Analysis and Reproduction of Geopolymer Concrete

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B.S. in Civil and Environmental Engineering Massachusetts Institute of Technology, 2019

Submitted to the Department of Civil and Environmental Engineering in Partial Fulfillment of the Requirements for the degree of

MASTER OF ENGINEERING IN CIVIL AND ENVIRONMENTAL ENGINEERING at the

MASSACHUSETTS INSTITUTE OF TECHNOLOGY

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ABSTRACT

Geopolymers are inorganic polymers based on aluminosilicates that are produced from synthesizing pozzolanic compounds or aluminosilicate source materials with highly alkaline solutions. Geopolymer concrete is a stronger, more durable and more environmentally friendly alternative to ordinary Portland cement (OPC) concrete. Based on Joseph Davidovits' recipe for geopolymer concrete, we varied the ratios of the materials in an attempt to produce the ideal formula for the concrete that withstands maximum compressive strength. Through our iterations, we found the optimum texture was produced when the amount of sodium carbonate and lime are proportionally increased relative to the rest of the materials.

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Chapter 1 - Introduction

The Pyramids of Giza are structural wonders that have lasted years of weathering. There have been theories about the long-standing structures and the components used to construct their concrete. One such theory is from Joseph Davidovits, a mid 1980's French materials scientist, who suggested that the stones had been casted using geopolymerization. Geopolymerization is the binding of aluminosilicates and highly alkaline solutions that hold aggregates together. Geopolymers have ceramic-like properties that exhibit stronger fire resistance than conventional concrete produced by ordinary Portland cement.

1.1 - Motivation

The geopolymer theory that Davidovits romanticized is appealing; however, there is no documentation dating back to Ancient Egypt that would tell us the exact formula of concrete used for the Pyramids of Giza. Moreover, there is hardly any proof that the Ancient Egyptians had access to lime which is the main ingredient of Davidovits's formula and all derivative theories. Although we were unable to confirm the usage of lime in Ancient Egypt, we experimented with the assumption that lime was used. Therefore, we created mixtures in an attempt to improve mechanical qualities of geopolymer concrete using the amount of information ascertained from Professor Hobbs' sample as the base case. Unfortunately, we were unable to test these mechanical qualities of our reproductions due to the COVID-19 outbreak.

1.2 - Literature Review

1.2.1 - Geopolymer Definition

"Geopolymer is a term used to describe inorganic polymers based on aluminosilicates and can be produced by synthesizing pozzolanic compounds or aluminosilicate source materials with highly alkaline solutions" [L.Y. Kong and Jay]

Geopolymer is a binding chemical used to create concrete with fire resistant properties. It has been tested that ordinary Portland cement (OPC) loses strength as temperatures increase which causes chemical changes and weakens the binding between aggregates. The increase of temperature also degrades the concrete cover which can potentially expose any rebar within the concrete. Therefore, geopolymers have been explored as an alternative concrete binder.

1.2.2 - Previous Studies of Geopolymer Concrete

There have been multiple studies of how geopolymers act as a concrete binder. Hardjito et al. noted the difference in geopolymer concrete's strength under varying environments such as temperature, curing time, and type of activator used for the concrete binding reaction. In general, higher curing temperature leads to shorter curing time. Using soluble silicates as activators created geopolymer concrete with an increased compressive strength. The paper's literature review mentioned that "Van Jaarsveld, van Deventer, and Lukey … found that curing for a longer period of time at elevated temperature weakened the microstructure. [And] Barbosa, MacKenzie, and Thaumaturgo stated that the water content played an important role on the properties of geopolymer binders, besides the chemical composition of the oxides used as activators". The figures below describe the results from the experiment by Hardjito et al. where various geopolymer concretes were tested for compressive strength under various environments while considering the constraint of workability.

