EFFECT OF TRIISOPROPANOL AMINE AND CALCIUM NITRATE ON THE
STRENGTH DEVELOPMENT OF GLASS FIBER REINFORCED CONCRETE

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ABSTRACT

EFFECT OF TRIISOPROPANOL AMINE AND CALCIUM NITRATE ON THE STRENGTH DEVELOPMENT OF GLASS FIBER REINFORCED CONCRETE

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This thesis work aims to research the possibility of acceleration in strength development by the use of Calcium Nitrate ($Ca(NO_3)_2$) and Triisopropanol Amine (TIPA) in the production of glass fiber reinforced concrete, also known as GFRC or GRC.

In conventional cementitious systems, $Ca(NO_3)_2$ is used as an accelerator, and TIPA is typically used as a grinding agent for cement. In this thesis study TIPA is used in order to increase the ultimate strength of the concrete whose early strengths are increased by $Ca(NO_3)_2$.

In order to achieve the goal, the required values of the admixtures were prepared and their 6 h compressive and flexural strength were measured. Also their strength development in 7 and 28 days were noted.

The results revealed that TIPA could be used to increase the ultimate strength of GRC. The use of 0.05-0.1% TIPA and 6-8% $Ca(NO_3)_2$ is suitable for gaining the highest early and ultimate strengths.

ÖZ

TRİİSOPROPANOL AMİN VE KALSİYUM NİTRAT'IN CAM ELYAFI İLE GÜÇLENDİRİLMİŞ BETONUN MUKAVEMET GELİŞİMİNE ETKİSİ

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Bu tez, GRC yahut GFRC adı ile de bilinen cam elyafı ile güçlendirilmiş betonun, Triisopropanol Amin (TIPA) ve Kalsiyum Nitrat ($Ca(NO_3)_2$) kullanımı ile dayanım gelişiminin mümkün olup olmadığını tespit etmeyi amaçlamaktadır.

Klasik çimentolu sistemlerde $Ca(NO_3)_2$ priz hızlandırıcı olarak kullanılırken, TIPA genellikle çimento öğütme katkısı olarak kullanılmaktadır. TIPA bu çalışmada, erken dayanımı $Ca(NO_3)_2$ tarafından arttırılmış betonun nihai dayanımını arttırmak amacıyla kullanılmaktadır.

Çalışmada, 6 saatlik maksimum eğilme ve basınç mukavemet değerlerine ulaşabilmek için gereken katkı miktarları tespit edilmiştir. Bu numunelerin 7 ve 28 günlük mukavemet gelişimleri de gözlenmiştir.

Çalışma sonunda, TIPA'nın GRC'nin nihai dayanımını arttırmak amacıyla kullanılabileceği tespit edilmiştir. Sonuçlar, %0.05-0.1 TIPA ve %6-8 $Ca(NO_3)_2$ katkı kullanımıyla GRC'nin en yüksek erken ve nihai dayanımlara ulaşmak için kullanılabileceğini göstermektedir.

Anahtar Kelimeler: GRC, GFRC, cam elyafı, prekast beton, priz hızlandırıcılar, beton katkıları, TIPA, kalsiyum nitrat

ACKNOWLEDGEMENTS

TABLE OF CONTENTS

LIST OF TABLES

TABLES

xiv

LIST OF FIGURES

FIGURES

NOMENCLATURE

Aft: Alumina ferric oxide tri-sulfate

AFm: Alumina ferric oxide mono-sulfate

ANOVA: Analysis of variance

ASTM: American Society for Testing Materials

COV: Coefficient of variation

EN: European Standards

GRC: Glass fiber Reinforced Concrete

GRCA: Glass fiber Reinforced Concrete Association

ITZ: Inter Transition Zone

LOI: Loss On Ignition

MOR: Modulus of Rupture

MPa: Mega Pascal

SEM: Scanning Electron Microscope

TEA: Triethanolamine

TIPA: Triisopropanol amine

W/C: Water to Cement Ratio

XRD: X-Ray Diffraction

CHAPTER 1

INTRODUCTION

1.1. Glass Fiber Reinforced Concrete

Glass Fiber Reinforced Concrete (GRC or GFRC) is a kind of cement based composite material which is reinforced by the use of chopped glass fibers in order have superior flexural and compressive strength. It is regulated internationally by the institute called International Glass fiber Reinforced Concrete Association (GRCA Technical Manuals, 2013).

GRC basically consists of a cement, a fine aggregate (sand), an alkali resistant (AR) glass fiber, water, and if necessary, some co-polymers (acrylic, vinyl acetate, styrene etc.) (GRCA Technical Manuals, 2013). It is generally used for the production of concrete facade and panels used for coating and some esthetical purposes. These coatings can be in the shape of straight panels or they can have a complex geometry. After being manufactured they are fixed to the facade of the building by the use of steel frames which are mounted at the back of these GRC panels.

The GRC products are manufactured in molds specific for the architectural design because of being in different and complex shapes. Taking the panel out of the mold is a challenging process for a conventional concrete since it needs a certain flexural behavior. In order to improve the flexural properties glass fibers are embedded. In the presence of glass fibers concrete behaves like a polymer product and can go through substantial bending as compared to conventional concrete. The use of the same mold for a specific GRC panel limits the production capacity. Therefore, it would be beneficial to increase the speed of production by decreasing the time to take the panel from its mold. Thus, manufacturing a GRC strong enough to pass the regulation limits

while decreasing the costs is among the biggest challenges that engineers face during the production of GRC.

1.2. Objective

The main objective of this study is to research a new and cost effective way for the production of the GRC that gains sufficient strength in about 6 h without using any steam curing or any other equipment. The Triisopropanol amine (TIPA) and calcium nitrate, $Ca(NO_3)_2$, were taken as the admixtures to accelerate the strength. Various combinations of these two admixtures were used in order to maximize the early strength at approximately 6 h. The ultimate goal of the research is to determine the optimum amounts of these admixtures to gain the maximum early strength without compromising the ultimate strength.

1.3. Scope

This thesis consists of five chapters. Chapter 2 presents a literature review and gives a general background on GRC production, concrete admixtures for early strength gaining, and newly discovered admixtures serving for this purpose.

Chapter 3 presents the experimental procedure including the preparation of the samples, the ingredients used in the samples, and the testing procedures. In order to determine the performance of the GRC mixes their compressive and flexural strengths were measured. Also, setting time and slump test results were noted.

Chapter 4 presents the results of the test program focusing on the accelerated setting and the ultimate strength of the samples. Moreover the effect of the admixtures is explained.

Chapter 5 gives the conclusions withdrawn throughout the thesis study. Furthermore, possible future research areas that will complement the findings of this thesis are also included.

CHAPTER 2

LITERATURE REVIEW AND BACKGROUND

2.1. What is Glass Fiber Reinforced Concrete

Glass fiber reinforced concrete, also known as GFRC or GRC, is a type of fiber-reinforced concrete which contains chopped glass fibers that are embedded in a cementitious matrix. GRC is mainly used in the exterior building facade as precast panels. Just like shotcrete, GRC is sprayed and cast into expensive molds that mainly govern the price of the product. Basically, GRC is a kind of shotcrete but, differently, it has an initial setting more than 1 hour, preferably more than 1.5 h because of workability issues, such as spraying and compaction by rollers.

2.2. Constituent Materials

GRC basically consists of a cement, a fine aggregate (sand), an alkali resistant glass fiber, water, and if necessary, some co-polymers such as acrylic, vinyl acetate, styrene etc. In the following subsequent subsections the main ingredients of GRC will be explained.

2.2.1. Alkali-resistant glass fiber

The glass fiber used in GRC have to be alkali-resistant continuous silicate filament fiber developed and formulated to have high strength retention in hydraulic cement environments. The glass fiber must conform to EN 15422:2008 or approved National Standard (ASTM C1666/C 1666M) (GRCA Technical Manuals, 2013). Glass fiber

mainly consists of silica (SiO_2) and some other metal oxides but its alkali resistant behavior depends on the zirconia (ZrO_2) content. It is generally stated that after 19% of zirconia the glass fiber behaves as alkali resistant (Palmer, 2014, GRCA Technical Manuals, 2013).

Figure 1: Appearance of chopped alkali resistant glass fibers

2.2.2. Cement

Usually Portland Cements having high early strength and ultimate strength such as CEM I 52.5 R are used in the production of GRC. Use of blended cements and mineral admixtures are not preferred since they slow down the early strength gain.

2.2.3. Fine aggregates

The aggregates used in the production of GRC must be very fine in order to be sprayed by the air guns. Also aggregate or sand must be washed and dried to remove soluble matter and permit accurate control of the water/cement ratio. The particle shape is usually round that has a smooth surface. For spray GRC the maximum particle size is usually limited to 1.2 mm. However for premix GRC the maximum particle size is limited to 2.4 mm (GRCA Technical Manuals, 2013). In both cases the fine fraction, i.e. sand passing a 150 micron sieve, must be less than 10% of the total weight of sand.

Silica sands are widely used and should conform to the specification given in Table-1. Sands with more moisture content may be used provided that the moisture content and the mix design is altered accordingly. Sands other than silica sands may be used but the producer should provide evidence of their suitability. Soft building sands must not be used (GRCA Technical Manuals, 2013).

Table 1: Specification for silica sand (GRCA Technical Manuals, 2013.

Silica content	>96 %
Moisture content	<2 %
Loss-on ignition	<0.5 %

2.2.4. Water

Water used in the production of GRC must be clean and free from deleterious matter, conforming to EN 1008 or another approved national standard (GRCA Technical Manuals, 2013).

2.2.5. Admixtures

Admixtures are permitted and their use is encouraged as they can enhance the properties of GRC. They should always be used strictly in accordance with the suppliers' recommendations and the producer must ensure that their use has no adverse effect on the product. Calcium chloride-based admixtures must not be used if the GRC component contains steel reinforcement, fixing sockets or other cast-in devices (GRCA Technical Manuals, 2013).

2.2.6. Acrylic polymers

Acrylic thermoplastic polymer dispersions can be used in accordance with the instructions quoted by the manufacturers and should conform to the specifications given in Table 2.

Table 2: Specification for polymer curing aid (GRCA Technical Manuals, 2013)

Compound type	Aqueous thermoplastic polymer dispersion
Polymer type	Acrylic based
Solids	45-55%
Appearance	Milky white, creamy, free from lumps
Minimum film-formation temperature	7-12 C
Ultraviolet resistance	Good
Alkali resistance	Good

The main purpose of using acrylic polymers is to gain extra compressive and flexural strength which otherwise could not be gained by conventional ingredients. The polymers give extra binding force and a flexible behavior to the concrete (GRCA Technical Manuals, 2013).

2.2.7. Pigments

Powder pigments or dispersions may be used to produce colored GRC. The pigments should conform to international or national standards. It must be recognized that color variation may occur (GRCA Technical Manuals, 2013).

2.2.8. Other possible ingredients

Other component materials (e.g. silica fume, metakaolin, fly ash, reinforcing fillers, admixtures, meshes, vinyl acetate), may be added to modify the properties of the mix

unless it is proved that their use will not adversely affect the properties of the GRC (GRCA Technical Manuals, 2013).

2.3. Mix Design of GRC

GRC can be produced with different mix designs according to the production method and desired properties. The mix design must be such that the mechanical properties of the GRC are achieved and that these requirements are consistent with the engineering design of the product. The mix designs presented in Table 3 are intended as a guide; designs falling outside of these guidelines may be acceptable but should be scrutinized before use.

There are 3 main grades of GRC; grade 8, grade 10, and grade 18. Grades are named by the number referring to the minimum value of modulus of rupture (MOR) defined in the standards for GRC. Also a P letter comes with the standard referring to the use of acrylic thermoplastic polymer emulsion in the GRC mix design. As the number increases the MOR increases and the strength of the material is defined by the Water to Cement (W/C) ratio and the amount of glass fiber included in the mix.

