HASAN KALYONCU UNIVERSITY
GRADUATE SCHOOL OF
NATURAL & APPLIED SCIENCES

EFFECT OF MOLARITY OF NAOH ON STRENGTH OF GEOPOLYMER MORTAR

M. Sc. THESIS
IN
CIVIL ENGINEERING

BY
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DECEMBER 2016

Effect of molarity of NaOH on strength of geopolymer mortar

M.Sc. Thesis
In
Civil Engineering
Hasan Kalyoncu University

Supervisor
Assoc. Prof. Dr. Kasım MERMERDAŞ

By
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December 2016

T.C.
HASAN KALYONCU UNIVERSITY
GRADUATE SCHOOL OF NATURAL & APPLIED SCIENCES
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Exam date: 02 - 12 -2016

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Gailani Mohammed Mahmud Pirazhn

ABSTRACT

EFFECT OF MOLARITY OF NAOH ON STRENGTH OF GEOPOLYMER MORTAR

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M.Sc. in Civil Engineering

Supervisor: Assoc. Prof. Dr. Kasım MERMERDAŞ

November 2016, 59 pages

Geopolymers have received considerable attention due to their properties and applications as environmentally friendly alternatives to ordinary Portland cement. Geopolymer mortar (GPM) is an emerging alternative to ordinary Portland cement mortar (OPCM), and is produced via a polycondensation reaction between aluminosilicate source materials and an alkaline solution. As a relatively new material, many engineering properties of geopolymer mortar are still undetermined. In this study, the compressive strength, have been studied experimentally. A total of 12 geopolymer mix mortar were tested for the above mentioned characteristics, the results indicate that the combination of the above constituents at 90 ^{o}C has a positive impact on the strength of geopolymer mortar .The experimental data was analyzed by an analysis of variance (ANOVA) utilizing MINITAB16 statistical software. This method establishes the magnitude of the total variation in the results and distinguishes the random variation from the contribution of each variable. A conventional level of significance ($p < 0.05$) was used for the statistical analysis.

Key words: Geopolymer mortar; Fly ash; Alkaline solution, Compressive strength, ANOVA

ÖZET

NAOH'UN MOLARİTESİNİN GEOPOLİMER HARÇLARINA ETKİSİ

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İnşaat Mühendisliğ Yüksek Lisans

Danışman: Doç. Dr. Kasım MERMERDAŞ

Aralık 2016, 59 pages

Geopolimerler, sıradan Portland çimentosuna çevre dostu alternatifler olarak özellikleri ve uygulamaları nedeniyle büyük ilgi görmektedir. Geopolimer harç (GPM) sıradan Portland çimento harcı (OPCM) için ortaya çıkan bir alternatiftir ve alüminosilikat kaynak malzemeleri ile alkalin bir çözelti arasındaki bir polikondansasyon reaksiyonu vasıtasıyla üretilmektedir. Nispeten yeni bir materyal olarak, geopolimer harcının bir çok mühendislik özelliği hala tam olarak malzemne belir lenememi tir. Bu çalışmada basınç dayanımı deneysel olarak incelenmiştir. Toplam 12 geopolimer harç yukarıda belirtilen özelliklere göre test edilmiştir. Sonuçlar, yukarıda belirtilen bileşenlerin 90° C'de kombinasyonunun geopolimer harcının mukavemetini olumlu etkilediğini ortaya koymuştur. Deneysel veriler MINITAB16 istatistiksel yazılım ile bir yönlü varyans analizine (ANOVA) tabi tutulmuştur. Bu yöntem sonuçların toplam varyasyonunun büyüklüğünü belirler ve rastgele değişimi her değişkenin katkısından ayırır. İstatistiksel analiz için konvansiyonel bir anlam seviyesi ($p < 0.05$) kullanılmıştır.

Anahtar kelimeler: Geopolimer harç; Uçucu kül; Alkali çözelti, Basınç dayanımı, ANOVA

ACKNOWLEDGEMENT

In the name of Allah, the Entirely Merciful, the especially merciful. First of all, I want to express my gratitude and thankfulness to the God almighty who is creator, the sovereign, and the sustainer of the universe and creatures. It is only through his mercy and help this work could be completed and I am hoping that this little effort be accepted by him.

I would like to express my gratitude to my supervisor Assoc. Prof. Dr. Kasım MERMERDAŞ, for his invaluable help, advices and directions during this thesis.

Special thanks are reserved for Assist. Prof. Dr. Dia Eddin NASSANI for suggestions to improve the quality of the thesis.

My special thanks are reserved for my parents and my wife's parents, all my family members, they have given me an endless enthusiasm and encouragement.

My special thanks are reserved for my friends Ph.D. students Mr. Safie Oleiwi and Mr. Mohamed Moafak ARBILI

Finally, I would like to express my sincere gratitude to anyone who helped me throughout the preparation of the thesis.

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LIST OF SYMBOLS/ABBREVIATIONS

AA	Alkali Activated
C-S-H	Calcium Silicate Hydrate
CTL	Control
CO_2	Carbon dioxide
KOH	Potassium hydroxide
NaOH	Sodium hydroxide
M	Molarity
FA	Fly Ash
HM	Hydration Modulus
Ms	Modulus of Activator
OPC	Ordinary Portland Cement
SSD	Saturated Surface Dry
w/b	Water to Binder Ratio
w/s	Water to Solid Ratio

CHAPTER 1

INTRODUCTION

1.1 General

Around the World, the most widely used material in construction sector is concrete. In the production of concrete, cement plays the main role. To occupy the amount of cement needed for concrete production. Huge amount of raw material and energy needed. During the processes of cement production some of harmful gases or pilotable gases are released to the atmosphere, causing pollution and effect greenhouse. Day by day and due to the increased demand for concrete production of cement was increased. According to the studies, it was estimated that the 2 billion tons of cement is manufactured annually. Moreover, there is an annual increase in production about 3%, thus processes of cement production increases release of CO_2. Since each 1 ton of cement production release about 1 tone of CO_2 to atmosphere, due to combustion of fossil fuels and cement production processes as result of limestone de-carbonation of in the kiln (Roy 1999), it is one of the major components of the green house emission. The amount of CO_2 released to atmosphere about 7% of total emissions of greenhouse gases, and CO_2 contributes about 65% of global warming.

Among studies that specialize durability of concrete against weather condition, it was observed that many concrete structures that subjected to corrosive environment deteriorated after time about 20-30 years. We have to think about new the technology of materials for structure construction that has green properties and better durability properties like geopolymers that used waste materials such as fly ash and less emission of gases.

The huge amount of fly ash generated worldwide from thermal power plant and lead to the problem of waste management. Fly ash based, binder system produce alternative to concrete eliminating cement is called “Geopolymer concrete”. Geopolymer binder are used together with aggregate to produce geopolymer concrete which are ideal for building and repairing infrastructure and for pre-casting unit. The new product that called geopolymer has properties of using waste materials on its production and saves up to 80% of CO_2 emission, gaining high final strength in a short time, better resistance to freezing and thawing, high resistance to sulphates, corrosion, and low shrinkage strains.

Davidovits in 1978 was the first researcher that introduces the term "Geopolymer" and described as a member family of mineral binders with an amorphous microstructure and has chemical composition similar to zeolites. Geopolymer used as an alternative to ordinary portland cement for concrete production, Davidovits (1988) used waste materials or by product materials such as slag, husk ash and fly ash that has an aluminum (Al) and silicon (Si) with an alkaline activator to produce a binder.

Further we can consider that the geopolymer is an environmentally friendly product, has better properties compared with conventional ordinary Portland cement. Chindaprasirt et al. (2009) through a research used fly ash as a base material to produce geopolymer concrete, with 3 different concentration of alkaline activator made by NaOH (5, 10 and 15M) with Na_2SiO_3, used heat curing at constant temperature at 65 ^oC for 48 hours. By using different methods of analyses SEM, FT-IR and DSC thermogram, indicated that the fly ash has a good reactivity and give good degree of geopolymerisation. It was that concentration of 10M is the best for alkaline activator for geopolymer production.

1.2 The Aim of the Study

The aim of this study is to obtain good alternative to ordinary Portland cement, the best option is to find an environmentally friendly product with the same time using waste or by product materials to get useful construction material. This study with obtaining suitable FA based geopolymer mortar by evaluating the strength development through using different molarity with different ratio of Na_2SiO_3 to NaOH, in same time using computer software to evaluate the mix design through analyzing strength results.

1.3 Arrangement of the Organization

The thesis contains five chapters; Chapter 1 deals with the general introduction and aim of study. Chapter 2 reviews about OPC concrete, geopolymer concrete, production method for geopolymer, and the best mixes of materials used for production of geopolymer concrete. Chapter 3 materials, mixtures, casting, curing conditions, test methods, software analyzer are described. Chapter 4 includes discussion of test results. Chapters 5 summarizes the main results of this thesis.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter provides a review of previous studies in the field of geopolymer mortar. There are several published papers that investigated the effect of sodium silicate and sodium hydroxide on compressive strength and effect of the age on compressive strength. Moreover, the chapter also includes the previous studies utilization of statistical analysis to demonstrated effects factors on compressive strength.

2.2 Geopolymer

In 1978, Davidovits improve a binder called "geopolymer" to depict another cementitious material which has ceramic-like properties. Geopolymer could be created by joining a pozzolanic compound or aluminosilicate source material with highly alkaline solutions. The reaction of Al_2SiO_5 with alkali polysilicates produces a shapeless to semi-crystalline three-dimensional structure of polymeric sialate (Si-O-Al-O) bonds create are inorganic materials that polycondense practically identical to natural polymers called geopolymer.

Sialate tetrahedral arrangements, alkali silica-oxo-aluminate abbreviation, the figure 2.1 clarify calcium, sodium, lithium or potassium being the alkali (Davidovits 1978).

