

**RESPONSE OF SELF-CONSOLIDATING PASTE
SYSTEMS CONTAINING ACACIA GUM AS AN ORGANIC
ADMIXTURE**



FINAL YEAR PROJECT UG 2012

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This is to certify that the

Final Year Project Titled

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for the undergraduate degree

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Abstract

This study investigates the response of Self-Consolidating Paste systems (SCPs) with the addition of an organic admixture, Acacia Nilotica Gum, a species of trees grown in Pakistan belonging to the genus “Acacia”. Its gum is extracted from the tree and is used in a number of application primarily as an emulsifier. This research uses NMR and FTIR experimental techniques to report the characterization of Acacia Gum (AG). The primary aim of this study is to investigate the usage of Acacia Gum in SCPs as a Viscosity Modifying Admixture (VMA). VMAs are used in SCPs to impart stability on the mix and reduce bleeding and segregation. The gum was grinded to powdered form and added in SCPs in ratios of 0.33%, 0.66% and 1% with respect to the cement content and its effect on a number of properties of SCPs was studied through experimentation including Water demand and Setting time determination, Flow test, Calorimetry, Shrinkage and Strength Tests. This study records and analyzes the results of these tests to evaluate the suitability of using Acacia Gum as an organic admixture in SCPs.

Furthermore, Secondary Raw Materials (SRMs) were incorporated in two additional formulations in the presence of AG and the response of SCPs was studied. The effects of SRMs on the properties of SCPs are well-established in literature and the knowledge has been utilized to achieve improvement of SCPs containing Acacia Gum.

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INTRODUCTION

1.1 Self-Consolidating Cementitious Systems

Self-consolidating cementitious systems include self-consolidating pastes, mortars and concrete. Self-compacting Paste (SCP) systems are the vehicles for transport of aggregates during flow in self-compacting mortar (SCM) systems and self-compacting concrete (SCC) systems. The properties of modern concretes like high performance concrete (HPC) and SCC depend significantly on the properties of SCP systems [1].

Self-Consolidating Concrete has properties that differ considerably from conventional slump concrete. SCC is highly workable concrete that can flow through densely reinforced and complex structural elements under its own weight and adequately fill all voids without mechanical vibration and segregation, excessive bleeding, or other separation of materials [2].

1.1.1 Criteria for Self-Consolidating Systems:

There are three basic criteria for attaining self-compaction: High deformability, high passing ability and high resistance to segregation. High flow is achieved by using super-plasticizer and high segregation resistance is achieved by using either low water-powder ratio or a moderate water-powder ratio with viscosity enhancing agent [3].

1.1.2 Advantages and Disadvantages

Self-Consolidating concrete is used in the construction industry nowadays due to a number of advantages it presents over the conventional high slump concrete. Firstly, SCC reduces construction time and manpower requirements due to its ability to compact under its own weight, thereby eliminating the requirement for manual vibration which is time consuming and also results in differential compaction of concrete and hence different durability. Secondly, SCC results in an overall better finished surface and better homogeneity of the concrete mix due to absence of mechanical compaction which also eliminates the possibility of worker error. SCC also improves the workplace environment by reducing noise pollution and eliminating health problems related to the use of vibration equipment such as 'white fingers' and deafness. SCC is therefore called the 'quiet revolution in concrete construction' [4]. One disadvantage of SCC is its higher cost of production especially in country like ours compared to conventional concrete but that is mostly offset by the economic advantages gained through the requirement of a smaller workforce, lesser time to achieve concreting and higher durability. Sometimes SCC is the only solution in a typical application.

1.2 Acacia Gum (AG)

Acacia Gum is a natural organic ooze-out of the various species of acacia tree. It is one of the world's most common gums and its usage dates back to ancient times for purposes as varied as mummification and inks for hieroglyphics. In modern times, acacia gum finds its usage in medicine, food industry, printing, paint production, glue, cosmetics and various industrial applications. Acacia Gum is obtained from a wide variety of Acacia trees grown in large parts of the African continent and also towards east in the South-East Asian countries of Pakistan and India. Sudan is the largest producer of acacia gum in the world which produces 70-85% of world production, and produces most of acacia gum in Sudan from the tree Acacia Senegal [6]. In Pakistan it is extracted from the Acacia Nilotica tree which is indigenously known as "Keekar" [7].

The chemical composition of acacia gum is a complicated mixture of polysaccharides and glycoproteins which gives it the properties of a glue and binder suitable for human consumption. Due to its viscous nature acacia gum has also found its usage as a viscosity controller in inks and textile industries. In concrete, the application of acacia gum is relatively limited although research has shown some benefits of using it as an organic admixture in suitable quantities and in suitable form which can be explored. In self consolidating cementitious systems, acacia gum has the potential of being used as a substitute for the Viscosity Modifying Admixture (VMA) required in such systems due to its viscosity enhancing properties along with some good engineering properties.

1.3 Secondary Raw Materials (SRMs):

Secondary Raw Materials are added in cementitious mixtures primarily to economize the mix by replacing a percentage of the cement and enhancing a number of properties depending on the type of SRM used. SRMs can be classified into two distinct categories depending on the mechanism of their action in cement pastes; either chemical or physical. SRMs, also known as supplementary cementitious materials (SCMs), that react chemically with cement pastes are classified as pozzolanas and those that react physically are termed as fillers.

Pozzolanas are natural or artificial material containing silica in reactive form. ASTM 618-94a describes pozzolana as:

"A siliceous and aluminous material which in itself possess little or no cementitious value but will, in finely divided form and in the presence of moisture, chemically react with calcium hydroxide obtained during cement hydration at ordinary temperature to form compounds possessing cementitious properties" [5]

For the pozzolanas to undergo an effective chemical reaction, it is important for the silica to be amorphous, that is, glassy, because crystalline silica has very low reactivity [5]. These materials are often added to concrete to make concrete mixtures more economical with increased strength, reduced permeability and improve other concrete properties [8].

The other category of SRMs are called Fillers, also called Mineral Powders (MPs) or Rock Powders, which are chemically inert and their effect is predominantly physical [8]. A filler is a finely ground material, of about the same or even lesser fineness as Portland cement, which, owing to its physical properties, has a beneficial effect on some properties of concrete, such as workability, density, permeability, volume stability, durability, capillarity, bleeding or cracking tendency. Limestone powder, marble powder and quarry dust are examples of Fillers commonly used. [5]

Generally, the use of SRMs may reduce the shrinkage and heat of hydration of cementitious mixes due to replacement of cement. [8]

1.4 Research Objectives:

The objectives of this research are twofold:

- Firstly the research aims to characterize the organic gum obtained as an ooze-out of the Acacia Nilotica (local name: Keekar) trees of Pakistan through NMR and FTIR experimental techniques.
- Secondly, the acacia gum is mixed in powdered form in SCPs in varying ratios and its effect on the various properties of SCPs is recorded using experimental techniques.

LITERATURE REVIEW

2.1 Self-Consolidating Paste Systems (SCPs)

Self-Consolidating Cementitious systems include Self Consolidating Pastes Systems (SCPs), Self-Consolidating Mortar Systems (SCMs) and Self Consolidating Concrete Systems (SCCs). This study focuses on SCPs which are the basic medium for carrying the aggregates in SCMs and SCCs. Most studies are carried out at SCP level as presence of aggregates interferes with the results of some tests. The optimization of SCPs would, thus, improve the quality of both SCMs and SCCs which is the ultimate aim from a construction point of view.

SCPs differ from conventional cement pastes in that they have high flowability characteristics owing to the presence of a chemical admixture called a superplasticizer. In addition, another criterion for SCP is stability or resistance to segregation which is achieved through the use of a Viscosity Modifying Admixture (VMA).

2.1.1 Superplasticizer (SP):

ASTM C 494-92 refers to superplasticizers as “water-reducing, high range admixtures”, classifying them as Type F admixtures or when retarding effect is also induced, as Type G admixtures [5]. Chemically, superplasticizers consist of long molecules which act by wrapping themselves around the cement particles and giving them a highly negative charge so that they repel each other. This results in deflocculation and dispersion of cement particles. The resulting improvement in workability is exploited in two ways: by producing concrete with a very high workability or concrete with a very high strength at low water contents [5]. For the purpose of this study, superplasticizers are used for the purpose of achieving greater workability at a given water/cement ratio.

2.1.2 Viscosity Modifying Admixture:

Viscosity modifying admixtures (VMAs) are water-soluble polymers that increase the viscosity of mixing water and enhance the ability of cement paste to retain its constituents in suspension [9]. Viscosity Modifying Admixtures enhance stability of the mixture by preventing bleeding and segregation. They increase the viscosity of the highly flowable SCC to ensure all the materials of the mixture remain in a single matrix and flow as a uniform mixture instead of separating out or segregating. Various VMAs are available in the market as chemical additives. Organic materials with viscosity enhancing properties can also be used as VMAs in SCCs. For the purpose of this research, Acacia gum is used as an organic VMA and its effect on the various properties of SCPs is studied accordingly.

2.1.3 Viscosity and Yield Stress Measurement of SCPs:

Viscosity of a SCP can be estimated by measuring the time taken by the paste to reach a spread of 25 cm in a flow experiment using the mini-Hagerman cone apparatus. This time is denoted by T25cm in seconds. Greater the T25cm (sec) value, more viscous is the paste. Greater viscosity may be desirable when the concrete has to fill congested reinforcement or deep sections whereas lesser viscosity may be appropriate for concrete that has to travel long distances without much obstruction.