Table 3: Mix designs of GRC grades (GRCA Technical Manuals, 2013).

Premix Grade	Description	Aggregate/ cement ratio	W/C ratio	Glass fiber content (% by weight of total	Polymer solids content (% by weight of cement)
Grade 8	General purpose premix	0.5-1.50	0.30-0.40	2.0-3.0%	Nil
Grade 8P		0.5-1.50	0.30-0.40	2.0-3.0%	4-7%
Grade 10	Sprayed premix or high quality cast premix	0.5-1.50	0.30-0.38	2.0-3.5%	Nil
Grade 10P		0.5-1.50	0.30-0.38	2.0-3.5%	4-7%
Grade 18	Direct sprayed GRC	0.5-1.50	0.30-0.38	4.0-5.5%	Nil
Grade 18P		0.5-1.50	0.30-0.38	4.0-5.5%	4-7%

Until 2014, the minimum allowed strengths according to the GRCA was 50 MPa of compressive strength and 18 MPa of flexural strength at 28 days. By the 2015 update of the standard the minimum compressive strength is limited to 80 MPa and the minimum flexural strength is limited to 22 MPa (GRCA Technical Manuals, 2015). These new limits require polymeric admixtures and extra glass fibers to be used in the mix of GRC. The experimental study of this research was initiated and completed before the update of the standard. Therefore, all mix designs are prepared according to the former standard.

2.4. Production Method of GRC

The three grades of GRC could be produced by different ways for various applications as summarized below:

1. A pourable grade (grade 8) containing premixed chopped fibers which can be cast into a mold to form any shape, texture or feature required by the specifier.

2. A sprayable premixed grade (grade 10), similar to the others, which can be sprayed into a mold to form any shape, texture or feature required by the specifier.

3. A sprayable grade (grade 18) containing a high fiber content which can be sprayed into a mold to form any shape, texture or feature required by the specifier.

The pourable grade (grade 8) of premixed material provides a lower strength and is less ductile than the sprayable grades.

The sprayable premixed grade (grade 10) provides a lower strength than the sprayable grade material.

The sprayable grade containing a high fiber content (grade 18) provides a higher tensile strength and is more ductile than the other two grades (GRCA Technical Manuals, 2013).

2.4.1. Procedure of GRC production

Basic steps of GRC production are illustrated in Figure 2. The cement, sand and water is mixed together to form a cement slurry as shown in Figure 2.a. Glass fiber filaments (Figure 2.b) are chopped and mixed with the slurry. By the use of an air gun (Figure 2.c) the slurry prepared is sprayed into specific molds (Figure 2.d) having certain shapes arranged according to the architectural design.

(a) The slurry being mixed in the tank   (b) Glass fiber filaments used in GRC

(c) Air gun used for spraying the premix   (d) Spraying procedure and the mold

Figure 2: Basic steps of GRC production

A typical example of a complex shaped mold and GRC panel taken out of this mold is shown in Figure 3.

(a) A mold in a complex geometry.
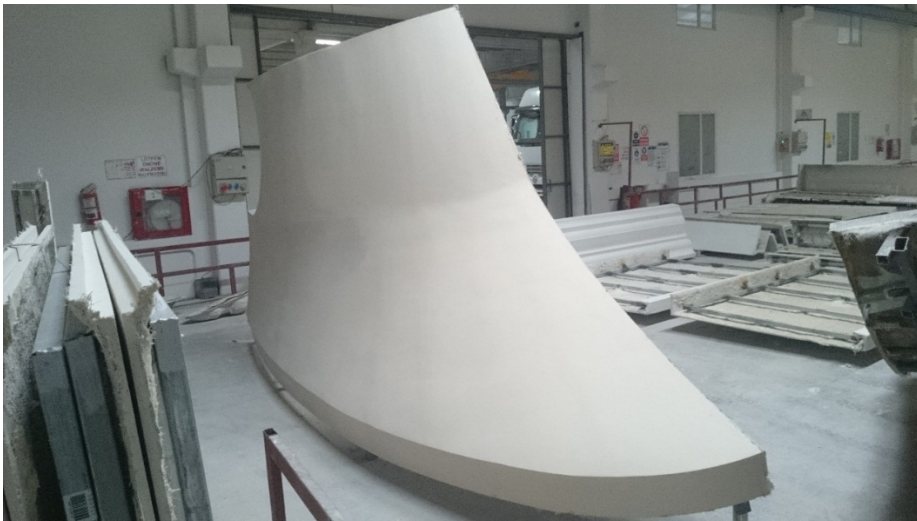
(b) A GRC panel taken out of the mold.

Figure 3: Complex shaped mold and GRC panel.

Whatever the shape is, the thickness is usually limited to reduce the weight of the panel. The thickness of 1.8 cm is commonly taken as the standard depth of the hardboard panels used during the production of the molds. After spraying, the GRC in

the mold is vibrated in order to have a certain compaction. Then, steel frames are anchored to the back side of the GRC panel and then the molds are stored in ambient conditions for 24 h before taking the GRC panel out of the mold. 24 h is needed in order to have enough strength to take the concrete out of the mold properly. During this process a crane which holds the steel frames of the GRC panel is used. While taking out the mold the GRC is subjected to tensile stresses which can cause cracking. Therefore, GRC needs a certain flexural behavior. Glass fibers are used in this kind of material to achieve the required flexibility.

2.4.2. Disadvantages of the current production technique

The common production technique of GRC comes with a definite disadvantage as it needs specific molds designed for each project. The molding process is a very difficult, sensitive and long process in itself and increases the costs.

Another reason that increases the cost of the GRC panel is the extended (24 h) curing process which means every mold can be used only once a day. Since the molds are expensive and there is always a deadline, the curing process increases the molding costs enormously. In other words, fibers, molds and the production rate determines the costs. The glass fiber must be alkali resistant in order to have a high service life, thus it is always expensive and there are only a few producers in the world. A decrease in the glass fiber cost is almost impossible. Therefore, if a technique is developed to increase the production rate, the costs of the GRC panel can be decreased. For example, if a mold can be used twice a day, the molding cost is decreased by 50%. Accelerating the early strength of GRC such that it can be taken out of the mold within 6 h, enables producers to use the same mold twice a day.

2.5. Improving the Early Strength of Concrete by Admixtures

The present economic constraints require acceleration in the speed of work in the construction industry. The need for a concrete with sufficient strength at a very early age is in many situations very important. Such concrete is obtained through the use of some admixtures such as water-reducing admixtures, set accelerators and hardening accelerators. Standards about admixtures for concrete and mortar differentiate between set and hardening accelerators namely;

– Set accelerator is defined as an admixture that decreases the initial setting for the transition of the mix from the plastic to the rigid state.

– Hardening accelerator is defined as an admixture that increases the rate of development of early strength in the concrete with or without affecting the setting time (Aggoun et. al., 2008).

Various kinds of materials are used for accelerating the concrete's setting. Some additives are blended in the dry mix, and some are admixtures which are added into the concrete's mixing water. Most of these admixtures aim to decrease the water requirement of the concrete and therefore increase the early strength of concrete. But since GRC has no coarse aggregate inside and it has a higher amount of cement content in spite of having a low W/C ratio, the water content can't be decreased more as it can cause micro cracks causing a dramatical decrease in the ultimate strength which is not desirable. GRC has to gain at least 50 MPa of compressive strength within 28 days as stated in the GRCA standards. Also the admixture must be liquid or at least soluble in water, since solid admixtures are difficult to blend in the silos used in the production technique.

2.5.1. Superplasticizers

Most common admixtures used in the production of GRC are super plasticizers which are polycarboxylates and melamine sulfonates. They both decrease the water

requirement by repelling the molecules and increasing the workability of the slurry. They should not be used more than a certain amount as liquid content is limited in the GRC production. Therefore, they are not considered as the correct admixtures for increasing the strength below 6 h.

2.5.2. Chlorides

Calcium chloride has been the most widely used set accelerator in this field. However, the presence of chloride causes serious problems regarding corrosion of reinforcing bars embedded in concrete members. This has renewed interest to develop a number of chloride-free admixtures. Non-chloride, non-corrosive set accelerating salts was reviewed. It was realized that the calcium formate, $Ca(CHO_2)_2$, could be used as a set accelerator (Dodson, 1965).

2.5.3. Calcium nitrate

The second salt to fulfill requirements is calcium nitrate, $Ca(NO_3)_2$, which is patented in 1969. It is commonly known to be an agricultural fertilizer and it is very cheap. The calcium ions accelerates formation of calcium hydroxide thus precipitation and setting. This is why calcium nitrate is commonly used as an accelerator for concrete. It was initially assumed that the accelerating effect was dependent on the aluminate content of the cement. This assumption was not verified for cements at low temperature (5°C). The results obtained revealed that calcium nitrate does function as a set accelerator for cement between 7 and 20 °C, but the set accelerating efficiency of calcium nitrate depends very much on the cement type used. It was also noted that the set accelerating efficiency of calcium nitrate seems to increase with increasing belite content in cement or other cement characteristic promoting belite formation in the clinker process (Angtadt and Hurley, 1969, Justnes, 1995). This phenomenon is the reason why calcium nitrate loses its effect after the early age of the strength development. It is also a very effective corrosion inhibitor for metal embedded in concrete. The nitrate ions

lead to formation of iron hydroxide, whose protective layer reduces corrosion of the concrete reinforcement (Al-Amoudi et. al., 2003).

2.5.4. Triethanolamine (TEA)

Triethanolamine (TEA), a tertiary alkanolamine which has a low molecular weight, is used as a grinding aid in cement manufacture and as a constituent in certain admixture formulations in concrete practice. Depending on the cement type and addition rate, TEA can produce either set acceleration or retardation. Addition of 0.02% to the type I Portland cement, TEA acts as a set accelerator, at 0.25% as a mild set retarder and at 0.5% a severe retarder and at 1% a very strong accelerator (Rixon,Mailvaganam, 1999). The effect on the strength development in cement pastes is also dependent on the added amount of TEA (Heren and Ölmez, 1996).

2.5.5. Triisopropanol amine (TIPA)

A patented material named as Triisopropanol amine (TIPA) is reported to provide an increased strength for the concrete at 7 and 28 days (Gartner and Myers, 1989). TIPA is basically a tertial amine. Basic chemical expression of TIPA is $C_9H_{21}NO_3$. It is simply a molecule which has nitrogen in the center and 3 alcohols around it. A TIPA molecule is shown in Figure 4. TIPA is most commonly used in cement industry as a grinding agent. But it is also used as surfactant, emulsifier, stabilizer, neutralizer in chemical industry (DOW Chemical Product Manual, 2014).
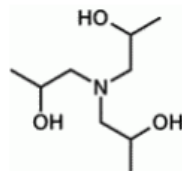
Figure 4: TIPA molecule.

The addition of small amounts of TIPA results in interesting increases in the strength of cement pastes at different ages (Gartner and Myers, 1993). A recent study carried out on the strength enhancing mechanism of TIPA presented compressive strength data for 10 Portland cements tested as cement paste after 28 days of hydration. The average strength improvement with 200 ppm TIPA added to the mix water was 10% (Sandberg and Doncaster, 2014).

