

Prediction of the Compressive Strength from Resonant Frequency for Low-Calcium Fly
Ash-Based Geopolymer Concrete

by

Chen Yang
MEng, University of Victoria, 2017

A Thesis Submitted in Partial Fulfillment
of the Requirements for the Degree of

MASTER OF ENGINEERING

in the Department of Mechanical Engineering

© Chen Yang, 2017
University of Victoria

All rights reserved. This thesis may not be reproduced in whole or in part, by photocopy
or other means, without the permission of the author.

Supervisory Committee

Prediction of the Compressive Strength from Resonant Frequency for Low-Calcium Fly
Ash-Based Geopolymer Concrete

by

Chen Yang
MEng, University of Victoria, 2017

Supervisory Committee

Rishi Gupta, Department of Civil Engineering
Supervisor

Mohsen Akbari, Department of Mechanical Engineering
Departmental Member

Abstract

Due to its usage in large quantity, the concrete industry contributes to 5% of the annual anthropogenic carbon production. The main reason is the production of traditional concrete binder materials, Ordinary Portland Cement (OPC), involves a high carbon emission. Therefore, finding a greener binder material has been considered as the most effective way to reduce the carbon footprint for the concrete industry. In recent years, fly ash, a waste material from power plants, has gained public attention owing to its similar binding properties as OPC. Therefore, this study focuses on using fly ash to produce a cement-free geopolymer concrete. Instead of using sodium-based activator, this study adopts potassium-based activator that consists of hydroxide and silicate, due to its better contribution on workability and strength. Under the heat curing condition, the effect of the constituent of activator was investigated in this study, by ranging the concentration of hydroxide (10M, 12M and 14M), the ratio of silicate to hydroxide (2.0, 2.5 and 3.0). The results have shown the compressive strength increases with the increasing hydroxide concentration and the decreasing silicate to hydroxide ratio. Besides, attempts were made to propose a non-destructive methodology for the prediction of compressive strength from resonant frequency. The dynamic elastic modulus was determined from resonant frequency test, following by compression test. The predictive equation was proposed by conducting a multiple regression analysis between dynamic elastic modulus and compressive strength. Given the relationship between resonant frequency and dynamic elastic modulus from ASTM Standard Code, the proposed equation was successfully correlated to resonant frequency. The accuracy of proposed equation was then evaluated with experimental data and validated with previous work from another researcher.

Supervisory Committee

Rishi Gupta, Department of Civil Engineering
Supervisor

Mohsen Akbari, Department of Mechanical Engineering
Departmental Member

Table of Contents

Supervisory Committee	2
Abstract	3
Table of Contents	4
List of Tables	5
List of Figures	6
Acknowledgments	7
Dedication	8
Chapter 1 Introduction	9
Chapter 2 Literature Review	10
2.1 Geopolymer Chemistry	10
2.2 Geopolymer Concrete Constituents	10
2.3.1 Aggregates	10
2.3.2 Binder Materials	11
2.3.3 Alkaline Activator	11
2.4 Factors Affecting Geopolymer Compressive Strength	12
2.4.1 Concentration of Hydroxide & Ratio of Silicate to Hydroxide	12
2.4.2 Ratio of Alkaline Liquid to Fly Ash	12
2.4.3 Water Content	12
2.4.4 Curing Temperature and Curing Time	13
2.5 Resonant Frequency Testing Method	13
2.6 Relationship between Compressive Strength and Elasticity	14
Chapter 3 Experimental Investigation	17
3.1 Materials and Preparation Work	17
3.2 Mixture Proportions Development	17
3.3 Mixing and Curing	20
3.3 Testing Investigations	20
3.3.1 Sample Preparations	20
3.3.2 Resonant Frequency Test	21
3.3.3 Compression Test	22
Chapter 4 Results and Discussion	23
4.1 Factors Affecting the Compressive Strength of Geopolymer Concrete	23
4.2 Correlation Between Resonant Frequency and Compressive Strength	24
4.2.1 Resonant Frequency of Geopolymer Concrete	24
4.2.2 Elastic Modulus of Geopolymer Concrete	26
4.2.3 Model Development	27
4.2.4 Model Validation	28
4.2.5 Applicability and Limitations	29
Chapter 5 Conclusions	30
Bibliography	31
Appendix A Materials Information	34
Fly Ash	34
Aggregates	35
Potassium Silicate	38
Potassium Hydroxide	40

List of Tables

Table 1 Relationships between Static Elasticity and Strength.....	14
Table 2 Mixture Proportions (kg/m ³).....	18
Table 3 Constituents for KOH in Different Concentration.....	19
Table 4 Compression Test Results.....	23
Table 5 Results of Resonant Frequency Test.....	25

List of Figures

Figure 1 Atomic Structure of Geopolymer	10
Figure 2 Schematic of Longitudinal Resonant Frequency Test.....	14
Figure 3 Static Elasticity and Compressive Strength for Geopolymer Concrete.....	15
Figure 4 Dynamic Elastic Modulus and Compressive Strength for OPC.....	15
Figure 5 Dry Mix of Geopolymer Concrete	20
Figure 6 Geopolymer Concrete Specimen.....	21
Figure 7 Experimental Setup for Resonant Frequency Test.....	21
Figure 8 Experimental Setup for Compression Test.....	22
Figure 9 Compressive Strength Development	24
Figure 10 Compressive Strength vs. Resonant Frequency	25
Figure 11 Compressive Strength vs. Dynamic Elasticity	26
Figure 12 Regression Analysis in Matlab.....	27
Figure 13 Compressive Strength Predictions.....	29

Acknowledgments

I am grateful to my supervisor, Mr. Rishi Gupta who provided guidance and support to me over my study at the University of Victoria, so make it possible for me to finish my degree smoothly. Thanks for your kind advice regarding the topic of my research. I am proud of being one of your students.

I would thank Mr. Peyman Azarsa and Ms. Fernanda Belforti for giving me generous help for my manual mixing and casting work and your contribution to my data analysis part, this study could have not been so successful without their contribution.

I am also thankful for Mr. Adham El-newihy for your guidance on the resonant frequency test which is not possible to discover by my own. Also, thanks for encouraging me with your motivating words.

At last, I would like to express my gratitude to all professors, co-workers and friends who provided guidance and encouragement.

Dedication

I dedicate this thesis to my parents, who have taught the way of my life, and offered unwavering support and unconditional love. It is not possible for me to complete my Master's degree overseas without their mental and financial support. Words can never be enough to express my gratitude.

Chapter 1 Introduction

Concrete production contributes to 5% of the annual anthropogenic carbon production due to its usage in large quantities [1]. According to the Cement Association of Canada [2], five billion cubic yards of concrete is produced annually, which is roughly equivalent of one ton concrete consumed by every individual on the earth each year. Additionally, the production of binder material of traditional concrete, Ordinary Portland Cement (OPC), involves high carbon emission. It is estimated that each ton of modern OPC releases about 800kg of carbon dioxide (CO₂) [3]. As the increasing global demand, by 2050, the consumption of concrete is predicted to reach four times of 1990 level [1]. Unquestionably, the greatest challenge that concrete industry faces in future is the CO₂ emission reduction.

Driven by the rising concern regarding the environment issues, it is imperative for the concrete industry to take actions to reduce carbon emission. In many investigations, using supplementary cementing material, such as fly ash, as an alternative to OPC is a proven strategy to reduce carbon emission for concrete industry. Most recently, some researchers have invented a new type of concrete that uses fly ash straight as a binder material, which is known as geopolymer concrete [4]. Since the fly ash is a locally available waste material from power plant stations, the geopolymer concrete can take advantage of cheaper cost over OPC concrete, with similar mechanical properties and reduced CO₂ emission. These superior properties have already offered geopolymer materials a greater opportunity for construction application. The largest geopolymer concrete project in the world so far was unveiled in Toowoomba, Queensland in November 2014 [5], where an airport was built with 100,000 tons of geopolymer concrete, which made this project a significant milestone in engineering.

Even though there are many existing studies on geopolymer concrete, most of them only focused on the investigation of ideal mix design for the improvement of strength [4] [6]. As another aspect, understanding the relationship between strength and physical properties is also important for geopolymer concrete materials at the early stage of development, but there are very limited works addressing this aspect. Therefore, this study proposes a predictive relationship between compressive strength from resonant frequency using a non-destructive method.

This report begins with the literature review in Chapter 2 regarding the background information on geopolymer concrete, such as the chemistry and constituents of geopolymer concrete, as well as some essential factors that affect the strength of geopolymer concrete. Chapter 2 also reviews the principles and the current development of non-destructive testing methodology for concrete. Chapter 3 briefs the experimental investigations, which includes the raw materials information, mixture proportion development, and equipment setups. In Chapter 4, the test results were discussed and the process of equation modeling was described. At last, the accuracy of the proposed equation was evaluated with experimental data and validated with previous work from another researcher.

Chapter 2 Literature Review

2.1 Geopolymer Chemistry

The concept of geopolymer was first introduced by Joseph Davidovits in 1972, which is defined as the chains or networks of mineral molecules linked with repeating unit of (-Si-O-Al-O-) [7]. As shown in Figure 1, geopolymer skeleton consists of tetrahedral Al and Si atoms arranged in a highly cross-linked structure, with negative charges associated with tetrahedral AlO_4 groups that charge-balanced by non-framework sodium ions. These sodium atoms are bonded with some water molecules in the presence of moisture [8].

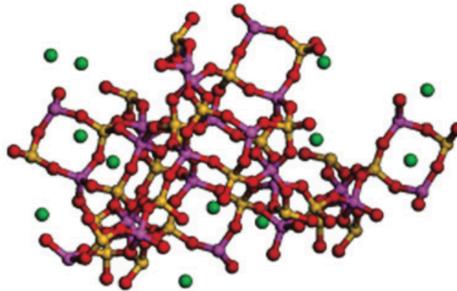


Figure 1 Atomic Structure of Geopolymer [8]

The term “polymer” was named to this material is owing to the polymerization process that takes place in Si-Al materials under alkaline condition. First, Under the alkaline condition, Si-O-Si and Al-O-Al bonds in aluminosilicate materials are broken first through an exothermic process, so the silicon and aluminum ions will be dissolved into solution, then these ions will transport and condense in poorly ordered orientation to form the monomers, followed by the polymerization of monomer into polymeric structure [7].

It should be noticed that these reaction steps overlap each other, which means the dissolution, ions transportation, poly-condensation and polymerization take place simultaneously. Besides, water in the admixture does not cause any chemical reaction, since the equivalent reacted water was eventually produced from the polymerization of the monomers. This means the production of geopolymer binder is water independent, but water is required to improve the workability of mixture in practical [7].

2.2 Geopolymer Concrete Constituents

There are three main components in geopolymer concretes, namely aggregates, binder materials, and alkaline activator.