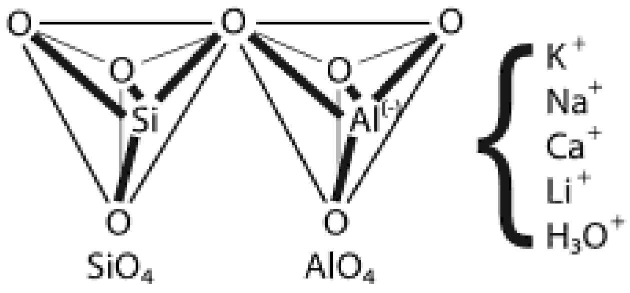

Figure 2.1 Sialate tetrahedral arrangements (Davidovits 1978)

Lee and Jang (2016) investigated the effect of FA properties on the development of FA based geopolymer strength, observed of delayed development of high strength geopolymer. Chemical and physical properties of FA were observed by particle size analyzer, X-ray fluorescence test and X-ray diffraction test. They applied multi-technical descriptions using SEM & EDS, MIP and FT-IR to obtain a thorough understanding of the relationship between microstructure, reaction products, and strength growth according to the ripeness of geopolymer. It was concluded that the properties of FA significantly affect the characteristics of geopolymer.

The geopolymer binder is a low-CO_2 cementious material. It doesn't depend on the calcination of limestone that produces CO_2. This innovation can set aside to 80% of CO_2 emissions created by the cement and aggregate industries. (Raijiwala et al., 2013)

The properties of geopolymer incorporate high early strength, low shrinkage, freeze-thaw resistance, sulfate resistance and corrosion resistance described by Raijiwala et al., (2013). These high-alkali binders don't generate any alkali-aggregate reaction described.

Also Ryu et al (2013) in their research showed the polmarization reaction by Figure 2.2

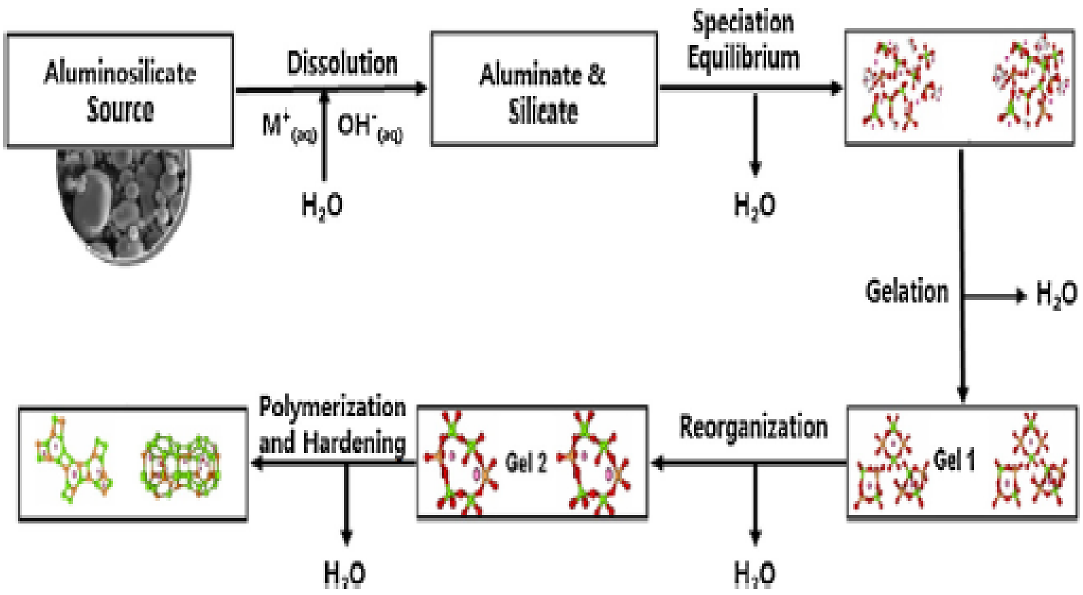

Figure 2.2 Geopolymerisation process (Ryu et al., 2013)

From source material Al_2SiO_5 dissolves by the action of hydroxide ions (-OH).

Foreboding ions then arrange into polycondense with monomers to form polymeric structures (Hardjito and Rangan, 2005). The below chemical formula explain poly sialate (Davidovits 2011).

$$Mn\ [-(SiO_2)_z\text{-}A_{l2}]_n,\ wH_2O \qquad \text{Eq (2.1)}$$

The manufacture of geopolymer concrete is carried out using the usual concrete technology methods. Hardjito et al., (2004) utilized geopolymer as the binder, in place of cement paste, to procreate concrete. The laboratory experiment results, low calcium (class F) FA from Western Australia was utilized as the source material. The chemical composition of the FA, as determined by X-Ray Fluorescence (XRF) analysis, is given in Table 2.1.

Table 2.1: Sythesis of FA as determined by XRF (mass %)

(Hardjito et al.,2004)

Oxides	**Batch I**	**Batch II**	**Batch III**
SiO_2	53.36	47.80	48.00
Al_2O_3	26.49	24.40	29.00
Fe_2O_3	10.86	17.40	12.70
CaO	1.364	2.42	1.78
Na_2O	0.37	0.31	0.39
K_2O	0.8	0.55	0.55
TiO_2	1.47	1.328	1.67
MgO	0.77	1.19	0.89
P_2O_5	1.43	2.00	1.69
SO_3	1.70	0.29	0.50
ZrO_2	-	-	0.06
Cr	-	0.01	0.016
MnO	-	0.12	0.06
LOI	1.39	1.10	1.61

2.3 Fly Ash

The aluminous-siliceous material and due to FA ability to react chemically with calcium hydroxide $Ca(OH)_2$ is classified as a pozzolanic to form cementitious compounds. FA is a by-product of combustion a coal, mainly it’s collected at energy power plants available through worldwide. FA can be classified according to the type of coal burned, FA produced from bituminous coals and anthracite is classified as Class F, and FA Class C produced from burning of lignite and sub-bituminous coals.

Because of the appearance of high amounts of calcium oxide CaO uncleanness in FA Class C geopolymer precursors interact to produce C-A-H and C-S-H and has parallel formation to direction of Si-O-Al-O bonds. During compounds hydration the alkalinity of the mixture rise and encourages rapid poly condensation and dissolution. Presence of calcium oxide (CaO) the chemical reaction improved and the strength of the geopolymerisation is increases (Diaz-Loya et al., 2011). FA reactivity depend on the proportion of SiO_2 and its nature (Hemmings and Berry, 1988). Figure 2.4 showed the chemical properties of FA

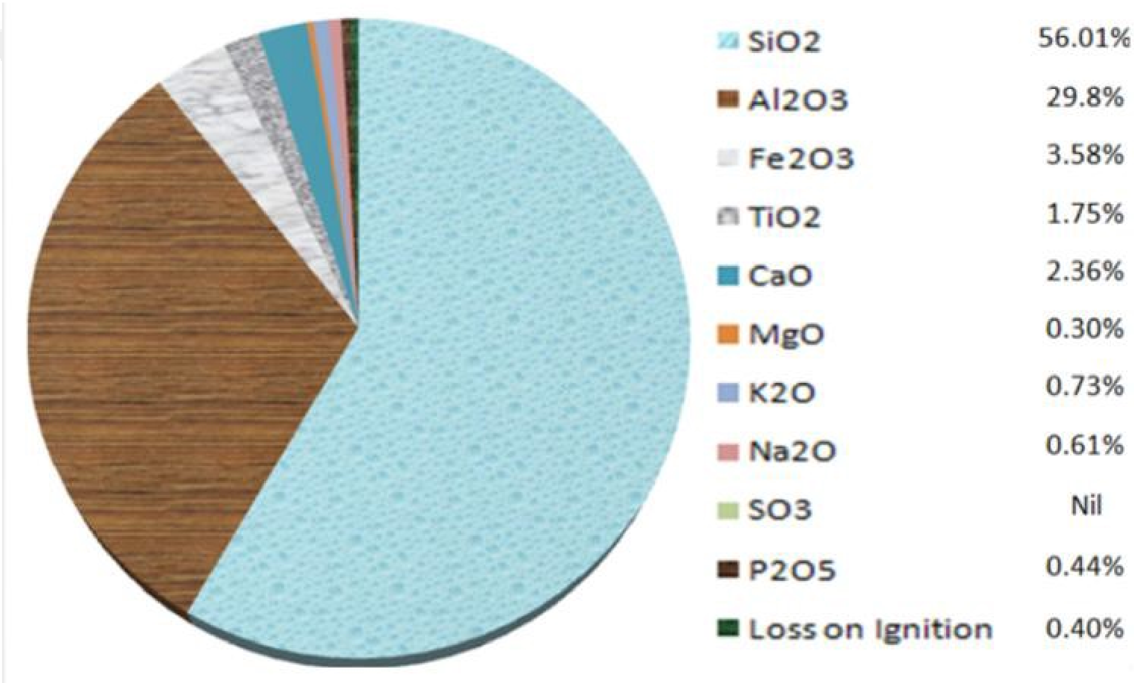

Figure 2.3 chemical properties of F A (Adam, 2009)

2.4 Alkaline Activators

The type of activator has the main role in the geopolymerisation product and when the alkaline activator contains soluble silicate, high rate of reactions happen and either potassium silicate (KOH) or sodium silicate (NaOH), compared with utilization of alkaline hydroxides only (Palomo et al. 1999).

The combination of sodium hydroxide and sodium silicate solutions are used for the activation of FA based geopolymer concrete, utilized by Ayachit et al (2016). Single activator either sodium hydroxide or sodium silicate alone is not much effective as clearly seen. Due to increase in concentration of sodium hydroxide solution in terms of molarity (M) makes the concrete more brittle with increased compressive strength. It is observed that the compressive strength of geopolymer concrete increases with increase in concentration of sodium hydroxide solution and or sodium silicate solution with increased viscosity of fresh mix.

Van Deventer and Xu (2000) approved that the solution made by adding sodium hydroxide (NaOH) solution to the sodium silicate (Na_2SiO_3) solution to make alkaline activator enhance the reaction between the solution and source material. Hence, by study of the geopolymerisation of 60 natural Si-Al minerals, they found in general the potassium hydroxide KOH solution caused a lower range of dissolution of minerals than the sodium hydroxide NaOH solution; therefore sodium hydroxide NaOH is better.

