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On the Development of 8,000 psi Geopolymer Mortar for Practical Use in Field Applications

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On the Development of 8,000 psi Geopolymer Mortar for Practical
Use in Field Applications

A thesis submitted in partial fulfillment
of the requirements for the degree of
Master of Science in Civil Engineering

by

Remington Reed
University of Arkansas
Bachelor of Science in Civil Engineering, 2016

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Abstract

Although there are several perceived benefits from the use of geopolymer binder as an alternative to ordinary portland cement in concrete materials, it is not widely used in field applications. Geopolymer materials have no set guidelines for mixture design and often require heat curing to facilitate strength gain. Additionally, many geopolymers are created using carefully controlled and high quality materials and activator chemicals. These factors greatly affect the practicality of the material. The goal of this research was to create a geopolymer material capable of achieving high compressive strengths at 28 days without heat curing, and using only readily available and cost effective materials. The material developed in this study is expected to see practical use in field applications. As a result of this study, a non-heat cured geopolymer mortar achieving a compressive strength of over 8,000 psi at 28 days was developed using local sand, Class C fly ash, and readily available commercial grade chemicals. Modulus of elasticity was also examined during the course of research. Through the process of refining a mixture design, several design factors affecting compressive strength were considered. The results of the several batches made in the refinement process could be used in the development of future materials with a target compressive strength and stiffness.

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Thank you to my advisor, Dr. Hale, for encouraging me to pursue my graduate education and trusting me with this project. This research, along with your support and insight has given me the chance to grow as a student, travel the world, and meet friends and colleagues I will keep in touch with for a lifetime. Thank you to my committee for supporting my research both in the lab and the classroom.

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1 Introduction

Geopolymer, or inorganic aluminosilicate polymer, is an alternative to ordinary portland cement (OPC) as a binder in concrete materials. Concrete and mortars made with geopolymer binder exhibit similar strengths and behavior to materials made with traditional ordinary portland cement [1]. Geopolymer binder is a long chain amorphous crystalline structure comprised of silicates and aluminates. This structure is created by combining a pozzolanic material with a chemical activator solution to facilitate polymerization and in turn, strength gain [2].

Geopolymer binder can be used as an alternative to fully or partially replace OPC in concrete material mixture designs [3]. These materials are often considered environmentally friendly alternatives to OPC binders, and exhibit desirable characteristics for several applications. Despite functioning in a manner similar to OPC materials, geopolymers are not widely used in industry for a variety of reasons that will be discussed in following sections.

2 Background

2.1 Geopolymer Makeup

Geopolymer binder is composed of alumina and silicate in a long chain amorphous crystalline structure. This “chain” is comprised of a series of monomers created by the alignment of alumina and silicates from some source material rich in both [2]. In most cases, this source material is a pozzolan possessing properties that will be discussed in following sections. A chemical activator solution must be added to the source material. The chemical activator frees the alumina and silicate present in the pozzolan allowing them to restructure and polymerize with the aid of additional silicate substrate also found in the solution. As the crystalline structure is formed, water from the activator solution exits the matrix [2, 3, 4]. During the strength gain process, most producers of geopolymer materials use heat or steam curing in a range of 60 to 90

Celsius [6]. The heat expedites the reaction and helps drive water from the matrix therefore facilitating quicker strength gain [2, 6]. This is different from traditional OPC concrete in that water in solution is not needed for hydration and the production of typical hydration products is not responsible for the strength of the geopolymer binders [5].

Pozzolanic materials are composed of siliceous and aluminous compounds [2, 5]. They are both found naturally and created as a byproduct of several manufacturing processes. Many different pozzolans contain the necessary substrates to form the alumino-silicate polymer, but require the addition of a chemical activator solution. In most cases, fly ash is used as the reactive pozzolan for geopolymer production. However, rice husk ash, slag cement, and natural pozzolans are also acceptable materials [1]. Fly ash is the most commonly used aluminate/silicate source due to its availability and high alumina and silica content [1, 2]. Class F, or low calcium fly ash (FFA), is the more commonly used for geopolymer production than Class C, or high calcium (CFA), fly ash. FFA is considered a better material for geopolymer production because of its finer particle size and low calcium content. It is accepted that finer fly ash produces stronger final materials, and calcium is not necessary for strength gain in geopolymers [6]. There is less research into the use of CFA, and in some cases opinions differ on the effect of the calcium content [8]. Some sources maintain that the calcium interferes with the polymerization process, resulting in lower strengths [2]. Others maintain that it is possible that the calcium in CFA could add an additional source of strength gain in the form of typical hydration. The calcium leads to the formation of typical hydration products, and in turn creates a water deficit that enhances the polymerization process [7]. This standard hydration of calcium in CFA does bring with it some concerns of flash setting [5]. In most cases it is agreed upon that the fly ash quality/composition

is one of the most important factors affecting a geopolymer material's fresh and hardened properties [1, 4, 6, 8].

Most chemical activator solutions are a two part system consisting of a strong base and an additional silicate source [2, 3, 5]. Traditional geopolymer production uses sodium hydroxide or potassium hydroxide as a strong base. This strong base “activates” the alumina and silicate present in the fly ash allowing them to enter solution and then polymerize. The sodium silicate portion of the solution enhances the reaction between the solution and pozzolan. Sodium hydroxide (NaOH) is the most commonly used chemical for activators because it exhibits a high rate of dissolution of the alumina and silicate compounds necessary for strength gain [2]. Most geopolymer research and production ensures these chemicals are fully dissolved in water at the time of addition to the other materials to ensure even distribution.

2.2 Applications and Use

In practice, geopolymer materials are currently not seeing widespread use despite the perceived benefits gained by using this binder type [5]. Due to the use of pozzolans, which are primarily considered waste products, geopolymer materials are often considered a “green” alternative to OPC [5, 9]. Portland cement production is responsible for 85 percent of the energy used in concrete production and accounts for up to 7 percent of the total anthropogenic carbon dioxide emission each year [5, 7, 9, 10]. Fly ash, rice husk ash and slag cement are all byproducts of other industries and therefore are responsible for no carbon dioxide production. Geopolymer concretes can achieve compressive strengths similar to traditional OPC concrete: anywhere from 30 to 80 MPa, (4,400 psi to 11,600 psi) [7]. In addition to the environmental benefit, research has shown that geopolymer materials are more resistant to acid attack and exhibit more durability than traditional opc materials [3, 5].

