GEOPOLYMER CEMENT CONCRETE: A SENSITIVITY ANALYSIS

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ABSTRACT

Geopolymer cement is an alternative binder manufactured from fly ashes that can fully replace portland cement in traditional reinforced concrete applications. As a viable material for precast components, geopolymer features very rapid strength development and the ability to be prepared using typical concrete equipment. This study focuses on a sensitivity analysis of three base mix designs of geopolymer cement concrete with compressive strengths ranging from 1500-6000 psi. The main purpose of the sensitivity analysis is to determine the level of influence of typical production variables on the resulting mechanical properties of the concrete. The production variables, such as, *w/cm*, curing temperature, and curing duration are each known to impact the compressive strength and elastic properties of GCC. Regression analysis was used to determine the level of influence of each production variable on mechanical properties. The interactions between changes in the production variables were also studied to determine if a combination of variables may have a greater net impact on the mechanical properties of the concrete. From the analysis, we find that as we adjust production variables we can influence the resulting compressive strength. These relationships have been illuminated with the regression models.

Keywords: Geopolymer, Alternative Binder

INTRODUCTION

This research was undertaken following the prototype production of precast geopolymer cement concrete panels in a PCI producer-member plant. The panels were used to construct a highly energy efficient single family residence for the US Department of Energy's Solar Decathlon. The house and production process were described in (Tempest et al 2015).[1] Following this production experience, it was evident that if geopolymer cement concrete (GCC) is to enter more routine production, new specifications for production variables will be needed to ensure quality in the cured material. Current tolerances for water additions, curing conditions, and other variables that might be affected in the course of precast concrete production and apply to portland cement concrete (PCC), do not apply to GCC.

In general, GCC is prepared by mixing an alumina-silicate source material, such as fly ash, with an alkaline solution that "activates" cementitious properties in the alumina-silicate. This combination of "activating solution" and alumina-silicate material may be mixed with aggregates and formed much like PCC. However, unlike PCC, GCC attains its compressive strength characteristics after a period of elevated temperature curing. Therefore, in addition to conditions, such as water content and curing environment, that are maintained for PCC, are factors in GCC production and must be similarly controlled.

The purpose of this study was to determine the impact of controllable variables in the geopolymer concrete mixing and curing process to the mechanical characteristics of the cured concrete. The goal is to determine the level of importance of each variable and to provide data that can be used to draft tolerances and specifications. The three variables which were analyzed were the water-cementitious materials ratio (*w/cm*), the curing temperature, and the duration of elevated temperature curing. The analysis determined which of these variables have the most influence on the mechanical properties of geopolymer concrete. A combination of these variables may exist and it is important to understand interactions between variables. Brief descriptions of these variables follow.

WATER-CEMENTITIOUS MATERIALS RATIO (W/CM)

In this study the *w/cm* is defined as the weight ratio of the mixing water and water contained in the sodium silicate solution to the weight of the sodium silicate solids, the fly ash and the sodium hydroxide. As with PCC, the *w/cm* is an important determinant of several mechanical and durability properties of GCC. Because of only limited dependence on hydration processes, the presence of water is not required during the curing process of GCC. However, it has been observed that the initial presence and later loss by evaporation of the water molecules creates pockets of air which affect the mechanical properties of GCC. Additionally, some amount of water is essential to achieve adequate workability. Therefore, in this study, the degree of detrimental effects on the mechanical properties by increasing the water content for better workability was determined by creating concrete specimens at one of three *w/cm* (as described in Table 1. The mix design with *w/cm* 1 is a stiff mix, *w/cm* 2 contains a ten percent increase in water content over *w/cm* 1 and allows for some workability, and *w/cm* 3 contains a 20% increase in water content.

HIGH TEMPERATURE CURING TEMPERATURE

The curing temperature of GCC has largely been observed to play a role in the mechanical properties of the concrete, primarily in the compressive strength[2-5]. An increase in curing temperature has a positive effect on the properties. In this study the intent was to quantify the temperature impact to the development of compressive strength by varying the curing temperature. A curing temperature range of 27°F from 140°F to 167°F was evaluated to determine this relationship between curing temperature and compressive strength.

HIGH TEMPERATURE CURING DURATION

The curing process of GCC consists of three phases. Immediately after mixing, the GCC typically rests in the forms for a period of from several hours to multiple days. Following this rest period, the temperature of the concrete is elevated for a period of time, during which most of the strength gain occurs. Finally, additional strength gain may further occur in the weeks and months following the high temperature phase. The high temperature curing duration of GCC is a variable of great importance in the mechanical properties with previous research indicating an increase in compressive strength when allowed to cure for a longer period of time[2-5]. In this study the role of the curing duration was evaluated such that samples were cured for either 12, 24, 36, or 48 hours.