The influence of alkaline activator ratio on the compressive strength of FA-based geopolymers showed by (Al Bakri, 2011). Figure 2.3 shows that increasing in the water glass/NaOH solution mixing ratio leads to an increase in the ratio of SiO_2/Al_2O_3

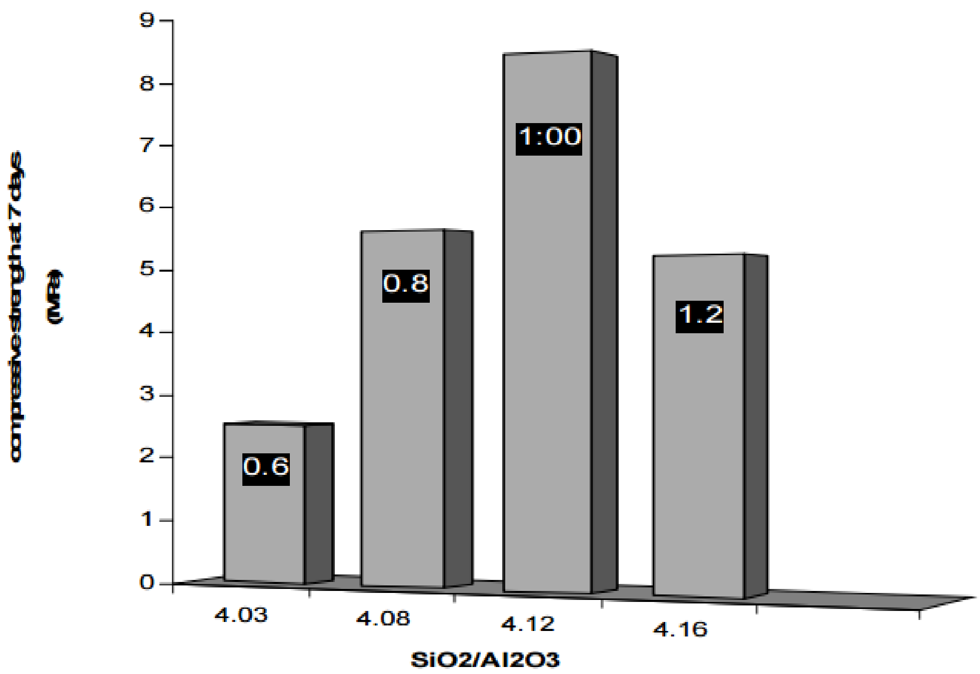

Figure 2.4 Influence SiO_2/Al2O_3 ratio on the compressive strength (Al Bakri, 2011)

2.5 Aggregates

Generally the aggregate used in concrete production must be with no unwanted chemicals, clean as possible, no absorbed clay and other fine materials that cause the distortion of the hydration process and distorts concrete characteristics. The main mechanical properties of concrete durability, hardness and strength, are the three main properties being important for concrete structures. Recycled aggregates should not be used in concrete in areas that has contact with chloride ions and/or sulfate. In general concrete made by recycled aggregate has high absorption rates low specific gravities compared with conventional gravel aggregate concrete. The concrete which is produced with recycled aggregate possesses unfavorable workability with low compressive strength. Because the compressive strength of concrete depends on the strength properties of the original aggregate and the W/C ratio.

Generally aggregates occupy about 70-77% of total concrete volume and it is obtained separately as fine and coarse aggregates. A part from natural aggregates and recycled aggregates detained from construction demolition, aggregate can also be made from slag or bottom ash and might be utilized as alternative to natural aggregate to produce concrete. Commonly aggregates are stored by different sizes; graded to be utilized to satisfy the required grading.

To reduce water request for mixing, Girawale (2015) in his research described that the fine aggregate should amount to smooth rounded particles, it is recommended that the grading should lie on the coarser side of the limits, a fineness modulus of 3.0 or greater recommended, both to reduce the water demand and to improve the workability of paste-rich mixes. The sand too must be free of silt or clay particles regarding in Table 2.2

Table 2.2 Aggregates specification (Girawale, 2015)

Properties	Coarse aggregate	Fine aggregate
Type	Crushed angular	Spherical (river sand)
Maximum size	20 mm	4.75 mm
Specific gravity	2.78	2.64
Material finer than 75 micron	NA	1.25%
Water absorption	1.10 %	1.46 %
Silt content (%)	0.4	1.1
Bulk density (g/cm^3)	1.53	1.90
Organic matter	NA	NA

Recycled aggregates should not be used in concrete in areas that has contact with chloride ions and sulfate. In general concrete made by recycled aggregate has high absorption rates low specific gravities compared with conventional gravel aggregate concrete.

2.6 Application of Geopolymer

Geopolymers are being investigated in many scientific and industrial disciplines, including modern inorganic chemistry, physical chemistry, colloid chemistry, mineralogy, geology and other engineering process technologies. Geopolymers have potential uses in numerous industries in addition to the concrete industry as previously discussed. A brief description of specific examples of how geopolymer systems can be used in several industries are as follows:

2.6.1 In-Situ Concrete Industry

A geopolymer paste is suitable as a partial or full replacement of the cement slurry in concrete products and is currently used commercially in this capacity by Iveron Materials in Florida as well as by Zeobond in Australia. For in-situ concrete applications the three critical design considerations are the set time, the strength, and how safe the material is to handle. Two common concerns about the use of geopolymer paste as a cement replacement in concrete are how it will impact steel rebar, and if alkali-aggregate reactions will occur from their use (Davidovits, 1994)

2.6.2 Precast Concrete Industry

The precast concrete industry can benefit even more than the in-situ concrete industry from the use of geopolymers. This industry already has the required system in place to produce optimal geopolymer concrete due to the capability of curing specimens at elevated temperatures. Exposure to elevated temperature curing has been demonstrated to produce higher strength geopolymers much faster than ambient conditions (Chindaprasirt et al., 2007). Precast geopolymer concrete is currently being produced commercially by Antonello Precast Concrete in Australia.

2.6.3 Composites Industry

Geopolymer pastes are excellent resins for heat and fire resistant composites. Carbon fiber reinforced geopolymer composites were found to be an ideal material for use in aircrafts due to their high specific strengths, high temperature capabilities, and non-combustibility (Federal Aviation Administration, 1981). A recent patent filed by Airbus indicates they will be using geopolymer composites to make conduits in areas of the plane that see high temperatures, like the power plant PQ Corporation. Another industry currently using carbon fiber-reinforced geopolymer composites is the racing industry, where the geopolymer composites have replaced titanium in the exhaust systems of several Formula One and Indy Car race cars.

2.6.4 Architectural Industry

Geopolymer pastes can be used in combination with a filler to create decorative stone wall tiles and pavers. Geopolymer pavers have very good wear resistance compared to some other typical pavers such as synthetic marble. In addition to this, they are stable to ultraviolet and infrared radiation (Davidovits, 1988).

2.6.5 Thermal Insulation

Foamed geopolymer pastes with mica fillers have been shown to be a very effective thermal insulator, especially when very high temperature thermal insulation is necessary (Lizcano et al., 2012).

2.6.6 Pharmaceutical Industry

In industry, geopolymers have been proposed and tested as a high strength pellet to contain and allow for controlled release of highly potent opioids used to treat chronic pain (Kovalchuk et al., 2007). For this application the rate at which the drug is released is a critical parameter to control. Too high of a rate could be fatal, and too low of a rate could prove ineffective. It is also critical for the pellet to have a fairly high strength in order to prevent rapid release of the entire dose due to accidental breakage from chewing, and also to deter recreational abuse of these drugs by crushing them. Only certain base materials can be used in this application due to concerns of toxicity.

2.6.7 Toxic Waste Immobilization

For this application, an encapsulation matrix with an extremely low leach rate is imperative. A study on this particular application indicates that geopolymers are capable of immobilizing uranium waste (Ra-226) as well as several heavy metals such as mercury and lead (Criado et al., 2010)

2.7 Compressive Strength of Geopolymer Concrete

Barbhuiya et al. (2009) studied the influence of using calcium hydroxide with silica fume to produce concretes and substituted by 30% FA of the OPC based content. The amount of silica fume used was 5% by mass of the OPC content. The amount of hydrated lime was about (0.05) to total mass of the binder cementious (FA+ OPC) materials. Room curing was used at first 24 hrs, at 20°C. They see that with increasing the amount of hydrated lime the workability decreased a super plasticizer added. Early compressive strength of the GPC mixes was found increased due to the addition of calcium hydroxide and silica fume. For age 72 hrs. it was show that the strength of hydrated lime with silica fume mixes was greater by 6 MPa than the standard concrete mix mesasured 24 Mpa.

The specimens were cured at three different temperatures of 60° C, 80°C and 100°C for 24 h in the oven, for the process needs curing at high temperature. They were then left at open air (room temperature 25°C) in the laboratory until testing. Tests were carried out on triplicate specimens and average compressive strength values were recorded. From the table 2.5, it can be seen that the geopolymer concrete cured at 80°C gives the best results.

Table 2.3 compressive strength

Days	Compressive strength (Mpa) for 50%NaOH+50%KOH					
	M25	60°	80°	100°	12M NaOH	12M KOH
1	4.92	26.84	31.14	29.9	20.14	23.1
7	25.36	34.74	37.22	36.12	31.05	33.16
14	28.42	42.38	48.86	44.08	35.38	39.12
28	30.33	50.24	55.26	52.18	39	42.44

The compressive strength result for 1st, 3rd, 7th and 28th days of testing are shown in Figure 2.8 reported in the study of (Nuruddin et al., 2011). For testing, 12M NaOH solutions demonstrated the highest compressive strength of 47.83, 48.52, 49.44 and 51.52 Mpa respectively. It was observed that an increase in compressive strength from 8M to 12M but decreased from 12M to 14M for all days of testing.