Although these materials have been researched and seen isolated use for the last 30 years, they are not part of common practice [5]. This could be a result of many things. Because there are many combinations of pozzolans and chemical binders, there is not a set mixture design process for these materials [3, 7]. In addition to this, the standard practice of heat curing and using Class F fly ash make it difficult to produce and implement these materials in many places. This, in many cases, has relegated geopolymers to use in precast facilities [2, 5]. By researching the effect of each major part of the mixture design, the use of local materials, and simple mixing and curing procedures, it is possible to develop ways to increase the use of geopolymer materials as alternatives to traditional OPC concretes and mortars.

3 Research Plan

3.1 Research Goals

The goal of this research was to develop a geopolymer material for industrial use containing only local/cost effective materials. This material is expected to achieve compressive strengths of 8,000 psi and be compatible with current industry practices for work time and functionality, including curing at ambient temperatures. As a result of developing this material, the research also lends itself to use as a guide for future geopolymer mixture design procedures. By demonstrating the effects of different variables on the functional properties of standard geopolymer materials, a simple mixture design process for geopolymer materials becomes possible.

For the purposes of this research, mortar was chosen as the primary testing material. This decision was made to reduce the variability that results from the addition of coarse aggregate. Additionally, the batch size for mortar is much smaller and allows for more effective use of

materials, curing space and laboratory time. The testing was conducted in two phases: strength testing, and modulus of elasticity testing.

3.2 Methods and Materials

Strength Phase

Compressive strength was considered the primary factor in comparing the mixture designs. All mortar mixture designs were broken into two parts, a dry and wet mix. The dry mix consisted of Class C fly ash from the White Bluff Power Plant in Redfield, AR and oven dried Arkansas River sand passing the number 8 sieve. The wet mix contained the chemical activator solutions. The activator chemicals were treated as part of the wet mix because of the need for them to be in solution in order to ensure complete dissolution and distribution within the final material. For strength testing, NaOH pellets were used for the alkaline portion of the solution, and sodium metasilicate pentahydrate was chosen as the silicate addition. These materials are produced in bulk quantities and are readily available for industrial applications. Four major variable parameters were considered in the strength testing phase: water to fly ash ratio, sand to fly ash ratio, activator concentration, and NaOH to silicate ratio.

Water to fly ash ratio was treated similarly to the water to cementitious material ratio that is typically used in OPC mixture designs. This parameter varied from 0.25 to 0.4 by mass. The purpose of the water in the polymerization process is to serve as the solvent for the chemical activator solution and to adequately facilitate the dispersion of the dissolved chemical throughout the mix.

Sand to fly ash ratio was described using percentage of the total dry materials (this excludes chemicals). Due to the similar costs of these two materials, very high fly ash contents were considered. Both Class C fly ash and sand cost approximately 25 to 50 percent less than

portland cement. The main portions of the dry mix were 25, 30, 40, and 50 percent by volume fly ash.

Activator concentration referred to the molarity of only the NaOH portion of the activator solution. Once the water content was established for a specific mixture design, the total weight of water was then split evenly to create the two separate solutions; NaOH and silicate. The molarity for the NaOH solution ranged from 8 molar to 16 molar.

Activator ratio was based on the mass of the solutions. This is a mass based ratio of NaOH solution to silicate solution added to the mixture design. The baseline ratio of NaOH to silicate was one to one, meaning that the silicate solution was simply the addition of an equal mass of silicate to an equal amount of water as the alkaline solution. The decision to use a mass based ratio for the two activator chemicals was due to the large difference in molecular weight. Equal molar concentrations of the chemicals would have led to gross oversaturation of the sodium metasilicate pentahydrate. The activator ratios, NaOH to silicate, investigated were 1:1, 1.25:1, 1.50:1, and 1:75:1. When varying the activator ratio, the mass ratio of the water was first changed in order to determine the amount of NaOH to add to reach the desired alkalinity. Then a mass ratio of silicate was added to the remaining water. This was done to ensure both the volume of solution and amount of chemical used were at the desired activator ratio.

During the strength phase, each of the four parameters was modified while the others remained constant. Several mixture designs were batched and tested with the goal of optimizing strength. The first mixture designs were used to guide refinement of subsequent mixture designs in order to yield high compressive strengths. The mixtures designs used for this process are displayed in Table 1.

Table 1. Basic parameters of strength phase mixture designs

Design Number	Sand/FA Ratio	Activator Concentration	Activator Ratio	Water Content
1	2.75	8	1.00	0.40
2	2.75	10	1.00	0.40
3	2.75	12	1.00	0.40
4	2.75	8	1.00	0.40
5	2.75	10	1.00	0.40
6	2.75	12	1.00	0.40
7	2.75	14	1.00	0.40
8	2.75	16	1.00	0.40
9	2.75	10	1.00	0.25
10	2.75	10	1.00	0.30
11	2.75	10	1.00	0.35
12	2.75	10	1.00	0.26
13	2.75	10	1.00	0.27
14	2.75	10	1.00	0.34
15	2.75	10	1.00	0.36
16	2.75	11	1.00	0.35
17	2.75	12	1.00	0.35
18	2.75	13	1.00	0.35
19	1.00	10	1.00	0.30
20	1.50	10	1.00	0.30
21	2.33	10	1.00	0.30
22	1.00	10	1.00	0.25
23	1.50	10	1.00	0.25
24	1.50	10	1.50	0.30
25	1.50	10	1.50	0.30
26	1.50	10	1.00	0.27
27	1.50	10	1.50	0.25
28	1.50	10	1.25	0.30
29	1.50	10	1.75	0.30
30	2.33	10	1.00	0.27
31	1.50	10	1.00	0.25
32	1.50	10	1.00	0.25

The mortar was mixed using a tabletop Blakeslee paddle mixer as shown in Figure 1. The dry materials were mixed for 5 minutes. During this time the activator chemicals were fully

dissolved into the mixture water, creating the activator solution. At the end of five minutes, the activator solution was added to the mixer. After three minutes, the mixer was turned off and the mortar was stirred using a large spoon to ensure that no dry pockets had occurred within the mixed materials. The mortar was then mixed for another two minutes. For the strength testing phase, 2 inch cubes were cast. Nine cubes were cast per mixture design. The cubes were then placed in an environmental chamber at 70 degrees Fahrenheit and left uncovered for 24 hours.



Figure 1. Tabletop mixer

After 24 hours, the cubes were demolded, and the compressive strength of 3 cubes was tested at 1, 7, and 28 days, respectively, in accordance with ASTM C109 as shown in Figure 2 [11]. Once specimens were demolded they were returned to the environmental chamber and cured at a constant 70 degrees Fahrenheit and 50 percent relative humidity. The results were used as they were recorded in order to develop future mixture designs. This process was repeated until

each of the four factors (water to fly ash ratio, sand to fly ash ratio, activator concentration, and NaOH to silicate ratio) had been examined and refined. This process continued and resulted in several mixes having all four of the major variables adjusted to maximize compressive strength gain.



