Statistical software to improve the accuracy of geopolymer concrete mix design and proportioning

Carlos Montes[1], Sergio A. Gomez[1], Niraj Khadka[1], Erez N. Allouche[1]

[1]Louisiana Tech University, Alternative Cementitious Binders Laboratory, Trenchless Technology Center 1207 Agriculture Drive, Ruston, LA 71270;

KEYWORDS: geopolymer, mix design, statistical analysis

ABSTRACT

The mix design and proportioning of geopolymer concrete is a complex process due to more variables being involved compared to Portland cement systems. ACI 211.1 is the standard practice used to create proportionings for Portland cement concrete mix designs but it is unable to account for changes in compressive strength and slump associated with geopolymer key parameters like activator solution concentration, fly ash type, curing conditions among others.

The present paper describes the development of statistical geopolymer mix design software based on ACI 211.1 that incorporates the aforementioned geopolymer parameters of fly ash type, activator solution concentration and others. A design of experiments for those variables was created and their effect on target compressive strength and slump evaluated. This data was incorporated in the software to improve its prediction accuracy and new trial batches were prepared to evaluate these improvements. Results show that with a few modifications, it is possible to create mix designs and proportioning of geopolymer concrete with the guidance of ACI 211.1.

INTRODUCTION

ACI 211.1 is the current practice utilized for the proportioning of normal, heavyweight and mass concrete in the United States and worldwide [1]. Even though this guideline produces accurate proportionings based on specifications like compressive strength and slump, its use is limited when it comes to the proportioning of geopolymer concrete (GPC) [2]. This new type of fly ash-based cementitious binder requires the use of more design parameters like activator solution to fly ash ratio (in substitution of the water/cement ratio), the concentration and ratio of the activator solutions, curing conditions, etc., [3] which means that the mix design and proportioning process becomes more complex than when working with ordinary Portland cement (OPC) systems.

These geopolymer activator parameters have a great influence in its fresh properties. [4]. Several researchers have reported the rheological behavior of geopolymer dependent on the molar concentration of the silicate and the ratio of silicate to hydroxide solutions [5, 6, 7]. And naturally, these same parameters also affect the hardened properties of geopolymer concrete [8].

The absolute volume method described by ACI 211.1 can be used for proportioning of GPC, however, the incorporation of the aforementioned geopolymer parameters into the proportioning process is essential to produce more accurate results.

A mix design and proportioning software developed by the Trenchless Technology Center (TTC) of Louisiana Tech is a tool created to address these important issues. Even though there is still the need to produce trial batches to confirm the predictions of the software, the intention is that the predictions are accurate enough to minimize the trial and error process after the theoretical calculations. Such kind of softwares exist to help on the propotioning process of Portland cement concrete [9, 10], but so far there is no software to help on the proportioning of geopolymer concrete.

Statistical information for the software was gathered from an testing plan based on a design of experiments created on MINITAB. Testing was conducted based on current ASTM standards. The software is designed to accumulate experience and improve by receiving feedback from the user.

MATERIALS AND METHODS

Design of experiments

Three activator solution control and two response variables were selected to evaluate the accuracy of the ACI 211.1 practice to predict the compressive strength of GPC. Two design variables were included to analyze the results at three different levels of compressive strength and slump. Also, five factors (control parameters) were kept constant throughout the experimental plan. The variables and control parameters are summarized in Table 1.

Table 1. Variables used for the design of experiments.

CONTROL VARIABLES	**RESPONSE VARIABLES**	**CONTROL PARAMETERS**
ACTIVATOR SOLUTION VARIABLES	1-day oven cured compressive strength in 4”x8” cylinders	Curing time: 24 hrs
Silicate type (SiO_2/Na_2O ratio of 2.0 and 3.2)	Slump	Curing temperature: 140 F (60 C)
Hydroxide concentration (10, 12 and 14 M)		Fine aggregate: river sand
Silicate/hydroxide ratio (1, 1.5 and 2)		Coarse aggregate: gravel with some crushed particles
DESIGN VARIABLES		
Compressive strength (3000 and 5000 psi [20 and 35 MPa)	Slump (1-2 in [25-50 mm], 3-4 in [75-100 mm], 6-7 in [150-175 mm])	

A design of experiments for the activator solution control variables for each combination of the two variables was created using the software MINITAB. MINITAB provided the experiments required and a randomized order of testing. Also, MINITAB provided the analysis of variance of the results and the deviation from the expected outcome, providing data that was fed into the mix design and proportioning software.