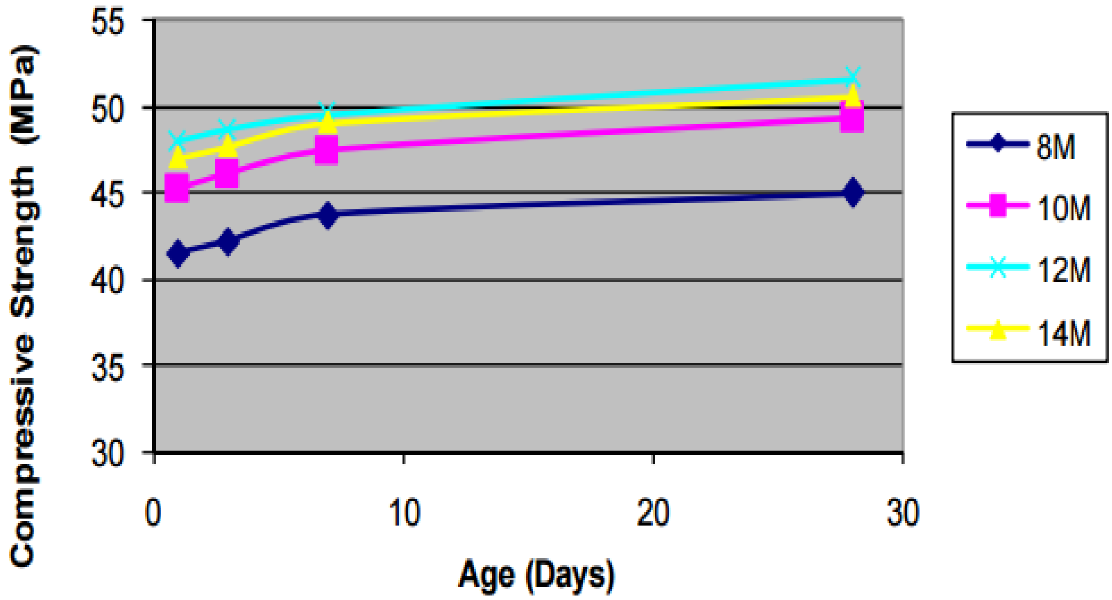

Figure 2.5 influnce of NaOH molarity on compressive strength (Nuruddin et al., 2011)

The early age compressive strength at the age of 3 days for the mix with 14 M was found between 55 and 77% of the 28-day strength and this is higher than the corresponding strength of mixes with 12 M shown in Figure 2.9, development of early age compressive strength.

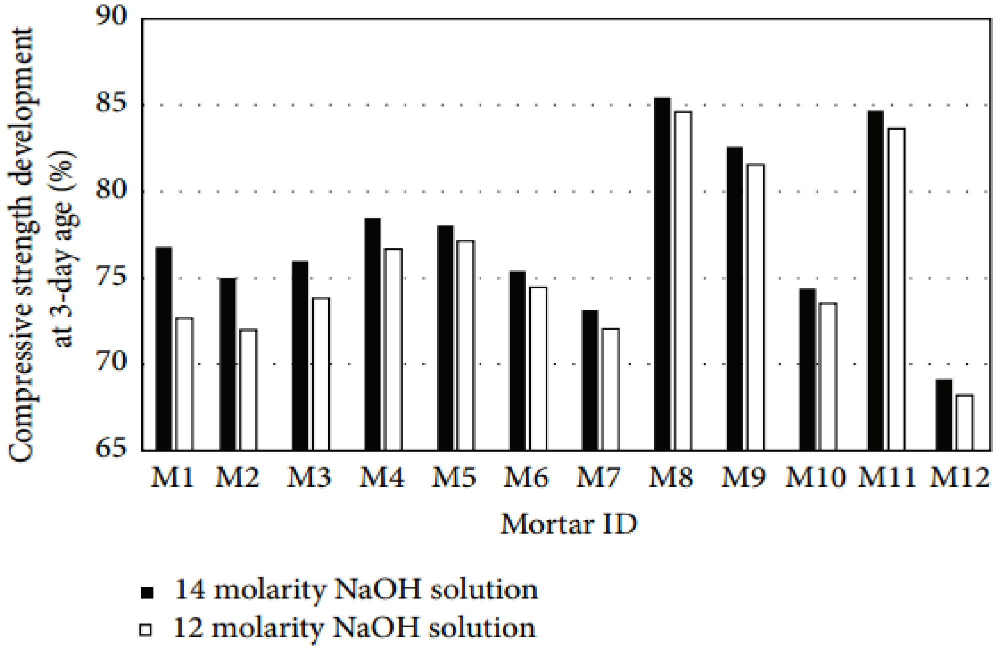

Figure 2.6 Early age compressive strength (Nuruddin et al., 2011)

2.8 Analysis of Variance (ANOVA)

The use of statistical Analysis (ANOVA) have been demonstrated as useful and applicable. The models have considerable advantages; they are generally robust and produce powerful tests (Hill & Lewicki, 2007). ANOVA-models concern to a class of linear models suitable when modeling a continuous response variable against one or several qualitative explanatory variables, generally called factors, that are measured either on a nominal or ordinal measurement scale.

To determine how the value of the response variable is altered by the manipulation of factors it is by utilize ANOVA-models, but foremost to study differences in means between factor levels (Sawyer, 2009). Frank Yates presented methods for unbalanced data analysis in the 1930's (Herr, 1986). Following in the footsteps of Yates, numerous authors have been addressing unbalanced data in ANOVA-models, some of the more recent being Fujikoshi (1993), Weber and Skillings (2000), Rencher (2000), Bao & Ananda (2001) and Langsrud (2003).

2.8.1 Univariate Analysis of Variance

To estimate the factor level means, it is necessary to observe several outcomes, called replicates, given a certain combination of factor levels. If the number of replicates for each factor combination is equal, the data is referred to as balanced.

However it is often the case that the number of replicates varies over factor levels. ANOVA is a tool for estimating the effects of factors on a continuous response variable with the goal of detecting differences in means for different factor categories, called levels (Sawyer, 2009).

Effects models are part of a larger set of general linear models including random effects models and mixed models. Thus, since factors are assumed to be fixed, levels of factors are not considered to be random samples from a larger populations of levels. Hence, inference from fixed effects models is only valid within the specific population and factors included in the model (Sawyer, 2009).

The ANOVA-model relies on several assumptions:

- Normality: The observed sample is assumed to be drawn from a normally distributed population.
- Independence: Observations in the observed sample are independent of each other.
- Homoscedasticity: The variance-covariance matrices are equal across levels of factors.

CHAPTER 3

EXPERIMENTAL STUDY AND METHODOLOGY

3.1 Introduction

This chapter deals with the process of producing FA based geopolymer with different molarity of NaOH. Two ratios of alkaline activator based on ratio of Na_2SiO_3 to NaOH were used. The strength development of the produced geopolymer mortars were evaluated. The results were analyzed using statistical software.

3.2 Materials

3.2.1 Fly Ash (FA)

In this research dry FA (ASTM low calcium, Class F) was used as the base materials, obtained from local power plant. The table below show the chemical and physical properties of FA used in research was obtained as a demonstrated (Table 3.1)

Table 3.1 Chemical and physical properties of FA

Physical and chemical analysis (%)	FA
CaO	2.2
SiO_2	57.2
Al_2O_3	24.4
Fe_2O_3	7.1
MgO	2.4
SO_3	0.3
K_2O	3.4
Na_2O	0.4
Loss on ignition	1.5
Specific gravity	2.25
Specific surface area (m^2/kg)	379

3.2.2 Alkali Activator

In this research a mix of sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) solution was chosen alkaline activator. They were selected based on the activation of sodium because it was cheaper than potassium. Sodium hydroxide used has technical grade in the form of flakes (3 mm), with a specific gravity of 2.130, 98% purity, and PH 14. The molar mass is 40 g / mol and the materials were obtains from a local supplier.

Figure 3.1 Sodium Hydroxide

To obtain the solution of (NaOH), the flakes were dissolved in water by proper amount depending on the chemical proportions to get proper molarity of the solution. The main purpose of the research is the effect of NaOH on the strength of geopolymer mortar. Therefore different concentrations of NaOH were obtained. Solid mass of sodium hydroxide in solution varied depending on the concentration of the solution in terms of the molarity M. For example, sodium hydroxide solution concentration 8M consists 262 grams of solid sodium hydroxide (in flake or pellet form) per liter of the solution, where 40 is the molecular weight of sodium hydroxide. Similarly, the mass of solidy sodium hydroxide measuring per kilogram of solution are 10M: 313 grams, 12M: 361 grams, 14M: 404 grams, 16 M: 444 grams. Note that the mass of solids as sodium hydroxide is only part of the mass of the sodium hydroxide solution, and water is the main ingredient. Figure 3.2 shows the sodium hydroxide solution

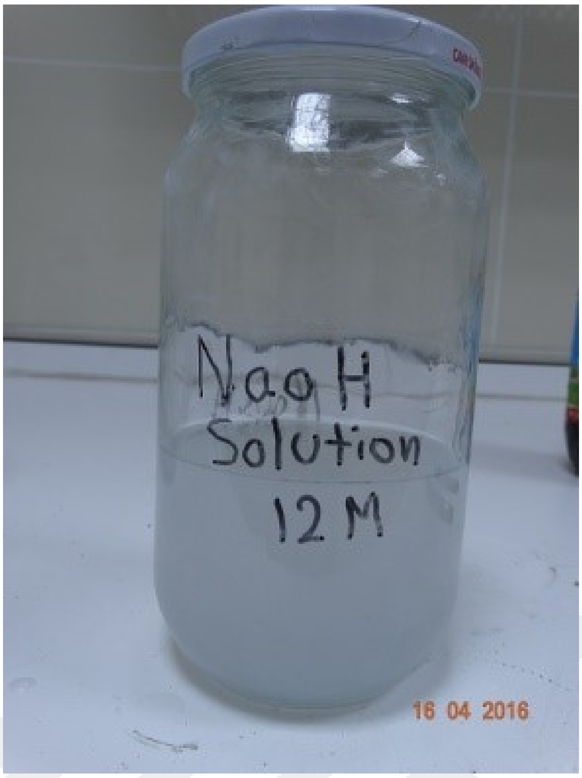

Figure 3.2 Sodium Hydroxide Solution

The chemical composition of a solution of sodium silicate Na_2O = 14.7%, SiO_2 = 29.4%, and 55.9% of the water mass. The other characteristics of a solution of sodium silicate are specific gravity 1.48 and viscosity at 20^{o}C 400 CP