2.6. Previous Studies

Because of being a relatively new subject, only a few researchers investigated the usage of TIPA as an admixture. One of the most significant research about this subject was presented by Aggoun et.al. in 2006. They investigated the effect of using some admixtures such as calcium nitrate, TIPA and TEA, on the setting and hardening process of cement pastes at 20 °C. Tests were performed on samples from various mixes considering two types of cements. The results showed that calcium nitrate acted mainly as a setting accelerator with efficiency depending on the cement chemical composition. In the long term, its effect on the strength increase was insufficient to be considered as a hardening accelerator. Regardless of the cement type used, both TEA and TIPA performed well as a hardening accelerator at all ages. The combination of calcium nitrate with either TEA or TIPA resulted in their joining effects with time, translated by a reduction in the initial and final setting time and a strength enhancement at all ages of the cement pastes, particularly at early ages. Used with equal dosage, TIPA is far more efficient in terms of strength increase than TEA. In detail, they reported that calcium nitrate works with low C_3S containing cements, and TIPA accelerates better than TEA. It was reported that for the cements having higher C_4AF TIPA has a retarding effect by about 40%. By the addition of 0.05%, it also improved considerably the compressive strength at all ages about 185% at 1 and 3 days, 130% at 7 days and 100% at 28 days. The joining effects were also illustrated with the use of calcium nitrate in combination with TIPA. The initial and final setting times compared with control mix values were reduced by 45%. But the increase in

compressive strength for short and long term ages was respectively more than 90% for the lower C_3S containing cement.

In another study, Perez et al. (2003) recently proposed that TIPA does not improve the mechanical properties of hydrated portland cement paste, but rather improves mortar and concrete strength by acting on the interfacial transition zone (ITZ) between the portland cement paste and sand or aggregate. Perez et al. based their conclusion on studies of a model portland cement that did not display any increase in the degree of hydration or compressive strength in the presence of TIPA, but displayed significant strength enhancement of mortar made with the model portland cement and limestone.

Ichikawa et al. (1997) presented evidence that TIPA, besides enhancing the hydration of ferrite and alite, also promotes the hydration of limestone, thereby indicating an effect of TIPA on the ITZ between portland cement paste and limestone fines in addition to the effect of TIPA on hydrated portland cement paste without limestone fines.

Sandberg and Doncaster (2003) presented data on the compressive strength of hydrated portland cement paste and mortar after 28 days hydration, thereby addressing the effect of TIPA on the mechanical properties of hydrated portland cement paste and, indirectly, the effect of TIPA on the transition zone between the paste and siliceous sand. The 28-day compressive strength testing of parallel samples of hydrated portland cement paste and mortar using 10 different portland cements resulted in significant strength gains in both paste and mortar treated with TIPA. The average strength improvement with TIPA was 10% in the hydrated portland cement paste and 9% in the mortar. The results clearly show that the strength enhancement is not dependent on an ITZ mechanism. The observed strength gain in hydrated portland cement paste confirms that TIPA is able to enhance the mechanical properties of hydrated portland cement paste without any paste–aggregate ITZ being present.

Gartner and Myers (1993) proposed that TIPA is a facilitated transporter that chelates Fe^{3+} from the hydration product of ferrite and then releases it into the aqueous phase, increasing the dissolution of ferrite and promoting the overall silicate reaction.

18

Hong and Xiaodong. (2010) presented a research investigating the hydration mechanism of a P.II 52.5R cement in the presence of TIPA, glucose or both. Calorimetry tests showed that the interaction effect of TIPA and glucose significantly enhance the degree of hydration of the cement after 7 days. X-ray diffraction analysis confirms that the addition of glucose promotes C_4AF dissolution by TIPA. The accelerated C_4AF hydration yields a reduced CH content and an increased amount of chemically combined water. Due to the delayed acceleration effect of glucose, the hydrate products are characterized by a high surface area. The presence of TIPA also favors the aluminate reaction, reflected by the accelerated AFt (alumina, ferric oxide, tri-sulfate) to AFm (alumina, ferric oxide, mono-sulfate) conversion. For the pastes containing TIPA, the hydration products comprises AFt, AFm and hemicarboaluminate. In the cement with both TIPA and glucose, the hydration of C_4AF is inhibited before 4 days. However, it is significantly promoted afterwards compared with TIPA alone. The interaction effect of TIPA and glucose promotes the hydration of cement up to 28 days, as validated by calorimetry and thermal analysis. The promoted hydration results from the addition of glucose, which strengthens the ''facilitated transportation'' effect of TIPA and accelerates ferrite dissolution. The pore structure of the cement paste is modified by this interaction effect, as well. TIPA alone tends to reduce the specific surface area of the paste and increases the average pore diameter. The addition of glucose, on the contrary, enhances the specific surface area and reduces the average pore diameter. The interaction effect of TIPA and glucose is more pronounced, and the corresponding paste has the highest specific surface area and the lowest average pore diameter at 28 days.

CHAPTER 3

EXPERIMENTAL PROCEDURE

In order to accelerate the GRC's early strength at 6 h without compromising the ultimate strength, TIPA and $Ca(NO_3)_2$ were added into common GRC mix. Compressive and flexural strength tests were carried out to determine the suitability of the resulting material to the standards. Strength test results were evaluated to see whether 6 h of molding is enough to take out of the piece from the mold or not. For each mixture, 3 samples with the nominal dimensions of 5x5x5 cm (width x depth x length) for compressive strength test and 3 samples with the nominal dimensions 4x4x16 cm (width x depth x length) for flexural strength test were prepared. In the preparation stage of the samples, the GRC samples were manufactured by manual molding without the use of air gun. For all the mixes the same production technique but different admixture amounts were applied. Compressive and flexural strength tests were conducted after 6 h, 7 and 28 days of sample production. The results were compared with the standard mix which is treated in the same procedure with other samples.

3.1 Material Properties

The ingredients, their supplier, and amount to prepare the GRC mixes are given in Table 4. For every mix, the amounts of the cement, sand, water and glass fiber were kept constant whereas the amounts of TIPA and $Ca(NO_3)_2$ were varied.

Table 4: Mix ingredients, their supplier, and amount.

Ingredient	Type	Amount (g)
Cement	ÇİMSA CEM I 52.5 R White	1750
Sand	ÖZTAŞ 40-45 AFS Silicate	2250
Water	Tap water	650
Glass fiber	AR class	60
TIPA	BASF Amix TI %99.5	variable
$Ca(NO_3)_2$	Basic agricultural fertilizer	variable

According to the GRCA Technical Manuals, 2013, the minimum amount of glass fiber must be more than 3%. But, since the slurries used in the study were prepared by a simple mixer and during the molding an air gun is not used, in order to assure a sufficient workability for the proper mixing, the glass fiber amount was reduced to 1.5% which resulted in a sufficiently workable mix for manual molding. Hence, a decrease in the flexural strength of the samples is expected because of the reduction in fiber amount. However, flexural behavior depends mainly on the amount of the glass fibers in the mix not the admixtures thus the increase in the compressive strength is expected to be more dramatical.

The aggregate used in the preparation of the samples was 40-45 AFS silica sand. Its chemical composition and size distribution are given in Tables 5 and 6, respectively.

Table 5: Chemical composition of the sand used in the samples.

SiO_2	Fe_2O_3	Al_2O_3	K_2O	Na_2O	CaO	MgO	LOI
97.17	0.38	1.78	0.22	0.05	0.03	0.03	0.31

Table 6: Size distribution of the sand used in the samples.

Sieve	%
1.0	0.18
0.71	3.03
0.5	8.42
0.355	29.97
0.25	39.95
0.18	16.25
0.125	1.69
0.09	0.42
0.063	0.10
Bottom	0.00
Total	100.00

The TIPA used in the preparation of the samples was a product of BASF chemical company. It has a commercial name of Amix TI which contains 97% Triisopropanol amine. The technical specifications of TIPA used are given in Table 7.

Table 7: Technical specifications of Amix TI (BASF Product Manual, 2007).

Ingredient	Value
Triisopropanol amine	97% (min)
Water	1% (max)
Melting Temperature, ºC	39.00
Boiling Temperature, ºC	240-370
Density @20ºC, g/cm^3	1.01
Flash Point, ºC	155
Ignition Temperature, ºC	295

The cement used in the preparation of the samples was a white Portland cement labelled as CEM I 52.5 R according to EN197-1. The technical specifications of the cement are given in Table 8.

Table 8: Technical specifications of the cement

	Value	Standard or Test Method	Minimum Allowed Value	Maximum Allowed Value
Chloride content, %	0.0119	EN 196-2		0.10
Sulfur dioxide content, %	2.83	XRF		4.00
Whiteness, %	86.9	EN 196-1,3	-	-
First Setting, min	115	EN 196-1,3	60	-
Expansion, mm	1.0	EN 196-1,3	-	10
2 day compressive strength, MPa	33.3	EN 196-1,3	30	-
28 day compressive strength, MPa	52.6	EN 196-1,3	52.5	62.5

3.2. Sample preparation & experimental procedures

The GRC samples were manufactured by manual molding without the use of air gun. No steam curing was applied. The specimens that were prepared for 6 h of testing were left in lab conditions whereas the samples that will be tested at 7 and 28 days were kept in the water tanks at 20 °C during the curing period.

3.2.1. Setting time

In order to determine the final setting time of the samples, a manual Vicat Needle was used. Vicat apparatus has a steel needle in the form of a right cylinder of effective length 50 ± 1 mm and diameter 1.13 ± 0.05 mm which is moving upwards and

downwards in order to determine the plunge depth over the specimen. The total mass of moving part must be 300 ± 1 g. Test method starts with filling the circular mold by paste of standard consistence and after getting a flat surface over it, the mold is placed into the Vicat apparatus. The needle must be over the molded specimen at conveniently spaced position, not less than 10 mm from the rim of the mold or from each other. After cleaning the Vicat needle, it is dropped down over the specimen in order to plunge into the specimen and the depth of plunge is measured. This measurement is repeated at conveniently spaced intervals of time, e.g. at 15 min intervals. Initial setting time is the time elapsed from time zero to the time at which distance between the needle and the base-plate is 4 ± 1 mm. Final setting time is the time elapsed from time zero to the time at which the needle first penetrates only 0.5 mm into the specimen.

3.2.2. Flow test

Flow test is applied to determine the consistency of concrete where the nominal maximum size of aggregate does not exceed 38 mm using flow table apparatus. Before commencing the test, the table top and inside of the mold was wetted and cleaned of all gritty material and the excess water was removed with a rubber squeezer. The mold was firmly held on the center of the table and filled with concrete in two layers, each approximately one-half the volume of the mold and rodded with 25 strokes with a tamping rod, in a uniform manner over the cross section of the mold. After the top layer was rodded, the surface of the concrete was struck off with a trowel so that the mold was exactly filled. The mold was then removed from the concrete by a steady upward pull. After that, the table was raised and dropped from a height of 12.5 mm, 15 times in about 15 s. The diameter of the spread concrete is the average of six symmetrically distributed caliper measurements read to the nearest 5 mm.

3.2.3. Compressive and flexural strength tests

The prepared samples were tested after 6 h, 7 and 28 days of production using a computer aided strength test apparatus (Alşa CP5/80 type uniaxial strength tester) in order to determine the flexural and compressive strengths. A consistent and continuously increasing force was applied to the test specimens by a hydraulic testing machine. The maximum force required to break the sample and the actual dimensions of each sample were noted. In this study samples with the nominal dimensions of 5 x 5 x 5 cm (width x depth x length) for compressive strength test and samples with the nominal dimensions 4 x 4 x 16 cm (width x depth x length) for flexural strength test were used.

After molding the samples, the first two set of experiments were done after 6 h. No curing was applied to the samples. But for the third set of the experiments, the samples were cured for 7 and 28 days at room temperature in tap water. Later, they were dried in the oven and then exposed to compressive and flexural strength tests.

The compressive strength was calculated by dividing the maximum load to the cross sectional area of the sample according to the formula given below.

$$\sigma = \frac{F}{A}$$

Where σ is the compressive strength (MPa), F is the maximum load (N) and A is the cross sectional area (mm^2).