Yield stress of an SCP may be estimated in a similar way by measuring the time to reach 30 cm mark on the mini-Hagerman Cone flow apparatus. This time is denoted by T30cm (sec).

2.1.4 Rheology:

Rheology is the science of the deformation and flow of materials. It is a complex discipline used to understand the workability characteristics of SCC. The two most important properties of SCC's rheology are:

Yield Stress: the measure of the amount of energy required to make SCC flow. To be considered SCC, concrete must flow easily under its own weight, so its yield stress must be very low.

Plastic viscosity: the measure of the resistance of SCC to flow due to internal friction. SCC must have a high viscosity in order to suspend aggregate particles in a homogenous manner within the concrete matrix without segregation, excessive bleeding, excessive air migration, or paste separation.

In summary, SCC must have low-yield stress and high viscosity. [2]

2.2 Acacia Gum (AG)

Acacia gum is an edible biopolymer obtained as exudates of mature Acacia trees which grow in different parts of the world, principally in the African region of Sahe in Sudan. The exudate is rich in soluble fibers, and its emanation from the stems and branches usually occurs under stress conditions such as drought, poor soil fertility, and injury. [10]

2.2.1 Uses of AG

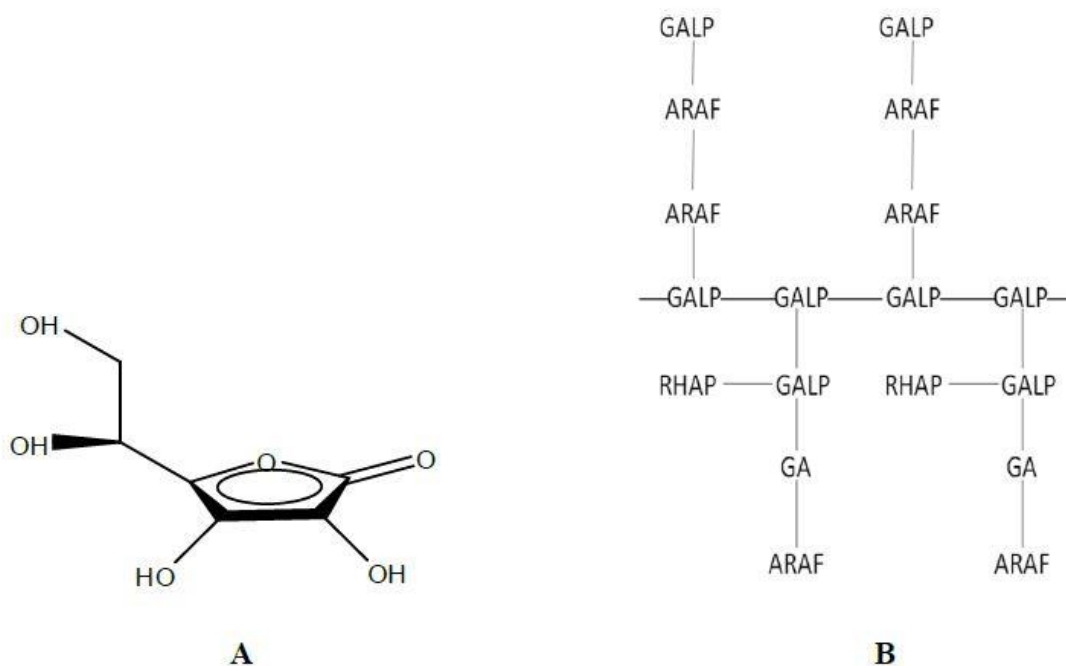
The use of Acacia Gum dates back to the second millennium BC when the Egyptians used it as an adhesive and ink. Chemically, Acacia Gum is a complex mixture of macromolecules of different size and composition (mainly carbohydrates and proteins). Today, the properties and features of AG have been widely explored and developed and it is being used in a wide range of industrial sectors such as textiles, ceramics, lithography, cosmetics and pharmaceuticals, encapsulation, food, etc. Regarding food industry, it is used as a stabilizer, a thickener and/or an emulsifier agent (e.g., soft drink syrup, gummy candies and creams). In the pharmaceutical industry, AG is used in pharmaceutical preparations and as a carrier of drugs since it is

considered a physiologically harmless substance. Additionally, recent studies have highlighted AG antioxidant properties, its role in the metabolism of lipids, its positive results when being used in treatments for several degenerative diseases such as kidney failure, cardiovascular and gastrointestinal. [10]

2.2.2 Chemical Composition

The chemical composition of Acacia Gum consists of multiple fractions consisting mainly of highly branched polysaccharides; typically 42% galactosyl, 27% arabinosyl, 15% rhamnosyl, 14.5% glucuronosyl, and 1.5% 4-O-methyl-glucuronosyl, and a protein-polysaccharide complex as a minor component. GA consists of mainly three fractions: the major one is a highly branched polysaccharide consisting of galactose backbone with linked branches of arabinose and rhamnose, which terminate in glucuronic acid (found in nature as magnesium, potassium, and calcium salt) [11].

The composition can vary with Acacia Gum's source, the age of the trees, soil environment and climatic conditions. One of the many proposed structures of Acacia Gum is shown in the figure below: [11]



1. Structure of monosaccharides (A), Segment of Arabic Gum molecule (B). The polysaccharide backbone is composed of D-Galactopyranose (GALP), with linked branches of L-Arabofuranose (ARAF), L-Rhamnopyranose (RHAP), and D-Glucuronic Acid (GA).

Figure 2.1: Acacia Gum Structure

2.2.3 Acacia Nilotica

Acacia nilotica Lam (Mimosaceae) indigenously known as ‘Babul’ or ‘Kikar’ is a medium sized tree and is widespread in subtropical and tropical Africa from Egypt to Mauritania southwards to South Africa, and in Asia eastwards to Pakistan and India [7]. This research studies the effects of *Acacia Nilotica* on SCPs. Gum is present in the bark and tends to be dark in colour. This species may indeed have been the original source of true gum arabic which is now obtained commercially from *Senegalia senegal*. The *Acacia nilotica* gum, samogh or samuk (arabic) is sold in balls and is commercially of inferior quality. It has been used as an emulsifying agent and emollient. It is edible and is used to relieve throat and chest complaints. In addition, It has an inspiring range of medicinal uses and is used as an astringent, emollient, liver tonic, antipyretic and antiasthmatic. [7] Its taxonomic tree is shown as following section.

2.2.3.1 Taxonomic Tree

Domain: Eukaryota

Kingdom: Plantae

Phylum: Spermatophyta

Subphylum: Angiospermae

Class: Dicotyledonae

Order: Fabales

Family: Fabaceae

Subfamily: Mimosoideae

Genus: *Acacia*

Species: *Acacia nilotica*

2.3 Secondary Raw Materials (SRMs):

Secondary Raw Materials are used to enhance the properties of cementitious mixtures and also make them cost-effective by replacing a portion of the most expensive commodity in the mix, cement. The SRMs used in this study are Fly Ash and Limestone Powder.

2.3.1 Fly Ash:

Fly Ash, also known as pulverized-fuel ash, is the most common artificial pozzolana. It is the ash precipitated electrostatically or mechanically from the exhaust gases of coal fired power stations. The fly ash particles are glassy and spherical (which is advantageous from the water requirement point of view) and have a very high fineness. It also has a high specific surface which means that the material is readily available for reaction with Calcium Hydroxide. [5]

Class F: It is the most common fly ash. It is derived from bituminous coal and is mainly siliceous. The main requirements of ASTM C 618-94a are: a minimum content of 70% silica, alumina and ferric oxide taken all together. The pozzolanic activity of Class F fly ash is in no doubt, but it is essential that it has a constant fineness and a constant carbon content. From an

appearance point of view, this fly ash imparts a darker color to the resulting concrete due to the presence of carbon in the ash [5].

Class C: It is a high-lime ash, containing as high as 24% lime (CaO) content, originating from lignite coal [5]. The total content of silica, alumina and ferric oxide is 50% [3]. High-lime ash has some cementitious properties of its own but, because its lime will combine with the silica and alumina portions of ash, there will be less of these compounds to react with the lime liberated by the hydration of cement. The carbon content is low, the fineness is high, and the color is light. The behavior of high-lime ash is sensitive to temperature: specifically in mass concrete when a rise in temperature occurs, the products of reaction may not be of high strength [5].

When mixed with portland cement and water, fly ash will react with the calcium hydroxide released by the hydration of portland cement to produce various calcium-silicate hydrates (C-S-H) and calcium-aluminate hydrates. These pozzolanic reactions are beneficial to the concrete in that they increase the quantity of the cementitious binder phase (C-S-H) and, to a lesser extent, calcium-aluminate hydrates, improving the longterm strength and reducing the permeability of the system. Both of these mechanisms enhance the durability of the concrete.

Fly ash is normally incorporated in cementitious mixes at ratios ranging from 15% to 25% however higher ratios (30% to 50%) have also been used in mass concrete applications (e.g. dams, foundations) to control temperature rise. In recent decades, even higher levels (40% to 60%) have been used in structural applications, producing concrete with good mechanical properties and durability. Increasing the amount of fly ash in concrete is not without shortcomings. At high levels problems may be encountered with extended set times and slow early strength development, leading to delays in the rate of construction. These drawbacks become particularly pronounced in cold-weather concreting. For any given situation there will be an optimum amount of fly ash that can be used in a concrete mixture which will maximize the technical, environmental, and economic benefits of fly ash use without significantly impacting the rate of construction or impairing the long term performance of the finished product. The optimum amount of fly ash will be a function of wide range of parameters and must be determined on a case-by-case basis [12].