MIX DESIGN

The mix design used in this study is the same one used to cast the residential building described in (Tempest et al 2015).[1] The initial mix design was developed with trial and adjustment methods for ash:activator ratios and cement:aggregate ratios. The Fuller method was used to proportion aggregates in order to achieve both density and workability in the fresh concrete. Table 1 details the three mix designs used in this study. The variable ingredient of Mixes 1, 2 and 3 is the water content, which increases by 10% between Mix 1 and Mix 2, and 20% between Mix 1 and Mix 3. Table 2 outlines the breakdown of the mix, curing duration, and curing temperature for each sample type.

The coarse aggregates used for this study conformed to ASTM C33[6] size #57 and the fine aggregates conformed to ASTM C33[6] grading requirements for fine aggregate. The coarse aggregate was prepared to saturated-surface dry conditions and the fine aggregate to oven-dried conditions prior to all mixing. The fly ash used in the study is classified as Class F per ASTM C618[7].

Table 1. Mix designs used for samples, pounds per cubic yard.

Mix	1	2	3
Water, lb./yd.^{3}	2028	2230	2433
Sodium Silicate, lb./yd.^{3}	7479		
Sodium Hydroxide, lb./yd.^{3}	977		
Fly Ash, lb./yd.^{3}	21244		
Fine Aggregate, lb./yd.^{3}	1370.3		
Coarse Aggregate, lb./yd.^{3}	1370.3		
Total, lb./yd.^{3}	3915.7	3923.2	3930.7

PRODUCTION

Batches were mixed in the early morning, typically, over a five to six hour period with prior setup and collection of materials. The morning mixing time was crucial for proper mixing as ambient temperatures were in the range of 80˚F to 90˚F. The consistent mixing prior to the highest heat of the day was important to maintain a mixing procedure preventing the concrete from drying out too quickly. A standard three cubic foot capacity concrete mixer was used to prepare all the mixes which required a somewhat cautious and slower mixing process. The mixing process consisted of mixing half the total coarse aggregate with half the total fine aggregate, adding half the total fly ash and allowing to mix for approximately fifteen (15) minutes. Following this, the total activating solution was added to the mix along with the total water and the concrete mixed for another fifteen (15) minutes. Lastly, the last half of the fine and coarse aggregate was added and the concrete mixed for a final fifteen (15) minutes. The mixing procedure was organized such that consistency between batches could be achieved and it allowed the mixers to handle the large quantities of materials. The basic regimen consisted of mixing a portion of the aggregates followed by a portion of the fly ash, this process minimized the material lost after each addition. The fly ash tends to escape out of the mixer very easily when mixing and can create significant losses and changes in the mix if too much is lost. The possibility of losing fly ash is also the key reason for adding the activating solution and water halfway through, this process allows the fly ash to settle and mix into the proper paste and concrete.

The casting of samples was performed within thirty (30) minutes of final mixing. Plastic 4"x8" cylinders were used to cast the samples for compressive strength testing. After casting, samples were matured at room temperature for twenty-four (24) hours and then moved to a lab oven and heat cured at the temperatures designated in Table 2. Samples were removed from the oven as they reached the appropriate high heat curing duration. The high heat curing durations for each sample group type can be found in Table 2**.**

Table 2. Sample type designation and description.

Mix	Curing duration, hr.	Temperature, °F		
		140°F	158°F	176°F
1	12 and 24	1.60.C	1.70.C	1.80.C
	36 and 48	1.60.B	1.70.B	1.80.B
	24 and 48	1.60.A	1.70.A	1.80.A
2	12 and 24	2.60.C	2.70.C	2.80.C
	36 and 48	2.60.B	2.70.B	2.80.B
	24 and 48	2.60.A	2.70.A	2.80.A
3	12 and 24	3.60.C	3.70.C	3.80.C
	36 and 48	3.60.B	3.70.B	3.80.B
	24 and 48	3.60.A	3.70.A	3.80.A

The fresh concrete properties were measured as part of the correlation to the durability test procedures. The primary properties measured were the slump flow of the concrete and the air content of the concrete. All of these properties were measured on mixes with an "A" designation.

SLUMP FLOW

Due to its consistency, GCC may produce erroneous results if workability is measured by the ASTM C143, Standard Test Method for Slump of Hydraulic-Cement Concrete[8]. As a result, the preferred alternative method was the Standard Test Method for Slump Flow of Self-Consolidating Concrete (ASTM C1611)[9] used as a means of measuring the consistency of the fresh concrete. The test consists of two pieces of data; the first being a visual stability index and a measurement of the circular spread of the concrete. Test procedure B was used in the testing of the concrete.