The flexural strength was measured by a 3 point bending setup. The MOR was calculated according to the following formula:

$$\sigma_f = \frac{3FL}{2bd^2}$$

Where, σ_f is the flexural strength (MPa), F is the maximum load (N), L is the distance between the legs (mm), b is the width (mm) of the sample, d is the thickness (mm) of the sample.

3.2.4. Microstructural Characterization

After the strength measurement tests, the strongest and the weakest samples were characterized by X-Ray Diffraction (XRD) and scanning electron microscope (SEM) techniques.

3.2.4.1. X-Ray diffraction (XRD)

An X-Ray Diffractometer (Rigaku D/MAK/B) which is available in METU Metallurgical and Materials Engineering Department was used at the speed of 2Θ/min in order to identify the phases present and mineral composition of samples. X-Ray diffractograms of the selected samples were evaluated in Rigaku-4.2 software with control of PC directly linked to diffractometer by using the Inorganic Crystal Structure Database (ICSD) of International Center for Diffraction Data (ICDD) and Rigaku Database.

3.2.4.2. Scanning electron microscope (SEM)

FESEM Nova Nanosem which is available in METU Metallurgical and Materials Engineering Department was used in high vacuum mode to investigate micro morphology of the selected samples. The samples were dried for 2 h at 60 ºC prior to analysis. The SEM images represent the typical morphology of the samples.

3.3. GRC Compositions

The optimum value for TIPA is 0.05% of cement amount (Aggoun et. al., 2008). Therefore, for the first set of the experiments TIPA amount was set at 0.05%, while changing $Ca(NO_3)_2$ amount from 0 to 14%. A W/C ratio of 0.37 was selected that will yield sufficient workability. The mix features for this set are presented in Table 9. All samples were tested for their flexural and compressive strength after 6 h of the

production. In order to express the contents of the mixtures, code names referring to the ingredient amounts were used. Every mixture has a code name starting with a C and T that is followed by the % amount of the calcium nitrate and TIPA. For example the sample C0-T0 has neither calcium nitrate nor TIPA in the mixture and C3-T25 indicates that there's 3% calcium nitrate and 0.025% TIPA in the mixture.

In the second step, in order to investigate the combined effect of TIPA with $Ca(NO_3)_2$, variable amounts of the two admixtures were used in the samples. In the preparation of these samples the peak point of $Ca(NO_3)_2$ that was obtained in the first set of the experiments was considered. This set of the experiments gave a matrix and a surface graph of the results which may be helpful to understand the behavior of the GRC mixes. The mix features of the second set of experiments are given in Table 10.

According to the results of the second set of the experiments a third set of the experiments were designed in order to assess the standard 7 and 28 days strengths of GRC mixes. For the third set of experiments 5 samples were produced again. These samples had the highest compressive strengths gained within 6 h and were taken as the control samples. The chosen mixes are labelled with a * in Table 10.

After obtaining the maximum and the minimum values, the chosen two samples were characterized with X-Ray Diffraction Spectroscopy (XRD) and Scanning Electron Microscopy (SEM) techniques.

Table 9: Ingredients used in the preparation of the samples tested in the first set of the experiments.

| Sample | Ca(NO$_3$)$_2$ | | TIPA | | Water | Cement | Sand | Glass |
	%	g	%	g	(g)	(g)	(g)	fiber (g)
C0-T0	0	0.00	0	0.00	650	1750	2250	60
C0-T5	0	0.00	0.05	0.88	650	1750	2250	60
C1-T5	1	17.50	0.05	0.88	650	1750	2250	60
C1.25-T5	1.25	21.88	0.05	0.88	650	1750	2250	60
C1.50-T5	1.5	26.25	0.05	0.88	650	1750	2250	60
C1.75-T5	1.75	30.63	0.05	0.88	650	1750	2250	60
C2-T5	2	35.00	0.05	0.88	650	1750	2250	60
C3-T5	3	52.50	0.05	0.88	650	1750	2250	60
C4-T5	4	70.00	0.05	0.88	650	1750	2250	60
C5-T5	5	87.50	0.05	0.88	650	1750	2250	60
C6-T5	6	105.00	0.05	0.88	650	1750	2250	60
C7-T5	7	122.50	0.05	0.88	650	1750	2250	60
C8-T5	8	140.00	0.05	0.88	650	1750	2250	60
C9-T5	9	157.50	0.05	0.88	650	1750	2250	60
C10-T5	10	175.00	0.05	0.88	650	1750	2250	60
C11-T5	11	192.50	0.05	0.88	650	1750	2250	60
C12-T5	12	210.00	0.05	0.88	650	1750	2250	60
C13-T5	13	227.50	0.05	0.88	650	1750	2250	60
C14-T5	14	245.00	0.05	0.88	650	1750	2250	60

Table 10: Ingredients used in the samples of the 2nd set of the experiments.

Sample	Ca(NO$_3$)$_2$		TIPA		Water (g)	Cement (g)	Sand (g)	Glass fiber (g)
	%	g	%	g				
C0-T0	0	0.00	0	0.00	650	1750	2250	60
C4-T0	4	70.00	0	0.00	650	1750	2250	60
C5-T0	5	87.50	0	0.00	650	1750	2250	60
C6-T0	6	105.00	0	0.00	650	1750	2250	60
C7-T0	7	122.50	0	0.00	650	1750	2250	60
C8-T0	8	140.00	0	0.00	650	1750	2250	60
C0-T25	0	0.00	0.025	0.44	650	1750	2250	60
C4-T25	4	70.00	0.025	0.44	650	1750	2250	60
C5-T25	5	87.50	0.025	0.44	650	1750	2250	60
C6-T25	6	105.00	0.025	0.44	650	1750	2250	60
C7-T25	7	122.50	0.025	0.44	650	1750	2250	60
C8-T25	8	140.00	0.025	0.44	650	1750	2250	60
C0-T5	0	0.00	0.050	0.88	650	1750	2250	60
C4-T5	4	70.00	0.050	0.88	650	1750	2250	60
C5-T5	5	87.50	0.050	0.88	650	1750	2250	60
C6-T5*	6	105.00	0.050	0.88	650	1750	2250	60
C7-T5*	7	122.50	0.050	0.88	650	1750	2250	60
C8-T5*	8	140.00	0.050	0.88	650	1750	2250	60
C0-T75	0	0.00	0.075	1.31	650	1750	2250	60
C4-T75	4	70.00	0.075	1.31	650	1750	2250	60
C5-T75	5	87.50	0.075	1.31	650	1750	2250	60
C6-T75	6	105.00	0.075	1.31	650	1750	2250	60
C7-T75	7	122.50	0.075	1.31	650	1750	2250	60
C8-T75*	8	140.00	0.075	1.31	650	1750	2250	60
C0-T1	0	0.00	0.100	1.75	650	1750	2250	60
C4-T1	4	70.00	0.100	1.75	650	1750	2250	60
C5-T1	5	87.50	0.100	1.75	650	1750	2250	60
C6-T1	6	105.00	0.100	1.75	650	1750	2250	60
C7-T1	7	122.50	0.100	1.75	650	1750	2250	60
C8-T1*	8	140.00	0.100	1.75	650	1750	2250	60

(*) indicates the chosen samples for the third set of the experiments

CHAPTER 4

RESULTS AND DISCUSSION

4.1. First Set of the Experiments: Determining the optimum value for $Ca(NO_3)_2$

First set of the experiments were conducted to determine the optimum amount of $Ca(NO_3)_2$. Data gained through the first set of the experiments were documented to establish a reference point for the further studies to be carried out to accomplish the objective of this study. The TIPA amount was kept constant at 0.05% of the cement content whereas the $Ca(NO_3)_2$ amount were changed gradually up to 14% of the cement content.

The slump values and the final setting time of the GRC samples produced through the first set of the experiments are tabulated in Table 11. Despite of having a low W/C ratio of 0.37 the addition of the admixtures resulted in a sufficiently workable mix. During the tests no segregation was observed. As compared to the mixture that did not contain any admixture, hereafter it will be called standard mix throughout the manuscript, the slump value increased but the final setting time decreased upon incorporation of the two admixtures. Moreover, the slump value increased but the final setting time decreased as the amount of $Ca(NO_3)_2$ is increased. The increase in the slump continues until 1.5% $Ca(NO_3)_2$ addition and reaches a maximum value of 17.5 cm while the standard mix had a slump value of 16 cm. Further addition of $Ca(NO_3)_2$ beyond 1.5% did not cause any change in the slump values. $Ca(NO_3)_2$ is known to have a certain plasticizing effect in cement mixes. The increase in slump value of the samples was attributed to the plasticizing effect of $Ca(NO_3)_2$. The results revealed that about 9% increase is gained in slump value upon addition of small amounts of both admixtures.

Table 11: Setting times and slump values of the samples of the first set of the experiments

Sample	Slump (cm)	Setting Time (h)
C0-T0	16.0	5.0
C0-T5	17.0	5.5
C1-T5	17.0	4.5
C1.25-T5	17.0	4.0
C1.5-T5	17.5	3.5
C1.75-T5	17.5	3.5
C2-T5	17.5	3.5
C3-T5	17.5	3.5
C4-T5	17.5	3.5
C5-T5	17.5	3.5
C6-T5	17.5	2.0
C7-T5	17.5	2.0
C8-T5	17.5	2.5
C9-T5	17.5	2.5
C10-T5	17.5	2.5
C11-T5	17.5	3.0
C12-T5	17.5	3.0
C13-T5	17.5	3.5
C14-T5	17.5	3.5

Final setting time of the standard mix is 5 h, but by the addition of the admixtures it dramatically decreases and with 6% of $Ca(NO_3)_2$ addition final setting time reaches to 2 h which corresponds to a 60% reduction. After 7% of $Ca(NO_3)_2$ addition, final setting time starts to increase again and at 14% of $Ca(NO_3)_2$ addition it reaches to 3.5h. The decrease in setting time is due to the addition of both admixtures. Only TIPA addition by 0.05% led to increase in setting time. However addition of 0.05% TIPA together with 1% $Ca(NO_3)_2$ resulted in a decrease in setting time.