2.3.2 Limestone Powder:

Limestone powder (LSP) is produced as by-product of limestone crushers. Large volumes of this powder are accumulated and it is a big problem. Utilization of this by-product may solve the problems related to disposal, environmental pollution and health hazards. Limestone powder has been used to produce cement in some countries, and in the recent EN197-1 specification, it is mentioned that up to 35% of limestone powder can be added to produce Portland limestone cement and Portland composite cement. The use of limestone powder in concrete, particularly in SCC, has been widespread in Sweden and France, where limestone powder is stored in silos alongside the cement in ready-mix concrete plants. The addition of fine limestone powder can

significantly improve the workability of self-compacting concrete and it has shown to enhance the rate of cement hydration and strength development, as well as to improve the deformability and stability of fresh SCC. Preliminary studies carried out as part of a major European research project on SCC suggested that fine limestone powder could be used effectively in SCC. The SCC mixes containing fine limestone powder showed improved fresh properties and excellent surface finish. This was attributed to improved particle packing and water retention of the fresh mixes, as well as to possible chemical reactions involving cement hydrates and CaCO_3 . [13]

2.4 X-Ray Fluorescence (XRF)

XRF is an experimental technique used to determine the complete chemical analysis of a sample by giving the quantities of the chemical compounds or elements present in the sample. It uses X-rays or Gamma rays bombardment to excite the material under experimentation, thereby resulting in the production of characteristic X-rays from the materials. XRF technique is frequently used for the chemical analysis of cement and SRMs [16]. It provides identification of metallic oxides but is of little consequence for the characterization of organic admixtures.

2.5 Nuclear Magnetic Resonance (NMR):

NMR technique is used for structural determination of molecules. It uses a large magnet to probe the intrinsic spin properties of atomic nuclei. Like all spectroscopies, NMR uses a component of electromagnetic radiation (radio frequency waves) to promote transitions between nuclear energy levels (Resonance). Upon absorption of the frequencies, the nuclei resonate and different atoms within a molecule resonate at different frequencies. This observation allows a detailed analysis of the structure of a molecule, especially of organic origin.

2.6 Fourier Transform Infrared Spectroscopy (FTIR):

FT-IR stands for Fourier Transform InfraRed, the preferred method of infrared spectroscopy. In infrared spectroscopy, IR radiation is passed through a sample. Some of the infrared radiation is absorbed by the sample and some of it is passed through (transmitted). The resulting spectrum represents the molecular absorption and transmission, creating a molecular fingerprint of the sample. Like a fingerprint no two unique molecular structures produce the same infrared spectrum. Therefore, infrared spectroscopy can result in a positive identification (qualitative analysis) of every different kind of material. In addition, the size of the peaks in the spectrum may be direct indication of the amount of material present. This makes infrared spectroscopy useful for several types of analysis. [13]

EXPERIMENTAL PROGRAM

3.1 Materials Used

The following materials were used in the study.

3.1.1 Cement

‘Bestway’ Ordinary Portland Cement (OPC), grade 43, type I manufactured in Pakistan, conforming to ASTM standard C150-04 was used.

3.1.2 SRMs

Class F German Fly ash was obtained and kept in a sealed container to prevent contact with moisture. Limestone powder was obtained as Margalla crush. It was washed, oven dried and ground to powder to be used in cement mixes. It was also kept in a container similar to FA. It was made sure that both SRMs were free from lumps before using them to prepare mixes.

3.1.3 Superplasticizer

Melflux 2651F by BASF Germany, poly-carboxylate ether (PCE) type powder superplasticizer have been used in the study. It is a third generation high performance superplasticizing powder admixture for cement based construction materials. It is especially optimized for plasticization and water reduction of cementitious construction materials which helps in high early strength development. It’s anti-bleeding and segregation properties make it more effective when used with cement.

3.1.4 Acacia Gum (AG)

Acacia Nilotica Gum, indigenously known as Keekar Gum, was obtained locally from a retailer in Rawalpindi, Pakistan.



Figure 3.1: Acacia Nilotica Gum

3.2 Formulations Studied

The following formulations are studied.

Serial #	Cement	FA %	LSP %	AG %	SP %	W/C %	Formulation Name	Formulation Abbreviation
1	C1	0	0	0	0	27	C1-0-0-0-27	0
2	C1	0	0	0.33	0	28.5	C1-0-0.33-0-28.5	0.33AG
3	C1	0	0	0.66	0	32	C1-0-0.66-0-28.5	0.66AG
4	C1	0	0	1	0	34.5	C1-0-1-0-34.5	1AG
5	C1	0	0	0	0.131	27	C1-0-0-0.131-27	0-SP
6	C1	0	0	0.33	0.65	28.5	C1-0-0.33-0.65-28.5	0.33AG – SP
7	C1	0	0	0.66	0.72	32	C1-0-0.66-0.72-32	0.66AG - SP
8	C1	0	0	1	0.80	34.5	C1-0-1-0.80-34.5	1AG - SP
9	C1	10	10	0.33	1.2	28.5	C1-FA10LSP10-0.33-1.2-28.5	0.33AG-FA10LSP10-SP
10	C1	20	0	0.33	0.80	28.5	C1-FA20-0.33-0.80-28.5	0.33AG-FA20-SP

Table 3.1: List of Formulations

The formulations are read as “Type of Cement” - “SRMs (%)” - “Acacia Gum (%)” - “Superplasticizer (%)”- “Water Content (%)”.

The primary focus of the research is on the formulations 5-10 containing SP since the research is dealing with SCPs. However, formulations containing no SP, 1-4, are required to determine the water demands on the basis of which flow tests are performed to determine the SP demand for each formulation and hence establish new formulations (5-10) on which Strength, Calorimetry and Shrinkage tests were performed. Furthermore, Calorimetry test was also performed on formulations 1-4 containing no SP.

3.3 Mixing Regimes

Standard DIN-196 mixing regime was followed for all tests. A Hobart mixer was used for mechanical mixing at two different rpm. The mixing regime is as follows:

- I. Manual addition of cement+additive to water in Hobart mixer. Time for this addition not exceeding 10 seconds.
- II. Slow mixing for 90 sec in Hobart mixer at 145 rpm.
- III. Break for 30 sec. Time for cleaning the sides of the mixer.
- IV. Fast mixing for 90 sec in Hobart mixer at 285 rpm.



Figure 3.2: Hobart mixture

3.4 VICAT Water Demand and Setting Time test:

The water demand and setting times were determined using the standard VICAT apparatus in accordance with ASTM C 187, C 191 and B.S 12 for WD and ASTM C 150 and B.S 12:1978 for setting time.



Figure 3.3: VICAT Apparatus

Water demands of the following formulations were determined.

1. C1-0-0-0-27	3. C1-0-0.66-0-28.5
2. C1-0-0.33-0-28.5	4. C1-0-1-0-34.5

Setting times of the following formulations were determined.

1. C1-0-0-0-27	5. C1-0-0-0.131-27
2. C1-0-0.33-0-28.5	6. C1-0-0.33-0.65-28.5
3. C1-0-0.66-0-28.5	7. C1-0-0.66-0.72-32
4. C1-0-1-0-34.5	8. C1-0-1-0.80-34.5

Setting times for formulations 5-8, containing SP, were determined for the purpose of comparison with Shrinkage and Calorimetry curves.

For determining water demands, the percentage of water content at which the VICAT needle penetrates up to 5 ± 2 mm from the bottom of the mould was noted. For determining the initial setting time, the initial set needle was used and the time taken for the paste to gain sufficient stiffness so that the needle could not penetrate beyond 5 ± 2 mm from the bottom of the mould was noted. For final set time, the final set needle was used and the time taken for the paste to gain enough stiffness so that the needle leaves no circular impression on the cement sample was recorded.

3.5 Flow Test

The flow test is conducted using a standard Hagerman Cone of $6\times 7\times 10\text{cm}^3$ dimensions to determine the flow ability of Self Consolidating Paste as a measure of its spread, with a target of 30 ± 1 cm. The spread is measured by taking two readings in orthogonal direction using a scale or a measuring tape and averaging them to obtain the spread value. The flow test was conducted to determine the SP requirement for the formulations containing AG at varying ratios with respect to the cement content.

The amount of superplasticizer required for each formulation to reach a spread of 30 ± 1 cm was determined. In addition, to estimate the viscosities and yield stresses of the formulations, the T25cm and T30cm times were measured respectively using stop watches.



Figure 3.4: Hagerman mini-cone slump apparatus

3.6 Calorimetry

F-CAL 8000 field calorimeter was used to generate calorimetric curves for the following formulations:

1. C1-0-0-0-27	5. C1-0-0-0.131-27
2. C1-0-0.33-0-28.5	6. C1-0-0.33-0.65-28.5
3. C1-0-0.66-0-32	7. C1-0-0.66-0.72-32
4. C1-0-1-0-34.5	8. C1-0-1-0.80-34.5
	9. C1-FA10LSP10-0.33-1.2-28.5
	10. C1-FA20-0.33-0.80-28.5

Each sample was kept for a period of 120 hours. The purpose of calorimetry is to study the effect of AG and SP on the heat of hydration and reaction kinetics.