Mixture	Concentration of NaOH liquid in molarity (M)	Sodium silicate/NaOH liquids ratio by mass	7-day compressive strength after curing at 60 °C for 24 h, MPa
A-1	8M	0.4	17.3
A-2	8M	2.5	56.8
A-3	14M	0.4	47.9
A-4	14M	2.5	67.6

Table 2—Effect of parameters on compressive strength



Figure 1: The effect of curing temperature on two mixtures specified in the table



Figure 2: The relation between curing time and compressive strength

Figures 1 and 2 clearly show that the geopolymer concrete surpasses the ordinary Portland cement in compressive strength. Ordinary Portland cement typically has a compressive strength of 53 MPa ["Concrete Technology.", 2020]. Hardjito et al. also noted that the fresh geopolymer concrete was stiff after mixing. Even though the mixture was fluid enough to compact into cylinders, significant improvement in workability was desired.



Figure 3: Effect of superplasticizer on compressive strength



Figure 4: Effect of various superplasticizers on compressive strength

Kong et al. used superplasticizers in an attempt to overcome the lack of workability in fresh geopolymer concrete. Figure 3 shows that superplasticizers have a significant effect on the compressive strength of concrete. Figure 4 shows that even though various superplasticizers were used, the mix without superplasticizers was consistently stronger. Although adding superplasticizers to geopolymer concrete improved workability, the improvement was not significant enough to make up for the decreased compressive strength. "Superplasticizers do not improve [the] workability of fly ash-based geopolymer ... Superplasticizer[s] therefore is not beneficial in reducing water content as observed in the case of OPC concrete". [Kong et al., 2009]



Figure 5: Effect of water to solids ratio on compressive strength

Hardjito and Rangan observed the correlation between water to solid ratio in the concrete and its compressive strength. Figure 5 clearly shows that the geopolymer concrete, reacting similar to OPC, is weaker at the higher water to solid ratio. The graph also shows that the relative strength of the geopolymer concrete increases as the curing temperature increases. Hardjito and Rangan noticed water was expelled during the geopolymer chemical reaction provided by Davidovits. They explained that the "water ... leaves behind discontinuous nano-pores in the matrix, which provide benefits to the performance of geopolymers ... plays no role in the chemical reaction that takes place ... [and] merely provides the workability to the mixture during handling". [Hardjito and Rangan, 2005]

Unfortunately, Hardjito et al. and Kong et al. did not provide the chemical formula they used for the binder. Yang et al. on the other hand, provided their chemical reaction as:

 $(2SiO_2 \cdot Al_2O_3 \cdot 2H_2O) + NaHCO_3 + CaO + H_2O \rightarrow Na_2O \cdot 2SiO_2 \cdot Al_2O_3 \cdot 3H_2O + CaCO_3$ It is the same as Davidovits' formula except NaHCO₃ which is typically replaced by Na₂CO₃ (Sodium Carbonate). Yang and her team had to use NaHCO₃ as an alternative due to lack of availability.

Kong et al. on the other hand described their mixing process. They started by dry mixing the coarse and fine aggregates in a pan mixer for 3 minutes. Then the alkaline solutions were added into the aggregates and mixed for an additional 7 minutes. Finally, the concrete was put into 100 mm diameter by 200 mm high cylindrical molds and compacted using a vibration table, then sealed to prevent evaporation.

The mixtures were all cured at room temperature for 24 hours and then subjected to 80°C and 93% humidity for another 24 hours. Some samples were further autoclaved at 800°C for an hour before they were allowed to cool to room temperature in the oven.

1.2.3 - Environmental Impact of Geopolymers

According to the National Ready Mix Concrete Association, 927 kg of carbon dioxide is emitted for every 1000 kg of ordinary Portland cement. There have been serious efforts to create a more environmentally friendly concrete. Some suggest changing the production process of concrete to lower carbon footprint, while others believe the key is finding alternative ingredients for concrete. For example, high-volume fly ash (HVFA) increased the concrete performance while using the OPC by-product, fly-ash, to create new concrete. Hence, we can be more environmentally friendly if we were able to replace ordinary Portland cement with geopolymer concrete.

1.2.4 - Davidovits' Recipe

Davidovits detailed his geopolymer concrete mixing formula for reproducing the Ancient Egyptian concrete based on his study in "They Built the Pyramids". The following is his recipe:

Prepare nummulitic limestone outcrop (fossil shells), naturally friable, from
 Tracy-le-Val south of Saint-Quentin (France). It resembles the limestone used for Pyramids of
 Giza but does not contain kaolinitic clay, which has to be added.

