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Investigating the Rapid Curing Possibility of Geopolymer Concrete

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INVESTIGATING THE RAPID CURING POSSIBILITY OF GEOPOLYMER CONCRETE

by

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ABSTRACT

Recent studies of heat-cured fly ash based green geopolymer concrete have shown its suitability for fabrication of structural members. Fabrication of these structural members requires continuous moderate heating (145°F) for 24 hrs, and an oven essential, for a large member can quickly turn into an energy guzzler, potentially eliminating the green credentials of the product. The proposed research involves the development of a frontal polymerization (FP) method that achieves rapid curing of geopolymer at ambient condition after short-term heat application. Initial work shows thermal FP in geopolymer is a possibility and might be a solution to the problem. The process includes blending of monomer, initiator, cross-linkers, and geopolymer slurry, which upon application of heat, polymerizes locally, and the reaction front moves forward to complete the procedure resulting in a solid finished product. Preliminary strength evaluation revealed the strength of FP cured geopolymer mortar specimens are compatible with traditional cement mortar samples. Therefore, frontally cured geopolymer has the potential to save significant time and energy required in the curing of geopolymer product and can create a new horizon in fly ash utilization, the waste often deserted in the environment as a landfill material. Therefore, the research has the potential in generating green credits and means for meeting federal requirements for reduced carbon footprints on federally-funded projects and save taxpayers' money.

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CHAPTER 1

INTRODUCTION

1.1 Background

The use of cementing materials to build structures is not a new concept. People have been using cementing materials to build houses and various other constructions. The discovery of cement has revolutionized the approach people have been using to construct buildings. Concrete is the mix prepared from cement, water, and aggregate along with filler material such as sand. With its high strength, durability and low maintenance feature, cement has been the dominant primary cementing material.

Moreover, cement concrete can be cast to any chosen shape at the working site making it more desirable than any other material such as wood or steel that possesses abilities to be a building material. The use of concrete in construction has sky-rocketed so much these days that it is now second to water for the most utilized substance in the world. However, the production of cement imposes impacts on the environment. The heating of limestone (calcium carbonate) breaks down to lime (calcium oxide), a component of cement and releases carbon dioxide (CO_2). This CO_2 is responsible for 8% of total CO_2 emissions (Eq.1).

$$CaCO_3(s) \rightarrow CaO(s) + CO_2(g) \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \text{ Eq.1}$$

Besides emissions, cement production uses natural resources such as limestones, fuel fossil, and considerable energy. Attempts have been made to reduce this loss by using alternative cementing materials such as blast furnace slag, silica fume and fly ash. This thesis/research focuses on the cementing material made up of fly ash.

Fly ash is the by-product from a coal power plant and is often deserted with no or minimum use. One of its green alternative applications is to make geopolymer concrete. Geopolymer concrete utilizes fly ash and slag along with alkaline activators which polymerize to form a hardened binder. The use of these concrete reduces the waste stocks and carbon emission from cement production. However, one of the challenges in using geopolymer is its requirement for high-temperature curing which is often fulfilled by an oven set to a certain temperature. This requirement not only imposes geopolymer to consume energy but also restricts its application to smaller structures that can fit into the oven. The work described in the following chapters will outline a possibility of a curing method that requires heat application in a small area for a short duration of time and has an ability to propagate throughout the body of the structure to offer a finished product. Implementation of this curing method into geopolymer eradicates its restriction of oven curing constraints and reduces carbon footprints to obtain green credentials.

1.2 Objective

The objective of this thesis is to provide a possible alternative curing method that can be used to eliminate the limitations possessed by a traditional geopolymer concrete. The next objective is to utilize industrial waste, fly ash, which is often abandoned into landfills. This not only reduces the waste stocks but also saves land areas required for desertion. The work shows a significant reduction in curing time needed to obtain a hardened product as well. This property opens up an application of geopolymer concrete that requires quick setting but an early full strength. Finally, this project discusses the capabilities to cure using perpetual energy sources such as sunlight to make it greener and eventually eliminating environmental impacts and savings in energy.

1.3 Thesis Organization

In addition to this introductory chapter, the thesis has four more chapters: Literature review, Methods, Results, and Conclusions.

Chapter 2 reviews the relevant literature on Cement Concrete (CC), Geopolymer Concrete (GPC) and FP technique. A review consisting of previous work and study performed by FP technique is presented along with an earlier application.

Chapter 3 discusses the methods that were used to prepare the mix and create the specimen. Also, this chapter describes the procedure adopted to synthesize organic initiator Aliquat Persulfate (ALPS) through ionic exchange and a filtration process to remove the inhibitor from a monomer. In addition, multiple trials performed with various chemicals in different proportions are also tabulated alongside the outcome for each mix and are recorded in Appendix B.

Chapter 4 discusses the results shown by the samples in terms of compressive strength. This chapter also compares the results for CC, GPC, and Frontally Polymerized Geopolymer Concrete (FPGPC) samples. It presents the Scanning Electron Microscopy (SEM) image protocol and analyzes the image produced for FPGPC samples.

Chapter 5 presents the conclusion from the research presented herein and outlines the recommendations for future improvements and implementations of the research along with possible expansion of the study .

CHAPTER 2

LITERATURE REVIEW

2.1 Cement Concrete

In the early days of Greek civilization, calcinated gypsum was used as a cementing material, whereas Romans used lime from limestone to build houses, which was the primary construction material that existed. The development of early Portland cement started in the late 18th Century in Britain. It consists mainly of Calcium Oxide (CaO), Silicon dioxide (SiO_2), Aluminum Oxide (Al_2O_3), Ferric Oxide (Fe_2O_3), and Sulfur dioxide (SO_2).

After its discovery, Ordinary Portland Cement (OPC) has been a significant construction material (concrete, mortar, and grouts) due to its high strength, workability, and ability to remain hardened from moisture. However, along with OPC's versatility, its production procedure releases a considerable amount of CO_2 in the atmosphere, contributing a significant role in the greenhouse effect. The developed countries are already concerned and some regulations regarding the limitation of CO_2 are taken into consideration. The gas released while producing OPC is almost equal to 1.35 billion tons, which accounts for 7% of the total greenhouse gases [1]. Mehta recommended the use of less natural resources and energy will minimize the emission of CO_2, thus producing environment-friendly concrete [2].

With various methods to reduce CO_2, use of alternative energy sources, full or partial replacement of clinker with supplementary material and carbon capture and storage system are few of the most effective ones. Among the factors, the replacement of clinkers with inert filler materials such as limestone has proven to be the most practical approach [3].

In addition to the release of CO_2, the cement concrete requires 28-days curing time to gain its full strength. Hardening of cement is a hydration process which involves water, usually provided externally to cure the concrete mix. The optimum performance of OPC depends upon the curing time and temperature [3]. Compressive strength and sorptivity are two properties that can influence the durability of the structures [4]. Hazare *et al.* have mentioned curing time as a factor to determine sorptivity while Ozer *et al.* have investigated the sorptivity for different specimen having various air or water curing time and found that the curing time plays a significant role in sorptivity of OPC and requires a minimum curing of seven days to show any gain in strength [5], [6]. Studies have shown to have a reduction of 50% in compressive strength of the sample cured in water and air [7]. Furthermore, the sample cured over the water showed a progressive increase in compressive strength over a five year period, but the sample cured over air reached its peak at 28 days and remains constant [8].

2.2 Geopolymer Concrete

GPC uses fly ash or slag in partial replacement with the OPC that acts as a binder in a concrete and has been introduced to subsidize the CO_2 emission problem. It was first studied by the French researcher Davidovits with Kaolinite and the alkaline activator in 1978 [9]. Several studies have indicated that the partial replacement (up to 30%) of supplementary cementing materials such as microfine cement and stone dust with fly ash and slag can have a positive impact on the mechanical properties of the cement concrete [10], [11]. The replacement of this kind of microfine material on a high level into the specimen leads to having better workability. In addition to the workability, the specimens showed an increase in compressive strength for most of the samples as compared with the regular cement mix. Furthermore, the samples with the partial replacement of 15% microfine cement and 7.5% of stone dust were able to demonstrate higher split tensile strength, flexural strength, higher absorption of impact energy, higher pull out failure load, and higher modulus of elasticity [12]. However, the replacement of stone dust has to be limited, and with the proper amount it can help augment the mechanical properties through better hydration and improve the pore structure of the concrete [12], [13].

Fly ash is the byproduct of the coal combustion plant and is abundantly used as landfill material. It is also known as coal ash or flue ash. Since 1998, fly ash production is more than 390 million tons per year and only less than 15% was used [14]. With the current development pace in countries like the US, China, and India, it can be easily predicted to hit at a high level. The ASTM C618 identifies two different types of fly ash (Class F and Class C) based on the presence of calcium oxide (CaO), silicon dioxide (SiO_2), ferric oxide (Fe_2O_3), and aluminum oxide (Al_2O_3). One major difference between the two types of fly ash is the amount of CaO present: Class F fly ash has less than 7% of CaO while Class C fly ash contains more than 20% of CaO in it [15]. This allows Class C fly ash to harden without having any activator while class F requires specific cementing material for hardening and gaining strength [16].

In addition, the former has a minimum of 70% of oxides by mass while the number is 50% for the latter. Besides the difference in contents, Class F fly ash is produced from the burning of mature anthracite or bituminous coal and is pozzolanic while Class C fly ash is produced from burning younger lignite or sub-bituminous coal and has cementitious properties along with pozzolanic properties [15].

Class F fly ash, when induced with Alkaline activators, forms an alumino-silica gel, which then binds with aggregates to form a hardened geopolymer concrete upon application of heat (140°F ~ 190°F) [17]. However, it has been studied that GPC can be cured in an ambient condition but requires longer curing time to reach the target strength. Most commonly utilized alkaline activators in GPC are Sodium Silicate (Na_2SiO_3) and Sodium Hydroxide (NaOH) or Potassium Hydroxide (KOH). For the best results, the solution should be mixed 24 hours before use, and the molarity of the NaOH used should be in the range of 8 M to 16 M. The proportions of the materials and their constituents can impact the workability and compressive strength of the geopolymer mix [18]. Furthermore, increasing the ratio of Na_2SiO_3 to NaOH from 0.5 to 2.5 increases viscosity and decreases the workability of the GPC. However, the ratio of $Na_2SiO_3$and NaOH is 1:1.5 for the oven and 1:2 for ambient curing [19]. In another study by Chindaprasirt *et al.*, the optimum ratio for the mix was found to be in the range of 0.67 to 1 [20].

The calcium content present on the fly ash has a considerable influence in the bonding properties, setting time, workability, and strength of the concrete. With constant ratio for an alkaline solution to fly ash and Na_2SiO_3 to NaOH, the increment in water to fly ash ratio increases the workability and setting time with all ratios. Furthermore, the addition of water with precise ratios of alkaline solution to fly ash leads to higher compressive strength, which is due to an increase in calcium silicate hydration from additional water [19]. Higher calcium content helps to form calcium silicate hydrate, aluminum modified calcium silicate and sodium aluminosilicate hydrate, which are responsible for the early age strength of the concrete, but it severely affects the workability and setting time of the concrete [20] - [22].

The presence of a high amount of calcium resulting in calcium silica hydration in a specimen allows it to harden and gain strength at ambient temperature [16]. However, Gomaa *et al.* conclude that the compressive strength of the oven cured samples were higher when compared with the samples cured at ambient temperature [19]. To increase the utilization of the fly ash in concrete, it must demonstrate the performance that is comparable to conventional Portland cement products. Studies recorded shows that high volume fly ash (HVFA) concrete replacing 60% of the OPC has outstanding mechanical properties and durability, even higher than the OPC Concrete [23]. Fly ash based GPC demonstrated other good vital properties like acid resistance, low creep, and low shrinkage [24].

The curing temperature, curing process, and the fineness of the fly ash content play a significant role in the development of compressive strength for the geopolymer. With proper curing temp (140°F ~ 190°F), the GPC can gain strength of 5690 ~ 7100 psi [25]. In another study conducted by Palomo *et al.*, the compressive strength was not affected by the alkaline solution to fly ash ratio [26].

2.3 Frontal Polymerization

This is a process in which a monomer with the presence of an initiator cures into a polymer through propagating the reaction at the front due to thermal diffusion. It has a wide range of application in rapid polymer synthesis and reduction in energy requirement. There are three different kinds of FPs: photo, isothermal, and thermal.