2.3.1 Aggregates

Geopolymer concrete uses the same types of aggregate as cement-based concrete, and usually occupies 70~85% of total volume. Generally, crushed granite rock and natural river sand are used as coarse and fine aggregates respectively. The combination of fine to coarse aggregate in a proper ratio can give the concrete the least air void volume in the concrete. Benny Joseph studied the influence of aggregate content on the behavior of fly ash-based geopolymer concrete [9]. The geopolymer concrete with 70% total aggregate content by

volume, and the ratio of fine aggregate to total aggregate between 0.3~0.4 was found to present good mechanical properties in his investigation.

2.3.2 Binder Materials

Davidovits proposed that the aluminosilicate materials with the ability to react with alkaline liquid are the best candidates for geopolymer binder materials [7]. The most common material, fly ash, was reported to be very effective to dissolve, polymerize with alkaline activator, condense on particle surface and solidify to matrices ultimately with strength and stability [10]. At the same time, as the waste from thermal power plants, the low cost and local availability allow its potential application in concrete industry. In 2000, it was estimated that 600 million tons of fly ash was produced worldwide [11], where Canada produced 5 million tons of fly ash, but only about 5% was used as supplementary binder materials [12].

According to the content of calcium, fly ash is classified into two main types, namely ASTM Class C and Class F. Class F usually contains no more than 15% of lime in the composition, with a greater content of aluminosilicate materials and iron (more than 70%) than Class C. While, Class C fly ash generally comes out from power plants with higher lime content, which is more than 15% and up to 30% [13].

The content of calcium of fly ash plays an important role in polymerization process and the development of compressive strength. It was revealed by J. Temujin [14] that the presence of carbon compounds improves the mechanical properties of geopolymer concrete that is cured at ambient temperature. This is because calcium compound improves the dissolution of fly ash in the alkaline medium, leading to a faster polymerization. However, when fly ash-based geopolymer cured at elevated temperature, the presence of calcium compound in significant quantity will interfere with polymerization process. The presence of calcium possibly does not allow the formation of three-dimensional polymeric network, consequently insufficient polymeric network was formed, resulting a reduced final strength [14]. Since the elevated temperature curing regime will be used in this study, thus Class F fly ash is preferable than Class C as a binder material to make geopolymer concrete.

2.3.3 Alkaline Activator

Alkaline activator is another essential factor in the polymerization process, which is required for the dissolution of Si and Al from the source materials. The most commonly used alkaline activator is the combination of hydroxide and silicate from soluble alkali metals, either sodium (Na^+) or potassium (K^+). Most the studies have focused on the use of sodium-based activator to synthesize geopolymer concrete, owing to the smaller ionic size, which enables sodium based activator to be more active in the dissolution of source materials, therefore higher compressive strength. [15]

However, the most recent study [16] has found the similar mechanical properties can be obtained by using either of potassium or sodium-based alkaline activator [17]. It was revealed that the use of potassium-based activator has more benefits in solubility of

aluminosilicate materials, compatibility with other additives, and the workability of geopolymer concrete, despite the higher cost than sodium based activator [17].

The reason of using the combination of hydroxide and silicate is that the reactions occur at a higher rate than use hydroxide merely. Because the addition of silicate solution provides silicon atoms in aqueous form, which is helpful in activating the precursor of geopolymer materials, subsequently beneficial to the dissolution of source materials and the activation of polymerization process, leading to better ultimate mechanical properties [15] [16].

2.4 Factors Affecting Geopolymer Compressive Strength

The compressive strength of low-calcium fly ash-based geopolymer concrete is highly dependent on the factors that discussed in this part, therefore these factors must be considered carefully when designing mixing proportions.

2.4.1 Concentration of Hydroxide & Ratio of Silicate to Hydroxide

The concentration of hydroxide and the ratio of silicate to hydroxide have been identified as the key factors affecting the properties of the geopolymer. D. Hardjito et al. [10] have found the compressive strength increases significantly with the concentration of hydroxide and the ratio of silicate to sodium. The best 7-day compressive strength was obtained as 67 MPa, with the concentration hydroxide of 14M and the ratio of silicate to hydroxide of 2.5. Another study [19] revealed that the workability and setting time were reduced with the increase of concentration of hydroxide due to more entrapped air in concrete.

2.4.2 Ratio of Alkaline Liquid to Fly Ash

Xu et al [15] has suggested the ideal ratio of alkaline liquid to fly ash should be around 0.33 to allow the geopolymerization reaction to take place in real practice. Another researcher, Palomo [16] has researched the effect of liquid to fly ash ratio (0.25 and 0.3) using different alkaline activators. Even though the compressive strength was not investigated as directly relevant to liquid/fly ash ratio, the sodium based geopolymer concrete with the compressive strength as high as 67 MPa was obtained after 24 hours curing at 65 °C with the liquid/fly ash ratio of 0.3. As the similar curing condition will be applied to this study, therefore, the alkaline liquid to fly ash was remained at about 0.35 without variation.

2.4.3 Water Content

Unlike OPC, the water in low-calcium fly ash-based geopolymer does not cause a reaction, because the reacted water will be expelled from the final product eventually. Previous research by D. Hardjito et al. [10] has revealed that the water content in mix design plays an important role on the strength of geopolymer by affecting the workability, despite it is not a part of reaction. Water content is usually expressed by water-to-geopolymer solids ratio (w/s), which is equal to total mass of water in the mixture (the water in alkaline activator + the mass of extra added water) divided by the total mass of geopolymer solids, which includes the mass of fly ash, silicate solids and hydroxide solids. Like OPC concrete, an increase of w/s ratio reduces the compressive strength of concrete too. The accepted massive ratio of water to fly ash ranges between 0.17 up to 0.3.

2.4.4 Curing Temperature and Curing Time

The mechanical properties of geopolymer concrete is also highly dependent on curing temperature and curing time. Like all chemical reactions, the geopolymerization process will take place at a higher rate under an elevated temperature. Therefore, curing at a higher temperature, which is known as heat curing, can help geopolymer concrete develop early-age mechanical properties rapidly. Despite geopolymer with similar compressive strength can be produced by curing at ambient temperature, but a much longer curing time is required.

A higher temperature cannot always help building stronger geopolymer concrete. Pavel Rovnaník [20] observed that the bulk density of hardened concrete slightly decrease with rising temperature. This phenomenon was accounted by the formation of accumulative larger pore at elevated temperature, which will reduce the bulk density, and thus reduced final properties of geopolymer concrete [20]. The temperature between 60°C and 80 °C is considered as an acceptable range for heat curing, the geopolymer concrete that was cured at 60°C is regarded as the most preferable heat curing temperature for fly ash-based geopolymer concrete. [20] [21].

In addition, curing time is another factor that need to be considered to achieve the desired curing effect. Curing at elevated temperature for very short time does not help the development of strength, but prolonging the cure time over 24 hours can be a waste of energy. Because the fast strength development will only take place in the first 24 hour at elevated temperature, during which about 75% of target strength can be developed theoretically. The increase of strength will become moderate beyond 24-hour curing. [21]

Instead of curing at ambient temperature, the heat curing is adopted in this study curing regime to develop the strength of geopolymer concrete rapidly. The geopolymer concrete will be cured at 60°C for 24 hours, followed by ambient temperature curing at 30°C until testing.

2.5 Resonant Frequency Testing Method

The traditional destructive testing methods were found not feasible, as large number of samples and complex equipment are required for the determination of mechanical properties, the process of which is always time-consuming and costly. Most importantly, the destructive instinct of conventional tests makes it not possible to conduct multiple testing on the same sample.

The recently emerging non-destructive testing (NDT) technology has partially tackled this issue. NDT is defined the process of inspecting, testing, or evaluating materials, components or assemblies without destroying the serviceability of the part or system [22]. Instead of determining the mechanical properties by reaching to the failure mode directly, NDT attempts to predict the mechanical properties indirectly by exploring the indication of properties, or establishing the relation between mechanical and physical properties. [23]

For example, resonant frequency test is an indirect mean for the determination of elastic modulus. As the natural vibration frequency of a structural member is related to the elastic

modulus and density. Therefore, the elastic modulus can be determined by measuring either of longitudinal, transverse and torsional resonant frequency, with the measured mass and dimensions of a specimen.

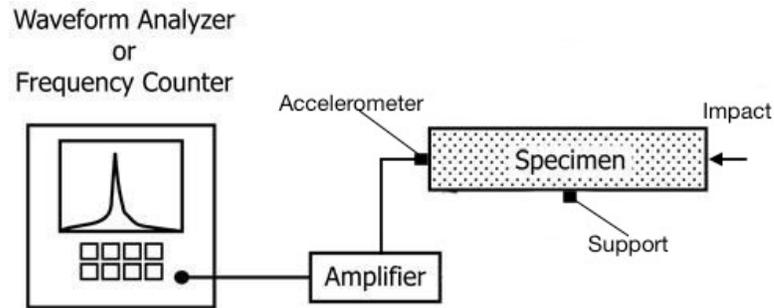


Figure 2 Schematic of Longitudinal Resonant Frequency Test [24]

ASTM C215-14 [24] illustrated the details of standard test method for fundamental longitudinal resonant frequency of concrete specimens using the impact resonance method, the experimental setup of which is shown in Figure 2. With a support placed under the middle of specimen and an accelerometer attached to the center of one end, the concrete specimen was perpendicularly struck by a lightweight rigid steel impactor to the center of the other end, the acceleration signal then was picked up by the accelerometer and recorded by data acquisition system. After the processing in the waveform analyzer, the fundamental longitudinal resonant frequency was output eventually. The dynamic modulus of elasticity (G_d) can be calculated by substituting the longitudinal frequency in Equation 1 [24]:

$$G_d = 5.093(L/d^2)Mn^2 \quad (1)$$

where: n : fundamental longitudinal frequency (Hz)
 L : length of concrete cylinder (m)
 d : diameter of cylinder (m)
 M : mass of cylinder (kg)

2.6 Relationship between Compressive Strength and Elasticity

Because the destructive mean of static elastic modulus determination gives more reliable results, most international codes only focus on the prediction of compressive strength (f'_c) from static elastic modulus (E_c). Table 1 summarized predictive relationships for OPC-based concrete that recommended by some international standard codes.

