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MASTER OF SCIENCE (MSc) THESIS

VISUAL INSPECTION AND MECHANICAL TESTING OF FLY ASH-BASED FIBROUS GEOPOLYMER COMPOSITES UNDER FREEZE-THAW CYCLES

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DEPARTMENT OF CIVIL ENGINEERING

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The study entitled **"Visual inspection and mechanical testing of fly ash-based fibrous geopolymer composites under freeze-thaw cycles"** prepared by Zana Muhamad MAHMOOD under supervision of Assoc. Prof. Dr. Kasım MERMERDAŞ was unanimously accepted as a MASTER of SCIENCE thesis on 23/12/2020 by the following committee in Graduate School of Natural and Applied Science, Department of Civil Engineering, at Harran University.

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ABSTRACT

MSc Thesis

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Geopolymer is a binder that can be considered as an alternative to Portland cement. The utilization of geopolymer concrete as an alternative to the Portland cement-based concrete adds sustainability to the environment by reducing the energy consumption and greenhouse gas emission associated with cement production. Fiber reinforcement is a popular practice known to improve performance of construction materials. In this thesis, an experimental study was conducted to evaluate freeze-thaw behaviour of fiber reinforced geopolymer composites. To build up an extensive experimental program, four different fiber types, namely, basalt fiber (BF), steel fiber (SF), and glass fiber (GF) with two different lengths and three fiber volume fractions of 0.4, 0.8, and 1.2% were utilized for geopolymer composite production. To monitor freeze-thaw resistance, 100, 200, and 300 cycles were applied as per ASTM C666. In the current study, the solid binding component of geopolymer composite was the fly ash with a fixed dosage of 550 kg/m^3 while the alkaline activators were Na_2SiO_3 and NaOH solutions. A constant alkaline activator-to-binding material (solid binding component) ratio of 0.5 was assigned. In total, 13 different fly ash-based geopolymer mixtures were designed. Based on the results of the experimental program it was found out that the workability of the fresh geopolymer composites was adversely affected by the fiber addition. A continuous increase in both compressive and flexural strengths was achieved when the volume fraction of the BF and SF type fibers were increased. Another critical finding is that utilization the fiber decreased the damage of the freeze-thaw cycles. Therefore, it was proved that the use of fiber can be an effective way to increase the durability of the geopolymer composites against the freeze-thaw cycle. Based on the visual inspection of the specimens throughout the whole exposure period, it was found out that the geopolymer specimens maintained their integrity with varying levels of mass loss even after being subjected to the 300 freeze-thaw cycles.

KEYWORDS: Geopolymer, Freeze-thaw, Fibers, Durability, Visual inspection

ÖZET

Yüksek Lisans Tezi

DONMA-ÇÖZÜLME DÖNGÜLERİNE MARUZ KALAN UÇUCU KÜL BAZLI ELYAF TAKVİYELİ GEOPOLİMER KOMPOZİTLERİN GÖRSEL MUAYENESİ VE MEKANİK OLARAK TEST EDİLMESİ

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Geopolimer, Portland çimentosuna alternatif olarak düşünülebilecek bir bağlayıcıdır. Geopolimer betonun çimento esaslı betona alternatif olarak kullanılması çimento üretiminden kaynaklanan enerji tüketiminin ve sera gazı emisyonunun azaltılmasından dolayı çevresel sürdürülebilirliğe katkı sağlar. Fiber takviyesi yapı malzemelerinin performansını iyileştiren popüler bir uygulamadır. Bu tezde, lif takviyeli geopolimer kompozitlerin donma çözülme davranışlarının incelenmesi için deneysel bir çalışma yapılmıştır. Ayrıntılı deneysel bir program kurmak için dört farklı lif seçilmiştir. Basalt elyaf (BE) çelik elyaf (ÇE) iki farklı boya sahip cam elyaf (CE) hacimce %0,4, %0,6, %0,8 ve %1,2 oranlarında kullanılmışlardır. Donma çözülme direncini belirlemek için ASTM C666'ya göre 100, 200, 300 donam çözülme döngüsü uygulanmıştır. Geopolimer için katı bağlayıcı bileşen 550 kg/m^3 uçucu kül iken, alkalin aktivatör ise Na_2SiO_3 ve NaOH çözeltileridir. Sabit bir alkalin aktivatör katı bağlayıcı oranı olarak 0,5 belirlenmiştir. Toplamda, 13 farklı uçucu kül bazlı geopolimer karışımlar tasarlanmıştır. Deneysel çalışmada, fiber eklenen taze geopolimer kompozitlerin işlenebilirliklerinin olumsuz etkilendikleri görülmüştür. Ayrıca BE ve ÇE türü fiber oranlarının artmasıyla basınç ve eğilme dayanımında sürekli artış olmuştur. Diğer kritik bir bulgu ise, fiber kullanımının donma çözülmeden kaynaklı hasarı azaltmada etkili olduğudur. Böylece, fiber kullanımının geopolimer harçlarının donma çözülme etkilerine karşı dirençlerini artırdığı kanıtlanmıştır. Bütün döngü periyodu boyunca yapılan görsel muayene sonuçlarına göre, geopolimer numunelerin 300 döngü sonunda bile çeşitli seviyelerde kütle kaybı ile bütünlüklerini korudukları anlaşılmıştır.