Mix design and proportioning software

A custom mix design software based on ACI 211.1, currently under development at the Trenchless Technology Center, was utilized to formulate the various mix designs incorporating three geopolymer variables (silicate type, hydroxide concentration and silicate/hydroxide ratio) in addition to the activator solution to fly ash ratio (the water/cement ratio of traditional Portland cement mixes). The software utilizes input parameters from the aggregates (e.g., specific gravity, bulk density, etc.), powder and liquid precursors to produce an initial formulation to meet strength and slump requirements for a specified volume. A screen of the software is shown in Fig. 1.

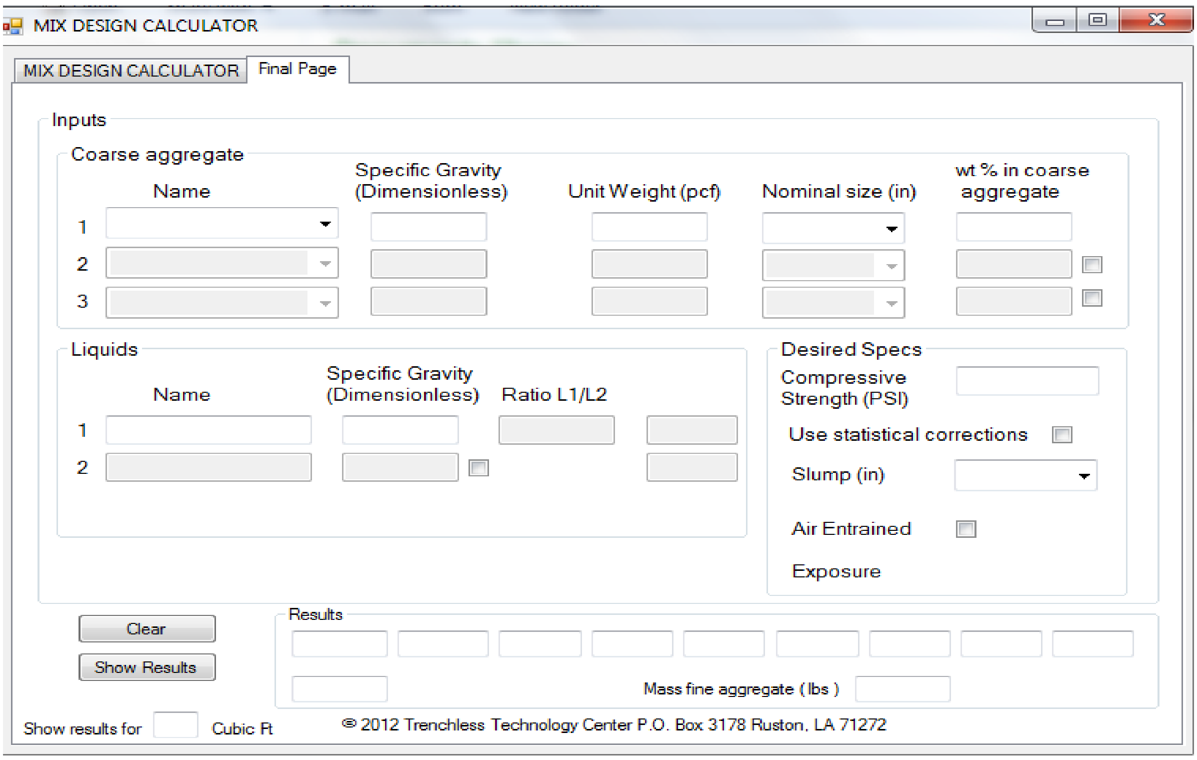

Fig. 1. Geopolymer software screen shot containing user input information.

