T. R.
VAN YUZUNCU YIL UNIVERSITY
INSTITUTE OF NATURAL AND APPLIED SCIENCES
DEPARTMENT OF CIVIL ENGINEERING

MECHANICAL AND DURABILITY PROPERTIES OF THE GEOPOLYMER CEMENT PRODUCED WITH PUMICE POWDER AND FLY ASH

M.Sc. THESIS

PREPARED BY: Maadh Abdulmuttaleb Hamid HAMID
SUPERVISOR: Asst. Prof. Dr. Namık YALTAY
CO-SUPERVISOR: Asst. Prof. Dr. Mehmet TÜRKMENOĞLU

VAN-2021

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ACCEPTANCE and APPROVAL

This thesis entitled " **Mechanical and Durability Properties of Geopolymer Cement Produced with Pumice Powder and Fly Ash** " presented by Maadh Abdulmuttaleb Hamid HAMID under supervision of Asst. Prof. Dr.Namık YALTAY in the department of civil engineering has been accepted as a M. Sc. thesis according to Legislations of Graduate Higher Education on 9 / 4 / 2021 with unanimity majority of votes members of the jury.

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Maadh Abdulmuttaleb Hamid HAMID

ABSTRACT

MECHANICAL AND DURABILITY PROPERTIES OF THE GEOPOLYMER CEMENT PRODUCED WITH PUMICE POWDER AND FLY ASH

HAMID, Maadh Abdulmuttaleb Hamid
M.Sc. Thesis, Civil Engineering
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A new and improved invention of binder materials has become an urgent need in order to reduce reliance on Portland cement while minimizing environmental pollution and energy consumption associated with its manufacture. Geopolymer cement can be used as a possible alternative. Its production requires use of silica-alumina materials, an alkaline-activator solution, and a curing time. In this research, pumice powder and fly ash, both high in silica and alumina have been used to produce geopolymer paste. Six pumice-fly ash percentages and six alkaline solution-to-binder (A/B) ratios were used. NaOH as an alkaline activator solution was constant at a concentration of 12M with curing at 60°C for 2, 4, and 6 days. Several laboratory tests such as consistency, strength tests, and UPV test as well as for elevated temperature resistance and freezing-thawing cycles resistance tests were used to investigate the behavior, performance of various geopolymer mixtures. The results confirm that pumice powder with fly ash can be utilized to create good geopolymeric binders with optimum compressive strengths of (31.02, 32.03, 36.76, 50.43, 57.17, and 69.90 MPa) for mixtures (P100-R0.46, P80-R0.46, P60-R0.42, P40-R0.38, P20-R0.34, and P0-R0.30) respectively. The results revealed that the increase in the content of fly ash led to the development of the different properties of geopolymer samples tested. The best curing time for the best values was found in 6 days. The results also showed that the alkaline solution to binder ratios and consistency had a significant effect on the geopolymers.

Keywords: Alkali activator, Curing time, Fly ash, Geopolymer, Pumice powder.

ÖZET

POMZA VE UÇUCU KÜL İLE ÜRETİLEN GEOPOLİMER BETONLARIN MEKANİK VE DURABİLİTE ÖZELLİKLERİ

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Portland Çimentosunun üretiminin çevre kirliliğine ve yüksek enerji tüketimine neden olması, birçok araştırmacıyı, çimento yerine yeni bir bağlayıcı bulmaya yönlendirmiştir. Silika ve Aluminanın, alkali aktivatör ile kürlenmesi sonucu üretilen Geopolimer bağlayıcılar Çimento yerine kullanılabilecek bir malzeme olarak düşünülmektedir. Bu çalışmada Geopolimer bağlayıcı yüksek oranda silika ve alümina içeren pomza tozu ve uçucu kül, ile üretilmiştir. Bu amaçla 6 farklı yüzde oranında pomza ve uçucu kül karışımı, 6 farklı alkali/bağlayıcı oranında denenmiştir. Alkali aktivatör olarak NaOH çözeltisi, 2,4 ve 6 gün boyunca 60 ° C'de kürleme ile 12M sabit konsantrasyonda kullanılmıştır. Kıvam, basınç dayanımı, eğilme dayanımı ve tahribatsız ulrases hız geçişi testleri ve yüksek sıcaklık ve donma çözülme dirençleri farklı karışımlar için belirlenmiştir. Test sonuçlarına göre pomza tozu ve uçucu kül karışımları içeren geopolimer bağlayıcılar, optimum (31.02, 32.03, 36.76, 50.43, 57.17 ve 69.90 MPa) basınç dayanımlarında sırasıyla (P100-R0.46, P80-R0.46, P60-R0.42, P40-R0.38, P20-R0.34 ve P0-R0.30) karışımları için elde edilmiştir. Uçucu kül oranının artmasıyla geopolimer örneklerinin farklı özelliklerinin iyileştiği görülmüş, en iyi kürleme süresi 6 gün olarak belirlenmiştir. Test sonuçları alkali/bağlayıcı oranı ve kıvamın geopolimer numunelerin özellikleri üzerinde önemli derecede etkili olduğunu göstermiştir.

Anahtar Kelimeler: Alkali activator, Geopolimer Pomza tozu, Kür süresi, Uçucu kül.

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2021

Maadh Abdulmuttaleb HAMID

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SYMBOLS AND ABBREVIATIONS

Some symbols and abbreviations used in this study are presented below, along with descriptions.

Symbols	Description
°C	Degree centigrade
Al	Aluminum
Al_2O_3	Aluminum oxide
$Ca(OH)_2$	Calcium hydroxide
CaO	Calcium oxide
CO_2	Carbon dioxide
Fe_2O_3	Iron oxide
K_2SiO_3	Potassium silicate
Kg/m^3	Kilogram/cubic meter
KN	Kilo-Newton
KOH	Potassium hydroxide
M	Molarity
MgO	Magnesium oxide
mm	Millimeters
MPa	Mega Pascal
Na_2SiO_3	Sodium silicate
NaOH	Sodium hydroxide
Si	Silicon

Abbreviations	Description
A/B	Activator to binder ratio
AAC	Alkali-activated cement
ASTM	American society for testing and materials
BS EU	British Standards Institute
CSA	Canadian Standards Association
FA	Fly ash
GGBS	Ground granulated blast slag
OPC	Ordinary Portland cement
P	Pumice
POFA	Palm oil fuel ash
RHA	Rice husk ash
SFA	Silica fume ash
SS/SH	Sodium silicate to Sodium hydroxide
UPV	Ultrasonic pulse velocity
W/B	Water to binder ratio

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1. INTRODUCTION

1.1. General

One of the problems of Portland cement is that its production contributes nearly (5-7%) CO_2 emission of the global (Benhalal et al., 2013; Romanoa et al., 2013; Singh and Middendorf, 2020) and with the high need for cement due to the increase of population in the world, an increase in infrastructure and industrialization, meanwhile the sustainability and environment issues should be considered (Turner and Collins, 2013). With the previously mentioned drawback of using cement in construction, there are many other points like energy consumption and consuming high quantities of raw materials. Therefore, alternative material binders for Portland cement should be looked for. Geopolymer could be the future alternative for use in building and construction applications and this geopolymer cement could have positive points comparing with Ordinary Portland cement such as decrease carbon dioxide pollution, less cost of production, possibility to use waste materials, and reducing consumption of raw materials (Habert et al., 2011; Davidovits, 2013; Duxson et al., 2007; Lloyd and Rangan, 2010; Zakka et al., 2021).

To create geopolymer cement, alumina-silicate resource materials, alkaline-activator is required with curing temperature (Davidovits, 1991; Van-Deventer et al., 2007). These raw materials could be industrial waste by-products materials such as GGBFS and fly ash or natural raw materials such as kaolinite and calcined clays (Farhan et al., 2020; Ren et al., 2021). The need to protect the natural environment from pollution requires the development of new building materials that depend on waste generated by power plants such as fly ash to reduce their negative impact (Merinkline et al., 2013; Dwivedi and Jain, 2014; Abdul-Aleem and Arumairaj, 2012)

According to previous studies, several factors influence the fresh, hardened properties and interactions of geopolymers material such as chemical composition, fineness of raw materials, alkaline to binder ratio, the concentration of alkaline solution curing time, and temperature (Ibrahim and Maslehuddin, 2021).

Choice of geopolymer cement materials requires studying some factors such as cost, availability, application of type and confirms that their use was economically beneficial in that country in which it is available.

In this study, pumice (a natural material) with fly ash (industrial by-products) was used for the production of the geopolymer cement paste.

1.2. Aim and Objective

The main aim of this search is to explore the possibility of using pumice with fly ash as source material to produce geopolymer cement paste. To obtain environmentally friendly and locally produced products with excellent strength performance and durability properties that can be used in building and construction applications. To achieve this aim we will be pursued the following objectives:-

1. To explore the possibility of using pumice with fly ash as source material for the production of geopolymer cement and the possibility of being a future alternative material binder for Portland cement.
2. Trying to mix different proportions of pumice with fly ash (P%+FA%) and study the effect of this change in properties of geopolymer cement paste.
3. Investigate the influence of different alkali activator solution to binder ratio (A/B) on the strength properties of P-FA geopolymer paste.
4. To experimentally investigate the hardened properties of the product such as the compressive strength and flexural strength and other tests that will be performed.
5. To investigate the effect of the cure time on the strength of P-FA geopolymer paste.
6. To obtain high compressive strength of geopolymer product.
7. To investigate the freezing-thawing durability properties of P-FA geopolymer paste.
8. To investigate the influences of elevated temperature on the P-FA geopolymer samples.

9. To investigate the values of Ultrasonic Pulse Velocity and estimate the correlation equation between them and compressive strength as well as density.

1.3. Research Significance

The research importance of this thesis is to use geopolymer cement as a possible replacement to Portland cement for use in building and construction applications. Creating geopolymer cement requires a silica-alumina material, an alkaline-activator solution, and curing time and temperature. it is a result of a chemical reaction. There are no previous studies about different mixes proportion of pumice with fly ash to produce geopolymer cement paste, there are studies only with fly ash (an industrial by-product) or only with pumice (a natural material), and my study is an attempt to mix natural material with industrial by-products. Portland cement production can therefore be significantly reduced by increasing the use of geopolymer technology, which could potentially reduce greenhouse gas emissions. A large quantity of raw pumice in Turkey, easily obtained at low cost, and the manufacture of its powder produces very little pollution and environmental effects, as opposed to the manufacture of Portland cement. Also, fly ash, as industrial waste to be used in geopolymer cement, will also help to protect the environment from pollution. The finding of this experimental research will be useful in determining the effect of various proportions of (pumice with fly ash), different alkali activator solution to binder ratios (A/B), and curing time on the strength characteristics, as well as the resistance of geopolymer paste to elevated temperature and freezing-thawing cycles. The end of this thesis provides the basis for further studies on the use of this product in certain practical applications.

1.4. Layout of Thesis

The thesis is arranged into five sections:

Section 1: This section includes the introduction, which mainly contains a general background of geopolymers, the aim, and the objective in addition to the significances of this study.

Section 2: This section includes the more relevant literature review to the presented study, contains general background, defines geopolymer cement and geopolymer concrete, and defines source materials and alkaline solutions that used in the research, geopolymer synthesis and chemical structure, general literature about pumice and fly ash and the factors that effect of the geopolymers.

Section 3: Displays the details of the used material in this work and their characterizations, work program, describe the detailed followed methodology of the experimental work, the test procedures and curing conditions were described in this section.

Section 4: Presents the results of all tests with descriptions of the effect of the content percentage of pumice and fly ash, the alkali solution to binder (A/B) ratio, and curing time on the strength of geopolymer pastes. Also, the influence of the freezing-thawing cycle and the elevated temperature resistance were discussed.

Section 5: Summarizes the main conclusions about the results and made some recommendations for further studies.

2. LITERATURE REVIEW

2.1. General

Portland cement is considered to be the best and most important material binder used for various applications. Cement production requires great energy and consumes a lot of raw materials, as well as it is one of the biggest contributors to CO_2 emissions global (Okonkwo et al., 2019; Signh and Middendorf, 2020). With the increase in global demand for cement, one can imagine the negative impact that will have on the environment due to increased production. Therefore, there is a necessity for further investigation into cement products with lower environmental impacts and better economic benefits. Geopolymer cement is considered as a new sustainable building material that has received wide interest from researchers around the world. As many research works have been published in various fields of geopolymers since geopolymer cement binders can be used as a possible replacement for Portland cement since providing comparable performance in similar civil engineering applications in addition to its usefulness in reducing pollution and greenhouse gas (Ren et al., 2021).

The term *geopolymer* is commonly used to refer to the amorphous alkali alumina silicate. In fact, during the development studies of geopolymers, several terminologies have been used as inorganic polymers, alkali-activated cement, alkali bonded ceramics, geo cement, soil cement, hydro ceramics, etc. Generally speaking, these terms are all describing synthesized material (binder) that is produced using the similar initial ingredients of a recurrent unit of sialate monomer (–Si–O–Al–O–) (Duxson et al., 2007; Davidovits, 2013; Singh, 2015). The reactions that result in geopolymer called *geopolymerization* (Davidovits, 1994; Davidovits, 2005)

While geopolymer technology is regarded as new, the technology has old roots and was considered to be the building material for Giza's pyramids, as well as other old constructions (Barsoum and Ganguly, 2006).

In 1978, Joseph Davidovits (a French professor) was the first to coin the name *Geopolymer* to describe a broad variety of materials characterized by chains or networks of inorganic molecules (Davidovits, 1991; Davidovits, 2005). The use of alkaline

activator solution led to creating three-dimensional (3D) polysialates chains (similar to fly ash and slag), which resulted in hydroxylation and poly-condensation reactions (Van Chanh et al., 2008; Davidovits, 1991).

Between 1979 and 1995, Davidovits and his team published numerous papers and obtained patents on geopolymerization. These patents involve, among other things, the development of a geopolymeric binder and the development of a silica-aluminate mineral polymer (Komnitsas and Zaharaki, 2007).

Researches on geopolymer accelerated, and they appeared as published papers in late 1990 (Provis et al., 2015). Research efforts worldwide should focus on the production of *user-friendly* geopolymer cement that can be used in the same way as in Portland cement conditions (Davidovits, 2008). However, some drawbacks to geopolymer cement have restricted its use. These include lack of standards, risk of handling, costs, and availability. (Okonkwo et al, 2019)

2.2. Source Materials

Geopolymers have two main requirements: resource materials and alkaline activator solutions. Resource materials must contain alumina-silicate in their chemical composition, and these materials may either originate from natural sources or perhaps waste by-products materials (Xu and Van-Deventer, 2000; Davidovits, 2013; Duxson et al., 2007; Singh and Middendorf, 2020; Lloyd and Rangan, 2010; Davidovits, 1999; Farhan et al., 2020; Ren et al., 2021). Waste by-products materials include industrial, agricultural and municipal wastes (Farhan et al., 2020; Ren et al., 2021).

In addition to the above, a variety of source materials for the production of geopolymer cement besides the (types of alkaline activator) are shown in Table 2.1, which is used by the researchers for the production of geopolymer cement. Table 2.2, Table 2.3 shows the main chemical composition of some natural and industrial waste materials used in the production of geopolymer cement.

Table 2.1. Source materials for production geopolymer cement (Xu and Van-Devender, 2000; Davidovits, 2013; Duxson et al., 2007; Singh and Middendorf, 2020; Farhan et al., 2020; Ren et al., 2021).

Silicate Alumina Materials	Alkaline Activator
Natural materials :- calcined clays, kaolinite clays ,metakaolin clays, dolomite, pozzolan , zeolite ,volcanic ashes ,Volcanic rocks, pure Al–Si minerals,	NaOH, KOH, $Ca(OH)_2$, $NaOH + Na_2CO_3$, $K_2CO_3 + Ca(OH)_2$, Sodium silicate , (other silicates also)
Industrial waste by-products:- fly ash, GBFS, silica fume, red mud, copper and hematite mine tailings, etc.	
Agriculture waste by-products:- rice-husk ash, corncob ash, palm oil fuel ash, etc.	
Municipal waste materials:- waste glass, construction waste, etc.	

Table 2.2. Main chemical composition of some industrial waste materials (Amran et al., 2020; Garcia-Lodeiro, 2015; Shekhovtsova et al, 2018).

Property content (%)	FA class F	RHA	GGBS
SiO_2	38 to 55	>90	30 to 40
Al_2O_3	20 to 40	>9	5 to 20
Fe_2O_3	6 to 16	>28	<2
CaO	1.8 to 10	1-2.2	35 to 40
MgO	1 to 5	>1	5 to 18
Na_2+K_2O	1.25 to 5	-	∼4

Table 2.3. Main chemical composition of some natural materials(Safaria et al., 2020; Occhipinti et al., 2020; Yadollahi et al., 2015b; Salmani et al., 2017; Ferone et al 2013, Gharzouni et al., 2016; Metekong et al, 2021; Djobo et al., 2016; Lemougna et al., 2020; Baenla et al., 2019; Bims Sanayicileri Dernegi, 2006).

Property content (%)	Pumice	Metakaolin clay	Volcanic ash
SiO_2	52 to 75	53-54	40-46
Al_2O_3	10 to 18	42-46	14-16
Fe_2O_3	0.5 to 5	0.9-1.6	12-13
CaO	0.5 to 7	0-0.17	8-11
MgO	0.2 to 3	0-0.19	6-8
Na_2+K_2O	3.0-9.0	0-1	0.8-5.3

The most source materials used for geopolymer and their typical composition range are illustrated in Figure 2.1 As shown; pumice and fly ash contain a high percentage of silica and alumina, making them suitable as source materials for the production of geopolymer cement and concrete.

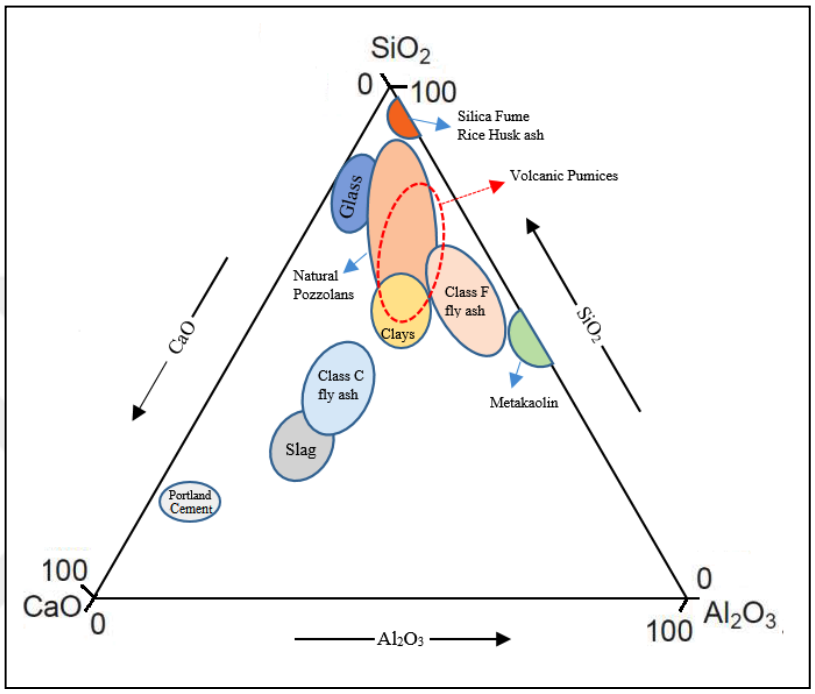


Figure 2.1. The range of source material compositions depending on the ternary Al_2O_3-CaO-SiO_2 (Mahmood, 2019).

Xu and Van-Deventer (2000) experimentally investigated a large array of alumina-silicate source materials for geopolymers. The examination included fifteen source materials which included ortho-, di-, and ring silicate, chain silicate, sheet silicate, also framework silicate (Clay group, Feldspar group, Sodalite group, and zeolite groups). The alkaline solution was NaOH or $Ca(OH)_2$, the better results were with potassium hydroxide. They discovered that the greatest compressive strength can be acquired in the elements that have higher portability on dissolve. The important influence was observed on compressive strength from some factors like Ca or K_2 oxide content, the molar concentration, Si /Al ratio in material, and A/B alkali ratio.

2.3. Geopolymer Synthesis and Chemical Structure

The synthesis of geopolymer cement is the product of a reaction between a source materials containing alumina-silicate with a high concentration of alkaline solutions by an inorganic polycondensation reaction (Davidovits, 1991; Palomo, 2014).

The transformation of the alumina silicate particles during geopolymerization is commonly described as the dissolution of the aluminosilicate source material by hydrolysis due to an alkaline activator (Duxson et al., 2007).

Generally, the process-taking place in geopolymerization of the source material is shown in Figure 2.2 (Singh et al., 2018).

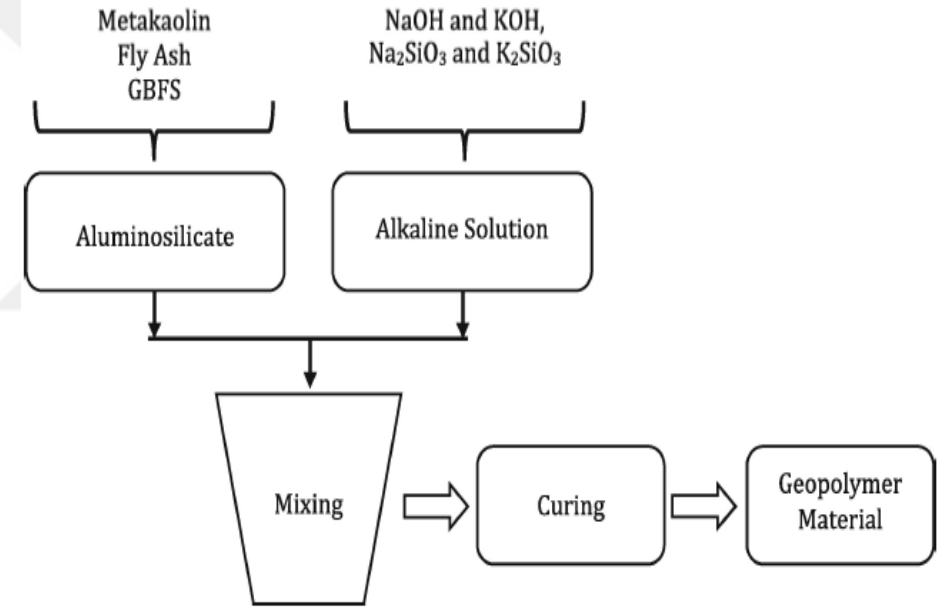


Figure 2.2. Geopolymer cement synthesis (Singh et al., 2018).

The setting and hardening mechanisms of the geopolymers are not clear. However, the following steps are included in the proposed mechanism for geopolymerization reactions (Van Chanh et al., 2008):

- Through the action of the hydroxide ions, the atoms of Si and Al dissolve in.
- Condensation occurs and precursor ions are converted into monomers.
- Polymeric structures are formed by polycondensation

Davidovits (2005) developed a logical scientific terminology for silicate and aluminosilicate materials based on different chemical units, which are categorized according to the Si: Al atomic ratio, as shown in Figure 2.3.

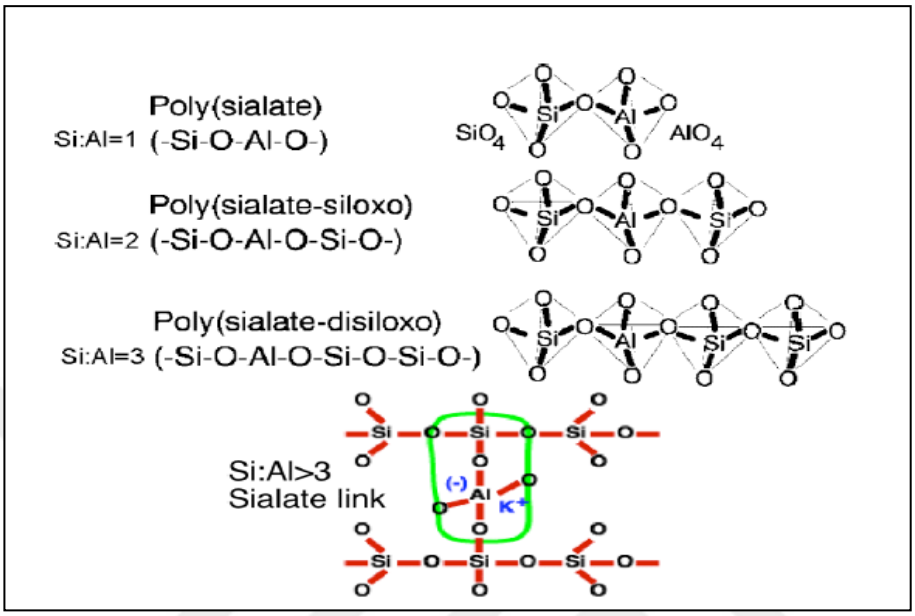


Figure 2.3. Geopolymer terminology and structure of polysialates.(Davidovits, 2005).

Geopolymerization process includes a chemical reaction in an alkaline solution that produces a three-dimensional (3D) polymeric chain with sialate bonds (-Si-O-Al-O) in a ring structure. The schematic of geopolymer formation can be understood as shown in "Eq 2.1" and "Eq 2.2" (Davidovits, 1991; Van Chanh et al., 2008).

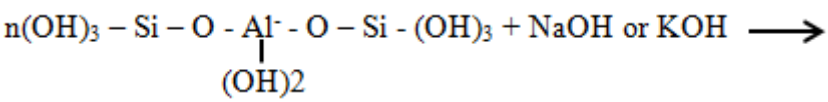
(Si-Al source) + Silicates + Water + Alkaline activator =

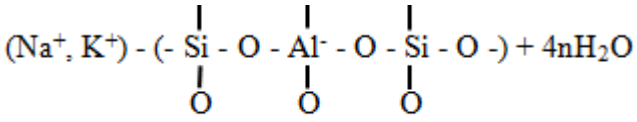
$$n(Si_2O_5, Al_2O_2) + 2nSiO_2 + 4nH_2O + NaOH \text{ or } KOH \longrightarrow$$

$$Na^+, K^+ + n(OH)_3 - Si - O - Al^- - O - Si - (OH)_3 \quad (2.1)$$
$$\qquad\qquad\qquad\qquad\qquad\qquad\qquad\qquad\qquad\quad |$$
$$\qquad\qquad\qquad\qquad\qquad\qquad\qquad\qquad\quad (OH)2$$

(Geopolymer Precursor)

Geopolymer Precursor + Alkaline Ions





(Geopolymer Backbone) (2.2)

These frameworks (-Si-O-Al-O-) called polysialates which are chain and ring polymers included a building unit of silicon-illuminate consisting of AlO_4 and SiO_4 linked by the sharing the oxygen atoms. The Sialate Si-O-Al bridge works to interlinked and to form the rings and chains. The positive ions of Na^+ or K^+ are present in order to balance the negative ions of Al. The three-dimensional silica-aluminate structures, ranging from amorphous to semi-crystalline are categorized as geopolymer (Davidovits, 1994).

Duxson et al. (2007) suggested a model for the process of polymerization as shown in Figure 2.4 .This consists of five stages:- a) dissolution, b) speciation equilibrium, c) gelation d) reorganization, and finally e) polymerization and hardening.

In the dissolution stage, the alkaline activator causes the aluminosilicate source to dissolve by hydrolysis, resulting in silicate and aluminate particles. After that, they then incorporated and produced a highly saturated solution of aluminosilicate. This leads to the formation of a gel and because of condensation, big networks have been developed and the release of water occurs in this stage. After the gelation stage, the system goes on rearranging, resulting in the three-dimensional (3D) aluminosilicate network, which is called geopolymers (Duxson et al. 2007; Nawaz et al., 2020).

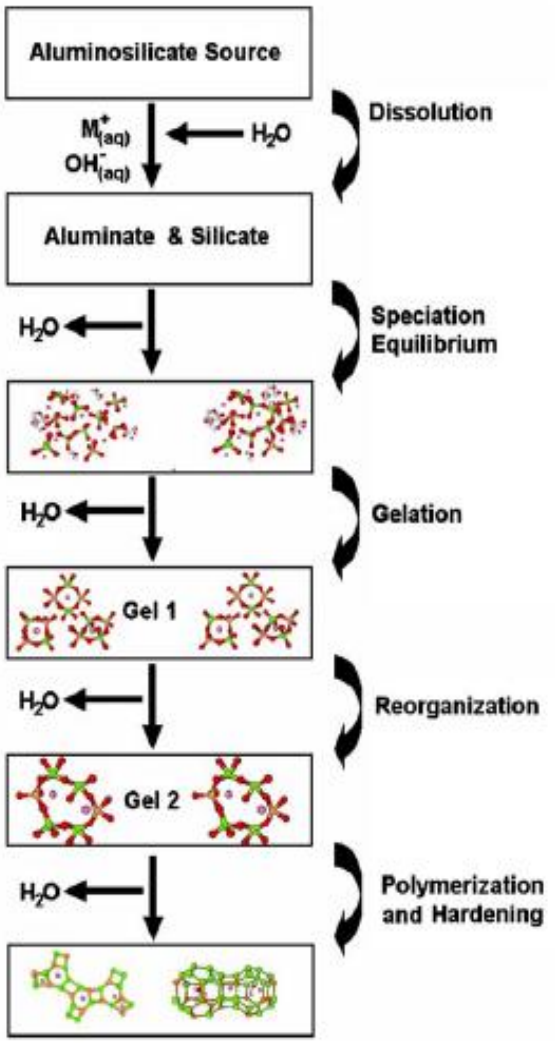


Figure 2.4. Model for the process of polymerization (Duxson et al., 2007).

2.4. Geopolymer Cement and Geopolymer Concrete

The meanings of these two terms *geopolymer concrete* and *geopolymer cement* are often confused. Cement refers to a binder, while concrete refers to a composite material resulting from a mixture of cement and aggregates. (Davidov, 2013; Okonkwo et al., 2019)

However, any geopolymer is produced by the reaction of the raw material with an alkaline activator. A polymerization process happens due to the chemical reaction. In this condition, geopolymers can show a wide diversity of properties, based on the

selection of the raw material, the type of curing and its temperature, procedure of mixing and concentration of the alkaline solution, etc. In general, geopolymer concrete possesses advantages over normal concrete such as earlier growing strength, big chemical resistance, good resistance to sulfate attack and acid. All these parameters make geopolymer concrete preferable to be used (Rangan, 2014).

2.5. General Literature About Pumice Geopolymers

Pumice is a volcanic natural pozzolanic material that is high in alumina and silica allowing it to be used as one of the geopolymer source materials, resulting in more sustainable and cost-effective products of geopolymer cement and concrete. Recently, several researchers have conducted studies on pumice-based geopolymer.

Allahverdi et al. (2008) used the pumice to produce geopolymer cement activated by the NaOH and Na_2SiO_3 solutions with changed alkali solutions based on NaOH and Na_2SiO_3. The water to binder (W/B) ratios were at 0.36, 0.40, and 0.44 respectively, and curing temperature at 25°C. The influence of the water/binder ratio, the concentration of sodium oxide, and silica modulus were studied. The results showed that the quality of this geopolymer cement depends on the composition of activator and w/b ratio, also chemical composition. Suitable compressive strengths up to 47 MPa with silica modulus, Na_2O concentration, and the ratio of W/B of 0.60,10 wt.%, and 0.36 respectively in 28 days tested.