ANAHATAR KELİMELER: Geopolimer, Donma-Çözülme, Elyaf, Dayanıklılık, Görsel inceleme.

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SYMBOLS AND ABBREVIATIONS

ACI	American Concrete Institute
GGBFS	Ground Granulated Blaste Furnace Slage
BF	Basalt fiber
SF	Steel Fiber
GF-3	Glass fiber 3^{mm}
GF-6	Glass fiber 6^{mm}
Al_2SiO_3	Alumina silicate
CKD	Cement Kiln Dust
Al_2O_3	Alumina (Aluminum oxide)
FA	Fly Ash
PC	Portland Cement
K_2SiO_3	Pottasium silicate
GPC	Geopolymer Concrete
GPM	Geopolymer mortar
CaO	Calcium oxide
CO2	Carbon Dioxide
F	Maximum load
f'_c	Concrete compressive strength
Fe_2O_3	Iron oxide
f_t	Concrete flexural strength
K_2O	Potassium oxide
KOH	Potassium hydroxide
M	Molar
MgO	Magnesium oxide
Na_2SiO_3	Sodium silicate
NaOH	Sodium Hydroxide
OPC	Ordinary Portland cement
P_2O_5	Phosphorus oxide
GPCs	Geopolymer concrete copmosite
SP	Superplasticizer
OPC	Ordinary portland cement
Al	Aluminium
Si	Silicone
FA	Fly ash based geopolymer
SFRC	Steel fibre reinforced concreter

1. INTRODUCTION

1.1. General

The conventional concrete concept usually covers the adoption of Portland cement and other materials such as water, aggregates (fine and coarse), which exert an influence on the properties of concrete during blending. Portland cement manufacturing has certain drawbacks such as incurring high energy consumption, generating a great amount of CO_2 emissions, requiring a vast quantity of resources, and releasing a lot of greenhouse gases such as CO, CO_2, etc. (Bosoaga et al., 2009; Lämmlein et al., 2019; Xie et al., 2019). Manufacturing 1 kg of ordinary Portland cement (OPC) causes a generation of carbon emission approximately ranging between 0.66 and 0.82 kg that is almost 5-7% of the human-induced global CO_2 emissions (Peng et al., 2012). Theatrically, cement manufacturing has expanded as the infrastructural, and industrial development increased enormously (Habert et al., 2010). Jindal (2019), utters that as regards the International Cement Review Research, in 2018, the estimated value of the global cement demand is more than 4.216 billion metric tons of cement, meaning the required energy consumption to manufacture this amount of cement is approximately 9.476x107 Joules/ton. So, there are many attempts to decrease and/or cut the Portland cement employment in concrete such as the partial substitution of Portland cement with cementitious products and the pursuit of Portland cement alternatives.