2) Pour 160 kg of kaolinitic clay, followed by 60 kg of sodium carbonate (natron) and 80 kg of slaked lime (hydrated lime), into 2 kiloliters of water in a pool.

3) Mix the geological glue from step 2 with 4500 kg of limestone from step 1 using a simple wooden paddle.

After drying, the final mixture contained between 18-20% weight water.



Figure 6: Pyramid of Davidovits' mixture from Davidovits' "They Built the Pyramids"

The purpose of this test was to demonstrate that the type of limestone used in step 1 was perfect for re-agglomeration. "We have disaggregated this soft material with water, then mixed the muddy limestone and its fossil shells with kaolin clay, and a simple geopolymeric binder. Then, the limestone mud was packed into the mould (a pyramid shape!). This re-agglomerated limestone, bonded by a geochemical reaction, thus hardened into a resistant block, much harder than the original material. We have strengthened the stone and made it more resistant to pollution, acid rain, and freezing." [Davidovits, 2008]

Chapter 2 - Methodology

2.1 - Geopolymer Concrete Reproduction

The following section describes the methodology used to reproduce Davidovits' recipe described in the previous section [Section 1.2.4].

2.1.1 - Materials

The reproduction of concrete is initiated by procuring materials of geopolymer concrete. Specifically, the necessary materials include lime (CaO), sodium carbonate (Na2CO3), kaolinite (Al2O3 2SiO2·2H2O), limestone (CaCO3) and polycarboxylate superplasticizer.

Each material was procured from the following companies:

- 1. Quicklime: CaO stones from Admir Masic's lab
- Sodium Carbonate: Sodium carbonate anhydrous from VWR Chemicals [Lot number: 19F1056472]
- 3. Kaolinite: Kaolin powdered kg from Ward's Science [Lot number: 438590]
- 4. Limestone: 100% Natural Pulverized Limestone (5 pounds) from Amazon

5. Polycarboxylate superplasticizer: ADVA 198 Pail from GCP Applied Technologies Inc. However, we were lacking sodium carbonate (Na2CO3) for our initial (6) tests and replaced it with sodium chloride (NaCl). The sodium chloride was from Macron Fine Chemicals [batch number 0000118188].

2.1.2 - Experimental setup

Lime can be prepared from quicklime which is a powder form of CaO. To increase reactivity, we re-calcilined the quicklime by heating it in an oven at 1100 degrees celsius for 8 hours and kept it at 100 degrees celsius until the day before application.

After the materials were measured out in correct quantities detailed in Section 2.1.3, the materials were added in the following order [as seen in Figure 7 below]:

- 1. Water
- 2. Kaolinite
- 3. Sodium Carbonate (Sodium Chloride for the first 6 batches)
- 4. Lime
- 5. Limestone
- 6. [Optional] Superplasticizer



Figure 7: Each material is measured and laid out before mixing

Each batch was mixed in a hobart mixer [Figure 8] for 3 minutes unless superplasticizers were required. If required, superplasticizers were added by trial after 1 minute and mixed for an additional 3 minutes, resulting in 4 minutes of total mixing.



Figure 8: Hobart Mixer we used to mix the materials

Each batch of concrete is casted in four cylinders and a prism mold. As shown in Figure 9 and 10, the cylinder is 2.5 cm by 10 cm, and the prism mold is 2" by 2" by 8". The small volume of each mold allows us to create multiple samples at once to test with precision and accuracy. As

seen in Figure 11, the prism mold was personally designed and crafted by Stephen Rudolph after we gave rough guidelines on how the mold should look like.



Figure 9: Measurements of the cylinder mold



Figure 10: Prism Mold







Figure 11: Concrete Prism Mold plan courtesy of Stephen Rudolph

The typical curing time for geopolymer concrete is 24 hours at room temperature of $22 \pm 2^{\circ}$ C. The mixture is usually autoclaved at 180-190 °C (steam pressure 175 psi) within an hour of mixing and maintained at this temperature for 4-8 hours since heat improves the binding strength of geopolymer. The mixture is then cooled and transferred to a standard curing room until testing.

Another curing method is soaking the mixture in water for 28 days to induce an exothermic reaction that increases the binding strength. However, we ruled out this method due to the limitation of time.