Kani and Allahverdi (2009) produced an inorganic polymeric binder from pumice and discussed the influence of curing period and temperature on strength improvement. Na_2SiO_3 and NaOH solutions have been used as alkaline activators, three different chemical formulations mixes were made with different stream-curing temperatures and period and (45, 65, and 85°C) and (5, 10, 15 and 20 hours) respectively for curing. Optimal compressive strength was 37.5 MPa in the atmosphere at 25 °C, 37.5 MPa, by hydrothermal treatment for 20 hours at 85 °C and 57.5 MPa after 7 days for 20 hours respectively.

Kani and Allahverdi (2011) focused on the shrinkage performance of a geopolymer made from natural pozzolan (pumice). The effect of chemical composition,

SiO_2/Na_2O ratio, Na_2O/Al_2O_3ratio, and water to Al_2O_3 ratio as the parameters were studied. The hydrothermal curing was used to study the influence of curing on shrinkage. The results demonstrated that the chemical composition and the molar ratio of the alkaline activator mostly affect the shrinkage behavior of natural pozzolan-based geopolymer products. The amount of shrinkage was reduced by applying the hydrothermal curing with short procuring in a humid atmosphere.

Bondar et al (2011a) investigated adding mineral additives such as kaolinite, lime, and other calcined pozzolans to strengthen and improve the properties of geopolymer cement produced from Taftan natural pozzolan under normal and autoclave curing. KOH and sodium silicate have been used as alkaline activator. The study has reported that can be compensated the needs of CaO, Al_2O_3, and SiO_2 content in the natural pozzolan-based geopolymer paste, by using mineral additives to improve its properties.

Yadollahi et al(2014) investigated the use of pumice to fabricate geopolymer. NaOH and Na_2SiO_3 as an alkaline solution, superplasticizer, different A/B were used. Different curing temperatures from 25 to 85°C for 48 had been used. The test result showed that 65°C was the best temperature for pumice activation. The curing of the samples before 28 days was a notable effect on compressive strength, while after 28 days the effect on all temperatures of the heat treatment was negligible.

Yadollahi et al. (2015a) studied the elevated temperature effects on pumice-geopolymer which activated by the $Na_2SiO_3$and NaOH solutions with Superplasticizers, The W/B ratio were different values of 0.36, 0.40, and 0.44 and curing for 48 h at 65°C. Elevated temperature was (100, 200, 300, 400, 500, 600, 700 and 800 °C) for 3 hours. They found that with increasing elevated temperature levels the compressive strength decrease. Compare to the OPC the strength loss of geopolymer concrete is less. According to the results were reported pumice-based geopolymer has an excellent fire resistance property.

Yadollahi et al (2015b) investigated the influence of silica modulus and aging on properties of pumice-based geopolymer cement by using several various parameters. NaOH with sodium silicate solutions as an alkali activator, variable silica modulus of 0.52, 0.6 and 0.68, W/B of (0.36, 0.40 and 0.44) and Na_2O content (4%, 7% and 10%)

have been investigated. The curing temperature was 48 h at 65°C. They found out that optimum compressive strength obtains about 40 MPa in the mixture with Na_2O= 0.10%, SiO_2/Na_2O=0.68, and water/binder ratios= 0.36, the results revealed that compressive strength of pumice-geopolymer was adequate to use in building and construction material.

Almalkawi et al. (2017) studied geopolymer cement-based pumice. A mechanochemical processing and heat treatment were used with other raw materials of alkali and /or alkaline earth metal (included lime, sodium sulfate, and soda ash as chemical reagents, and gypsum as additive) into hydraulic cement. They investigated the hydration heat of cement particles, particle size distribution, bond structure, chemical composition, thermal attributes, mineralogy and cement particles morphology, and hydrated cement paste. The results showed that it is possible to produce hydraulic cement with applicable structures and development of strength.

Yener and Karaaslan (2019) used a ground clinker as a resource of calcium to improve the setting time also the rate of gaining strength at ambient temperature for pumice-based geopolymer paste activated by 12M of sodium hydroxide solution. curing at ambient temperature and 70°C for 5 and 24h were applied. AL/B ratio fixed 0.4. A conclusion of this work showed that adding clinker will shorten the setting times and compressive strength increased with increasing curing time and temperature for pumice-based geopolymers paste.

Safari et al (2020) investigated the hardening properties of pumice-geopolymer paste. It was activated by six NaOH alkali solution concentrations ranging from 8 to18 M, mixed with sodium silicate, the superplasticizer used, the A/B ratio was constant at 0.35 with curing temperature at 23, 60, 80, and 100 °C for 24, 48, 72 and 120 hours until the test at 7 and 28 days. They reported that the highest flexural and compressive strength found at 60°C for curing at 120h with 12M alkali solution, low curing time, required high temperatures to achieve good strength, while low temperatures required more curing time.

Occhipinti et al (2020) examined and evaluated the possibility of using pumice based geopolymers cement as restoration materials in heritage applications either alone or in binary mixtures with metakaolin at different weight ratios, were activated by

NaOH (8M) and sodium silicate solutions as alkali activator, A/B ratio various from 0.46 to 0.72 and cured at 25 ± 3C. Several tests have been done. The results showed that pumice powder mixed with 20–30 wt.% of metakaolin is suitable to make up lightweight geopolymers with satisfying compressive strength up to 12 MPa, water absorption of about 18.0%, and porosity up to 30%. These values indicate good characteristics to the potentiality for use in the proposed heritage applications.

A few studies have been conducted on the use of filling materials to produce pumice-based geopolymer concrete.

However, Bondar et al (2011b) studied some properties of concrete prepared with alkali-activated natural pozzolans. Potassium hydroxide (KOH) with Na_2SiO_3, fine and coarse aggregate, curing temperature (20 ± 2, 40 ± 2, and 60 ± 2°C), and W/B (0.42, 0.45 and 0.55) were used as parameters. generally, they concluded that natural pozzolan -geopolymer had a low compressive strength from ordinary Portland cement at an early age of curing while reaches the same or higher strengths than ordinary Portland cement mixtures after long curing. Also, the results showed that the compressive strength decreased with increases in the A/B ratio.

Ibrahim et al. (2017) studied some influences of binder content and alkaline activators on some properties of geopolymer concrete produced from natural pozzolan Also they examined the influences of curing time. The mass ratio of Na_2SiO_3 and NaOH(14M) varied from 2.0 to 2.75 and the volume of the binder in the range of 350 kg/m3 to 450 kg/m3. The fine and coarse aggregate content were 0.35 and 0.65, respectively, from the total aggregate. The curing time at 25±2°C for 24 h after that used 60°C for 0.5, 1, 3, 7, 14, and 28 days. They reported that the alkaline activator composition and binder content substantially had affected the properties of geopolymer concrete and the strength would improve with curing for up to 7 days. However, further, an increase in curing temperature up to 28 results to reduce the strength of geopolymers. The optimum strength and microstructural characteristics were obtained when the mass ratio of Na_2SiO_3/NaOH was 2.5, and the mass ratio of A/B is 0.525.

Ibrahim et al (2019) inspected the effect of activator solution on Alkali-activated binder by using natural pozzolan as precursor material. varying the of sodium silicate/NaOH ratio(2.0, 2.25, 2.5 and 2.75), the molarity of NaOH from 8,10 and 14 M

fine and coarse aggregate, curing at 60°C for ½, 1, 3, 7, 14 and 28 d and AL/B ratio 0.52 as parameters in the study were used. The conclusion of this work showed that the compressive strength increased with an increase in NaOH concentration of and the optimum compressive strength was gained in a ratio of 2.5 for SS/SH and NaOH concentration in 14 M.

2.6. General Literature About Fly Ash Geopolymers

Fly ash is an industrial waste produced by coal-fired power stations, formed after the use of coal as a fuel for the generation of electrical energy. The worldwide coal ash amount to about 600 million tones, 75-80% (nearly 500 million tons) from it contains fly ash. (Ahmaruzzaman, 2010) .Fly ash materials are caused by environmental problems, the recycled use by construction will decrease the cost of removal and lessen the costs of concrete manufacturing overall (Okonkwo et al 2019). Fly ashes are fine, glassy powder, particles size are with a range from 0.5 μm to 100 μm and contain essential quantities of SiO2, Al_2O_3, Fe_2O_3, and CaO. Also, lesser quantities of sodium, potassium, magnesium, and titanium are presented (Watile and Mhaisane, 2015).

Fly ash classification used by some investigators is shown in Figure 4.5., showing the two ASTM and CSA systems. According to (ASTM C618, 2003) fly ash is grouped into 2 types, the first is class F and the second is class C, the total composition of SiO_2+Al_2O_3+Fe_2O_3 is at least 70% for Class F and 50% for Class C by weight. While Canadian Standard, (CAN/CSA-A3000-03, 2003) is classified the fly ash into three types dependent on CaO contents (wattimena et al., 2017).

A large number of researches have been conducted with fly ash to produce geopolymer cement or concrete.

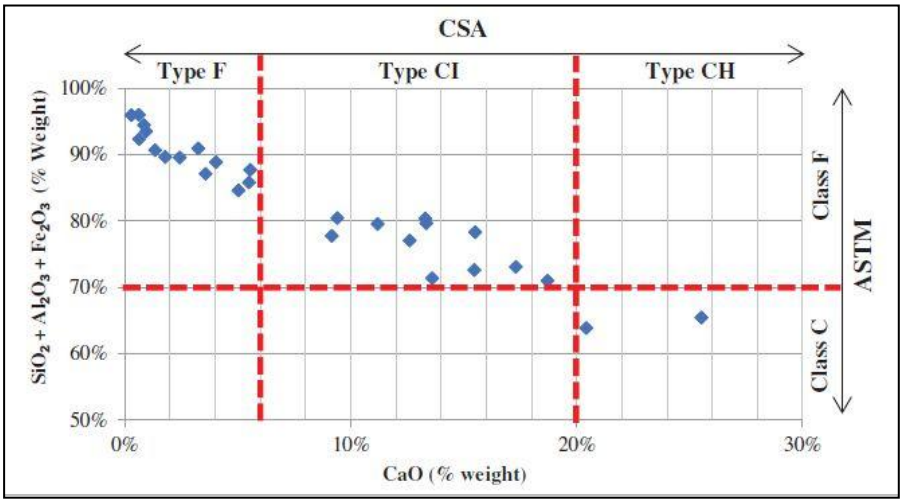


Figure 2.5. Fly ash classification that used in geopolymer studies (Wattimena et al., 2017).

2.6.1. Fly ash-based geopolymer cement pastes or mortars

Palomo et al. (1999) described the activation mechanism of fly ash (class F) in a highly alkaline solution. Four activators were utilized as activator: NaOH, KOH, NaOH+sodium silicate, KOH + potassium silicate. Some influences of some variables on compressive strength were studied. Curing time was (2 h, 5 and, 24 h), temperature (65°C, and 85°C) and A/B (0.25 and 0.35). The geopolymer cement made base on fly ash has been resulted to have about 60 MPa in compressive strength after curing at 85°C for 5 h.

Panias et al (2007) inspected the effect of NaOH, sodium silicate, and water contents on the compressive strength of fly ash geopolymers. Also, tests that deal with the microstructural composition of the produced samples were conducted which cured at 60 ◦C for 72 h. The conclusions of this work showed that the increased water content negatively affects the compressive strength. The increase in sodium silicate has been shown to improve the strength characteristics of the manufactured specimens.

Temuujin et al. (2009a) investigated the significance of CaO and $Ca(OH)_2$ on the compressive strength and stress-strain curves of fly ash geopolymer pastes. 1, 2, and 3% of fly ash were replaced by calcium compounds, sodium silicate solution was used. Curing in ambient was temperature and 70°C. As a general finding, they found that the

addition of calcium compound enhanced mechanical properties at ambient temperatures while lessening these properties at 70°C for cured samples.

Temuujin et al. (2009b) conducted a comparative study about fly ash geopolymers in terms of mixing procedure. They carried out their work using normal and mechanical mixing procedures. They concluded that the mechanical mixing exhibited a strength improvement of 80% when compared to the normal mixing procedure. Also, the microstructural observations revealed the fact that using the mechanical method to mix yielded more dissolved fly ash and less particle size which in turn led to more active geopolymerization and hence more powerful performance under the strength acquired by the mentioned mixing method.

Komljenovic et al. (2010) used six kinds of fly ash to study the hardening characteristics of fly ash geopolymers mortar with different chemical activators which were sodium hydroxide, potassium hydroxide, NaOH+Na_2CO_3, Ca(OH)2, and Na_2SiO_3 with various concentrations, period of curing (24 hours at 20±2°C and 6d at 55±2°C). Their findings stated that, the activator type and concentration represent a main reason concerning the mechanical and the microstructural behavior of the matrix. The maximum compressive strength was in fly ash, which had the highest content of fine particles; also, the high strength was strongly related to the high Si/Al ratio of geopolymer product and with use sodium silicate, the maximum compressive strength was gained.

Temuujin et al. (2010) studied the hardening properties of the manufactured sample by fly ash geopolymer mortars with different sand contents. NaOH (in 14 M) and Sodium silicate as alkali activator and cured at 70°C for 24 h were used. The results demonstrated that there was a link relationship between sand content and alkali activator. Increasing the amount of sand without increasing the amount of alkaline activator had a negative effect, resulting in a lower degree of geopolymerization. Compressive strength with 60 MPa was obtained and these values of strength did not change significantly with the addition of up to 50 wt.% sand.

Abdullah et al (2011a) studied some factors that affect fly ash geopolymer paste compressive strength. Different FA/activator solution ratios, curing temperature, NaOH molarities and Sodium silicate to NaOH ratios were used. The results exposed that the

maximum strength obtained with Sodium hydroxide molarity, Na_2SiO_3 to NaOH ratio, and FA/activator ratios were in 12 M, 2.5, and 2.0, respectively after 7 days. The compressive strength was up to 70 MPa at a temperature of 60°C.

Al Bakri, et al., (2011a) studied the influence of the NaOH morality on the early age compressive strength of fly as based geopolymer cement. NaOH with sodium silicate as an activator solution was used. NaOH concentrations were 6, 8, 10, 12, 14, and 16. Curing time constant at 70°C for 24 hours and the samples were tested at 1, 2, 3, and 7d. The best compressive strength obtained was with 12M sodium hydroxide concentration.

Al Bakri et al. (2011b) conducted an attempt to synthesize a geopolymer mix made from fly ash and studied the influence of curing heat on chemical and physical properties. Cured was at ambient temperature and 50 to 80ºC for 24h. Alkaline activators were sodium silicate with NaOH (in12m). The best compressive strength results were 67.04 MPa in samples cured with 60°C. The results showed that the compressive strength reduced after 60°C.

Al Bakri et al (2012) studied the effect of sodium hydroxide molarity and Na_2SiO_3 to NaOH ratios on compressive strength of fly ash class F using geopolymer cement. Alkaline activators were sodium silicate with NaOH, different ratios of Na2SiO3/NaOH, six different NaOH concentrations from 6 to 16 M, a fixed activators solution/ratio at 0.40, and fixed curing at 70° for 24 hours were used. The results showed that the highest compressive strength obtained in the $Na_2SiO_3$to NaOH ratio was 2.5 and the NaOH concentration was 12M.

Rickard et al. (2012) studied the thermal properties of geopolymers made from five different types of fly ash to investigate the performance of geopolymers under elevated temperatures. Sodium aluminate and sodium silicate solutions with sodium hydroxide were used. The cure period was 24 h at 70 °C and elevated temperature at 1000°C. They studied the mechanical, thermal, and microstructural characteristics of the fabricated samples. In general, they concluded that Si/Al ratio was an important factor in the thermal and the mechanical behavior of the samples. This ratio strongly affected the strength trend and the stability of the produced specimens, to be more specific, Si/Al ratios of more than 5 yielded a significant improvement in strength as well as clear

dimensional stability while the ratios which were less than 2 exhibited fewer strengths and weaker stability in terms of specimens dimensions.

Ryu et al. (2013) tested the effect of alkaline activators solution on the performance of FA geopolymers mortar by conducting strength and microstructural experiments on the fabricated specimens. Parameters consisted of series 1) was made of 6, 9, and 12 M of NaOH, series 2) was made of NaOH in 9M with 5 dosages of sodium silicate, cured for 24 hours at 60 °C and tested in 7 Classes (1, 3, 7, 14, 28, 56, 91 days). They concluded that the composition and the change in the chemicals have caused a significant influence on investigated properties. They also found that the compressive strength increase with higher molarity (12M) and a ratio of 1:1 NaOH: sodium silicate achieves a remarkable compressive strength of approximately 47 MPa. Also, a relationship between the splitting tensile and compressive strength of geopolymer concrete was proposed.

Hanjitsuwan et al. (2014) studied the influence of various NaOH molarities on the strength properties and electrical performance of high calcium fly ash geopolymer pastes. Sodium silicate and NaOH (in 8, 10, 12, 15 and 18 molar) as alkali activators, A/B ratio 0.4, and the temperature at (25°C for 1 hour +40°C for 24 hours) were used. The major finding of research revealed the fact that sodium hydroxide concentration mainly affected the setting time and the strength properties. Also, the dielectric constant and conductivity was increased with the increased concentrations of sodium hydroxide.

Patankar et al (2014) studied the impact of cure temperature and NaOH concentration on compressive strength of FA geopolymer mortar. Sodium silicate solution with different NaOH molarity (from 2.91 to 15.08 mol) was used as an activators solution, different activators solution/FA ratio from 0.35 to 0.45, different curing heat from 40 to 120°C for 1 day were used. After 3 days of curing time, the samples were tested. Findings have shown that the increase in the concentration of NaOH leads to an improvement in compressive strength and workability and the increase in a curing temperature was essential to accelerating the increase in strength. It was recommended that 13M be used as a NaOH activator solution based on better com strength and workability.

Adam and Horianto (2014) studied geopolymer mortar based on fly ash. They investigated the influence of the temperature and duration of the curing on the strength. Na_2SiO_3 and NaOH were activator solutions. The curing temperature was 80 to 120°C and curing time was 20, 6 and 4 h. The highest temperature was given the highest strength. So the curing temperature at 120°C for 20h gave the greatest strength.

Morsy et al (2014) study of FA geopolymer mortar to explore the influence of different Na_2SiO_3 to NaOH ratios (0.5 to 2.5) on geopolymer synthesis at curing in 80◦C for 24 h. The finding showed that the strength increases with increasing curing time. The compressive strength increases as the ratio of Na_2SiO_3 to NaOH increases (0.5 to 1.0) but then decreases.

Atis et al. (2015) manufactured high-strength geopolymer mortars from Class F fly ash by conducting more than one experiment concerning heat curing, the concentration of chemical activators, and curing times. The parameters chosen were: NaOH was an alkali activator at 2 to 20 M with 2M increment, curing temperatures 45 to 115 with 10°C increment step. Curing durations time at 24, 48, and 72 h, Mortar mixture sand to binder ratio 3:1 and 1/3 for water to binder ratio. They found out that up to an optimum point, both curing temperature and chemical activator's dosage were considered as the major factors that increase strength. The optimum compressive strength was 120 MPa also the optimum flexural strength was 15 MPa.

Zhang et al (2016) conduct a comparative study between five types of fly ashes that low in CaO. To study the effect of some parameters (such as fineness, particle density, mineralogical composition, specific surface area, etc.) of physical, crystallographic, and chemical characteristics on geopolymerization performance and the compressive strength, by using sodium silicate with 12 M NaOH solution and cured time at 40°C for 1 day. The result has been shown that these parameters have significant effects on the compressive strength of the fly ash-geopolymer cement.

Pavithra et al (2016) studied the influence of alkaline solutions on the behavior of fly ash geopolymer mortar. The parameters included Na_2SiO_3 to NaOH ratio from 1to 2.5, NaOH molarity from 10 to 18 mol as an alkaline activator, and Curing temperature was at 70°C for 1 day. The result showed that the optimum compressive strength values gaining at the 1.5 ratios for Na_2SiO_3 /NaOH of. It was found that the

compressive strength of the fly ash-geopolymer mortar increased by increasing the molarity of NaOH up to 16M and subsequently, by further increasing NaOH molarity the strength decreased.

İlkentapar et al. (2017) Fly ash of class F for geopolymer mortar was used to study the effect of heat cure time and rest period after heat cure time. In their study were used curing at 75°C for 4 hours, 1, 2, 3 and 7 days with23± 2°C until the test at 28 days, 50±5% relative humidity, just NaOH as an alkaline liquid activator with a concentration of 10 M and A/B ratio 0.46. The result showed that compressive strengths were proportionally increased with increasing curing from 4 168 hours. Also a strength development was observed after the rest period. The optimum strength obtained :11.82, 45.30, 64.78 , 70.03 and 75.69 ,In 4h ,1,2,3 and 7 days respectively in 28 day tested.

Shekhovtsova et al (2018) in their research, a technique for estimating the reactivity of fly ash geopolymer cement was developed. NaOH solution as alkaline activators and curing temperature at 60°C for 24h were used. The supposed technique depends on the (K-value) that contains 3 characteristics of low calcium fly ash including the specific surface area by Blaine measurement, amorphous phase percentage and a calculated parameter represent polymerization degree of silica. The result showed that the K value could be used to estimate the compressive strength of the fly ash alkali-activated cement and that there is a good relationship between them, with a correlation coefficient equal to 0.961-0.833 for the compressive strength of 1 and 91 days respectively.

Durak et al (2021) study about the effect of a pre-rest time before cure temperature on the properties of FA geopolymer mortars. The samples pre-rested under laboratory conditions for 0, 1, 2, 3, 7, 14, and 28 days before heat curing, and thereafter, they were cured to heat at 75°C for 2 days. The study results showed that the strength with the pre-rest period increased considerably compared to samples without pre-resting and it was determined that pre-resting improves properties.

A considerable amount of research has been done to optimize the behavior of the geopolymeric matrix in terms of different properties. One of the procedures carried out is to mix more than one raw material to achieve better results compared to the

performance of the geopolymeric cement produced by a single precursor source material.

Swanepoel and Strydom (2002), Examined the effect of cure temperature and curing time on strength of geopolymers produced by using FA and kaolinite. The NaOH and sodium silicate solutions and 40 to70°C as curing time for 72, 48, 24, and 6 hours. The optimum compressive strength was achieved when curing was at 60°C for 48 hours.

Songpiriyakij et al. (2010) studied the influences of the replacement of FA with rice husk ash and bark ash by five percentages on the compressive strength, degree of reaction, and microstructure of the manufactured geopolymers. NaOH(14M and 18M) and sodium silicate solutions were used, cured at 27°C+60°C for 24 hours, and compressive strength tested at 3, 7, 14, 28, and 90 days. The results of the study have shown that the quality of the source materials plays a large role in the improvement of the geopolymers produced.

Sinsiri et al. (2012) investigated the effect of the use of diatomite with FA on characteristics of geopolymer mortar, where the proportions of FA replacement with diatomite was 60 % and 80 % of fly ash by weight, in addition to using 100 % for both sources. The parameters that used were NaOH and Na_2SiO_3 as alkaline activator, $Na2SiO_3$/NaOH ratios of 1.5, NaOH concentrations of 15M, A/B ratio from 0.40 to 0.70, cured at (ambient temperature for 1 day +75°C for 1day). They argued that the increase in the A/B ratio from 0.4 to 0.5 increased the compressive strength, while the increase in the A/B ratio from 0.5 to 0.7 was led to a reduction in the compressive strength by about 45%. A compressive strength of between 22.0 and 81.0 MPa was obtained from mixtures with the A/B ratio of 0.50 for 7 days.

Sukmak et al (2013) studied the clay and FA geopolymer and the factors that affect its strength. A liquid alkaline activator was Na_2SiO_3 with 10M NaOH; Na_2SiO_3 to NaOH ratio from 0.4 to 2.3, A/B ratio was 0.4 to 0.7were used. The sample was cured at (ambient temperature for 24 h + at (65, 75, and 85°C) for (24, 48, and 72 h) and compressive strength tested after 7, 14, 28, 60, and 90 days. They concluded that the strength would increase as the alkaline solution increases to 0.6 A/B, but decreases as the alkaline solution volume increases. The compressive strength would increase with

an increase in heating up to a certain value, but the overheating with excess curing time caused micro-cracks on the samples, which led to a decrease in strength. They found the optimum results for geopolymer were: Na_2SiO_3 to NaOH ratio of 0.7, A/B ratio of 0.6, and optimum temperatures between 65 and 75 °C.

Zhang et al (2014) studied the effect of the differential factor on the characteristics of geopolymers manufactured from fly ash (class F) and red mud. Sodium tri silicate of 2 M and NaOH of 50% by weight as alkali solution were used. The curing temperature at 80°C for 1d, 50°C for 7d, and at room temperature. The study also focused on the effect of chemical composition in geopolymers. The result of the study found that the Na to Al ratio in 0.6 to 0.8 with a fixed Si to Al ratio of 2 is a better value for synthesizing geopolymers from fly ash and red mud.

Duan et al. (2016) tested the strength properties of geopolymers made from fly ash and metakaolin. Also, sulfate attack was investigated. FA was replaced by metakaolin 0,5, 10, 15, and 20% by weight. NaOH (in10M) and sodium silicate as alkaline activators were used. The results show that the existence of metakaolin improved the resistance against sulfate attack; the increasing content of partially replaced metakaolin was responsible for improving the performance of the manufactured geopolymer samples. In terms of microstructural composition, the presence of metakaolin increased the compactness of the resulting matrix with the improvement of strength properties and resistance to sulfate attack.

Marcin et al.(2016) carried out experimental tests to identify the effect of adding GGB slag to the mechanical behavior of fly ash geopolymers. Fly ash was replaced by 15%, 30%, 50%, and 65% by weight of slag, NaOH (in 10M) as alkali activator was used. The samples were cured for 6 h in a hot air drying chamber at a temperature of 80°C and tested after 7, 28, and 90d. The results showed that the addition of GGBS was had a little influence on the compressive strength whereas flexural strength was reduced with the increased slag content. However, the porosity properties were observed to be better with the increasing slag content.

Saha and Rajasekaran (2017) conducted a study on the geopolymer paste properties to understand the hardening effect of the geopolymeric matrix. they realized that setting time property is not computable in reasonable ranges when the matrix

consists of fly ash only, therefore they examined the influence of adding from 10% to 50% in of GGBS. The sodium silicate solution to NaOH ratio = 1.0 and AL/B = 0.4 and cast samples tested after 7, 28, and 56 d. Their conclusion showed that compressive strength and setting time significantly improved trends that occur by an increasing trend when GGBS was added to the mixes.

Wei et al (2021), studied the influence of low heating treatment of alkali-activated FA and GGBS mortar. 50% FA and 50% GGBS, the cure time at -5 °C, 0 °C, 5°C, and 20°C were used. A retarder, antifreeze, and a superplasticizer were used. NaOH solution 12 M with Na_2SiO_3 was the alkali solution. The findings showed that low cure temperature decreased the compressive strength of the tested samples. The low temperature slowed the hydration process and resulted in harmful pores.

2.6.2. Fly ash with filling materials based geopolymer concrete

Hardjito et al. (2004) study was about geopolymer concrete development with using FA class F and studied the parameters influences to compressive strength, sodium silicate with NaOH (8 and 14 M) as the alkaline liquid was used, and (30 to 90°C in 6 to 96 h) was curing temperature and time respectively. A/B ratio was kept fixed at 0.35. The most prominent results include that 60°C curing temperature and 4 days Curing was the better for the compressive strength. A high molarity of NaOH solution give results better than low molarity, and the compressive strength reached 90 MPa for FA-geopolymer concrete.

Mishra et al (2008) studied the effect of different cure times and concentration of activator solution and cure time on strength and water absorption of FA geopolymer concrete with sodium silicate and NaOH concentration of 8M, 12M, and 16M. A/B ratio was taken as 0.5. Curing was in 100°C for one hour then for 1, 2, and 3d at 60°C. Results of the investigation indicated that compressive strength increase with an increase in curing time and NaOH concentration. Maximum compressive strength was up to 46 MPa in 3d curing time. Also, they concluded the water absorption decreased with increase curing time and in NaOH concentration.

Kong and Sanjayan (2010) performed an investigation of the influences of very high heating on FA geopolymer concrete, mortar, and paste. Alkaline activators KOH (in7M) and Na_2SiO_3 solutions, Superplasticizers, solid/alkaline liquid ratio of 3.0, curing temperature 80°C for 24h were used as parameters. The 800°C for 1 h as an elevated temperature was used to study its effect on geopolymer. The study showed that aggregate size and specimen size are the two most important factors that influence geopolymer behavior at high temperatures. The aggregate in sizes of more than 10 mm led to better strength at ambient temperatures and very higher temperatures.

Diaz et al. (2010), study about the factors affecting the suitability of FA to use in geopolymers. They used different fly ashes performance in terms of chemical composition, behavior, and setting time along together with the microstructural characterization. NaOH (in14 M) and Na_2SO_3 were used as activator liquid. Cure time was for 3d at 60°C and tested after 24 h. Conclusions stated that the degree of the vitrification, location of the maximum diffraction of glass, and particle size of the FA represented the main factors that affect the characteristics of the resulting specimens

Nuruddin et al. (2011) investigated the effects of NaOH solution concentration and super plasticizer on the workability, compressive strength, and microstructure of low calcium FA self-compacting geopolymer concrete. The curing temperature was 70°C at 48 hours. Results have shown that compressive and workability have improved as the dosage of the superplasticizer and NaOH concentration have increased. The compressive strength increased when the concentration of NaOH solution was 8-12 M but decreased when it was 12-14M. The maximum compressive strength of 51.52 MPa was obtained when tested at 12M on 28 days.

Memon et al. (2011) investigated the cure conditions influences on the compressive strength FA self-compacting geopolymer concrete. Sodium silicate and NaOH(in 12M) as an alkaline activator, the curing time in 24, 48, 72 and 96 hours, curing temperature of 60 to 90°C, superplasticizer dosage of 7% by weight, A/B of 0.5, 2.5 the ratio of Na_2SO_3/NaOH were used as parameters. Some tests like L-box, Slump flow, and V-funnel were used to evaluate workability properties. The results show that the compressive strength is increasing as the curing time increases. The

compressive strength increase was not significant after 48 hours. The highest compressive strength was achieved with concrete specimens cured at 70°C.

Josepha and Mathew (2012), tried an attempt to understand the influences of different contents of coarse and fine aggregate with different parameters in characteristics of FA geopolymer concretes. Na_2SiO_3 and NaOH(in varying molarity) as an alkaline solution with a superplasticizer, an A/B ratio of 0.35 to 0.65, and curing temperature of 30°C to 120°C with curing time of 6 t0 72 hours that were used as parameters. The results showed that geopolymer concrete with better values of parameters could be produced with better properties than OPC concrete. The maximum compressive strength obtained was 56 MPa after 28 days.

Jamkar et al. (2013) studied the influence of different fineness of FA on the strength properties of geopolymer concrete. A highly alkaline solution of $Na_2SiO_3$and NaOH (in 13 M) solution was used to active 5 different fly ashes. The Blaine fineness of fly ash were 265, 327, 367, 430, and 542 m2/kg, A/B ratio of 0.35, curing temperature of 90°C for 4 to 24 h were used. The results indicated that the fineness of FA plays an essential role in geopolymer activation. When the fineness of fly ash increased, both workability and compressive strength improved. It was also mentioned that the finer particles of fly ash lead to an increase in the reaction rate and require less curing period to achieve enough strength.

