

In a side investigation [24], the response of SCPs containing Acacia Nilotica gum as an admixture was studied. While both Acacia Nilotica and Acacia Modesta gums yielded different results, their overall behavior in cementitious systems was somewhat similar as discussed in the following lines.

- Both Acacia Modesta and Acacia Nilotica gums when incorporated in SCP systems increased flow and setting times
- Both these types of Acacia gums increased dormant period and reduced heat of hydration as evident from the respective calorimetric curves
- The density of the gum based systems decreased due to increase in the air content
- Both types gave increased strength values when the respective SCP samples were cured in the laboratory relative to the samples which were cured in a water tank

CHAPTER 5

INDUSTIAL APPLICATIONS

In common parlance, Acacia gum behaves as a chemically inert material in the cementitious system. It adsorbs water physically and forms a coat over the cement particles. This coat delays the hydration process. When this coat eventually breaks due to difference in temperature, acacia gum disengages the adsorbed water and the hydration process gains momentum. AM gum based samples are porous; also AM lumps are sensitive to moisture. Therefore if water curing is done, these samples lose their strength.

This behavior resulted in AM gum delaying setting times and reducing heats of hydration. This property makes such systems suitable for hot weather and mass/distant concreting where longer setting times are preferred. Also AM gum in SCPs reduces their density and increases air voids. This property may be useful for in-fill panels. Finally, we observed that the incorporation of AM gum in SCPs made the respective systems more resilient. A resilient concrete structure provides an extended service life, adaptive re-use and the challenges of natural and man-made disasters (National Ready Mixed Concrete Association, 2009).

CONCLUSIONS AND RECOMMENDATIONS

1. The setting times of SCP systems containing Acacia Modesta gum increased almost in proportion to the amount of Acacia Modesta gum used in these samples whereas early heat of hydration in such systems decreased. Therefore it may be concluded that AM behaves as a typical retarder in such systems and may be suitable for hot-weather and distant concreting where delayed setting times are preferred.
2. SCP formulations show an increase in the viscosity and air content (of the fresh samples) and a respective decrease in the density values (of the hardened samples). Acacia Modesta gum based systems are therefore effective in creating lighter concrete constructions. This makes such system suitable for in-fill panels.
3. Compressive strength of the gum based SCP formulations reduced by approximately 30 % with water curing at 28 days and by 15 % when cured in the laboratory air. This strength reduction may be taken care of by using suitable SRMs. Also note that Acacia

Modesta gum has been used in its very raw form (i.e., it is obtained directly from the Acacia Modesta tree). The polymers, which are usually available in market, are present in the processed form. Therefore the gum in question has almost zero processing cost. It may be subjected to further chemical processing for use an admixture to help in the improvement of the properties of the cementitious systems.

4. Acacia Modesta gum seems to behave as an internal curing agent in the SCP systems and effectively reduces the total shrinkage in the respective cementitious systems. However more work needs to be done on this aspect.
5. Both Acacia Modesta and Acacia Nilotica gums (studied in a side investigation) exhibit similar trends in results in terms of calorimetry, flow response, setting times and strength when used as admixtures in the SCP systems. Acacia Modesta gum based samples however exhibited better results.
6. Acacia Modesta gum based samples show an improvement in the resilience of the respective systems. This area also provides the potential direction for future investigations.

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APPENDIX

Formulation No.	Formulation Type
1	C1-SP0.175-AM0.0-27
2	C1-SP0.28-AM0.25-27
3	C1-SP0.35-AM0.50-27
4	C1-SP0.42-AM0.75-27
5	C1-SP0.52-AM1.00-27

1. Particle Size Distribution

Sieve No.	Sieve diameter (m)	Mass retained (g)	Cumulative mass retained (g)	Cumulative mass retained (%)	Cumulative mass passing (%)
16	1.18	0.102	0.102	0.058	99.942
30	600	15.23	15.332	8.74	91.26
40	425	28.05	43.382	24.75	75.25
50	300	55.05	98.432	56.159	43.841
100	150	45.33	143.762	82.022	17.978
140	106	12.99	156.752	89.433	10.567
200	75	11.41	168.162	95.943	4.057
Pan	-	7.11	175.272	100	0
Total		175.272			

(% Error = 1.5)

2. Flexure Test Results

Formulation	Day 3	Day 7	Day 14	Day 28
1	0.82	11.602	15.7	15.8
2	4.3	2.69	12.07	12.1875
3	0.49	16.289	15.093	14.64

4	0	9.61	14.53	12.304
5	0	5.51	7.5	13.36

3. Compressive Test Results

Formulation	Day 3	Day 7	Day 14	Day 28
1	40.5	54	62.42	71.5
2	39.15	49.5	55.2	58.15
3	25	32.15	46.5	52.05
4	0	25.31	35.31	46.75
5	0	8.5	20.5	40.5

4. Flow Test Results

Formulation	T25 (s)	T30 (s)
1	2	8
2	3	17
3	4	20
4	6	20
5	6	22