The photo FP uses a photo-initiator, which has a high UV light absorbance and decomposes when exposed to the light forming free radical that carries the front forward [27]. Isothermal FP involves adding a seed of consequential polymer into a solution of monomer and initiator. The polymer should be soluble in the solution, which has a viscous region where the polymerization rate is higher due to the Norrish – Trommsdorff effect [28].

Chechilo *et al.* first studied thermal FP. This kind of FP generates heat, which carries the reaction forward making a chain of polymers. The reaction is divided into three different stages: Initiation, Propagation, and Termination. In the first stage, a radical heat is generated from the initiator with the application of external heat or energy source which attaches with the monomer to form a monomer radical, capable of growing into a chain of polymers by reacting with other nearby monomers (propagation). Finally, a stable bond is formed between two radicals leading the reaction to termination. The study by Chechilo was conducted with methyl methacrylate (MMA) as a monomer and benzoyl peroxide as an initiator. Later, Pojman *et al.* revived the frontal polymerization and studied traveling of fronts in thermal free radical initiators over different kinds of monomers at ambient pressure [29].

The FP reaction can be varied over different temperature parameters and velocity of the front depending upon the type of monomer and its properties. The study conducted by Nason *et al.* on the multi-functional methacrylate induced by UV light finds Trimethylolpropane triacrylate (TMPTA) to have the shortest time to reach the peak temperature and the highest velocity among the other monomers as well [30]. The front temperature and velocities increase by the addition of more initiators [31]. The unstable initiator decomposes faster with high temperatures. An experiment performed by Goldfeder *et al.* shows the effect of the initial temperature of the monomer in the reaction and initiator concentration in the maximum temperature of the front, final degree of conversion, and propagation velocity [32].

Peroxide when used as the initiator usually has a high temperature, and when a monomer with a low boiling point and an unstable initiator is used, the reaction produces a substantial amount of gas that forms bubble [33]. Application of pressure during the reaction is one of the techniques to eliminate bubble formation, but it was not found to be suitable for the study [33], [34]. Another approach is to use an initiator like Aliquat persulfate (APS) or some reactive monomer - initiator solution that can be used in high boiling solvent such as Dimethyl Sulfoxide (DMSO) or Di-methyl Formamide (DMF) to propagate the front in ambient pressure which produces few or no gas bubbles [35]. Acrylamide polymerization with ammonium, potassium, or sodium persulfate as an initiator propagates with no gas production [36], [37].

CHAPTER 3

METHODS

3.1 Materials Used

The chemicals that were used in the development of frontally polymerized geopolymer concrete were filtered MMA, TMPTA, DMSO, ALPS, and Na2SiO3. All the chemicals were obtained from Sigma Aldrich and used as it was received, except for ALPS and filtered MMA. ALPS was synthesized by ionic exchange from APS and Tricaprylmethylammonium Chloride (Aliquat 336®). The MMA received was filtered through with alumina column to remove Mequinol (MEHQ) inhibitor. In addition to the chemicals, Class F fly ash was used along with sand as a filler material. The sand passing through sieve 20 and retaining at sieve 50 was used to make the 2" × 4" cylindrical samples, which were used to evaluate for the compression strength.

3.1.1 Synthesis of Aliquat Persulfate

Materials required:

- Tricaprylmethylammonium Chloride also known as Aliquat 336®
- Ammonium Persulfate 228.2 g/mol
- Diethyl ether
- Water
- Anhydrous Sodium carbonate

Procedure:

- 42.35 g APS was dissolved into 150 mL of DI water, then placed into an ice bath to cool.
- 50 g of Aliquat 336® was dissolved into 50 mL of diethyl ether.
- The Aliquat-ether solution was placed in an ice bath on a magnetic stir plate.
- The cold APS-water solution was slowly added as the Aliquat-ether solution was continuously stirred. The solution was covered and stirred in the ice bath for 30 minutes (Figure 3-1).
- The solution was poured into a separatory funnel and allowed to stand until the organic and aqueous phases separated. The aqueous layer was removed, and the organic phase was placed back into the ice bath (Figure 3-2).
- 100 mL of cold DI water was added to the organic solution and stirred for 10 minutes. The aqueous layer was removed with a separatory funnel. This procedure was repeated two times for a total of three items of washing.
- After the third wash, the organic phase was decanted into an Erlenmeyer flask. Approximately, 2 grams of anhydrous sodium carbonate was added to remove any water in the organic phase.
- The organic phase was placed into a 300 mL round bottom flask and roto-vaped to remove ether from the solution (Figure 3-3).

Figure 3-1: Stirring of Aliquat-Ether Solution in an Ice Bath.

Figure 3-2: Separation of Organic Phase Through a Separatory Funnel.

Figure 3-3: Rotovaping of the Mix to Remove Excess Ether Present.

3.1.2 Filtration of MMA to Remove MEHQ Inhibitor

A 50 ml burette is washed thoroughly with acetone to clean any residue of other chemicals before use. A small piece of cotton wool is added at the bottom of the alumina column to screen any alumina from passing into the filtered MMA. The setup is clamped with a funnel on top and MMA is poured and allowed to flow through the column (Figure 3-4). The MMA dripping out of the alumina column is collected in a jar and stored before use.

3.2 Chemical Properties and their Structures

The description of various materials used along with their molecular formula is presented below. The chemical structure of the materials used can be found in Table 3-1.

3.2.1 Geopolymer Mix

3.2.1.1 *Sodium Silicate (SS)*

SS is a general name for chemical compounds of silica and sodium with one of the following formulas:

- $Na_{2x}SiO_{2+x}$
- $(Na_2O)_x \cdot SiO_2$

These compounds include sodium metasilicate, sodium orthosilicate, and sodium pyrosilicate. SS is generally a white or colorless powder that is soluble in water. When dissolved in water, it produces a basic or alkaline solution. It is used in a wide variety of industrial applications and is an important component of cement and silica materials.

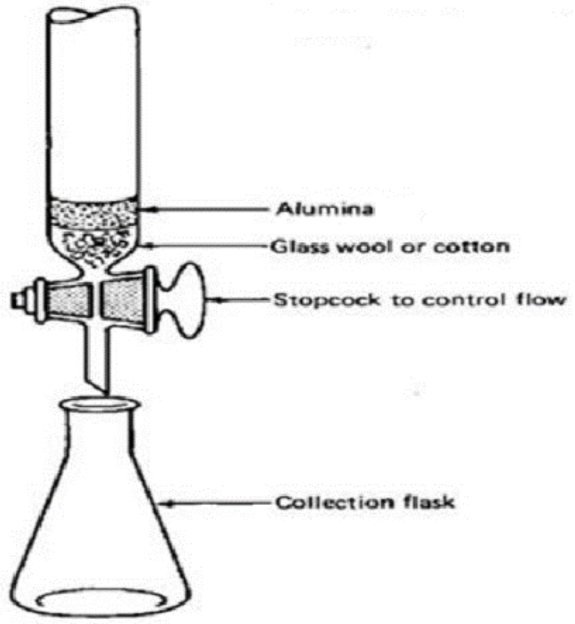
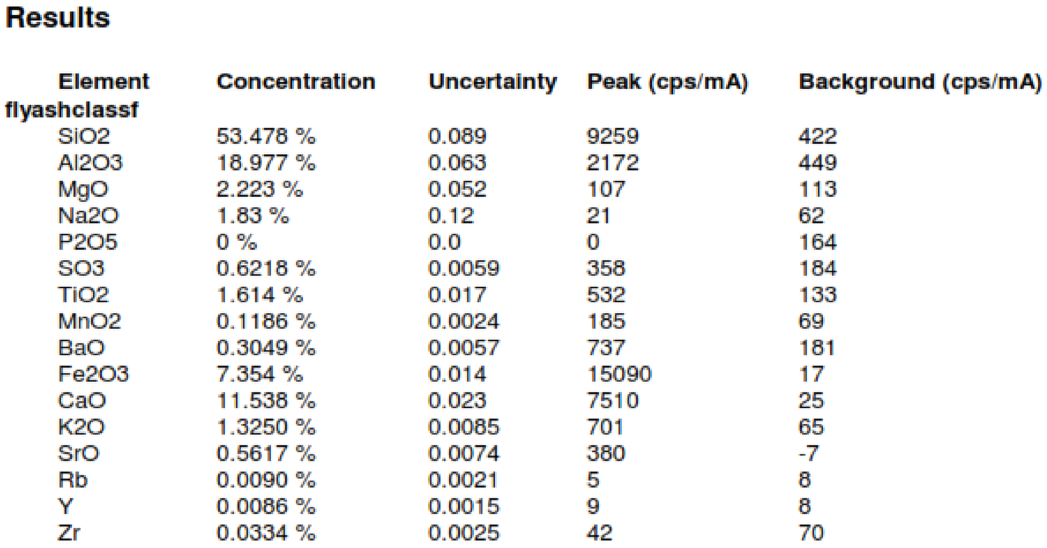


Figure 3-4: Setup to Remove MEHQ from MMA with Alumina Column.

3.2.1.2 *Fly Ash*

Fly ash is a product of coal combustion. Its chemical composition varies that contains SiO2, Al2O3, Fe2O3, and CaO. In addition to unburnt carbon, it may contain traces of metals such as arsenic, beryllium, boron, cadmium, chromium, cobalt, lead, manganese, mercury, molybdenum, selenium, strontium, thallium, and vanadium. Fly ash was previously a waste product of coal-fired power plants, but it is now used as a building material as a replacement for Portland cement. Fly ash is pozzolanic and can form cement. Fly ash particles form in the air from exhaust gases and tend to be generally spherical, ranging from 0.5 to 300 µm (Figure 3-5). The XRF (X-ray fluorescence) results for Class F Fly ash has been listed in Figure 3-6.

Figure 3-5: Class F Fly Ash.



Results

Element	Concentration	Uncertainty	Peak (cps/mA)	Background (cps/mA)
flyashclassf				
SiO2	53.478 %	0.089	9259	422
Al2O3	18.977 %	0.063	2172	449
MgO	2.223 %	0.052	107	113
Na2O	1.83 %	0.12	21	62
P2O5	0 %	0.0	0	164
SO3	0.6218 %	0.0059	358	184
TiO2	1.614 %	0.017	532	133
MnO2	0.1186 %	0.0024	185	69
BaO	0.3049 %	0.0057	737	181
Fe2O3	7.354 %	0.014	15090	17
CaO	11.538 %	0.023	7510	25
K2O	1.3250 %	0.0085	701	65
SrO	0.5617 %	0.0074	380	-7
Rb	0.0090 %	0.0021	5	8
Y	0.0086 %	0.0015	9	8
Zr	0.0334 %	0.0025	42	70

Figure 3-6: XRF Report of Fly Ash Used.

3.2.1.3 *Sand*

Sand is a naturally occurring quartz composed of SiO2. It may contain various other traces of minerals depending on source or location. The size of sand particles can vary widely, and it may also include other small fragments of mineral or biological origins such as granite or mollusk shells. Sand is used as a precursor material in the production of glass, quartz, and silica.

Figure 3-7: Sand Used as Filler Material.

3.2.2 Monomer

3.2.2.1 *Methyl Methacrylate (MMA)*

MMA is an organic monomer with the chemical formula of CH2 = C (CH3) COOCH3 and a molecular weight of 100.12 g/mol. It is a methyl ester (CH3-O) of methacrylic acid, an industrially important component of many plastics. It is a clear liquid with a density of 0.94 g/cm and is miscible with water as well as most organic solvents. MMA is used for the synthesis of poly (methyl methacrylate) or PMMA. It is also used as a basis for other plastics and as a co-monomer for hybrid plastics such as styrene, vinyl, and hydrogels.

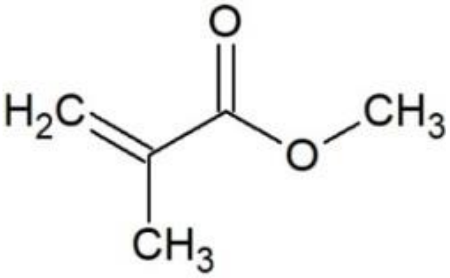


Figure 3-8: Chemical Structure of MMA.