Memon et al (2013), their study focused on the effects of NaOH concentration on the compressive strength and fresh properties of FA self-compacting geopolymer concrete. The parameters used in this study were NaOH of 8 to14 M, curing temperature of 70°C for 2d, Superplasticizer, A/B ratio fixed at 0.5 and Na_2SiO_3/NaOH ratio fixed at 2.5. It was also used four times to compressive strength tests at 1, 3, 7 and 28d were used. The findings showed that the change in the concentration of NaOH had little effect on workability. As the concentration of alkaline solution increased, the workability was slightly reduced. The maximum compressive strength was obtained in 12M of NaOH solution.

Nath and Sarker (2014) studied the effect of adding the GGBS to the FA to synthesize geopolymer concrete cured in ambient temperatures that do not require heat treatment, takes less time to harden and are mostly comparable to other types of binders.

The FA replaced GGBFS from 10 to 30%, NaOH with Na_2SiO_3 as the activator solution, and the A/B 0.3 and 0.45. The main conclusions of the obtained results proved the benefit of using GGBS within the geopolymeric matrix, which made the resulted composites comparable to the conventional OPC binders in setting time and strength properties.

Chindaprasirt and Chalee (2014) examined the effects of the use of changed molarities of NaOH on the strength behavior, resistance to chloride, and corrosion phenomena of FA geopolymer concrete after exposure to seawater. The alkaline liquids were NaOH (from 8 to 12 M) and sodium silicate solutions. A/B was fixed in 0.60 and curing temperature between 26 and 35C. Higher molarities were found to yield better results in terms of corrosion and turn better performance regarding strength because the corrosion effect is always correlated to the strength behavior.

Zaetang et al. (2015) studied the influences of NaOH concentration, curing temperature, and the partial incorporation of OPC with FA on some properties of geopolymer concrete. NaOH (in 10 and 15 M)with Na_2SiO_3 as an activator solution, a fixed A/B ratio of 0.3, and curing temperature at 60 to 120°C for 2 days were used in this study. The results showed that with increasing the replacement level of ordinary Portland cement and increasing the molarity of NaOH from 10 to 15 M, the compressive strength of geopolymer concrete would increase. Curing of 90°C was recommended for optimum strength.

Reddy et al. (2018) used FA and GGBFS to produced geopolymer concrete. The ambient temperature was for the treatment, different A/B in the range of 0.4 to 0.8 and the NaOH(in14M) with Na_2SiO_3 as the alkaline liquids were used in the study. They reported that the increase in the A/B ratio from 0.4 to 0.8 resulted in a 50% reduction in compressive strength, which was in the range from 66 to 32 MPa.

Naghizadeh and Ekolu (2019) produced a geopolymer concrete by a new mix design method using FA as a binder. A combination of $Na_2SiO_3$and NaOH was used as an activator solution. The mixture design method was based on some factors such as the mass ratio of alkali activator to FA, the Na_2SiO_3/NaOH ratio of 2, and the NaOH concentration. The workability and compressive strengths values of geopolymer concrete were satisfactorily comparable to the designed values and it was possible to

apply the concept of the proposed method to make mix design charts for different types of geopolymer concrete.

Ekmen et al (2021) investigated to evaluate the influences of the parameters of the different properties of lightweight geopolymer concrete using FA as binder and pumice as aggregate. NaOH of 10 to 14 M with Na_2SiO_3 as an alkaline activator, Na_2SiO_3/NaOH ratio of 1.5 to 2.5, AL/B ratios of 0.45 and 0.55, amount of Superplasticizer and curing time of 75 C for 24 h were used as mixing parameters. The results showed that the ratios A/B and Na_2SiO_3/NaOH significantly influenced the behavior of the viscosity. There was a strong relationship between mechanical properties and the composition of oxides. The maximum strength was obtained by increasing the molarity of NaOH from 10-12 M for the ratio of A/B to 0.55 and the ratio of A/B to 0.45, the maximum strength was by increasing the molarity of NaOH from 12-14M. Lightweight geopolymer concrete can be used in the precast industry.

2.7. Alkaline activators

Many alkaline activator solutions can be used for alkali activation in the geopolymerization process NaOH, KOH, $Ca(OH)_2$, NaOH+Na_2CO_3, K_2CO_3+$Ca(OH)_2$, Sodium silicate, and other silicates (Singh and Middendorf, 2020).

The alkaline solution may be either alone or obtained by mixing alkali hydroxides at specific molarity with sodium silicate solution or other silicates. The use of alkaline activators depends on some factors, such as the cost, availability, and reactivity required. Several studies have been conducted on alkaline activator influences of geopolymers, some of them listed in previous sections.

Joshi and Kadu (2012) Joshi and Kadu (2012) identified the effect of several different alkaline activator parameters on geopolymer concrete and the relationship with the strength of concrete. The parameters used in this study include NaOH solution at concentrations of 8 to 16, Na_2SiO_3/NaOH ratio of 1.75 to 3.0, A/B ratio of 0.25, 0.30, 0.35 and 0.4, curing temperature at (60°, 75°, and 90°) and curing time of (1and 2 days). The conclusions of this study showed that there is a close relationship between the parameters used and the compressive strength. An improvement in strength was noticed

when the molarity increased. They recommended the use of NaOH in 12 M, Na_2SiO_3/NaOH ratio of 2.50, and A/B of 0.35 in curing conditions at 75°C for 24 hours to obtain optimum compressive strength in FA geopolymer concrete.

Wang et al. (2012), investigated the influence of alkali activators on the strength of metakaolin geopolymer cement by using different molarity of NaOH of 8 to 16M with different amounts of sodium silicate. The strength value was found to improve with the NaOH molarity from 8 to 16M for the 7day age sample. Optimum compressive strength was at 12 and 16M solution concentrations during 7 days of curing.

Leong et al. (2016) conducted a comprehensive study on the effect of different types of alkaline activator solutions and their contents of K_2O and Na_2O ratios on strength in fly ash geopolymer. Na_2SiO_3, KOH, NaOH and $Ca(OH)_2$ with varying proportions and quantities were used as activator solutions. Also the study included the effect of Na_2SiO_3/NaOH, Na_2SiO_3/KOH and Na_2SiO_3/$Ca(OH)_2$ on workability and compressive strength and to investigate variations in compressive strength because of oxide molar ratios. Results showed that the bigger ratios of molar oxide did not inevitably result in greater compressive strengths. The weakest workability and strength were when $Ca(OH)_2$ was used as an alkali activator compared to that produced by KOH or NaOH.

2.8. Factors Affecting Mechanical Properties of Geopolymer

According to previous studies, several factors are affecting fresh and hardened properties and interactions, of the geopolymer cement, mortar, and concrete like the composition and fineness of the source materials, the type and concentration of alkali activators, heat degree, and the curing period. Some of the main parameters affecting the properties of geopolymer composites are explained as follows:-

2.8.1. Chemical composition

The composition of the resources determines the combination and type of alkali activator solutions in the synthesis system of geopolymer (Ibrahim and Maslehuddin,

2021). Generally, a low amount of calcium in resource materials required curing at high temperature. While the increase of calcium in precursor materials shorten the setting times and improve the rate of gaining strength with curing at ambient temperature.(Yener and Karaaslan , 2019; Ibrahim and Maslehuddin, 2021) .Otherwise, a large quantity of Ca may be interfering with the polymerization process so it has a fast setting time and may affect the microstructure (Gourley, 2003). Silica and aluminum oxide have been indicated as the Si/Al ratio, which the increase in it leads to an improvement in the compressive strength of the geopolymers (Gunasekara et al., 2014; Komljenovic et al., 2010). The Si/Al ratio limits the application domains (Abdullah et al., 2011b). The (Si: Al ratio) was used by Davidovits (1999), to categorize the different forms of geopolymer applications. However, according to Chindaprasirt et al. (2012), a high increase in Si/Al reduces both the setting time and the compressive strength.

2.8.2. Fineness of binder

The fineness and particle size of the binder material (like fly ash, bottom ash, etc.) significantly affect the workability of concrete and cement, the hardened properties, the durability characteristics of the geopolymers, and the activity of the alkali liquids. Highly fine is accelerated the polymerization mechanism, and when the fineness of the binder increased both the compressive strength and the workability improved. (Sathonsaowaphak et al., 2009; Patankar et al., 2013; Yusuf et al.,2014; Weng et al., 2005; Brough et al., 2000) .

2.8.3. Concentration of alkaline activators

In general, most literature researches used NaOH and Na2SiO3 or NaOH alone as an alkaline activator in geopolymers. Many previous researches reported that an increase in the concentration of the alkaline activator leads to accelerating chemical dissolution and alumina-silicate solubility improves. Also the compressive strength increases with increasing molarity up to a certain concentration (Safari et al, 2020; Najamuddin et al., 2019; Abdullah et al 2011a; AlBakri, et al., 2011a; 2012; Ryu et al. 2013; Hanjitsuwan et

al., 2014; Patankar et al, 2014; Atis et al.,2015; Pavithra et al, 2016; Mishra et al 2008; Nuruddin et al., 2011; Memon et al, 2013; Chindaprasirt and Chalee, 2014; Ekman et al,2021; Huseien et al., 2016; Ghafoori et al., 2016; Ibrahim et al, 2019).

There are no specific controls for selecting the molarity concentration of the alkaline activator, but many studies have indicated that 12 M of NaOH at curing temperatures between 60-70°C to give the best results concerning the compressive strength and some other characteristics (Safari et al, 2020; Abdullah et al, 2011a; AlBakri, et al., 2011a; 2012; Ryu et al., 2013, Nuruddin et al., 2011; Memon et al, 2013). However, Patankar et al (2014) were recommended to use13M of NaOH for better workability and compressive strength, while Pavithra et al. (2016), Ibrahim et al (2019), Ekmen et al (2021) founded that 14M NaOH giving a better result.

2.8.4. Alkaline solution to binder ratio A/B

Alkali activator solution to binder ratio is considered one of the most important factors, which affect the strength and durability properties of geopolymer so should determine carefully. Most activator solutions consist of water and when the quantity of activator solution increases, the quantity of water will also increase. As a result of the water found inside the alkaline-activator solution resulting in a lower microstructure in the geopolymer mixture, this leads to a decrease of the compressive strength (Bondar et al., 2011b; Reddy et al. 2018).

According to some previous studies, there is no optimum value or limited range of alkali activator to binder ratio, due to the difference in characteristics of source materials using in geopolymers such as chemical composition fineness, properties of filling materials, using superplasticizer, and humidity. However , According to Bondar et al (2011b), Ibrahim et al. (2017) Ghafoori et al. (2016), and Occhipinti et al (2020), it possible to the conclusion that alkali activator/ binder ratio between 0.45 to 0.55 to produce natural pozzolans based geopolymer. According to Palomo et al (1999), Al Bakri et al (2012), Patankar et al (2014) the ratio between 0.30 to 0.40 is better to produce based fly ash cement. While from the studies of Sinsiri et al. (2012); Joseph and Mathew(2012), Sukmak et al. (2013), and Reddy et al. (2018) the range between 0.45 to 0.55 is the most

recommended to produce fly ash-based geopolymer when adding another raw material or filling materials (fine and coarse aggregate).

2.8.5. Sodium silicate/sodium hydroxide ratio

Several studies have shown that the ratio of Na_2SiO_3 to NOH is considered to be another parameter affecting the mechanical durability properties of geopolymers. There are no optimum values for the Na_2SiO_3/NaOH mass ratio due to the different composition of the resource materials and the influence of other parameters such as the alkali activator properties, the A/B ratio, and the curing conditions. However, Morsy et al. (2014) stated that the strength of geopolymers was improved when of Na_2SiO_3/NaOH mass ratio increased from 0.5 to 1, while Sukmak et al. (2013), Pavithra et al.(2016) stated that the maximum compressive strength can be achieved when the mass ratio was 1.5. Oleiwi et al. (2018), Nagaraj and Babu, (2018) reported that the optimum Na_2SiO_3/NaOH was 2 and finally Abdullah et al. (2011a), Joseph and Mathew (2012) recommended using the ratio of 2.5 for the production of geopolymers.

2.8.6. Curing conditions

The curing temperature and curing time are required for geopolymerization processes and to obtain the early strength property of geopolymers, also accelerates the setting time. Many researchers are trying to find an appropriate balance between the curing temperature and the duration of treatment in order to find the best curing conditions, which achieve the best mechanical properties of the geopolymers concrete or cement. Geopolymers cement can be produced at the ambient temperature but the gain strength will be at a slow rate as time compares to heat curing. The increase in the heat curing leads to an increase in the strength of geopolymer concrete, also the increase the curing period develops the properties of geopolymer concrete (Al Bakri et al., 2011b; Patankar et al. 2014; Adam and Horianto, 2014; Sukmak et al, 2013; Josepha and Mathew, 2012). While the opposite leads to a negative impact on mechanical strength development due to high

temperatures that will cause cracks in geopolymer cement, it seems that mild curing improves mechanical properties (Ibrahim and Maslehuddin, 2021).

So it can inference from several previous studies that the optimum compressive strength has obtained with the curing temperature in range (60–75)°C, and curing time in a range of (5 -7) day for pumice based geopolymer (safari et al 2020; Ibrahim et al.,2017) and (1– 4) day for fly ash-based geopolymer (Al-Bakri et al., 2011b; Sukmak et al, 2013; Hardjito et al., 2004; Mishra et al., 2008; Memon et al., 2011). Nevertheless, some studies showed that curing in (90 – 100) °C and (1-2) days for FA-based geopolymer is most appropriate for curing (Patankar et al. 2014).

3. MATERIALS AND METHODS

3.1. General

This section includes three parts. The first describes the type and specification of the materials that used for the production of geopolymer paste. The second part is about the experimental work, such as the description of work mixtures contents, the mixing procedure, and curing described in this part. The third part describes the geopolymer specimens tests program as compressive strength, flexural strength, water absorption, as well as tests after exposure specimens to freezing-thawing cycle and elevated temperature.

This section also includes pictures of the devices used in the work and the models that were produced.

Figure 3.1 shows the work program flow chart in this study includes the three parts outlined in this section.

3.2. Materials

3.2.1. Pumice powder

Used pumice was brought from the kocapinar region near Ercis Town, eastern Turkey. The powder of pumice was made in the laboratory by using a ball mill machine to grind the crushed aggregates of pumice for 4 hours. Table 3.1 includes details of chemical composition and Figure 3.2 the distribution of particle size for pumice used in the research. A sample of the crushed and powder of pumice that used is shown in Figure 3.3 and the ball mill machine as in Figure 3.4, respectively.

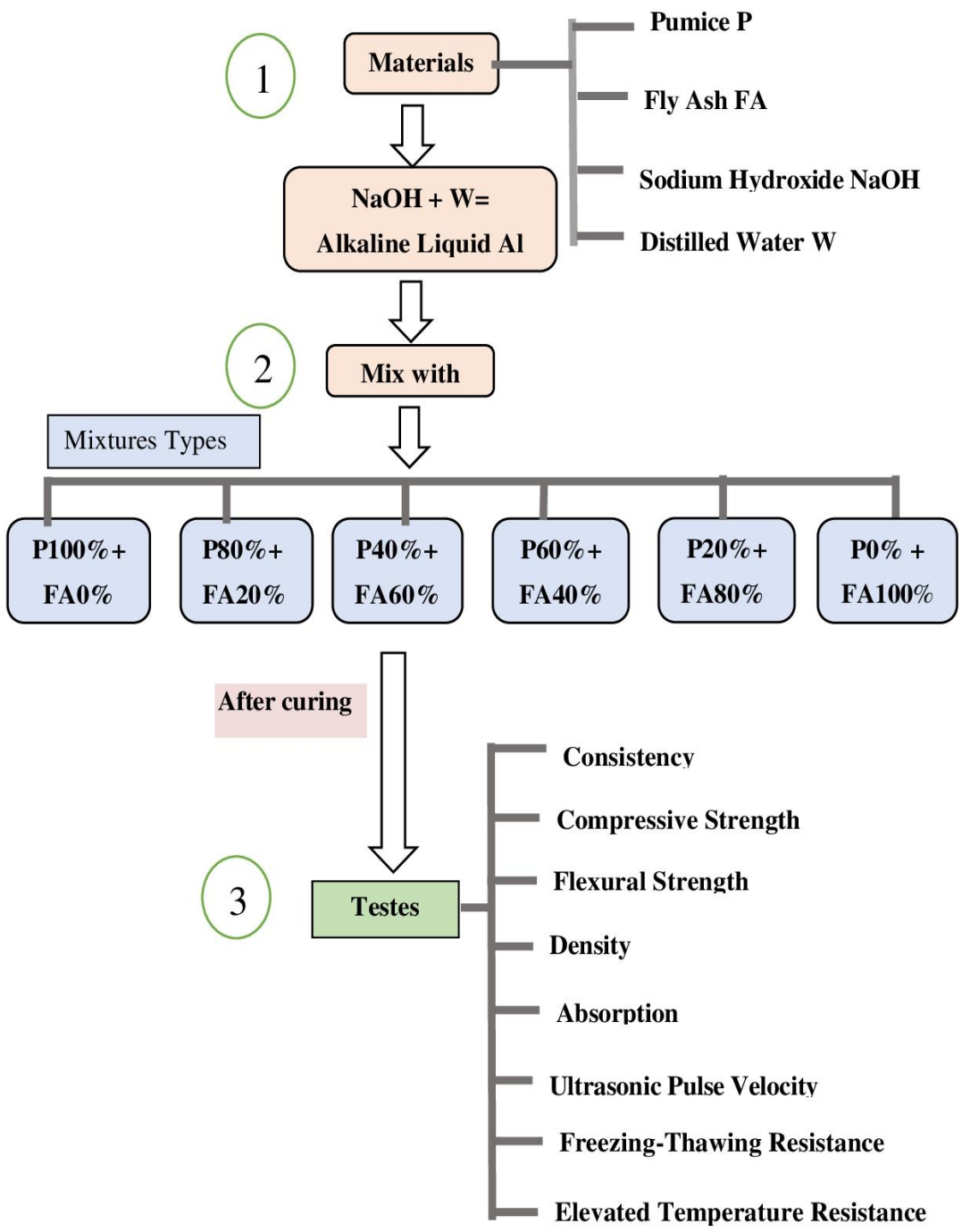


Figure 3.1.The flow chart of the work program.

Table 3.1. Chemical composition of Ercis pumice powder (Safari et al., 2020).

Chemical Composition	Percentage%
SiO_2	75.23
Al_2O_3	14.04
Fe_2O_3	1.95
K_2O	5.05
MgO	0.22
CaO	0.52
Na_2O	2.09
TiO_2	0.11
P_2O_5	0.03
MnO	0.22
SO_3	0.29
Cl-	0.04

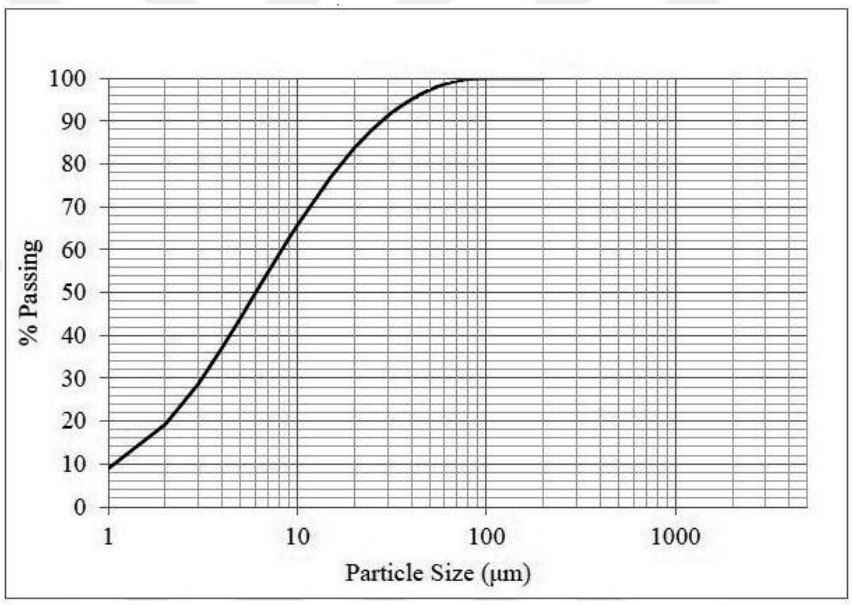


Figure 3.2. Ercis pumice powders particle size distribution (Safari et al., 2020).



Figure 3.3. Crushed and powder pumice.



Figure 3.4. The ball mill machine.

3.2.2. Fly ash

The fly ash, which was used in the search, was provided from Sugözü Thermal Power Plant located in Sugözü village of Yumurtalık district in Adana province in Turkey Table 3.2 shows the chemical composition also the physical properties of Sugözü fly ash data and Figure 3.5 shows a sample of fly ash that was used. The results of chemical composition show that the used FA satisfied the requirement of ASTM 618, 2008 class F with a low CaO content as the sum of (SiO_2, Al_2O_3, and Fe_2O_3) is more than 70%, as well as the CaO ratio by weight, is less than 10%.



Figure 3.5. Sample of fly ash used.

Table 3.2. Chemical composition of Sugözü fly ash (Atabey, 2017).

Chemical Composition	Percentage%
SiO_2	60.51
$Al2O_3$	21.69
Fe_2O_3	7.85
K_2O	2.58
Na_2O	0.92
MgO	1.65
CaO	1.52
SO_3	0.53
Cl-	0.010
Free CaO	0.183

3.2.3. Alkaline activator solution

Sodium hydroxide flakes with 98% purity that are commercially available are used in this study to prepare alkaline activator solution. It was obtained from Koray Chemistry, Istanbul, Turkey. Sodium hydroxide was used because it was the most used substance in the previous studies.

Solution of NaOH can be obtained by dissolving solid flakes in an amount of distilled water. Depending on the concentration of the alkaline activator solution that required the amount of NaOH to be mixed with water. For example where NaOH alkaline activator solution with a concentration of 1 Molar consists of 40g of NaOH solid per 1 liter of alkaline solution, thus if 12 molar consists 440g NaOH solid per 1liter of alkaline solution. Figure 3.6 is shown (NaOH) in flakes form and NaOH alkaline activator solution. Sodium hydroxide reaction was exothermic; therefore, it is very important to take care

while preparing the solution such as wearing safety gloves, costume, safety, shoes, glasses, and the solution should be prepared in a special place. (Figure 3.7). According to some previous recommendations, after the solution mixed left for at least 24 hours waiting period before using it, to complete the interaction.

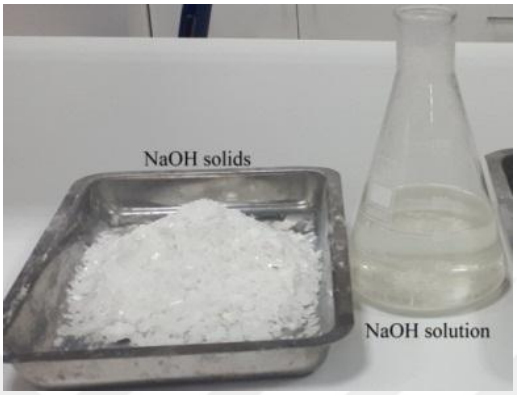


Figure 3.6. NaOH in flakes form and solution.



Figure 3.7. The place for preparing NaOH solution.

3.2.4. Water

Distilled water was used to dissolution the NaOH flakes for mixtures of geopolymer. It was made in the laboratory from tap water by distilled water machine (Figure 3.8).



Figure 3.8. Distilled water machine.

3.3. Experimental Details

In this study, there are six main mixes of work illustrated in Table 3.3. with the description of the mixture contents.

Table 3.3. A brief description of work mixtures.

Mix. No.	Geopolymer Powder Content %	Pumice Powder g	Fly Ash g	Alkali to Binder Ratio A/B	NaOH Concentration mol	Curing (day)
1	Pumice100%+ fly ash 0%	1200	0	0.30,0.34, 0.38, 0.42, 0.46, and 0.50	12	2,4,and 6
2	Pumice 80%+ fly ash 20%	960	240	0.30,0.34, 0.38, 0.42, 0.46, and 0.50	12	2,4,and 6
3	Pumice 60%+ fly ash 40%	720	480	0.30,0.34, 0.38, 0.42, 0.46, and 0.50	12	2,4,and 6
4	Pumice 40%+ fly ash 60%	480	720	0.30,0.34, 0.38, 0.42, 0.46, and 0.50	12	2,4,and 6
5	Pumice 20%+ fly ash 80%	240	960	0.30,0.34, 0.38, 0.42, 0.46, and 0.50	12	2,4,and 6
6	Pumice 0%+ fly ash 100%	0	1200	0.30,0.34, 0.38, 0.42, 0.46, and 0.50	12	2,4,and 6

3.3.1. Mixing, casting, and curing

According to Table 3.3, six mixes with different percentages of pumice and fly ash from 0% to 100% have prepared, six alkali to binder ratios (A/B) have used 0.30, 0.34, 0.38, 0.42, 0.46, and 0.50. NaOH as an alkaline activator solution was constant at a concentration of 12M with curing at 60°C for 2, 4 and 6 days. After preparing the alkaline solution, it was left for one day before using it in the mixing process. The dry powder of (P%+ FA%) for each type of mixtures was manually and carefully mixed until the mix homogenized (Figure 3.9). The mixing of pastes was done mechanically according to BS EN 196-1 by using an electric mixer machine (Figure 3.10). At first, the solution pours into the bowl of mixing, and then the binder powder was added. Then immediately the mixer started at low speed. The total time of mixing was 3 minutes. After mixing, the paste was directly poured into the molds (with dimensions 4cm×4cm×16cm), which were oiled well to guarantee easy molding (Figure 3.11). The casting was also done according to BS EN 196-1 by spreading with two layers and, after clamping the molds to the jolting table (Figure 3.12) the first layer was compacted using 60 jolts, then compacted the second layer with further 60 jolts. While performing each mixture, a small part was taken to test the consistency of that mixture by using the Vicat machine. After finishing each layer of casting the specimens were remained at room temperature for a 24-hour rest period and with nylon, vacuum film was covered to minimize evaporation of liquid solutions of geopolymer paste specimens (Figure 3.13). When the rest time ended, the samples were placed in the dry oven with a curing temperature of 60°C for different curing times of 2,4, and 6 days (Figure 3.14). After the curing time was ended, the samples were taken from the oven then left at room ambient temperature in the laboratory until the test time (Figures 3.15).

Figure 3.16 shows the specimens notation method that was used for different types of geopolymer mixtures for example P40%-R0.34-6d means P40 is the mixture with pumice 40% +fly ash 60%, R0.34 is alkaline to binder ratio (A/B) and 6d is the curing time of sample for period 6 days.



Figure 3.9. Sample of powder for 6 type mixtures.



Figure 3.10. Mixer machine.



Figure 3.11. Cast the mixture in the molds.



Figure 3.12. Jolting table.



Figure 3.13. Prepared samples before curing.

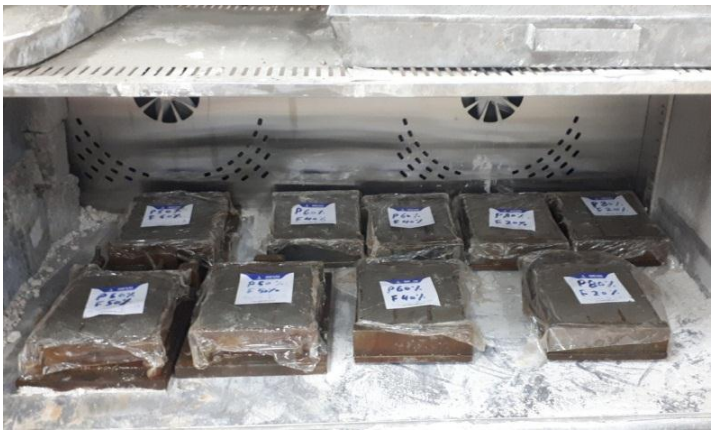


Figure 3.14. Curing of the samples in the oven.



Figure 3.15. Geopolymer samples for six mixtures.

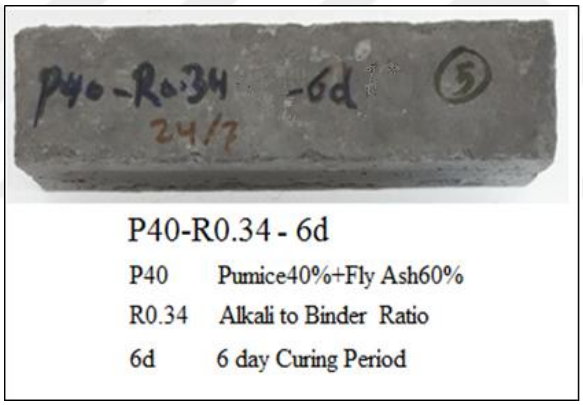


Figure 3.16. Specimen notation for different geopolymer paste.

3.4. Experimental Tests

3.4.1. Consistency test

Consistency test is a quick and easy test used to estimate the geopolymer paste consistency and workability for the fresh samples. The test for the fresh samples paste was carried out by using Vicat apparatus (Figure 3.17) according to BS EN 196-3. After mixing the geopolymer paste directly, a part of it placed in the mold of the device on a

glass plate and a needle with a diameter of 1.05 mm is set so that it is in contact with the surface and then it is allowed to fall and the amount of penetration is read.



Figure 3.17. Vicat apparatus.

3.4.2. Compressive strength

The compressive strength was determined using a hydraulic test machine type UTES (Figure 3.18). The test was conducted according to BS EN 196-1. The compressive strength test was carried out on the two halves of the prism specimens 4cm×4cm×16cm broken by suitable means or which obtained from the flexural test with compression on the side of the specimens over an area of 4cm×4cm and an increase in the rated load of 2200 N/s (Figure 3.19). The test was done at Geology Engineering Department in Van Yuzuncu Yil University.



Figure 3.18. Compressive strength machine.



Figure 3.19. Compressive strength test.

3.4.3. Flexural strength

A prism of 4cm×4cm×16cm was used to measure the flexural strength according to BS EN 196-1 by using a simply supported beam with a 10 mm span length (Figure 3.20). Flexural strength for geopolymer pastes was studied at age of 28 days. The loading rate was 50 N/s. Three specimens were used for each type of mixture. According to standard, the flexural strength value R_f in N/mm^2 was calculated according to equation "Eq. 3.1":

$$R_f = \frac{1.5 \times F_f \times l}{b^3} \quad (3.1)$$

Since R_f represents flexural strength in N/mm^2, F_f is the applied load in the middle of the specimen, l is equal to the space between two supports in mm and b is equal to the width of the prism side.



Figure 3.20. The flexural strength test

3.4.4. Ultrasonic Pulse Velocity

In general, Ultrasonic Pulse Velocity (UPV) tests are conducted in order to check the homogeneousness, the existence of possible cracks and defects. In this examination, the UPV test was carried out for the manufactured samples to evaluate the performance of the matrix with the studied parameters and the investigated categories. Pundit UPV tester was used to measure the transit time of wave passed through the specimens (Figure 3.21).