Alternatively, we considered using steam curing or oven curing. Steam curing heats the mixture with the temperature increasing by 60°C/hour from 0°C to 90°C (total of 1.5 hours). The temperature is then maintained at 90°C for another 4 hours. Afterwards, the mixture is cooled to room temperature and subsequently cured in a standard curing room until testing.

On the other hand, the oven curing heats the mixture in an oven up to 600°C over 4 hours. The mixture is then cooled down to room temperature in the oven overnight and subsequently transferred to a standard curing room until testing. 600°C is claimed to be the ideal temperature to increase the compression strength of the mixture. However, the reaction also calls for pressure controls which the autoclaves in our lab were not equipped with.

Additionally, concrete must be stiff enough to be hard pressed and released with the air compressor in order to be casted in the cylindrical mold. Due to the limitation of time, we sped up the curing process by putting the cylinders in the oven at 60°C for 24 hours rather than letting it harden at room temperature before demolding.

The following is the curing method we finally used: Demold the four samples from each batch after heating them at 60°C for 24 hours. Place each sample in the furnace [as seen in Figure 12] at 600°C for 4 hours. Finally, allow the sample to cool in the furnace overnight. We also left one unheated sample per batch and cured it at the lab room temperature of $22 \pm 2^{\circ}$ C until demolding and testing.



Figure 12: Furnace used to bake quicklime at 1100°C and oven curing of samples at 600°C for 4 hours

2.1.3 - Batches

The following charts describe the composition of materials used in each batch of concrete we created.

Batch #	Water (g)	Lime (g)	Kaolinite (g)	Sodium Chloride (g)	Limestone (g)	Superplasticizer (g)	Water/Cement ratio (%)
1	1000	40	80	30	2250	0	41.67
2	500	40	80	30	2250	0	20.83
3	500	40	80	30	2250	0	20.83
4	500	40	80	30	2250	2.7	20.95
5	400	40	80	30	2250	21.5	17.56

6	300	40	80	30	2250	97.4	16.56

Batch #	Wate r (g)	Lime (g)	Kaolinite (g)	Sodium Carbonate (g)	Limestone (g)	Superplasticizer (g)	Water/ Cement ratio (%)	Temp (Celsius)	Density (g/cm ³)
7	500	40	80	30	2250	23.8	21.83	26.1	6.7344
8	400	40	80	30	2250	89.9	20.41	26.6	6.8768
9	300	40	80	30	2250	181.2	20.05	25.8*	6.896
10	300	80	80	30	2250	221.6	21.38	27.4	6.8816
11	400	80	80	60	2250	200.9	24.32	31.6	6.6864
12	400	40	80	60	2250	111.5	21.05	27.0	6.6688

*Note: There was a mishap during mixing and had to stop for 40 seconds to adjust mixer

For batches 1 through 6, we used sodium chloride instead of sodium carbonate by mistake. Out of curiosity, we let the batches sit for a few days to cure. However, the visible weakness of the cured batches prevented us from running further analysis, and we moved on to mixing with the correct chemical, sodium carbonate.

Superplasticizers were added as needed depending on how well the materials were binding after a minute of mixing. If the materials were not binding well, we slowly added superplasticizer by trial and simply denoted the total amount used in the tables. The water to cement ratio was calculated by dividing the total amount of water and superplasticizer by the rest of the materials. Temperature denotes the temperature of the mix after settling it for one minute. Density is calculated by dividing the mass of concrete mixture by the volume of the mold.

2.2 - Analysis Method

We varied the water to cement ratio for batch 7 to 9 while we focused on varying the amount of lime and sodium carbonate for batch 10 to 12. Our goal was to observe the strength and flexure at a macro and micro scale after varying the amount of materials in each mixture.

On the macro scale, each sample would be crushed to test the compressive strength and subjected to the three point bending test to observe the flexural bending of the concrete. On the micro scale, each sample would be subjected to scratching and indentation to observe the creep and flexural toughness. The analysis between two scopes would allow us to verify the mechanical properties of the concrete.



Figure 13: The machine on the left is a three point bending machine and on the right is a compression machine which we were not able to apply

2.2.1 - Scratching and Indentation

After the compression test, we took pieces of the crushed concrete and put them into resin in order to bind the loose pieces. After a thorough polishing of each sample, we put them in the scratching and indentation machine for analysis.