3.2.3 Crosslinker

3.2.3.1 *Trimethylolpropane Triacylate(TMPTA)*

TMPTA is a trifunctional monomer containing three acrylate functional groups. Its chemical formula is C15H10O6 and has a molecular weight of 236.32 g/mol. At a standard state (25°C, 1 atm), it is a clear liquid with a density of 1.06 g/cm. It is used as a crosslinking agent to interconnect polymer strands by covalent bonds. TMPTA can be blended with other monomers and subsequently copolymerized into a polymer matrix. The purpose of cross-linking polymers is to alter physical characteristics of a polymer such as strength, rigidity, swelling and melting point, or to combine attributes of different polymers.

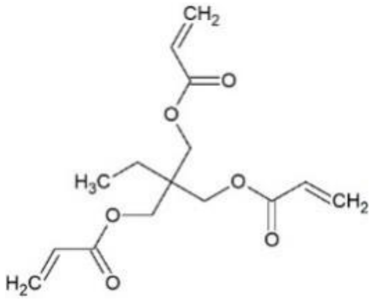


Figure 3-9: Chemical Structure of TMPTA.

3.2.4 Solvent

3.2.4.1 *Dimethyl Sulfoxide (DMSO)*

DMSO is an organosulfur compound. Its chemical formula is (CH3)2SO with a molecular weight of 78.13 g/mol. It is a colorless liquid with a density of 1.1004 g/cm. It is a very commonly used polar aprotic solvent that can dissolve polar and nonpolar compounds and is miscible with most organic solvents and water. Aprotic polar solvents like DMSO have large dipole moments but do not have hydroxyl (-OH) or amine (-NH) bonds and cannot hydrogen bond with themselves. DMSO is used as a solvent for chemical reactions involving salts and is relatively non-toxic, so it is widely used in biological applications.

3.2.5 Initiator

3.2.5.1 *Aliquat Persulfate (ALPS)*

ALPS is synthesized from Aliquat 336® by substituting the negatively charged chlorine ion with a persulfate anion. The positively charged quaternary amine cation coordinates with the persulfate anion forming a new ionic liquid. The process is performed in a two-phase reaction scheme with an organic and an aqueous layer. The layers are agitated to allow transfer of the respective anions (Figure 3-11). After the reaction has completed, the layers are separated, and the organic layer is dried to remove water and rotovaped to remove the residual solvent. The resulting viscous liquid is translucent and pale yellow. The chemical formula is 2[C25H54N+].[S2O82-].

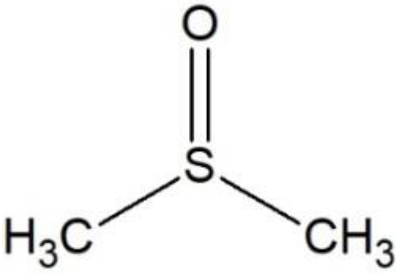


Figure 3-10: Chemical Structure of DMSO.

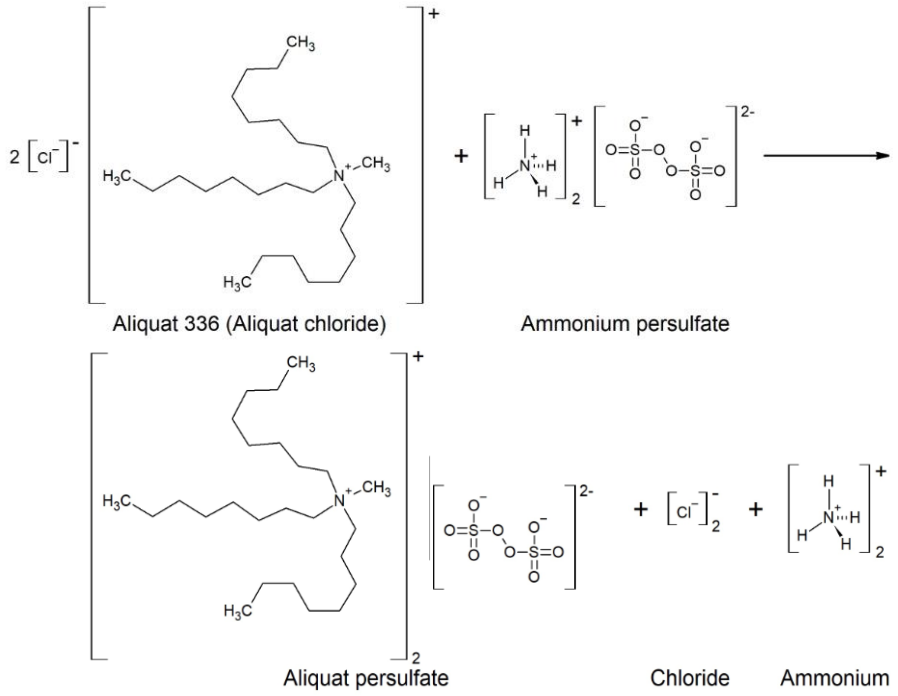


Figure 3-11: Schematic Drawing of the Ionic Exchange Reaction to Synthesize ALPS.

3.2.5.2 *Aliquat 336®*

Aliquat 336® is a quaternary ammonium salt. It has a chemical formula of C25H54N+Cl- and a molecular weight of 404.16 g/mol. It is a viscous liquid at 25°C with a density of 0.884 g/cm. It is an ionic liquid which is salt in a liquid state. It is widely used as a phase transfer catalyst. The chlorine anion can be substituted for many other negatively charged ions. The chemical structure of Aliquat 336® is shown in Figure 3-12.

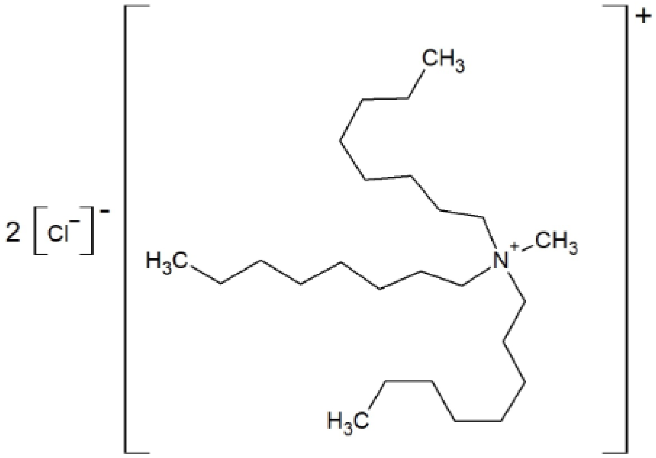
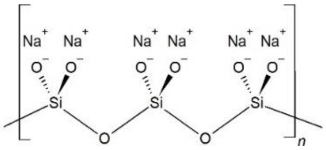
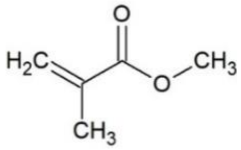
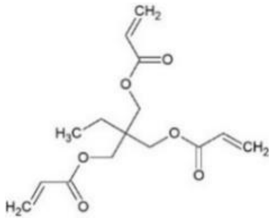
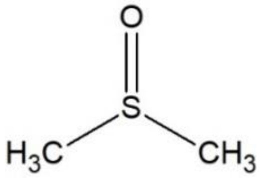
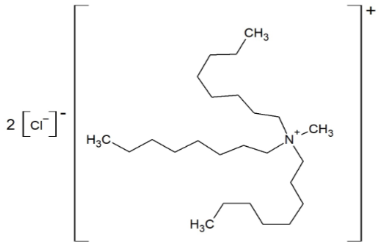
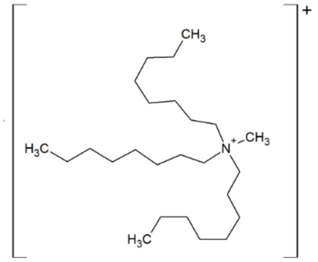
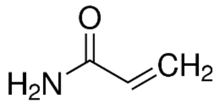


Figure 3-12: Chemical Structure of Aliquat 336®.

Table 3-1: List of Chemicals Used, their Chemical Formula and its Structure.

Chemical Name	Name Used	Chemical Formula	Chemical Structure
Sodium Silicate	SS	Na_2SiO_3	
Methyl methacrylate	MMA	$C_5H_8O_2$	
Trimethylolpropane Triacrylate	TMPTA	$C_{15}H_{20}O_6$	
Dimethyl sulfoxide	DMSO	$(CH_3)_2SO$	
Aliquat persulfate	ALPS	$2[C_{25}H_{54}N^{+}]^{\cdot}$ $[S_2O8^{2-}]$.	
Tricaprylmethylammonium Chloride	Aliquat 336®	$C_{25}H_{54}N^{+}Cl$	
Acrylamide	AAM	C3H5NO	

3.3 Preparation of Cylindrical Samples

3.3.1 Frontally Polymerized Geopolymer Concrete Sample

To evaluate the compressive strength for the frontally polymerized cylinders, seven samples of each were prepared in three batches to test for 28, seven and three days' strength. Filtered MMA and TMPTA in specific ratio were poured in a beaker and dissolved in DMSO to prepare the mix. The mixture was blended with the help of a shear mixer for about 10 minutes. Later, Na2SiO3 and ALPS was added into the beaker and stirred for another 10 minutes. The ratio was thoroughly mixed followed by adding fly ash and sand into the mixture to form a thick workable paste before pouring (Figure 3-13).The mortar was prepared following the above steps and poured into a 2" × 4" cylinder mold and was tampered 50 times by a steel rod to ensure uniformity throughout the sample (Figure 3-14). The samples were then placed below the halogen lamp R7s (200 W, 120 V) for eight minutes. The heat produced from the lamp was used as a curing medium. It took almost 40 minutes for the whole sample to cure and harden (Figure 3-16). The samples were stored for specified curing days before they can be tested for compressive strength.

Figure 3-13: Mixing of Chemicals with Sand and Fly Ash (Left), Cylindrical Molds to Prepare Samples (Right).

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Figure 3-14: Tampering of the Mixture (Left), Mixture Poured into a Cylindrical Mold (Right).

Figure 3-15: Application of Heat through a Heat Lamp.

Figure 3-16: Samples Obtained After Heat Application.

3.3.2 Fly Ash Based Geopolymer Concrete

Three batches of seven samples were prepared for 28, seven and three days' curing. GPC cylinders of 2" × 4" were prepared to test for compressive strength. The alkaline solution (i.e. Na2SiO3 and NaOH) was prepared with a ratio of two to one in a separate beaker by mixing it evenly for 5-10 minutes. The sand and fly ash of the same batch were used to prepare the samples to make the comparison more reasonable. The sand to fly ash ratio was three to one while the proportion of sand-fly ash to the alkaline solution was 5.2:1. This ratio of 5.2 was concluded with multiple trials to make the mix workable and most suitable. This final mix was stirred well with the help of a mixture and poured in a cylinder mold and tampered 100 times to fill in any voids present (Figure 3-18). After the mix was poured, the mold was wrapped in a plastic sheet to prevent evaporation and placed in a hot oven at 140o F for 24 hours before it could be tested for compression (Figure 3-20).

Figure 3-17: Mixing of Sand, Fly Ash, and Alkaline Solution.

Figure 3-18: Pouring of the Mixture into Molds.

Figure 3-19: Covering the Samples before Oven Curing.

3.3.3 OPC Samples

OPC Samples were prepared to have a relative study of compressive strength with the strength developed by the geopolymer and frontally polymerized cylinder. The samples were prepared with sand to cement ratio of three to one and a water content of 0.48. The sand-cement and water were mixed with the help of a kitchen mixer and poured into the cylinder. The mix was tampered 100 times with a rod to ensure all the air voids are fulfilled (Figure 3-21). The samples were air dried for 24 hours and cured with tap water for 28 days to gain the full strength. Three sets of seven cylinders are prepared for each batch to test the strength for 28, seven and three days (Figure 3-22).

Figure 3-20: Placing of Samples in an Oven (Left), Prepared Samples (Right).

Figure 3-21: Mixing of Cement, Sand, and Water.

Figure 3-22: Pouring of the Mixture in the Mold (Left) and Samples Obtained After Curing (Right).