Figure 3.21. Ultrasonic pulse velocity tester.

3.4.5. Density and water absorption

Density and water absorption percentages were calculated for dry geopolymer samples of all types of mixtures according to the ASTM C642-13 to determine their influence by different percentages of (pumice%+fly ash%) and (alkali to binder ratio). The samples were first dried in the oven for 24 hours and then the mass was calculated. After the samples were placed in the water pool for 24 hours (Figure 3.22), the mass was calculated by removing surface moisture with a towel. The values of the properties were calculated according to the "Eq. 3.2"and "Eq. 3.3".

$$\text{Density (g/cm}^3) = \frac{M_a}{V} \tag{3.2}$$

$$\text{Water absorption\%} = \frac{M_a - M_b}{M_a} \times 100\% \tag{3.3}$$

Since M_a is the weight of the dry sample (g), M_b is the weight of the saturated sample (g)and V is the volume of the sample (cm^3).



Figure 3.22. The samples in the water pool.

3.4.6. Freezing-thawing test

All mixtures of geopolymer paste specimens at age 28 days were tested for freezing-thawing cycles according to ASTM C666/C666M-15. One day before the testing time all samples were placed into the water to absorb the water, then they were put in a freezer cabinet and subjected to five freezing-thawing cycles (Figures 3.23). The freezing period was 24 hours at -30 ℃ for each cycle, and the thawing period was also 24 hours at room temperature. After five cycles were finished, the compressive strength was performed to compare the result with the specimens that were not exposed to the freezing-thawing cycles.

3.4.7. Elevated temperature test

Samples of the three maximum compressive strength for each type of mixture were exposed to 200, 500, and 800°C for 1 hour in the elevated temperature oven (Figure 3.24) then the samples were cooled to room temperature**.** The aim of this test is to evaluate the effect of high temperature on geopolymer specimens and also to compare compressive strength between before and after exposure to high temperatures.



Figure 3.23. Freezing-thawing freezer.



Figure 3.24. Oven for elevated temperature.

4. RESULTS AND DISCUSSION

4.1. Introduction

Several tables and figures were used to show the findings of the experimental work tests for different parameters. In order to keep the sequence of the investigation in terms of the investigated parameters, the results were presented and discussed as follows:

- Consistency test results.
- Compressive strength results.
- Flexural strength results.
- Density results.
- Water absorption results.
- Freezing-thawing resistance results.
- Elevated temperature test results.
- Ultrasonic Pulse Velocity (UPV) results.

4.2. Consistency Test Results

Usually, the Vicat apparatus is used in tests of Portland cement to find the consistency of cement which represents the standard percentage of water to add to the cement. There are currently no special specifications for determining the standard consistency of geopolymers. In this research, by using different A/B ratios, the inference was made by penetrating the needle of Vicat apparatus to give an idea of the consistency, workability, and its effect on the degree of compaction, subsequently its relationship on A/B ratio and compressive strength of geopolymer past.

Results of penetration tests in the Vicat device (see Table 4.1.) indicate that with increasing of A/B ratio to a certain value, the penetration and workability increase like to water-cement ratio in ordinary Portland cement. Results showed that the pumice as source material with a high A/B ratio as 0.50 ratio gave only 18 mm penetration, while

after percentage of fly ash increases the penetration and workability increased. Where the precursor was 100% fly ash the penetration of 30 mm has achieved by the A/B ratio of only 0.30 (Figure 4.1). This due to the fly ash is characterized by very high fineness while the pumice powder is lesser in fineness and has high absorption characteristics.

Figure 4.2 illustrates the relationship between consistency penetration (mm) by Vicat apparatus and compressive strength for samples curing at for 6d at 60°C tested at 28 days, the result indicates that the best compressive strength achieved with (11, 17, 18, 19, 28, and 30 mm) for P100%+FA0%, P80%+FA20, P60%+FA40, P40%+FA60, P20%+FA80%, and P0%+FA100% respectively.

Table 4.1. The results of consistency penetration (mm) by Vicat apparatus with different mixtures and A/B.

Precursor	A/B	Workability Description	Penetration (mm)	Precursor	A/B	Workability Description	Penetration (mm)
Mix1 P100%+ FA0%	0.30	hard	1	Mix4 P40%+ FA60%	0.30	hard	2
	0.34	hard	1		0.34	medium	9
	0.38	hard	1		0.38	medium	19
	0.42	hard	5		0.42	soft	39
	0.46	medium	11		0.46	very soft	40
	0.50	medium	18		0.50	very soft	40
Mix2 P80%+ FA20%	0.30	hard	1	Mix5 P20%+ FA80%	0.30	medium	8
	0.34	hard	1		0.34	soft	28
	0.38	hard	5		0.38	soft	39
	0.42	medium	12		0.42	very soft	40
	0.46	medium	17		0.46	very soft	40
	0.50	soft	29		0.50	very soft	40
Mix3 P60%+ FA40%	0.30	hard	1	Mix6 P0 %+ FA100%	0.30	soft	30
	0.34	hard	5		0.34	soft	39
	0.38	medium	10		0.38	very soft	40
	0.42	medium	18		0.42	very soft	40
	0.46	soft	39		0.46	very soft	40
	0.50	very soft	40		0.50	very soft	40

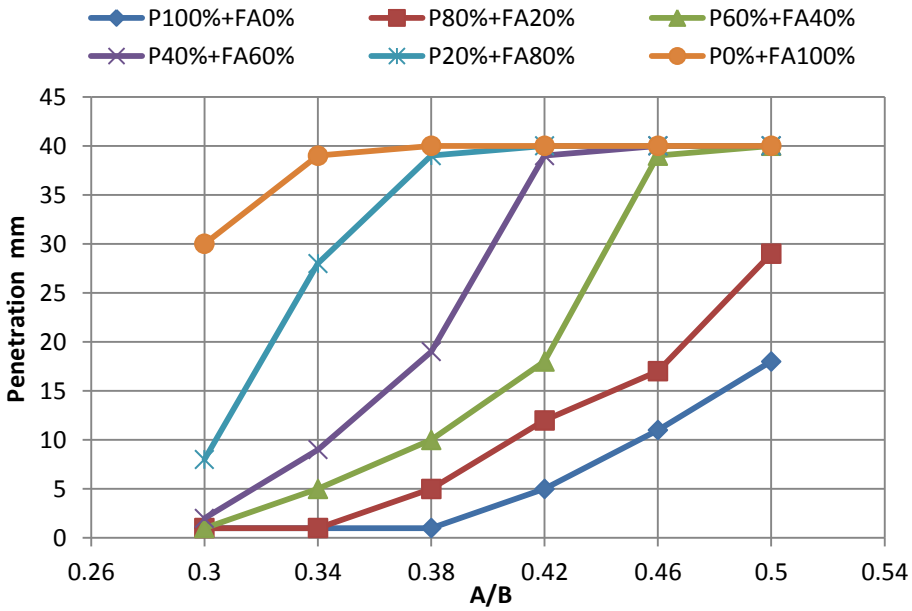


Figure 4.1. Relationship between A/B and consistency penetration (mm).

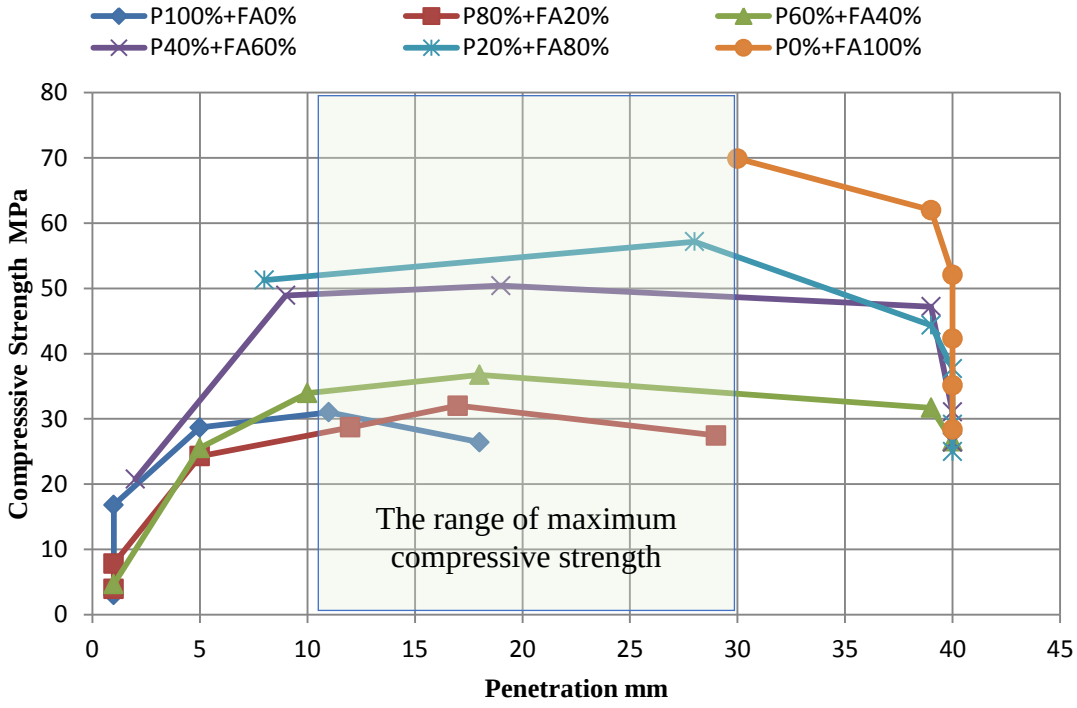


Figure 4.2. Relationship between consistency penetration (mm) and compressive strength (MPa).

4.3. Compressive Strength

To calculate the compressive strength, at least 3 tests were performed for 108 types of samples made with (6 type mixtures with changed content of P%+FA%, 6type of A/B ratio varying from 0.30 to 0.50, and 3 types of curing time at 60°C) tested at 7 and 28 days. The results average of tested specimens were recorded and all are shown in Table 4.2.which shows that the compressive strength at 7 days age was about 90% of that at 28 days. This means that there is no significant increase of compressive strength with time until 28 days, which is important in building construction if the speed of the construction process and the cost of curing are considered. This could be justified according to Bakharev et al. (1999). In their view, the speed of reaction rate of the alkali-activated material or geopolymer cement significantly higher than the hydration rates of ordinary Portland cement due to that it has a more open microstructure than Portland cement. However, the age of 28 days was adopted for all the other tests. The maximum compressive strength of the mixture with P100% was (28.64 MPa), this value increased by increasing FA content. A FA100% binder content gave the highest compressive strength 64.46MPa at 7 days.

4.3.1. Influences of the alkali to binder (A/B) ratio on compressive strength

Generally, the compressive strength decrease when the A/B ratio increase (Bondar et al., 2011b; Reddy et al. 2018). This like the water/cement ratio of Portland cement, when the alkali activator solution in the mixtures is increased, the amount of water also increases because the water within the alkali activator, resulting in a weaker microstructure (Bondar et al., 2011b). Figure 4.3, shows the effect of the A/B ratio in the compressive strength. In the case of a mixture with a pumice powder P100%-based geopolymer, the compressive strength increases when A/B increases from 0.30 to 0.46, and after that, it has decreased (in A/B ratio=0.50). Conversely, when the FA100% (P0%+FA100%) the compressive strength decreases as the A/B increased from 0.3 to 0.5.

Table 4.2. All results of compressive strength with concentration of alkaline in 12M, A/B ratio 0.30 , 0.34, 0.38, 0.42 , 0.46, and 0.50 for (2 , 4 and 6 day).

Sample Code	A/B	Compressive Strength MPa in 7 days test			Compressive Strength MPa in 28 days test		
		2d cured	4d cured	6d cured	2d cured	4d cured	6d cured
P100-R0.30	0.30	3.34	3.03	3.00	3.35	3.23	3.00
P100-R0.34	0.34	3.46	3.77	3.00	3.48	3.86	3.32
P100-R0.38	0.38	14.06	14.61	15.70	14.10	16.43	16.81
P100-R0.42	0.42	17.21	24.65	25.20	19.60	26.09	28.69
P100-R0.46	0.46	15.94	24.50	28.64	18.37	26.12	31.02
P100-R0.50	0.50	13.46	22.69	25.60	16.27	25.53	26.43
P80-R0.30	0.30	3.06	3.86	3.45	3.10	4.67	3.95
P80-R0.34	0.34	6.45	7.40	6.37	6.66	7.92	7.83
P80-R0.38	0.38	21.59	23.28	23.26	23.00	24.48	24.30
P80-R0.42	0.42	21.20	25.49	26.50	25.15	27.51	28.70
P80-R0.46	0.46	20.10	26.47	30.98	23.10	27.09	32.03
P80-R0.50	0.50	17.19	24.66	27.44	20.42	24.93	27.46
P60-R0.30	0.30	3.87	4.26	4.20	3.88	4.59	4.68
P60-R0.34	0.34	24.18	25.23	24.57	25.23	26.59	25.54
P60-R0.38	0.38	25.58	30.01	31.93	25.77	30.63	33.95
P60-R0.42	0.42	24.43	30.21	33.73	26.56	31.15	36.76
P60-R0.46	0.46	19.21	25.17	29.62	22.63	25.71	31.70
P60-R0.50	0.50	16.27	21.02	25.50	18.97	23.10	26.54
P40-R0.30	0.30	20.76	21.17	20.43	20.90	21.43	20.75
P40-R0.34	0.34	36.16	44.46	45.37	40.81	47.61	48.92
P40-R0.38	0.38	33.49	43.86	47.88	38.49	45.83	51.43
P40-R0.42	0.42	23.38	36.37	39.72	27.40	39.53	47.21
P40-R0.46	0.46	20.55	28.40	30.12	27.92	30.28	31.06
P40-R0.50	0.50	20.24	21.93	24.96	21.60	23.90	26.43
P20-R0.30	0.30	37.15	48.07	50.96	42.33	50.80	51.28
P20-R0.34	0.34	36.82	50.56	53.46	43.72	52.11	57.17
P20-R0.38	0.38	34.24	40.13	43.95	36.15	41.44	44.35
P20-R0.42	0.42	24.95	36.56	37.49	25.54	37.05	37.71
P20-R0.46	0.46	21.48	25.26	28.92	25.39	26.60	29.14
P20-R0.50	0.50	18.28	23.17	24.01	19.09	24.33	25.01
P 0-R0.30	0.30	52.63	59.64	64.46	56.52	63.77	69.90
P 0-R0.34	0.34	50.51	54.09	58.67	55.25	56.42	62.01
P 0-R0.38	0.38	37.45	41.67	52.05	48.70	51.36	52.10
P 0-R0.42	0.42	33.81	39.80	42.17	39.63	41.81	42.29
P 0-R0.46	0.46	26.55	30.06	34.49	27.72	31.79	35.13
P 0-R0.50	0.50	24.72	25.74	27.90	25.69	26.72	28.38

The most appropriate A/B ratio to achieve maximum compressive strength for each mixture type was different. Accordingly, it was revealed that there is a significant relationship between the A/B ratio and the compressive strength. The compressive strength of the pumice-fly ash geopolymer specimens develops with an A/B ratio to a certain value representing the optimum-active ratio of A/B, the compressive strength decreases more or less than this value. When the A/B ratio lower than the active-optimum value the development of compressive strength decreases because the amount of alkaline solution is not sufficient to complete the geopolymerization process. Decreased compressive strength will also occur when the A/B ratio exceeds the active-optimum value due to alkaline activator solution skipping the needs of geopolymerization process. The best A/B ratio that achieved the highest compressive strength was (0.46, 0.46, 0.42, 0.38, 0.34 and 0.30) for mixtures (P100%+FA0%, P80%+FA20, P60%+FA40, P40%+FA60, P20%+FA80% and P0%+FA100%) respectively. For the mixture of P100%, the correlation equation between A/B and compressive strength was (*fc=6.06(A/B)-3*) with a coefficient of determination (R^2*=0.8071*). For the mixture of FA100% the correlation equation was (*fc=-8.51(A/B) +78.10*) and a coefficient of determination (R^2*=0.9951*) indicates a good relationship between.

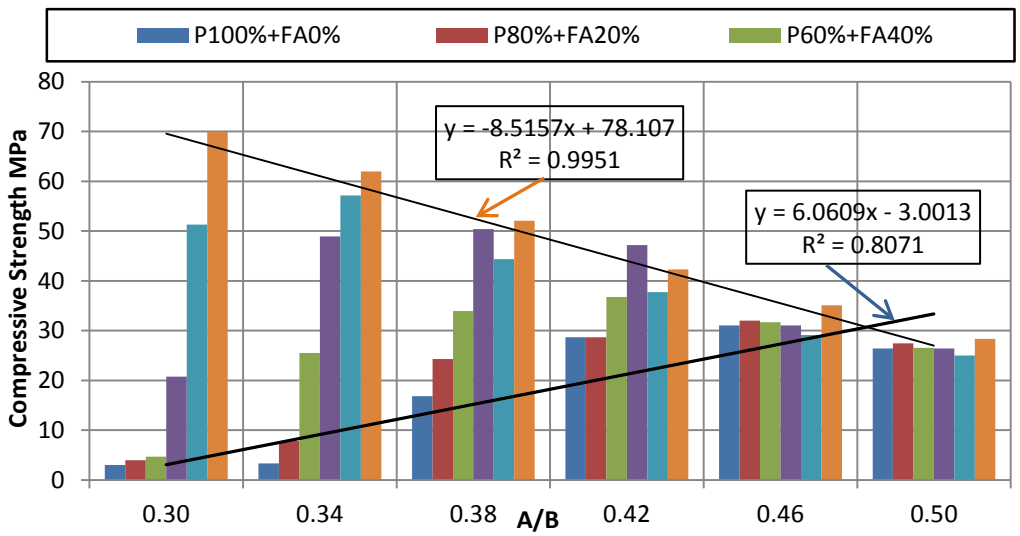


Figure 4.3. Compressive strength of P-FA geopolymer for different A/B at 6 days curing tested at 28 days.

4.3.2. Effect the cure time on compressive strength

Figures 4.4.to 4.9 show how the curing time affects the compressive strength of geopolymer cement at three types of curing times 2,4 and 6 days. Figures show that the increase in compressive strength increases as the curing time lengthen from 2 to 6 days. Optimum strengths were obtained at 6 days with the optimum-active A/B ratio for 6 different types of mixtures. Figure 4.4 which includes the mixture content 100% of pumice, illustrates that the development of strength at 60 °C is weak in the 2 days curing time, this strength increases by a high variance when curing time at 6 days, the percentage of strength gained in 2 days was 59.22 % after 2 days and 84.20% after 4 days from maximum strength of P100-R0.46 mixture tested at 28 days. This means pumice-geopolymer cement needs more time to achieve maximum strength, this results also consistent with some other studies' results (safari et al., 2020; Ibrahim et al., 2017). While the proportion of FA in mixtures increased from 20% to 80% (Figure 4.5 to 4.8), the speed of gaining strength gradually increased. Also, the variance in results between 2 and 6 curing time was decreased. When the mixture content 100% of FA (Figure 4.9.) the gain strength percentage from 6 days compressive strength becomes 80.86% after 2days and 91.23% after 4 days tested at 28 days (for P0-R0.30 mixture), this indicates that the fly ash needs a shorter period to develop and gain strength compared to pumice. These results are also consistent with several other studies results (Sukmak et al., 2013; Hardjito et al., 2004; Mishra et al., 2008; Memon et al., 2011)

Also, noticed that if the curing time continues to a further optimum-active A/B ratio, the strength decreases. This may be due to the alkaline solution interaction ended and the polymerization has been finished, with continuous curing time on the oven leading to micro crack development. Figure 4.10 shows the relationship between cure time and the compressive strength for maximum values. For the mixture of P100%, the correlation equation between cure time and compressive strength was ($fc=3.16T+12.52$) with the coefficient of determination ($R^2=0.9834$). For the mixture of 100%FA, The correlation equation was ($fc= 3.34T+50$) and the coefficient of determination ($R^2=0.9977$) indicates a good relationship between.

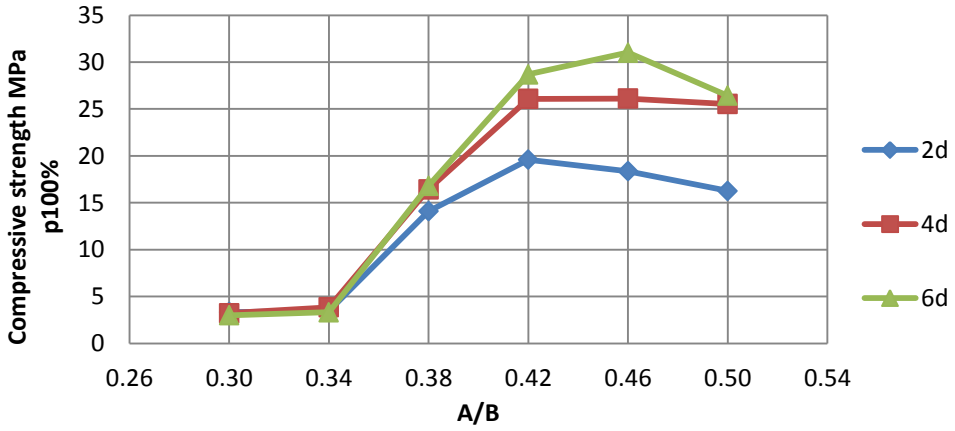


Figure 4.4. Effect the cure time on the compressive strength for P100%+FA0%.

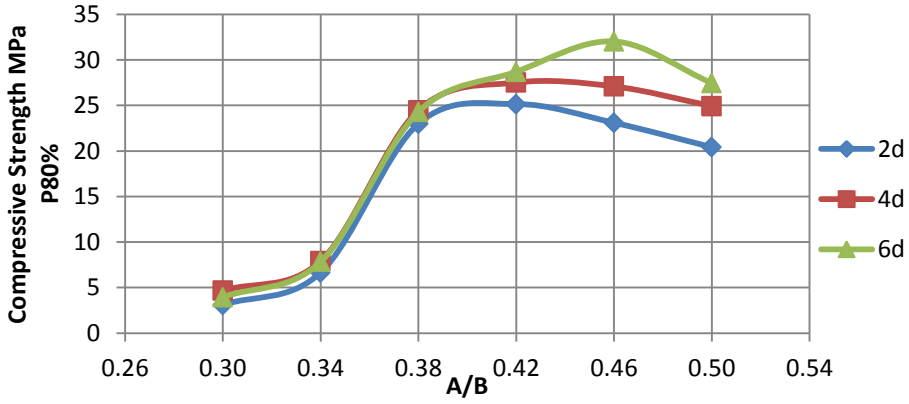


Figure 4.5. Effect the cure time on the compressive strength for P80%+FA20%.

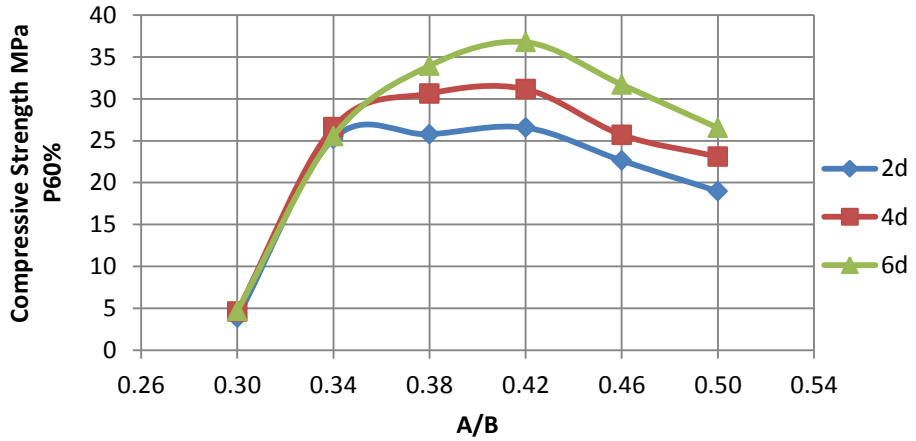


Figure 4.6. Effect the cure time on the compressive strength for P60%+FA40%.

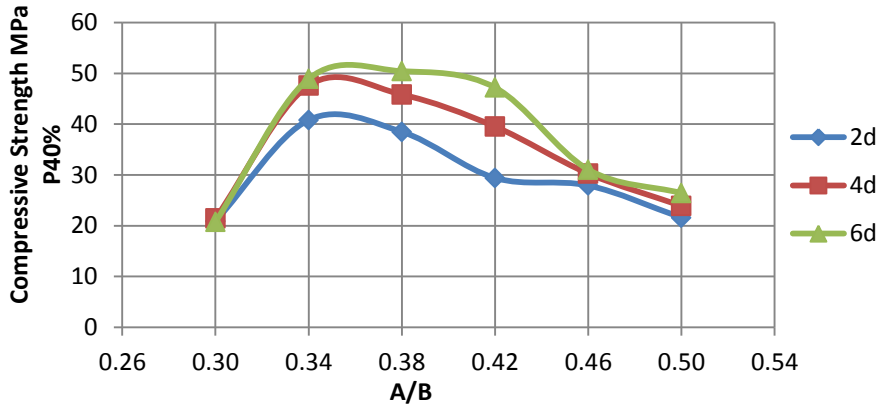


Figure 4.7. Effect the cure time on the compressive strength for P40%+FA60%.

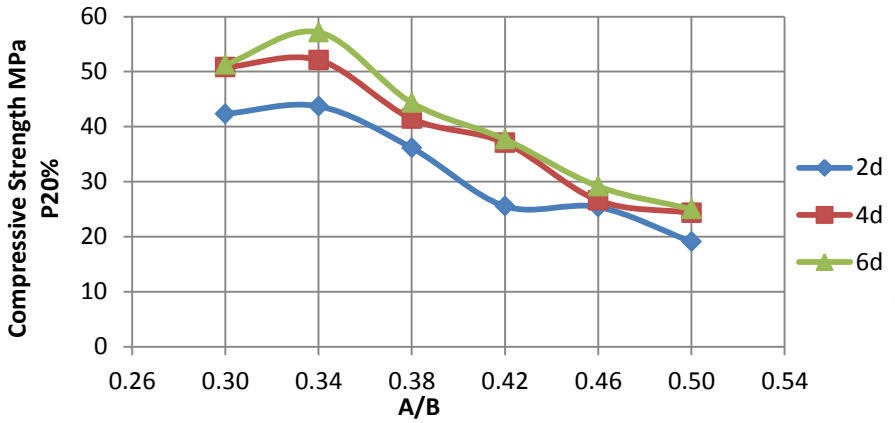


Figure 4.8. Effect the cure time on the compressive strength for P20%+FA80%.

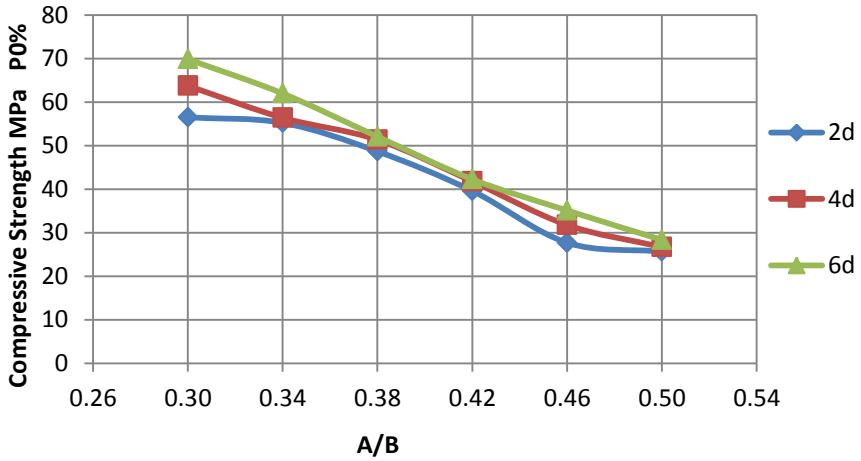


Figure 4.9. Effect the cure time on the compressive strength for P0%+FA100%.

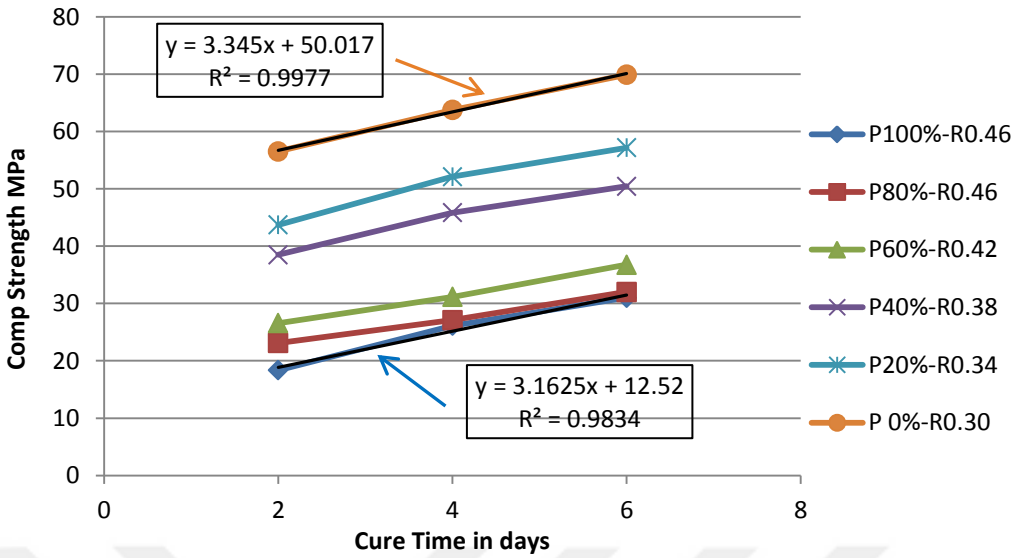


Figure 4.10. Relationship between cure time and the compressive strength.

4.3.3. Influence of the percentage pumice and fly ash on compressive strength.

The most important objective of the research is to know the changes in the compressive strength of the new geopolymeric cement through the use of a two-part source material (pumice+fly ash) and investigate the impact of varying the percentages from 0 % to 100% alternately on compressive strength. FA is widely used as a base material for producing geopolymers. Pumice powder was blended with FA to produce geopolymer paste. Based on the powder content, 6 types of the mixture are classified as geopolymer cement. Figure 4.11 illustrates the compressive strength for different types of mixtures. When the paste content was 100% Pumice, the maximum compressive strength at age 28 days (6d curing at 60°C) was 31.02 MPa as shown in the mix (P100-R0.46), while the maximum compressive strength was 69.90 MPa when the paste contained 100% fly ash, as shown in the mix (P0-R0.30).

It was found that if the percentage of replacement FA increases in the binder, the compressive strength increases (Figure 4.12). At the 20% ratio of the FA, the strength increased by 3.26 percent as in the (P80-R0.46), at 40% ratio of FA the strength increased 18.50 % as in the (P60-R0.42), at 60% ratio of FA the strength increase 62.57 % as in the (P40-R0.38) and the strength increases by 84.30% at 80% ratio of FA as in

the (P20-R0.34). This means that pumice with fly ash can be used to produce geopolymers.