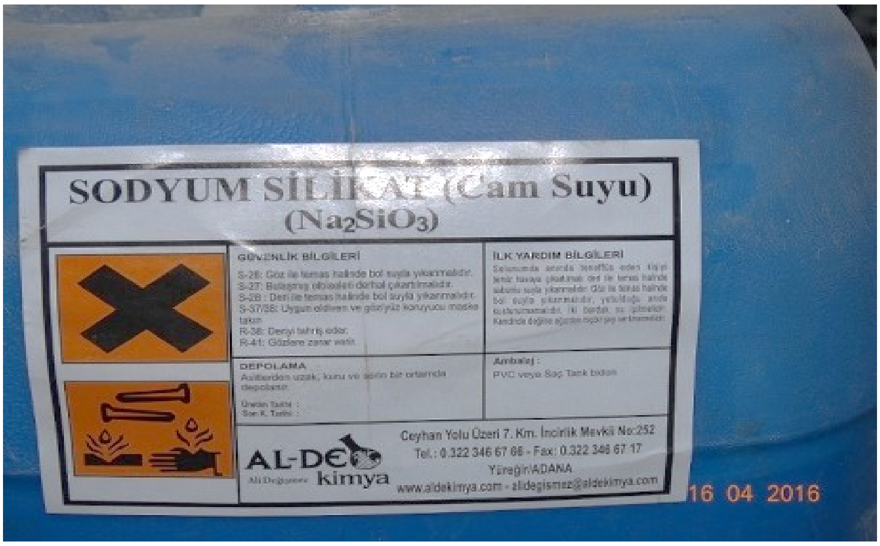

Figure 3.3 Sodium Silicate

3.2.3 Fine Aggregate

Aggregate were used in research was provided from local river quarry (river sand), just fine aggregate was used (0-4 mm), sieve of size (4 mm) was used to obtain aggregate grade from (0-4 mm). Aggregate was stored in laboratory specified gravity of aggregate obtained according to ASTM by using a sample of aggregate and weigh of 250 gram by using clean water and glass can. The specific gravity was (2.64).

Figure 3.4 Aggregate

3.2.4 Superplasticizer

To provide the workability of the mortar polycarboxylate ether type chemical admixture was used by amount of 6 % of FA weight in all mixtures.

Table 3.2 Properties of superplasticizer

Properties	Superplasticizer
Name	Glenium 51
Color tone	Brown
State	Liquid
Specific gravity (kg/1)	1.07
Chemical description	Polycarboxylate ether

3.3 Mix Proportion

The primary difference between geopolymer mortar and Portland cement concrete is the binder. Table 3.3 presents the composition of the mix designs.

Table 3.3 Geopolymer Concrete Mix Designs

NaOH : Na_2SiO_3 ratio (%)	Molarity	Na_2SiO_3 solution (kg/m^3)	F A (kg/m^3)	River sand aggregate (kg/m^3)	NaOH solution (kg/m^3)	Superplasticizer (kg/m^3)
2	6	225	600	1295.747	75	36
2	8	225	600	1291.544	75	36
2	10	225	600	1286.95	75	36
2	12	225	600	1281.953	75	36
2	14	225	600	1276.511	75	36
2	16	225	600	1271.271	75	36
3	6	517.5	1380	2980.218	172	82
3	8	517.5	1380	2970.552	172	82
3	10	517.5	1380	2959.984	172	82
3	12	517.5	1380	2948.492	172	82
3	14	517.5	1380	2425.372	172	82
3	16	517.5	1380	2415.415	172	82

3.4 Preparation of Fresh Mortar, Casting and Curing

A total 12 mixtures were prepared by varying Na_2SiO_3/NaOH ratio and molarities. The binder and the sand were first mixed together in a rotary mixer for about 2 min. The alkaline liquid was then added to the dry materials and the mixing was continued for further 5 min to produce the fresh mortar as shown in Figure 3.5.

Figure 3.5 Fresh mortar of geopolymer

The fresh mortar was compacted and to achieve mortar with less air voids. The molds were covered by plastic film to avoid evaporation of alkaline solution. For each mortar mixture, set of 12 specimen by dimension of (50x50x50) mm cube were cast to determine the compressive strength.

Figure 3.6 Preparation of mortar and casting

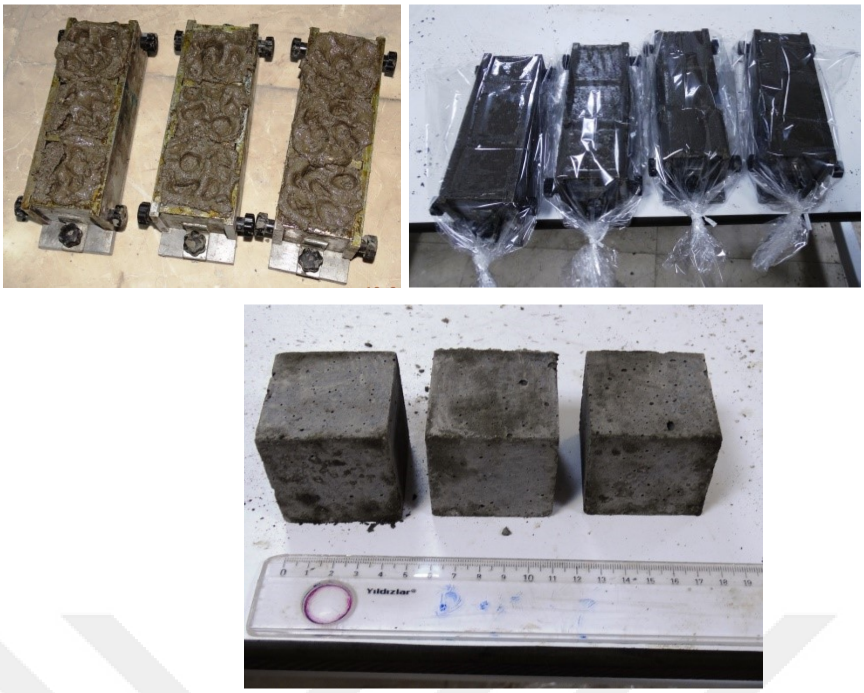

Figure 3.7 Preparation of mortar and casting

Immediately after casting, the test specimens were covered with plastic film to minimize the alkaline activator evaporation during curing at an elevated temperature as shown in Fig. 3.7. The test specimens were cured in an oven at 90°C for 2, 6, 8, 24, 48 and 72 h. After the curing period, the test specimens were left in the molds and demolded.

3.5 Testing

3.5.1 Compressive Strength

The compressive strength test of geopolymer mortar was obtained by means of 3000 KN capacity hydraulic testing machine by 50x50x50 mm cubes and tested according to ASTM C39 (2012), the test was performed on the test specimens at the age of 2, 6, 8, 24, 48 and 72 hrs. With loading rate 0.5 Mpa/sec. The compressive strength was calculated from average of three specimens at each testing age.

Figure 3.8 Compressive strength test

3.6 Analytical Techniques

To evaluated compressive strength, in terms of amount of NaOH to be used for gaining highest compressive strength, the data was subjected to an analysis by using statistical software MINITAB16 for analysis of variance (ANOVA). This method determines the size of the total variation in results and distinguishes random variation of the contribution of each variable. For statistical analysis ($P < 0.05$) was used as traditional level of importance.

3.6.1 ANOVA (Analysis of Variance)

ANOVA helps us to compare variability's within experimental data. In this research ANOVA table is made with help of **MINITAB 16** software. When performance varies 1 determines the average loss by statistically averaging the quadratic loss. The average loss is proportional to the mean squared error of Y about its target T. The initial techniques of the analysis of variance were developed by the statistician R. A.

Fisher in the 1920s and 1930s, and are sometimes known as Fisher's ANOVA or Fisher's analysis of variance, due to the use of Fisher's F-distribution as part of the test of statistical significance.

CHAPTER 4

RESULT AND DISCUSSION

4.1 Influence of Molarity on the Strength of FA based Geopolymers

For this experiment mortar of geopolymer manufactured by the polymeric reaction of FA and alkali activated solution is used. The effect of molarity on compressive strength of geopolymer mortar have revealed as below

- When the molarity increases, compressive strength increase with rate of (Na_2SiO_3/NaOH solution = 2) excepting 14M and 16M.It has been shown in Fig (4.1).
- The rate of (Na_2SiO_3/NaOH solution = 3) it has been demonstrated that as the molarity increases, compressive strength increase for all molarities. Fig (4.2)

It can be realised that Mix with molarity 12M demonstrated higher strength than the other mixtures. The highest strength in 12M mix in the age of 72 hrs is found 44 MPa. The lowest strength in 16M mix at the age 72 hrs is found to be lower than mix with molarity 12M. Compressive strength developments of the mortars are illustrated in Fig. 4.1;

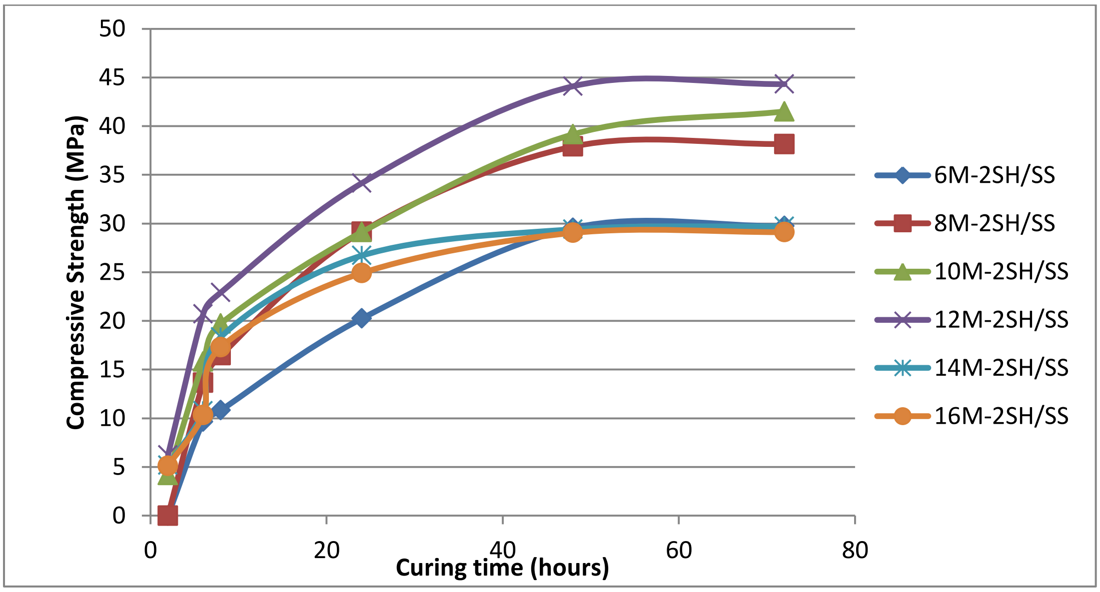

Figure 4.1 Compresive strengh of geopolymer with rate of (Na_2SiO_3/NaOH solution =2)

Figer 4.2 demostrated the increase in the molarity leads to increase the compressive strength in the rate of $Na_2SiO_3/NaOH$ solution =3, it was observed the mix with molarity 6M reported higher strength in the age 72 hrs.