The average compressive and flexural strength results of the first set of the experiments are presented in Table 12. The variation of the compressive strength of GRC mixes with $Ca(NO_3)_2$ is plotted in Figure 5. As seen in the figure, as $Ca(NO_3)_2$ amount increases the compressive strength values reach an optimum that corresponds to 6% of $Ca(NO_3)_2$ addition with a 169% increase in compressive strength. This optimum value occurs probably because of the saturation of $Ca(NO_3)_2$ in the medium causing an adverse effect after 7% addition to decrease the compressive strength. In Figure 6, flexural strength values increase rather irregularly, probably because of the agglomeration of the glass fibers present in the mixture. This is an expected result for a manually mixed GRC instead of being sprayed by an air gun. The accelerated setting increases the flexural strength and it gives a similar peak at 6-7% of $Ca(NO_3)_2$ with a 194% increase in flexural strength but it is still defined by the homogeneity and the position of the glass fibers inside the mix. The second peak on the in the figure and the increased variation between the flexural strength values are probable results of this phenomenon.
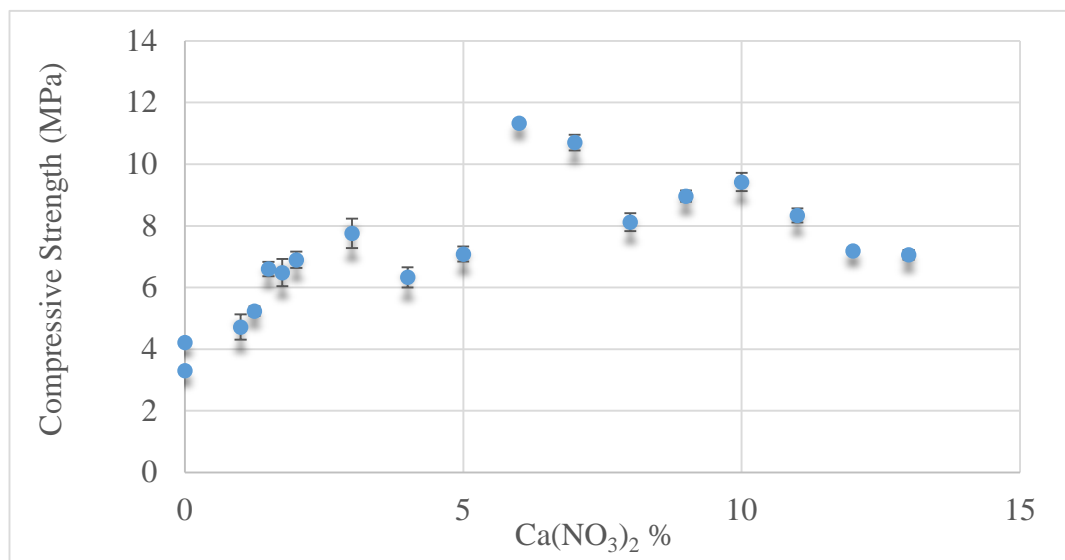
Figure 5: Compressive strength of the samples with respect to $Ca(NO_3)_2$ content at 6 hours

Table 12: Results of the first set of the experiments

Sample	Av. Compressive Strength (MPa) ± Standard Deviation	Av. Flexural Strength (MPa)
C0-T0	4.2 ± 0.05	1.8 ± 0.43
C0-T5	3.3 ± 0.10	1.6 ± 0.10
C1-T5	4.7 ± 0.41	1.7 ± 0.14
C1.25-T5	5.2 ± 0.16	1.9 ± 0.10
C1.5-T5	6.6 ± 0.23	2.4 ± 0.41
C1.75-T5	6.5 ± 0.44	2.0 ± 0.21
C2-T5	6.9 ± 0.27	2.5 ± 0.60
C3-T5	7.8 ± 0.48	2.9 ± 0.29
C4-T5	6.3 ± 0.33	2.6 ± 0.45
C5-T5	7.1 ± 0.25	3.6 ± 0.39
C6-T5	11.3 ± 0.11	5.0 ± 1.17
C7-T5	10.7 ± 0.25	5.3 ± 1.14
C8-T5	8.1 ± 0.29	3.3 ± 0.38
C9-T5	9.0 ± 0.18	3.1 ± 0.10
C10-T5	9.4 ± 0.29	4.1 ± 0.40
C11-T5	8.3 ± 0.23	2.6 ± 0.20
C12-T5	7.2 ± 0.10	3.4 ± 0.92
C13-T5	7.1 ± 0.16	3.6 ± 0.77
C14-T5	6.3 ± 0.17	5.0 ± 0.56
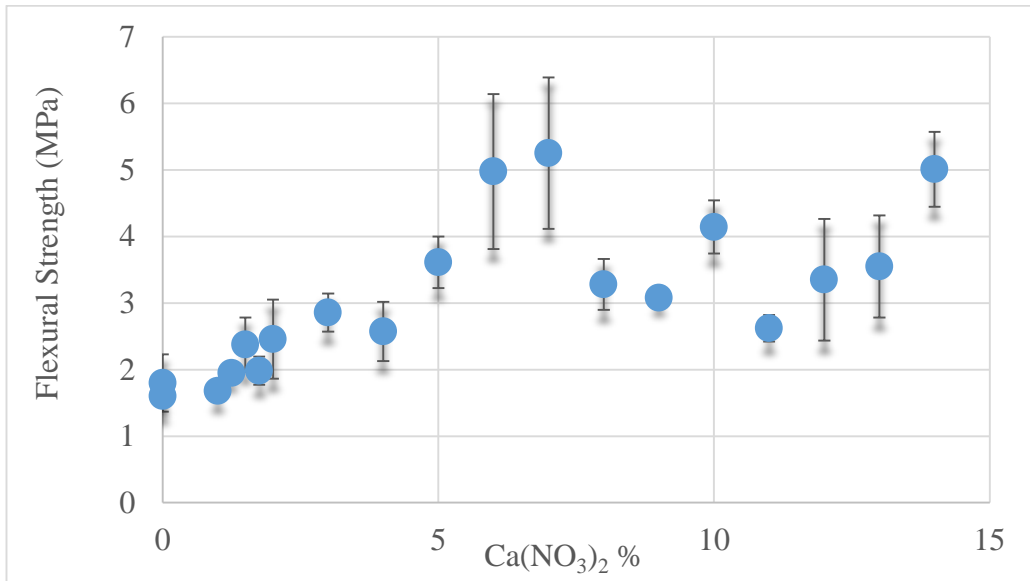
Figure 6: Flexural strength of the samples with respect to $Ca(NO_3)_2$ content at 6 hours

In the first set of the experiments, all samples, except C0-T5, showed greater compressive and flexural strength than the standard sample during the tests, which indicates that the addition of $Ca(NO_3)_2$ has a positive effect on early strength and 0.05% TIPA addition has no effect on early strength.

4.2. Second Set of the Experiments: Determining the combined effect of TIPA and $Ca(NO_3)_2$ at 6 h

In the second set of the experiments, the peak strength obtained for 6% of $Ca(NO_3)_2$ and 0.05% of TIPA addition was taken, and both of the amounts of the TIPA and the $Ca(NO_3)_2$ was changed around that peak point in order to determine its effect on 6 h strength. The results gave a matrix and a surface graph, stating the optimal combined effect of the two admixtures.

Tables 13 and 14 present the fresh properties of the mixes. During the second set of the experiments no segregation or workability issue was observed. Therefore no problem with the consistency of the mixes occurred.

Table 13: Slump values recorded in the second set of the experiments (cm)

TIPA (%)	Ca(NO$_3$)$_2$ (%)					
	0	4	5	6	7	8
0.000	16.0	17.5	17.5	17.5	17.5	17.5
0.025	17.5	17.5	17.5	17.5	17.5	18.0
0.050	17.5	17.5	17.5	17.5	17.5	18.0
0.075	18.0	18.0	18.0	18.0	18.0	18.5
0.100	18.0	18.0	18.0	18.0	18.0	18.5

Table 14: Setting times recorded in the second set of the experiments (h)

TIPA (%)	Ca(NO$_3$)$_2$ (%)					
	0	4	5	6	7	8
0.000	5.0	4.0	4.0	3.0	3.0	3.5
0.025	4.5	4.0	4.0	2.5	3.0	3.0
0.050	4.0	3.0	3.5	2.0	2.0	2.5
0.075	3.5	3.0	3.5	2.0	2.0	2.5
0.100	3.5	3.0	3.0	2.0	2.0	2.5

Although not being stated in the patent of Gartner and Myers (1989), during the second set of the experiments it was seen that TIPA has a definite plasticizing effect which is more dominant than Ca(NO$_3$)$_2$. As seen in Table 13, for all the samples for a given amount of Ca(NO$_3$)$_2$ addition, as the amount of TIPA increases the slumps also increases. The increase in slumps ranges between 9.4% and 15.6%. The samples which have the greatest slump are C8-T75 and C8-T1 by the increase of 15.6%. Having the greatest slump values at the highest addition of TIPA shows the plasticizing effect of the admixture. It is obvious that TIPA is more effective than Ca(NO$_3$)$_2$ on the slump values.

Also as seen in Table 14, the final setting time seems to decrease by the increasing amount of Ca(NO$_3$)$_2$ for a given amount of TIPA. The setting times seem to be affected

mainly by $Ca(NO_3)_2$ within 6 h as the results shows that TIPA addition could not change the setting time of the samples as much as the $Ca(NO_3)_2$ addition. The samples having 6 and 7% $Ca(NO_3)_2$ and 0.05-0.1% TIPA showed the lowest setting time which corresponds to 2 h and 60% decrease. Therefore $Ca(NO_3)_2$ seems to be the main ingredient giving the required reduction in final setting time. Figure 7 shows the change in final setting time.
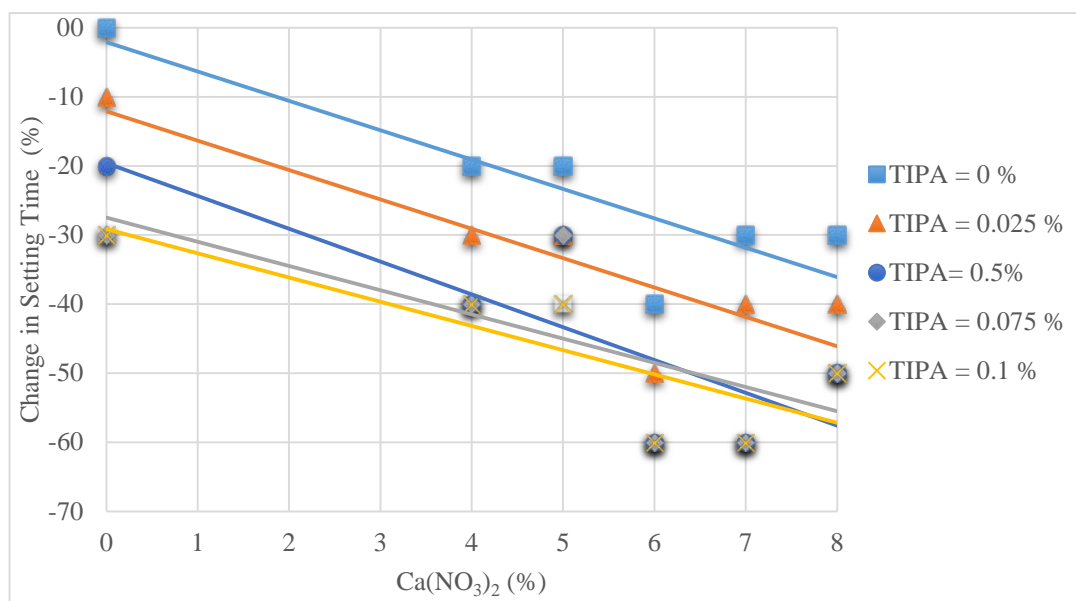
Figure 7: Change in setting time of the samples tested in the second set of the experiments

Tables 15 and 16 present the compressive and flexural strengths of GRC mixes in the second set of the experiments. At constant TIPA concentration, compressive strength increases with increasing amount of $Ca(NO_3)_2$ (Figure 8). But at constant $Ca(NO_3)_2$ concentration, compressive strength does not increase by the increasing amount of TIPA, which seems to have an optimal value at the concentration of 0.05%. Results of the second set of the experiments indicate that C6-T5 remains the peak point of the compressive strength graph with a value of 11.33MPa and 169% increase. After that point the change in compressive strength decreases, which tells us the profitable

margin has exceeded. Meanwhile the change in flexural strength is about 194% which gives a similar peak with the compressive strength, but since it is mainly defined by the amount and the position of the glass fibers used in the concrete and 1.5% glass fiber is used in the samples, the values it reached seems to be under limitations. There's also an irregularity between values which is probably caused by the agglomeration of the glass fibers in the concrete that is caused by the manufacturing of the samples manually (Figure 10&11). Yet, the overall increase in flexural strength is distinctive.

Table 15: Compressive strength test results of the 2nd set of the experiments (MPa)

TIPA (%)	$Ca(NO_3)_2$ (%)					
	0	4	5	6	7	8
0.000	4.22	4.44	5.51	5.35	5.34	6.56
0.025	3.52	5.22	4.53	6.08	6.28	6.98
0.050	3.27	6.33	7.09	11.33	10.7	8.12
0.075	2.78	4.4	4.39	4.88	5.98	9.34
0.100	4.33	4.31	6.56	5.91	6.1.	7.89

Flexural strength test results given in Table 16 indicate that concentration of the admixtures is not the only parameter determining the flexural strength as it changes randomly.