The difference in behavior between pumice and FA can be noticed. The reason is that the fly ash possesses good workability as well as the optimum chemical composition corresponds considerably to the requirements of the geopolymers. The optimum compressive strength was (31.02, 32.03, 36.76, 50.43, 57.17, and 69.90) for mixtures (P100-R0.46, P80-R0.46, P60-R0.42, P40-R0.38, P20-R0.34, and P0-R0.30) respectively at the ages 28 days(cure for 6 days at 60°C).

The correlation equation between FA% content and compressive strength for optimum values was (*fc=0.4FA%+25.96*) with a coefficient of determination (R^2*=0.9404*) indicate a good relation between FA% content and compressive strength.

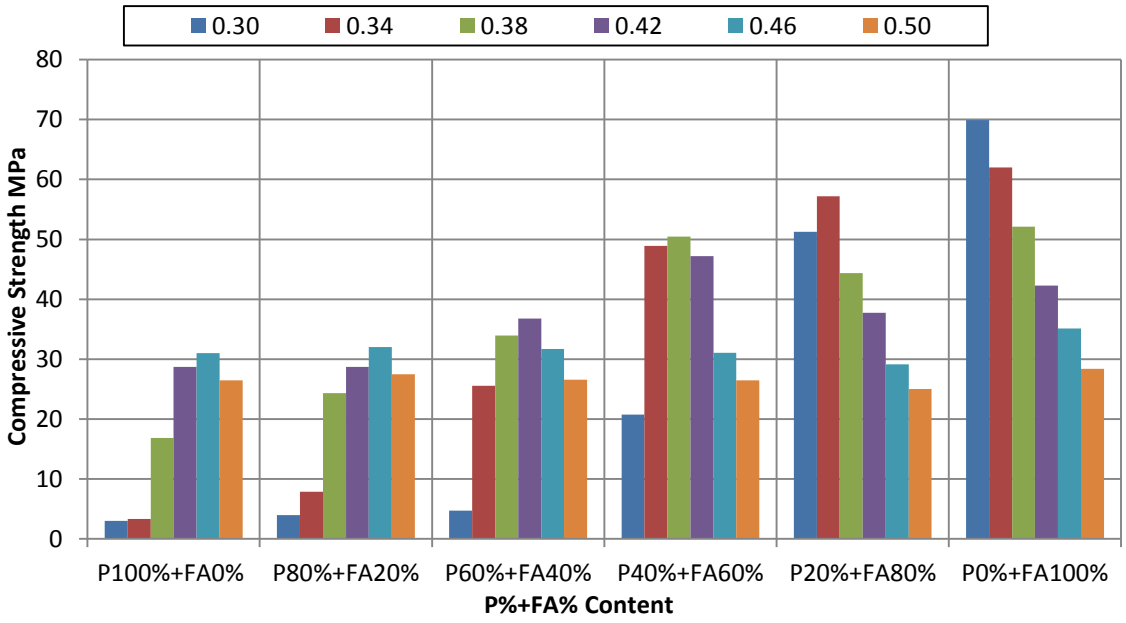


Figure 4.11. Compressive strength of geopolymer paste for different mixtures (cure for 6 days at 60°C) tested at 28 days.

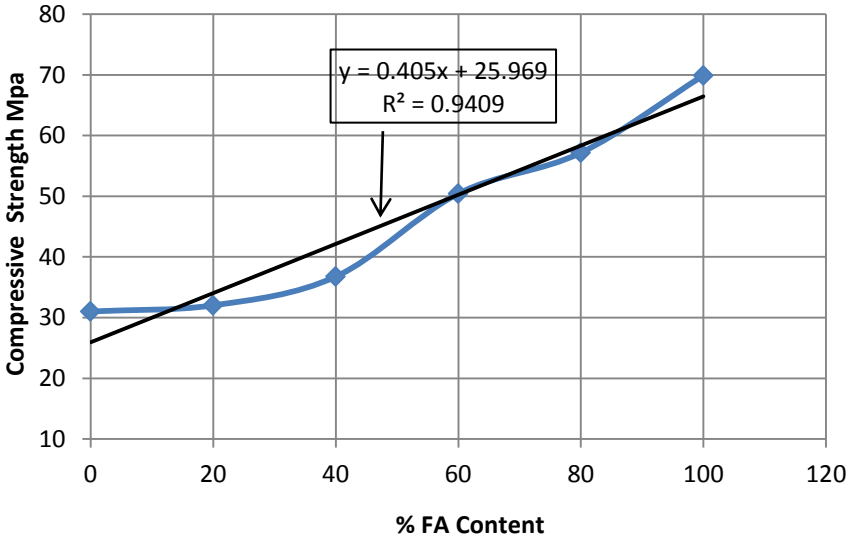


Figure 4.12. Effect of FA% content on compressive strength for optimum values.

4.4. Flexural Strength

Prisms of 4cm×4cm×16cm were used to perform this test at 28 days. Table 4.3 and Figures 4.13 to 4.18 present the results of flexural strength for 3 types of curing time, showing the influence of A/B and curing time on flexural strength for each type of geopolymer paste by the percentage of pumice powder and fly ash. It appears from the figures that the behavior of the affecting factors with flexural strength nearly similar to its behavior of compressive strength. Maximum flexural strength was obtained at 6 days with the optimum-active A/B ratio for 6 different types of mixtures, any increase or decrease of optimum-active A/B ratio would result in a decrease in flexural strength. The reason for this behavior is discussed in section 4.3 related to the compressive strength and there is no need for repetition.

It is noticeable, however, that where the geopolymer specimen consists of 100 % pumice powder, the flexural strength will be high and then drop until P60%+FA40% after that (on P40%+FA60%) returns to increase (Figure 4.19). The optimum flexural strength was (8.98, 8.23, 6.02, 6.26, 6.75 and 8.79) for mixtures (P100-R0.46, P80-R0.46, P60-R0.42, P40-R0.38, P20-R0.34, and P0-R0.30) respectively at 28 days.

Table 4.3. Results of flexural strength (MPa) with concentration of alkaline in 12M, A/B ratio 0.30, 0.34, 0.38, 0.42, 0.46, and 0.50 for (2, 4 and 6 day).

Sample Code	A/B	Flexural Strength MPa		
		cure at 2d	cure at 2d	cure at 6d
P100-R0.30	0.3	0.75	1.29	1.22
P100-R0.34	0.34	1.27	1.57	1.64
P100-R0.38	0.38	4.01	4.45	4.90
P100-R0.42	0.42	6.21	7.10	7.62
P100-R0.46	0.46	6.61	7.97	8.98
P100-R0.50	0.5	6.19	7.34	8.53
P80-R0.30	0.3	1.29	1.27	1.24
P80-R0.34	0.34	2.86	3.23	2.74
P80-R0.38	0.38	4.24	4.92	4.76
P80-R0.42	0.42	6.12	6.63	7.01
P80-R0.46	0.46	6.68	7.55	8.46
P80-R0.50	0.5	6.56	7.05	7.80
P60-R0.30	0.3	2.88	2.79	2.84
P60-R0.34	0.34	4.34	4.10	3.82
P60-R0.38	0.38	4.90	4.97	5.30
P60-R0.42	0.42	4.73	5.34	6.02
P60-R0.46	0.46	4.50	4.99	5.41
P60-R0.50	0.5	4.01	3.35	3.21
P40-R0.30	0.3	4.57	4.24	4.45
P40-R0.34	0.34	4.62	5.27	5.98
P40-R0.38	0.38	4.69	5.46	6.26
P40-R0.42	0.42	5.16	5.41	5.88
P40-R0.46	0.46	4.73	4.76	4.97
P40-R0.50	0.5	4.48	3.82	3.52
P20-R0.30	0.3	5.06	5.81	6.54
P20-R0.34	0.34	5.18	6.21	6.75
P20-R0.38	0.38	4.99	5.51	6.61
P20-R0.42	0.42	4.69	4.73	5.30
P20-R0.46	0.46	4.45	3.82	3.77
P20-R0.50	0.5	2.74	2.70	2.67
P0-R0.30	0.3	7.50	8.27	8.79
P0-R0.34	0.34	6.33	6.63	7.31
P0-R0.38	0.38	5.39	6.49	7.08
P0-R0.42	0.42	5.27	5.74	5.60
P0-R0.46	0.46	5.23	5.30	4.85
P0-R0.50	0.5	5.09	4.85	3.96

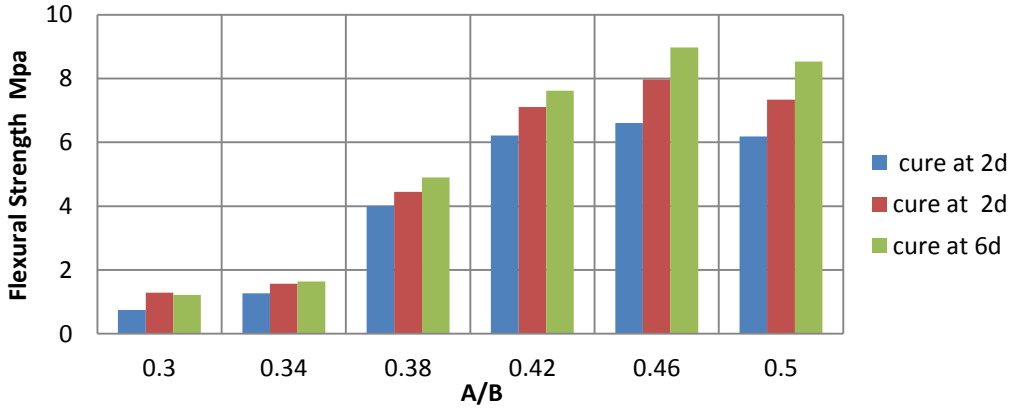


Figure 4.13. Flexural strength for P100%+FA0% geopolymer pastes at 28 days.

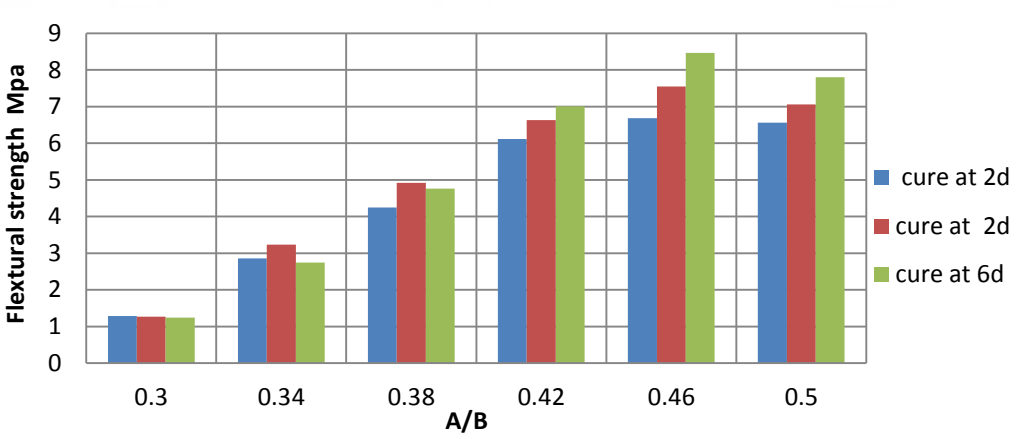


Figure 4.14. Flexural strength for P80%+FA20% geopolymer pastes at 28 days.

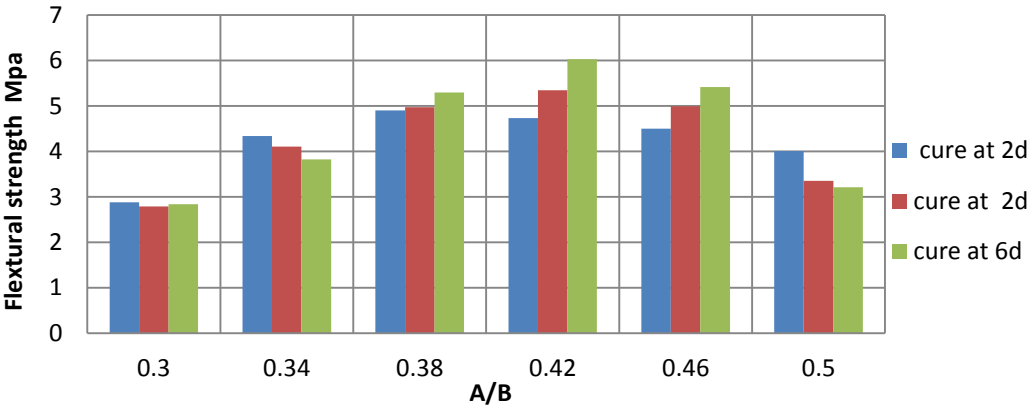


Figure 4.15. Flexural strength for P60%+FA40% geopolymer pastes at 28 days.

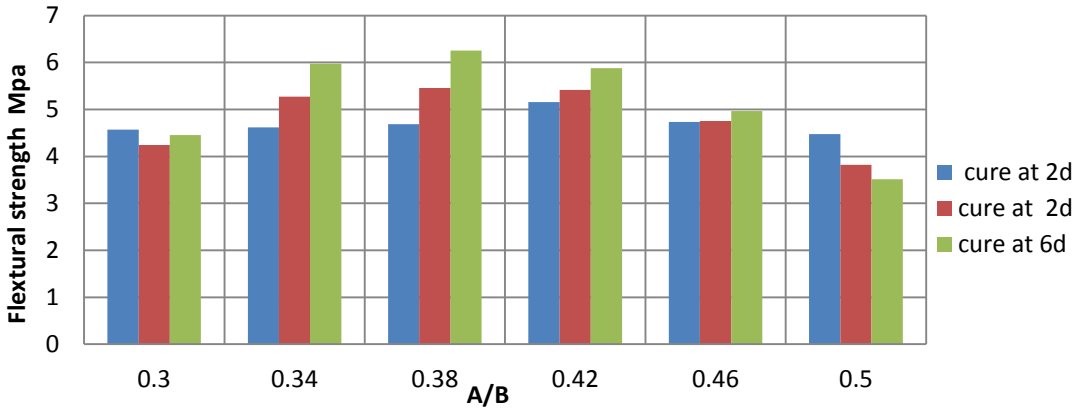


Figure 4.16. Flexural strength for P40%+FA60% geopolymer pastes at 28 days.

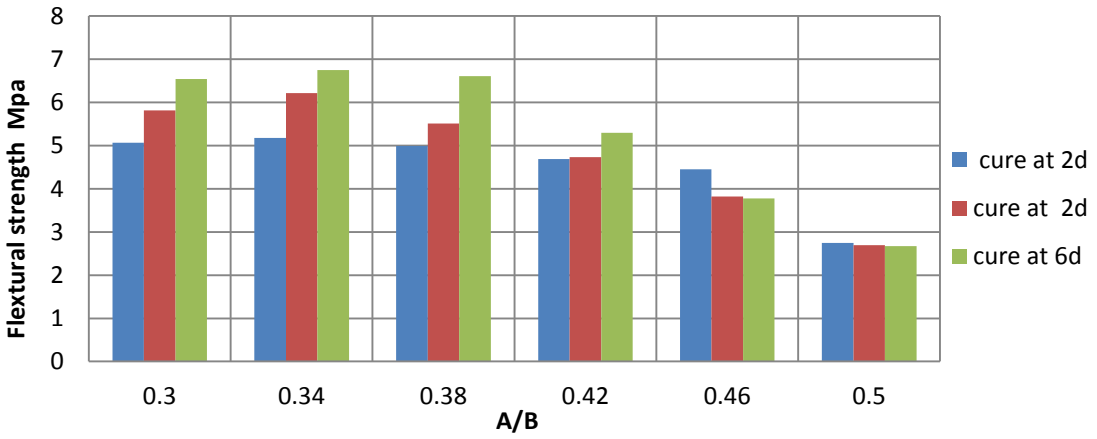


Figure 4.17. Flexural strength for P20%+FA80% geopolymer pastes at 28 days.

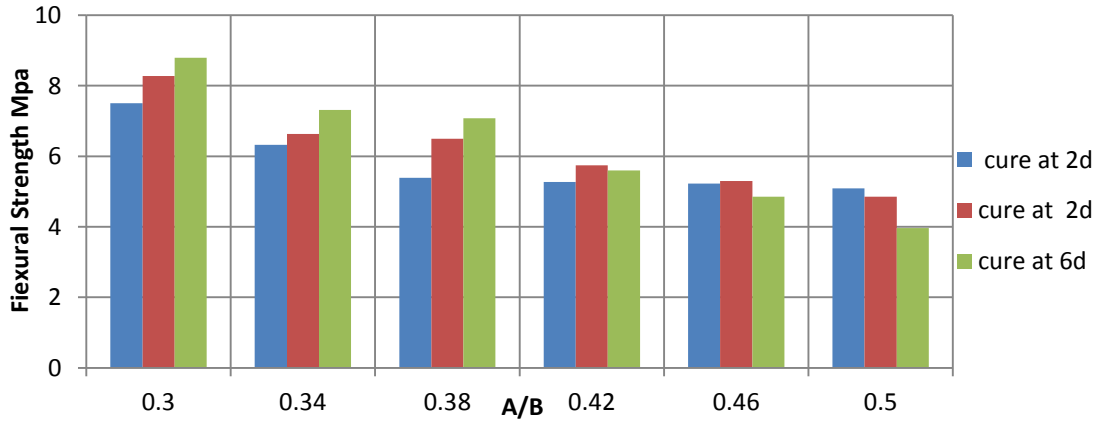


Figure 4.18. Flexural strength for P0%+FA100% geopolymer pastes at 28 days.

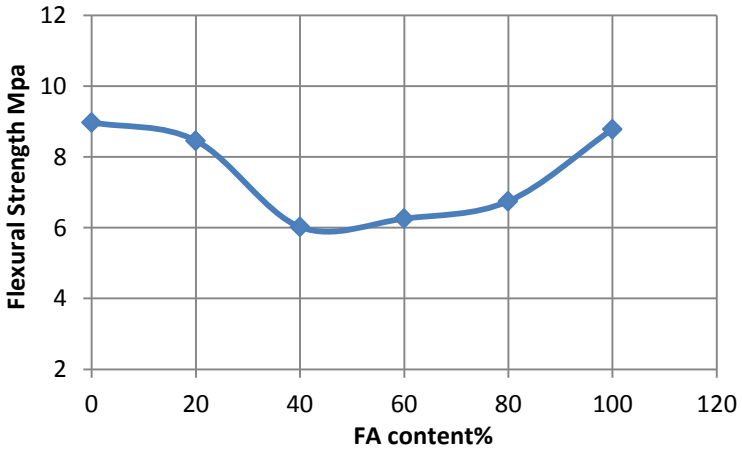


Figure 4.19. Flexural strength and FA% content for optimum values.

4.5. Density Test Result

All samples were weighted and calculated density for 3 type curing time (Table 4.4.). In general, for all samples with a rise in the cure time from 2 to 6d at 60°C the density decreases. This is normal due to a polymerization reaction completed at 6 days and more water evaporation occurs. As an example Figure 4.20 and Figure 4.21, this showed the effect of cure time in density.

Figure 4.22 shows the density of geopolymer paste for different mixtures at 6 days curing time. The density values decreased slightly with an increase in the rate of the pumice powder used in the preparation of the geopolymer paste. The reason, perhaps, for the specific gravity of the pumice powder is less than the specific gravity of the FA, therefore, the density value increased when the percentage of fly ash is high. For any type of mixtures, the density values vary depending on the A/B ratio, when the A/B ratio is lower than the optimum value with hard workability (like P100%+FA0% and P80%+FA20% mixtures), the density will be reduced, because the alkaline solution is not sufficient to complete the polymerization process and the vacuums are formed. When the A/ B ratio is higher than the optimum A/B ratio and mixtures with very soft workability (like P20%+FA80%and P 0%+FA100% mixtures) the density will decrease due to the high percentage of water that causes formation. The density values for the

maximum compressive strength were (1770, 1758, 1727, 1750, 1797, and 1809 Kg/m^3) for (P100-R0.46, P80-R0.46, P60-R0.42, P40-R0.38, P20-R0.34, and P0-R0.30).

Table 4.4. The results of density Kg/m^3.

Sample Code	A/B	Density Kg/m3		
		2d	4d	6d
P100-R0.30	0.3	1016	1012	1008
P100-R0.34	0.34	1211	1203	1188
P100-R0.38	0.38	1660	1641	1617
P100-R0.42	0.42	1797	1789	1766
P100-R0.46	0.46	1801	1793	1770
P100-R0.50	0.5	1832	1809	1789
P80-R0.30	0.3	1125	1121	1059
P80-R0.34	0.34	1332	1344	1293
P80-R0.38	0.38	1758	1680	1672
P80-R0.42	0.42	1797	1766	1758
P80-R0.46	0.46	1797	1785	1758
P80-R0.50	0.5	1805	1789	1766
P60-R0.30	0.3	1250	1211	1191
P60-R0.34	0.34	1719	1695	1672
P60-R0.38	0.38	1734	1711	1688
P60-R0.42	0.42	1813	1758	1727
P60-R0.46	0.46	1789	1719	1695
P60-R0.50	0.5	1719	1641	1602
P40-R0.30	0.3	1484	1445	1438
P40-R0.34	0.34	1816	1789	1730
P40-R0.38	0.38	1836	1797	1750
P40-R0.42	0.42	1832	1762	1738
P40-R0.46	0.46	1766	1719	1652
P40-R0.50	0.5	1703	1680	1621
P20-R0.30	0.3	1840	1828	1789
P20-R0.34	0.34	1859	1840	1797
P20-R0.38	0.38	1801	1793	1758
P20-R0.42	0.42	1793	1766	1738
P20-R0.46	0.46	1777	1750	1680
P20-R0.50	0.5	1641	1590	1578
P0-R0.30	0.3	1824	1816	1809
P0-R0.34	0.34	1805	1793	1785
P0-R0.38	0.38	1789	1742	1758
P0-R0.42	0.42	1758	1707	1684
P0-R0.46	0.46	1750	1691	1660
P0-R0.50	0.5	1699	1633	1586

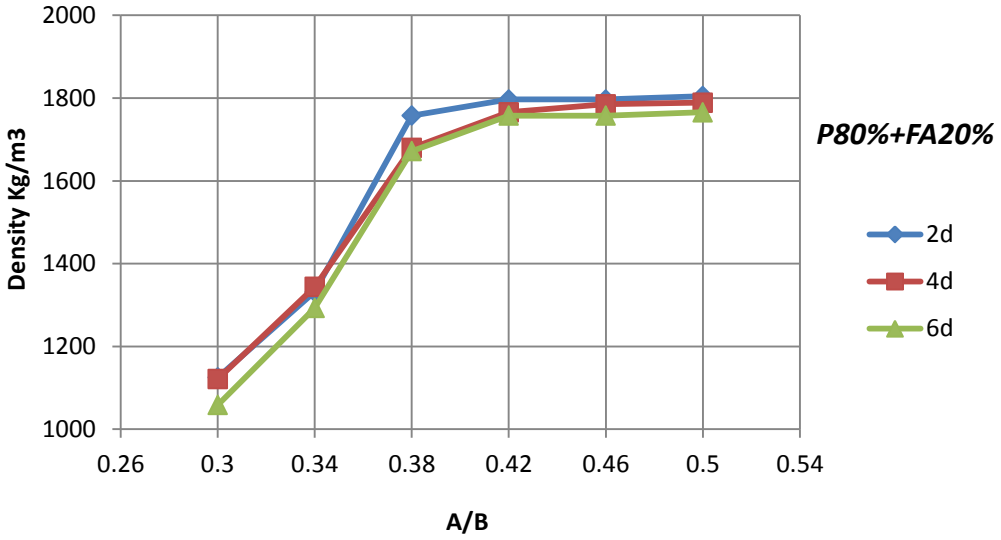


Figure 4.20. The density of geopolymer paste for P80%+FA60%.

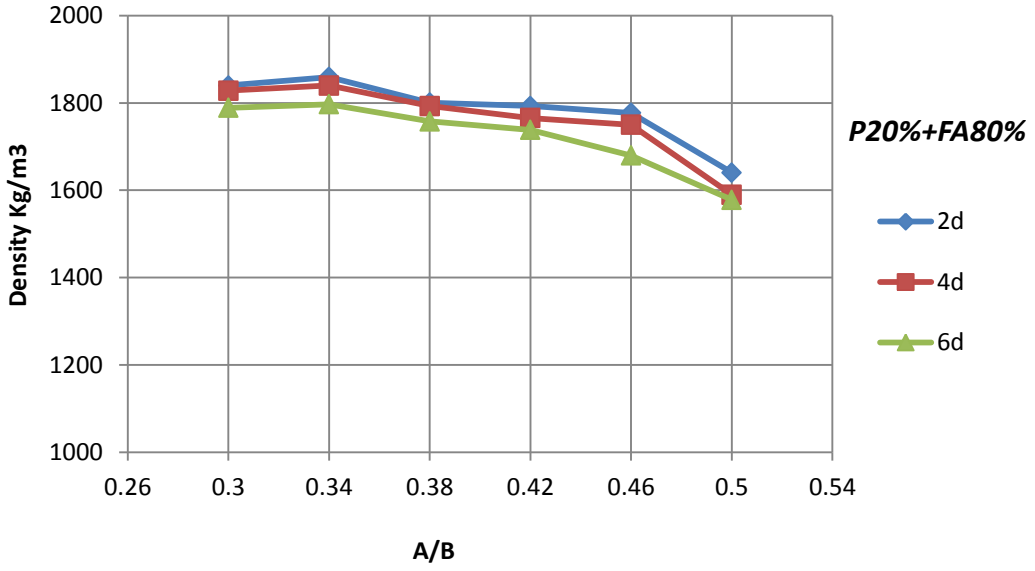


Figure 4.21. The density of geopolymer paste for P20%+FA80%.

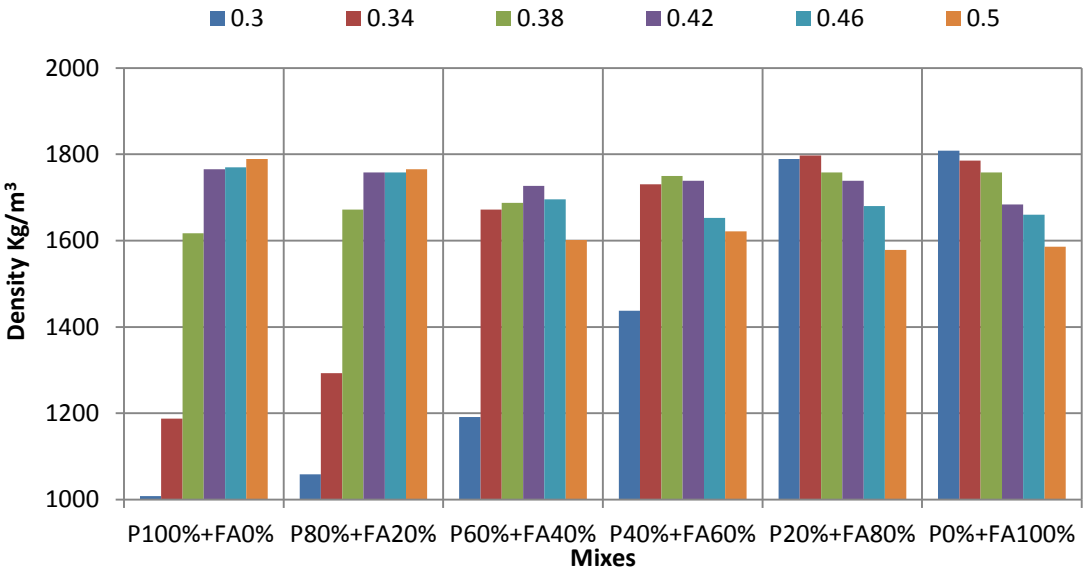


Figure 4.22. The density of geopolymer paste for different mixes at 6 days curing time.

4.6. Water Absorption

Water absorption of the binder is important to detect the strength and durability of the structure. Tests of water absorptions were made for mixes at 6 days curing time. As shown in Table 4.5 and Figure 4.23 that the mixing between pumice and fly ash led to a decrease in the water absorption. So the reduction in water absorption could be due to a high fineness for FA particles which interfere in between pumice powder particles (which are larger size) with optimum A/B ratio and through the polymerization process, the pores are closed so the water absorption decrease. When the A/B ratio is low in samples with P100% and P80%, the water absorption was high, because the alkaline liquid is not adequate to complete the polymerization process and voids are formed. When the A/ B ratio is high in samples with fly ash FA%, so the water content will increase, due to form the voids and leads to increased water absorption. In this test, water absorption values of fewer than 10 % were obtained (for optimum compressive strength and A/B ratio). All the high-quality concretes have fewer than 10 % water absorption values (Neville, 2005).

Table 4.5. Water absorption at 6 days curing time.

A/B	P100%+ FA0%	P80%+ FA20%	P60%+ FA40%	P40%+ FA60%	P20%+ FA80%	P0%+ FA100%
0.3	21.71	15.79	12.73	9.57	9.42	9.04
0.34	13.55	10.34	9.48	8.16	9.15	9.18
0.38	9.57	7.65	4.80	6.76	9.33	10.79
0.42	5.38	5.60	4.81	6.52	9.60	11.11
0.46	4.04	4.41	4.17	6.31	8.77	12.38
0.5	4.20	4.45	4.40	7.27	7.59	14.53

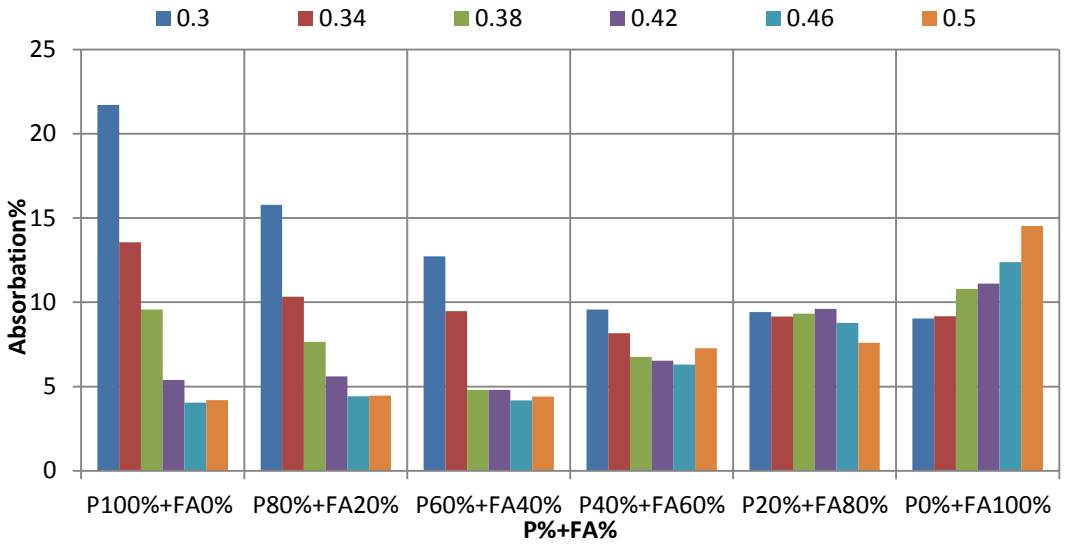


Figure 4.23. Water absorption of geopolymer paste for different mixes.