In this context, instead of conventional concrete, a new concept that is not in need of the OPC has been developed by a French material scientist (Davidovits J., 1994). This concept is mainly based on the polymerization reactions taking place between the aluminosilicate raw material and alkaline activator, which conduce to the formation of Si-O-Al-O bonds that creates the geopolymers (Ma et al., 2018; Chowdhury et al., 2020). So, silicon- and aluminum-rich raw materials and alkaline activator solutions are required for geopolymer materials. Within this framework, the waste -products like rice husk ash, fly ash, ground granulated blast furnace slag, etc.

released owing to the growing industrialization can be adopted as suitable raw materials for the polymerizing process. Thereby, a lower level of energy consumption (around 40%) and greenhouse gas emissions (about 70%) is achieved when the geopolymer is of concern. In another saying, cement is not required as a binder in the manufacturing of the geopolymeric materials, which makes them environmentally-friendly construction material. This is because Davidovits explained that the amount of CO_2 released in the case of the geopolymers is about 0.184 ton per ton of binder. Aldin et al. (2019) which means 80% less CO_2 emission compared to OPC (Turner and Collins, 2013). In this aspect, the polymerization reaction can be likened to a more energy-efficient, eco-friendlier, and more sustainable form of hydration reaction occurring between the cement and water. However, the role of water in the geopolymer is not significant as in the hydration reaction since it does not participate in the polymerization reaction, just takes a part in the workability of the mixture (Jindal, 2019).

Another important issue in the geopolymer concrete is the curing regime applied after manufacturing that differentiates from the that applied to the traditional cement-based concrete. Since the polymerization reaction is activated by the elevated temperature, a heat curing regime is applied to the geopolymer concrete instead of water curing. The experimental studies available in the literature have revealed that in general, the ideal curing temperatures are between 60 and 100 °C (Kong and Sanjayan, 2009; Duan and Zhou, 2015; Aydin and Baradan, 2012). Besides, it has been stated that the desired mechanical performance, especially compressive strength, cannot be achieved when an ambient temperature curing regime is applied to the geopolymer concrete (Xie and Ozbakkaloglu, 2015; Noushini etal., 2016; Hake et al., 2018). Apart from the temperature of the curing regime, the chemical compositions of the raw materials and alkaline activator type also remarkably exert an influence on the performance of the geopolymer concrete (Zhang et al., 2020). Rice husk ash, fly ash, and ground granulated blast furnace slag are the industrial by-products, most-widely utilized raw materials in the geopolymer concrete manufacturing since they are rich in alumina (AL_2O_3) and silica (SiO_2) and can be procured easily due to their wide availability (Puertas et al., 2011). In this regard, sodium hydroxide (NaOH)-sodium silicate (Na_2SiO_3) and potassium hydroxide (KOH)-potassium silicate (K_2SiO_3) patterns are the most common alkaline solution ingredients handled to activate aluminosilicate available in the raw materials (Palomo et al., 1999). Although NaOH provides less alkalinity than KOH, in practice, since the capacity of NaOH for silicate and aluminate monomers liberation is higher, it has rather a preferability than the KOH (Davidovits, 1994).