Table 16: Flexural strength test results of the 2nd set of the experiments (MPa)

TIPA (%)	$Ca(NO_3)_2$ (%)					
	0	4	5	6	7	8
0.000	1.76	1.55	2.29	2.47	3.21	2.82
0.025	2.14	2.38	1.97	3.49	3.23	3.84
0.050	1.55	2.57	3.61	4.98	5.25	3.28
0.075	2.00	2.07	1.45	2.06	3.09	3.90
0.100	2.51	1.93	2.55	3.38	2.47	3.09
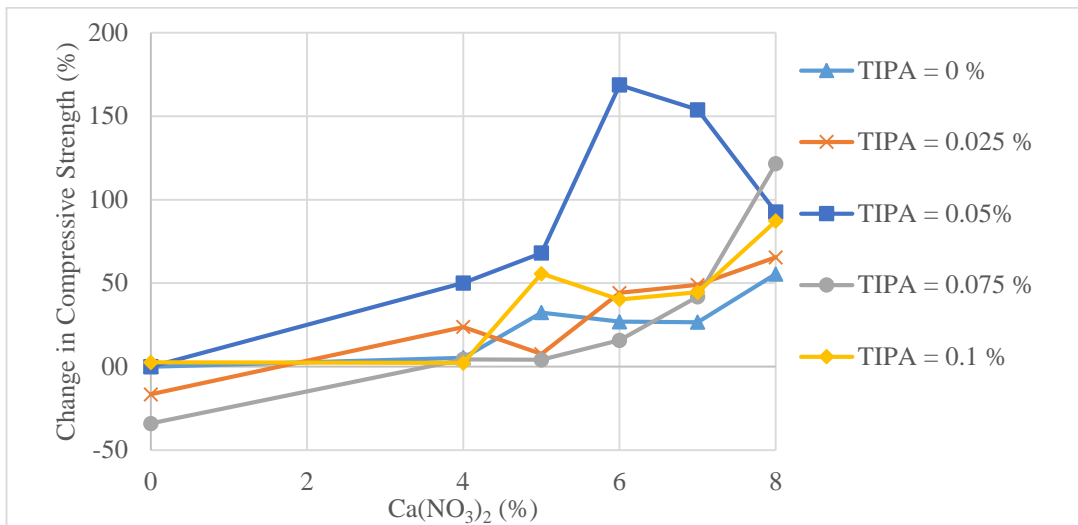
Figure 8: Change in compressive strength of the samples tested in the second set of the experiments
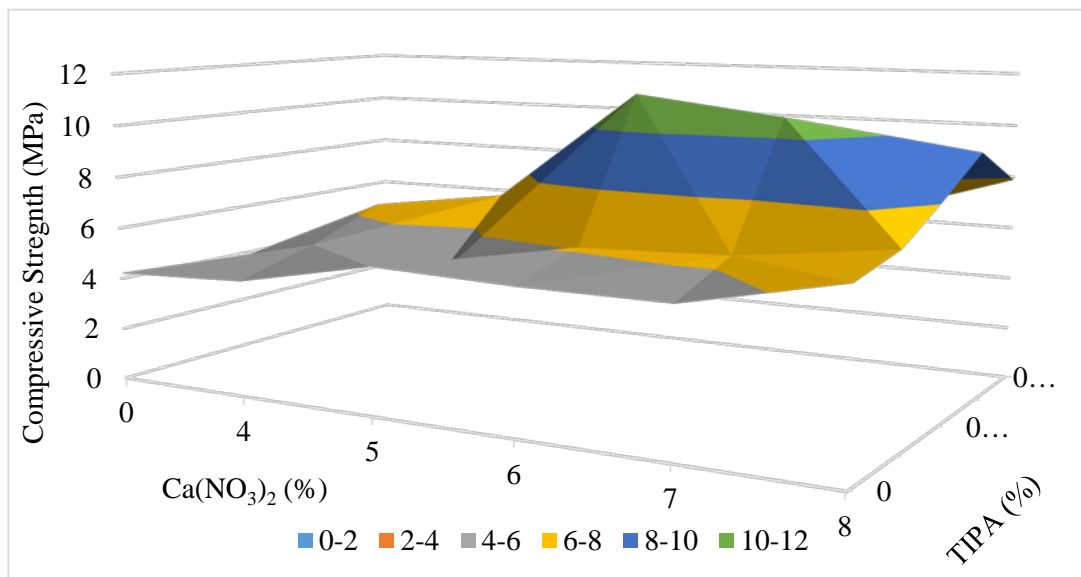
Figure 9: Surface graph of the compressive strength results of the 2nd set
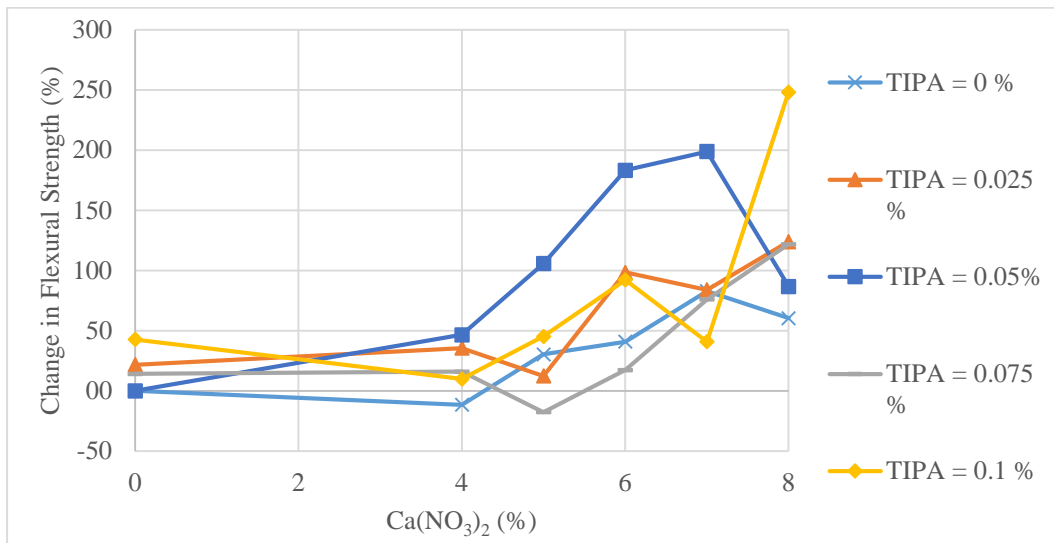
Figure 10: Change in flexural strength of the samples tested in the second set of the experiments
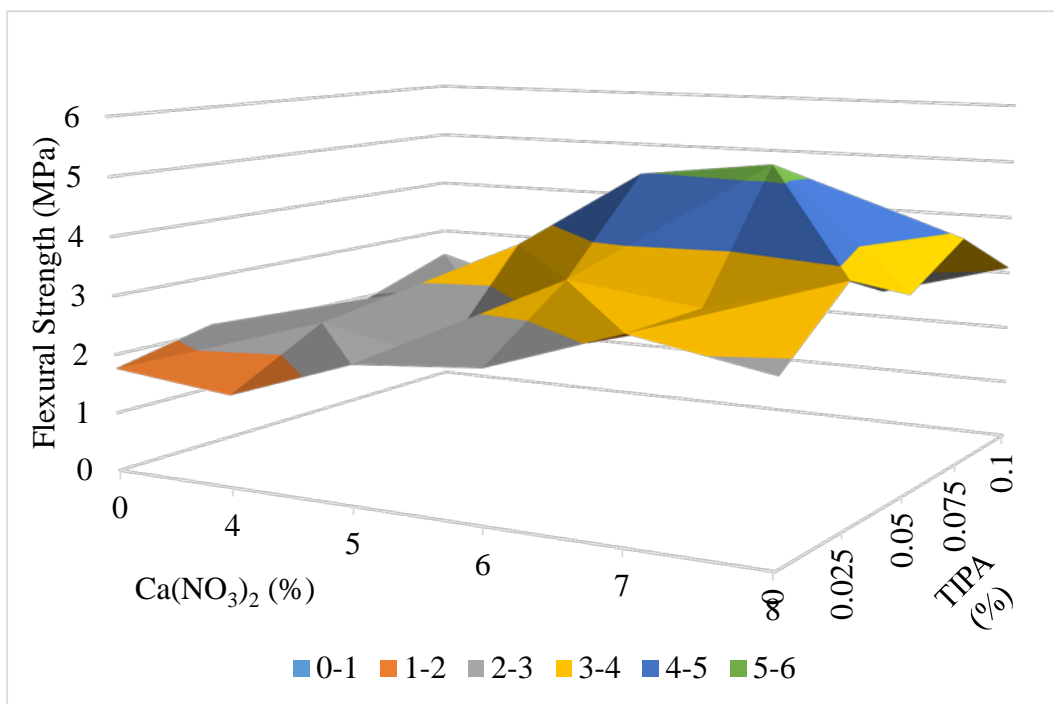
Figure 11: Surface graph of the flexural strength results of the 2nd set

4.3. Third Set of the Experiments: Strength tests at 7 and 28 days for the strongest 5 samples

In the third set of the experiments, the strongest 5 samples (C6-T5, C7-T5, C8-T5, C8-T75 and C8-T1) determined in the second set of the experiments are chosen and produced again to determine their 7 and 28 day strengths. Also in order to determine the effect of the TIPA on the strongest sample, which is C6-T5, a modified sample (C6-T0) was produced and tested. The compressive and flexural strength test results at 7 and 28 days are given in Tables 17 and 18.

Table 17: All results of the third set of the experiments at 7 days

Sample	Av. Compressive Strength (MPa) ± Standard Deviation	Av. Flexural Strength (MPa) ± Standard Deviation
C0-T0	42.17 ± 3.01	9.47 ± 0.91
C6-T5	53.13 ± 1.46	10.90 ± 0.56
C7-T5	53.43 ± 2.30	9.23 ± 0.78
C8-T5	45.63 ± 3.13	9.13 ± 0.45
C8-T75	53.73 ± 3.70	9.90 ± 0.20
C8-T1	55.70 ± 1.48	11.03 ± 1.50
C6-T0	40.03 ± 2.40	9.66 ± 1.89

The compressive strength test results at 7 days indicate that the admixtures increase the strength development such that all of the samples except C8-T5 reaches the GRCA limits of 28 days in the first week. The peak point of the first two set of the experiments C6-T5 showed an increase of 26% in compressive strength according to the standard mix, but surprisingly C8-T1 showed a greater increase in compressive strength by the amount of 31.8%. This is probably caused by the increasing effect of the TIPA during

the strength development, and as the amount of TIPA increases the compressive strength increases too. Probably the dispersant effect of TIPA creates a better medium for hydration reactions and result in higher later age strength. C6-T0 showed a lower compressive strength than the standard sample at 7 days, which seems to be because of the negative effects of acceleration of the setting in the absence of TIPA. It was the control sample of the C6-T5 which has 32.7% increase in strength according to C6-T0 at 7 days. This result indicates that TIPA has a more pronounced effect during the strength development, and $Ca(NO_3)_2$ loses its effect on the strength during the process and in the absence of TIPA, $Ca(NO_3)_2$ can't increase the strength at 7 days. Whereas the flexural strength values remain similar since it's basically determined by the amount of the glass fiber inside the mix and the glass fiber used in the samples are the same.