4.7. Freezing-Thawing Resistance

In this search, the influence of freezing-thawing on compressive strength for pumice and fly ash geopolymer paste specimens was investigated. The investigation included exposing all types of samples to 5 cycles of freezing-thawing then testing the compressive strength to know how the curing time, percentage of P%+FA%, and A/B ratio effect in, also to compare the results between before and after exposing to freezing-thawing. The Figure 4.24 to 4.29 illustrates compressive strength before and after the freezing-thawing cycle. As a primary observation, the compressive strength after freezing-thawing cycles of specimens was less than from which was obtained without exposed to any freezing-thawing resistance.

Table 4.6. The results of compressive strength (MPa) after freezing-thawing.

Sample Code	A/B	Compressive Strength MPa After Freezing-Thawing		
		2d cured	4d cured	6d cured
P100-R0.30	0.30	0.50	0.50	0.50
P100-R0.34	0.34	0.60	1.00	1.00
P100-R0.38	0.38	5.27	9.67	9.80
P100-R0.42	0.42	7.45	13.27	17.57
P100-R0.46	0.46	7.35	13.27	17.56
P100-R0.50	0.50	6.18	11.63	15.41
P80-R0.30	0.30	1.65	2.00	2.06
P80-R0.34	0.34	3.54	4.12	4.06
P80-R0.38	0.38	13.90	14.40	14.20
P80-R0.42	0.42	15.10	16.04	17.48
P80-R0.46	0.46	13.50	16.66	19.96
P80-R0.50	0.50	10.34	12.80	13.99
P60-R0.30	0.30	1.09	1.95	2.52
P60-R0.34	0.34	20.52	20.52	20.36
P60-R0.38	0.38	21.82	24.69	27.43
P60-R0.42	0.42	19.94	23.72	29.02
P60-R0.46	0.46	15.01	17.03	19.81
P60-R0.50	0.50	12.74	14.15	16.38
P40-R0.30	0.30	9.51	9.30	10.82
P40-R0.34	0.34	32.00	36.72	39.26
P40-R0.38	0.38	30.01	36.01	41.30
P40-R0.42	0.42	22.66	30.84	37.56
P40-R0.46	0.46	19.57	23.56	24.76
P40-R0.50	0.50	19.01	19.22	21.12
P20-R0.30	0.30	33.07	35.42	36.74
P20-R0.34	0.34	34.52	42.78	45.95
P20-R0.38	0.38	28.95	33.03	34.81
P20-R0.42	0.42	20.31	29.09	29.32
P20-R0.46	0.46	19.59	20.61	23.08
P20-R0.50	0.50	14.71	15.56	15.90
P 0-R0.30	0.30	50.45	56.98	63.93
P 0-R0.34	0.34	40.03	41.50	53.40
P 0-R0.38	0.38	33.00	34.95	37.18
P 0-R0.42	0.42	27.28	26.62	30.25
P 0-R0.46	0.46	15.35	17.92	24.73
P 0-R0.50	0.50	17.40	18.39	20.29

4.7.1. Influence of cure time on freezing-thawing resistance

Figure 4.24 to 4.29 show the influence of the curing period on the freezing and thawing resistance of geopolymer pastes at different A/B (0.30, 0.34, 0.38, 0.42, 0.46, and 0.50).In general, the compressive strength after 5 cycles of freezing-thawing gradually increased with increased curing time from 2 to 6 days. This may be attributed to continuing of the reactivity between aluminosilicate materials and alkaline solution, also most of the polymerizations are complete after long periods of curing. That led to improvement in freezing and thawing durability property of geopolymer paste in 6 days. Figure 4.30 shows the percentage of compressive strength loss for optimum values. In general the strength properties of geopolymer paste decrease after 5 cycles of freezing-thawing. The results were revealed that the amount of drop in the 6 days curing time less than 2 and 4 days, this was very clear in the P100% mixture, where the percentage of reduction (%) at curing 2, 4, and 6 days reached (59.99%, 51.07%, and 43.39 %) respectively. This percentage decreases with increase fly ash percentage, as it was reached (10.56 %, 10.51%, and 8.54%) at a curing 2, 4 and 6 days respectively. It is also noted that the strength loss percentage was converged at three types of curing time when the FA% was 40% to 80% in mixtures. The results indicate the significance of an adequate curing period to complete the reaction process with convenient temperature.

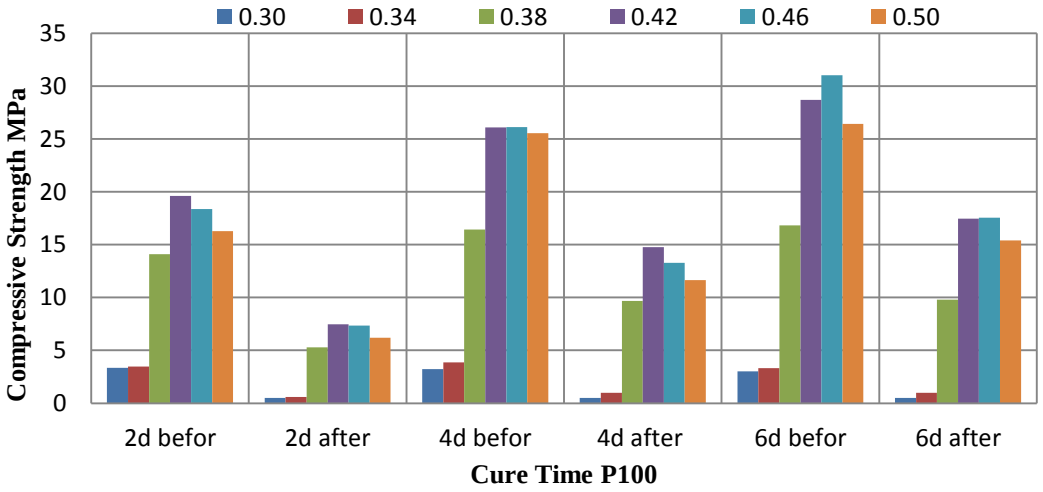


Figure 4.24. Influence of cure time on the compressive strength after 5 cycles of freezing-thawing for P100%+FA0%.

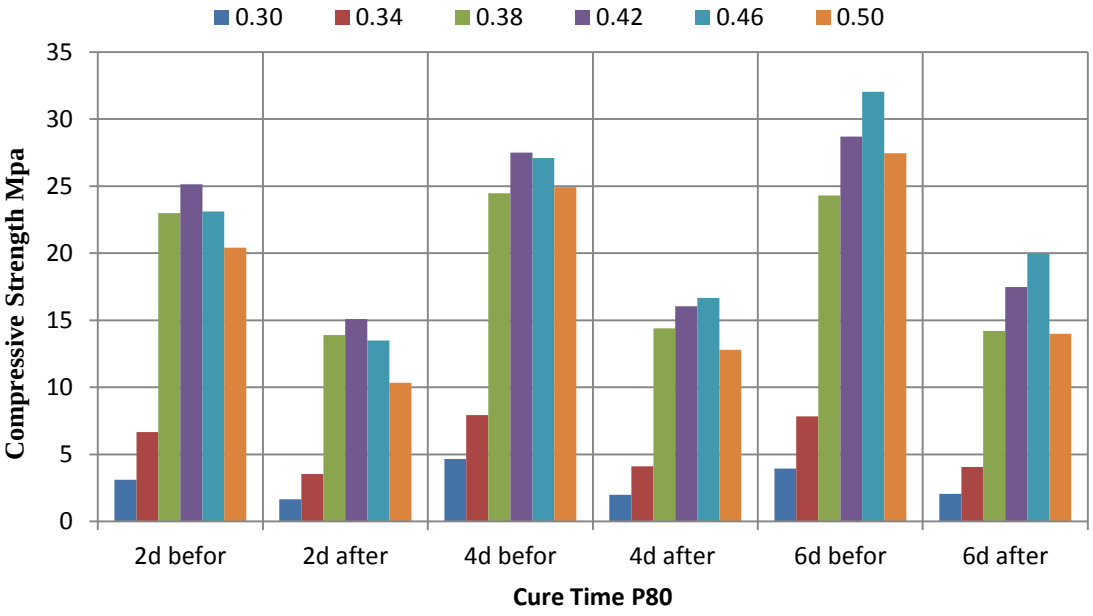


Figure 4.25. Influence of cure time on the compressive strength after 5 cycles of freezing-thawing for P80%+FA20%.

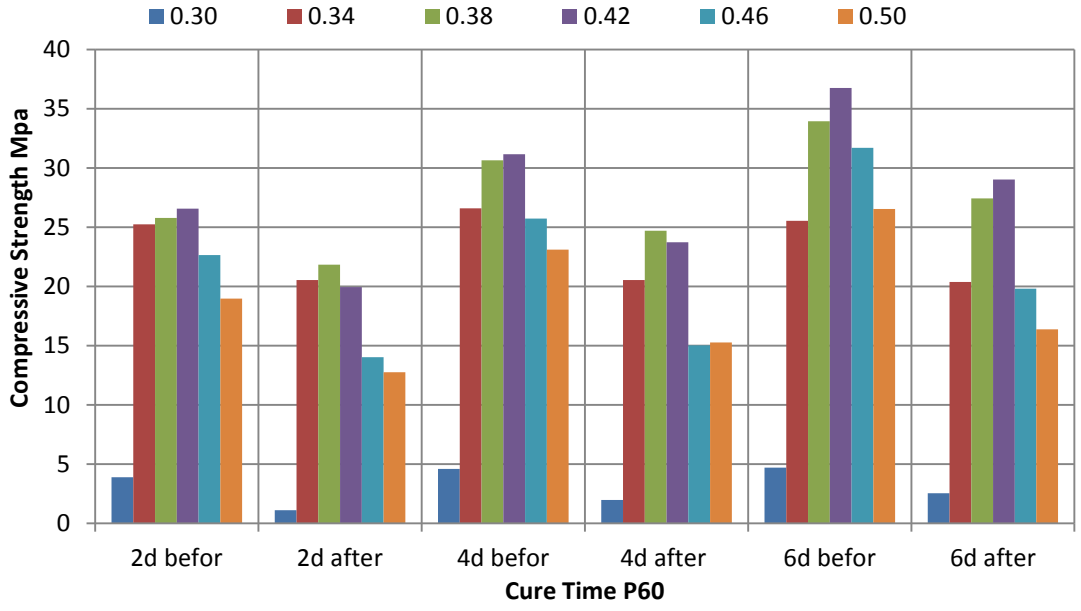


Figure 4.26. Influence of cure time on the compressive strength after 5 cycles of freezing-thawing for P60%+FA40%.

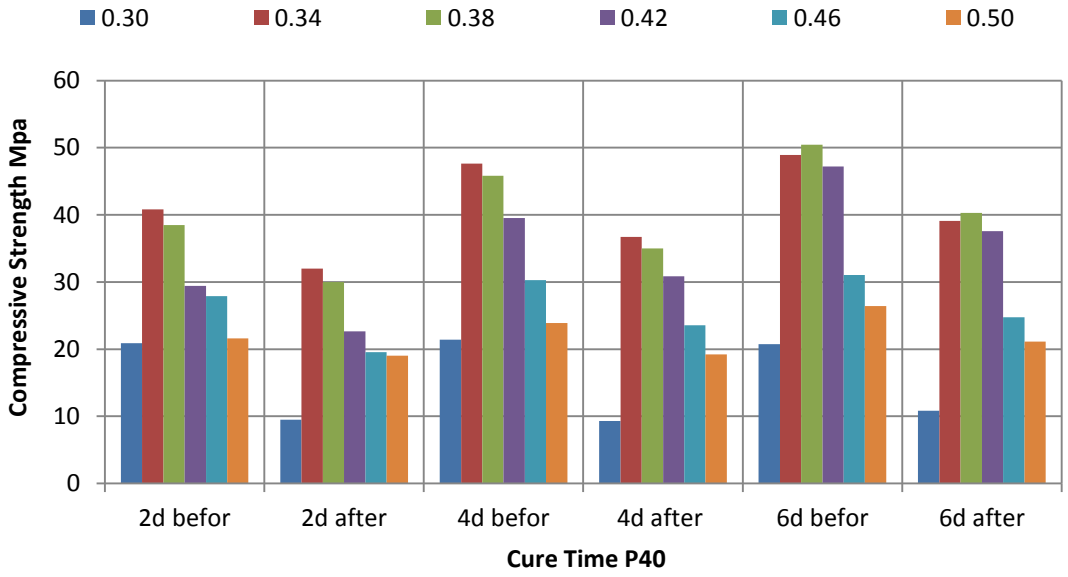


Figure 4.27. Influence of cure time on the compressive strength after 5 cycles of freezing-thawing for P40%+FA60%.

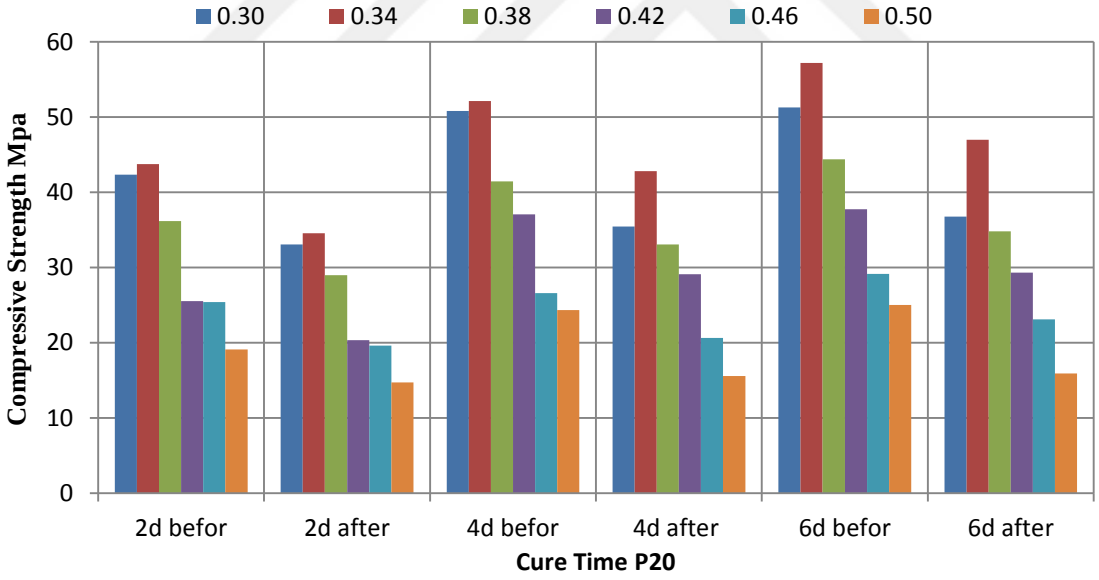


Figure 4.28. Influence of cure time on the compressive strength after 5 cycles of freezing-thawing for P20%+FA80%.

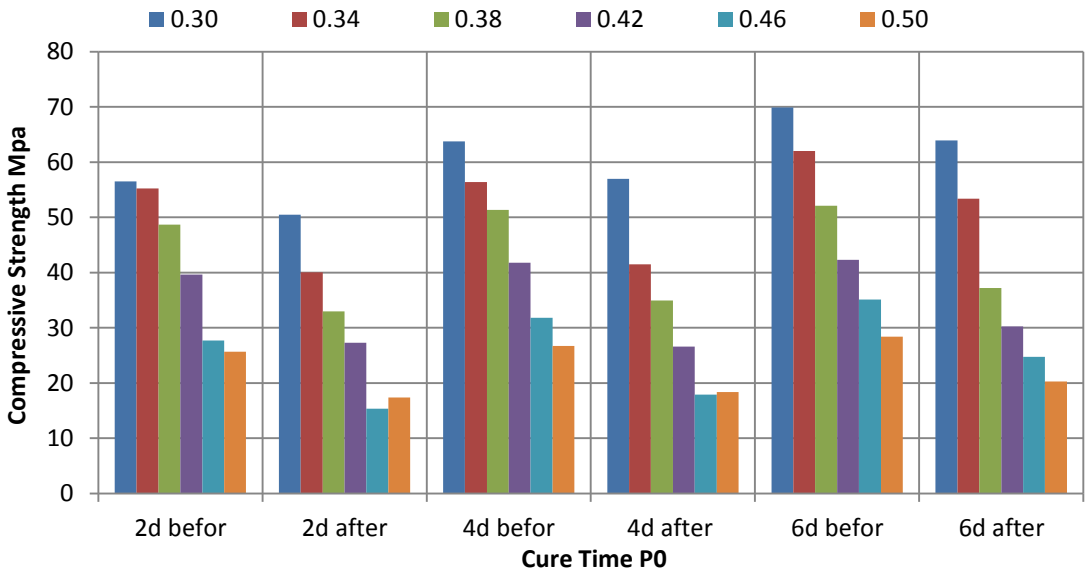


Figure 4.29. Influence of cure time on the compressive strength after 5 cycles of freezing-thawing for P0%+FA100%.

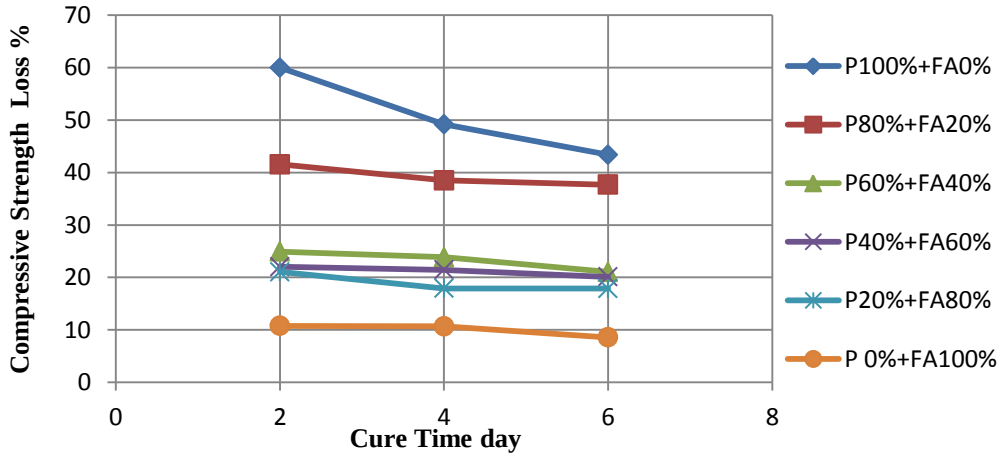


Figure 4.30. Compressive strength loss (%) after freezing-thawing with cur time for optimum values.

4.7.2. Influence of A/B ratio on freezing-thawing resistance

The alkaline solution to binder ratio (A/B) significantly influences the freezing-thawing properties of geopolymer pastes. The use of optimum A/B ratio in geopolymer mixtures leads to the better dissolution of pumice powder and/or fly ash as well as tends to improve the geopolymerization reaction, which results in optimum compressive

strengths. Figure 4.31 reveals the A/B ratio effect on compressive strength of P-FA-based geopolymer after five cycles of freezing-thawing after 28 days with 6-day curing time. The results indicate that the compressive strength after freezing-thawing increases by increasing the A/B ratio up to a certain value with behavior similar to before freezing-thawing. The compressive strengths after freezing-thawing test increase as the A/B ratio increase from 0.30 to (0.46, 0.46, 0.42, 0.38, and 0.34) for mixtures contain (P100%+FA0%, P80%+FA20, P60%+FA40, P40%+FA60, and P20%+FA80%) respectively, contrariwise in the mixture with FA100% the compressive strength decreases as the A/B ratio increase from 0.30 to 0.50.Figure 4.32 shows the amount of the compressive strength loss percentage after 5 cycle freezing-thawing with A/B ratio at 6 days curing time. Generally, the compressive strength loss% decreases as the A/B ratio is increased to a certain value (optimum ratio for each type mixture) that is necessary to complete geopolymerization process. However, the strength loss was (43.39%, 37.68%, 21.06%, 20.09%, 17.88 %, and 8.54%) in (P100-R0.46, P80-R0.46, P60-R0.42, P40-R0.38, P20-R0.34, and P0-R0.30) respectively at the ages 28 days cure for 6 days for the optimum compressive strength.

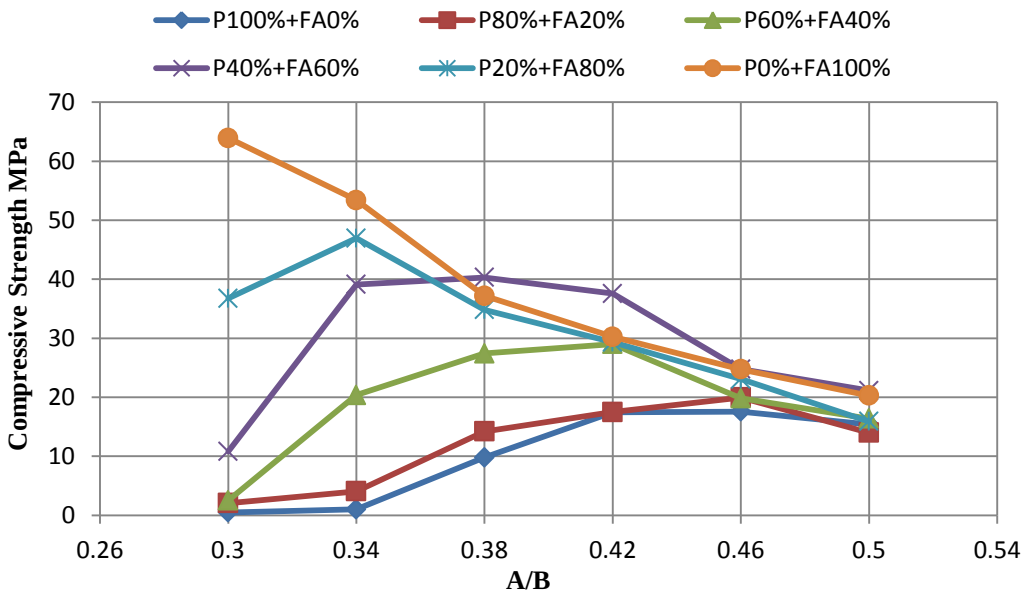


Figure 4.31. Compressive strength after 5 cycles of freezing-thawing for different A/B ratio at 6-day curing, tested at 28 days.

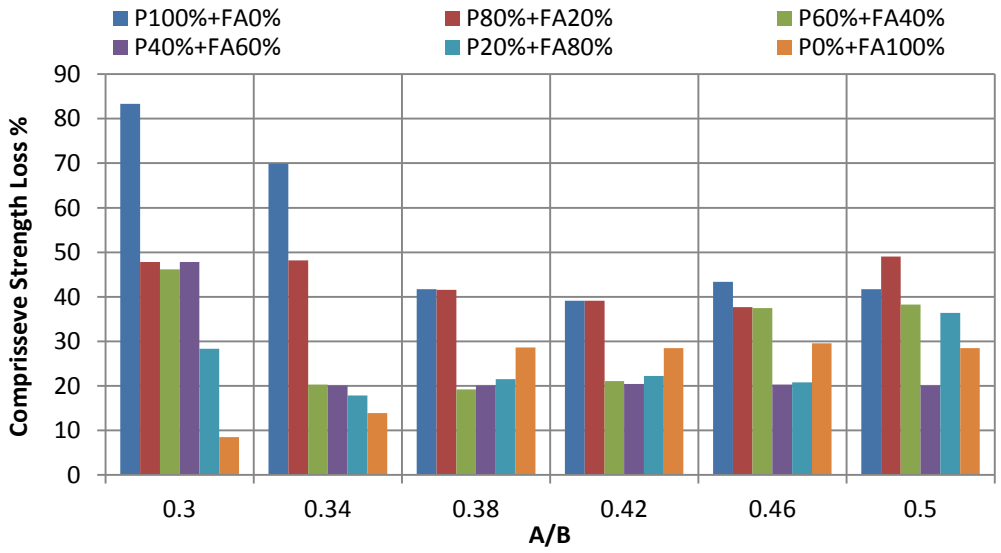


Figure 4.32. Compressive strength loss% after freezing-thawing with A/B ratio.

4.7.3. Influence of the pumice powder and FA on freezing-thawing resistance

Figure 4.33 explains the influence of FA% content on compressive strength (for optimum values) before and after freezing-thawing. The highest percentage for compressive strength loss after 5 freezing-thawing cycles was with P100% based geopolymer paste reached to 43.39 %. While the less percentage for compressive strength loss was with FA100% based geopolymer paste 8.54%, the increase of fly ash contain in mixtures lead to a decrease in the percentage for compressive strength loss after freezing-thawing.

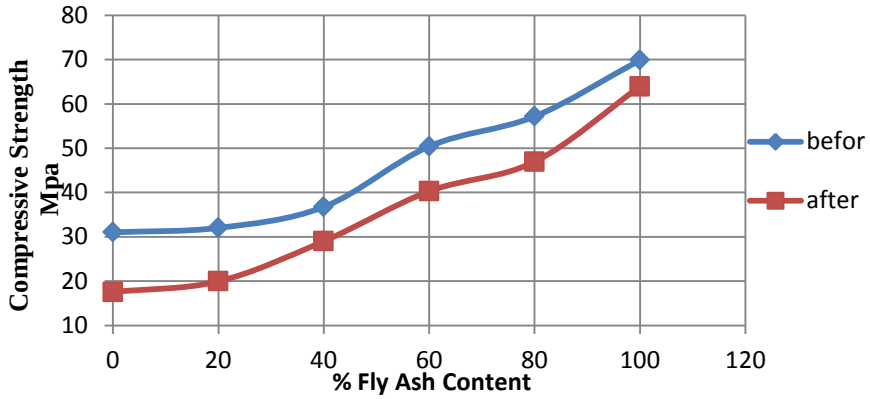


Figure 4.33.The influence of FA% content on compressive strength before and after freezing-thawing for optimum values.

4.8. Elevated Temperature Resistance

The influence of elevated temperature on the compressive strength of and visual appearance of geopolymer paste specimens was studied at temperatures of 200, 500, and 800°C. To do this test, from 6 mixture types the samples with the highest three optimum results in compressive strength have chosen (cured at 6o°C in the oven for 6 days).

Following high-temperature exposure, Figure 4.34 shows colors change and the shape of geopolymer specimens. The surface of the samples warmed up to 200 °C was not visible effect. At a temperature of 500°C the samples P100%+FA0%, P80%+FA20, and P60%+FA40 changed their color from grey to a light brown and enlarged their size, while the rest did not occur a clear effect in size except for a slight lightening in color. At 800°C, the samples P100%+FA0%, P80%+FA20, and P60%+FA40 increased in size relatively significantly and changed color to white and light green, the sample with P40%+FA60 is slightly larger with a slight color change. For the samples P20%+FA80% and P0%+FA100%, the size did not change, as for the color, it becomes light brown.

Table 4.7 shows all geopolymer paste specimen compressive strength after high temperatures exposed. In general, compressive strength decreases as elevated temperature increases to 200, 500, and 800°C at 28 days.

Figure 4.35 shows compressive strength loss % after elevated temperature. It is noticeable that in the mixtures with a high content of the pumice, a large loss of compressive strength occurs, so the result for P100%-R0.46 was (57.83%, 81.30%, and 86.85) at (300, 500, and 800°C) respectively. When the content of the FA increases in the geopolymer sample the loss% of compressive strength will decrease after elevated temperatures which were (0.11, 18.15, and 32.83) at (300,500, and 800°C) respectively. This indicates that FA is more tolerant of high temperatures from pumice. Figure 4.36.shows the influence of FA% content on compressive strength for different elevated temperatures, in 200°C there is a small drop in strength for high FA content mixes. The maximum compressive strength for geopolymer paste specimens that were obtained was (69.82,57.21, and 46.95 MPa) at (300,500 and 800 °C) respectively.

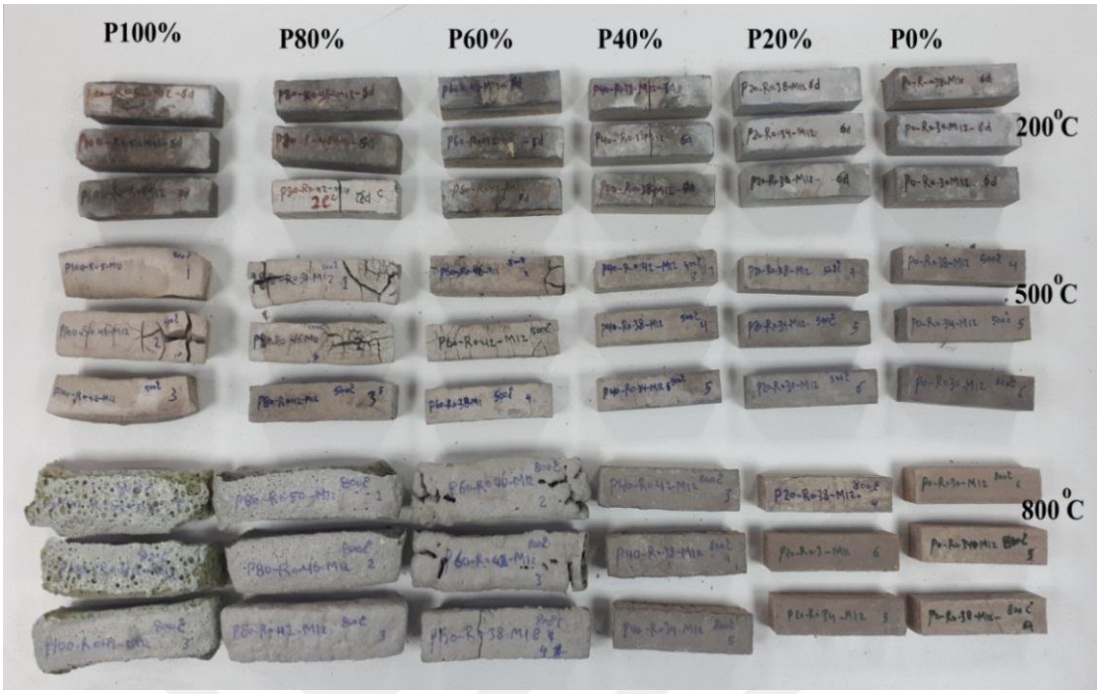


Figure 4.34. Geopolymer samples at 200°C, 500°C, and 800°C.

Table 4.7. Compressive strength (MPa) of geopolymer samples at 200°C, 500°C, and 800°C.