3.4 Selection and Optimization of the Mixer

The experiment began with powdered Acrylamide (AAM) as a monomer and APS as an initiator because both the monomer and initiator were water soluble. One gram of Acrylamide powder was dissolved in an equal amount of water; 0.10 g of APS was added to the last and mixed for the next five minutes. Heat from the soldering iron rod was used to initiate the reaction. Polymerization was noticed along with the gas released due to decomposition of APS into ammonium ions. Also, the polymer produced was feeble and disintegrated quickly. Later, 0.10 g of MBAA was added as a cross-linker along with the acrylamide and dissolved into an equal amount of water. The entire mixture was stirred for 15 minutes to ensure that all the components are dissolved completely. The solution was placed into the glass plate, and heat from the soldering iron rod was used to initiate the front. The reaction propagation was seen after a few seconds, and the final product looked robust than earlier. The problem with the mix was the front quenched without terminating the reaction.

The amount of MBAA and the initiator (APS) was increased further without changing the amount of water in the solution and mixed for 15 minutes. The mixer consisted of one gram of Acrylamide, MBAA, and APS with an equal amount of water. This produced a hazy thicker solution, which was poured into the glass plate and heat was applied at one end. Rapid frontal polymerization was noticed, but the reaction stopped upon removal of the heat source.

After numerous attempts, the best amount that would carry the front and give the final robust product was one gram of acrylamide, MBAA, and 2.10 g of APS with one gram of water. The monomer was mixed 10 minutes using a shear mixture until completely dissolved. MBAA was added to the solution and mixed for another 10 minutes without the initiator and an additional five minutes after the initiator was added. The much thicker paste was observed, and the mixture was placed on the glass plate. The tip of the iron rod acted as a heat source and was applied for a few seconds and removed after which the front carried forward on its own to give a brittle polymerized material.

The final solution that functioned as a frontal polymerization was mixed with fly ash along with Na_2SiO_3 and NaOH. The ratio of fly ash to the alkaline was 0.35 while the ratio of Na_2SiO_3 to NaOH was two to one. The thick paste was formed while mixing all the chemicals (without NaOH) and fly ash. When introduced into the paste, NaOH would turn the paste into dry grains making it impossible to apply heat and see the polymerization. Hence, it was excluded from the fly ash mixture.

For the next trial, 0.98 g of MBAA and Acrylamide was dissolved into an equal amount of water by stirring for about 10 minutes. Then, 1.30 g of Na_2SiO_3 was added to the mixture and stirred for an additional five minutes. After that, 2.06 g of APS was added to the solution and mixed well. This thick solution was able to absorb 3.70 g of fly ash turning it into a workable paste that can be poured into a cubical mold. The heat was applied through the soldering rod for 10 to15 seconds, and polymerization was observed. Initial observation revealed a hard, smooth surfaced sample. Several similar specimens were prepared, and the results seemed identical (Figure 3-23). During multiple trials, it was found that the fly ash mortar had a very short pot life period of about five to 10 minutes after the addition of the initiator. The specimens were then submerged into water to see if it remains intact with water contact. The samples slowly disintegrated and broke apart after a while.

The monomer (AAM) was replaced with MMA rejected hereafter. Since MMA is an organic monomer, it was not able to dissolve APS into itself. Water was added to the solution to dissolve APS and MBAA. The ultimate combination contained MMA, MBAA, Water, SS, and APS. This mix was blended with fly ash, and heat was applied via soldering iron rod. The paste was thinner than before and polymerization was not observed and hence disregarded.

MMA was found to have an inhibitor MEHQ into it, which might hinder the polymerization process. The MMA was made to pass through an alumina column to filter out the inhibitor and collected in a separate jar. ALPS was synthesized by ionic exchange and was used as the organic initiator because ammonium present in APS was substituted by aliquat which would reduce the formation of ammonium ions upon application of heat. TMPTA was identified as a suitable monomer and as a cross-linker for the polymerization process.

Early attempts included mixing of one gram of MMA and one gram of TMPTA for five minutes; 0.30 g of ALPS was added into the solution and mixed well for the next five minutes. The solution was poured in a capped test tube, and heat was applied at the bottom of the tube through a soldering iron rod. Polymerization was observed after multiple touches at a different location, and it took 47 seconds to complete the reaction. The same mixture was repeated with eight gram of fly ash and poured into a capped glass tube. Multiple touches from the rod tip were required and took almost five minutes to complete the reaction. The final product looked smooth and robust and remained intact when submerged into water.

Figure 3-23: Specimen Prepared with Various Shapes.

DMSO is an organic solvent introduced into the solution. One gram of DMSO was added at the beginning for the mixture without fly ash and poured into a capped test tube. The bottom of the tube was made in contact with the tip of the iron rod for a short period (almost eight seconds). The polymerization was observed and the whole reaction completed within 50 seconds. The same mix was used for the next batch. The heat was provided via a heat lamp at the bottom side of the tube and the front traveled upwards after 20 seconds of heat applied and continued on its own to complete the reaction.

Sand was introduced into the mixture along with fly ash to prepare a thicker concrete paste. The sand was a sieve for uniformity. The sand was made to pass through no 20 sieves and retained at no 40 sieves after sieving it for 20 minutes. The percentage by weight was 8.98% for MMA, TMPTA, and ALPS, 4.49% of DMSO, 45.7% of Sand and 22.87% of fly ash. With each chemical added, it was mixed for five minutes using a kitchen mixer and was poured in the one inch cubical mold. Heat from the lamp was applied as a heat source for one and a half minutes and removed. The reaction took eight minutes to complete, and the final product was a solid cube. However, the solid cube had a rough surface and patchy at some locations (Figure 3-24). Thus, more fly ash was needed for a smoother surface.

Figure 3-24: 2" × 2"× 2" Cube Sample with a Rough Surface.

Na_2SiO_3 is one of the vital components in geopolymer in the mix. In the presence of heat, it binds with fly ash to form a solid geopolymer concrete. With its addition, the percentage of ratio was changed to 4.9% for MMA, TMPTA and ALPS, 2.1% of DMSO, 39.1% of Sand, 42.6% fly ash, and 1.5% of sodium silicate. The mixer was added into two inch cubical mold and heat was applied from a heat lamp for eight minutes. The reaction took about 45 minutes to complete and gave a smoother finish product, and thus was introduced into the mix (Figure 3-25).

Figure 3-25: 2" × 2"× 2" Cube Sample with a Smoother Surface.

The same ingredients percentage by weight was mixed and poured into a 2" × 4" cylinder mold. Heat from the lamp was operated for eight minutes, and the reaction took about 50 minutes to complete. The mold was torn apart, and a solid cylinder with a smooth surface was discovered. The ratio was found to be best working for frontal polymerization with sand and fly ash. Figure 3-26 shows some of the cylinder sample prepared with the ratio stated. Figure 3-29 to Figure 3-31 show various trials used to optimize the procedure. Table A- to Table A- have a summary of the combination of chemicals used in different ratios and the results observed.

Figure 3-26: Frontally Polymerized 2" × 4" Cylinder.

3.5 Testing of Samples

Testing of the samples was performed in accordance to ASTM C39. The samples were prepared with three different materials: cement, geopolymer, and frontally polymerized geopolymer cured for 28, seven, and three days were developed in different batches and stored separately. These samples, when they reached the number of curing days stated, were tested for their compressive strength. The compression test was conducted on an ADMET 300 Kips compression testing machine (Figure 3-1). The loading channel applied was 450 lb., and the home rate was 0.05 in/sec. The jog rate during the test was set to be 1.0 in/sec and the loading rate used was (35 ± 7) psi.

Figure 3-27: Testing of Samples.

Figure 3-28: Samples After the Compressive Test.

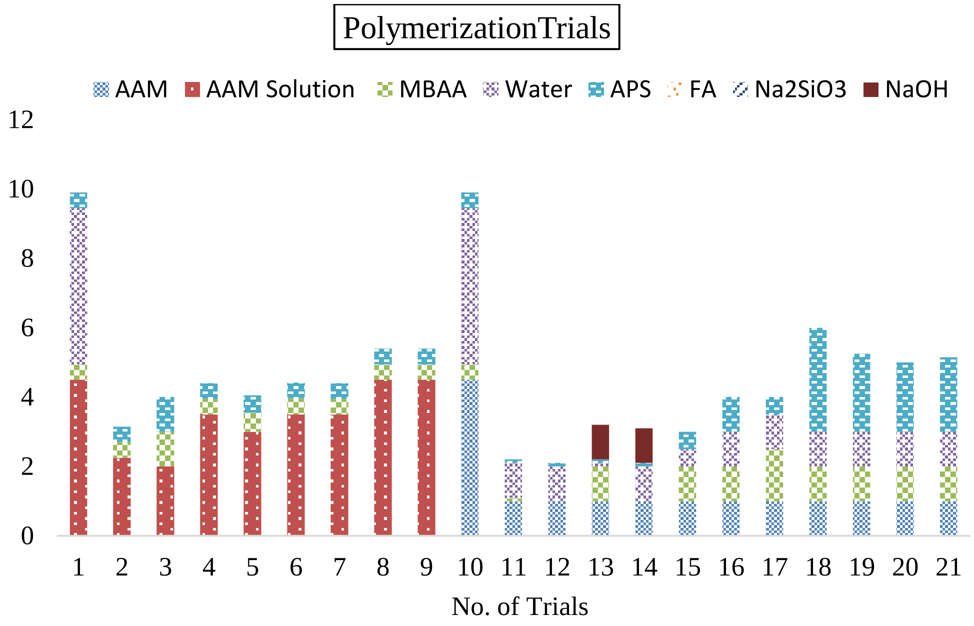


Figure 3-29: Polymerization Trials with Different Chemicals – I.

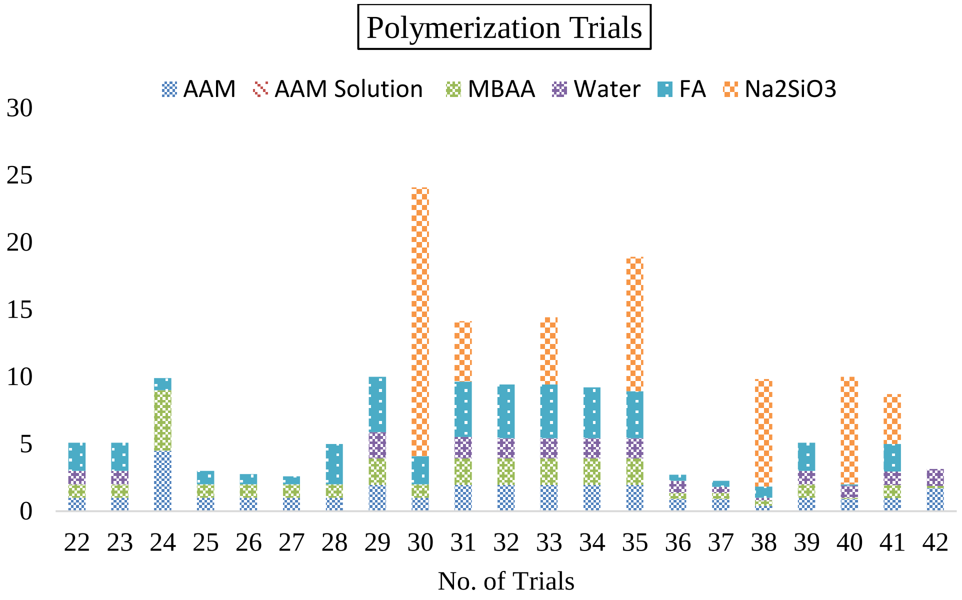


Figure 3-30: Polymerization Trials with Different Chemicals – II.