We can customize the number of scratches per row and column as well as the length and speed of each scratch for the scratch test. We also have the option of pre-scanning to calibrate the final results in case the original sample is not truly flat and/or smooth. This is necessary since even a thorough polishing does not guarantee the surface to be truly flat and smooth. Although the pre-scan improves accuracy of the scratch test, it almost doubles the run time for each scratch.

Indentation has similar yet stricter requirements. The sensitive balance embedded in the diamond tip will cause the indenter to be stuck on the first run if the surface is not truly smooth.



Figure 14: Image on the left details a scratch test while the right details an indentation

Chapter 3 - Results

3.1 - Analysis of Professor Hobbs' Sample

MIT Professor Linn W. Hobbs provided us with a cylinder of geopolymer concrete made in 2008 which we compressed until failure and analyzed using scratching and indentation. A quick visual analysis showed the sample was heterogeneous with clear striated layers of materials. Without more information from Professor Hobbs, we were unable to verify if the layering was produced on purpose or as a result of aging.





Figure 15: Professor Linn Hobbs' Cylinder right after demolding



Figure 16: Professor Linn Hobbs' samples embedded in resin to be polished for testing

We were provided the mixing formula used for Professor Hobbs' sample by Linda Seymour who took Professor Hobbs' concrete mixing class. Figure 17 shows Linda's notes on the geopolymer sample created in the aforementioned class.

Mixing "/ Professor Hobbs Binder Mix: 258.2 g Kaolin 106 g Na (D3 56.1 g CaO 18g H2 O (Will likely need more) yields 338.29 Feldspathid 100 mil playground Sand = 131.19 7g binder = 5.1% (131+7=138g total) 14 Kecipe 64.59 KaOlin (ISOmL) 26.59 NaCo (JomL) 14.09 CaO (IOmL) *4.59 Hz O *additional ~ 70.09 of water added } 74.59 of Hz O *additional ~ 70.09 of water added } total 90% Sand 10% binder \$ 0.9 Ws + 0.1 Wander = Wtot 6455 + 26.5 + 14 + 74.5 = 179.6 g total produced 29.6g in distes 150 g remain 91.5g binder (not including excess water $\begin{array}{c} (y) \text{inder} = 205.8 \text{ cm}^3 & 205.8 \text{ cm}^3 \\ \text{sand} = 131 \text{ g} / \frac{1003}{3} \\ \hline 1 \text{ glinder} & 242.6 \text{ g} \text{ sand} \\ \hline \frac{70}{179.6} = \frac{27.9}{5 \text{ fraction}} = 0.39 \\ \hline 160 \text{ g} \text{ mat'}(R = > 58.5 \text{ g} \text{ water} \end{array}$

27 g binder (179.69 mix 109.69 binder) = 44.29 mix Final mto: 44.2 g mix ZI cylinder 242.8 g send ZI cylinder measured remaining mix = 144.59 mix 3.24 cylinders (144.59 mix 44.29 Giunders 793.1 g Sand regid + 80 g H20 for workability

Figure 17: Linda Seymore's notes from Professor Hobbs' class

3.1.1 - Scratching and Indentation of Professor Hobbs' Sample

Scratch test discovers the sample's fracture toughness while indentation shows elastic modulus and creep. While investigating how to operate the scratcher and indenter, we created multiple sample pieces from Professor Hobbs' sample. From indentation, we discovered that Professor Hobbs' sample had an elastic modulus of 17 GPa. Note that a traditional piece of concrete has an elastic modulus of 40 GPa.

For each scratch test, we increased the vertical force at a constant rate from 0.5 N to 100 N with a loading rate of 99.5 N/min. I conducted a total of 5 scratch tests in an effort to get

acquainted with the equipement. Using the following formula of fracture toughness from Hoover and Ulm, $\frac{2pA}{R^3} = \frac{F_T^2}{R^3 K_C^2} = \alpha (\frac{d}{R})^3 + \delta (\frac{d}{R})^2 + \gamma (\frac{d}{R})$, I determined the coefficients alpha, delta and gamma to be 4.85097930068111e-05, -1.22613277359900e-05 and 5.75255011173120e-07 respectively. After several scratch tests on the Professor Hobbs' sample shown in Figure 18, we determined the fracture toughness to be 0.8MPa \sqrt{m} . Also note that the results in Figure 19 show some unexpected peaks from x=0 to x=0.2. Those are due to the scratches unexpectedly tearing around the surrounding surface which was evident under the microscope.