Sample Code	A/B	Compressive Strength MPa			
		60°C	200°C	500°C	800°C
P100%-R0.42	0.42	28.69	7.59	4.92	3.62
P100%-R0.46	0.46	31.02	13.08	5.80	4.08
P100%-R0.50	0.50	26.43	7.56	4.94	4.20
P80%-R0.42	0.42	28.59	15.12	7.40	4.19
P80%-R0.46	0.46	32.03	24.68	7.98	4.85
P80%-R0.50	0.50	27.46	13.69	7.38	4.55
P60%-R0.38	0.38	32.95	30.01	9.15	5.12
P60%-R0.42	0.42	34.76	32.45	9.85	4.99
P60%-R0.46	0.46	30.70	27.97	8.41	4.45
P40%-R0.34	0.34	48.92	46.52	24.95	17.46
P40%-R0.38	0.38	51.43	49.11	25.75	17.99
P40%-R0.42	0.42	47.21	44.65	21.95	16.35
P20%-R0.30	0.30	51.28	49.75	32.47	28.37
P20%-R0.34	0.34	57.17	55.82	39.76	34.91
P20%-R0.38	0.38	44.35	42.53	26.88	26.50
P 0%-R0.30	0.30	69.90	69.82	57.21	46.95
P 0%-R0.34	0.34	62.01	61.90	50.60	41.17
P 0%-R0.38	0.38	52.10	46.17	40.52	33.17

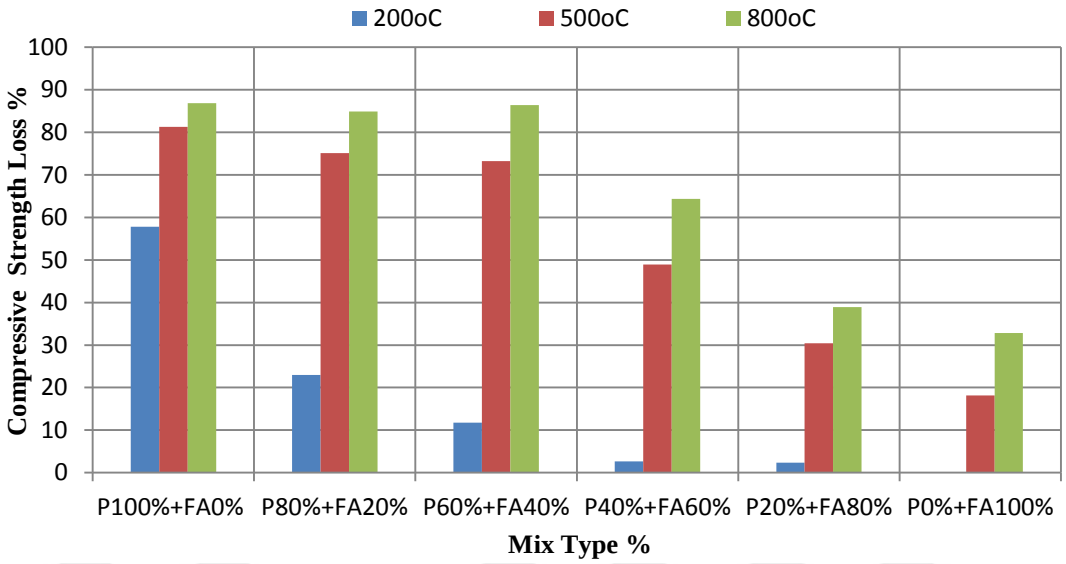


Figure 4.35. Compressive strength loss% after elevated temperature.

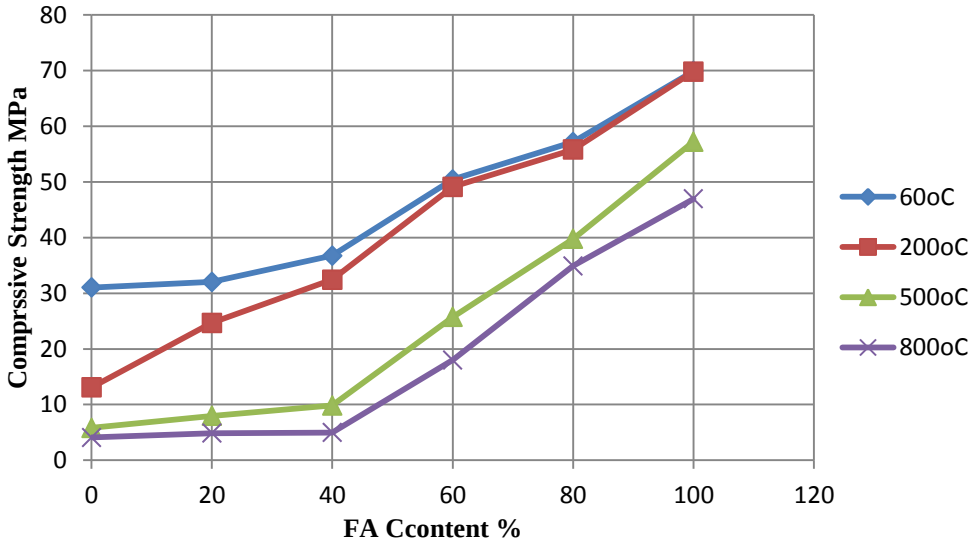


Figure 4.36. Influence of FA% content compressive strength for different elevated temperatures.

Figure 4.37 to 4.39 Show the relationship of A/B to compressive strength at elevated temperatures, it is noticed that there is increasing in the compressive strength with reaching to certain A/B ratio(which represent the optimum ratio), with similar behavior to compressive strength results at 60°C. The best A/B ratio was obtained(0.46, 0.46, 0.42, 0.38, 0.34, and 0.30) for mixtures (P100%+FA0%, P80%+FA20,

P60%+FA40, P40%+FA60, P20%+FA80%, and P0%+FA100%) respectively, which achieved the highest compressive strength after elevated temperature exposure.

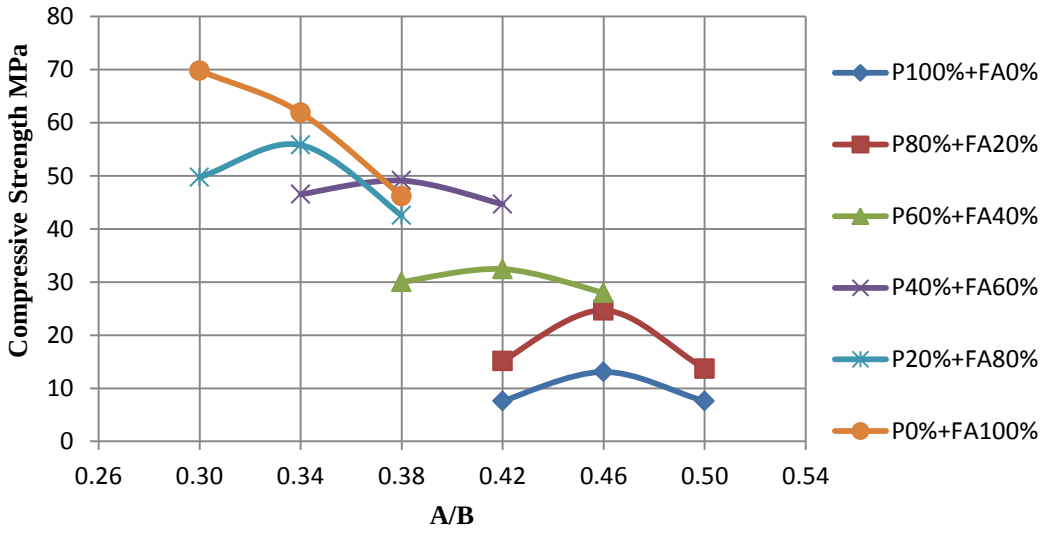


Figure 4.37. Compressive strength after 200°C for different A/B ratios.

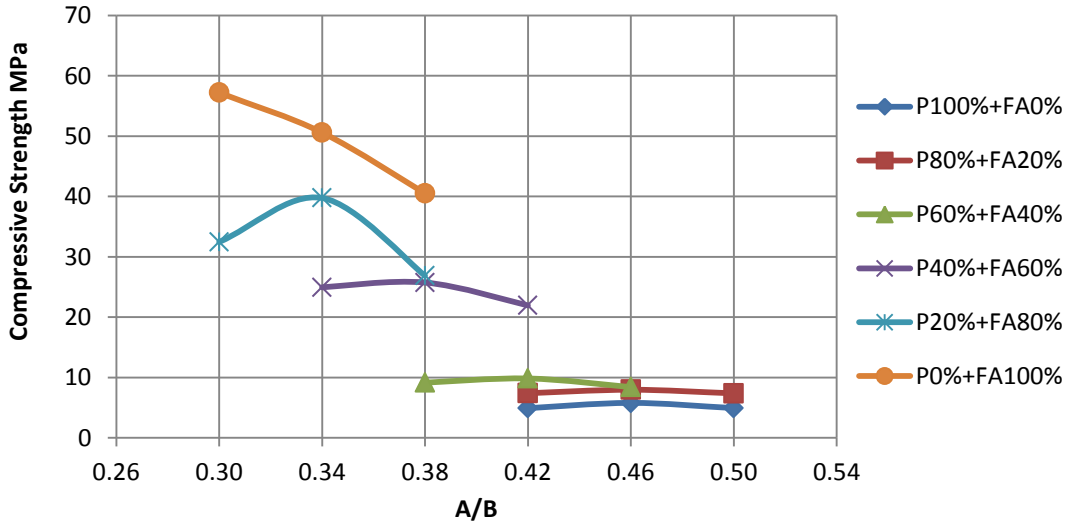


Figure 4.38. Compressive strength after 500°C for different A/B ratios.

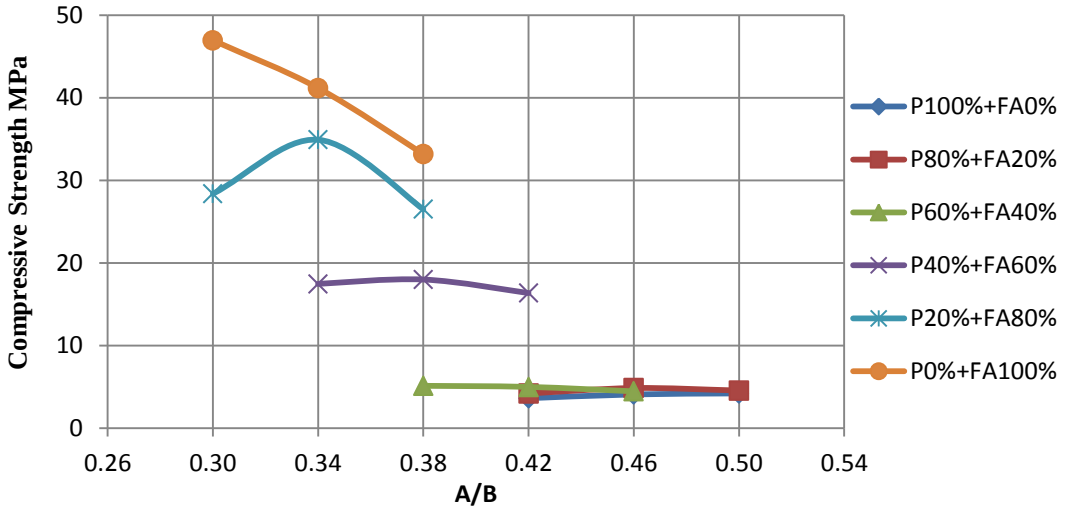


Figure 4.39. Compressive strength after 800°C for different A/B ratios.

4.9. Ultrasonic Pulse Velocity (UPV)

In this investigation, ultrasonic pulse velocity (UPV) testing of the geopolymeric samples was done to examine the effects on the ultrasound waves of the samples formed with different conditions and different proportions of the source material. All 108 type from specimens were tested which represent the pumice-fly ash-based geopolymer made with (6 type mixture with changed content of P%+FA%, 6 types of A/B ratio that vary from 0.30 to 0.50 and 3 types of curing time at 60°C), all results of the UPV are listed in Table 4.7

Generally, The results reveal the fact that the UPV value increase as the cure time increase from 2 to 6 days, this is due to the polymerization process which didn't complete in 2 or 4 days. Similar behaviors were observed at the results of UPV which giving a high speed to wave transfer at the same sample that carries out a high strength. When the density decreases, the UPV value decreases, which may be due to the presence of voids that are formed when there were high workability and water content is high, or the UPV value decreases because there is not a sufficient A/B ratio to complete the polymerization and the reactions. The results as well reveal that adding fly ash had no effect on the wave flow of the specimens were neither positive nor negative.

To estimate the equation relating compressive strength with UPV the trend line was drawn as shown in Figure 4.40. The equation of ($fc=0.0397\ UPV^{5.6535}$**)** can be used to demonstrate the correlation between compressive strength and UPV for all samples. The coefficient of determination ($R^2=0.7614$) (as a value between 0.0 and 1.0 for the *goodness of fit*) reveals decent relevance between two variables, this means approximately 76.14 % of the compressive strength can be predicted by equation and the ultrasonic pulse velocity data points.

Figure 4.41 shows the relationship between UPV and density. The correlation equation between density and UPV was ($fc=533.01UPV–60.78$). The coefficient of determination ($R^2=0.8139$) indicates a good relationship between density and UPV and approximately 81.39% of density can be predicted by correlation equation.

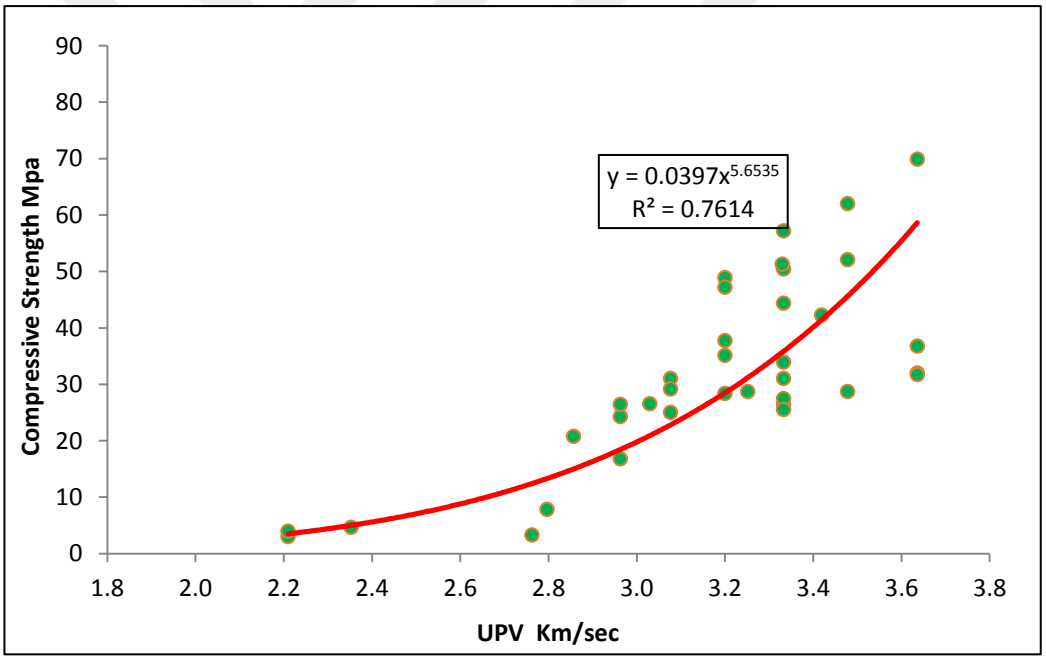


Figure 4.40. The correlation between compressive strength and UPV values.

Table 4.8. The results of Ultrasonic Pulse Velocity (UPV).

Sample Code	Ultrasonic Pulse Velocity m/sec		
	2d	4d	6d
P100-R0.30	1806	1923	2210
P100-R0.34	2100	2597	2763
P100-R0.38	2597	2963	2963
P100-R0.42	2985	3008	3252
P100-R0.46	2778	3077	3333
P100-R0.50	2759	3077	3333
P80-R0.30	1843	1923	2210
P80-R0.34	2649	2797	2797
P80-R0.38	2667	2920	2963
P80-R0.42	2985	3008	3478
P80-R0.46	3077	3333	3636
P80-R0.50	2837	3150	3333
P60-R0.30	2139	2162	2353
P60-R0.34	2667	3030	3333
P60-R0.38	2581	3279	3333
P60-R0.42	2667	3333	3636
P60-R0.34	2581	3250	3636
P60-R0.30	2424	2985	3030
P40-R0.30	2500	2667	2857
P40-R0.34	2500	2759	3200
P40-R0.38	2500	2857	3333
P40-R0.42	2353	2581	3200
P40-R0.46	2353	2548	3077
P40-R0.50	2353	2548	2963
P20-R0.30	2817	3077	3330
P20-R0.34	2817	3077	3333
P20-R0.38	2759	3077	3333
P20-R0.42	2581	2963	3200
P20-R0.46	2581	2963	3077
P20-R0.50	2424	2778	3077
P0-R0.30	3200	3478	3636
P0-R0.34	3101	3478	3478
P0-R0.38	3077	3400	3478
P0-R0.42	2759	3333	3419
P0-R0.46	2667	3200	3200
P0-R0.50	2500	3030	3200

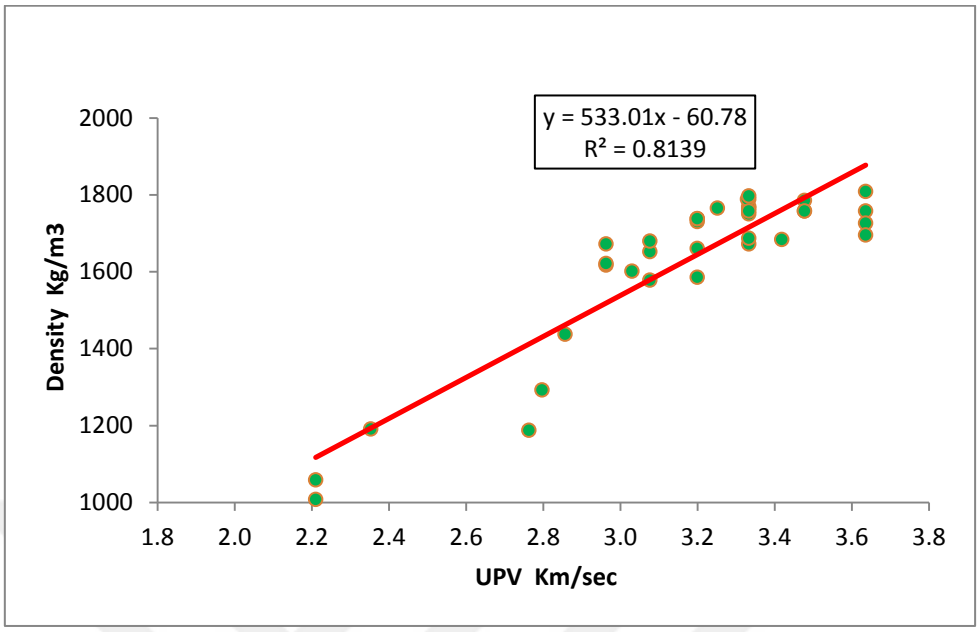


Figure 4.41. The correlation between density and UPV values.

5. CONCLUSIONS

5.1. Conclusions

Through the results obtained and the graphs that have been made, the following conclusions can be confirmed about the P-FA geopolymer paste.

There is significant relationship between consistency penetration (mm) by Vicat apparatus and strength, the results indicate that the best strength achieved with (11, 17, 18, 19, 28, and 30 mm) for P100%+FA0%, P80%+FA20, P60%+FA40, P40%+FA60, P20%+FA80% and P0%+FA100% respectively.

At 7 days, the compressive strength gained about 90% of that at the age of 28 days. This means that geopolymer binders will gain most of the strength early, which is important when considering the speed of the construction process. The maximum compressive strength of the 100% Pumice was (28.64 MPa), this value increased by increasing FA contain. When the mixture contained 100% FA achieved the highest value of 64.46MPa at 7 days.

The test results indicated that A/B ratios had an important influence on the geopolymer strengths. The strength of P-FA specimens increased by increasing the A/B to a certain value (representing the optimum-active A/B ratio for each type of mixture), the compressive strength decreases more or less than this value. The best A/B ratio was obtained (0.46, 0.46, 0.42, 0.38, 0.34, and 0.30) for mixtures (P100%+FA0%, P80%+FA20, P60%+FA40, P40%+FA60, P20%+FA80%, and P0%+FA100%) respectively, which attained the highest compressive strength.

The strength(compressive and flexural) of P-FA geopolymer past increased by the increase of the curing time from 2 to 6 days. The highest strength can be achieved with 6 days of curing time at 60°C. The speed of gaining strength gradually increases by increase fly ash content, so for optimum values at 6 days, it was achieved 80.86% after 2days and 91.23% after 4 days while pumice mixture was gained 59.22 % and 84.20 % at 2 and 4 days, respectively.

The result of this study confirms that it is possible to utilize pumice with FA to create good geopolymer binders. As the FA percentage of replacement increased in mixtures, the strength increased. The 20% ratio of FA leads to an increase in compressive strength by 3.26 %, 40% ratio of FA increased by 18.50 %, 60% ratio of FA the increased by 84.30 %, and 80% ratio of FA the compressive strength increased by 84.3% for mixture with optimum values.

The optimum compressive strength values that have obtained were (31.02, 32.03, 36.76, 50.43, 57.17, and 69.90) for mixtures (P100-R0.46, P80-R0.46, P60-R0.42, P40-R0.38, P20-R0.34, and P0-R0.30) respectively, tested at 28 days (cured for 6 days at 60°C).

The optimum flexural strengths were (9.40, 8.70, 6.14, 6.38, 6.75, and 8.86) for mixtures (P100-R0.46, P80-R0.46, P60-R0.42, P40-R0.38, P20-R0.34, and P0-R0.30) respectively, were tested at the ages 28 days.

The densities for specimens that achieve the maximum compressive strengths were (1770, 1758, 1727, 1750, 1797, and 1809 Kg/m3) for (P100-R0.46, P80-R0.46, P60-R0.42, P40-R0.38, P20-R0.34, and P0-R0.30) respectively. At all studied samples (which have an optimum A/B ratio), the water absorption values were less than 10 %. This means that the pumice-fly ash geopolymer binder has high quality for water absorption

The compressive strength of all specimens after 5 cycles of freezing-thawing cycles was less than the compressive strength of samples for which not exposed to any freezing-thawing resistance. The result indicates that increase the fly ash content in geopolymer paste has a significant effect on freezing-thawing resistance. The results were revealed that the strength drop amount in the 6 days curing time less than that at 2 and 4 days.

The compressive strength of tested samples after freezing-thawing increases by increasing the A/B ratio up to a certain value with behavior similar to before freezing-thawing. The loss percentage of compressive strength for mixtures with the optimum compressive strength was (43.39%, 37.68%, 21.06%, 20.09%, 21.37%, and 8.54%) in (P100-R0.46, P80-R0.46, P60-R0.42, P40-R0.38, P20-R0.34, and P0-R0.30) respectively at the ages 28 days cure for 6 days.

In general, the compressive strength decreases with increasing of elevated temperature at 200, 500, and 800°C at 28 days cured for 6 days in the oven with temperature 60°C. The strength increases with reaching to certain A/B ratio, which represents the optimum ratio, with similar behavior to compressive strength results at 60°C. The mixtures with a high content of the pumice, a large loss of compressive strength occurs. They were (57.83%, 81.30%, and 86.85%) at (300, 500 and 800°C) respectively. When the content of the FA increase in geopolymeric paste the loss of compressive strength will decrease after elevated temperatures which were (0.11%, 18.15%, and 32.83%) at (300, 500, and 800°C) respectively. Fly ash is more tolerant of high temperatures from pumice. At 200 °C, there is a small drop in strength for high FA content mixes. The maximum compressive strength for geopolymer specimens that were obtained was (69.82, 57.21, and 46.95 MPa) at (300, 500, and 900°C) respectively for the mixture of P0-R0.30.

The results reveal the fact that the UPV value increase as the cure time increase from 2 to 6 day. There was a relationship of the UPV with density and compressive strength. The equation of ($fc=0.0397UPV^{5.6535}$) with the coefficient of determination ($R^2=0.7614$) was found to relate the compressive strength and UPV so the correlation equation between density and UPV was ($fc=533.01UPV–60.78$) with a coefficient of determination ($R^2=0.8139$).

5.2. Recommendations

The findings of this research have shown its achievements clearly, but several future investigations are recommended as follows:

1. Investigation using aggregate to produce pumice-fly ash-based geopolymers concrete and study the different parameters affecting the strength and durability characteristics.

2. Conduction a more detailed investigation into the effect of fineness, activator concentration, and A/B ratio on fresh geopolymer properties such as workability, setting, and consistency.

3. Investigation the durability of the produced pumice-fly ash-based geopolymers by exposing them to seawater, chloride, and sulfate solution.

4. Investigation the possibility of using the produced pumice-fly ash-based geopolymers in structural applications by studying the behavior of steel reinforced members.

5. Investigation the impact of adding more different types of source material like lime, slag, and nano-silica, to improve the durability and strength of geopolymer products.

6. Experimentally investigation the water permeability, shrinkage, and creep of pumice-fly ash-based geopolymers.

7. Investigation the possibility to produce bricks, blocks, and pre-cast elements by this type of geopolymer and made a comparison with that produce from Portland cement.

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EXTENDED TURKISH SUMMARY (GENİŞLETİLMİŞ TÜRKÇE ÖZET)

1. ÖZ

Portland Çimentosunun üretiminin çevre kirliliğine ve yüksek enerji tüketimine neden olması, birçok araştırmacıyı, çimento yerine yeni bir bağlayıcı bulmaya yönlendirmiştir. Silika ve Aluminanın, alkali aktivatör ile kürlenmesi sonucu üretilen Geopolimer bağlayıcılar Çimento yerine kullanılabilecek bir malzeme olarak düşünülmektedir. Bu çalışmada Geopolimer bağlayıcı yüksek oranda silika ve alümina içeren pomza tozu ve uçucu kül, ile üretilmiştir. Bu amaçla 6 farklı yüzde oranında pomza ve uçucu kül karışımı, 6 farklı alkali/bağlayıcı oranında denenmiştir. Alkali aktivatör olarak NaOH çözeltisi, 2,4 ve 6 gün boyunca 60 ° C'de kürleme ile 12M sabit konsantrasyonda kullanılmıştır. Kıvam, basınç dayanımı, eğilme dayanımı ve tahribatsız ulrases hız geçişi testleri ve yüksek sıcaklık ve donma çözülme dirençleri farklı karışımlar için belirlenmiştir. Test sonuçlarına göre pomza tozu ve uçucu kül karışımları içeren geopolimer bağlayıcılar, optimum (31.02, 32.03, 36.76, 50.43, 57.17 ve 69.90 MPa) basınç dayanımlarında sırasıyla (P100-R0.46, P80-R0.46, P60-R0.42, P40-R0.38, P20-R0.34 ve P0-R0.30) karışımları için elde edilmiştir. Uçucu kül oranının artmasıyla geopolimer örneklerinin farklı özelliklerinin iyileştiği görülmüş, en iyi kürleme süresi 6 gün olarak belirlenmiştir. Test sonuçları alkali/bağlayıcı oranı ve kıvamın geopolimer numunelerin özellikleri üzerinde önemli derecede etkili olduğunu göstermiştir

2. GİRİŞ

Dünya nüfusunun artması ile altyapı ve sanayileşmenin artması nedeniyle çimentoya yüksek oranda ihtiyaç duyulmaktadır. Bu arada sürdürülebilirlik ve çevre konuları da dikkate alınmalıdır (Turner ve Collins, 2013). Portland çimentosunun en büyük sorunu, üretiminin küresel CO_2 emisyonuna %5-7 oranında katkıda bulunmasıdır (Benhalal vd., 2013; Romanoa vd., 2013; Singh ve Middendorf, 2020). Diğer sorun ise enerji tüketimi, yüksek miktarda hammadde tüketimi vb.'dir. Bu nedenle portland çimentosu için alternatif malzeme bağlayıcıları aranmalıdır. Geopolimer, bina ve inşaat

uygulamalarında kullanım için gelecekteki alternatif olabilir. Geopolimer çimento, sıradan portland çimentosuyla karşılaştırıldığında karbondioksit kirliliğinin azaltılması, daha düşük üretim maliyeti, atık madde kullanma imkanı ve hammadde tüketiminin azaltılması gibi olumlu yanlara sahip olabilir (Habert vd., 2011; Davidovits, 2013; Duxson ve diğerleri, 2007; Lloyd ve Rangan, 2010; Zakka ve diğerleri, 2021).

Geopolimer çimento oluşturmak için bir alümina silikat kaynağı ve alkali aktivatör gereklidir (Davidovits, 1991; Van-Deventer ve diğerleri, 2007). Alümina silikat kaynağı olarak hammaddeler, endüstriyel atık yan ürün malzemeleri ve doğal hammaddeler olabilir. Endüstriyel atık malzemeler GGBFS cürufu, uçucu kül ve benzeridir. Doğal hammaddeler süngertaşı, kaolinit, kalsine edilmiş killer ve benzeridir (Farhan ve diğerleri, 2020; Ren ve diğerleri, 2021). Doğal çevrenin kirlilikten korunması için, Uçucu Kül gibi santrallerin ürettiği atıklara bağlı yeni yapı malzemelerinin geliştirilmesine ihtiyaç vardır (Merinkline at el., 2013; Dwivedi ve Jain, 2014; Abdul-Aleem ve Arumairaj, 2012).

Önceki çalışmalara göre taze ve sertleşmiş özelliklere etki eden birkaç faktör vardır. Bunlar; geopolimer malzemelerin kimyasal bileşimi, hammaddelerin inceliği, alkali-bağlayıcı oranı, alkali sıvıların kürleşme süresi ve sıcaklığı vb. dir. (İbrahim ve Maslehuddin, 2021).

Bu çalışmada geopolimer çimento hamuru üretiminde doğal bir malzeme olarak pomza ve endüstriyel bir yan ürün olarak uçucu kül kullanılmıştır.

3. ÖNCEKİ ÇALIŞMALAR

Portland çimentosu, çeşitli uygulamalar için kullanılan en iyi ve en önemli malzeme bağlayıcı olarak kabul edilir. Çimento üretimi büyük enerji gerektirir ve çok fazla hammadde tüketir, ayrıca küresel olarak CO_2 emisyonlarına en büyük katkı sağlayan etmenlerden biridir (Okonkwo vd., 2019; Signh ve Middendorf, 2020). Küresel çimento talebinin artması ile artan üretimin çevre üzerinde yaratacağı olumsuz etkiler tahmin edilebilir. Bu nedenle, daha düşük çevresel etkilere ve daha iyi ekonomik faydalara sahip çimento ürünleri hakkında daha fazla araştırma yapılması gerekmektedir. Geopolimer çimento, dünyanın dört bir yanındaki araştırmacılardan büyük ilgi gören yeni bir sürdürülebilir yapı malzemesi olarak kabul edilmektedir.

Geopolimerler hakkında birçok araştırma çalışması yayınlanmıştır, çünkü geopolimer çimento bağlayıcıları, kirliliği ve sera gazını azaltmadaki yararlılığına ek olarak benzer inşaat mühendisliği uygulamalarında karşılaştırılabilir performans sağladığından Portland çimentosunun yerine kullanılabilir.

Geopolimer terimi genellikle amorf alkali alümina silikatı belirtmek için kullanılır. Aslında geopolimer üzerinde yapılan geliştirme çalışmaları sırasında; inorganik polimer, alkali ile aktive edilmiş çimento, alkali bağlı seramik, jeo çimento, toprak çimentosu, hidro seramik vb. olarak çeşitli terimler kullanılmıştır. Bu terimlerin tümü, sialat monomeri olarak benzer ilk bileşenler kullanılarak üretilen sentezlenmiş malzemeyi (bağlayıcı) tanımlamaktadır. (–Si – O – Al – O–) (Duxson ve diğerleri, 2007; Davidovits, 2013; Singh, 2015). Geopolimerle sonuçlanan reaksiyonlar geopolimerizasyon olarak adlandırılır (Davidovits, 1994; Davidovits, 2005).

Geopolimer teknolojisi yeni olarak kabul edilirken, teknoloji eski köklere sahiptir ve Giza'nın piramitlerinin yanı sıra diğer eski yapılar için yapı malzemesi olarak kullanılmıştır (Barsoum ve Ganguly 2006).