Figure 3.5: F-CAL 8000 Calorimeter

3.7 Shrinkage

Shrinkage test was carried out using the modified German Schwindrine apparatus. The apparatus measures the linear shrinkage of cementitious pastes with a sensitivity of 0.31 microns. The shrinkage test is required for determining the effect of varying AG doses on the shrinkage properties of SCPs. The test was performed on the following formulations which were kept for 150 hours in the apparatus:

C1-0-0-0-27	C1-0-1-0.80-34.5
C1-0-0-0.131-27	C1-FA10LSP10-0.33-1.2-28.5
C1-0-0.33-0.65-28.5	C1-FA20-0.33-0.80-28.5
C1-0-0.66-0.72-32	



Figure 3.6: Shrinkage channels



Figure 3.7: Shrinkage channels with sample

3.8 Strength Tests

Compressive and Flexure strength tests were carried out in accordance with EN 196-1: 1994. Samples were prepared in prisms having 4x4x16 cm³ dimensions. The samples were cured and tested in SSD state at the 7th, 14th, and 28th day. The weight of each sample was measured using a weighing machine immediately after demoulding to determine the densities of each sample. The following formulations were prepared and tested:

C1-0-0-0.131-27	C1-0-1-0.80-34.5
C1-0-0.33-0.65-28.5	C1-FA10LSP10-0.33-1.2-28.5
C1-0-0.66-0.72-32	C1-FA20-0.33-0.80-28.5

De-moulding was done 3 days after casting since formulations containing AG experienced a delayed setting time.

3.9 X-ray Fluorescence (XRF)

XRF Analysis was carried out on Axios Advanced WD- XRF PANalytical for all the major elements, using pressed pellet (Semi Quantitative Analysis). The analysis was performed on samples of cement, LSP and FA.

3.10 Fourier Transform Infrared Spectroscopy (FTIR)

Bruker Optics FT-IR Tensor 27 with standard KBr beam splitter technology was used for the analysis of Acacia Gum in the scanning range of 500-4000 cm^{-1} . The FTIR spectra would provide wavelength absorbance peaks for the corresponding functional groups and thus help in determining the various functional groups present in the Acacia Gum structure.

3.11 Nuclear Magnetic Resonance (NMR)

NMR was conducted using 300 MHz AVANCE series Switzerland spectrometer. 20 mg of AG was dissolved in 0.5 ml deuterated solvent to obtain the NMR.

3.12 pH test of AG

Acacia gum was mixed in water at room temperature at various concentrations and its PH was recorded using a pH meter. The acacia gum solution was made in a beaker and mixing was done manually using a stirrer. The results were recorded in the form of a table and a graph was plotted.

RESULTS AND DISCUSSIONS

4.1 Materials Characterization

4.1.1 Acacia Gum

FTIR and NMR tests were performed for the characterization of Acacia Gum. Results of both the tests are attached in Annexure B.

4.1.2 FTIR

From FTIR data functional groups present can be deduced. It can also be used for the determination of the structure of a compound. From the given FTIR it can be evaluated that the broad peak at 3344.8 cm⁻¹ can be due to the presence of water in acacia gum which may be absorbed from the atmosphere. Peaks at 1606.51 cm⁻¹ is due to C=C and 1417.15 cm⁻¹ indicate the presence of C-C stretch of the aromatic groups and 1026.71 cm⁻¹ indicate the presence of C-O bond. Peaks at 772 cm⁻¹ and 621 cm⁻¹ are in the fingerprint region and may be due to the halides ie C-Cl and C-Br respectively.

4.1.3 NMR

Proton NMR (¹H NMR) of acacia gum powder was taken on 300 MHz instrument in DMSO (Dimethyl sulfoxide) solvent. In the given NMR only two peaks can be observed. While rest of the peaks are highly populated or a noise can be observed in spectrum. Peaks in the region of 3.373 ppm represent the presence of electronegative oxygen atom which deshielded the proton on the alpha carbon. Peak at 2.515 ppm may be due to the aromatic group present in the acacia gum powder which are also confirm from double bond equivalent (DBE) formula. Peak at 5.122 ppm represent amide group. However presence of singlet and doublet is not so clear from the spectrum to detect the number of adjacent protons. NMR spectrum is too much noised to determine the exact formula of the acacia gum. So a rough conclusion can be drawn from DBE and the spectrum observed.

As the formula of acacia gum is C₂₆H₃₄N₂O₁₃ , so to determine structure;

Using DBE= $26 - (34/2) + 0.5(2) + 1 = 11$

It shows that at least two aromatic rings are present in the structure. However the number of aromatic rings may be 4 to accommodate the 26 carbons in AG monomer. The rest 7 DBE may represent double bonds.

4.1.3 pH of solution containing AG

The pH test was also performed on AG suspension in water. AG was mixed in water up to a concentration of 40% using a simple beaker and stirrer apparatus and manual stirring. The water temperature was 25°C. AG formed a suspension in water which became viscous with the addition of more AG until a 40% concentration was reached beyond which further addition was not possible.



Figure 4.1: AG suspension in water at 30% concentration



Figure 4.2: AG suspension in water at 40% concentration

The pH was measured using a pH meter and the results are shown in Figure 4.3. The pH behavior was noted to drop with an increase in the gum concentration. Beyond 1% concentration, the pH began to drop below 7.0 into the acidic scale and reached a lowest of 5.06 at a maximum concentration of 40%. It can be concluded that the Acacia Gum is not too acidic to be unsuitable for use in cementitious mixes especially because in cement applications very high concentrations of gum will not be used.

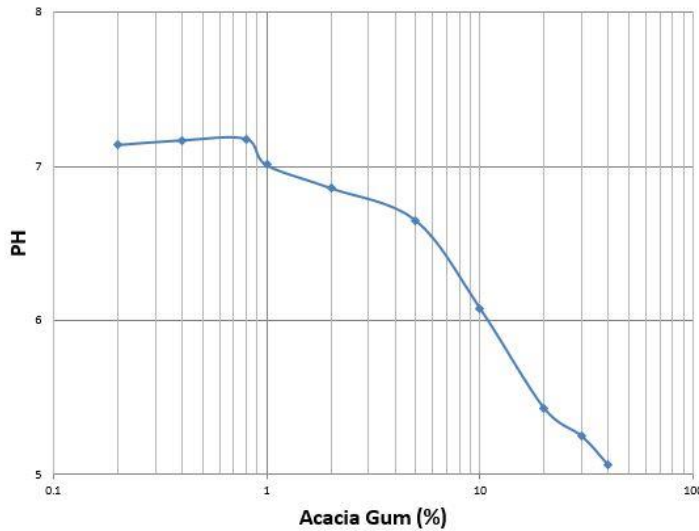


Figure 4.3: pH results with AG variation in water

4.2 Water Demands and Setting Times

The water demands and setting times, determined with the VICAT Apparatus, are shown in Figure 4.4 and Figure 4.5 respectively. The addition of AG increased the water demand of the paste system and also delayed the setting time considerably.

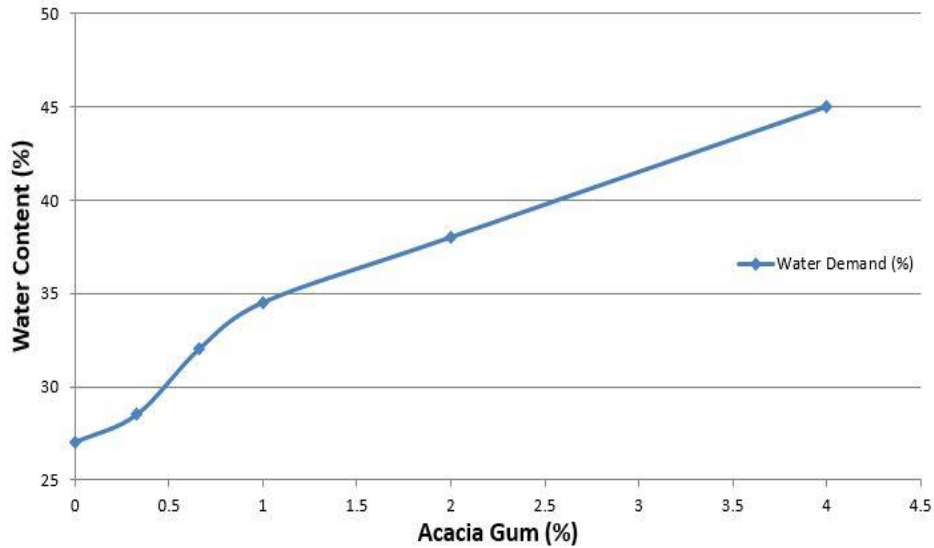


Figure 4.4: Water demand of cement paste with AG variation.