Table 1 Relationships between Static Elasticity and Strength

Code	Estimating Equation	Units
ACI 318-08 ^[25]	$E_c = 4700 f'_c{}^{0.5}$	E_c : MPa f'_c : MPa
ACI 363-92 ^[26]	$E_c = 3320 f'_c{}^{0.5} + 6900$	E_c : MPa f'_c : MPa
AS3600-05 ^[27]	$E_c = \rho^{1.5} (240 f'_c{}^{0.5} + 120)$	E_c : MPa f'_c : MPa ρ : kg/m ³
CSA A23.3-04 ^[28]	$E_c = 4500 f'_c{}^{0.5}$	E_c : MPa f'_c : MPa

As can be seen, all the equations indicate the elastic modulus tends to exhibit a positive linear relationship to the square root of the compressive strength. J. Wongpa et al. [29] also reported a similar trend for geopolymer concrete. As shown in Figure 3, the elastic modulus of geopolymer concrete is also linear to the square root of compressive strength but with a much lower static elastic modulus than that of OPC-based concrete, which means geopolymer concrete is not as brittle as OPC-based concrete.

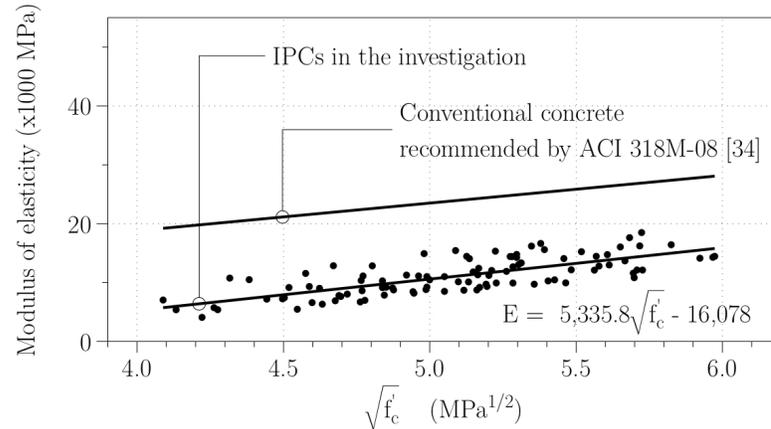


Figure 3 Static Elasticity and Compressive Strength for Geopolymer Concrete [29]

Owing to the non-destructive merit, some international code such as the British Code of Practice CP110:1972 [30] established a predictive relationship for compressive strength from dynamic elastic modulus using torsional resonant frequency for OPC-based concrete. The compressive strength was related to dynamic elastic modulus by Equation 2

$$G_d (GPa) = 22 + 2.8 f_c^{0.5} (MPa) \quad (2)$$

Another researcher Sharma et al. [30] proposed another empirical relationship between dynamic elastic modulus and the compressive strength for OPC-based concrete, which can be seen in Figure 4 [31]. This empirical equation has a prediction error $\pm 10\%$.

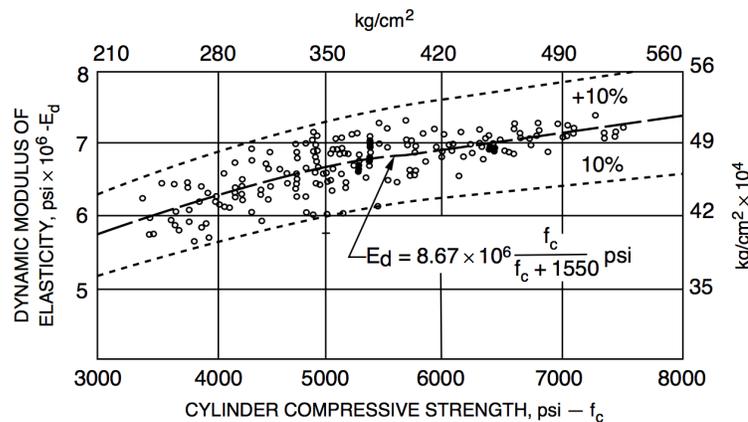


Figure 4 Dynamic Elastic Modulus and Compressive Strength for OPC [31]

Even though these empirical equations only hold for OPC-based concrete due to the lower elastic modulus that geopolymer concrete has, it suggests the possibility of prediction of the compressive strength from dynamic elasticity for geopolymer concrete by using NDT resonant frequency test.

Therefore, this project will establish a NDT predictive method for geopolymer concrete compressive strength. By varying the concentration of hydroxide and the ratio of silicate to hydroxide, totally nine different mixture proportions were developed, so that accuracy of the modeling equation can be ensured from data points in a wide range. The modeling equation was confirmed by conducting a regression analysis in Matlab first based on these data points, this was followed by a final validation by comparing it to results of another study.

Chapter 3 Experimental Investigation

3.1 Materials and Preparation Work

ASTM C618 Class F low-calcium fly ash was used as binder material, which was obtained from Centralia power plant Washington, USA with the calcium oxide contents of 14%.

Fine aggregates and coarse aggregates used were obtained from a quarry in British Columbia with relative dry density (SSD) of 2.671 and 2.713 respectively, and water absorption ratio of 0.79% and 0.69% respectively. The nominal size of coarse aggregate size is 12.5 mm, the sieve analysis of fine aggregates is given in Appendix A. All of them were prepared to be surface-saturated dry condition (SSD) before use.

The combination of potassium hydroxide (KOH) and potassium silicate (K_2SiO_3) were used as the alkaline activator. Since potassium hydroxide is more expensive than potassium silicate, therefore hydroxide and three different weight ratios of potassium silicate solution to potassium hydroxide solution (PS/PH Ratio) were selected, namely 2.0, 2.5 and 3.0.

The KOH pellets used in this study is produced by Sigma-Aldrich, with >85% purity in ACS grade. The KOH solution in three concentrations were used in this study, which were 10M, 12M and 14M. The KOH solution in these desired concentrations was prepared by dissolving measured KOH pellets in tap water one day in advance of use.

The soluble K_2SiO_3 in hydrous powder form (Model: AgSil 16H) was obtained from PQ Cooperation with the $SiO_2/K_2O=1.62$, and water weight percentage of 14.8%. The K_2SiO_3 solution in weight percentage of 56% is supposed to be used in the mix design. However, the attempts to prepare K_2SiO_3 solution by dissolving K_2SiO_3 powder into tap water or hydroxide solution were not successful. Undissolved K_2SiO_3 particles were observed to suspended in the solution and then turn into a gelatinous bulk at the bottom of the baker after mixing. To resolve this issue, K_2SiO_3 powder was first dry mixed with fly ash, sand and aggregate in advance of the addition of liquid. Then, the water required for potassium silicate solution was offset by adding extra calculated water.

In the beginning of the experiments, the geopolymer mortars from numerous mixtures in size of 50×50×50mm were tested before the final decision were made on the mixture to ensure the mechanical properties of the cylinder samples in the following step.

3.2 Mixture Proportions Development

Nine optimal mix designs shown in Table 2 were derived after the initial testing on mortars. The mixture proportions of fly ash, sand and aggregate were kept constant through all nine mix designs. The content of fly ash was kept 408 kg/m^3 , and the total alkaline activator content was held to 144 kg/m^3 , so the ratio of alkaline liquid to fly ash was remained to be 0.35 approximately. The total aggregate proportion, including sands was held to be 1795.2 kg/m^3 totally, which occupies around 74% of the total mass of the geopolymer concrete. The ratio of sand to total aggregate was kept at about 0.3 for the sake of minimizing the volume of air voids volume in the concrete.

Table 2 Mixture Proportions (kg/m³)

Mixture	Fly Ash	Sand	Aggregate	KOH Sol	K ₂ SiO ₃ Sol	K ₂ SiO ₃ Powder	Extra H ₂ O
10M2.0	408	530.4	1264.8	48	96	63.10	106.39
10M2.5	408	530.4	1264.8	41	103	67.70	110.21
10M3.0	408	530.4	1264.8	36	108	70.99	112.94
12M2.0	408	530.4	1264.8	48	96	63.10	109.93
12M2.5	408	530.4	1264.8	41	103	67.70	113.23
12M3.0	408	530.4	1264.8	36	108	70.99	115.59
14M2.0	408	530.4	1264.8	48	96	63.10	113.00
14M2.5	408	530.4	1264.8	41	103	67.70	115.85
14M3.0	408	530.4	1264.8	36	108	70.99	117.89

The concentration of KOH solution and the PS/PH Ratio are the only two varying parameters in the mix design. The KOH solution was prepared in three different concentrations: 10M, 12M and 14M, and each of them will be combined with K₂SiO₃ solution in three different PS/PH ratios, 2.0, 2.5 and 3.0 to make the alkaline activator. For the convenience of identity, the mix designs were named with the constituent of the alkaline activator that was used. For example, the mix design named with 14M3.0 means its alkaline activator consists of the KOH solution in 14M with the PS/PH ratio of 3.0, and so forth.

The ratio of water to geopolymer solid (w/s ratio) was selected to be 0.3 in all the nine mix designs, because relative higher w/s ratio can ensure a good workability of fresh concrete and longer setting time, along with a satisfactory strength. According to the definition of w/s ratio from last chapter, it can be calculated using following relationship:

$$w/s (wt) = \frac{\text{Extra Water Added} + \text{Water in PH Solution} + \text{Water in PS Solution}}{\text{Fly Ash} + \text{Solid in PH} + \text{Solid in PS}}$$

Therefore, given the constituent of alkaline activator and fly ash content, the water required to be added can be calculated by following procedures. Hereby, the mix design of 14M3.0 is taken as an example to illustrate the calculations.

a. Determination of the Water in PS Solution

The required K₂SiO₃ solution in 56% wt is 108 kg/m³, so the water in K₂SiO₃ solution in 14M3.0 is equal to 47.52 kg/m³.

b. Determination of the Water in KOH Solution

Table 3 summarizes the constituents of the KOH solution in different concentration. As can be seen, KOH solution in 14M contains 49.03% of water, and the required 14M KOH in mix design is 36 kg/m³, thus water in required KOH solution in 14M3.0 is equal to 17.65 kg/m³.

Table 3 Constituents for KOH in Different Concentration

Molarity (M)	Sol Density kg/m ³	Wt % of KOH	Wt % of H ₂ O	KOH Solid kg/m ³	Water kg/m ³
10	1397.59	40.39	59.61	564.42	833.17
12	1462.65	46.06	53.94	673.69	788.96
14	1523.01	50.97	49.03	776.30	746.70

c. Determination of Total Geopolymer Solid

The total mass of geopolymer solids includes the mass of fly ash, the solvents in KOH and K₂SiO₃ solutions. The mass of fly ash was given as 408 kg/m³. Similarly, the mass of KOH was determined as 18.35 kg/m³, and the mass of K₂SiO₃ was calculated from weight percentage as 108 kg/m³ × 0.56 = 60.48 kg/m³. Therefore, total geopolymer solid was equal to 408 + 18.35 + 60.48 = 486.83 kg/m³.

d. Determination of Total Water Required

The required water to maintain the w/s ratio at 0.3 was determined as (486.83 kg/m³ × 0.3) – (17.65 kg/m³ + 47.52 kg/m³) = 80.88 kg/m³.