Figure 1. Slump flow test setup using Procedure B of ASTM C1611[9].

Figure 2. Slump flow test sample once allowed to flow completely per ASTM C1611[9].

Table 3. Average reading of slump flow, in., over a minute and a half interval.

	Time elapsed at reading [seconds]		
Mix Design	30	60	90
1	22.75	23.50	24.38

	Time elapsed at reading [seconds]		
Mix Design	30	60	90
1	22.05	22.25	23.75
1	22.00	24.25	24.38
2	18.25	20.75	21.75
2	21.00	23.00	24.00
2	19.75	21.25	21.75
3	23.25	25.50	26.25
3	22.25	24.00	25.25
3	24.50	26.75	27.25

The results indicated some consistency between similar batches at a small degree. As expected, the highest water content had the highest slump flow values, however the change between the flow of *w/cm* 1 and 2 was not as expected and showed values much closer to each other.

AIR CONTENT

Air content of the fresh material was measured by both volumetric and pressure methods. Results are shown in Table 4. The measured values using both the volumetric (ASTM C173)[10] and pressure (ASTM C231)[11] methods varied only slightly. The samples ranged from a 1% air content to 4% air content. Taking into consideration the very small range of water content that was considered in the three mixes, the change in air content due to the constituent material volumes should be minimal. Also, no attempt was made to entrain additional air by using admixtures.

Table 4. GCC fresh concrete air content values measured with procedures from ASTM C173[10] and ASTM C231[11].

Mix Design	Volumetric ASTM C173	Pressure ASTM C231
1	1.25%	2.80%
1	-	3.00%
1	1.00%	-
2	1.75%	2.80%
2	-	2.80%
3	1.25%	2.40%
3	1.50%	4.00%

COMPRESSIVE STRENGTH TESTING

The results of the compressive strength testing are summarized in Table 5 and Figures 3-5.

Table 5. Twenty-eight (28) day compressive strength of mix designs 1, 2, and 3 following each curing regimen.

Mix Design	Elevated Curing Temperature [°F]	Elevated Curing Time [hours]	28-day compressive strength [psi]
1	140	12	2,715
		24	3,554
		36	3,298
		48	3,751
	158	12	3,250
		24	4,763
		36	5,517
		48	6,008
	176	12	5,153
		24	5,143
		36	5,843
		48	5,772
2	140	12	2,148
		24	2,631
		36	2,892
		48	3,358
	158	12	2,965
		24	3,904
		36	5,038
		48	5,231
	176	12	3,481
		24	4,347
		36	4,622
		48	5,407
3	140	12	1,048
		24	1,954
		36	2,511
		48	2,859
	158	12	2,950
		24	4,149
		36	3,973
		48	4,443
	176	12	3,691
		24	4,397
		36	5,485
		48	4,446

Figure 3. Relationship between *w/cm* and curing duration for samples cured at 140°F with data error bars showing the standard deviation.

Figure 4. Relationship between *w/cm* and curing duration for samples cured at158°F with data error bars showing the standard deviation.

Figure 5. Relationship between *w/cm* and curing duration for samples cured at 176°F with data error bars showing the standard deviation.

EFFECT OF W/CM RATIO

The general trend observed in Figures 1 – 3 is a decrease in compressive strength as a result of increase in *w/cm*. A lower *w/cm* is desirable, however, it is possible to achieve up to 80% of the total potential of a particular *w/cm* minimum conditions. For example, assuming the maximum potential of "*w/cm*: 1" was 6000 psi at 28-days then with the same conditions a strength of about 5,200 psi with "*w/cm:* 2" can be achieved and 4800 psi with "*w/cm*: 3." The control of water in this situation thereby increases the potential of the concrete by up to 120% of the baseline for the mix at maximum curing conditions.

Another example, examining the trends relating to maintaining the *w/cm* and changing the curing temperature and high heat curing duration, assuming the maximum potential of "*w/cm*: 1" is 6000 psi at 28-days then with varying production variables and keeping the same w/c, a minimum 40% of the compressive strength is possible. The same relationship for "*w/cm*: 2" from 5300 psi maximum potential is at a minimum 32% of the potential and with "*w/cm*: 3" a minimum 27% from the maximum potential of 4900 psi. This indicates that as the water content of GCC increases the ability to control the compressive strength is lost and a greater range of strengths is created. Therefore, the 10% increase in water content of the base mix design is not beneficial for a higher strength because a loss of approximately 1000 psi occurs in the 10% change.