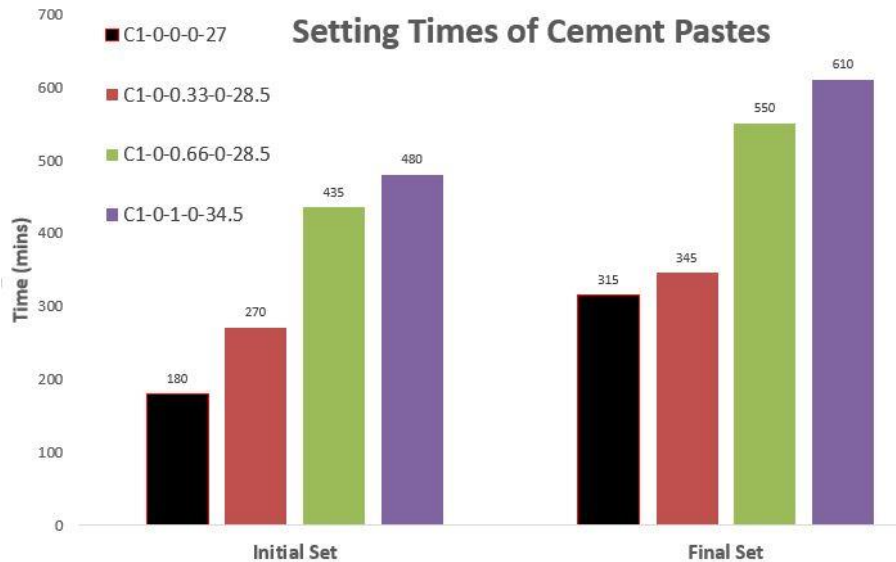


Figure 4.5: Setting times of formulations containing AG in varying ratios

To explain the role of AG in delaying the setting time of cement pastes, it's important to review its chemical composition. As mentioned in the previous section 2.2.2, AG consists of high percentages of galactose, rhamnose, arabinose and glucuronic acid, all of which fail to form

complexes with silicates present in cement [14]. This establishes the fact that the sugars present in AG do not react chemically with the silicates in cement. Rather, the process of hydration is inhibited due to the high loadings of multilayers of organic molecules adsorbed on the silicate surfaces, and the bulk-like characters of the multilayers tend to obscure the influences and interactions of the most-important first adsorption layer that is thought to be principally responsible for inhibiting hydration. The low absolute concentrations of organic compounds, such as saccharides or phosphonic acids, can strongly inhibit silicate hydration, which is attributed to their favorable adsorption properties compared to water at silicate particle surfaces [15]. This behavior explains the delay in setting time experienced in cement pastes containing AG.

4.3 Flow Test of SCPs

The flow test was conducted for each formulation to determine the SP demand for target flow and the corresponding T25cm and T30cm times for each formulation.

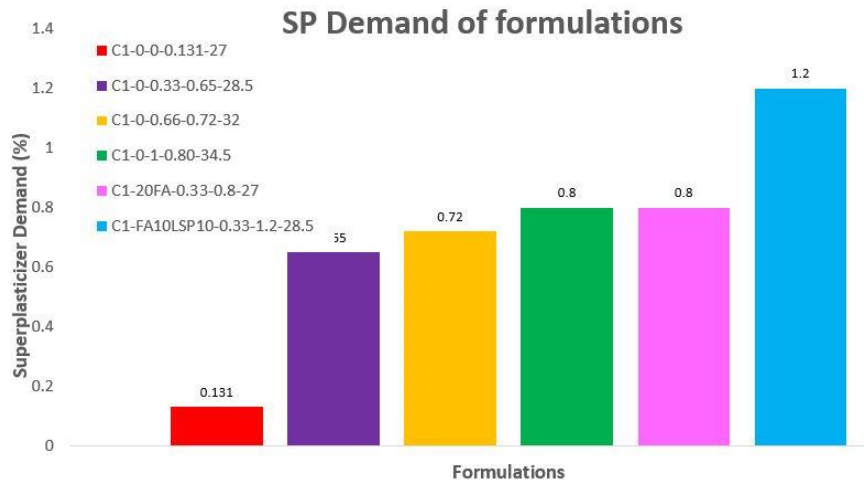


Figure 4.6: SP demand for each formulation

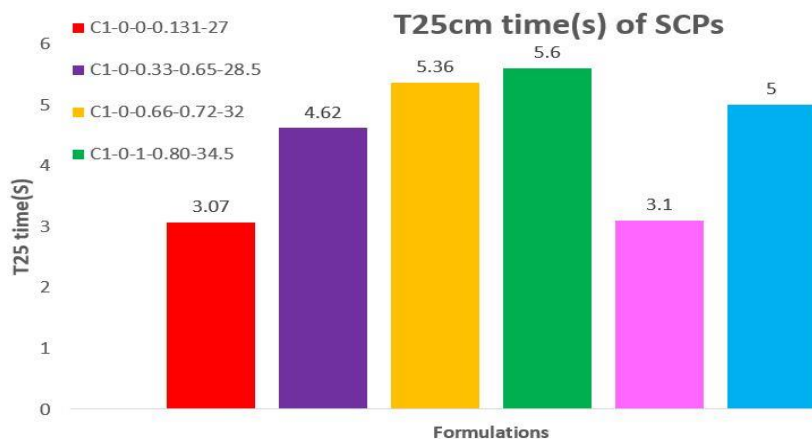


Figure 4.7: T25cm time for each formulation

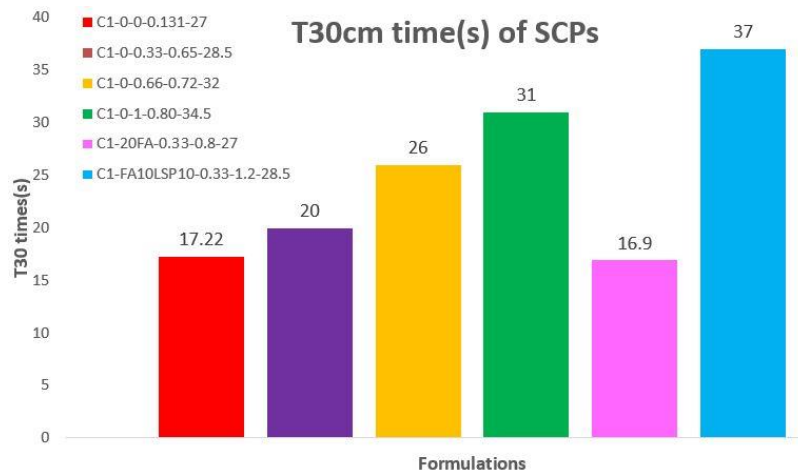


Figure 4.8: T30cm time for each formulation

As it can be observed from Figure 4.6, the SP demand for formulations without mineral admixtures increases as the AG content increases. It was observed during experimentation that the viscosity of each formulation increased with the subsequent addition of AG making the resulting formulations less flowable; thus, requiring greater content of SP to reach a spread of 30cm. T25cm time, which is the time taken to reach a spread of 25cm, also indicated the viscosity increase in formulations containing greater AG content. The results are illustrated in Figure 4.7. Also formulations with greater AG content exhibited greater yield stress which can be represented by the T30s time illustrated in Figure 4.8. T30s is the time required to reach the target spread of 30 cm. Greater the time a formulation takes to reach 30 cm mark, greater is the yield stress.

For formulations containing mineral admixtures, containing the same AG content of 0.33%, LSP increased both the SP demand and the T25 and T30 times. Like in the zero replacement formulations, the increase in SP demand, viscosity and yield stress compared to pure cement paste can be attributed to the addition of 0.33% AG. Whereas, the relative increase in SP, T25 and T30 in formulation with FA10LSP10 replacement compared to the formulation with only FA20 replacement can be attributed to the fact that FA consists of glassy spherical particles that reduce the internal friction of the paste during flow causing greater deformability potential whereas LSP increases the water demand of the system.

Since the addition of AG in SCPs increases their viscosity, it can be recommended for use as a Viscosity Modifying Admixture (VMA) in SCPs; which is the main aim of this study. Due to increase in viscosity, the SCPs exhibited lesser bleeding; rendering the pastes more stable. Such stable pastes would prevent the separating out of particles in the highly flowable mixture and make sure they remain in a single matrix which flows as a single unit. These pastes would enable further stability of subsequent SCMs and SCCs for which further studies are required.

4.4 Setting times of SCP formulations

The flow test established the SP demand for each formulation and consequently their initial and final setting times were determined using the VICAT apparatus again. The setting times are necessary for comparison with shrinkage and calorimetry curves later.

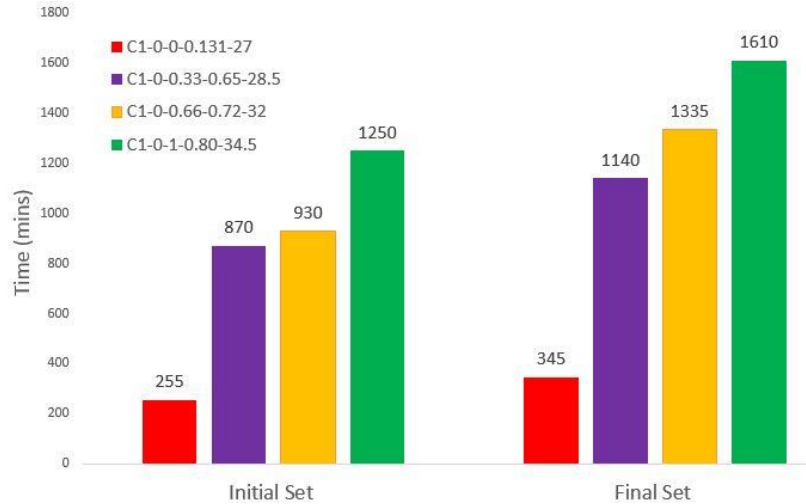


Figure 4.9: Setting Times for SCPs

In continuation with the trend observed in the setting times for conventional cement pastes, both the initial and final setting times of SCPs are delayed with the subsequent addition of AG. In addition, due to the retarding characteristics of SP, the SCPs exhibit an even greater delay in setting times compared to conventional cement pastes. The delay in setting time with the addition of AG is due to the inhibition of the process of hydration caused by the adsorption of organic molecules of AG on the silicate particle surfaces of cement, thereby preventing their contact with water which is necessary for hydration [15].