As mentioned in the previous part, instead of using K₂SiO₃ solution in 56% wt, the K₂SiO₃ in hydrous powder form containing 14.8% wt of water was used in the real mixture. Therefore, the water required to prepare silicate solution from hydrous powder should be added as the water offset. For the mix design 14M3.0, 108 kg/m³ of K₂SiO₃ solution in 56% is required, which means 60.48 kg/m³ K₂SiO₃ in 100% purity and 47.52 kg/m³ water is needed. Thus, 60.48 kg/m³ / (100% – 14.8%) = 70.99 kg/m³ K₂SiO₃ in hydrous powder is needed, and 47.52 kg/m³ – (70.99 kg/m³ × 14.8%) = 37.01 kg/m³ extra water is required to compensate the water hydrous powder. Therefore, 80.88 kg/m³ + 37.01 kg/m³ = 117.89 kg/m³ water is totally required for the mix.

The total required offset water for the rest of the mixture designs was calculated by following these similar calculation procedures.

3.3 Mixing and Curing

The preparation of fly ash-based geopolymer concrete is similar to that of cement-based concrete. But the preparation of geopolymer was conducted in a wheelbarrow by hand-mixing owing to the fast hardening time of these mix designs. As shown in Figure 5, all the measured fly ash, sand and aggregates, along with potassium silicate powder was first dry mixed manually for three minutes. Then, the prepared mixture was wet mixed for another three minutes using scoops after the addition of potassium hydroxide solution and extra water. This gave the fresh



Figure 5 Dry Mix of Geopolymer Concrete

geopolymer concrete a thoroughly homogeneous mix before the casting. Then, the unhardened geopolymer concrete was cast into six cylinders for each mix design in the size of 100mm × 200mm, followed by the consolidation on vibration table for two minutes to avoid excess of air voids accumulating inside of the fresh concrete. At very last step, the excessive fresh concrete was struck off using wooden float to prepare an even and flat finish surface. The geopolymer concrete cylinders were placed indoor with controlled relative humidity of approximately 70% for 24-hour resting period.

Once the geopolymer cylinders were demoulded after resting period, they were immediately placed into air-tight buckets that were filled with tap water. These cylinders in the buckets were then placed into a hot-oven at 60°C for heat curing for 24 hours. After this, the concrete cylinders were transferred into a water tank for ambient temperature curing at 25°C until testing. The reason for using water-immersed curing method is to prevent the strength and degradation of dynamic elastic modulus that is caused by moisture loss, especially over the heat curing period.

3.3 Testing Investigations

3.3.1 Sample Preparations

The surface of the cylinders was ground immediately after they were taken out from the water tank in a grinding machine. This provided the uniformly distributed load for compression test and a smooth surface for resonant frequency test. The test specimens were rested in the air at ambient temperature until SSD condition was achieved. The aim of this is to evaporate excessive water from the concrete so that interference of the moisture on the resonant frequency can be excluded.

Figure 6 which shows an untested geopolymer concrete cylinder with saturated dry surface, the brownish color is a feature that distinguishes fly-ash based geopolymer concrete from OPC-based concrete.

3.3.2 Resonant Frequency Test

The resonant frequency of geopolymer cylinder was measured using impact resonance method, and the testing procedures were conducted as the instructions in ASTM C215-14 standards [23]. The equipment setup is shown in Figure 7. The measurement system consists of an accelerometer, a power supply, a DAQ system and a PC with Labview installed working as a waveform analyzer. The cylindrical specimen was supported by a rubber bar underneath in the middle. The accelerometer manufactured by the PCB Piezotronics Inc (Model #: 352C33)

was attached to the center of one end, it has a sensitivity of $10.2 \text{ mV}/(\text{m}/\text{s}^2)$ and measurement frequency ranging from 0.5Hz to 10000Hz . A visible controlling platform was built in Labview software so that the sampling rate and points of data can be configured and the DAQ can be controlled. In the experiment, the sample rate and the sample size of the DAQ system were customized as 20000Hz and 2048 points respectively.

A ball-ended impactor that was made from rigid steel was used to strike the cylinder perpendicularly to the approximate center of the end surface. Simultaneously, the DAQ was triggered and the acceleration data was recorded after the detection of the first acceleration signal change. Then, repeated at least three times to until the deviation of the resonant frequency is no more than 10% from the average value. After this, the data from frequency domain was exported to a spreadsheet for the determination of resonant frequency by finding the frequency with maximum amplitude.

The length and the mass of the cylinder specimen were recorded for the calculation of dynamic elastic modulus before the compression test.



Figure 6 Geopolymer Concrete Specimen

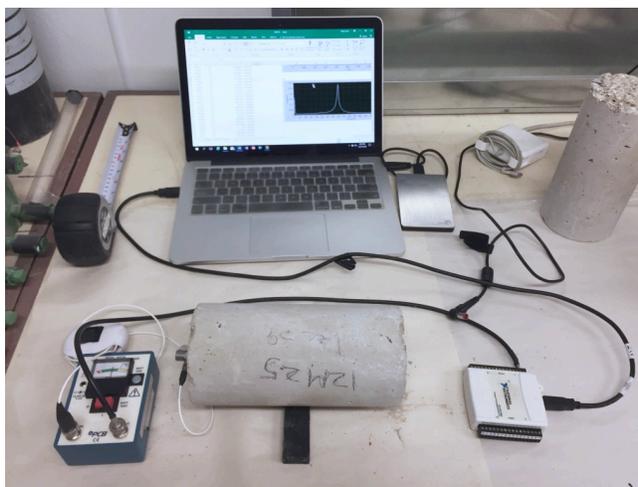


Figure 7 Experimental Setup for Resonant Frequency Test

3.3.3 Compression Test

Compression test were conducted right after the completion of resonant frequency by following ASTM C39-17 [18]. The equipment setup and loading pattern is shown in Figure 8.

The upper and lower bearing faces were wiped clean so that the load can be delivered evenly. Then, the cylinder was placed in between the upper and lower bearing surfaces carefully with the axes of cylinder and bearing blocks aligned with each other. After setting the load indicator to zero, the upper bearing block was lowered on the specimen. At the same time, the loading rate was rapidly adjusted to about 0.3 MPa/s, and the loading rate was remained same until the failure of the concrete cylinder. The maximum load in the unit of kN carried by the cylinder specimen was recorded for calculation. The obtained maximum loading value in kN was converted into compressive strength in MPa by dividing the cross-sectional area of the cylinder. Average value of the compressive strength of three cylinder specimens from each mix design were used to analyze the effect of alkaline activator on the compressive strength, compressive strength and resonant frequency from every single cylinder specimen was used for equation fitting.



Figure 8 Experimental Setup for Compression Test

Chapter 4 Results and Discussion

4.1 Factors Affecting the Compressive Strength of Geopolymer Concrete

Table 4 and Figure 9 give the information about the compressive strength development for all nine mixtures over the 28-day curing, where all the values represent the average compressive strength of three specimens. As expected, the geopolymer concrete cylinders after 28-day curing are much stronger than those were only cured for 7 days in general. This observation is in full accord with the discovery by Davidovits [7] that a longer curing time does not produce weaker geopolymer concrete. The compressive strength between each mixture did not have significant difference after curing for 7 days. It was not until the testing after 28-day curing that the mixture of 14M2.0 distinguished from other mix designs with the highest compressive at 21.65 MPa. It also can be noticed that about 60% of the strength was developed after first 7-day curing in average.

Table 4 Compression Test Results

Mixture	Average Compressive Strength after 7 Days (MPa)	Standard Deviation (MPa)	Average Compressive Strength after 28 Days (MPa)	Standard Deviation (MPa)	Average Strength Development on 7th Day (%)
10M2.0	11.79	0.478	19.57	1.398	60.25
10M2.5	11.61	3.468	16.14	1.418	71.93
10M3.0	5.94	N/A*	13.57	3.417	43.77
12M2.0	12.01	2.116	20.70	1.277	58.02
12M2.5	11.66	1.159	16.88	3.835	69.08
12M3.0	9.51	1.465	13.97	0.207	68.07
14M2.0	12.89	0.352	21.65	2.502	59.54
14M2.5	11.70	0.546	17.85	0.766	65.55
14M3.0	11.17	0.488	14.24	3.513	78.44

*Owing to fast setting time, the mixture 10M3.0 for 7-day test was not able to cast properly, only one specimen was cast successfully yet with unreasonably low compressive strength. Therefore, the standard deviation is not available.

Figure 9 gives the information about the effect of concentration of KOH and the PS/PH ratio on the compressive strength after curing for 7 days and 28 days respectively. Under the same PS/PH ratio, the mixture with a KOH solution in higher concentration yields a stronger geopolymer concrete, and the trends were both observed from 7-day and 28-day testing. In terms of PS/PH ratio, the test results suggest that a mixture with a smaller PS/PH ratio lead to a higher compressive strength.

When considering these two factors together, a higher concentration of KOH and a smaller PS/PH ratio produce strongest geopolymer concrete. It also can be noticed that the PS/PH ratio has more influence on the compressive strength of geopolymer concrete than the concentration of KOH solution, since a relatively bigger change on the compressive strength was observed over the variation on the PS/PH ratio, especially from the compression test results after curing for 28 days.