Table 18: All results of the third set of the experiments at 28 days

Sample	Avg. Compressive Strength (MPa)) ± Standard Deviation	Avg. Flexural Strength (MPa)) ± Standard Deviation
C0-T0	53.03 ± 1.74	10.83 ± 0.51
C6-T5	60.9 ± 6.89	12.64 ± 0.55
C7-T5	63.97 ± 5.23	10.33 ± 1.01
C8-T5	59.23 ± 3.23	10.4 ± 2.20
C8-T75	69.2 ± 4.36	11.23 ± 0.25
C8-T1	71.57 ± 2.60	10.07 ± 0.32
C6-T0	52.5 ± 3.20	10.83 ± 0.81

The compressive strength test results at 28 days are remarkable and they prove that TIPA increases the ultimate strength. The results of the third set of the experiments at 28 days showed that all samples have greater compressive strength values than the standard sample. The only sample remains below the standard sample is C6-T0 which has no TIPA inside, thus gives a barely lower compressive strength than the standard

sample by a value of 0.1% decrease in the compressive strength. The compressive strength test results showed that instead of the peak point of the first two set of the experiments which was C6-T5, surprisingly, C8-T1 gave the greatest compressive strength test result by a 34.9% increase. C6-T5 could just give a 14.8% increase in the compressive strength. This result proves that the increasing amount of TIPA causes a greater ultimate strength for all the samples although not being significantly effective within the first 6 h. Moreover after the very early strength gaining, $Ca(NO_3)_2$ loses its effect on strength development whereas TIPA dominates the development at long term. In contrary with the compressive strength values, all of the flexural strength values are similar since they are still defined by the amount of the glass fiber inside the mix.

In order to understand the strength development at all ages the Table 19 and Figure 12 could be useful. In Figure 12 it is clear that TIPA has an increasing effect on early age strength development, while $Ca(NO_3)_2$ loses its effect on long term. Although the change in compressive strength decreases by the time, the samples having higher TIPA remains stronger at all ages.

Table 19: Compressive strength development at all ages

| Sample	6 h	7 days	28 days
C0-T0	4.22	42.17	53.03
C6-T5	11.33	53.13	60.90
C7-T5	10.70	53.43	63.97
C8-T5	8.12	45.63	59.23
C8-T75	9.34	53.73	69.20
C8-T1	7.89	55.70	71.57
C6-T0	5.35	40.03	52.60
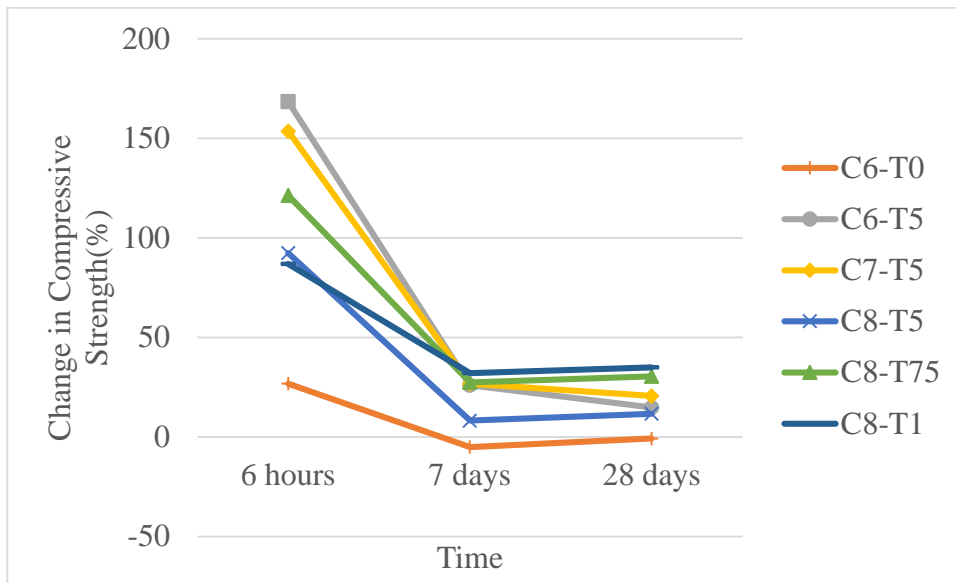
Figure 12: Change in compressive strength at all ages

4.4. Statistical Analysis

After recording all of the tests results, in order to determine the relation between them and truth of the hypothesis a statistical analysis which consists of multiple regression and ANOVA (Analysis of variance) is performed. The calculations are made for 6 h, 7 days and 28 days among the compressive strength values. Table 20 presents the 6 h data analysis.

Table 20: Statistical analysis of the results at 6 h

Regression Statistics	
Multiple R	0.681
R Square	0.464
Adjusted R Square	0.424
Standard Error	1.525
Observations	30

ANOVA

	df	SS	MS	F	Significance F
Regression	2	54.260	27.130	11.669	0.00022
Residual	27	62.773	2.325		
Total	29	117.033			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%
Intercept	3.123	0.723	4.318	0.000	1.639
$Ca(NO_3)_2$	0.518	0.108	4.801	0.000	0.296
TIPA	4.253	7.874	0.540	0.593	-11.903

The results given in Table 20 indicates that TIPA is more effective on the results of the first set of the experiments. The coefficient of TIPA is 4.253 where that of $Ca(NO_3)_2$ is 0.518. Also significance F<0.05 value indicates that the hypothesis arguing the possibility of using TIPA and $Ca(NO_3)_2$ as an admixture of concrete is true. Also the R^2 value which is the certainty of the estimation is 46% which is acceptable. In addition the difference between R^2 and $R^2_{adjusted}$ is too small, which means that the variation within variables is too low.

Table 21: Statistical analysis of the results at 7 days

Regression Statistics	
Multiple R	0.901
R Square	0.812
Adjusted R Square	0.717
Standard Error	3.379
Observations	7

ANOVA

	df	SS	MS	F	Significance F
Regression	2	196.704	98.352	8.616	0.03549
Residual	4	45.660	11.415		
Total	6	242.364			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%
Intercept	43.266	3.284	13.176	0.000	34.149
Ca(NO$_3$)$_2$ %	-0.383	0.701	-0.546	0.614	-2.328
TIPA %	176.697	54.630	3.234	0.032	25.021

The results given in Table 21 indicates that TIPA is more effective on the results at 7 days. The coefficient of TIPA is 176.697 where that of Ca(NO$_3$)$_2$ is -0.38. These numbers indicates that TIPA is increasing its effect on strength development. Also significance F<0.05 value indicates that the hypothesis is true. The R^2 value which is the certainty of the estimation is 81% which is quite acceptable. In addition the difference between R^2 and $R^2_{adjusted}$ is small, which means that the variation within variables is low.

Table 22 Statistical analysis of the results at 28 days

Regression Statistics	
Multiple R	0.972
R Square	0.945
Adjusted R Square	0.918
Standard Error	2.111
Observations	7

ANOVA

	df	SS	MS	F	Significance F
Regression	2	307.062	153.531	34.441	0.00301
Residual	4	17.831	4.458		
Total	6	324.893			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%
Intercept	53.068	2.052	25.862	0.000	47.371
$Ca(NO_3)_2$ %	-0.184	0.438	-0.419	0.697	-1.399
TIPA %	205.593	34.139	6.022	0.004	110.808

The results given in Table 22 indicates that TIPA is much more effective on the results at 28 days. The coefficient of TIPA is 205.593 where that of $Ca(NO_3)_2$ is -0.184. These numbers indicate that at 28 days TIPA is the only admixture effecting the strength development. Also significance F<0.05 value indicates that the hypothesis is true. The R^2 value is 94% which indicates that these estimations are quite reliable. In addition the difference between R^2 and $R^2_{adjusted}$ is too small, which means that the variation within variables is too low.

4.5 XRD and SEM Analysis of the Weakest and the Strongest Samples

In the third set of the samples, it is determined that Standard Mix (C0-T0) is the weakest one and the C8-T1 is the strongest one of the samples. In order to examine the possible reasons of this phenomena, XRD and SEM analyses were carried out. The analysis results are shown in Figure 13.

The XRD results indicate that in C8-T1 has a significant increase in the amounts of the hydrates occurred during the strength development. The hydrated crystals might be products of C_4AF phase. The SEM results also prove the effect of TIPA used in the C8-T1 which generates a more remarkable crystalline formation inside the mix.

According to the patent (Gartner and Myers, 1989), which is covered for 7 and 28 days and does not have any comment for 6 h, TIPA should be used in the range between 0-0.2 %, preferably below 0.1% and the optimal values are between 0.03 - 0.05 %. These amounts depend on the cement type. The results gathered in this study are in accordance with their study. Presence of the three bulky methyl groups in TIPA provides steric hindrance that minimizes adsorption of TIPA on the hydration products. Above 0.2%, TIPA solubilizes iron even after sulfate depletion. This allows the facilitated iron transport to continue beyond the sulfate depletion point. At late ages for cements with more than 4% C_4AF, it is possible to gain more strength. (Gartner and Myers, 1989). These mechanisms may lead the formation of hydrates of C_4AF phase as such monosulfoaluminate, monocarboaluminate or hemicarboaluminate and their byproducts (Ichikawa et. al., 1997). These minerals may be the reason for the change shown in XRD and SEM analyses given in Figure 13.

Standard Mix (C0-T0)   C8-T1
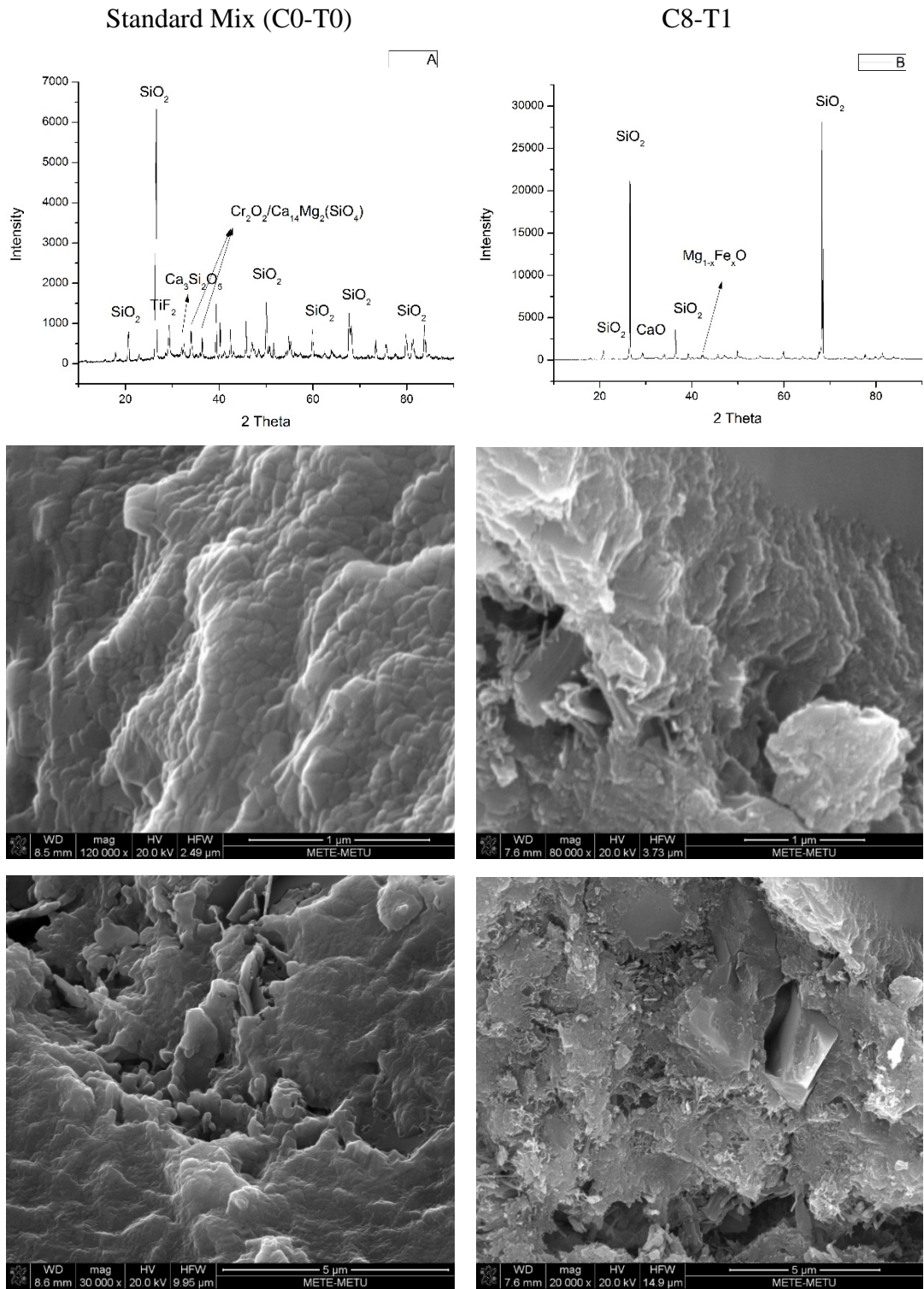
Figure 13: XRD and SEM analyses results

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

The results of the study indicates that the goal was achieved and a cheaper way of GRC production is possible by the use of both TIPA and $Ca(NO_3)_2$, it was shown that the results has led to the following conclusions.