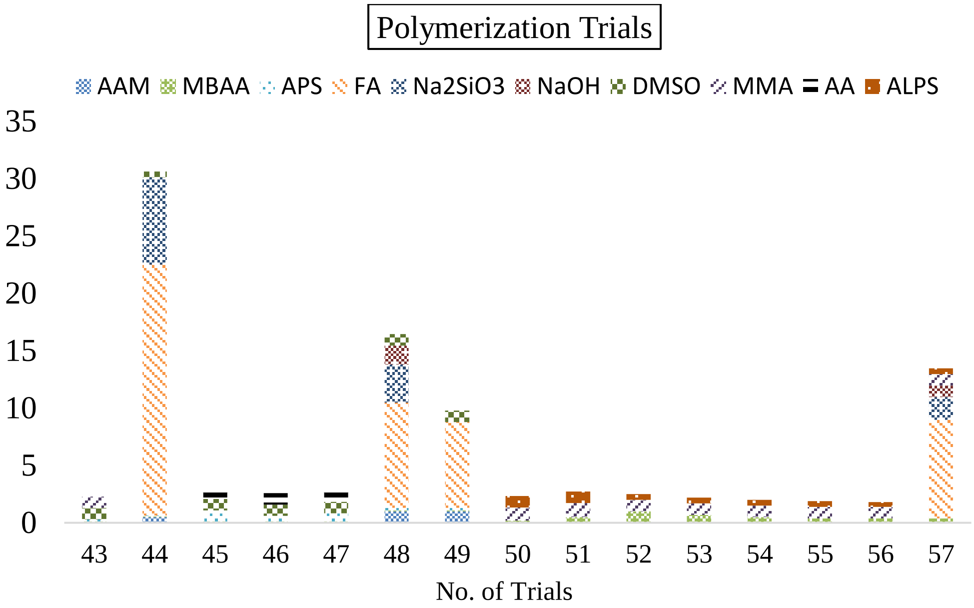


Figure 3-31: Polymerization Trials with Different Chemicals – III.

CHAPTER 4

RESULTS

4.1 Compressive Strength

Crushing the material in compression testing machine is the standardarized global method to test the material for compressive strength. It is one of the vital test performed for the material because it will determine the load the material can bear before failure. The samples prepared with different material for various curing periods were placed in between the plates of the compression testing machine and load was applied until the samples were crushed. The data for load, peak stress and modulus of elasticity were recorded for individual specimen. The data are tabulated below with the histogram for individual sample type.

4.1.1 Frontally Polymerized Sample

The compressive strength data for the samples after 28, seven and three days are listed in Table B- to Table B-. The average strength for the 28 days samples was 685 psi while the average strength for seven and three days was 876 psi and 910 psi, respectively. The strength of the samples seems to reduce by almost 33% from three days to 28 days period. The graph of the compressive strength for the individual sample is presented below for specific curing period.

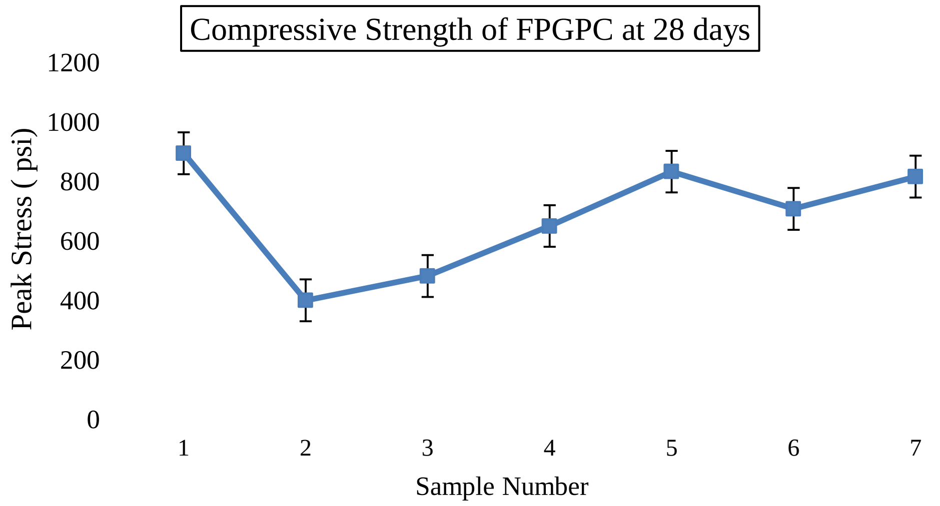


Figure 4-1: 28 Days' Compressive Strength of FPGPC.

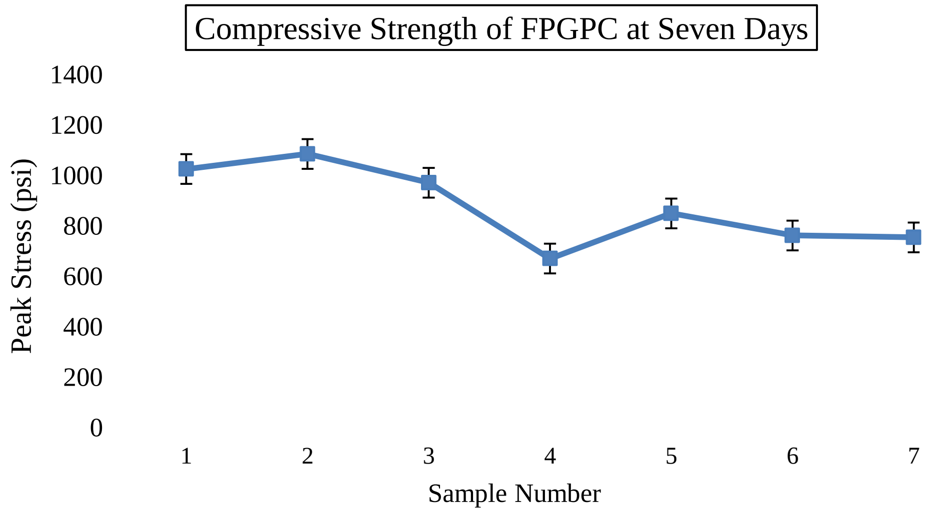


Figure 4-2: Seven Days' Compressive Strength of FPGPC.

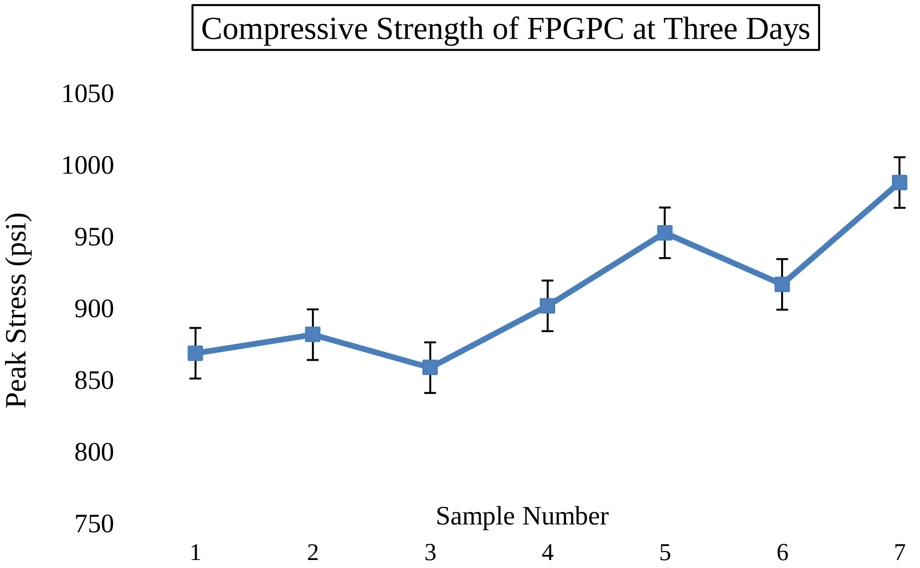


Figure 4-3: Three Days' Compressive Strength of FPGPC.

4.1.2 Fly Ash Based Geopolymer Concrete

Seven samples in three sets were prepared to test the compressive strength of the specimen for 28, seven and three days of curing period. From the test results, it was found that the average strength for the 28 days' samples was 2292 psi, while the average strength for seven and three days was found to be 3329 psi and 2817 psi. The strength of the samples seems to reduce by almost 45% from seven days to 28 days' curing period. However, the seven days' strength was higher by almost 18% when compared to three days' strength. The compressive strength data for the specimens is listed in Table B- to Table B- for 28, seven and three days of curing period, respectively. The histograms below show the peak stress for each curing period.

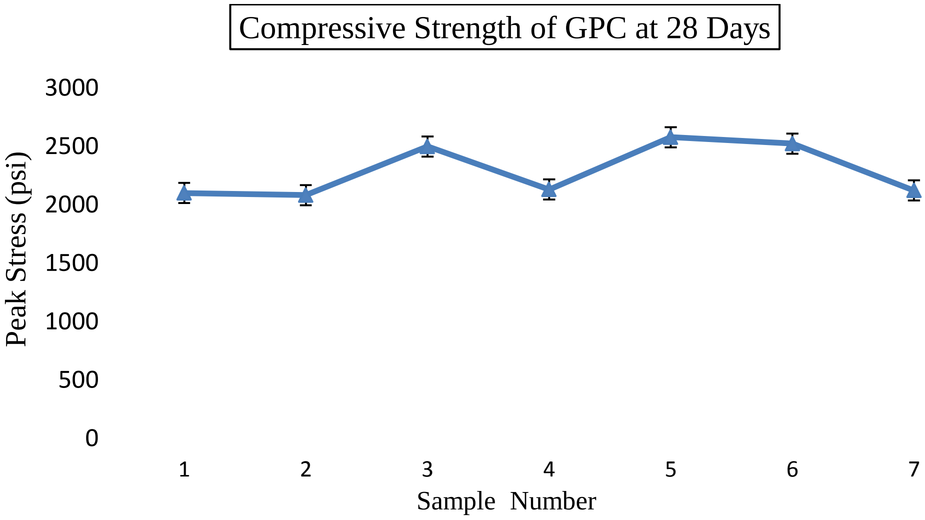


Figure 4-4: 28 Days' Compressive Strength of GPC.

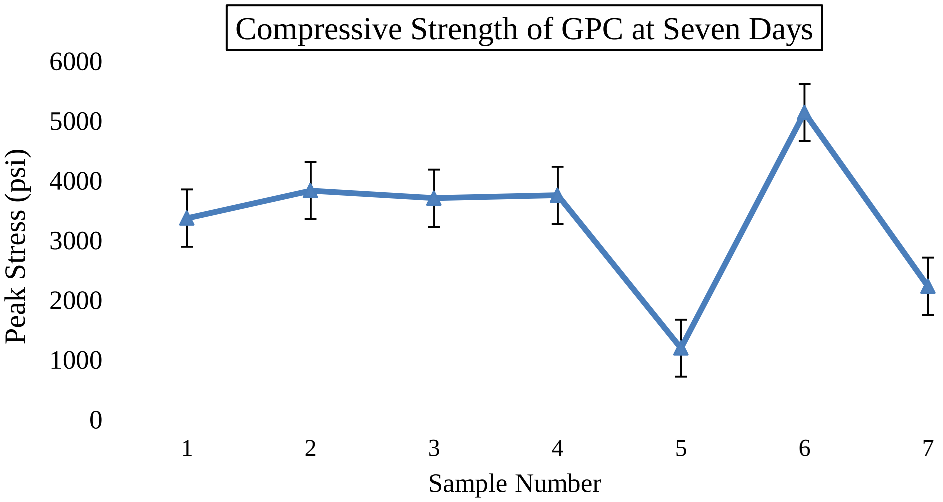


Figure 4-5: Seven Days' Compressive Strength of GPC.

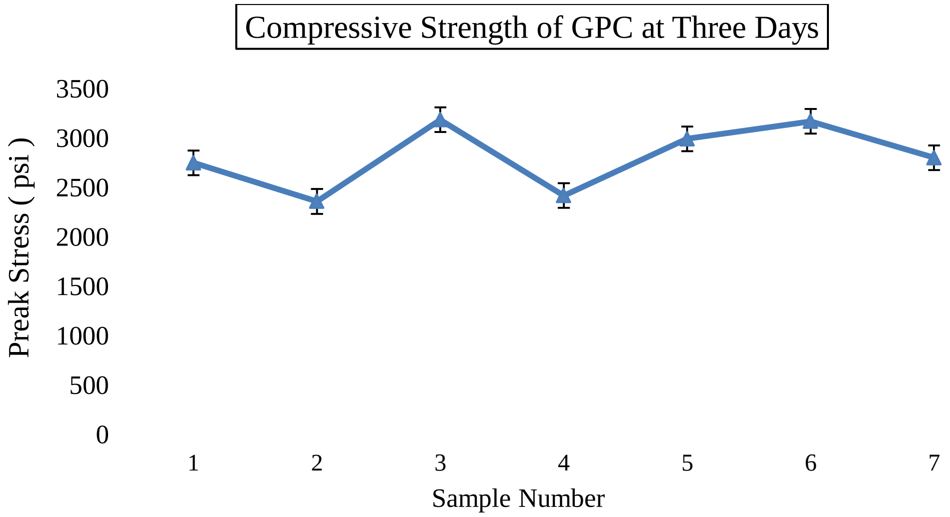


Figure 4-6: Three Days' Compressive Strength of GPC.