4.5 Calorimetry

Calorimetry curves were generated for both SCPs and conventional cement pastes to study the effect of AG on the hydration kinetics of cement pastes.

It is evident from these curves in Figure 4.10 and 4.11 that the addition of AG has a significant impact on the heat of hydration and the hydration kinetics of cementitious pastes. In both SCP and non-SCP mixes, the addition of AG has significantly lowered the peak temperature and also considerably retarded the time taken to reach the peak temperature. The retardation process can be explained due to the fact that AG inhibits the hydration of silicates by adsorbing on the surface of silicates thereby preventing contact with water. The greatest retardation was experienced by the SCP formulation containing 1% AG with the peak temperature arriving after around 80 hours. The lowering of peak temperature can also be attributed to the lack of cement-water interaction caused by AG.

Formulations containing SP experienced greater delays in reaching the peak temperature due to the retarding properties of SP [8] and also due to the SP-AG interaction which needs further study.

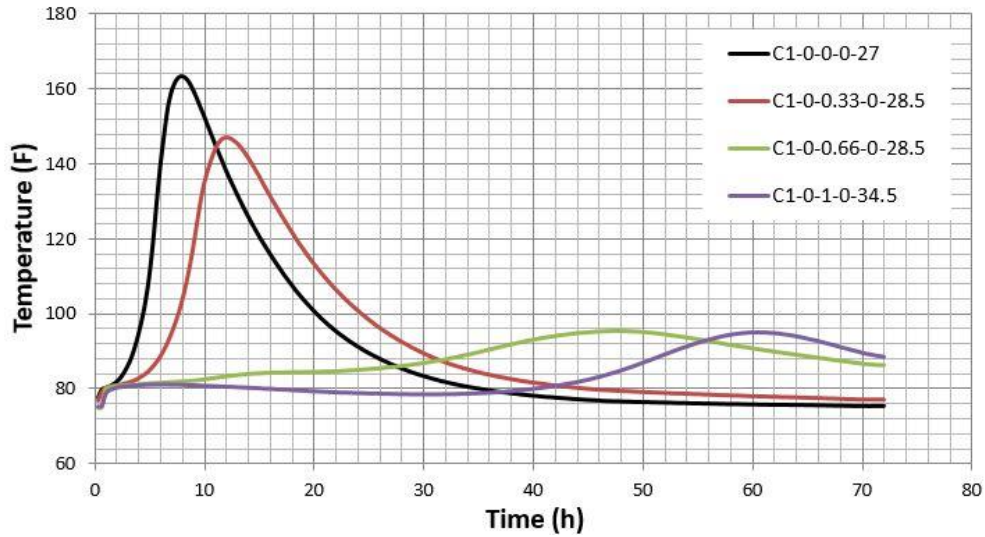


Figure 4.10: Calorimetry of cement pastes (without SP addition)

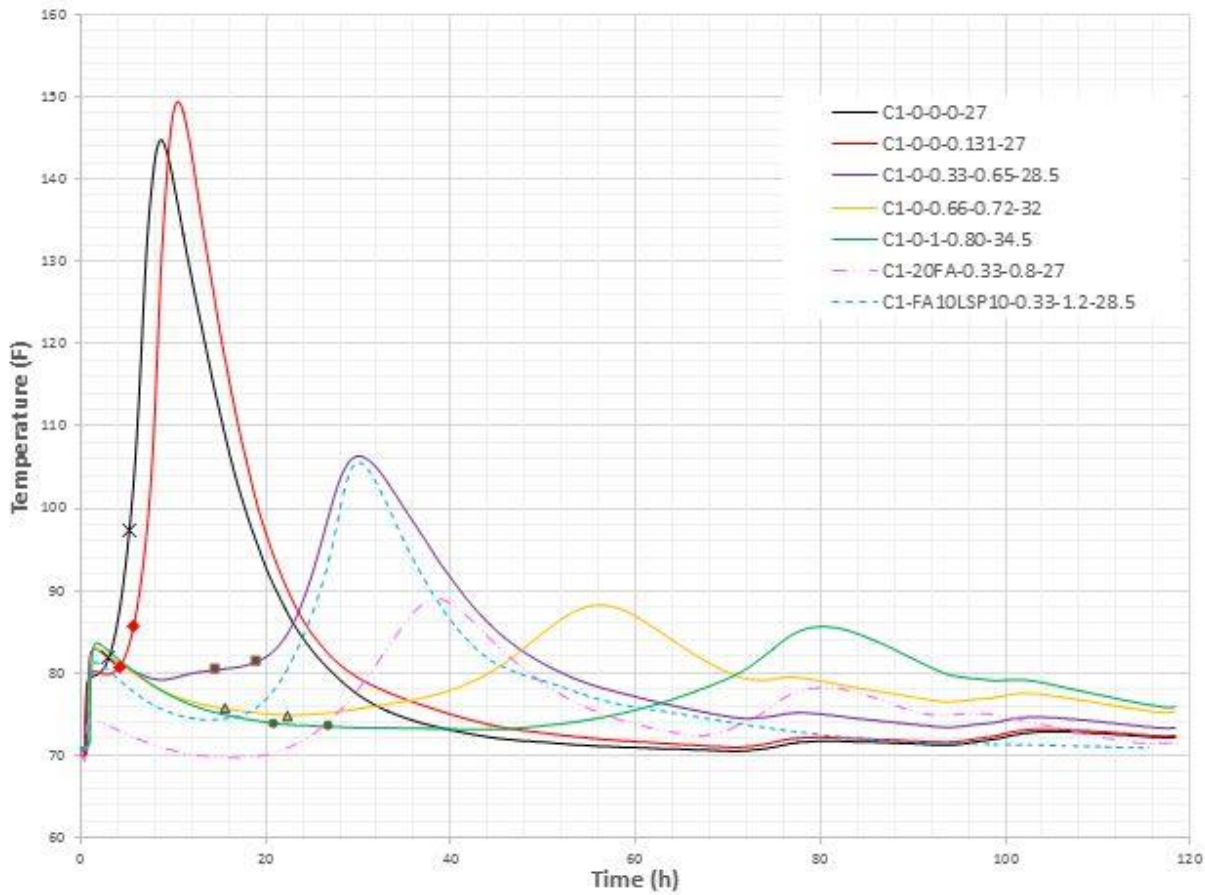


Figure 4.11 Calorimetry of SCPs

Furthermore, SCPs containing LSP and FA showed predictable trends. Formulation containing FA20 replacement showed a delay in peak of hydration due to Fly Ash's property of arresting calcium in cement pastes and thus making it unavailable for reaction in the early stages. The peak temperature is also lowered since cement, which undergoes a highly exothermic reaction, is replaced with a lower heat-producing material. The formulation containing FA10LSP10 replacement showed no delay or acceleration in peak of hydration because the accelerating and retarding properties of LSP and FA respectively cancel each other out.

4.6 Shrinkage

In SCPs the most important parameter after the flow is early volume stability [8]. This parameter is studied using the shrinkage apparatus. The shrinkage response generated for the formulations under study is shown in Figure 4.12.