Figure 2. Compressive strength testing

As a result of the ongoing refinement process, a mixture design was chosen for further testing. The criterion for this mixture design was primarily compressive strength, but also included consideration of raw material use and ease of handling. The purpose of this refinement was to create a suitable material to use as a high performance mortar developed for industrial practice. The selected mixture displayed high compressive strength foremost, but was also easy to replicate, made efficient use of materials. Additionally, the material was easy to place into molds and retained workability for a sufficient time to cast laboratory specimens. This material was further tested with changes in mixing procedure.

Modulus of Elasticity Phase

Once the selected mortar mixture design was chosen, research began on the modulus of elasticity (MOE) of the material. MOE testing was performed using cylinders due to the limitations of laboratory equipment. Available equipment for testing MOE, as seen in Figure 3, was not compatible with cube specimens. This presented some difficulty because of the limited capacity of the tabletop mixer as the batch size necessary to cast cylinders rather than cubes exceeded the capacity of the mixer.



Figure 3. Modulus of elasticity testing collar

In order to batch enough mortar to fill cylinder molds, it was necessary to change to a larger mixer. Batching was done using a small rotating drum mixer capable of mixing 1 cubic foot of material. The selected mixture design proportions were used to establish batch weights for approximately .7 cubic feet of material. The batch size was chosen to provide enough material to cast 8 4x8 inch cylinders without exceeding mixer capacity. Of the cylinders, 3 were

used to test compressive strength at each of 7 and 28 days. The purpose of strength testing was to ensure that the mortar was gaining strength in a similar manner to the batches mixed using the tabletop mixer. The additional 2 cylinders were used for the MOE testing.

Due to the change in mixer type, it was apparent that the mixing procedures used with the tabletop mixer were not adequate for fully mixing a material in a larger mix that behaved in the same way as was seen when using the tabletop mixer. Compressive strengths measured using ASTM C39 procedures showed reductions by over fifty percent when comparing mixtures cast using the tabletop mixer versus those cast using the rotating drum mixer [12]. The material did not behave in the same manner as the small batches. Dry material pockets were found in the broken cylinders. Because of this change in mixers, several mixing procedures were investigated and used in order to attain samples with similar performance to the original batches of the selected mortar with none yielding results analogous to that of the tabletop batches.

Following several attempts to use a rotating drum mixer, it was decided that another mixer may be more effective. In order to move forward with MOE testing, a double shear drum mixer was acquired in order to replicate similar mixing properties to the tabletop mixer. This mixer had two barrels with a spinning paddle within each that folded the material into itself and introduced higher shear and energy during mixing than with a typical rotating drum mixer. In addition to the change of mixer, the cylinder size was reduced to be better suited for testing mortar. Rather than using 4x8 inch cylinders, 3x6 inch cylinders were cast. The batch size was not changed. To accommodate the new specimen size, smaller MOE testing equipment was acquired. Nine cylinders were cast per batch, as well as several 2 inch cubes. The mixing procedures using the double shear drum mixer were kept in accordance with the provider's instructions. Half of the mixing water was added to the mixer, followed by all of the dry

material, the dry chemicals, and then the remaining mix water. To further increase the chance of proper chemical dissolution, granular NaOH was used instead of the pelletized form used in all previous mixtures. The sand was also changed to passing the number 10 sieve per the request of the mixer's provider. All testing using the double shear drum mixer was done on selected mixture designs from the initial strength phase.

In order to better understand the relationship between the initial mix design and MOE, several of the mixtures from the original strength phase were chosen for rebatching. These mixture designs were selected based on 28 day compressive strength at, or exceeding, 7,000 psi. During this phase, all of the mixing steps from the initial strength phase were repeated using the tabletop mixer. The mixes were then used to cast 6, 2 inch cubes, and 3, 3x6 inch cylinders. Three cubes were tested at 7 and 28 days respectively to ensure consistent compressive strength with the initial phase. Using these compressive strength results, a MOE test was performed on two of the cylinders with the third being used in the event of aberrant results such as a poorly cast or consolidated cylinder. Procedures consistent with ASTM C469 were used when testing modulus of elasticity [13]. It is important to note that the calculations for MOE were done assuming the compressive strength of the cylinders was identical to the cubes, and assuming a 15% strength reduction to account for the difference in specimen type. Assuming the cubes and cylinders were the exact same strength led to a more conservative calculation of MOE. At this point in the material testing, granulated NaOH was used instead of pellets. The NaOH was of the same grade as the pellets from the initial strength phase, and was mixed in accordance with the initial strength testing phase procedures.

4 Results and Discussion

4.1 Water content

During the strength testing phase of the research project, the optimal water content was determined to be between 25 and 30 percent of the fly ash content. These mixtures exhibited higher 28 day compressive strengths as well as yielded final material that was easy to form and cure. The higher compressive strengths of this range of water content are displayed in Figure 4. During this water content testing, all other mixture design variables were held constant. Shown in Figure 4 is the general decrease in compressive strength at 28 days as the water content increases.