EFFECT OF CURING TEMPERATURE

The overall trend observed is that the curing temperature is more significant when curing at 158°F or higher. The relationships between samples cured at 140°F to 158°F was a much more drastic change in strength versus the change between 158°F and 176°F. In particular, a very obvious difference in the mean compressive strengths is visible from 140°F to 158°F at every stage of the concrete but the difference varies at each stage when going from 158°F to 176°F. At age zero, the 158°F to 176°F change was greatest, at 14-days the difference is less significant, and at 28-days a slightly greater difference occurs. Essentially, the most important relationship to take away from this data is that regardless of the mix design, a 158°F minimum curing temperature is desirable to achieve a more precise range of strengths for the mix designs. Curing at temperatures lower than 158°F produces too much variability in the strengths.

Regardless of the *w/cm*, higher curing temperature and longer curing duration result in the same pattern of strength increase. Because of this, the goal of adjusting the production variables may become more concentrated on determining how *precise* and *controlled* a design concrete needs to be to meet mechanical property requirements.

EFFECT OF HIGH HEAT CURING DURATION

A significant increase in compressive strength is noted when increasing the curing duration from 12 hours to 24 hours. The same great gain does not apply to any curing above 24 hours, however, curing of 48 hours always produced higher compressive strengths. However, a more significant change over time is from the 36 hour to 48 hour curing duration where at age zero there is a greatest difference in strength occurred from the final 28-day strength. This indicates that a higher percentage of the total strength can be attained earlier on by curing for 48 hours.

SENSITIVITY ANALYSIS

The purpose of the sensitivity analysis presented here is to determine the level of influence of a production variable on the resulting mechanical properties. The production variables *w/cm*, temperature during high temperature curing, and high temperature curing duration are each known to impact the compressive strength and elastic properties of GCC. This analysis determines the level of influence by changing each variable or a combination of variables and measuring the outcome.

Multiple analyses were performed varying the dependent variables and measuring the outcome on the dependent variable (compressive strength). The study uses the compressive strength as the dependent variable and the *w/cm*, curing temperature, and curing duration as the three separate independent variables. The age of the sample is also taken into account as a separate variable. This portion of the analysis seeks to determine whether the level of importance of a variable increases or decreases as the concrete ages and the microstructure continues to develop.

The primary method of analysis for the data involved a stepwise multiple regression analysis. In the analysis, two multiple regression models are used. In the multiple regression, it is assumed that the compressive strength is the dependent or Y-variable. The production variables become the independent X_n-variables. The following is breakdown description of the variables:

Y: compressive strength

X_1: *w/cm*

X_2: curing temperature

X_3: curing duration

As there are three independent variables, interactions between the variables were also considered. It was previously known that each of the production variables can potentially affect the compressive strength. But the goal of this multiple regression analysis and model is to determine how more than one variable can affect the compressive strength. The analysis seeks 1) to identify the magnitude of the effect that each production variable has on the compressive strength outcome and, 2) to determine whether the effects of the production variables are fully independent to each other, or are dependent on the value of the other production variables. The goal becomes then to achieve the types of if-then scenarios for combinations or adjustments of variables.

The appropriateness of the model that is created is then measured by the *coefficient of determination* typically defined as a measure of model accuracy based on the experimental data. The coefficient of determination is best known as the "R^2" of a model equation and the closer the value is to one, the better the model fits the data. The relationship is given in Equation 1. The R^2value indicates what percentage of data points fall into the proposed model for the experimental data.

Equation (1)

In the case of these data, the statistical processer, Minitab, was used to create the regression models for this study (Minitab Inc.). The input to the processer were the production variables and the compressive strength results of all the samples that were prepared and tested to build the best fitting model. The processor builds each variable into the equation and performs a stepwise process to add in the pieces of the multiple regression model. Following each addition of a variable the R^2 value is checked for the fitting of the model. Once the processor completes the additions of all the variables it finalizes the model.

The multiple regression analysis of all the data considers the production variables, *w/cm*, curing temperature, and curing duration, and their impact to the compressive strength. The modeling results from the Minitab statistical processor are shown in Table 6**.** The first analysis is a single interaction model where the variables are individually taken into account creating a first-order linear model. The second model includes the possibility of interactions between variables by creating a full quadratic model, this is indicated by the polynomial and squaring of variables in the equation.

Table 6. Multiple regression models and optimization results of analysis of all data.