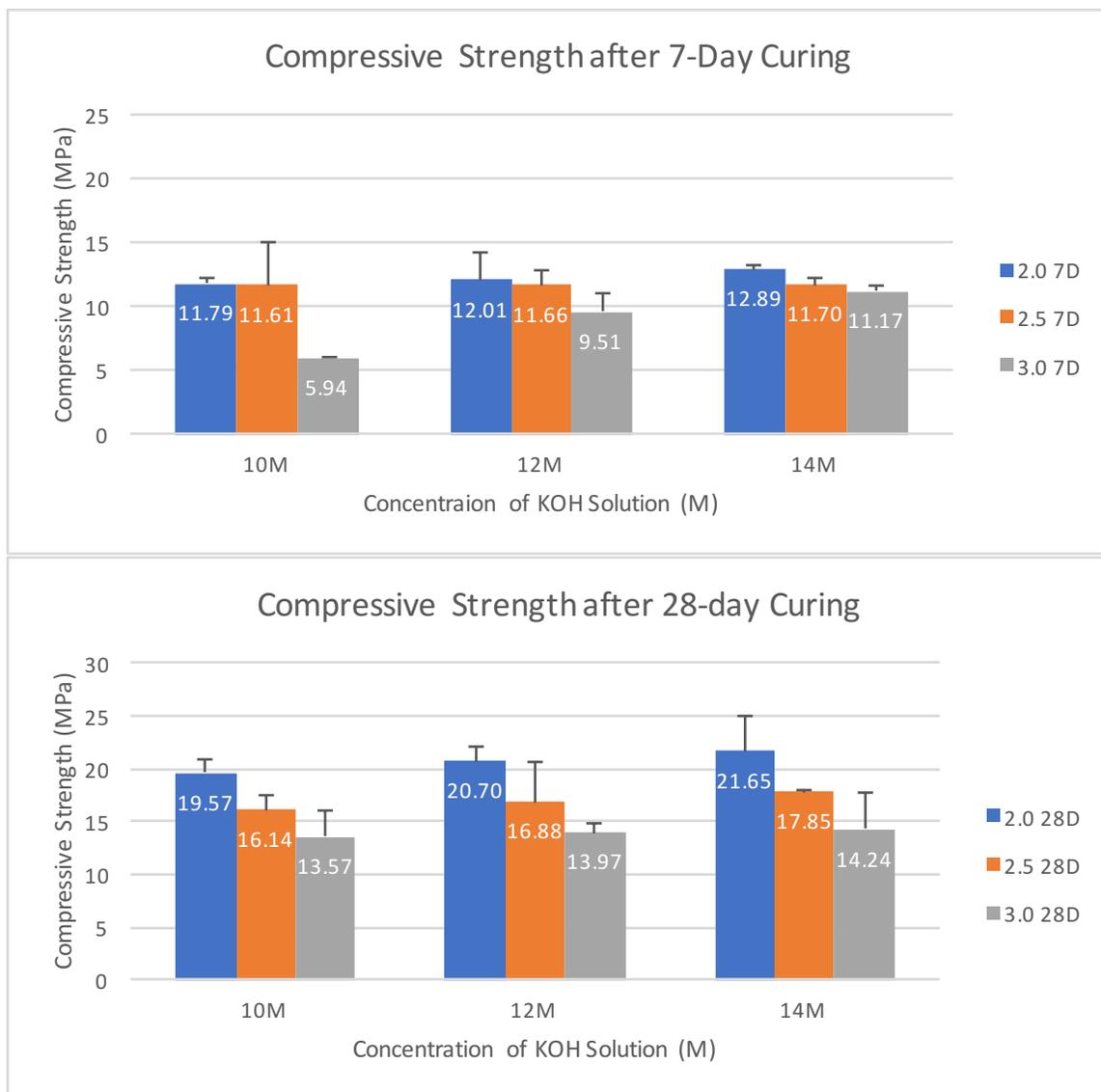


Figure 9 Compressive Strength Development

4.2 Correlation Between Resonant Frequency and Compressive Strength

4.2.1 Resonant Frequency of Geopolymer Concrete

Figure 10 plots the compressive strength over the resonant frequency, where the orange and blue dots represent the data collected from every single specimen after curing 7 days and 28 days respectively. As can be seen, the resonant frequency of geopolymer concrete in the size of 100mm × 200mm ranges from 3000 to 6000 Hz approximately. According to the previous investigation at the University of Victoria, the OPC-based concrete with average compressive strength of about 30 MPa has a resonant frequency between 9000 Hz to 9500 Hz, which is much higher than that of fly ash based geopolymer concrete with a similar compressive strength. This suggests geopolymer concrete has a much lower elastic modulus than OPC-based concrete. Previous study has investigated that the reduced elastic modulus was caused by the curing at elevated temperature. Curing at

ambient temperature can produce concrete with similar modulus of elasticity as OPC-based concrete, despite the slow development of strength [33].

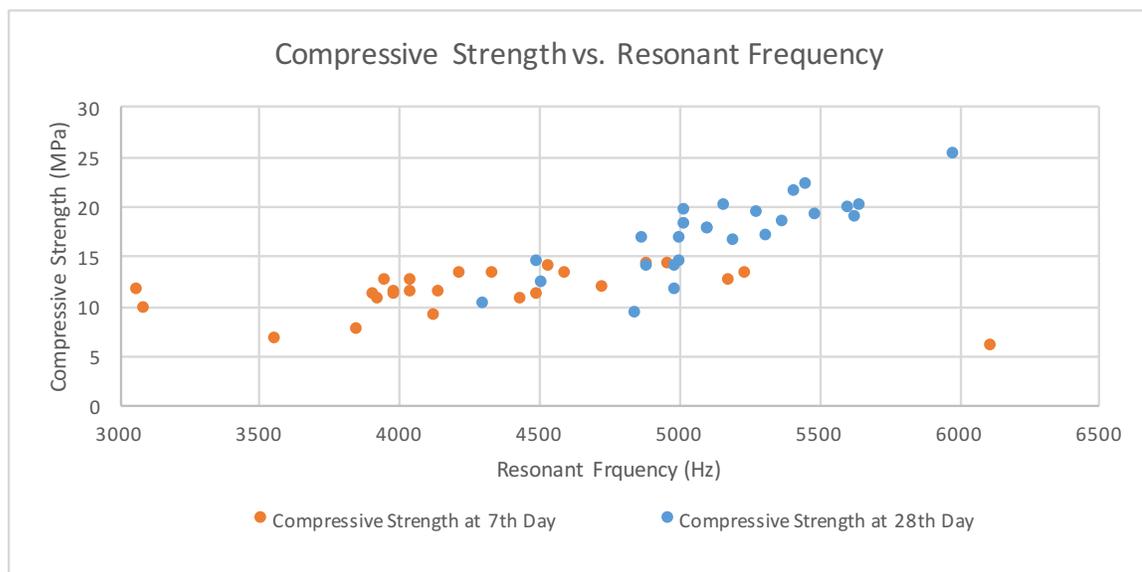


Figure 10 Compressive Strength vs. Resonant Frequency

For most mixtures, a higher resonant frequency was observed after curing for 28 days than that after 7 days. The increase in the resonant frequency suggests a longer the curing time could give the geopolymer concrete a more compact microstructure, thus should be higher compressive strength. This inference was confirmed by the test results in Table 5, which provides information about average resonant frequency and average compressive strength of each mixture. It can be seen that a higher resonant frequency generally corresponds to a higher compressive strength.

Table 5 Results of Resonant Frequency Test

Mixture	Average Resonant Frequency on 7th day (Hz)	Average Compressive Strength on 7th day (MPa)	Mixture	Average Resonant Frequency on 28th day (Hz)	Average Compressive Strength on 28th day (MPa)
10M3.0	6113.28	5.94	14M2.0	5742.19	21.65
14M2.0	5000.00	12.89	14M2.5	5345.05	17.85
14M3.0	4550.78	11.17	10M2.0	5305.99	19.57
10M2.5	4466.15	11.61	12M2.5	5136.72	16.88
12M2.0	4329.43	12.01	12M3.0	4954.43	13.97
14M2.5	4055.99	11.7	10M2.5	4928.39	16.14
12M2.5	4016.93	11.66	14M3.0	4902.34	14.24
10M2.0	3684.90	11.79	10M3.0	4726.56	13.57
12M3.0	3639.32	9.51	12M2.0	4658.20	20.7

However, an exception was observed from the mixture 10M3.0. It presented the maximum resonant frequency at about 6100 Hz after 7-day curing, but produced the minimum compressive strength of 5.94 MPa. This disagreement may result from the nature of the mixture. During the casting of 10M3.0, the hardening time was found to be too short to cast the fresh concrete into molds, the workability reduced significantly even before the compaction on the vibration table. As a result, some part of the fresh concrete became hardened over the manual mixing process without compaction. Therefore, three specimens tested after 7-day curing were not properly casted, a heterogeneous structure was observed after demoulding with a large volume of the air entrapped. Since the resonant frequency test and compression test are only effective for concrete in homogeneous structure, the data from 10M3.0 at 7 days was omitted for the correlation part.

4.2.2 Elastic Modulus of Geopolymer Concrete

The longitudinal resonant frequency data of every specimen, except those from the mixture 10M3.0 was converted to dynamic elastic modulus using Equation 1 in Chapter 2. The obtained results were plotted in Figure 11 and compared to the modeling equations derived by J. Wongpa [29], and ACI 318-08 [25]. It is obvious that ACI 318-08 standard overestimated the elasticity of geopolymer concrete. This is because the equation from ACI standard is only applicable for OPC-based concrete which usually has a higher elasticity than geopolymer concrete. While, the relationship proposed by J. Wongpa underestimated the experimental data. Especially for the compressive strength lower than around 8 MPa, it is not possible for concrete to have negative elasticity. The reason for the underestimation is the relationship was proposed to predict the compressive strength from static elastic modulus. For concrete materials with the similar compressive strength, static elastic modulus is always lower modulus than dynamic elastic modulus. From the comparison of experimental data and the relationship proposed by J. Wongpa, the dynamic elastic modulus of geopolymer was found to be higher than static elastic modulus by 30% approximately, when the strength ranges from 16 to 36 MPa.

Though these two relationships did not give accurate predictions on absolute values of the experimental data, they suggested the elastic modulus of concrete has a power law relationship to the compressive strength by the good predictions on the trend of data. Therefore, a power function will be used to propose the relationship between dynamic elastic modulus and compressive strength in following section.

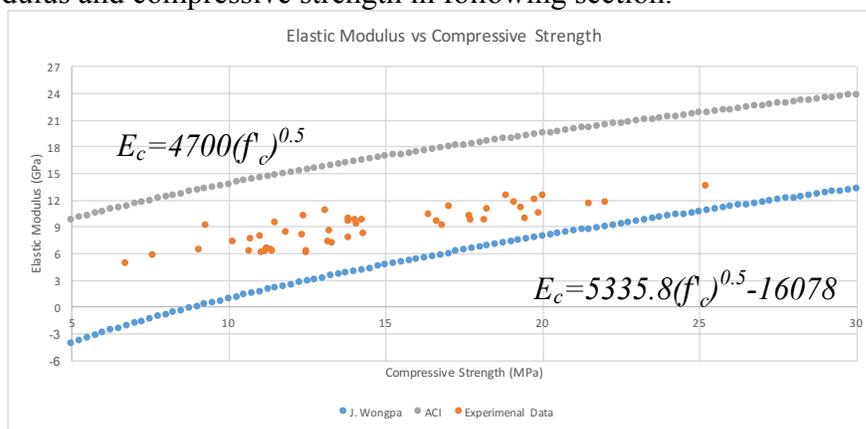


Figure 11 Compressive Strength vs. Dynamic Elasticity

4.2.3 Model Development

For the sake of convenience of modeling and the simplicity of the equation, this research proposed the general form of modeling equation as Equation 3:

$$G_d = a(f_c)^n \quad (3)$$

In order to improve the accuracy of the modeling equation and the range of applicability, two data points from another two mixtures with higher compressive that were developed by the geopolymer research group at the University of Victoria were also included for data fitting. These two mixtures were also fly ash-based geopolymer concrete, but they have much lower PH/PS ratio (0.25) and higher content of alkaline activator. The average compressive strength of these two mixtures was determined as 30.98 MPa and 35.29 MPa respectively, and the corresponding dynamic elasticity was 20.02 GPa and 22.12 GPa.