Figure 18: Best fit line out of the 5 scratch tests performed on Professor Hobbs' sample



Figure 19: Fracture Toughness of Professor Hobbs' sample

3.2 - Geopolymer Concrete Reproduction

3.2.1 - Water to Cement Ratio

Batches 7 to 9 were made with varied water input. Although water is a necessary active material in concrete, it also serves as a weakening agent if too much is applied. The superplasticizer also acted as additional water in our case since we did not use any cement in our mixture and produced a clay-like substance. There are no chemical or molecular interactions between the superplasticizers and the mixture due to the lack of cement, therefore the amount of

superplasticizers simply translates to water. All of our sodium carbonate batches had around 20% water to cement ratio, as shown in the batches table [Section 2.1.3].



Figure 20: From left to right, it is batch 7 [500g water], 8 [400g water] and 9 [300g water]. These samples are cured at room temperature for at least 72 hours.



Figure 21: Left is the first look into the furnace after heating. Right is a close up of the 300g water cylinder which held up the best, but even then the bottom is cracked and discontinued from the rest of the cylinder



Figure 22: From left to right, Batch 8 [400g water], Batch 9 [300g water], Batch 7 [500g water]

3.2.2 - Lime (CaO) and Sodium Carbonate (Na2CO3) Content

Sodium carbonate released significant heat when mixed into water and kaolinite. When the baked lime was added, there were visible bubbles, but no extreme visual or temperature change. Since lime is supposed to be extremely reactive in water, we were expecting more extreme exothermic reactions. Batch 11 doubled sodium carbonate and lime which released a significant amount of gas with strong odor that evaporated shortly after. Batch 11 also slightly bubbled after the lime input.

3.2.3 - Failed Sodium Chloride batches

The batches with sodium chloride (Batch 1 through 6) resulted in weak samples that did not harden at room temperature over the span of two weeks. Although we could not conduct further analysis due to the malleability of samples, we learned that 1000 grams of water is too much for any batch, which led us to start at 500 grams of water for the batches with sodium carbonate (Batch 7 through 12). We also learned to keep the lime as hot as possible before pouring it into the mix for stronger reactions. This is because the reactivity of lime decreases when the lime is exposed to moistures in the air and cooled. Lime pulls water molecules from the air and bonds with them, which decreases their reactivity in contact with water.

Chapter 4 - Conclusion

Unfortunately, we were unable to perform scratch and indentation tests on the cylinder and prism molds due to the COVID-19 quarantine. However, this research is a great foundation for future studies on geopolymer concrete.

Batches 7 through 9 mainly experimented with varying the amount of water in the mixture. Since the mixture containing less water became more granulated, we increased the amount of superplasticizers proportionally in an attempt to achieve the desired consistency. During this process, we discovered that superplasticizers behaved just like water because it did not have cement molecules to bind with. Therefore, we concluded water was not a major factor and used 400 grams of water for batches 10 through 12.

We doubled the amount of lime in batch 10 from batch 9 in hopes of a more exothermic reaction between the lime and water; however, batch 10 did not perform differently from the previous batches. Batch 11, which doubled the amount of lime and sodium carbonate from batch 8, visually had the optimum texture, the stickiest and most fluid out of all the batches. Batch 12, which doubled the amount of sodium carbonate from batch 8, had a stickier texture than batches 7 through 10, but not as much as batch 11. Batch 12 also had visible particles of lime after mixing and a firmer texture than batch 11 after settling from the vibration table. In summary, increasing lime and sodium carbonate proportionally to the rest of the materials resulted in the best texture.

Additionally, each batch formula makes just enough concrete to fill 4 cylinders and 1 prism mold. In order to experiment with various curing methods on the same batch of concrete,

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multiple prism mold samples are needed. We suggest proportionally increasing the amount of materials in our formula to increase the total volume of concrete while keeping the ratio of materials.