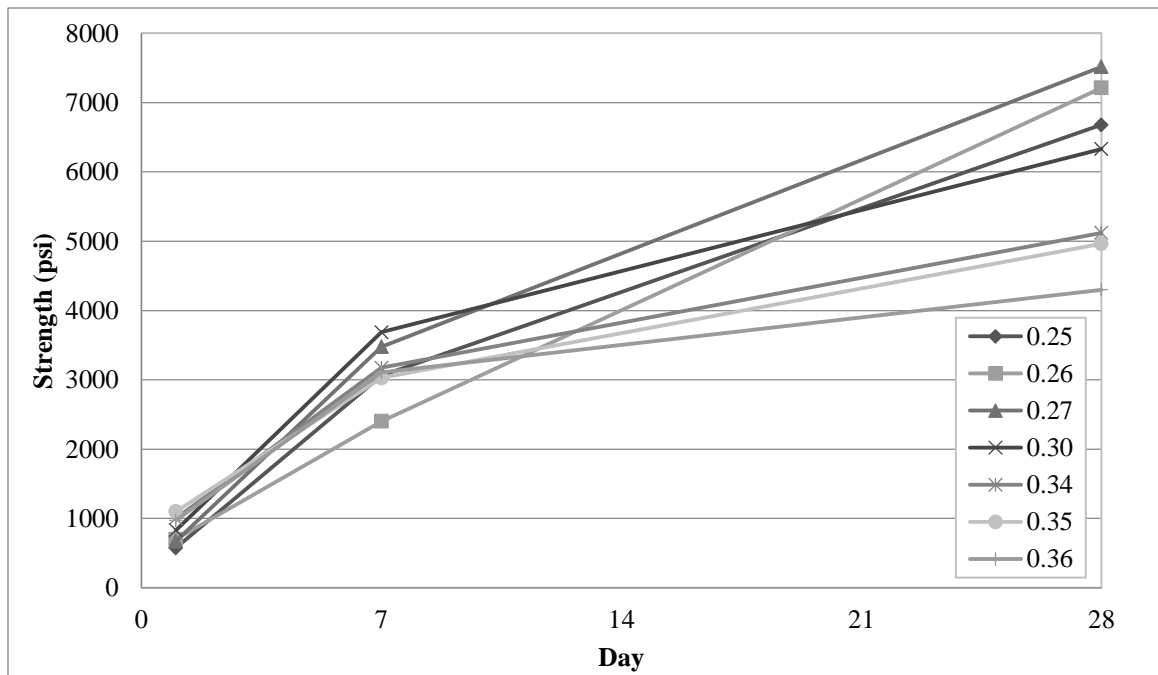


Figure 4. Compressive strength at 1, 7, and 28 Days as a result of water to binder ratio

Geopolymer binders do not hydrate in the same way that OPC binders do. The water present in the mixture serves only to facilitate the equal distribution of the activator chemicals: as

water leaves the matrix, the material gains strength. This varies from the behavior of traditional OPC materials due to the lack of a hydration process.

The greater the water content of the mixture design, the higher the moisture gradient between the specimen and the curing environment. The early strength gain is very comparable for all water contents as shown in Figure 5. The effect of water content is not fully apparent until 28 days of age.

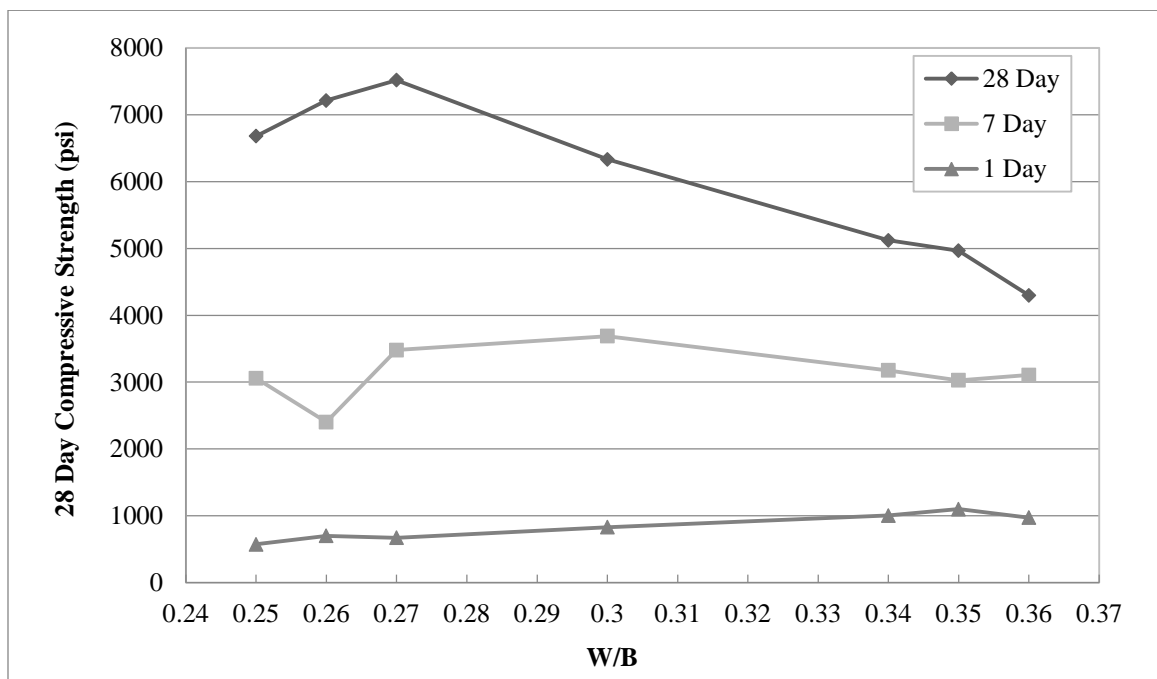


Figure 5. Compressive strength at 1, 7, and 28 Days as a result of water to binder ratio

Choosing to base subsequent mixture designs off of 7 day results proved an unreliable benchmark for performance as the 1 and 7 day results were not indicative of the 28 day strength, as is shown in Figure 5. By exiting the specimen more rapidly, the water may take with it some portion of the chemicals necessary for the polymerization process, thereby reducing the later age strength. This was further shown in a higher amount of efflorescence exhibited by the higher water to binder ratio mixture designs, as shown is Figure 6.

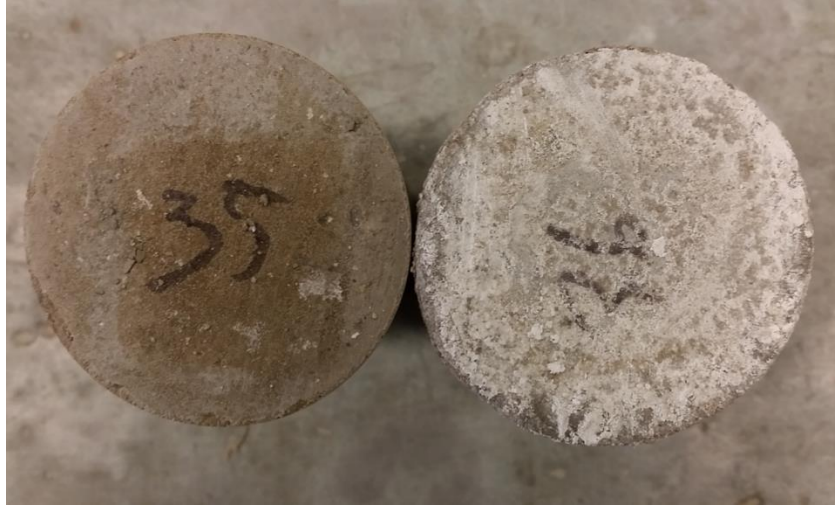


Figure 6. Chemical efflorescence

The optimal range in this study provided adequate water to completely dissolve and disperse the activator chemicals without increasing the water content to a point where it stifled late age strength gain. It is important to note that the complete dissolution of chemicals in the mixing water prior to addition to the dry materials allowed the water content to be lowered. When testing the same mixture design but treating the chemicals as a portion of a dry mixture, the samples exhibited visual pockets of undissolved chemicals in specimens as shown in Figure 7.