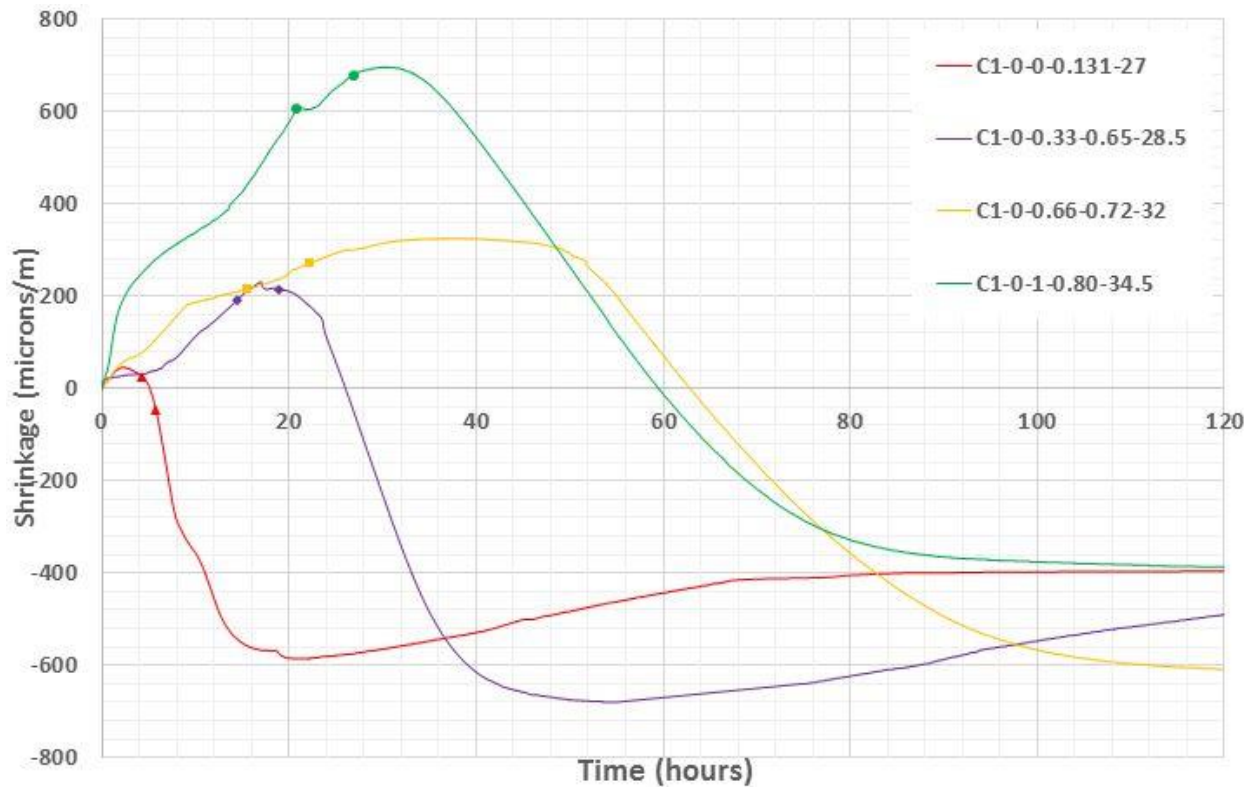


Figure 4.12 Shrinkage Response

It is observed from the shrinkage response that addition of AG causes the cement pastes initially to expand. The expansion response can either be caused by temperature increase or formation of crystals [8]. Since it is known from the calorimetry curves that AG lowers the temperature of mixes rather than increase it, so the early expansion response may be attributed to the formation of crystals in the cementitious matrix. This shrinkage reducing property of acacia gum can be utilized to improve the shrinkage response of SCCs.

The figures 4.11 and 4.12 shows markers on each curve which represent initial and final VICAT setting times respectively. In figure 4.11, the calorimetric response of formulations result in reaching peak temperature soon after encountering the final setting time. This is compared with the shrinkage curve in figure 4.12 which shows an increase in the shrinkage response of formulations soon after encountering final setting times. Thus, the late shrinkage can be related to the late rise in internal temperature of SCPs after they reach their final setting times as evident from the calorimetric curve in figure 4.11.

4.7 Strength Tests of SCPs

Compressive strength results are recorded at the 7th, 14th and 28th day. The results are shown in Figure 4.13 .

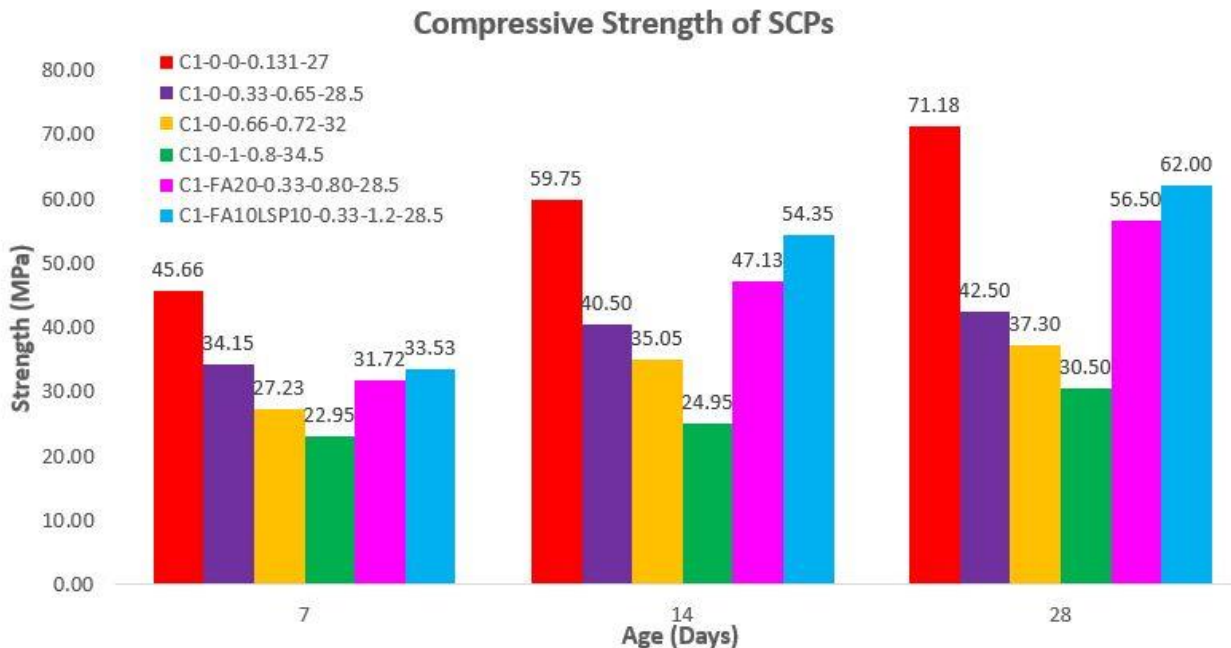


Figure 4.13: Compressive Strengths

The Figure 4.13 shows a decrease in strength with the addition of AG. Pastes with the greatest AG content exhibit the lowest strength. This can be attributed to the lower densities of pastes containing greater amounts of AG as explained in section 4.8. The pastes produced with AG addition showed greater amount of pores in their cross-section which can be seen with the naked eye after breaking the samples with a flexure testing machine, figure 4.14. The increase in porosity with the subsequent addition of AG could be the cause of lower strengths. Also, the fact that AG increased the water demand of formulations can also lower the strength of pastes due to higher w/c ratios.

However, the strength of SCPs containing AG increased with the addition of SRMs. FA10LSP10 replacement resulted in a 31.5% increase in the 28th day compressive strength of SCPs containing 0.33% AG. In other words, 88% of the strength of control sample was attained. Since

formulations containing AG are highly porous, the addition of SRMs decreases the porosity and therefore increases the strength.

4.8 Density

The samples prepared in moulds of 4x4x16 cm³ for strength testing were weighed at the time of de-moulding and divided by the volume of mould to obtain their densities. The results would determine the effect of acacia gum variation on the densities of SCPs.

Figure 4.14 shows a clear trend of reduction in the densities of SCPs with increasing acacia gum content. During experimentation, the formulations containing acacia gum showed a rough texture and the presence of bubbles. Also, when the samples were broken in two halves with the flexure testing apparatus so that their cross-sections are visible, the samples containing acacia gum showed greater porosity as shown in Figure 4.15.

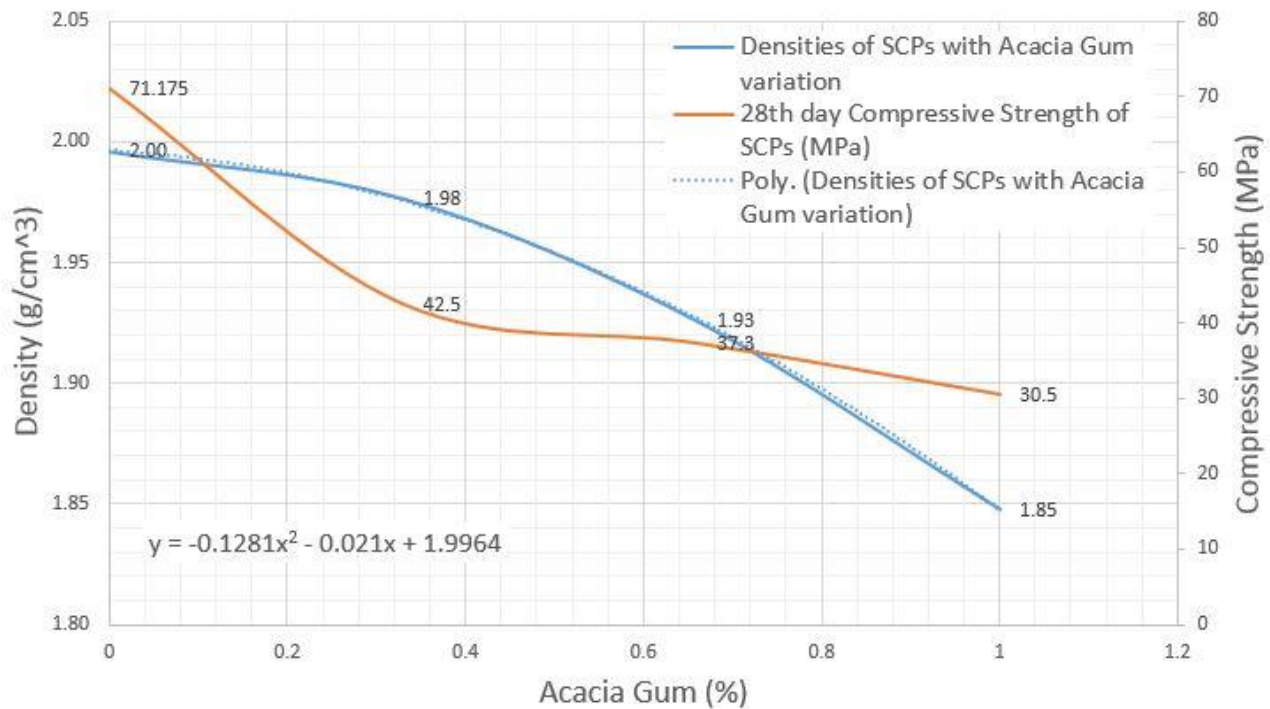


Figure 4.14: Variation in Densities of SCPs with respect to variation in Acacia Gum content

The greater porosity reflects greater air entrapment potential in cement pastes containing acacia gum. This property improves the insulation characteristics of such cement pastes in hardened state. However, the greater porous nature of these pastes is also responsible for their lower strength.