1. GRC panels could gain sufficient strength at about 6 h by the usage of an optimum amount of TIPA and $Ca(NO_3)_2$ as the admixtures without using any steam curing or any other equipment.

2. Setting time decreases but slump values increase by the addition of both admixtures. Final setting time decreases by 60% upon adding 6-7% $Ca(NO_3)_2$ and 0.05% TIPA. TIPA increases the ultimate strength of the GRC despite its less effectiveness in the first 6 h of the setting.

3. Within the first 6 h of production of GRC, $Ca(NO_3)_2$ is more effective than TIPA on the strength development. However, TIPA is much more effective than $Ca(NO_3)_2$ on strength development especially after 7 days. The maximum compressive strength at 6 h is gained by 6% $Ca(NO_3)_2$ and 0.05% TIPA addition with a 169% increase. This is the preferable amount of admixture for a faster production rate of GRC. The ultimate compressive strength obtained by the use of this amount of admixture is increased by 15%.

4. In the presence of TIPA and $Ca(NO_3)_2$, GRC could gain sufficient compressive strength at 7 days to meet the GRCA requirements of 28 day compressive strength.

5. By the addition of 8% $Ca(NO_3)_2$ and 0.1% TIPA, 35% increase in the 28 days compressive strength is achieved. This amount of admixture is preferable for higher ultimate strength.

6. X-ray Diffraction and scanning electron microscopy analyses reveal that TIPA increases the hydration of cement and promotes the creation of hydrates or mineral formation. In other words, at constant time the amount of the resulting hydrates of cement and their byproducts is different in the presence of TIPA.

Although not being intended in the study, it is determined that TIPA has a significant plasticizing effect on the concrete. This comes with an extra compaction effect which probably causes greater strength development. Therefore, for future studies, it is recommended to use these admixtures by decreasing the water content while keeping the slump value constant. Thus, greater strength values could be reached without losing workability.

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RESULTS OF THE 1ST SET OF THE EXPERIMENTS

Table 23: All results of the first set of the experiments

Sample	Slump (cm)	Setting Time (h)	Compressive Strength (MPa)					Flexural Strength (MPa)				
			#1	#2	#3	Av.	COV (%)	#1	#2	#3	Av.	COV (%)
C0-T0	16	5	4.3	4.2	4.2	4.2	1.1	1.3	2.0	2.0	1.8	24.6
C1-T5	17	4.5	4.4	4.6	5.2	4.7	8.7	1.8	1.6	1.6	1.7	8.3
C1.25-T5	17	4	5.2	5.1	5.4	5.2	3.0	2.1	1.9	1.9	1.9	4.9
C1.5-T5	17.5	3.5	6.7	6.3	6.8	6.6	3.5	2.8	2.2	2.1	2.4	17.1
C1.75-T5	17.5	3.5	6.1	6.4	7.0	6.5	6.9	2.2	2.0	1.8	2.0	10.6
C2-T5	17.5	3.5	6.6	6.9	7.2	6.9	3.8	1.8	3.0	2.6	2.5	24.2
C3-T5	17.5	3.5	7.4	7.6	8.3	7.8	6.2	2.8	3.2	2.6	2.9	10.0
C4-T5	17.5	3.5	6.0	6.5	6.6	6.3	5.2	2.6	2.1	3.0	2.6	17.3
C5-T5	17.5	3.5	6.8	7.3	7.1	7.1	3.5	3.7	4.0	3.2	3.6	10.7
C6-T5	17.5	2	11.2	11.3	11.5	11.3	1.0	6.1	3.8	5.0	5.0	23.4
C7-T5	17.5	2	11.0	10.5	10.7	10.7	2.4	4.5	4.7	6.6	5.3	21.7
C8-T5	17.5	2.5	7.8	8.1	8.4	8.1	3.6	3.4	3.6	2.9	3.3	11.6
C9-T5	17.5	2.5	9.0	9.1	8.8	9.0	2.0	3.0	3.2	3.0	3.1	3.1
C10-T5	17.5	2.5	9.8	9.2	9.3	9.4	3.1	4.5	4.3	3.7	4.1	9.7
C11-T5	17.5	3	8.5	8.1	8.5	8.3	2.7	2.4	2.7	2.7	2.6	7.6
C12-T5	17.5	3	7.1	7.2	7.3	7.2	1.3	4.3	2.4	3.4	3.4	27.3
C13-T5	17.5	3.5	7.0	7.0	7.2	7.1	2.2	4.4	3.2	3.0	3.6	21.6
C14-T5	17.5	3.5	6.2	6.2	6.5	6.3	2.8	5.6	4.5	4.9	5.0	11.2

RESULTS OF THE 2ND SET OF THE EXPERIMENTS

Table 24: All results of the second set of the experiments

Sample	Ca(NO$_3$)$_2$ %	TIPA %	Compressive Str. (MPa)					Flexural Str. (MPa)				
			#1	#2	#3	Avg.	COV (%)	#1	#2	#3	Avg.	COV (%)
C0-T0	0	0.000	4.3	4.2	4.2	4.2	1.1	1.3	2.0	2.0	1.8	24.6
C0-T25	0	0.025	3.3	3.5	3.7	3.5	5.7	2.7	1.5	2.2	2.1	29.6
C0-T5	0	0.050	3.3	3.2	3.4	3.3	3.1	1.5	1.6	1.5	1.6	3.9
C0-T75	0	0.075	2.3	2.9	3.2	2.8	16.7	2.2	2.0	1.8	2.0	12.0
C0-T1	0	0.100	4.3	4.1	4.5	4.3	4.2	2.2	2.8	2.6	2.5	12.5
C4-T0	4	0.000	4.1	4.9	4.3	4.4	8.9	1.6	1.8	1.3	1.6	14.6
C4-T25	4	0.025	5.0	5.2	5.5	5.2	4.7	2.0	2.5	2.6	2.4	12.4
C4-T75	4	0.075	4.3	4.3	4.6	4.4	3.7	2.0	1.8	2.3	2.0	10.6
C4-T1	4	0.100	4.3	3.9	4.7	4.3	10.1	1.5	2.0	2.3	1.9	19.0
C5-T0	5	0.000	5.5	5.3	6.0	5.6	6.3	1.7	2.5	2.7	2.3	24.1
C5-T25	5	0.025	4.3	4.7	4.6	4.5	4.9	2.6	2.0	1.4	2.0	29.2
C5-T75	5	0.075	4.4	4.5	4.3	4.4	2.3	1.2	1.8	1.3	1.4	19.1
C5-T1	5	0.100	6.4	6.8	6.6	6.6	3.0	2.2	3.0	2.5	2.6	16.9
C6-T0	6	0.000	5.4	5.3	5.4	5.4	1.2	2.5	2.3	2.7	2.5	7.1
C6-T25	6	0.025	6.0	6.0	6.2	6.1	1.7	3.8	3.2	3.4	3.5	8.7
C6-T75	6	0.075	4.8	4.9	5.0	4.9	2.6	1.6	2.4	2.2	2.1	18.6
C6-T1	6	0.100	5.8	6.0	6.0	5.9	1.7	3.4	4.0	2.7	3.4	19.0
C7-T0	7	0.000	5.6	5.3	5.1	5.3	5.0	3.5	3.4	2.7	3.2	14.7
C7-T25	7	0.025	6.0	6.4	6.5	6.3	4.5	4.0	2.4	3.4	3.2	24.7
C7-T75	7	0.075	5.8	6.1	6.1	6.0	3.4	2.7	3.4	3.1	3.1	11.2
C7-T1	7	0.100	5.6	6.4	6.3	6.1	6.8	2.5	1.9	3.0	2.5	21.8
C8-T0	8	0.000	6.4	6.5	6.8	6.6	3.0	3.2	2.1	3.2	2.8	21.4
C8-T25	8	0.025	7.1	7.1	6.7	7.0	3.1	3.6	4.4	3.8	3.9	10.6
C8-T75	8	0.075	9.1	9.4	9.5	9.3	2.7	4.6	3.2	3.8	3.9	18.1
C8-T1	8	0.100	8.0	7.7	8.0	7.9	2.4	6.0	7.1	5.3	6.1	14.7

RESULTS AT 7 DAYS

Table 25: Strength Results at 7 days

Sample	Compressive Strength (MPa)					Flexural Strength (MPa)				
	#1	#2	#3	Avg.	COV %	#1	#2	#3	Avg.	COV %
C0-T0	39.0	45.0	42.5	42.17	7.15	10.3	8.5	9.6	9.47	9.58
C6-T5	54.8	52.1	52.5	53.13	2.74	11.0	10.3	11.4	10.9	5.11
C7-T5	53.3	55.8	51.2	53.43	4.31	8.6	10.1	9.0	9.23	8.41
C8-T5	46.1	42.3	48.5	45.63	6.85	8.7	9.1	9.6	9.13	4.94
C8-T75	51.6	51.6	58.0	53.73	6.88	9.9	9.7	10.1	9.9	2.02
C8-T1	56.4	56.7	54.0	55.70	2.66	11.9	11.9	9.3	11.03	13.61
C6-T0	42.4	40.1	37.6	40.03	6.00	10.48	7.5	11.0	9.66	19.55

RESULTS AT 28 DAYS

Table 26: Strength Results at 28 Days

Sample	Compressive Strength (MPa)					Flexural Strength (MPa)				
	#1	#2	#3	Avg.	COV %	#1	#2	#3	Avg.	COV %
C0-T0	55.0	52.4	51.7	53.03	3.28	10.4	10.7	11.4	10.83	4.74
C6-T5	64.0	65.7	53.0	60.90	11.32	12.56	12.14	13.23	12.64	4.35
C7-T5	65.0	58.3	68.6	63.97	8.17	9.4	11.4	10.2	10.33	9.74
C8-T5	56.8	58.0	62.9	59.23	5.46	10.4	8.2	12.6	10.4	21.15
C8-T75	68.4	73.9	65.3	69.20	6.29	11.5	11.0	11.2	11.23	2.24
C8-T1	69.3	71.0	74.4	71.57	3.63	10.2	9.7	10.3	10.07	3.19
C6-T0	55.6	49.2	52.7	52.50	6.1	11.2	9.9	11.4	10.83	7.52