Figure 7. Undissolved chemicals in the mortar

Using data from the other parameter testing, it became evident the water content had a greater effect on compressive strength than fly ash content. When all other factors were constant and only water and fly ash content were varied, the 28 day compressive strengths were not equally changed by each factor as displayed in Figure 8.

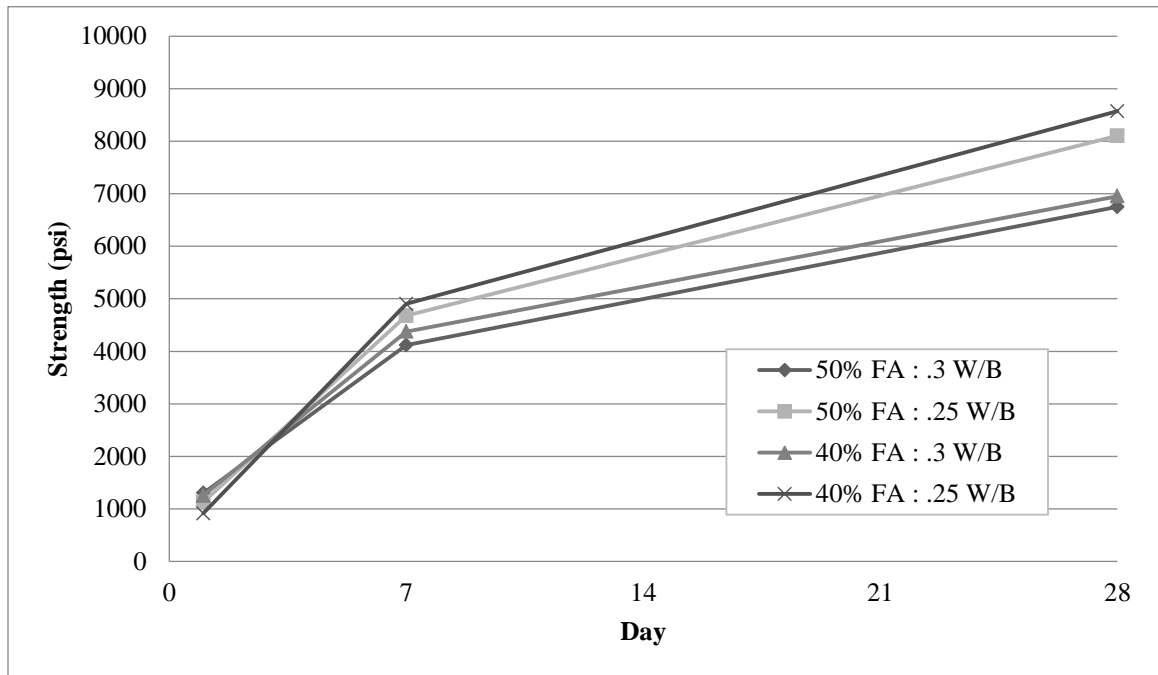


Figure 8. The effect of variable water content compared to the variable fly ash content

Figure 8 shows that when water content was held constant but fly ash content was changed, the 28 day strength results varied by approximately 500 psi or less. When fly ash content was kept constant and water content was varied, the 28 day compressive strength varied by up to 1500 psi. While both factors affect the strength of the material, the change in water content had the greater effect on the compressive strength of the material.

4.2 Activator Concentration

Molarity of the NaOH portion of the activator solution was varied from 8 to 16. Because the NaOH and silicate solutions were considered two separate activators, the NaOH portion of the activator ratio was only made using half of the mixture water. This means that the actual

molarity in total solution would be half that of the specified NaOH portion. Increasing NaOH concentration increased compressive strength up to a limit (10 to 11 molar) and then it decreased compressive strength as shown in Figure 9.

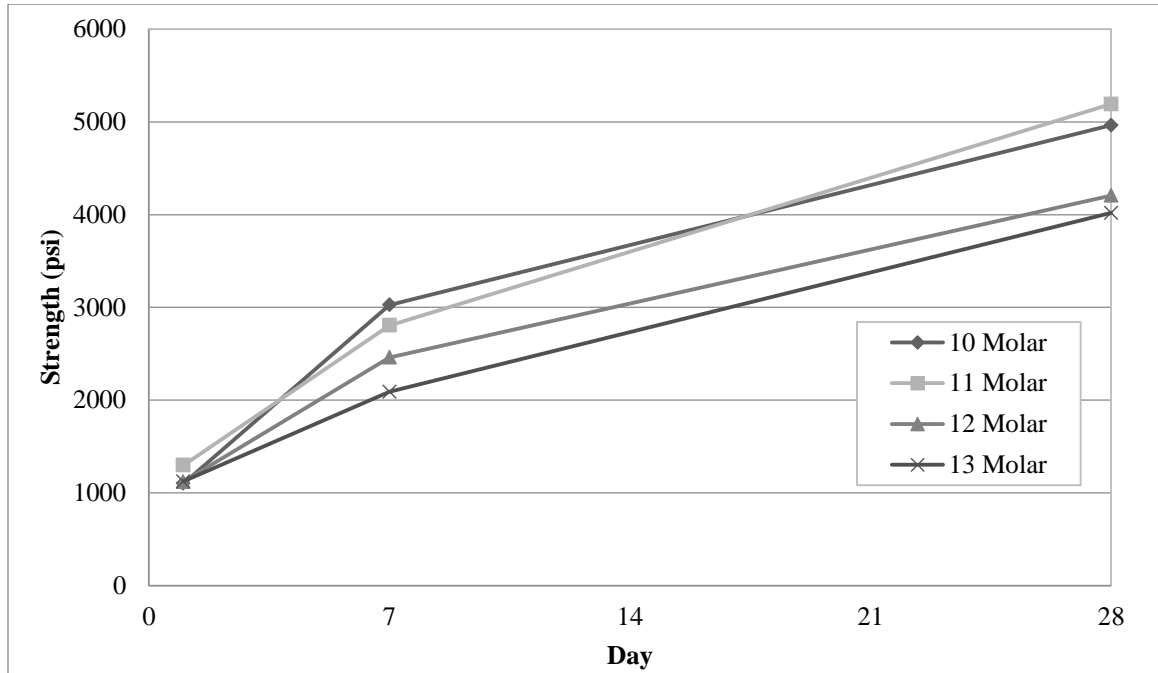


Figure 9. Effect of NaOH concentration on compressive strength

Keeping all other variables constant, testing showed that an activator molarity of 11 yielded the highest compressive strength specimens. Although the highest performing concentration was 11 molar, 10 molar solutions were used for the remainder of testing for the purposes of cost and safety. This slightly weaker solution performed comparably to the 11 molar solutions in its effects on compressive strength, but was deemed more cost effective and safer for lab use due to a slight reduction in caustic concentration.