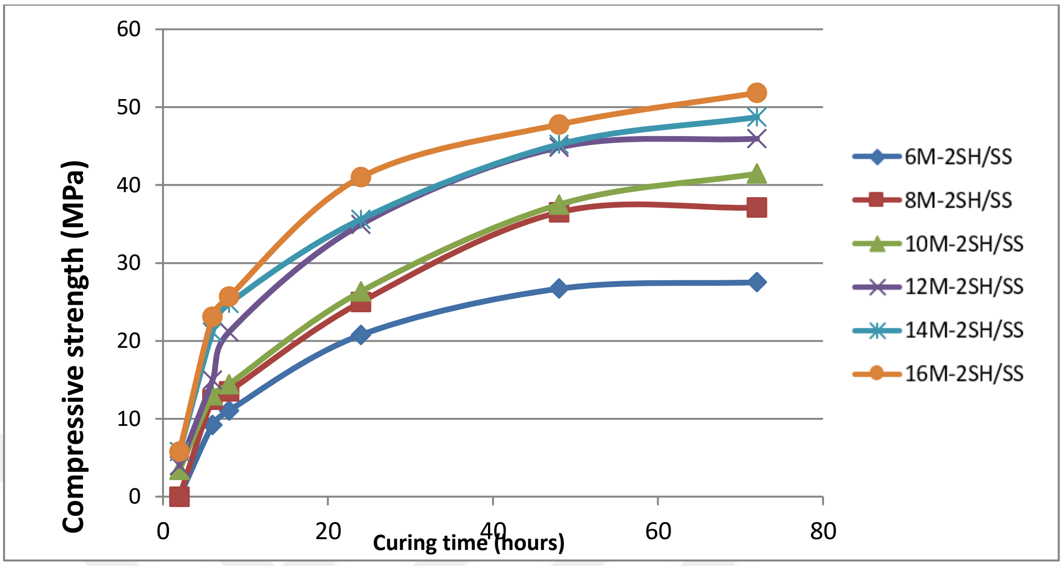

Figure 4.2 Influnce of mass ratio of Na2SiO3/NaOH on strength develepment of FA geoplymers

4.2 Influnce on Strength Development of FA Geopolymers by Mass Ratio of $Na_2SiO_3/NaOH$

Figures (4.3 to 4.8) indicates the lowest Na_2SiO_3 content is detected from the samples with $Na_2SiO_3/NaOH$ ratio of 2.0 in the (M14 and M16). This ratio composed the poorest strength development among the other ratios at all tested ages and thus presents the lowest geopolymerization reaction. Although, at the highest Na_2SiO_3 solution from the samples with a $Na_2SiO_3/NaOH$ mass ratio of 0.3, the geopolymer exhibits relatively poor strength development at the tested ages in the M6, M8, M10 and M12. The strength development in the specimens with $Na_2SiO_3/NaOH$ mass ratios of 3.0 and 2.0 mention the complexity of the geopolymerization regression and the significance of the concentration of the alkaline activator constituents.