In the literature, there are many experimental studies to investigate and reveal the influence of type and chemical compositions of raw materials Zhang et al. (2011); Puertas et al. (2003); Terzano et al. (2005); Temuujin et al. (2009); Van et al. (2002) , using binary or ternary utilization of raw materials Duan et al. (2015); Hake et al. (2018); Goriparthi and Roa (2017); Li and Liu (2007); Rovnaník and Śafrānkovā (2016) , incorporating ultra-fine materials such nanosilica and Alccofine Jindal et al. (2017); Adak et al. (2014); Singh et al. (2018); Deb et al. (2016), applying various curing types and temperatures Kong and Sanjayan (2010); Venkateswara et al. (2017); Adam and Horianto (2014); Kani and Allahverdi (2009), adding fibers like basalt, carbon, glass, etc. Ali et al. (2020); Guo and Xiong (2020); Mermerdaş et al. (2019); Mulapeer et al. (2019), and utilizing a different kind of alkaline activator with several mixing rates on the fresh Okeye et al. (2015); Abdul Rahim et al. (2014), mechanical, thermal, and durability characteristics of the geopolymer concrete. Ali et al. Ali (2020) added basalt fiber with two different lengths in the metakaolin-based geopolymer composite and reported that fiber addition leads to a slight increase in the compressive strength. However, they also expressed that improvement in the compressive strength due to using longer basalt fiber is much more than that owing to employing the shorter basalt fiber. Also, they explored the effect of basalt fiber addition and its length on the flexural strength and ultrasonic pulse velocity values. They deduced that adding basalt fiber leads to an increase in flexural strength, in particular, the longer basalt fiber addition augments the flexural strength more. Moreover, it was observed by Ali et al. (2020) that adding fiber to the metakaolin-based geopolymer composite results in an inconsiderable increase in the ultrasonic pulse velocity values. In the study conducted by Çelik et al. (2018), four kinds of synthetic fibers with various volume fractions were utilized in the metakaolin-based geopolymer composite manufacturing. They observed that the addition of the polyvinyl alcohol and basalt type fibers with the volume fractions of 0.4, 0.8, and 1.2% systematically conduces to an increase in the compressive strength, whereas polyolefin and modified polyamide type fibers increased the compressive strength till the fiber volume fraction of 0.8% and then, causes a decrease of the compressive strength at the volume fraction of 1.2%. Çelik et al. (2018) reported a conclusion similar to Ali et al. (2020) for the impact of the fiber addition on the ultrasonic pulse velocity. Besides, Ganesh and Muthukannan (2020) studied the effect of the glass fiber volume fraction on workability, compressive, splitting tensile, and flexural strengths of fly ash and fly ash/slag based geopolymer concrete. They reported that increasing the glass fiber volume fraction from 0 to 1.25% causes a decrease in the workability. Also, in accordance with the fiber volume fraction, they stated firstly an increase in the compressive, splitting tensile, and flexural strengths, and later a decrease. Additionally, Mermerdaş et al. (2019) investigated the effect of glass fiber content on the compressive and splitting tensile strengths of the fly ash-based geopolymer composite and found out that increasing glass fiber content from 0 to 1.2% leads to a remarkable increase in the compressive strength and a gradual rise in the splitting tensile strength of the geopolymer composites. The number of studies dealing with examining the effect of freeze-thaw cycles on the features of the fibrous geopolymer concrete is limited. However, when the researches about exploring the freeze-thaw impact on the mechanical properties of the geopolymer composite are investigated, it can be stated that the geopolymer composites have good resistance to the freeze-thaw action. Aygörmez et al. (2020) investigated the influence of freeze-thaw cycles on the mechanical and microstructural characteristics of the geopolymer composites. They stated a reduction in the compressive strength, flexural strength, and ultrasonic pulse velocity values and also microstructural deteriorations after 300 freeze-thaw cycles (cycling between -18 and 4 °C). Moreover, the SEM analysis conducted in this study revealed that the cracks in the micro-scale occur when the geopolymer composites were exposed to 300 freeze-thaw cycles. Zhao et al. (2019) also determined the freeze-thaw durability of the red mud slurry-fly ash-based geopolymer composites subjected to the 50 freeze-thaw cycles ranging between -10 and 4 °C. They observed that exposing the geopolymer composites to the freeze-thaw cycles results in diminishing of their compressive strength. Besides, they stated that one geopolymer composite mixture cured at ambient temperature was collapsed after the 44th freeze-thaw cycle.

1.2. Thesis Objectives

In study herein, the influence of fiber type and volume fraction on the workability feature, compressive strength, flexural strength, and ultrasonic pulse velocity properties of the fly ash-based geopolymer composites were experimentally investigated. Additionally, the effect of fiber addition on the freeze-thaw durability of such mortars was monitored by means of experimental investigation and visual inspection. Therefore, an extensive experimental program was built up in which four different fiber types, three fiber volume fractions, and three freeze-thaw cycles were designated. Besides, the results were statistically evaluated to demonstrate the effectiveness of the independent variables such as fiber type and volume fraction, freeze-thaw cycles on the dependent variables like workability, compressive strength, flexural strength, ultrasonic pulse velocity, and weight loss.

1.3. Thesis Outline

This thesis study consists of five chapters:

1. Chapter One: This highlights the value, durability and long-lasting of concrete and its effect on the nature and the use of environmentally-friendly concrete such as geopolymer concrete with its properties reviewed.
2. Chapter Two: This chapter presents an idea from previous studies that examined the production of geopolymer concrete and investigated several factors such as strengthened fibers which affect their freezing and thawing properties, the advantages and disadvantages of reviews and the compatibility of the classes with their views.
3. Chapter Three: This chapter describes the preparation of geopolymer concrete in terms of initialization, preliminary testing, compliance with the specifications necessary, the design of the mixtures, acquiring the required

resistance and preparing the specimens needed for the aggressive environment under the ACI318 requirements.