Mixture designs with NaOH concentrations above 13 molar did not set up within 7 days and posed increased danger to persons handling the activator chemicals and even the final material itself. Due to the exothermic nature of the reaction between NaOH and water, and the caustic nature of the activator solution, great care was used when mixing. Because of these

concerns, alternative means of mixing were used for all batches made using mixers larger than the tabletop version. Once in the mortar, the activator solution is dilute enough that only minor skin irritation occurs with contact. Additionally, the heat of mixing is dispersed throughout the material eliminating the risk of burns.

The caustic properties of the final mortar are still a concern and could be improved by the use of weaker activator solutions or alternative chemical choices. For the purposes of this research it was assumed that great care would be taken when handling the material. For the sake of avoiding heat curing and simple mixing procedures, this concern was not addressed immediately in the strength testing process.

4.3 Fly Ash Content

When all other variables were kept constant, compressive strength results showed that a mix with 40% fly ash by weight performed the best for compressive strength as displayed in Figure 10. A 1.5:1 sand to fly ash ratio provided enough reactive silicate and aluminates to produce strong bond formations. The higher performance of this ratio could also be caused by particle size distribution. Without performing a chemical analysis of the mortar batches it is unknown how much if any of the fly ash added acted as inert filler.

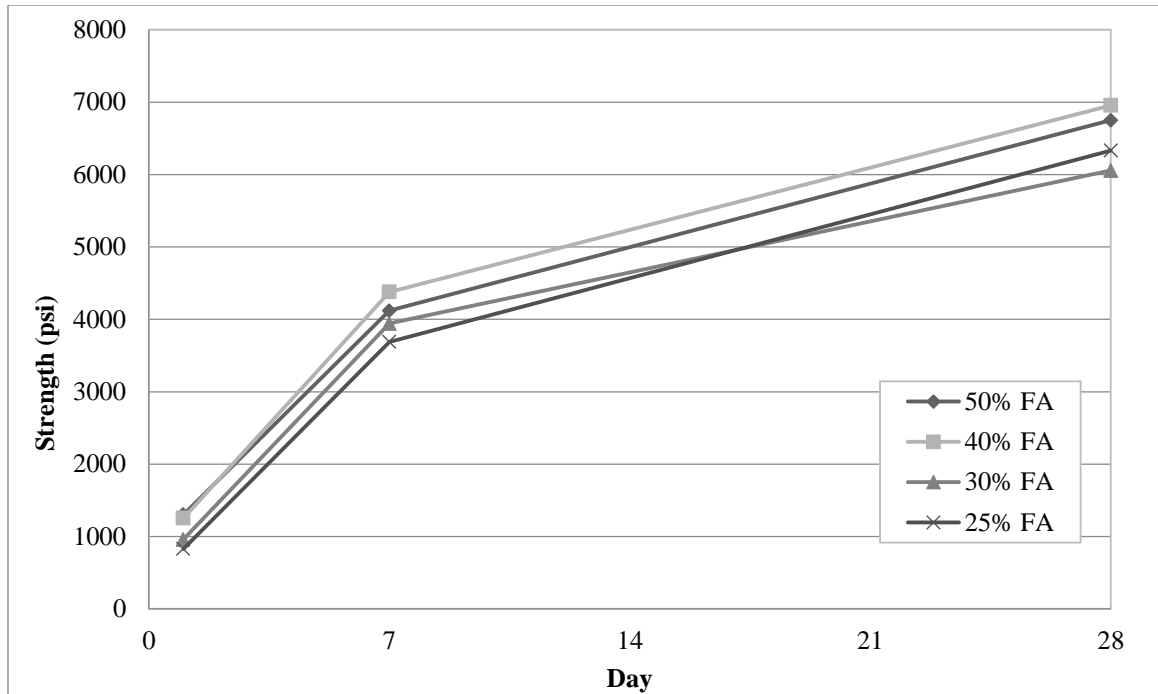


Figure 10. Effect of fly ash content on compressive strength

During the course of the research, all the fly ash used was from the same source but from separate batches. In repeat batches and follow up testing, this material performed the same despite being from multiple barrels. This suggests that general performance is not affected by small changes in the amounts of reactive silicate and aluminate in the fly ash. Further research into the behavior of fly ash as a filler material could help further refine this ratio of solid materials.

4.4 Activator Ratio

Activator ratio was altered to observe the effects of using an increased concentration of sodium silicate pentahydrate. There was a peak in compressive strength using 1:1.5 NaOH to silicate ratio as displayed in Figure 11. This specific mix, with all other variables held constant, yielded highest compressive strength. Increasing to a ratio of 1:1.75 resulted in decreased compressive strength. Reducing the ratio to 1:1.25 also decreased compressive strength.

However, these changes affected the fresh material properties of the mix. Rather than having low

flow/workable mortars, the mixtures with additional sodium silicate pentahydrate were almost liquid. When casting cubes with increased silicate, tamping was unnecessary and the mix could easily be poured into molds. This also delayed the setting time. Large amounts of efflorescence appeared on the outside of these samples. Because of this change in properties it was considered safer, more economical, and more practical to continue using a 1:1 ratio.