1978'de Joseph Davidovits (bir Fransız profesör), inorganik moleküllerin zincirleri veya ağları ile karakterize edilen çok çeşitli malzemeleri tanımlamak için Jeopolymer adını ilk kullanan kişiydi (Davidovits, 1991; Davidovits, 2005). Alkalin aktivatör çözeltisinin kullanılması, hidroksilasyon ve poli-yoğunlaşma reaksiyonları ile sonuçlanan üç boyutlu (3D) polisialat zincirleri (uçucu kül ve cürufa benzer) oluşturmaya yol açtı (Van Chanh ve diğerleri, 2008; Davidovits, 1991).

1979 ve 1995 yılları arasında Davidovits ve ekibi, geopolimerizasyon üzerine çok sayıda makale yayınladı ve patent aldı. Bu patentler, diğer şeylerin yanı sıra, bir geopolimerik bağlayıcının geliştirilmesini ve bir siliko-alüminat mineral polimerinin geliştirilmesini içermektedir (Komnitsas ve Zaharaki 2007).

Geopolimerik malzemeler üzerine uygulamalı araştırmalar hızlandırılmış ve 1990 sonlarında makaleler yayınlanmaya başlanmıştır (Provis vd., 2015). Dünya çapındaki araştırmacılar, Portland çimentosu koşullarında olduğu gibi kullanılabilen kullanıcı dostu geopolimer çimentoların üretimine odaklanmalıdır (Davidovits, 2008). Bununla birlikte, geopolimer çimentonun kullanımını kısıtlayan bazı dezavantajlar

vardır. Bunlar; standart eksikliği, kullanım riski, maliyetler ve kullanılabilirliktir (Okonkwo ve diğerleri 2019).

4. MATERYAL VE YÖNTEM

4.1. Materyal

4.1.1 Pomza Tozu

Deneylerde kullanılan süngertaşı Türkiye'nin doğusundaki Erciş ili yakınlarındaki Kocapınar bölgesinden getirilmiştir. Süngertaşı agregaları laboratuvarda bilyalı değirmen kullanılarak 4 saat öğütülerek toz ponza elde edilmiştir.

4.1.2. Uçucu kül

Araştırmada kullanılan uçucu kül, Adana ili Yumurtalık ilçesi Sugözü mevkiinde bulunan Sugözü Termik Santrali'nden temin edilmiştir.

4.1.3. Alkali Aktivatör Çözeltisi

Bu çalışmada alkali aktivatör çözeltisi hazırlamak için ticari olarak temin edilebilen % 98 saflıkta sodyum hidroksit peletleri kullanılmıştır. Koray Kimya, İstanbul, Türkiye'den alındı.

NaOH çözeltisi, katı peletlerin bir miktar damıtılmış su içinde çözülmesiyle elde edilmiştir. Bu çalışmada 12 Molar konsantrasyonlu alkali aktivatör çözeltisi kullanılmıştır.

4.1.4. Saf Su

Alkali aktivatör solüsyonunun hazırlanması için NaOH peletlerini çözmek için damıtılmış saf su kullanıldı. Laboratuvarda damıtılmış su cihazı kullanılarak musluk suyundan elde edilmiştir.

4.2. Yöntem

4.2.1. Karıştırma, döküm ve kürleme

% 0 ile % 100 arasında farklı süngertaşı ve uçucu kül yüzdesine sahip altı karışım hazırlanmış, altı alkali-bağlayıcı oranı (A / B) 0.30, 0.34, 0.38, 0.42, 0.46 ve 0.50 kullanmıştır. Tüm deneylerde, alkali aktivatör çözeltisi olarak 12 M NaOH ve 2, 4 ve 6 gün boyunca 60 ° C'de kürleme süreleri sabit tutuldu. Alkali çözelti hazırlandıktan

sonra karıştırma işleminde kullanılmadan önce bir gün bekletildi. Her tip karışım için kuru toz (% P +% FA) elle ve karışım homojen hale gelene kadar dikkatlice karıştırıldı. Pastaların karıştırılması, BS EN 196-1'e göre elektrikli bir karıştırıcı makine kullanılarak mekanik olarak yapılmıştır. İlk önce çözelti karıştırma kabına döküldü ve ardından bağlayıcı tozu ilave edildi. Daha sonra karıştırıcıda düşük hızda 3 dakika karıştırıldı. Karıştırıldıktan sonra, macun, kolay kalıplama sağlamak için iyice yağlanan kalıplara (4cm × 4cm × 16cm^3 boyutlarında) doğrudan döküldü. Döküm ayrıca BS EN196-1'e uygun olarak iki katmanla yayılarak yapıldı ve kalıplar sarsma masasına sıkıştırıldıktan sonra ilk katman 60 sarsıntıyla sıkıştırıldı, ardından ikinci katman tekrar 60 sarsıntıyla sıkıştırıldı. Her karışım yapılırken, Vicat makinesi ile o karışımın kıvamını test etmek için karışımdan küçük bir parça alındı. Her bir döküm tabakası bittikten sonra numuneler 24 saatlik dinlenme süresi boyunca oda sıcaklığında tutuldu ve geopolimer macun numunelerinin sıvı solüsyonlarının buharlaşmasını en aza indirmek için naylon vakumlu film ile kaplandı. Dinlenme süresi sona erdiğinde numuneler 2, 4 ve 6 günlük farklı kür süreleri için 60°C kürleme sıcaklığında kuru fırına yerleştirildi. Kürlenme süresi sona erdikten sonra numuneler fırından alındı ve test zamanına kadar laboratuvarda ortam sıcaklığında bırakıldı.

5. BULGULAR VE TARTIŞMA

5.1. Kıvam Testi Sonuçları

VICAT cihazında yapılan penetrasyon testlerinin sonuçları, A/B oranının belirli bir değere yükseltilmesiyle, penetrasyon ve işlenebilirliğin sıradan Portland çimentosundaki su çimento oranına benzer şekilde arttığını göstermektedir. Sonuçlar, 0.50 oranında yüksek A/B oranına sahip pomzanın kaynak malzeme olarak yalnızca 18 mm penetrasyon sağladığını, uçucu kül yüzdesi arttıktan sonra penetrasyon ve işlenebilirliğin arttığını ve öncü %100 uçucu kül olduğunda sadece 0.30 A/B oranında 30 mm penetrasyon olduğunu göstermiştir. Bunun nedeni, uçucu külün çok yüksek incelik ile karakterize edilirken, süngertaşı tozunun daha az incelikte ve yüksek su emme özelliklerine sahip olmasıdır.

5.2. Basınç Dayanımı

Tüm örneklerin 7 günlük ve 28 günlük deney sonuçları kaydedildi. 7 günlük basınç dayanımının 28. gündeki basınç dayanımının yaklaşık% 90'ı olduğu tespit edilmiştir. Bu, 28. güne kadar basınç dayanımında önemli bir artış olmadığı anlamına gelir. Bu, inşaat sürecinin hızı ve kürleme maliyeti dikkate alındığında, bina yapımında önemli bir parametredir.

5.2.1. Alkali-bağlayıcı (A/B) oranının basınç dayanımı üzerindeki etkisi

A / B 0,30'dan 0,46'ya çıktığında, ponza tozu (% P100) bazlı geopolimer içeren karışımda basınç dayanımı artar. Daha sonra A / B oranı 0,50 değerinde ise düşmüştür. Tersine, FA100% (P0% + FA100%), A / B 0,30'dan 0,50'ye yükseldikçe basınç dayanımı azalır.

Her karışım türü için maksimum basınç dayanımına ulaşmak için en uygun A / B oranı farklıdır. Buna göre A / B oranı ile basınç dayanımı arasında önemli bir ilişki olduğu ortaya çıkmıştır.

Pomza ve uçucu küllü geopolimer numunelerin basınç dayanımları A / B oranı ile değişmektedir. A/B oranı aktif-optimum değerden düşük olduğunda, alkali çözelti miktarı nedeniyle basınç dayanımının gelişimi geopolimerizasyon sürecini tamamlamak için yeterli değildir. Alkalin aktivatör çözeltisinin geopolimerizasyon işleminin ihtiyaçlarını atlaması nedeniyle A/B oranı aktif-optimum değeri aştığında da basınç dayanımının azalması meydana gelecektir. Karışımlar için en iyi A / B oranı (0.46, 0.46, 0.42, 0.38, 0.34 ve 0.30) (P100%+FA0%, P80%+FA20, P60%+FA40, P40%+FA60, P20%+FA80% and P0%+FA100%) ve en yüksek basınç dayanımı sırasıyla elde edildi.

5.2.2. Kürlenme süresinin basınç dayanımına etkisi

Kürlenme süresi 2 günden 6 güne uzadıkça numunelerin basınç dayanımı artar. 6 farklı karışım türü için, 6 günlük kürlenme süresinde ve optimum-aktif A / B oranında optimum basınç dayanımı elde edilmiştir.

5.2.3 Pomza ve uçucu kül yüzdelerinin basınç dayanımına etkisi

Süngertaşı tozu% 0 ile% 100 arasında uçucu kül ile harmanlanarak geopolimer macun elde edildi. Macun içeriği% 100 süngertaşı olduğunda, 28 günlük maksimum basınç dayanımı (60°C ve P100-R0.46'da 6 gün kürleme) 31.02 MPa idi. Macun% 100 uçucu kül içerdiğinde, P0-R0.30 karışımında maksimum basınç dayanımı 69.90 MPa

idi. Bağlayıcıdaki ikame uçucu kül yüzdesi artarsa basınç dayanımının artacağı belirlenmiştir.

Örnekler ilk 6 gün 60°C'de kürlenmiş ve (P100-R0.46, P80-R0.46, P60-R0.42, P40-R0.38, P20-R0.34, P0-R0.30) oranlarında hazırlanmıştır ve 28 günlük basınç dayanımları sırasıyla (31.02, 32.03, 36.76, 50.43, 57.17, 69.90 MPa)dır.

5.3. Eğilme Dayanımı

Eğilme dayanımlarını etkileyen faktörler, basınç dayanımlarını etkileyen faktörlerle hemen hemen aynıdır. Maksimum eğilme mukavemeti, 6 farklı karışım türü için optimum-aktif A/B oranında 6. günde elde edilmiştir. Optimum aktif A/B oranındaki herhangi bir azalma veya artış, eğilme mukavemetinde bir düşüşe neden olur. 28 günlük karışımlar (P100-R0.46, P80-R0.46, P60-R0.42, P40-R0.38, P20-R0.34 ve P0-R0.30) için en iyi eğilme dayanımları sırasıyla (8.98, 8.23, 6.02, 6.26, 6.75 ve 8.79) dır.

5.4. Yoğunluk Testi Sonuçları

Genel olarak, kürlenme süresinin 60°C'de 2 günden 6 güne çıkmasıyla tüm numuneler için yoğunluk azalır. Geopolimer macunun hazırlanmasında kullanılan ponza tozu oranının artması ile yoğunluk değerleri bir miktar azalmış, uçucu kül yüzdesi yüksek olduğunda yoğunluk değerleri artmıştır. Maksimum basınç dayanımı için yoğunluk değerleri (P100-R0.46, P80-R0.46, P60-R0.42, P40-R0.38, P20-R0.34 ve P0-R0.30) için (1770, 1758, 1727, 1750, 1797 ve 1809, Kg/m^3) tür.

5.5. Su Emme Oranı

Bu testlerde% 10'un altında su emme değerleri elde edilmiştir (optimum basınç dayanımı ve A/B oranı için). Tüm yüksek kaliteli betonlar% 10'dan daha az su emme değerine sahiptir (Neville, 2005). Pomza ve uçucu külün karıştırılması su emiliminin azalmasına neden olmuştur.

5.6. Donma Çözülme Direnci

4.6.1. Kürlenme süresinin donma-çözülme direncine etkisi

Genel olarak, 5 donma-çözülme döngüsünden sonra basınç dayanımı, 2 günden 6 güne artan sertleşme süresiyle kademeli olarak artmıştır. Geopolimer macunun mukavemet özellikleri, 5 döngü dondurma-çözülmeden sonra azalır. Elde edilen sonuçlara göre 6 günlük kürlenme süresindeki düşüş miktarının 2 gün 4 günden az olduğu ortaya çıktı. Bu, kürlemenin 2, 4 ve 6 günlerdeki azalma yüzdesinin (%) sırasıyla (% 59.99, % 51.07 ve % 43.39) ulaştığı P100% karışımında çok açıktı. Bu oran, kürleşmede sırasıyla 2, 4 ve 6 günde ulaşıldıkça uçucu kül yüzdesi arttıkça azalmaktadır (% 10,56, % 10,51 ve % 8,54).

4.6.2. Alkali çözelti-bağlayıcı oranının donma-çözülme direncine etkisi

Sonuçlar donma-çözülme direncinin, donma-çözülme öncesine benzer davranışla A / B oranını artırırken, donma-çözülme direncinin arttığını göstermektedir. Genel olarak, geopolimerizasyon işlemini tamamlamak için gerekli olan A/B oranı belirli bir değere (her tip karışım için optimum oran) yükseldikçe basınç dayanımı kaybı yüzdesi azalır. Bununla birlikte, güç kaybı 28 günlük örneklerde (P100-R0.46, P80-R0.46, P60-R0.42, P40-R0.38, P20-R0.34 ve P0-R0.30) karışımlarında (% 43.39, % 37.68, % 21.06, % 20.09, % 17.88 ve % 8.54) dür.

4.6.3. Pomza tozu ve uçucu külün donma-çözülme direncine etkisi

5 donma-çözülme döngüsünden sonra en yüksek basınç dayanımı kaybı yüzdesi P100% bazlı geopolimer macunun% 43.39'a ulaşmasıyla oldu. Donma-çözülme sonrası en düşük basınç dayanımı kaybı% 8.54 ile FA100% bazlı geopolimer macunda olmuştur. Karışımlarda bulunan uçucu külün artışı, donma-çözülmeden sonra basınç dayanımı kaybı yüzdesini düşürmektedir.

5.7. Yüksek Sıcaklık Dayanımı

Genel olarak, yüksek pomza içeriğine sahip karışımlarda 28 günde 200, 500 ve 800°C'ye yükseldikçe basınç dayanımı azalır. Dolayısıyla sonuçlar P100%-R0.46 için (300, 500 ve 800°C) sırasıyla (% 57.83, % 81.30 ve % 86.85) dır. Geopolimer hamurdaki uçucu kül içeriği arttığında, sırasıyla (300, 500 ve 800 ° C'de) (0.11, 18.15

ve 32.83) olan yüksek sıcaklıklardan sonra basınç dayanımı kaybı azalacaktır. Bu, uçucu külün pomza kaynaklı yüksek sıcaklığa daha toleranslı olduğunu gösterir.

5.8. Ultrasonik Darbe Hızı (UPV) Testi

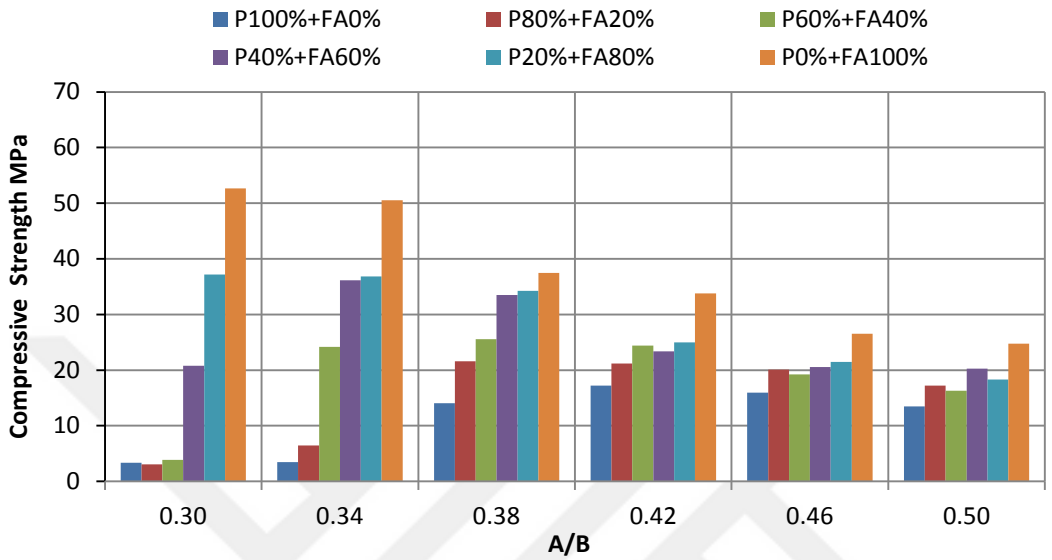
Genel olarak sonuçlar, kürleme süresinin 2 günden 6 güne çıkmasıyla UPV değerinin arttığını ortaya koymaktadır. Tüm geopolimer numunelerinin basınç dayanımı ve UPV değerleri arasındaki (fc = 0.0607e1.9101UPV) denklemi ve belirleme katsayısı (R2 = 0.7328) arasındaki ilişki. Yoğunluk ve UPV arasındaki korelasyon denklemi (fc = 533.01UPV – 60.78) idi ve belirleme katsayısı (R2 = 0.8139) iyi bir ilişkiye işaret ediyor.

6. SONUÇLAR

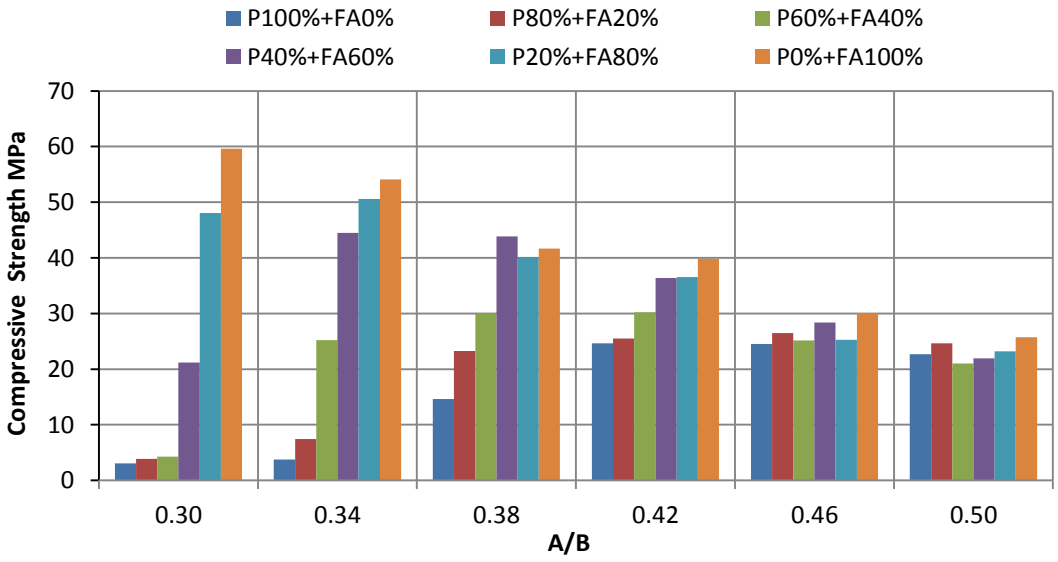
Sonuçlar, uçucu kül içeren pomza tozunun, karışımlar için (P100-R0.46, P80-R0) optimum basınç dayanımları (31.02, 32.03, 36.76, 50.43, 57.17 ve 69.90 MPa) olan iyi bir geopolimer bağlayıcılar oluşturmak için kullanılabileceğini doğrulamaktadır. Sırasıyla 46, P60-R0.42, P40-R0.38, P20-R0.34 ve P0-R0.30). Sonuçlar, FA içeriğindeki artışın, test edilen geopolimer numunelerinin farklı özelliklerinin gelişmesine yol açtığını ortaya koydu. En iyi değerler için en iyi kürlenme süresi 6 gün bulundu. Sonuçlar ayrıca alkali çözelti-bağlayıcı oranlarının ve kıvamının geopolimerler üzerinde önemli bir etkiye sahip olduğunu göstermiştir.

APPENDIX

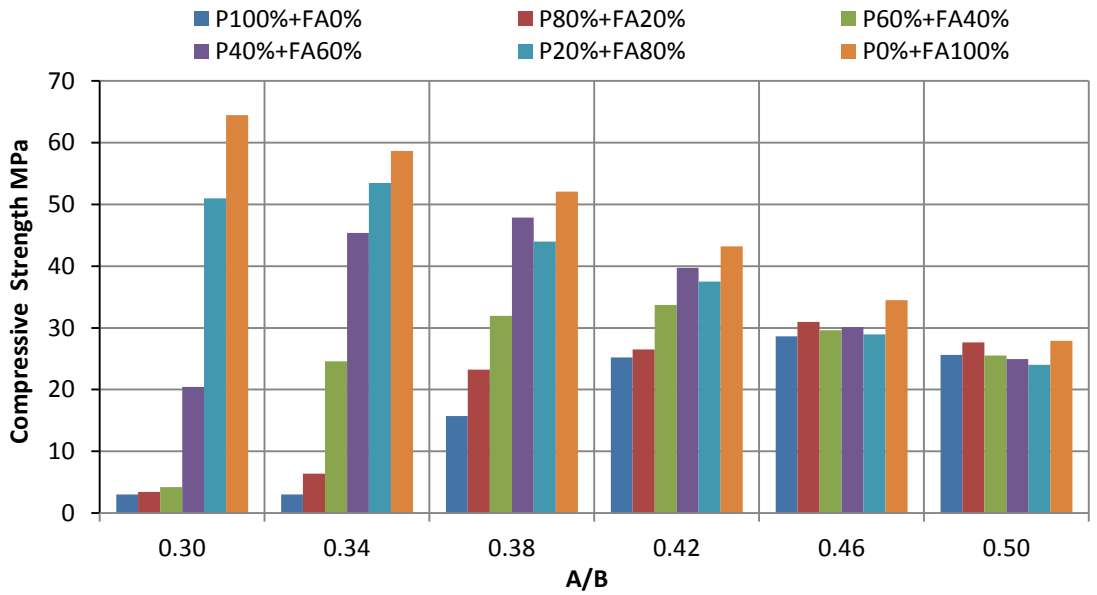
App 1. Extra figures of compressive strength.



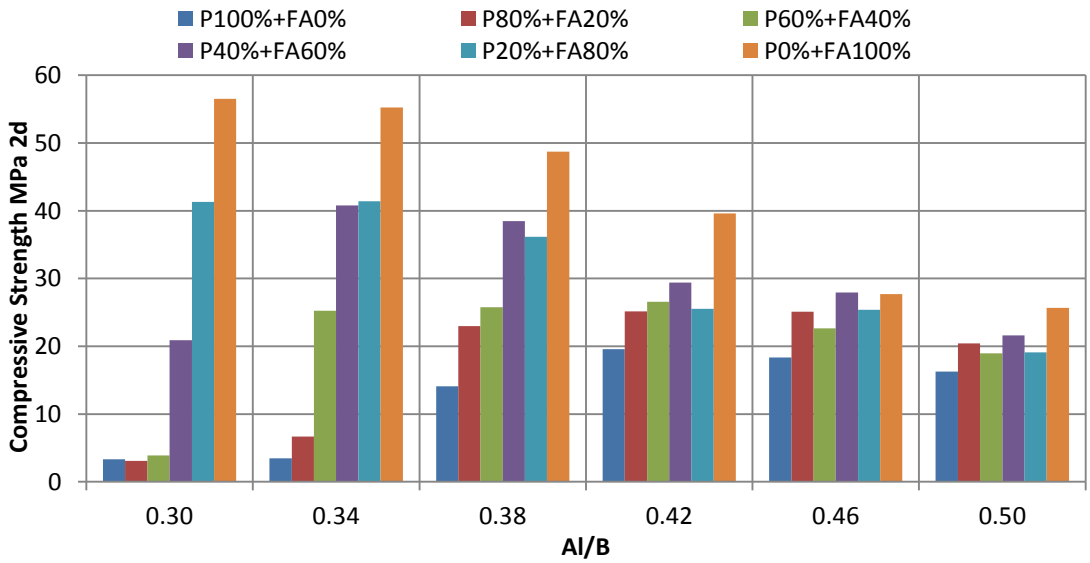
1. Compressive strength of P-FA geopolymer for different A/B at 2 days curing tested at 7 days.



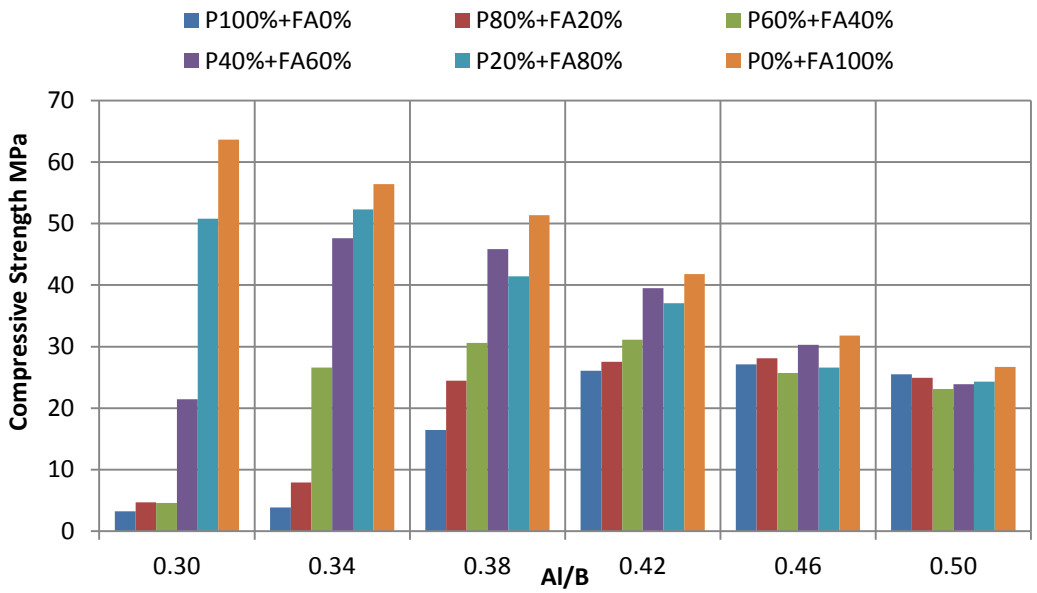
2. Compressive strength of P-FA geopolymer for different A/B at 4 days curing tested at 7 day



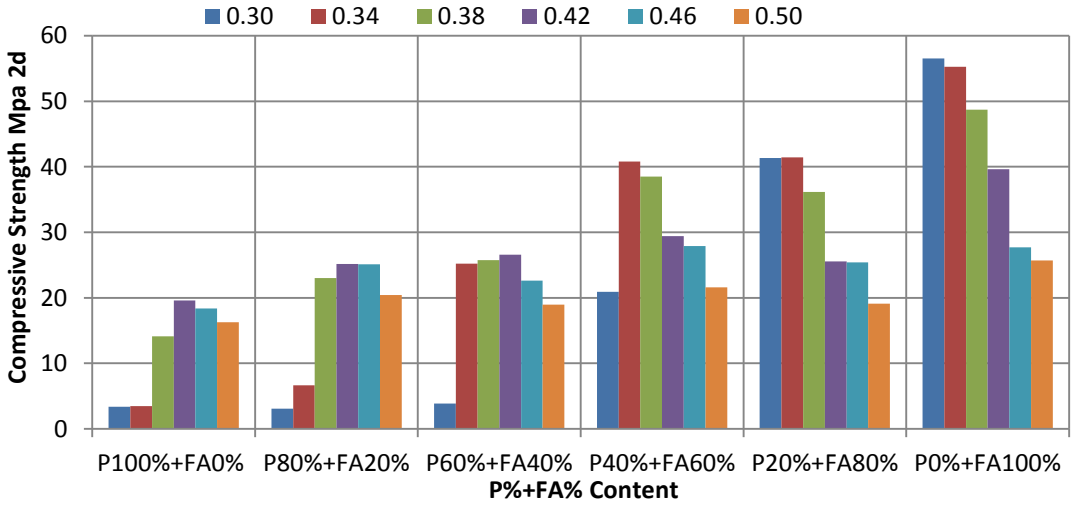
3. Compressive strength of P-FA geopolymer for different A/B at 6 days curing tested at 7 days.



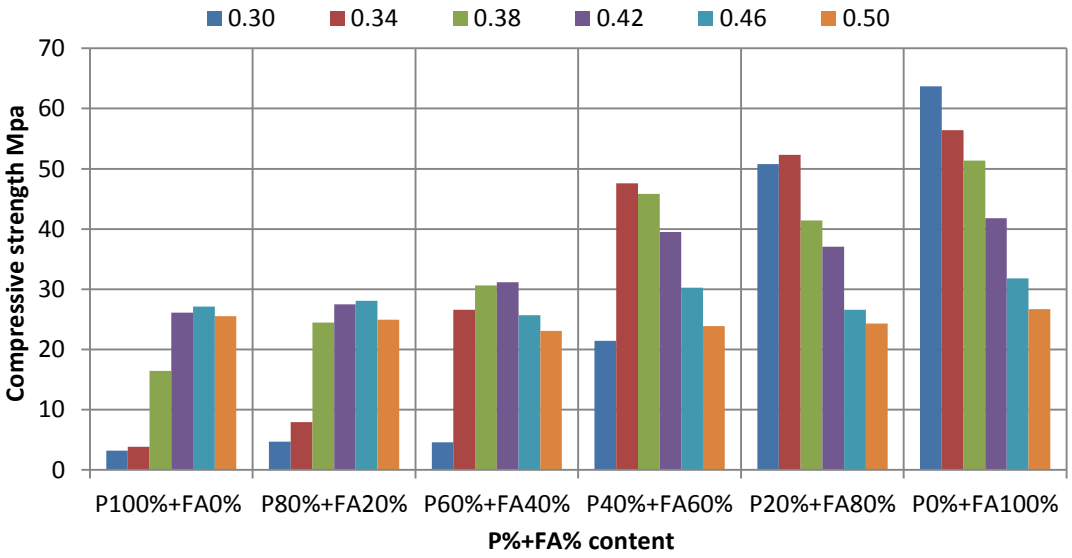
4. Compressive strength of geopolymer paste for different A/B at 2 days curing tested at 28 days.



5. Compressive strength of geopolymer paste for different A/B at 4 days curing tested at 28 days.



6. Compressive strength of geopolymer paste for different P+FA content at 2 days curing tested at 28 days.



7. Compressive strength of geopolymer paste for different P+FA content day curing tested at 28 days.

App 2. Extra figures of casting and samples.



1. Casting in molds with varied ratios A/B.



2. Samples kept in the laboratory until testing at 28 days.

CURRICULUM VITAE

Maadh Abdulmuttaleb Hamid HAMID completed high school in 1991. He holds a Bachelor of Civil Engineering from Anbar University in 1995. He holds a Higher Diploma in Building and Construction Engineering/University of Technology-Baghdad in 1999. He has worked in Anbar province projects from 2001 until now, with the degree of chief engineers, and holds a degree (consultant) in the Iraqi Engineers Union. In February 2019, he has started his postgraduate study in the Civil Engineering Department, Institute of Natural and Applied Sciences at Van Yüzüncü Yıl University.