The software output was an initial mix design based on volume calculations, data and tables from ACI 211.1 to obtain concrete specimens with the selected design compressive strength and slump. The compressive strength for ACI 211.1 is based entirely on the water/cement ratio. In the geopolymer software water/cement ratio is replaced by the activator solution/fly ash ratio of geopolymer concrete. However, other factors such as silicate type, hydroxide concentration and silicate to hydroxide ratio are not included in ACI 211.1. Therefore, several mix designs for selected levels of compressive strength and slump were created to estimate their effect on the prediction values. The deviation from design values of the results obtained from this design of experiments was estimated and incorporated in the software to further increase its prediction potential.

Materials

Fly ash from CLECO's Dollet Hills power plant located in Mansfield, LA was selected for this study. The chemical composition, phase composition and other characteristics of this fly ash can be seen in Table 2.

Table 2. Characteristics of the fly ash utilized in the study.

Chemical composition (% wt.)		**Phase composition (% wt.)**	
SiO_2	59.32	Quartz	12.2
Al_2O_3	19.72	Mullite	4.8
CaO	6.90	Amorphous	83
Fe_2O_3	7.22	**Other characteristics**	
MgO	2.23	Percent finer than 45 µm	62.97
Na_2O	1.11	Specific gravity	2.23
TiO_2	1.00		
Other oxides	2.35		
LOI	0.15		

Commercial sodium silicate solutions from PQ ® of two types (D and N) were used for this project. The specifications are on Table 3

Table 3. Characteristics of the sodium silicate utilized in the study.

Sodium silicate type	D	N
SiO_2/Na_2O ratio	2.0	3.2
Na_2O wt%	14.7	8.9
SiO_2 wt%	29.4	28.7
Specific gravity	1.53	1.38
Viscosity (cPoises)	400	180

Sodium hydroxide flakes of 99% purity were used to prepare sodium hydroxide solutions with a molarity of 10, 12 and 14.

Experimental procedure

After obtaining the proportioning for each experiment from the mix design software, geopolymer concrete was mixed using a paddle mixer (Fig. 2a) and poured in 6x12 inch [15x30 cm] cylinders according to ASTM C192 (Fig 2b). Samples were allowed to cure for 24 hours at 140 F [60 C] for 1 day (Fig 2c). Slump was measured according to ASTM C143 (Fig. 2d) and density was measured according to ASTM C138. Compressive strength was evaluated according to ASTM C39 (Fig 2e). Three cylinders were prepared for each combination and averages and standard deviations calculated. A total of 324 specimens were prepared for 108 experiments (Fig 2f).

Fig. 2 (a) Mixing, (b) preparing cylinders, (c) curing, (d) measuring slump, (e) compressive strength test, (f) test specimens.

RESULTS AND DISCUSSION

Three specimens per combination were tested and their results averaged. The results were fed into the MINITAB software for analysis of variance (ANOVA) and the calculation of means and standard deviations. Fig. 3 summarizes the ranges obtained by each one of the levels of the main variables. It can be seen that both silicates and silicate/hydroxide ratios were able to provide the full range of compressive strengths included in ACI 211.1 (2000 to 7000 psi). Since silicate N is more commercially available and less concentrated than silicate D. It was decided that it is not necessary to use silicate D to produce geopolymer concrete from this fly ash at this level of compressive strengths. Also, both silicate/hydroxide ratios were able to produce the full range of compressive strengths, therefore silicate/hydroxide ratio of 1.5 was selected because it produces better workability, finishability and it is also more economical.

Table. 3 Ranges of the levels of the variables of the design of experiments for compressive strength (psi [MPa]).