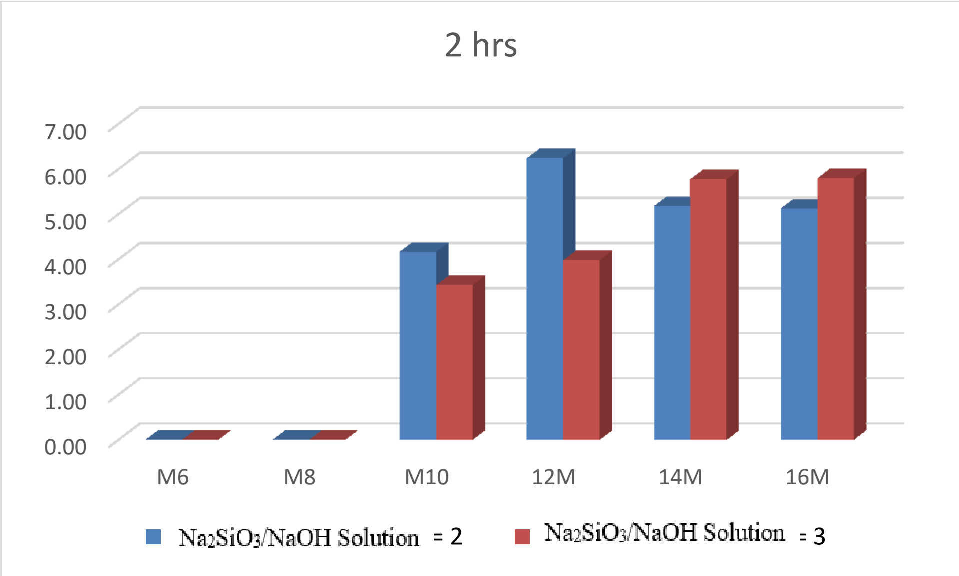

Figure 4.3 Compresive strengh of geopolymer mortar at 2 hrs

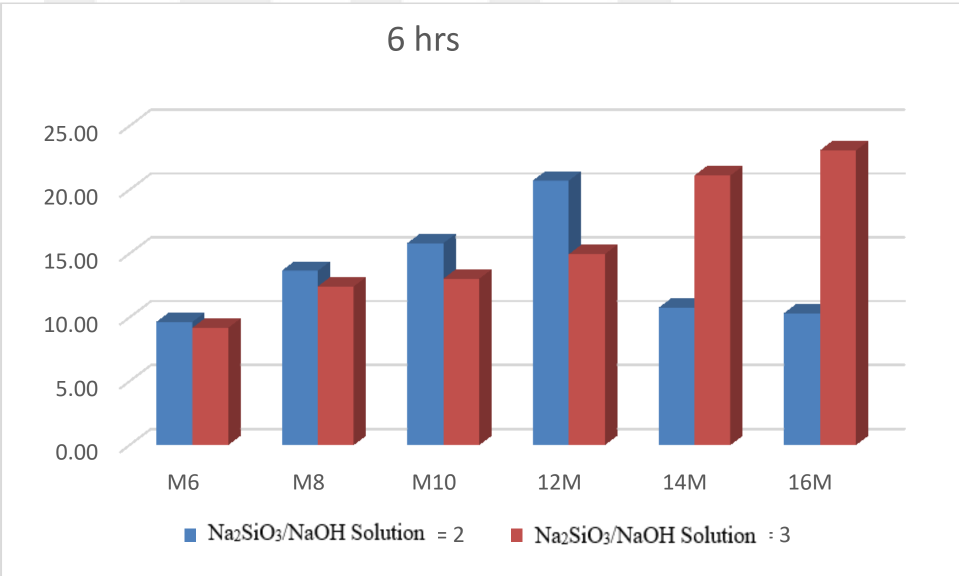

Figure 4.4 Compresive strengh of geopolymer mortar 6 hrs

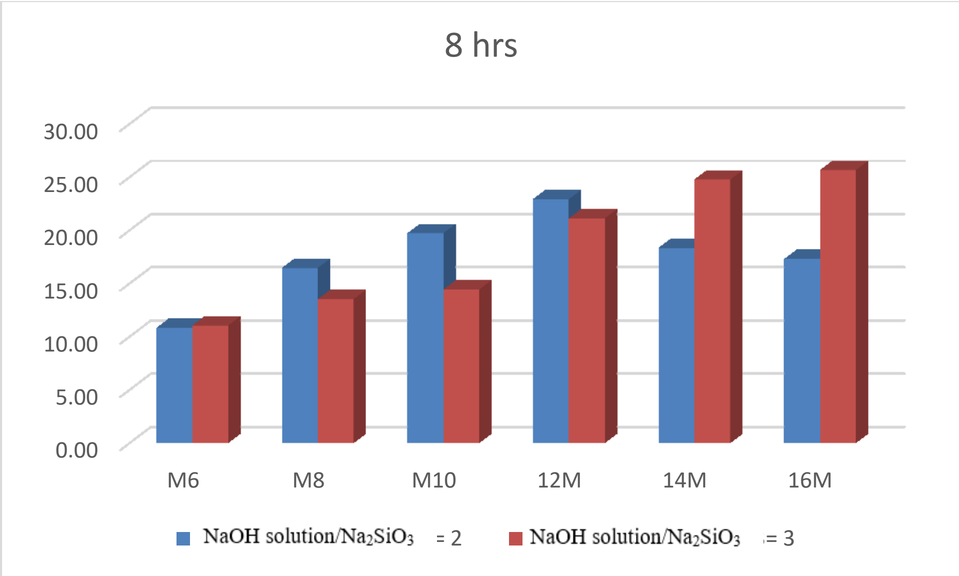

Figure 4.5 Compresive strengh of geopolymer mortar at 8 hrs

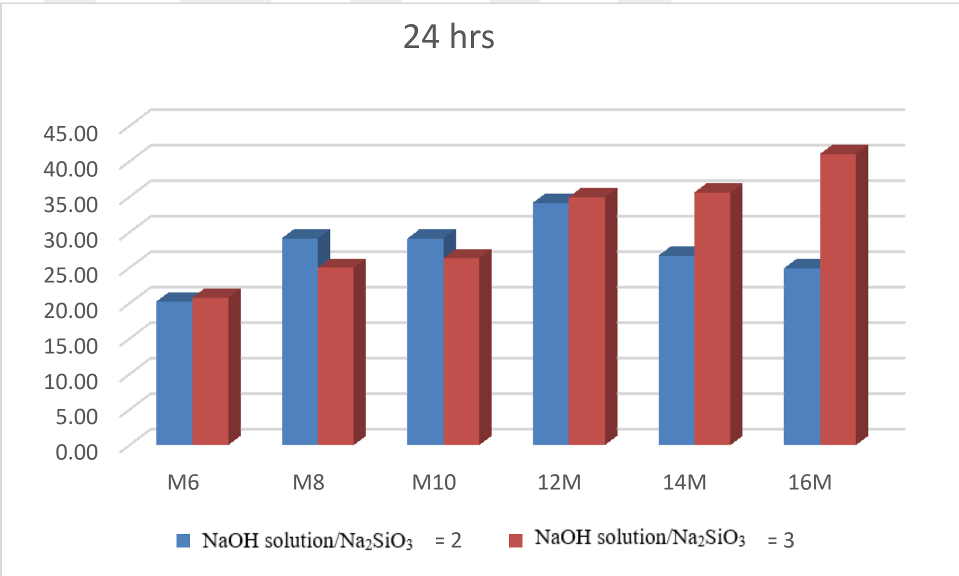

Figure 4.6 Compresive strengh of geopolymer mortar at 24 hrs

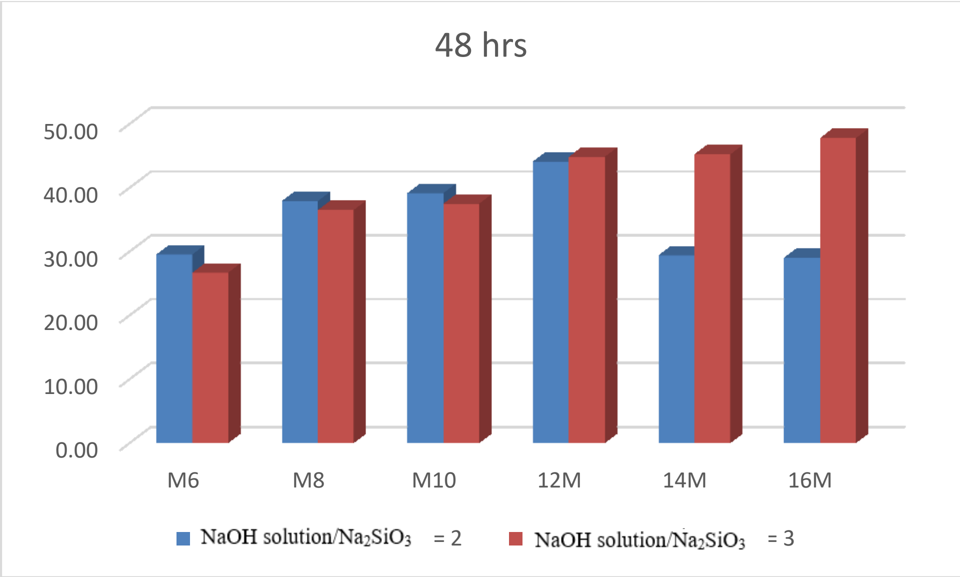

Figure 4.7 Compresive strengh of geopolymer mortar at 48 hrs

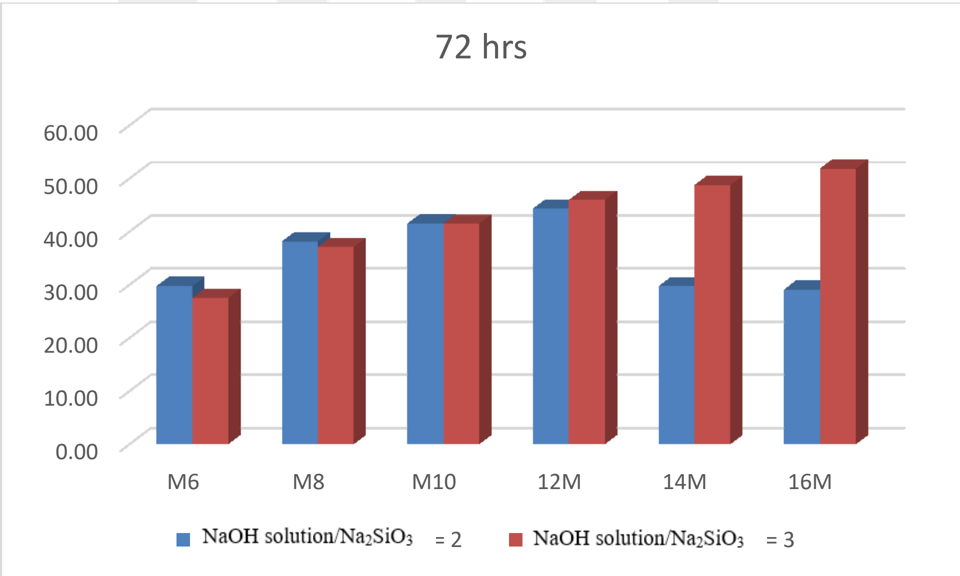

Figure 4.8 Compresive strengh of geopolymer mortar at 72 hrs

4.3 ANOVA and Effect of the Factors

In this study the ANOVA was utilized to analyze the influence of Na_2SiO_3 rate, duration and molarity on compressive strength. ANOVA is a statistical method used to determine the individual interactions of all control factors. The percentage distributions of each control factor were utilized to measure the corresponding influence on the quality characteristics.

The performed experimental plan was determined at 95% of confidence level. ANOVA values belonging to experimental results el of $Na_2SiO_3/NaOH$ rate (X_1), duration (X_2), molarity (X_3) and compressive strength (Y).

4.3.1 Regression Equation

The equations of compression strength were generated based on the control factors and their interactions.

General Regression Analysis: y versus x_1, x_2, x_3

Regression Equation:

$$Y = -5.71084 + 2.91764\, X_1 + 0.450419\, X_2 + 0.899768\, X_3 \quad (4.1)$$

Table (4.1) demonstrates regression equation of the analysis of variance (ANOVA). The data can be collect from table the coefficients SE- coefficients for all factors over responses. P value more than 0.050 indicate that $Na_2SiO_3/NaOH$ rate (X_1), can be ignored. While for other parameters the P value is less than 0.050.

Table 4.1 Coefficients of regression equation

Term	Coef.	SE Coef.	T	P
Constant	5.71084	5.32448	-1.0727	0.287
X1	2.91764	1.73978	1.6770	0.098
X2	0.45042	0.03411	13.2033	0.000
X3	0.89977	0.25468	0.001	0.001

4.3.2 General Linear Model for Compressive Strength

The analysis of variance (ANOVA) for compressive strength (Y) response, it demonstrated in Table (4.2). The important data can be collect here is the percentage effect of all factors over responses. P value less than 0.05 indicate model terms are significant. In this case $Na_2SiO_3/NaOH$ rate is significant model term.

The coefficient of determination (R^2) which indicates the goodness of fit for the model so the value of R^2 =0.915 which indicate the high significance of the model.

Table 4.2 Analysis of Variance for y, using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P
X1	1	153.2	153.2	153.2	7.74	0.007
X2	5	11660.9	11660.9	2332.2	117.82	0.000
X3	5	1034.2	1034.2	206.8	10.45	0.000

S = 4.44902

R-Sq = 0.915

R-Sq (adj) = 0.899

R-Sq called coefficient of determination indicates explanatory power of any regression model. Its value lies between +1 and 0. It can also been shown that R –sq is the correlation between actual and predicted value. It will reach maximum value when dependent variable is perfectly predicted by regression equation.

Figure 4.9 signifies that the residual follows a straight line and there are no unusual patterns or outliers. Moreover, the hypothesizes regarding the residual were not violated and the residuals are normally distributed.

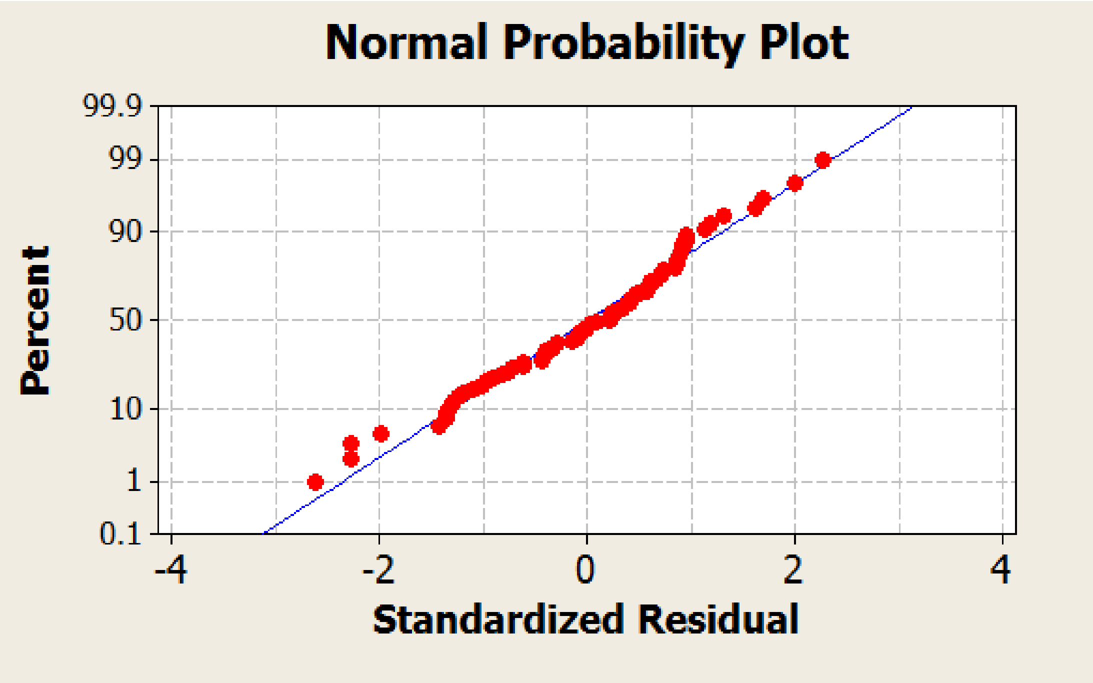

Figure 4.9 Residual plot for Y (compressive strength)