4.1.3 <u>OPC Samples</u>

Seven Cement cylinders were prepared in three different batches to test for 28, seven and three days' strength. The results show that the average compressive strength of the samples cured for 28 days was 3150 psi, while the average strength for samples cured for seven days was found to be 2650 psi. Similarly, the strength cured by samples cured for three days was 1757 psi. The strength seemed to increase significantly in the early days but was stable after it reached close to 28 days. In addition to that, the strength was increased by about 45% from three days to 28 days curing period. The compressive strength data for the samples can be found in Table B- to Table B-. The graphs below show the peak stress for each curing period.

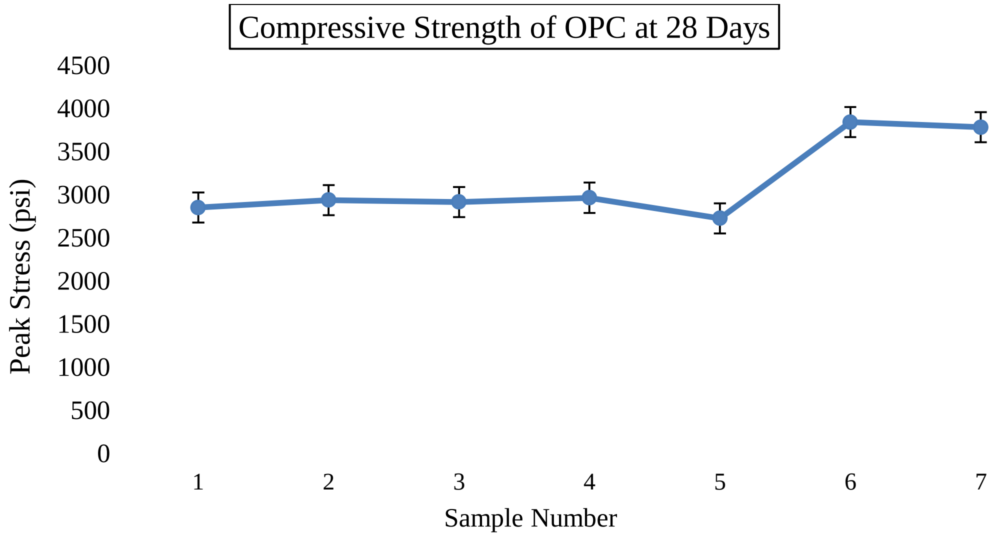


Figure 4-7: 28 Days' Compressive Strength of OPC.

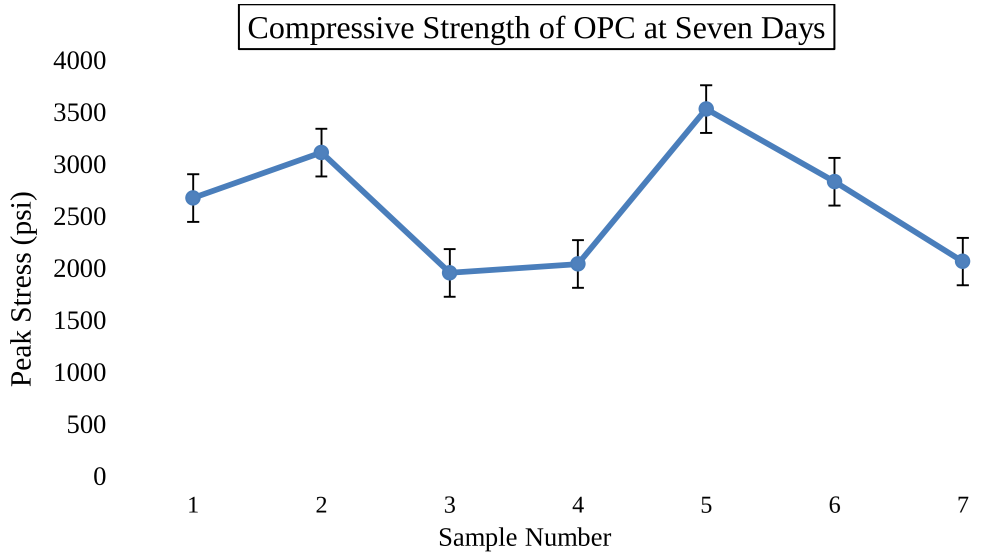


Figure 4-8: Seven Days' Compressive Strength of OPC.

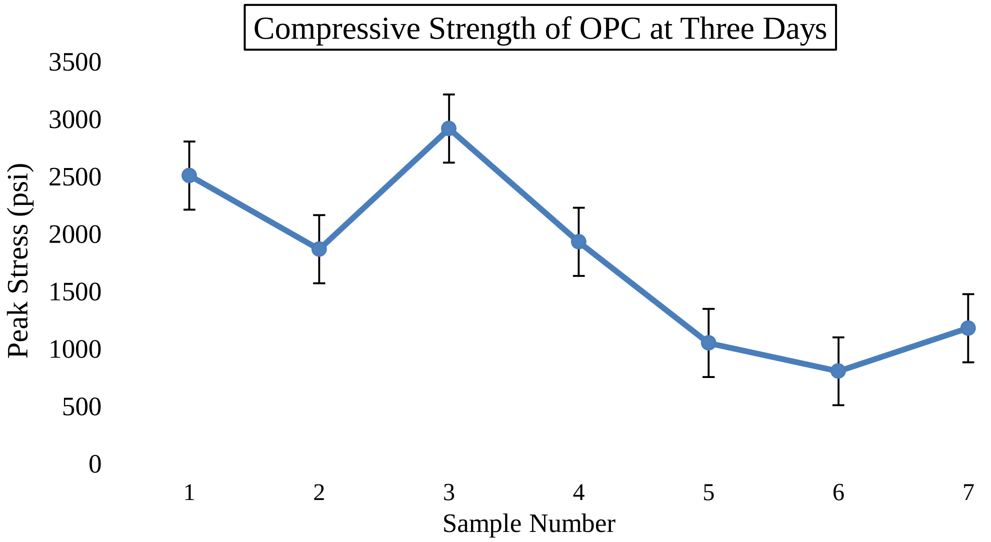


Figure 4-9: Three Days' Compressive Strength for OPC.

4.2 Comparison of Compressive Strength

The compressive strength for three different sample types cured for 28 days is plotted in Figure 4-10. The graph shows 28 days' strength for OPC samples to be the highest among all followed by GPC and FPGPC samples.

Figure 4-11 shows the average strength of the samples cured for seven days for the different samples. The strength for geopolymer and FPGPC samples were found to be increased compared to the 28 days' strength of the respective samples.

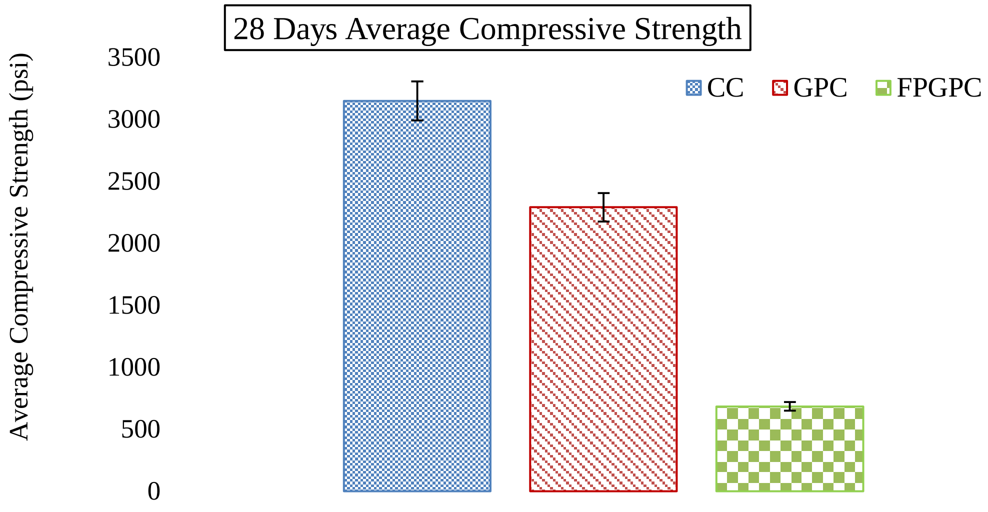


Figure 4-10: 28 Days' Average Compressive Strength for OPC, GPC, and FPGPC.

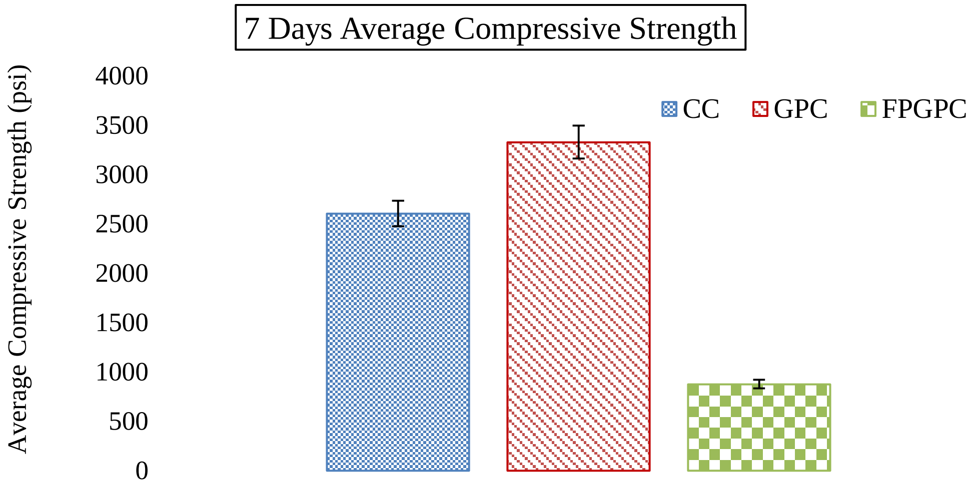


Figure 4-11: Seven Days' Average Compressive Strength for OPC, GPC, and FPGPC.

Similarly, Figure 4-12 shows an average compressive strength for samples cured for three days. The strength for cement concrete seems to decrease with a decrement in curing time. However, the geopolymer and FPGPC samples tend to increase the strength with lower curing time.

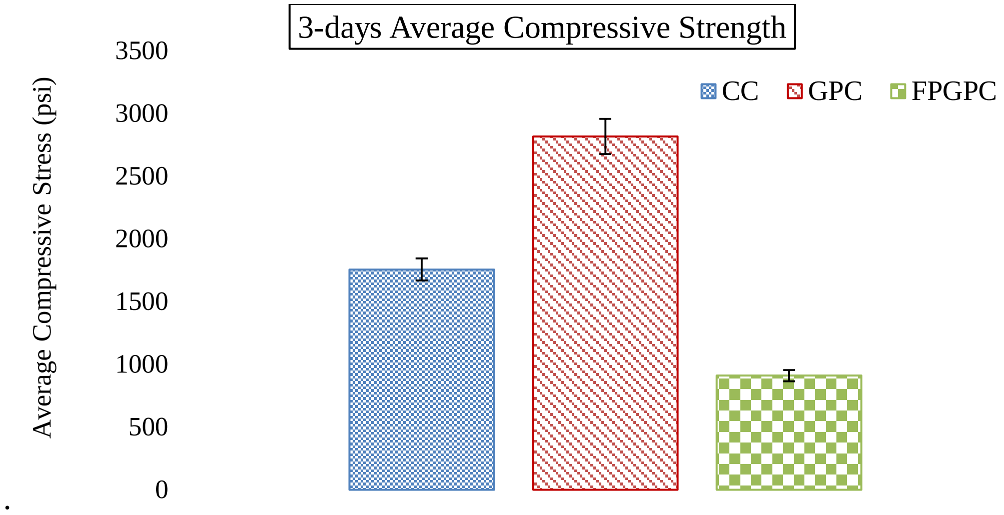


Figure 4-12: Three Days' Average Compressive Strength Data for OPC, GPC, and FPGPC.