The regression analysis was conducted in the Curve Fitting Toolbox of Matlab for the experimental data. As can be seen in Figure 12, the best fitting was achieved with coefficients a and n as 0.7618 and 0.921 respectively. The goodness of the fitting was also generated. The coefficient of determination (R-square) was determined as 0.8477, which means this equation can explain about 84% of data variance.

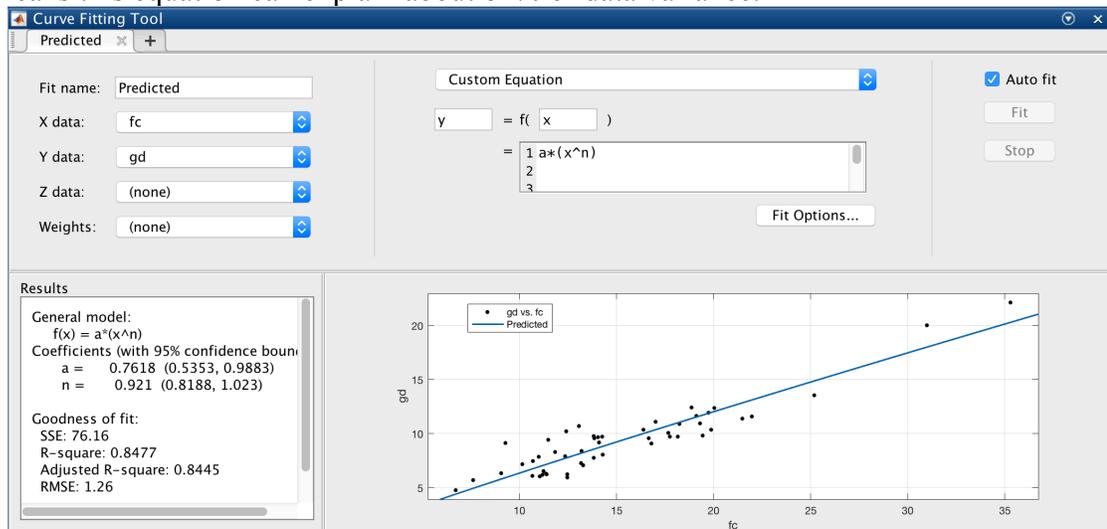


Figure 12 Regression Analysis in Matlab

Therefore, the relationship between dynamic elastic modulus and compressive strength for fly-ash based geopolymer concrete was established as Equation 4:

$$G_d = 0.7618 (f_c)^{0.921} \quad (4)$$

Other than being linear to the square root of compressive strength as investigated by J. Wongpa, the dynamic elastic modulus was found to be almost linear to compressive strength, since the coefficient n was determined to be 0.921. The reason may be the lack of data points in higher compressive strength. As can be seen, most data points distribute between 5 MPa and 22 MPa, with only two data points beyond 22 MPa. These only two

points are not enough to represent the real relationship between dynamic elasticity and compressive strength with good certainty, even though the overall R-square is 0.8477. Therefore, this modeling equation might yield unconfident prediction results for the geopolymer concrete with compressive strength higher than 22 MPa. However, the certainty of this modeling equation can be improved in future by integrating more data points from geopolymer concrete with higher compressive strength more than 22 MPa.

At last, by substituting Equation 1 in Chapter 2, an empirical predictive relationship for the compressive strength of fly ash-based geopolymer concrete from longitudinal frequency was proposed as Equation 5:

$$6.8648 \times 10^{-9} (L/d) M n^2 = (f_c)^{0.921} \quad (5)$$

where: n : fundamental longitudinal frequency (Hz)
 L : length of concrete cylinder (m)
 d : diameter of cylinder (m)
 M : mass of cylinder (kg)
 f_c : estimated compressive strength (MPa)

4.2.4 Model Validation

To validate the accuracy and the applicability, the developed modeling equation was used to compare with the experimental data and the modeling equation from previous work by Arkamitra Kar [32]. Arkamitra Kar [32] developed fly ash-based geopolymer concrete with compressive strength ranging from 20 to 90 MPa and he also established effective relationships for ultrasonic pulse velocity between and compressive strength, dynamic elastic modulus, which are shown in Equation 6, where v represents the velocity of ultrasonic pulse in the unit of m/s:

$$f_c = 7.78494v \text{ (MPa)} \quad G_d = 4.0849v \text{ (GPa)} \quad (6)$$

Even though the direct relationship between compressive strength and dynamic elastic modulus was not indicated in the original work directly, a relationship between these factors can be further derived by substituting these equations together, which shown Equation 7:

$$G_d \text{ (GPa)} = 0.52472 f_c \text{ (MPa)} \quad (7)$$

To compare the prediction results, the compressive strength from Equation 7 and Equation 4 were plotted together against dynamic elastic modulus, along with the experimental data in Figure 13. It can be seen these two equations have very similar trends on the prediction of compressive strength from dynamic elastic modulus. But the equation derived from Arkamitra's work seems to relatively overestimate the compressive strength. A similar conclusion can be drawn from the numerical evaluation to the goodness of fitting. The compressive strength was averagely overestimated by 2.966 MPa from Arkamitra Kar's work when compressive strength ranges from 5 to 48 MPa. The maximum difference was

about 3.49 MPa when dynamic elastic modulus is close to 23.69 GPa. The R-squared value was calculated as 0.7196 in a spreadsheet. The close R-squared values indicate both of these two predictive equations can give reasonable a prediction on the compressive strength, but the equation derived in this project can account for more data variance.

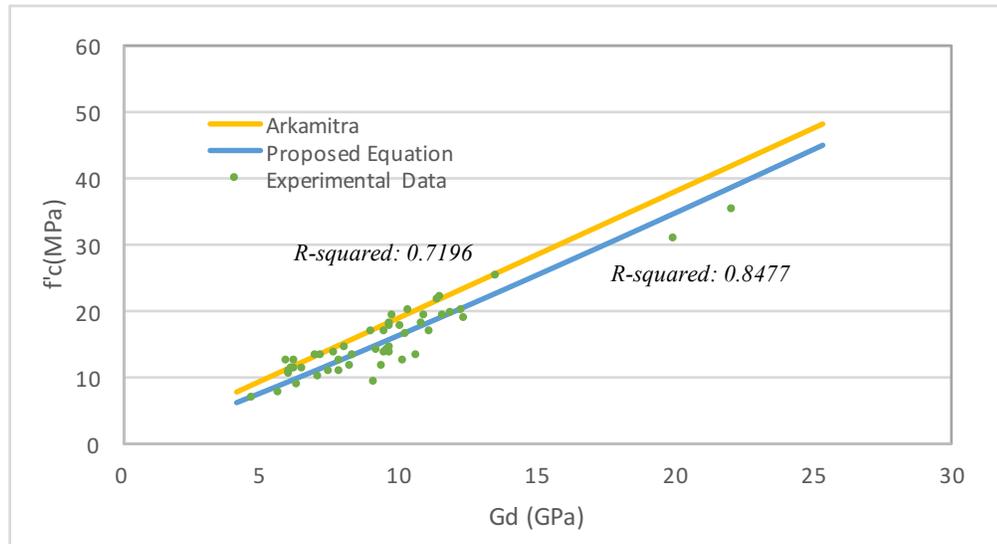


Figure 13 Compressive Strength Predictions

4.2.5 Applicability and Limitations

There are some situations where the estimation equation may not be applicable. First, the estimation equation was derived from fresh geopolymer concrete, so the estimation equation is only valid for geopolymer concrete in early age. Also, since the concrete specimens were prepared and tested by following ASTM Standards, the conditions indicated in ASTM C215-14 and ASTM 039-17 should be carefully followed. For example, ASTM C215-14 has clarified the geometry limitations on the test specimens that clearly indicate that the relationship is only valid for concrete specimens in cylinders or prism shapes. Therefore, the equations for the dynamic elastic modulus calculation may not be applicable to other shapes without the correction on shape factor.

Affected by boundary conditions, the estimation equation is probably not valid for in-situ field test without any implementing factors on the boundary conditions. For example, this estimation equation is not valid for a fly-ash geopolymer concrete cylinder under stress.

At last, the estimation equation is valid for fly ash-based geopolymer concrete as investigated so far, but the applicability for other types of geopolymer concrete, such as slag-based, is not known until further experimental investigations.

Chapter 5 Conclusions

Due to the significant role of alkaline activator in the compressive strength, therefore the mixture of fly ash-based geopolymer concrete was developed by researching on the impact of alkaline activator first. Instead of using ordinary sodium-based activator, the mixture was activated by potassium-activator due to the better contribution on the workability and compressive strength. The best mixture was determined by ranging the concentration of potassium hydroxide, the ratio of potassium silicate to potassium hydroxide, with holding the fly ash to alkaline liquid ratio, water content, and aggregate content at constant without any variation. The developed mixture was casted into concrete cylinders in the size of 100×200 mm, followed by resting for 24 hours and water immersion hot-oven curing. Since it was believed the hot-oven curing longer than 24 hours does not boost the compressive strength significantly. Therefore, cylinders were transfer to a water tank for ambient temperature curing after 24-hour heat curing until the test day.

The compressive test was conducted after curing for 7 days and 28 days respectively, and most concrete cylinders were found to exhibit good compressive strength, despite the fast setting time over the casting. The compressive strength of geopolymer cylinders was investigated to increase with a higher concentration of the potassium hydroxide solution and a lower ratio of potassium silicate to potassium hydroxide. The mixture with best compressive strength was found to have the highest potassium hydroxide solution of 14M and the lowest ratio of potassium silicate to potassium hydroxide 2.0 from both tests after curing for 7 days and 28 days, and the compressive strength of this mixture was found to increase by 67%, which were tested as 12.89 MPa and 21.65 MPa respectively. It was found most mixture developed about 60% in average of compressive over the first 7-day curing.

Besides, the present study also established the predictive relationship between the resonant frequency and compressive strength by using dynamic elastic modulus as an intermediate. The longitudinal resonant frequency of every cylinder was determined and dynamic elastic modulus was calculated by following ASTM C215-14 before the compression test. The fly ash-based geopolymer concrete was investigated to have a 33% to 66% less resonant frequency and dynamic elasticity than those of cement-base concrete in the same size. The obtained longitudinal resonant frequency for geopolymer concrete ranged between 3000 and 6000 Hz, while the cement-based concrete cylinder usually has a longitudinal resonant frequency around 9000 Hz. This may result from the different properties of binder materials. As for dynamic elastic modulus, it was found the higher the dynamic elastic modulus it has, the stronger the geopolymer concrete will be. The strongest cylinder with a compressive strength of 25MPa has a highest dynamic elastic modulus around 13.6 GPa.