From the discussion, it can be recommended that cement mixes containing acacia gum should be employed in applications where strength is not a major requirement, e.g. partition walls. The air entrainment characteristics would provide advantages for specific applications like improving

insulation and protecting against freezing and thawing action of water in cold climates. In addition, the reduction in weight of cementitious mixes containing acacia gum renders it suitable for preparing light weight concrete which also presents its set of advantages. The lower weight reduces the dead load of the structure which is an important consideration in tall structures and structures erected on weak soils. Also, in framed structures the weights of the walls are carried by the beams and columns. The reduction in the weights of walls would result in considerable economy.



Figure 4.15: Cross Section of SCP in hardened state containing 1% Acacia Gum

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

After careful evaluation and analysis of the results which have been discussed in the previous chapters, the following conclusions and recommendations can be drawn:

Conclusions:

- Acacia Gum increases the water demands of cementitious system
- AG increases the SP demand of SCPs to reach the target spread
- AG increases the viscosity and yield stress of SCPs as observed through T25 and T30 trends, thereby increasing the stability of SCPs. Due to this property, it can be used as a Viscosity Modifying Admixture (VMA) in SCPs to reduce the bleeding of pastes.
- AG lowers the heat of hydration of cement pastes as observed through calorimetry. This property renders it suitable for use in hot weather and mass concrete applications
- AG delays the setting time of cement pastes by adsorbing on the silicate particle surfaces and inhibiting hydration. It is concluded that where cementitious mixes are required to be hauled over long distances, the addition of small amounts of acacia gum would prevent early stiffening of such mixes.
- AG causes early expansion in SCPs as observed through their shrinkage response. This shrinkage-reducing property can be used to improve shrinkage response of SCCs.
- Addition of AG reduces the density and hence increases the porosity of SCPs. The greater porosity, or air entrainment, of SCPs containing AG presents a set of advantages over conventional mixes. The air entrainment improves the insulating characteristics of concrete and also the freezing and thawing resistance in cold weathers. The insulation property may be employed in hollow block concrete construction which is popular nowadays, especially in Karachi. The lower density would reduce the weight of concrete structures lowering the weight of partition walls and hence reducing the dead load requirements of beams and columns thereby reducing their dimensional and material requirements and producing better economy of a structure. Such structures may also be suitable for earthquake resisting due to lighter weight.
- Incorporation of AG reduces the compressive strength of SCPs, however introducing suitable SRMs in imaginative proportions, this trend can be offset.

Recommendations

- Determine an optimum mineral admixtures percentage addition to offset the strength reduction of SCPs caused by AG
- Further chemical analysis should be performed to determine the structure of AG
- Study the effect of AG on the properties of SCMs and SCCs

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Annexure A

Table A-1: Water Demands

Formulation	C1-0-WD	C1-AG0.33-WD	C1-AG0.66-WD	C1-AG1-WD	C1-AG2-WD	C1-AG4-WD
Water Demand	27 %	28.5%	32 %	34.5 %	38 %	45%

Table A-2: Setting Times

Formulation	Initial Setting Time	Final Setting Time
C1-0-WD	180 mins	315 mins
C1-AG0.33-WD	270 mins	345 mins
C1-AG0.66-WD	435 mins	550 mins
C1-AG1-WD	480 mins	610 mins
C1-0-0-0.131-27	255 mins	345 mins
C1-0-0.33-0.65-28.5	870 mins	1140 mins
C1-0-0.66-0.72-32	930 mins	1335 mins
C1-0-1-0.80-34.5	1250 mins	1610 mins

Table A-3: Flow Test Results

Formulation	% SP	T25 (sec)	T30 (sec)
C1-0-0-0.131-27	0.131	3.07	17.22
C1-0-0.33-0.65-28.5	0.65	4.62	20
C1-0-0.66-0.72-32	0.72	5.36	26
C1-0-1-0.80-34.5	0.80	5.6	31
C1-20FA-0.33-0.8-27	0.80	3.1	16.9
C1-FA10LSP10-0.33-1.2-28.5	1.20	5	37

Table A-4: Compressive Strengths

Formulation	7-Day (MPa)	14-Day (MPa)	28-Day (MPa)
C1-0-0-0-27	45.66	59.5	71.18
C1-0-0.33-0-28.5	34.15	40.5	42.5
C1-0-0.66-0-32	24.42	35.05	37.3
C1-0-1-0-34.5	22.95	24.95	30.5
C1-FA20-0.33-0.80-28.5	31.72	47.13	0
C1-FA10LSP10-0.33-1.2-28.5	33.53	52.25	62

Table A-5: PH Test

Acacia Gum (%)	PH @ 23 C
0.2	7.14
0.4	7.17
0.8	7.18
1	7.01
2	6.86
5	6.65
10	6.08
20	5.43
30	5.25
40	5.06

Table A-6: Densities

Sample	Weight	Density (g/cm³)	Acacia Gum (%)
C1-0-0-0.131-27	511	2.00	0
C1-0-0.33-0.65-28.5	506	1.98	0.33
C1-0-0.66-0.72-32	493	1.93	0.66
C1-0-1-0.8-34.5	473	1.85	1

Annexure B Materials

Table B-1: XRF

Sample Name	CEM E-880	LSP E-881	FA E-878
SiO ₂	19.19	3.00	59.06
TiO ₂	0.29	0.04	1.58
Al ₂ O ₃	4.97	0.69	27.58
Fe ₂ O ₃	3.27	0.27	5.14
MnO	0.04	0.01	0.05
MgO	2.23	0.67	1.27
CaO	65.00	52.67	1.66
Na ₂ O	0.58	0.30	0.54
K ₂ O	0.51	0.10	1.59
P ₂ O ₅	0.08	0.00	0.15
LOI	3.84	42.24	1.38

Figure B-5: FTIR - Acacia Gum

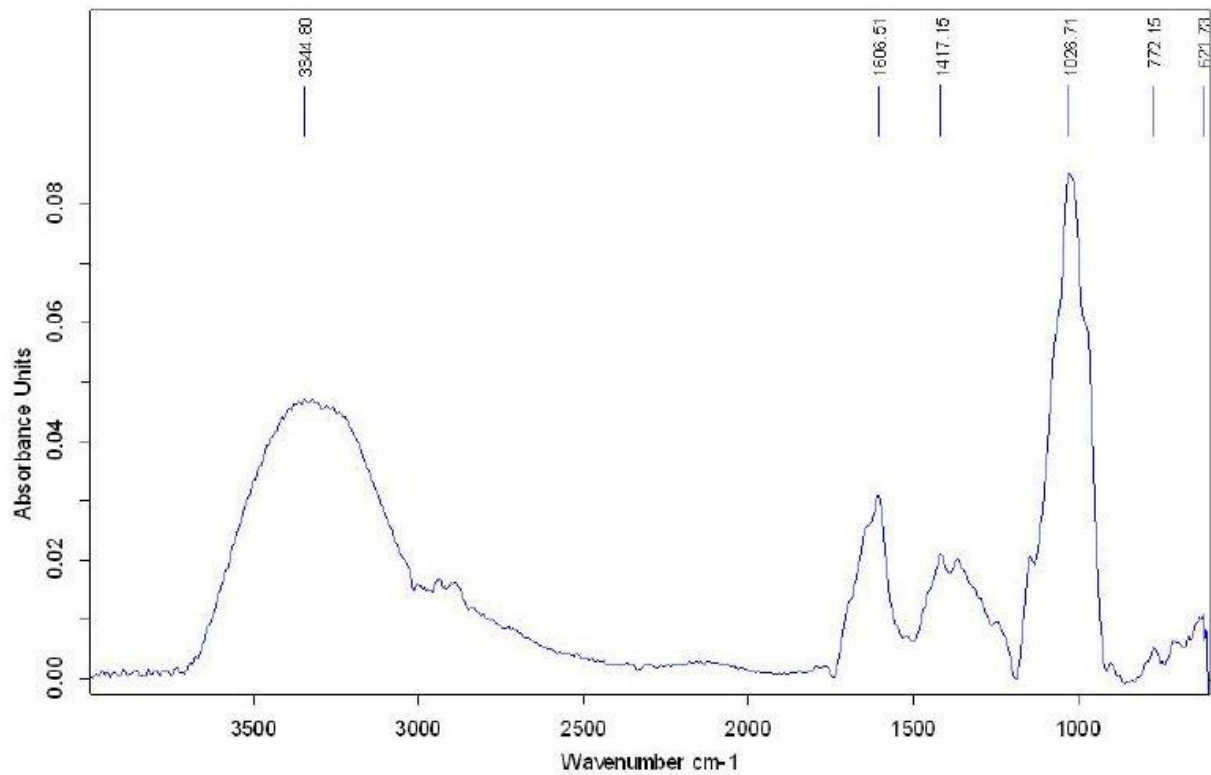


Figure B-6: NMR - Acacia Gum

WJ_1HNMR_DMSO