4.3 SEM Images

SEM produces images by exposing the surface of a sample to a focused beam of electrons. The electron beam interacts with the atoms of the sample and reflects electrons, emits secondary electrons, and produces electromagnetic radiation such as X-rays and cathodoluminescence. Each of these phenomena can be detected and analyzed by specialized techniques. In SEM, the signal is converted into digital images with a resolution between 1 nm to 20 nm.

4.3.1 Description of SEM (Alfred Gunasekaran, Ph.D. IFM Resources)

The AMRAY SEM (Model: 1830) is a low-resolution scanning electron microscope, currently fitted with a tungsten filament as the electron source. This SEM is generally operated at slightly higher electron beam energies (15–30 kV), and it has a resolution of ~ 5-10 nm under optimum conditions. The electron optical column is maintained at low pressures (< 10-6 Torr) by a turbo molecular pump and an ion pump. All the SEM controls are manual, and the alignment of the electron column can be easily performed. The specimen chamber has a drawer-like design for loading specimens, and the working distance can be manually adjusted. This configuration allows for imaging both thick specimens and thin wafers in this SEM. The available X-Y motorized specimen stage facilitates locating the region of interest on the sample's surface.

Capabilities:

- Ordinary materials, such as metals, ceramics, polymers, and machined or stamped materials, concretes, and other solid materials can be imaged.
- Elemental X-ray analysis can be performed, and the images can be captured in digital format.
- Thick specimens with a height approximately 1.0 mm to 25 mm can be imaged.

4.3.2 Sample Preparation Protocol

The samples of frontally polymerized geopolymer concrete were organized by mounting strips of double adhesive sided conductive carbon tape upon a steel mounting stage. A granulated portion of the sample was dusted onto the adhesive surface. The stage mounted samples were then placed into the SEM and images were focused and balanced before recording.

4.3.3 Sample Characterization

The samples were found to contain spheres of various diameters and a granular aggregate (Figure 4-13). One of the components of the material was fly ash (Figure 4-15). Fly ash is a byproduct of coal burning in power plants. It forms in hot air from exhaust gases and aggregates into spherical structures. The granular aggregate is probably composed of sand (silica) granules, small fly ash residue, and sodium silicate and organic polymers due to the frontal polymerization materials.

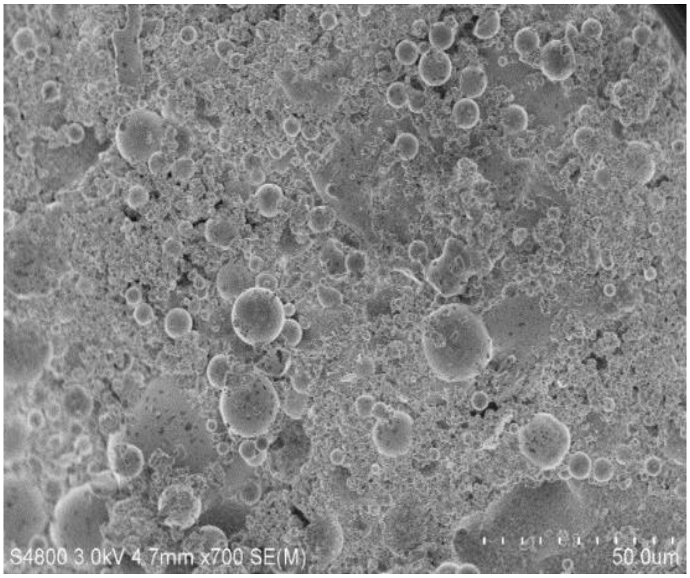


Figure 4-13: SEM Image of the Sample at Showing Spheres of Various Diameter.

Upon closer examination, it was found that the materials used in the preparation of the samples were not perfectly bonded but had smaller voids in between (Figure 4-14). These voids might be the cause for the samples to have low compressive strength in comparison to geopolymer or cement concrete. One of the reasons for the presence of voids might be due to the inability of the monomer or initiator to complete polymerization and form a solid polymer.

Also, the fly ash spheres found in the samples seem to be isolated with the remaining cluster of polymerized samples (Figure 4-15). To overcome this limitation, bonding is required between fly ash spheres with a polymerized cluster which might be obtained through an additional chemical like trimethoxysilane that has shown capabilities to enhance internal bonding of fly ash spheres.

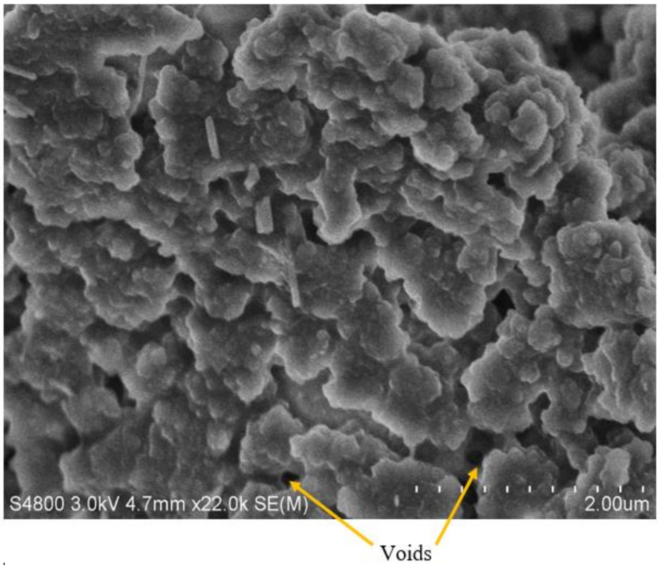


Figure 4-14: SEM Image Showing Voids Present in the Sample.

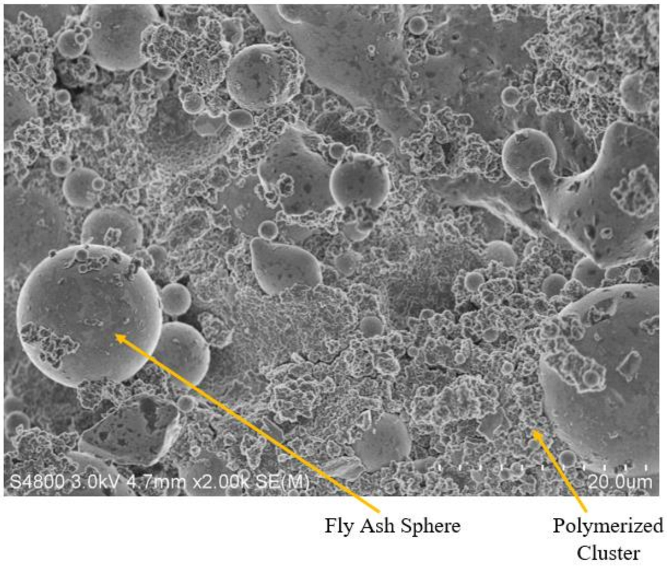


Figure 4-15: SEM Image Showing an Isolated Polymerized Cluster and Fly Ash Sphere.

CHAPTER 5

CONCLUSIONS AND FUTURE WORK

5.1 Conclusions

The early research and the experiment performed shows that the specimens cured by frontal polymerization technique can possess compressive strength comparable to geopolymer and cement concrete. From the study conducted, it was discovered that the strength of the specimen had an average compressive strength up to 910 psi, which is almost 50% of the strength developed by cement concrete when cured for three days. Also, the strength developed by the FPGPC for different curing period was relatively equal, which means it can gain early strength and does not depend upon the curing period to gain strength like CC. Besides these advantages, the experiment has the following useful benefits:

- The presented experiment reduces the curing time for a geopolymer concrete by a significant amount.
- The mix prepared has good pot life and workability.
- It can be blended with all sorts of different chemical such as magnesium hydroxide or sodium hydroxide to enhance its existing properties such as corrosion resistance, thermal resistance, or sulfate attack.
- The sample had air pockets on the sides which may be one of the reasons for its low performance in compression (Figure 5-1). One of the reasons for this

might be the presence of residual ammonium in the synthesized ALPS. Repetitive washing of ALPS with distilled water should help to get rid of the residue. Also, MMA is a very volatile monomer which might have been another reason for the presence of voids in the sample. Choosing a different non-volatile monomer might help to have a better product

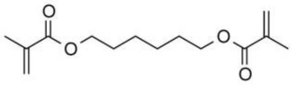
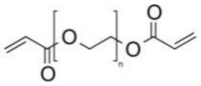
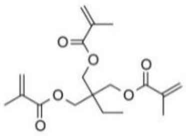
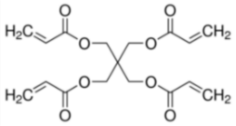
- Any existing chemicals used in the experiments can be altered to create a product with better performance and finish.
- Failure mode on the FPGPC samples were observed. Samples were spotted to have asymmetrical diagonal and vertical cracks along with complete crushing of samples from the bottom without a specific pattern of failure. One reason for this might be the friction along with the compressive force applied from the plates and the irregular voids present in the sample (Figure 5-2).
- Bonding abilities can be tested with several monomers and crosslinkers such as Hexamethylene diacrylate, Polyethylene (glycol) diacrylate, Trimethylolpropane ethoxylate triacrylates, and Pentaerythritol tetraacrylate to get materials with different strengths (Table 5-1).

5.2 Future Work and Implementation

The samples created using FPGPC have shown possibilities to utilize the industrial wastes (fly ash) and form a solid material with strength capabilities. The study can be multi-dimensional with curing time, different proportion of monomer-initiator used, the volume of the cross-linker, and the type of filler material. However, this research only focuses on studying the possibilities with different monomer and initiator ratios with fly ash and sand as a filler material.

Figure 5-1: Gas Pockets Seen on the Prepared Samples.

Table 5-1: Summary of Other Possible Monomers and Crosslinkers.

No.	Name of the Chemical	Chemical Structure
1	Hexamethylene Diacrylate	
2	Polyethylene (glycol) Diacrylate	
3	Trimethylolpropane Ethoxylate Triacrylates	
4	Pentaerythritol Tetraacrylate	

This study also emphasizes on curing the samples with a frontal polymerization technique, which has capabilities to propagate on its own after heat application for a short duration of time. This property integrated with the samples makes the process noble and different. The study can be expanded with changes in the curing method such as UV Cure or Solar cure, where sunlight can be used as a heat source to start the chain reaction. Also, different monomer and initiator with reformed chemical properties can be added in the mix to obtain the desired results.

Figure 5-2: Different Failure Pattern Observed in FPGPC Samples.

Figure 5-3 below shows an early study performed with solar curing to prepare the sample. The process took almost 12 minutes to complete and was exposed to concentrated light passing through a hand lens for about six minutes to give a finished product. Curing the samples with solar energy and utilizing fly ash makes the process environment-friendly to produce Green Concrete. This process not only utilizes the waste products but also saves significant curing energy overcoming the limitation possessed by traditional geopolymer concrete. In addition, the method saves land area used for discarding fly ash and cuts off CO2 released during cement production to acquire the green credits.

Figure 5-3: Curing Possibilities of the Sample with Sunlight.

Moreover, different chemicals can be integrated into the mix to obtain concrete with various properties. Some of the possibilities can be the addition of antibacterial chemicals to fight back against organics released in sewer lines, or chemicals that have abilities to sustain sulfate attack, prolonging the life of structures built, and overcoming some of the limitations of traditional concrete.

Figure 5-4 shows a possibility of the research in developing modified geopolymer product that has superior durability, corrosion resistance properties and better finish along with faster curing time, which might prove to be a great alternative to the existing system for sewer and water mains or rapid pavement development.

Figure 5-4: Some Areas of Future Implementation.

Other possibilities could be developing a 3D printing device integrated with a heat source that pour the modified concrete to the particular design and cure it simultaneously (Figure 5-5). The benefits of the study along with future possibilities can be summarized through Figure 5-6.

Figure 5-5: Research Possibilities in 3D Printing.