Geopolymers have the potential to replace cement in ordinary Portland cement concrete which has been used for the majority of construction in the past decade. Since cement is the main contributor of carbon emissions from ordinary Portland cement concrete, incorporating geopolymers and eliminating the use of cement will significantly decrease carbon emissions from concrete productions. Learning from the ancient methods could save our future climate.

4.1 - Future Work

The next step would be to test the pre-cured samples for the mechanical properties mentioned in Section 2.1.4. On a micro scale, we can conduct scratch tests to measure the fracture toughness as well as indentations to measure the creep and elastic modulus. On a macro scale, the strength and flexural bending can be found by compression tests and three point bending respectively. Additionally, we can measure shrink/expansion of the sample after curing, the slump of the fresh sample, and the pH difference before and after adding superplasticizers to the mixture.

We could also experiment with further varying the ratio of materials, especially the lime and sodium carbonate, since adjusting the ratio of these two materials significantly altered the physical characteristics of mixtures during our iterations.

Another possibility is to explore other curing methods. We were unable to obtain an autoclave to heat the samples in a controlled temperature and pressure setting. We suggest

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exploring the effects on concrete properties by curing methods such as autoclave, exposure to room temperature air, oven firing, and water which could cause additional reactions between the lime and water.

Chapter 5 - Contributions

I worked closely with Ahmed Omran, a visiting scholar, and under supervision of Professor Admir Masic. Ahmed and I prepared and analyzed the mixtures in a joint effort. For analysis, we utilized Professor Franz-Josef Ulm's lab for scratching and indentation of our concrete. Professor Linn W. Hobbs of Materials Science and Engineering and Nuclear Science and Engineering provided us with a cylinder of geopolymer concrete which initiated our interest into the subject. I would also like to thank Stephen W. Rudolph for providing access to the lab and assisting during experimentation.

Chapter 6 - References

- 1. "Concrete Technology." *Strength of Cement*, Building Research Institute, 2020, buildingresearch.com.np/services/ct/ct2.php.
- 2. Davidovits, Joseph. "They Built the Pyramids". Geopolymer Institute, 2008, pp. 71-76.
- Hardjito, D., and Rangan, B.V. "Development and Properties of Low-Calcium Fly Ash-based Geopolymer Concrete." Curtin University of Technology, 2005, https://www.researchgate.net/publication/228794879_Development_and_Properties_of_ Low-calcium Fly Ash Based Geopolymer Concrete.
- 4. Hardjito, Djwantoro, et al. "On the Development of Fly Ash-Based Geopolymer Concrete." *Nikanlink.com*, ACI Materials, 2004, nikanlink.com/Content/User%20Files/ رُوْيِلِيمر.pdf.
- Hoover, Christian, and Ulm Franz-Josef. "Experimental chemo-mechanics of early-age fracture properties of cement paste." ScienceDirect, Cement and Concrete Research, 2015,

https://reader.elsevier.com/reader/sd/pii/S0008884615001106?token=02A7F645C1880C F61DE4E913C0403E06F3342815D3EF70594D3DD26C3131B698831FC502416FF7DE E95587BC13CA8849.

- L.Y. Kong, Daniel, and Jay G.Sanjayanb. "Effect of Elevated Temperatures on Geopolymer Paste, Mortar and Concrete." *Cement and Concrete Research*, Elsevier, 17 Nov. 2009, www.sciencedirect.com/science/article/pii/S0008884609003007.
- National Ready Mixed Concrete Association (NRMCA). "Concrete CO2 Fact Sheet", June 2008,

http://www.nrmca.org/greenconcrete/concrete%20co2%20fact%20sheet%20june%20200 8.pdf?fbclid=IwAR1_UQnCNR4KWPh_QEG1mqy15adcDQaRtZ2MsOLHz5gIlw9Jz3X xNdLwU9g

- Rangan, B.V. "Fly Ash-based Geopolymer Concrete". Curtin University of Technology, 2008, https://www.researchgate.net/publication/230717147_Fly_Ash-Based_Geopolymer _Concrete.
- 9. Yang, Catherine, et al. "Geopolymers: Building Material of the Past and Sustainability for the Future." 2019.