Model Equation	Model R^2	% incremental increase in R^2
Linear Model $Y = 296 - 53188X_1 + 107.41X_2 + 47.78X_3$	82.36%	X_1, 10.9666 X_2, 47.1551 X_3, 25.6197
Quadratic Model $Y = -14630 - 306209X_1 + 876.8X_2 + 1711X_3 + 1288794X_1^2 - 5.510X_2^2 - 1.038X_3^2 - 595X_1{*}X_3$	88.39%	X_1, 11.5560 X_2, 50.9746 X_3, 26.0861

Both models show that the curing temperature has the largest impact on the compressive strength followed by the curing duration and *w/cm*. The impact defined by the model is the progressive adjustment of the model and the increasing of the R^2 value. Thus, the conditions that resulted in the greatest compressive strength were related to the highest curing temperature, the longest curing duration, and the lowest *w/mc*. The results consistently show that the higher compressive strengths depend on the maximized highest curing temperature, maximized curing duration, and minimized water content.

The quadratic model fits the data more closely. As the model changes from a first-order linear model to a quadratic model, an increase from R^2 = 82.36% to R^2 = 88.39% is observed. This indicates that the relationship between the production variables and the compressive strength is not linear, and also has some dependence on combinations of variables. The model shows interactions between the *w/cm* and the curing duration as a part of the equation (the -595X_1*X_2 portion). This demonstrates that the *w/cm* in conjunction with the curing duration have an effect on the compressive strength. The third column in Table 6 "% incremental increase in R^2," shows the individual production variables' impacts to the final model. It shows which independent variable led to a higher R^2 value for the model. The higher contribution to R^2 is associated with greater influence over compressive strength development. In both models, the curing temperature has the greatest influence to the R^2 value of the equation followed by the curing duration and the *w/cm*, respectively. This pattern was found in both the linear and non-linear models and indicates the importance of the curing temperature on the compressive strength. Based on the models, a lower *w/cm* is ideal. However, it is possible to overcome the benefit of a lower *w/cm* with a higher curing temperature and even a lower curing temperature with a higher curing duration.

Figure 6. Full quadratic model equation showing the model and experimental data for the three curing temperatures for *w/cm:* 1; concrete age is twenty-eight days.

Figure 7. Full quadratic model equation showing the model and experimental data for the three curing temperatures for *w/cm:* 2; concrete age is twenty-eight days.

Figure 8. Full quadratic model equation showing the model and experimental data for the three curing temperatures for *w/cm:* 3; concrete age is twenty-eight days.

The test data is shown with the quadratic model equation in Figures 4-6. The overall trend shows the lowest *w/cm* as having the highest compressive strengths throughout. However, observation of the results shows that that a range of strength between approximately 3500 psi to 4000 psi could be achieved using any of the *w/cm* by varying the curing duration and curing temperature. Greater curing duration consistently increases the compressive strength and the quadric models show the more significant impact of increasing from a 24 hour curing duration to a 48 hour curing duration. As the curing duration is increased the values of the compressive strength increase and the apparent impact of the *w/cm* increase as well.

Of the 158°F curing temperature series, the lowest curing duration of 12 hours for each *w/cm* is very similar to the 140°F and 48 hour curing samples. These trends are distinctly visible in Figures 4-6 and show the concurrent relationships between the production variables of GCC as they may impact the compressive strength. These production variables show that there is a degree of flexibility in mix designs if the target compressive strength is a plus or minus 500 psi, for most cases. Essentially, the relationships show that if too much water is added to the mix it can, potentially, be accommodated by increasing the curing temperature and curing duration to achieve the desired compressive strength

CONCLUSIONS

The mix design of GCC plays a significant role in the predicting its final mechanical properties. A proper balance of the necessary quantities of aggregates, fly ash, water, and activating solution are key, however, these results have shown that the curing regimen may be of greater importance within some ranges. Although this paper does not propose tolerances for these characteristics, it is apparent that an eventual specification would include some policy for water additions as well as curing temperature and duration.

The study described in this paper employed source materials from only one source. In particular, the single source of fly ash may not be representative of the performance of other ashes from other coal plants due to the great variability of this material. As such, the ash composition and quality is also a production variable but one that can not be controlled as easily as those discussed in this paper. Because of this aspect of GCC production, an eventual standard will most likely be largely performance based. The variables considered also did not leave a constrained range set by the research team. Only ranges that were known to produce acceptable workability and compressive strength results were attempted. One might expect greater non-linearity outside of these ranges. Nevertheless, the proposed combinations of production variables are limited in that this study only presents the optimization within the specified production ranges and has not concluded by determining the absolute optimum value for any production variable.

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