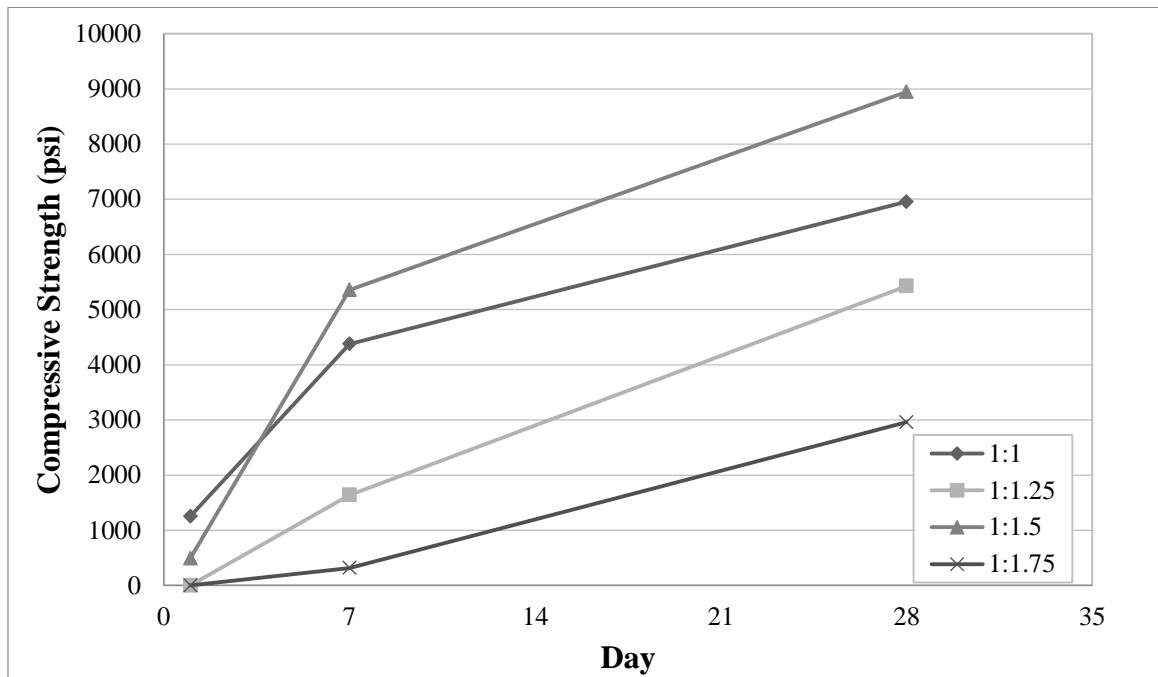


Figure 11. Effect of NaOH to sodium silicate on compressive strength

For the purposes of this research, the ratio was quantified by mass. When adjusting the ratio, the water was split in accordance with the ratio. For example, the 1:1.5 ratio, 40% of the water was considered part of the NaOH solution. Using that adjusted amount of water, the NaOH addition was the mass necessary to reach 10 molar. Then silicate mass addition was calculated by multiplying the mass of NaOH used by 1.5.

Despite the increased compressive strength of the higher ratio mixtures designs, they did not align with the goal of practical use for the study's final mortar design. The high flow resulted in materials that were difficult to work with and place. Additionally, the delayed setting time was

considered not ideal for field applications. Attempts were made to mitigate workability issues by lowering the water content. These mixes were slightly more practical due to lower flow, but did not result in higher compressive strength and therefore only furthered the result that a 1:1 mass ratio was best suited for continuing refinement.

4.5 Large Batch Size

Several attempts were made to increase the batch size and use larger mixers. Larger batches allowed for more specimens and to verify that the results were applicable for larger scale production. None of the mixes batched using a rotating drum mixer were within a satisfactory strength range to be considered analogous to the previous cube tests. In order to mitigate the danger of handling large volumes of a highly caustic solution, the first large batches were carried out by adding dry chemicals to the other dry materials. Though NaOH is highly hydrophilic, the size of the pellets and lack of mixing energy provided by the drum mixer lead to insufficient dissolution of the NaOH. When strength tests were performed, clusters and singular undissolved NaOH pellets were found in nearly all broken samples. This lead to differences of up to 1000 psi in compressive strengths between samples from the same batch. In order to mitigate this issue, some batches were attempted using the same mixing procedure as with the tabletop mixer, using fully dissolved solutions rather than adding materials. Regardless of the full dissolution, these mixes were still notably weaker, up to 3000 psi lower, than the small batches.

In order to get complete mixing, a double shear drum mixer was used. This involved scaling up the batch size in order to engage both drums of the mixer. Once batch size and procedure was determined, the strength results were comparable to those of the strength testing phase cubes. This was actively compared during testing by verifying strengths with cubes cast and tested at the same time as the cylinders.

4.6 Modulus of Elasticity

Modulus of elasticity of the materials showed no defined relationship with compressive strength. Figure 12 displays the relationship between MOE and 28 day compressive strength as it relates to the standard ACI equation for concrete: $E = 57,000 \sqrt{f'c}$.

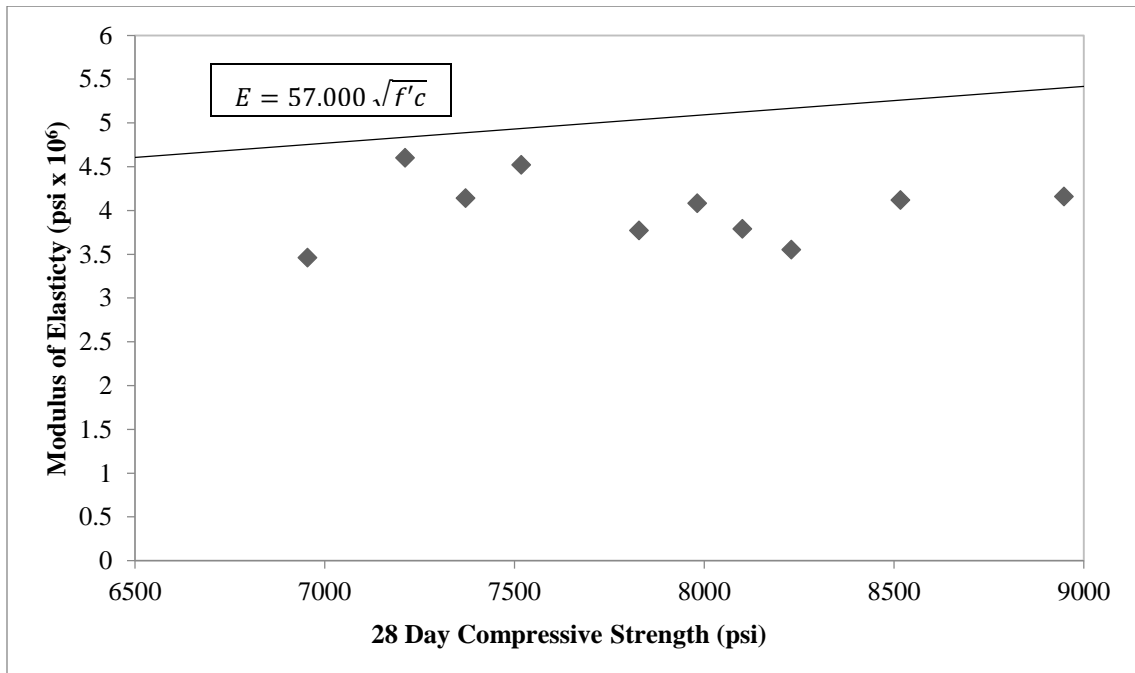


Figure 12. MOE of lab tested specimens compared to expected values

This equation was not applicable to the geopolymer mortar because of the difference in binder type and the lack of coarse aggregate. Figure 12 shows that in all tested specimens the ACI standard equation over predicts the MOE and that compressive strength of the geopolymer mortar is not the biggest indicator of MOE. The mixes with a higher MOE were those with a higher sand to fly ash ratio. The two mixes with the highest MOE, mixes 12 and 13 as seen in Table 1, were both made using a 2.75:1 sand to fly ash ratio. This is because the fine aggregate is coarser and stronger than the fly ash that is acting as filler material. Further testing to see exactly how each factor affects the stiffness of the mortar is necessary.