Compressive strength		GENERAL DESIGN		
		Min	Max	Range cover
Silicate	N	1438 [9.9]	8414 [58.0]	✔
	D	2039 [14.1]	8550 [59.0]	✔
Ratio	1	1438 [9.9]	8414 [58.0]	✔
	1.5	2039 [14.1]	8281 [57.1]	✔
NaOH Molarity	10	1585 [10.9]	6638 [45.8]	✔
	12	1830 [12.6]	8006 [55.2]	✔
	14	1438 [9.9]	9346 [64.4]	✔

From Table 3 it can be seen that not all the hydroxide molarities were able to reach the full range of compressive strengths specified in the design of experiments. Therefore it was necessary to determine which molarity would be optimal for a particular set of compressive strength and slump. From a cost perspective, it is desirable to reduce the molarity of hydroxide solutions as much as possible. In Fig. 4 the optimal molarities for each combination of compressive strength and slump are highlighted. It can be seen that a molarity of 10 is sufficient in most cases, and a molarity of 12 and 14 is required only in two specific cases of high strength. A compressive strength within 3 standard deviations from the design strength was considered appropriate for the particular combination because statistical correction factors would be applied to produce mix design for a particular minimum compressive strength.

Table 4. Experimental compressive strength values for the three levels of molarities for each level of compressive strength and slump (in [mm]).

	3000 PSI		
MOLARITY	1~2	3~4	6~7
10 M	3352 [23.1]	2157 [14.9]	2627 [18.1]
12 M	3473 [24.0]	2496 [17.2]	2379 [16.4]
14 M	3203 [22.1]	2359 [16.2]	2659 [18.3]

	5000 PSI		
MOLARITY	1~2	3~4	6~7
10 M	5327 [36.7]	4079 [28.1]	3857 [26.6]
12 M	4541 [31.3]	4059 [28.0]	4904 [33.8]
14 M	6000 [41.4]	5183 [35.7]	5306 [36.6]

	7000 PSI		
MOLARITY	1~2	3~4	6~7
10 M	4691 [32.3]	6395 [44.1]	5445 [37.5]
12 M	5868 [40.5]	6982 [48.1]	6297 [43.4]
14 M	7574 [52.2]	8281 [57.1]	7271 [50.1]

The same analysis procedure was followed for the slump (Fig. 5). In general, all levels for three variables were able to achieve slump values in the range of ACI 211.1 (1-7 inches).

Table 5. Ranges of the levels of the variables of the design of experiments for slump (in [mm])

Slump		GENERAL DESIGN		
		Min	Max	DR
Silicate	N	0	9 [229]	✔
	D	0	10 [229]	✔
Ratio	1	1 [25]	11 [229]	✔
	1.5	0	12 [229]	✔
NaOH Molarity	10	0	13 [229]	✔
	12	1	14 [229]	✔
	14	1 [25]	15 [229]	✔

Table 6. Experimental slump values for the three levels of molarities for each level of compressive strength and slump (in [mm]).

	3000 PSI		
MOLARITY	1~2	3~4	6~7
10 M	2 [50]	7 [178]	9 [229]
12 M	2 [50]	5 [127]	4 [102]
14 M	2 [50]	5 [127]	8 [203]

	5000 PSI		
MOLARITY	1~2	3~4	6~7
10 M	6 [152]	9 [229]	9 [229]
12 M	4 [102]	9 [229]	9 [229]
14 M	5 [127]	9 [229]	9 [229]

	7000 PSI		
MOLARITY	1~2	3~4	6~7
10 M	0	9 [229]	9 [229]
12 M	0	8 [203]	9 [229]
14 M	0	8 [203]	9 [229]

However, the slump values for the different levels of hydroxide molarity and most particularly for the optimal molarity levels for compressive strength were evaluated and it was found that in most cases (except two), the slump of the samples exceeded those predicted by ACI 211.1.

IMPROVEMENT OF THE PREDICTION CAPACITY OF THE SOFTWARE

The information gathered with the design of experiments was fed into the software to obtain an improved mix design. ACI 318 details standard deviation computations to use test data to create a modification factor for the design compressive strength. When tests for a particular mix design are less than 15, a conservative modification factor should be used. When there are more than 15 tests, a modified standard deviation can be used to calculate a modified design compressive strength. ACI 318 also requires that the concrete used for the calculation of the standard deviation must have a *f'(c)* that lies within 1000 psi of the strength required for the proposed work.