After the regression analysis in Matlab by fitting experimental data, the relationship between compressive strength and dynamic elastic modulus was derived as $G_d = 0.7618(f)^{0.921}$, this equation can account for 85% of data variance. Good accuracy and consistency were validated by the experimental data and modeling equation from previous work by Arkamitra Kar [32]. The final equation for the prediction of compressive strength from longitudinal resonant frequency for fly ash-based geopolymer concrete was confirmed as equation: $6.865 \times 10^{-9} (L/d^2) Mn^2 = (f)^{0.921}$

Bibliography

- [1] The Concrete Conundrum. Chemistry World | March 2008
Available: http://www.rsc.org/images/Construction_tcm18-114530.pdf. [Accessed: 08-May-2017].
- [2] “History of Cement - Cement Association of Canada.” [Online]. Available: <http://www.cement.ca/en/Manufacturing/History-of-Cement.html> [Accessed: 02-May-2017].
- [3] A. Nazari and J. G. Sanjayan, *Handbook of Low Carbon Concrete*. Elsevier Science, 2016.
- [4] W. K. Part, M. Ramli, and C. B. Cheah, “An overview on the influence of various factors on the properties of geopolymer concrete derived from industrial by-products,” *Construction and Building Materials*, vol. 77, pp. 370–395, Feb. 2015.
- [5] “100,000 tonnes Geopolymer Concrete: world premiere – Joseph Davidovits.” [Online]. Available: <https://www.davidovits.info/100000-tonnes-geopolymer-concrete-world-premiere/>. [Accessed: 08-May-2017].
- [6] R. Hooton, S. Kolluru, J. Popovics, and S. Shah, “Determining Elastic Properties of Concrete Using Vibrational Resonance Frequencies of Standard Test Cylinders,” *Cement, Concrete and Aggregates*, vol. 22, no. 2, p. 81, 2000.
- [7] J. Davidovits, *Geopolymer chemistry and applications*, 4th edition. Saint-Quentin, France: Institut Géopolymère, 2015.
- [8] Concrete Institute of Australia, *Recommended practice: geopolymer concrete*. North Sydney: Concrete Institute of Australia, 2011.
- [9] B. Joseph and G. Mathew, “Influence of aggregate content on the behavior of fly ash based geopolymer concrete,” *Scientia Iranica*, vol. 19, no. 5, pp. 1188–1194, Oct. 2012.
- [10] D. Hardjito, B. V. Rangan, and others, “Development and properties of low-calcium fly ash-based geopolymer concrete,” *Curtin University of Technology, Perth, Australia*, 2005.
- [11] V. M. Malhotra, “Making Concrete ‘Greener’ with Fly Ash,” *Concrete International*, vol. 21, no. 5, May 1999.
- [12] P. S. PEng and K. C. PEng, “Sustainability in Construction: Using Fly Ash as a Cement Replacement,”
- [13] S. V. A. Silva, C. L. Wijewardena, S. M. A. Nanayakkara, and others, “Development of fly ash based geopolymer concrete,” 2012.

- [14] J. Temuujin, A. van Riessen, and R. Williams, "Influence of calcium compounds on the mechanical properties of fly ash geopolymer pastes," *Journal of Hazardous Materials*, vol. 167, no. 1–3, pp. 82–88, Aug. 2009.
- [15] H. Xu and J. S. J. Van Deventer, "The geopolymerisation of alumino-silicate minerals," *International Journal of Mineral Processing*, vol. 59, no. 3, pp. 247–266, Jun. 2000.
- [16] A. Palomo, M. W. Grutzeck, and M. T. Blanco, "Alkali-activated fly ashes: A cement for the future," *Cement and Concrete Research*, vol. 29, no. 8, pp. 1323–1329, Aug. 1999.
- [17] D. Sabitha, J. K. Dattatreya, N. Sakthivel, M. Bhuvaneshwari, and S. A. J. Sathik, "Reactivity, workability and strength of potassium versus sodium-activated high volume fly ash-based geopolymers," *Current Science*, vol. 103, no. 11, pp. 1320–1327, 2012.
- [18] C. ASTM, "39-17.(2017). Standard test method for compressive strength of cylindrical concrete specimens," *Annual Book of ASTM Standards*, vol. 04.02.
- [19] B. Siva Konda Reddy, J. Varaprasad, and K. Naveen Kumar Reddy, "Strength and Workability of Low Lime Fly-Ash Based Geopolymer Concrete," *Indian Journal of Science and Technology; Volume 3, Issue 12, December 2010*, Dec. 2010.
- [20] P. Rovnaník, "Effect of curing temperature on the development of hard structure of metakaolin-based geopolymer," *Construction and Building Materials*, vol. 24, no. 7, pp. 1176–1183, Jul. 2010.
- [21] D. Hardjito, S. E. Wallah, D. M. Sumajouw, and B. V. Rangan, "Factors influencing the compressive strength of fly ash-based geopolymer concrete," *civil engineering dimension*, vol. 6, no. 2, p. pp–88, 2004.
- [22] G. L. Workman, P. O. Moore, and American Society for Nondestructive Testing, *Nondestructive Testing Overview*. American Society for Nondestructive Testing, 2012.
- [23] J. Helal, M. Sofi, and P. Mendis, "Non-destructive testing of concrete: A review of methods," *Electron. J. Struct. Engrnr*, vol. 14, pp. 97–105, 2015.
- [24] C. ASTM, "215-14.(2014). Standard test method for fundamental transverse, longitudinal, and torsional resonant frequencies of concrete specimens," *Annual Book of ASTM Standards*, vol. 04.02.
- [25] ACI Committee 318, American Concrete Institute, and International Organization for Standardization, *Building Code Requirements for Structural Concrete (ACI 318-08) and Commentary*. American Concrete Institute, 2008.

- [26] ACI Committee 363, American Concrete Institute, and International Organization for Standardization, *Building Code Requirements for Structural Concrete (ACI 363-92) and Commentary*. American Concrete Institute, 1992.
- [27] C. S. Standards Association of Australia. Committee BD-002, *Concrete Structures: AS 3600-2005*. Standards Australia, 2005.
- [28] M. Tumkur and Canadian Standards Association, *Design of concrete structures: A23.3-04*. Mississauga, Ont.: Canadian Standards Association.
- [29] J. Wongpa, K. Kiattikomol, C. Jaturapitakkul, and P. Chindaprasirt, “Compressive strength, modulus of elasticity, and water permeability of inorganic polymer concrete,” *Materials & Design*, vol. 31, no. 10, pp. 4748–4754, Dec. 2010.
- [30] R. E. Rowe and Cement and Concrete Association (Great Britain), *Handbook on the Unified Code for Structural Concrete (CP110:1972)*. Cement and Concrete Association, 1972.
- [31] Sharma, M.R. and Gupta, B.L., “Sonic modulus as related to strength and static modulus of high strength concrete”, *Indian Concrete Journal. (Bombay)*, vol. 34, no. 4, pp. 139–140, 1960.
- [32] A. Kar, U. B. Halabe, I. Ray, and A. Unnikrishnan, “Nondestructive characterizations of alkali activated fly ash and/or slag concrete,” *European Scientific Journal, ESJ*, vol. 9, no. 24, 2013.
- [33] M. Sofi, J. S. J. van Deventer, P. A. Mendis, and G. C. Lukey, “Engineering properties of inorganic polymer concretes (IPCs),” *Cement and Concrete Research*, vol. 37, no. 2, pp. 251–257, Feb. 2007.

Appendix A Materials Information

Fly Ash

Properties	Values (%)
Fineness Retained on 45 micron (No. 325 Sieve)	16.4
Loss of ignition	0.19
SiO ₂	47.1
Al ₂ O ₃	17.4
Fe ₂ O ₃	5.7
CaO	14
MgO	5.4
SO ₃	0.8

Aggregates



RELATIVE DENSITY AND ABSORPTION OF COARSE AGGREGATE CSA A23.2-12A

January 12, 2016
Project Number: 1530704/7000

LEHIGH MATERIALS, DIVISION OF LEHIGH HANSON MATERIALS LTD.
P.O. Box 1790
Sechelt, BC
V0N 3A0

ATTENTION: Mr. Nick Sawchuk

PROJECT: CSA Concrete Aggregate Testing, December 2015

Sample:	Coarse Aggregate
Source:	Sechelt Pit

Date sampled: December 2015
Date tested: January 12, 2016

Sampled by: Client
Tested by: DC

Trial No.	Mass (g)	Relative Density (Dry Basis)	Relative Density (SSD Basis)	Apparent Relative Density	Absorption (%)
1	3483.4	2.699	2.717	2.748	0.66
2	3808.0	2.690	2.709	2.743	0.72
AVERAGE		2.695	2.713	2.746	0.69

Reported by: I. Chung

Reviewed by: 
L. Hu, M.Sc.E., P.Eng.



Notice: The test data given herein pertain to the sample provided, and may not be applicable to material from other production zones/periods. This report constitutes a testing service only. Interpretation of the data given here may be provided upon request.
GOLDER ASSOCIATES LTD., 300 - 3811 North Fraser Way, Burnaby, BC Canada V5J 5J2 Tel: 604-412-6899 Fax: 604-412-6816



**RELATIVE DENSITY AND ABSORPTION
OF FINE AGGREGATE**
CSA A23.2-6A

January 12, 2016
Project Number: 1530704/7000

LEHIGH MATERIALS, DIVISION OF LEHIGH HANSON MATERIALS LTD.
P.O. Box 1790
Sechelt, BC
V0N 3A0

ATTENTION: Mr. Nick Sawchuk

PROJECT: CSA Concrete Aggregate Testing, December 2015

Sample:	Fine Aggregate
Source:	Sechelt Pit

Date sampled: December 2015
Date tested: January 7, 2016

Sampled by: Client
Tested by: DC

Trial No.	Mass (g)	Relative Density (Dry Basis)	Relative Density (SSD Basis)	Apparent Relative Density	Absorption (%)
1	500.5	2.649	2.669	2.705	0.79
2	500.8	2.652	2.672	2.708	0.78
AVERAGE		2.651	2.671	2.707	0.79

Reported by: I. Chung

Reviewed by: 
L. Hu, M.Sc.E., P.Eng.