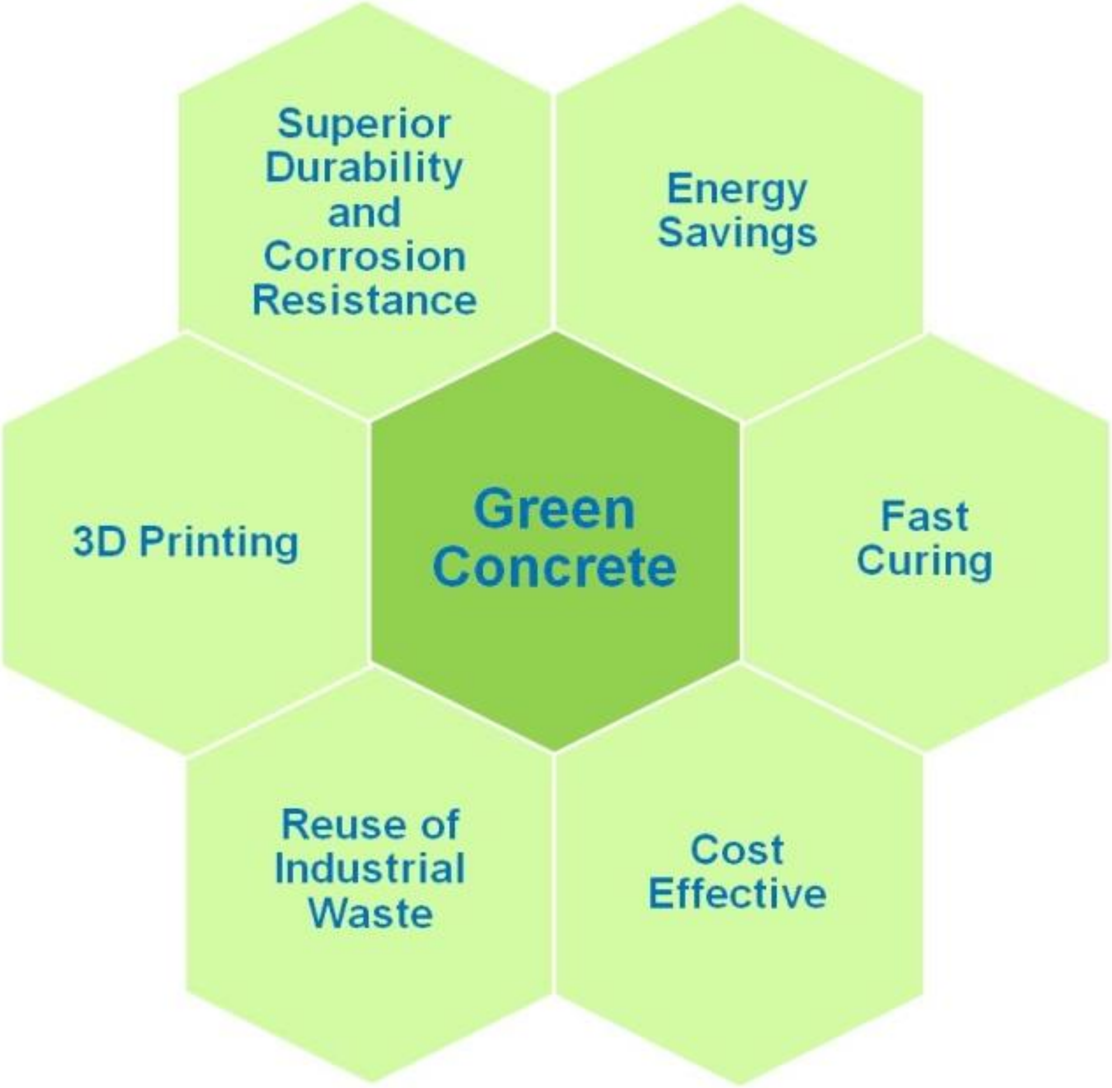


Figure 5-6: Summary of Benefits and Future Potentials.

APPENDIX A

SUMMARY OF OPTIMIZATION OF THE MIX

Table A-1: Mix Trials.

No.	AAM	AAM (aq)	MBAA	Water	APS	NaOH
1*	-	4.50	0.45	4.50	0.45	-
2	-	2.25	0.45	-	0.45	-
3	-	2.00	1.00	-	1.00	-
4	-	3.50	0.45	-	0.45	-
5	-	3.00	0.55	-	0.50	-
6	-	3.50	0.47	-	0.45	-
7	-	3.50	0.45	-	0.45	-
8*	-	4.50	0.45	-	0.45	-
9*	-	4.50	0.45	-	0.45	-
10	4.50	-	0.45	4.50	0.45	-
11	1.00	-	0.10	1.00	0.10	-
12	1.00	-	-	1.00	0.10	-
13	1.00	-	1.00	0.10	0.10	1.00
14	1.00	-	-	1.00	0.10	1.00
15	1.00	-	1.00	0.5	0.50	-
16	1.00	-	1.00	1.00	1.00	-

All units are in grams
*Unsuccessful Trials

Table A-2: Mix Trials (Continued)

No.	AAM	AAM (aq)	MBAA	Water	APS	FA
17	1.00	-	1.50	1.00	0.50	-
18	1.00	-	1.00	1.00	3.00	-
19	1.00	-	1.00	1.00	2.25	-
20	1.00	-	1.00	1.00	2.00	-
21	1.00	-	1.00	1.00	2.15	-
22	1.00	-	1.00	1.00	2.10	-
23	1.00	-	1.00	1.00	2.10	-
24	4.50	-	4.50	-	0.90	-
25	1.00	-	1.00	-	1.00	-
26	1.00	-	1.00	-	0.75	-
27	1.00	-	1.00		0.60	-
28	1.00	-	1.00	-	3.00	-
29	1.96	-	1.96	1.96	4.12	-
30*	1.00	-	1.00	-	2.10	20.00
31	1.96	-	1.96	1.60	4.12	4.50
32	1.96	-	1.96	1.50	4.00	-
33	1.96	-	1.96	1.50	4.00	5.00
34	1.96	-	1.96	1.50	3.80	-
35	1.96	-	1.96	1.50	3.50	-

All units are in grams
*Unsuccessful Trials

Table A-3: Mix Trials (Continued)

No.	AAM	MBAA	Water	APS	FA	SS	DMSO	MMA	AA	ALPS
36	0.90	0.45	0.90	0.45	-	-	-	-	-	-
37	0.90	0.45	0.45	0.45	-	1.58	-	-	-	-
38	0.40	0.4	0.20	0.82	8.00	2.80	-	-	-	-
39	1.00	1.00	1.00	2.10	-	1.00	-	-	-	-
40	0.90	0.10	0.90	0.10	8.00	2.80	-	-	-	-
41	0.98	0.98	0.98	2.06	3.70	1.30	-	-	-	-
42	1.70	0.17	1.28	0.14	-	-	-	-	0.80	-
43	-	0.10	-	0.20	-	-	1.00	1.00	-	-
44	0.50	0.05	-	0.10	22.00	7.64	0.50	-	-	-
45	-	0.10	-	1.00	-	-	1.00	-	1.00	-
46	-	0.10	-	0.50	-	-	1.00	-	1.00	-
47	-	0.10	-	0.75	-	-	1.00	-	1.00	-
48	1.00	0.10	-	0.20	9.20	3.30	1.00	-	-	-
49	1.00	0.10	-	0.20	7.50	-	1.00	-	-	-
50	-	0.20	-	-	-	-	-	1.15	-	1.00
51	-	0.50	-	-	-	-	-	1.22	-	1.00
52	-	1.00	-	-	-	-	-	1.00	-	0.50
53	-	0.70	-	-	-	-	-	1.00	-	0.50

All units are in grams
* Unsuccessful Trials

Table A-4: Mix Trials (Continued)

No	MBAA	FA	SS	NaOH	MMA	ALPS
54	0.50	-	-	-	1.00	0.50
55	0.40	-	-	-	1.00	0.50
56	0.40	-	-	-	1.00	0.40
57*	0.40	8.60	2.00	1.00	1.00	0.50

All units are in grams
*Unsuccessful Trials

APPENDIX B

COMPRESSIVE STRENGTH DATA

Table B-1: 28 Days' Compressive Strength Data for FPGPC.

Sample	Weight (lb.)	Load(lb.)	Peak Stress(psi)	Modulus of Elasticity(psi)
1	433	2,820	897	27,518
2	410	1,260	402	18,088
3	434	1,520	484	43,057
4	411	2,050	652	36,879
5	430	2,620	835	23,898
6	434	2,230	710	34,232
7	413	2,570	818	39,579
Average	**424**	**2,153**	**685**	**31,893**

Table B-2: Seven Days' Compressive Strength Data for FPGPC.

Sample	Weight(lb.)	Load(lb.)	Peak Stress(psi)	Modulus of Elasticity(psi)
1	412	3,230	1,027	72,645
2	410	3,420	1,087	69,210
3	414	3,060	973	90,050
4	415	2,110	672	39,795
5	416	2,670	851	62,755
6	415	2,400	764	59,790
7	415	2,380	756	42,745
Average	**414**	**2,753**	**876**	**62,427**

Table B-3: Three Days' Compressive Strength Data for FPGPC.

Sample	Weight(lb.)	Load (lb.)	Peak Stress(psi)	Modulus of Elasticity(psi)
1	407	2,730	869	57,865
2	411	2,770	882	63,518
3	416	2,700	859	62,420
4	413	2,830	902	77,914
5	404	2,990	953	79,163
6	400	2,880	917	64,384
7	408	3,100	988	66,042
Average	**408**	**2,857**	**910**	**67,329**

Table B-4: 28 Days' Compressive Strength Data for GPC.

Sample	Weight(lb.)	Load(lb.)	Peak Stress(psi)	Modulus of Elasticity(psi)
1	487	6,600	2,101	134,051
2	477	6,550	2,083	161,723
3	476	7,850	2,500	300,659
4	478	6,700	2,132	177,865
5	477	8,010	2,579	278,783
6	456	7,930	2,524	130,435
7	462	6,670	2,123	120,127
Average	**473**	**7,187**	**2,292**	**186,235**

Table B-5: Seven Days' Compressive Strength Data for GPC.

Sample	Weight(lb.)	Load(lb.)	Peak Stress(psi)	Modulus of Elasticity(psi)
1	431	8,660	3,381	128,729
2	424	7,430	3,843	148,282
3	419	10,030	3,716	158,972
4	413	7,610	3,764	142,789
5	422	9,420	1,205	124,708
6	430	9,980	5,150	191,137
7	422	8,820	2,241	84,021
Average	**423**	**8,850**	**3,329**	**139,805**

Table B-6: Three Days' Compressive Strength Data for GPC.

Sample	Weight(lb.)	Load(lb.)	Peak Stress(psi)	Modulus of Elasticity(psi)
1	430	8,543	2,755	118,278
2	424	7,210	2,365	139,567
3	419	9,980	3,192	144,812
4	413	7,325	2,424	136,611
5	422	8,975	2,998	114,254
6	430	9,858	3,176	149,752
7	422	8,627	2,807	110,560
Average	**423**	**8,645**	**2,817**	**130,548**

Table B-7: 28 Days' Compressive Strength Data for OPC.

Sample	Weight(lb.)	Load(lb.)	Peak Stress(psi)	Modulus of Elasticity(psi)
1	431	8,970	2,856	285,259
2	439	9,240	2,942	224,654
3	433	9,170	2,920	354,754
4	429	9,330	2,969	203,145
5	440	8,600	2,730	642,655
6	437	12,080	3,846	816,781
7	437	11,890	3,786	383,183
Average	**435**	**9,897**	**3,150**	**415,776**

Table B-8: Seven Days' Compressive Strength Data for OPC.

Sample	Weight(lb.)	Load(lb.)	Peak Stress(psi)	Modulus of Elasticity(psi)
1	431	8,410	2,678	208,100
2	439	9,790	3,115	222,462
3	432	6,150	1,958	174,284
4	439	6,420	2,043	181,227
5	440	11,110	3,535	279,015
6	444	8,910	2,836	195,106
7	440	6,490	2,067	276,029
Average	**438**	**8,183**	**2,605**	**219,460**

Table B-9: Three Days' Compressive Strength Data for OPC.

Sample	Weight(lb.)	Load(lb.)	Peak Stress(psi)	Modulus of Elasticity(psi)
1	444	7,900	2,514	330,336
2	442	5,800	1,872	460,130
3	441	9,180	2,923	392,188
4	439	6,080	1,936	308,378
5	442	3,320	1,056	108,653
6	443	2,540	810	491,406
7	441	3,720	1,185	168,102
Average	**442**	**5,506**	**1,757**	**322,742**

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