5 Conclusions

Although heat curing is often used to facilitate strength gain in materials made with geopolymer binders, it is not necessary for production. Geopolymer formation and strength gain is possible and replicable in ambient conditions. The goal of this research was to develop a geopolymer mortar using readily available/local materials that would achieve compressive strengths of at least 8,000 psi at 28 days without the aid of heat curing. Given a proper mixture design, it is possible for non-heat cured geopolymer mortars to achieve compressive strengths above 8,000 psi at 28 days. For the purpose of this study a .25 to .27 water content, 10 molar activator concentration, 1.5:1 sand to fly ash ratio, and 1:1 activator ratio were found to be ideal for the production of non-heat cured 8,000 psi geopolymer mortar. The following conclusions were also reached during the course of the project:

- Several mixture design factors must be considered when design and optimizing geopolymer materials mixtures portions: water to fly ash ratio, sand to fly ash ratio, activator concentration, and NaOH to silicate ratio.
- Early strength gain is similar for most mixture designs, and differences in 28 day strength are not apparent until after 7 days when heat curing is not involved.
- Small changes in water or chemical ratios and quantities affect both fresh and hardened properties of the material including changes in compressive strength and flow.
- Optimizing water content influences compressive strength more than fly ash content.
- Increasing chemical activator concentrations does not continue show increases in strength enough to warrant their use.

- Sand and fly ash contents have greater effects on stiffness than compressive strength.
- Raw material quality and consistency concerns can be overcome with careful mixture proportioning.
- Class C fly ash is suitable for geopolymer production as long as water content, fly ash content, and chemical activator solutions are carefully controlled.
- Geopolymer materials may need special mixing procedures or equipment to ensure consistent large scale production.
- These materials exhibit stiffness comparable to materials made with OPC binder, with the modulus of elasticity being slightly lower.

The design of these materials must be done by refining the mixture portions one by one to optimize performance. Water content has the greatest effect on binder strength. This is due to the reaction that occurs when the alumina silicate structure is formed, and because the water content determines the amount of activator chemical added to the mix. While the use of high grade activator solutions and Class F fly ash are commonplace, they do not prohibit the use of more cost effective or locally available materials. Some portion of the pozzolanic material used will act as inert filler and therefore affects the stiffness of the materials. Local and readily available materials along with careful mixture portioning allow for the development of non-heat cured geopolymer materials for industry use.

6 Additional/Future Research

Future research can further lead to the development of standard mixture design procedures for materials made with geopolymer binder. Because the development of this

material was directed toward high performance in field use, testing with additional materials and modifications was conducted. This was done periodically throughout the modulus phase in both the large and small mixes in order to observe how material additions affected the material. The two most notable changes were the addition of fiberglass fibers and OPC. These materials were added to observe their effect on compressive strength, specifically early age, and modulus of elasticity. In order to ensure consistent addition, they were added based on their percentage of total batch mass with proportions never exceeding 3 percent. Using previous batches as guides, these materials were incorporated to be optimized for performance. The fibers and OPC were considered part of the dry mix and were treated as such while keeping the mixing process identical to the standard batches being made at the time.

7 References

- [1] Chindaprasirt, P., Chareerat, T., Hatanaka, S., Cao, T. "High-strength geopolymer using fine high-calcium fly ash," ASCE Journal of Materials in Civil Engineering, Vol. 23, No. 3, March 2011, 264-270.
- [2] Hardjito, Djwantoro. 2005. Studies of fly ash-based geopolymer concrete. Ph. D. Curtin University of Technology, Dept. Of Civil Engineering
- [3] Davidovits, J. (1999) Geopolymer Chemistry and Applications, 2nd Ed., Geopolymer Institute, Saint-quentin, France.
- [4] Xu, H., Van Deventer, J.S.J., "The Geopolymerization of Alumino-Silicate Minerals," International Journal of Mineral Processing, Vol. 59., No. 3, June 2000, 247-266.
- [5] Concrete Pavement Technology Program, "Geopolymer Concrete," FHWA-HIF-10-014, March 2010.
- [6] Hardjito, D., Wallah, S., Sumajouw, D., Rangan, B. V. "On the development of fly ash-based geopolymer concrete," ACI Materials Journal, V. 101, No. 6, December 2004, 467-472.
- [7] Diaz-Loya, E. I., Allouche, E. N., Vaidya, S. "Mechanical Properties of Fly-Ash-Based Geopolymer Concrete," ACI Materials Journal, V. 108, No. 3, June 2011, 300-306.
- [8] Guo, X., Shi, H., Dick, W.A., "Compressive strength and microstructural characteristics of class C fly ash geopolymer," Cement and Concrete Composites, V. 32, November 2009, 142-147.
- [9] Habert, G., d'Espinose de Lacaillerie, J.B., Roussel, N. "An environmental evaluation of geopolymer based concrete production: reviewing current research trends," Journal of Cleaner Production, V. 19, March 2011, 1229-1238.
- [10] Marceau, M., Nisbet, M., VanGeem, M. "Life cycle Inverntory of Portland Cement Concrete," PCA R&D Serial, No. 3011, 2007.
- [11] ASTM C109/C109M-16a Standard Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or [50-mm] Cube Specimens), ASTM International, West Conshohocken, PA, 2016, https://doi.org/10.1520/C0109_C0109M-16A
- [12] ASTM C39/C39M-18 Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens, ASTM International, West Conshohocken, PA, 2018, https://doi.org/10.1520/C0039_C0039M-18
- [13] ASTM C469/C469M-14 Standard Test Method for Static Modulus of Elasticity and Poisson's Ratio of Concrete in Compression, ASTM International, West Conshohocken, PA, 2014, https://doi.org/10.1520/C0469_C0469M-14