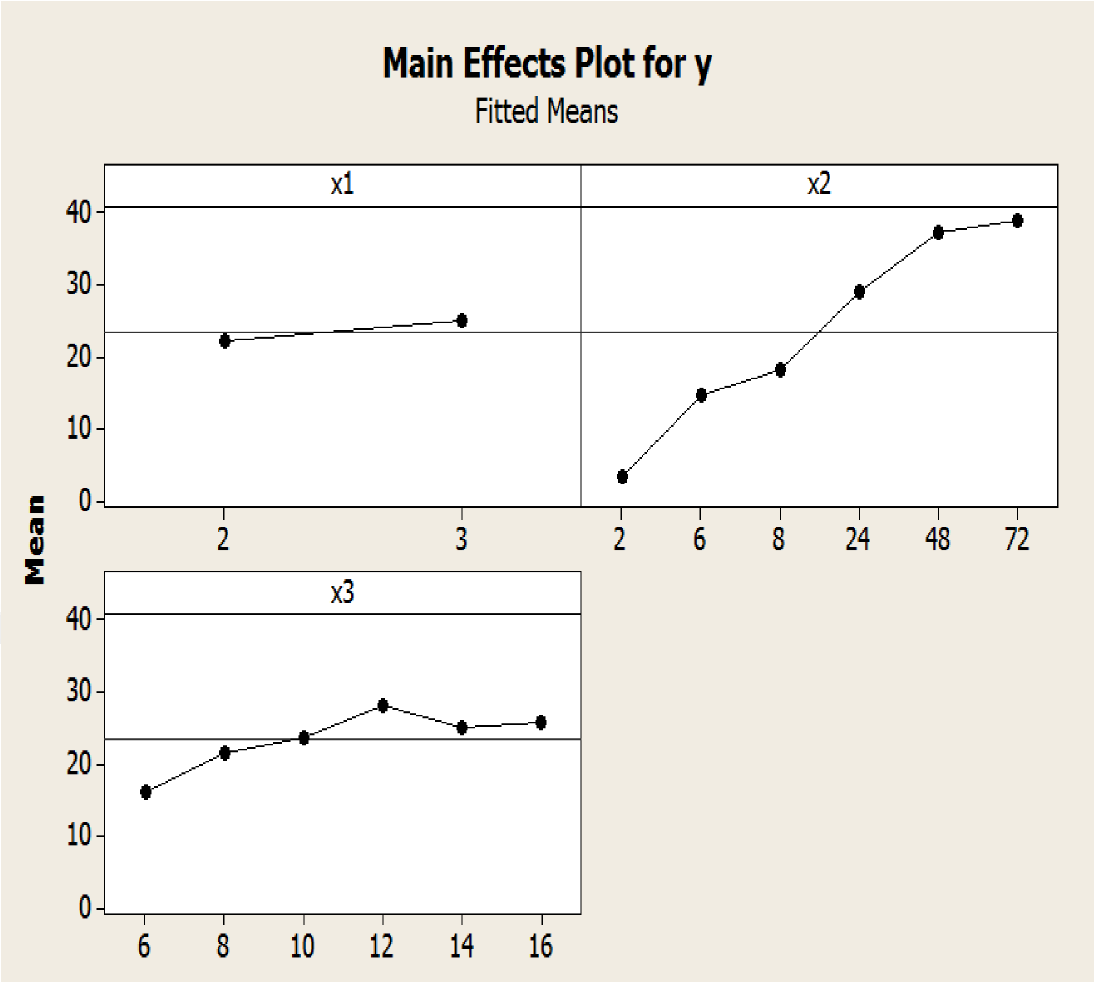

Figure 4.10 Main Effects plot for Y (Compressive strength) $Na_2SiO_3/NaOH$ rate (X_1), duration (X_2), Molarity (X_3).

From figure 4.10 it was observed that with increase in $Na_2SiO_3/NaOH$ rate (X_1) and with the duration (X_2) the compressive strength is increasing. While in part of the molarity (X_3) demonstrate the 12M has high compressive strength in the module.

Analysis of variance (ANOVA) demonstrates that the duration (X_2) have the highest influence on the compressive strength

CHAPTER 5

CONCLUSION

The results of the study carried out on FA-based geopolymer concrete was reported. The following conclusions are drawn from this study:

- The results illustrate that the combination of the above constituents at 90°C has a positive effect on the strength of geopolymer mortar.
- The molarity increases, compressive strength increase with rate of (Na_2SiO_3/NaOH solution = 2) excepting 14M and 16M. It has been shown that in Fig 4.1
- While with rate of (Na_2SiO_3/NaOH solution = 3) the molarity increases, compressive strength increase for all molarities. As shown in Fig 4.2.
- The coefficient of determination (R^2) which indicates the goodness of fit for the model so the value of R^2 0.915% which indicate the high significance of the model.
- The duration (X_2) have the highest impact on the compressive strength from Analysis of variance (ANOVA).
- P value more than 0.05 indicate that Na_2SiO_3/NaOH rate (X_1), may not be not significant. While for other variation the P value is less than 0.05, it indicates significance. This contradiction may be clarified by conducting further experimented study to generate more data samples.

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APPENDIX A
Experimental Data

Table A.1 Experimental Data

#	$Na_2SiO_3/NaOH$ X_1	Duration X_2	Molarity X_3	Comp. st Y
1	2	2	6	0
2	2	2	8	0
3	2	2	10	4.16
4	2	2	12	6.24
5	2	2	14	5.17
6	2	2	16	5.12
7	2	6	6	9.64
8	2	6	8	13.65
9	2	6	10	15.81
10	2	6	12	20.71
11	2	6	14	10.74
12	2	6	16	10.33
13	2	8	6	10.83
14	2	8	8	16.46
15	2	8	10	19.72
16	2	8	12	22.92
17	2	8	14	18.34
18	2	8	16	17.3
19	2	24	6	20.23
20	2	24	8	29.15
21	2	24	10	29.12
22	2	24	12	34.14
23	2	24	14	26.70
24	2	24	16	24.91

#	Na_2SiO_3/NaOH X_1	Duration X_2	Molarity X_3	Comp. st Y
25	2	48	6	29.55
26	2	48	8	37.90
27	2	48	10	39.16
28	2	48	12	44.1
29	2	48	14	29.40
30	2	48	16	29.03
31	2	72	6	29.73
32	2	72	8	38.16
33	2	72	10	41.51
34	2	72	12	44.31
35	2	72	14	29.72
36	2	72	16	29.11
37	3	2	6	0
38	3	2	8	0
39	3	2	10	3.42
40	3	2	12	3.98
41	3	2	14	5.77
42	3	2	16	5.79
43	3	6	6	9.18
44	3	6	8	12.43
45	3	6	10	13.03
46	3	6	12	14.95
47	3	6	14	21.11
48	3	6	16	23.08
49	3	8	6	11.05
50	3	8	8	13.53
51	3	8	10	14.43
52	3	8	12	21.12
53	3	8	14	24.77
54	3	8	16	25.66
55	3	24	6	20.77

#	$Na_2SiO_3/NaOH$ X_1	Duration X_2	Molarity X_3	Comp. st Y
56	3	24	8	24.99
57	3	24	10	26.34
58	3	24	12	34.95
59	3	24	14	35.61
60	3	24	16	41.02
61	3	48	6	26.69
62	3	48	8	36.50
63	3	48	10	37.5
64	3	48	12	44.84
65	3	48	14	45.22
66	3	48	16	47.78
67	3	72	6	27.52
68	3	72	8	37.10
69	3	72	10	41.47
70	3	72	12	45.95
71	3	72	14	48.73
72	3	72	16	51.85

APPENDIX B
Result of Minitab16 program

General Regression Analysis: y versus x1, x2, x3

Regression Equation

```
y  =  -5.71084 + 2.91764 x1 + 0.450419 x2 + 0.899768 x3
```

Coefficients

Term	Coef	SE Coef	T	P
Constant	-5.71084	5.32448	-1.0726	0.287
x1	2.91764	1.73978	1.6770	0.098
x2	0.45042	0.03411	13.2033	0.000
x3	0.89977	0.25468	3.5330	0.001

General Linear Model: y versus x1, x2, x3

Factor	Type	Levels	Values
x1	fixed	2	2, 3
x2	fixed	6	2, 6, 8, 24, 48, 72
x3	fixed	6	6, 8, 10, 12, 14, 16

Analysis of Variance for y, using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P
x1	1	153.2	153.2	153.2	7.74	0.007
x2	5	11660.9	11660.9	2332.2	117.82	0.000
x3	5	1034.2	1034.2	206.8	10.45	0.000
Error	60	1187.6	1187.6	19.8		
Total	71	14036.0				

S = 4.44902   R-Sq = 91.54%   R-Sq(adj) = 89.99%

Unusual Observations for y

Obs	y	Fit	SE Fit	Residual	St Resid	
30	29.0367	38.2767	1.8163	-9.2400	-2.28	R
35	29.7200	38.9250	1.8163	-9.2050	-2.27	R
36	29.1100	39.7326	1.8163	-10.6226	-2.62	R
60	41.0233	32.8828	1.8163	8.1406	2.00	R
72	51.8500	42.6503	1.8163	9.1997	2.27	R

R denotes an observation with a large standardized residual.

APPENDIX C

Photographic Views

Figure C 1 Photographic view during mortar production

Figure C 2 Photographic view of molded specimens

Figure C 3 Photographic view during preper Alkaline solution

Figure C 4 Photographic view of compressive strength testing