Notice: The test data given herein pertain to the sample provided, and may not be applicable to material from other production zones/periods. This report constitutes a testing service only. Interpretation of the data given here may be provided upon request.
GOLDER ASSOCIATES LTD., 300 - 3811 North Fraser Way, Burnaby, BC Canada V5J 5J2 Tel: 604-412-6899 Fax: 604-412-6818



SIEVE ANALYSIS OF FINE AND COARSE AGGREGATE

CSA A23.2-2A

LEHIGH MATERIALS, DIVISION OF LEHIGH HANSON MATERIALS LTD.
 P.O. Box 1790
 Sechelt, BC
 V0N 3A0

January 12, 2016
 Project Number: 1530704/7000

ATTENTION: Mr. Nick Sawchuk

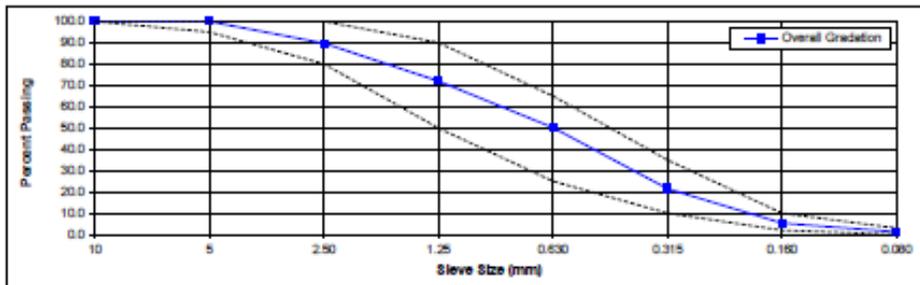
PROJECT: CSA Concrete Aggregate Testing, December 2015

Sample:	Fine Aggregate
Source:	Sechelt Pit

DATE SAMPLED: December 2015
 DATE TESTED: January 6, 2016

SAMPLED BY: Client
 TESTED BY: RZ

Sieve Size (mm)	% Retained	% Passing	Individual % Retained (Split values)		MATERIAL SPECIFICATION: CSA FA1	
			+ 5	- 5		
10	0.0	100.0	0.0		100	100
5	0.0	100.0	0.0		95	100
2.50	10.3	89.7		10.3	80	100
1.25	17.7	72.0		17.7	50	90
0.630	21.9	50.2		21.9	25	65
0.315	28.6	21.5		28.6	10	35
0.160	16.2	5.3		16.2	2	10
0.080	4.1	1.2		4.1	0	3
PAN	1.2	0		1.2		
Total	100.0		0.0	100.0		



Remarks: Fineness Modulus: 2.61

Reported by: I. Chung

Reviewed by: 
 L. Hu, M.Sc.E., P.Eng.



Notice: The test data given herein pertain to the sample provided, and may not be applicable to material from other production zones/periods. This report constitutes a testing service only. Interpretation of the data given here may be provided upon request.

Potassium Silicate

TESTIMONIALS

PQ Corporation
 Product of U.S.A.
 Produit des États-Unis

Manufactured by
 PQ Corporation
 P.O. Box 840
 Valley Forge, Pennsylvania, USA, 19482
 For emergency, call: / Pour les urgences, appeler: CHEMTREC: 800-424-9300
 General Information / Information générale: 610-851-4200

AgSil® 16H

Potassium Silicate/Silicate de Potassium GRADE: 0-0-32

GUARANTEED ANALYSIS
 Soluble Potash (K₂O).....32.0%
 Derived from Potassium Silicate
 F1253

Information regarding the contents and levels of metals in this product
 is available on the internet at <http://www.aapco.org/metals.htm>

WARNING
CAUSES EYE AND SKIN IRRITATION

Avoid contact with eyes, skin and clothing. Wash thoroughly after handling.

PRECAUTIONARY MEASURES: Wear suitable protective clothing, gloves, goggles and face protection.

FIRST AID: In case of contact, immediately flush eyes and/or skin with plenty of water for at least 15 minutes. Remove contaminated clothing or shoes. Wash clothing before reuse. Thoroughly clean shoes before reuse. Get medical attention if irritation persists.

SPILL OR LEAK: Carefully shovel or sweep up spilled material and place in a suitable container. Avoid generating dust. Use appropriate personal protective equipment (PPE).

For additional information, see Material Safety Data Sheet (MSDS) for this chemical.

CONTAINS/CONTIENT: Potassium Silicate/Silicate de Potassium – CAS Reg. No. 1312-76-1 and/et Water/Eau CAS Reg. No. 7732-18-5.

For Agricultural Use Only / Pour Usage Agriculture Seulement
 Keep Out of Reach of Children / Garder Hors de Porter des Enfants

AVERTISSEMENT
CAUSE L'IRRITATION AUX YEUX et à LA PEAU

Éviter le contact avec les yeux, la peau et les vêtements. Se laver soigneusement après la manipulation du produit.

MESURES PRÉVENTIVES: Porter un vêtement de protection approprié, des gants et une protection pour les yeux et le visage.

PREMIERS SOINS: En cas de contact avec les yeux ou la peau, rincer immédiatement à grande eau pendant au moins 15 minutes. Enlever tous vêtements et chaussures contaminés. Nettoyer les chaussures avant de les ré-utiliser. Laver les vêtements avant de les ré-utiliser. Obtenir l'attention médicale si l'irritation persiste.

DÉVERSEMENT OU FUIE: Ramasser ou balayer en prenant soin et placer le matériel dans un contenant approprié. Éviter la génération de poussière. Utiliser de l'équipement de protection (EPP) adéquat.

Pour de l'information supplémentaire, veuillez consulter la fiche signalétique pour ce produit.

NET WEIGHT: 50 LBS / 22.7 KG POIDS NET: 50 LBS / 22.7 KG

Rev. 10/07



THORNTON LABORATORIES
TESTING & INSPECTION SERVICES, INC.

1145 E. Cass St, Tampa, FL 33602
Phone: 813-223-9702 Fax: 813-223-8332
WWW.THORNTONLAB.COM

12-Oct-2015
Page 1 of 1

Report For: Custom Hydro
206 East 4th St.
Miller, MO. 65707
Attn: Brian Roethemeier

Sample Identification:

Soluble Potassium Silicate Powder
Id: AgSil 16H 0-0-32 with 52.8% SiO₂, PQ Corporation

Date Received: 25-Sep-2015

Laboratory Number: 384879

CERTIFICATE OF ANALYSIS

Method	Parameter	Result	Units
JAOAC, Vol. #96, #2, 2013	Silicon, Soluble (Si)	8.54	%

THORNTON LABORATORIES
Steve Fickett, III

Thornton Laboratories Testing & Inspection Services, Inc. responsibility for the above analysis originates with the client. Thornton Laboratories' responsibility for the above analysis or interpretations is limited to the invoice amount.



©2010 Infix Pro PDF Editor
To remove this notice, visit:
www.pdfeditng.com

Potassium Hydroxide

Specification Sheet

SIGMA-ALDRICH

Product Name	Potassium hydroxide, ACS reagent, ≥85% KOH basis (≤2.0% K ₂ CO ₃), pellets
Product Number	P6310
Product Brand	SIGMA
CAS Number	1310-58-3
Molecular Weight	56.11

TEST

APPEARANCE
ASSAY
CHLORIDE (CL)
NITROGEN COMPOUNDS (N)
PHOSPHATE (PO₄)
SULFATE (SO₄)
**AMMONIUM HYDROXIDE
PRECIPITATE**
HEAVY METALS (AS SILVER)
IRON (FE)
NICKEL (NI)
SODIUM (NA)
SHELF LIFE SOP QC-12-006

SPECIFICATION

WHITE PELLETS
NLT 85%, NMT 2.0% K₂CO₃
NMT 0.01%
NMT 0.001%
NMT 5 PPM
NMT 0.003%
NMT 0.02%
NMT 0.001%
NMT 0.001%
NMT 0.001%
NMT 0.05%
2 YEARS
DOCUMENT # P6310/4/30/93/1



SIGMA-ALDRICH

3050 Spruce Street
 Saint Louis, Missouri 63103 USA
 Telephone 800-325-5832 • (314) 771-5765
 Fax (314) 286-7828
 email: techserv@sial.com
 sigma-aldrich.com

Product Information

Potassium hydroxide ACS Reagent

Product Number **P 6310**
 Store at Room Temperature
22,147-3 is an exact replacement for P 6310

Product Description

Molecular Formula: KOH
 Molecular Weight: 56.11
 CAS Number: 1310-58-3
 Melting point: 360 °C, 380 °C (anhydrous)¹

This product is in the form of pellets. It is designated as ACS Reagent grade, and meets the specifications of the American Chemical Society (ACS) for reagent chemicals.

Potassium hydroxide (KOH) is a caustic reagent that is widely used to neutralize acids and prepare potassium salts of reagents. It is used in a variety of large-scale applications, such as the manufacture of soap, the mercerizing of cotton, electroplating, photoengraving, and lithography.¹

Potassium hydroxide is used in the analysis of bone and cartilage samples by histology.^{2,3} A protocol for the amplification of DNA from single cells by PCR that incorporates KOH has been reported.⁴ The use of KOH in studies of the binding of intercalating anticancer drugs to nucleic acids has been investigated.⁵

Precautions and Disclaimer

For Laboratory Use Only. Not for drug, household or other uses.

Preparation Instructions

This product is soluble in water (100 mg/ml), yielding a clear, colorless solution. Potassium hydroxide is also soluble in alcohol (1 part in 3) and glycerol (1 part in 2.5). The dissolution of potassium hydroxide in water or alcohol is a highly exothermic (heat-producing) process.¹

Storage/Stability

Potassium hydroxide rapidly absorbs carbon dioxide and water from the air and deliquesces.¹ Potassium hydroxide solutions should be stored in plastic bottles (polyethylene or polypropylene). KOH solutions will etch glass over a period of just a few days.

References

1. The Merck Index, 12th ed., Entry# 7806.
2. Philip, N. S., and Green, D. M., Recovery and enhancement of faded cleared and double stained specimens. *Biotech. Histochem.*, **75(4)**, 193-196 (2000).
3. Miller, D. M., and Tarpley, J., An automated double staining procedure for bone and cartilage. *Biotech. Histochem.*, **71(2)**, 79-83 (1996).
4. Pierce, K. E., et al., QuantILyze™: reliable DNA amplification from single cells. *BioTechniques*, **32(5)**, 1106-1111 (2002).
5. Cummings, J., et al., Determination of covalent binding to intact DNA, RNA, and oligonucleotides by intercalating anticancer drugs using high-performance liquid chromatography. Studies with doxorubicin and NADPH cytochrome P-450 reductase. *Anal. Biochem.*, **194(1)**, 146-155 (1991).

GCY/NSB 3/03

Sigma brand products are sold through Sigma-Aldrich, Inc.

Sigma-Aldrich, Inc. warrants that its products conform to the information contained in this and other Sigma-Aldrich publications. Purchaser must determine the suitability of the product(s) for their particular use. Additional terms and conditions may apply. Please see reverse side of the invoice or packing slip.