In our study, a conservative correction factor of 3 standard deviations (3S) was used to improve the accuracy of the mix design in terms of compressive strength and to include the mean in the calculations.

In order to compensate for the differences in slump to the predicted values a correction factor based on an interpolation between desired slump and water content from ACI 211.1 was introduced for compressive strengths between 4000 and 7000. Three new mix designs were obtained and tested for accuracy with predictions. The software selected a hydroxide of 10M for the first case and 12 M for the second. The results can be seen in Table 6:

Table 6. Trial batches to prove improved mix designs.

f'(c) / SLUMP	**COMPRESSIVE STRENGTH (psi)**	**SLUMP (in)**
4000 psi / (3-4 in)	4535	4
6000 psi / (6-7 in)	6898	7

CONCLUSIONS

A mix design and proportioning software created at the Trenchless Technology Center was used to formulate geopolymer concrete of different parameters of activator solution, namely, sodium silicate type, sodium hydroxide molarity and silicate/hydroxide ratio. A design of experiments was used to study the effect of these variables. Results showed that a concentrated silicate solution is not necessary to achieve the levels of strength covered by ACI 211.1 and also helped to determine the optimal silicate/hydroxide ratio. Three different molarities can be used depending on the required compressive strength and slump. The statistical data from the design was incorporated in the software to improve its formulation capacity and the quality of its predictions. Trial batches from two formulations were tested and compared to design parameters with satisfactory results. The geopolymer software prediction capabilities can be improved with more data fed into the system.

FUTURE WORK

Future features of the software will include fly ash source recommendations based on the TTC geopolymer database. These recommendations will be based on location, final applications, desired properties (e.g., early compressive strength, corrosion resistance, fire resistance), curing conditions (ambient, heat, etc.). It will also have the capability to produce mix design formulations for mortar and grout, lightweight and cellular geopolymer concrete, use of additives and set retarders.

REFERENCES

[1] ACI Committee 211, *Standard Practice for Selecting Proportions for Normal, Heavyweight and Mass Concrete*, ACI 211.1-91, reapproved 2009, American Concrete Institute, Farmington Hills, Michigan, 1991, 38 pages.

[2] Zubrod, R.W., *Performance Based Specification for Geopolymer Cement Binders*. SRMZ Technical, Inc., Farmington, NM, USA. Oral presentation. ASTM Symposium on Geopolymer Binder System. San Diego, CA. June 26-27, 2012.

[3] Montes, C., and Allouche, E.N. (2012). *Influence of Activator Solution Formulation on Fresh and Harden Properties of Low Calcium Fly Ash Geopolymer*. J. of Coal Comb. & Gasif. Products, Vol. 4, pp. 1-9.

[4] Chindaprasirt, P., Chareerat, T. and Sirivivatnanon, V. (2007). *Workability and strength of coarse high calcium fly ash geopolymer.* Cement and Concrete Composites Vol 29, (I 3), March, Pages 224-229

[5] Laskar, A.I., Bhattacharjee, R. *Rheology of fly-ash based geopolymer concrete*. ACI Materials Journal, V. 108, No. 5, Sep-Oct 2011, pp. 536-542.

[6] Palacios, M; Banfill, P.F.G. and Puertas, F., *Rheology and Setting of Alkali Activated Slag Pastes and Mortars: Effect of Organic Admixtures*. ACI Materials Journal, V. 105, No. 2, Mar-Apr, 2008, pp. 120-148.

[7] Criado, M., Palomo, A, and Fernandez-Jimenez, A., *Alkali Activated Fly Ash: Effect of Admixtures on Paste Rheology*, Rheologica Acta, V. 48, 2009, pp. 447-455.

[8] Wallah, S.E., and Rangan B.V., *Low calcium fly ash based geopolymer concrete: long-term properties*. Research Report GC2. Faculty of Engineering, Curtin University of Technology. Perth, Australia. 2003.

[9] Bentz, D., *Concrete Optimization Software Tool*, http://ciks.cbt.nist.gov/bentz/fhwa, National Institute of Standards and Technology, 2001.

[10] COMPASS, The Transtec Group, http://www.pccmix.com/, 2004.