4. Chapter Four: In this chapter, the findings and discussion are discussed.
5. Chapter Five: In this chapter, the critical findings presented in the previous chapter are summarized..

2. LITERATURE REVIEW

2.1. General

GPC has less carbon dioxide, which means it is environmentally friendly relative to traditional concrete. Davidovits has claimed that the aluminium (Al) and the silicon(Si). They can react with the natural source material such as fly and rice husk ash to make the binders with the aid of alkaline liquid. Davidovits coined the word 'geopolymer' as the chemical reaction occurring in a polymerization phase (Rangan, 2010).

It is produced from minerals such as kaolinite, clays, etc. or by-products like FA, GGBS, etc. which by its alkaline activation include alumina-silicate. Not only does the geopolymer minimize the concrete greenhouse footprint, but also improves its mechanical properties, in which chloride penetration and resistance to acid resist (Wallah and Rangan, 2006).

2.2. Geopolymer as an alternative to cement

There are several obstacles for the building industry to resolve climate change forces, global warming and sustainability without economic growth (Swamy, 2008). Sustainability is now a widespread word used in daily interactions, maintained for promoting or sustaining a process and aims at sustainability to conserve the planet's wealth and environment for the foresight.

Purnell (2012), for its simplicity, flexibility, performance and other factors, has concluded that cement concrete has been most commonly used in recent years. The large-scale production will use high amounts of raw materials, consume electricity and emit noxious gas and waste from the dust.

Jean (2009), analysed the use of materials used in concrete production, which is twice the combination of plastics, iron, aluminium and wood and second position after water consumption.

Figure 2.1 shows the global production of cement. In the year 2015, the world cement production was around 3.6 billion tones, and other researches published the cement production to reach about 4.2 billion tons in 2050. This required more than 3 billion tons of limestone for clinker manufacturing (Schneider et al., 2011).

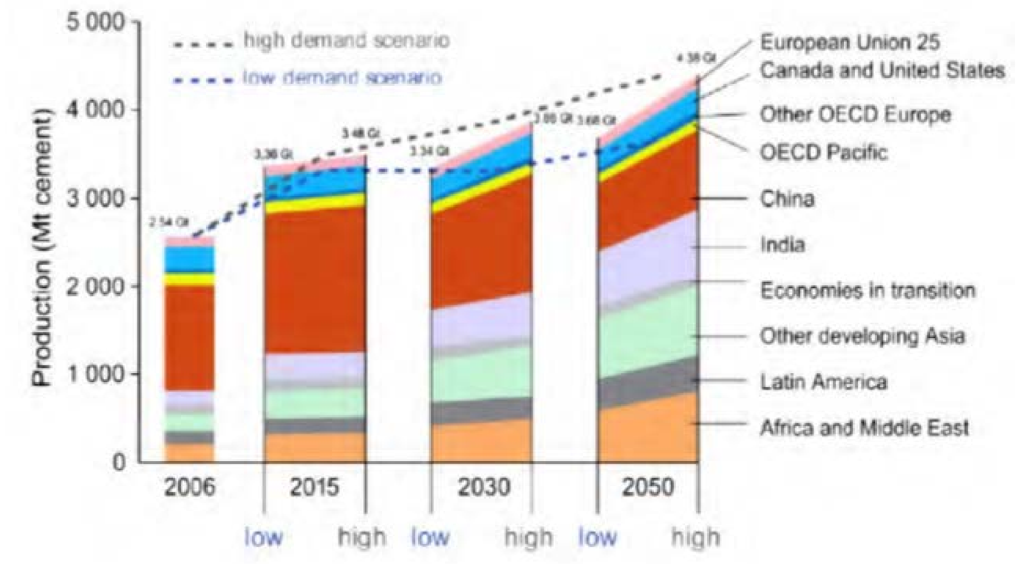


Figure 2.1. The global production of cement (Schneider et al., 2011)

Suitability has two main disadvantages:

- In the first point, approximately two-third tons of raw material needed to manufacture each ton of Portland cement which release nearly a ton of carbon dioxide during production contributing about 65% of global warming.
- Another reason for its exclusion from traditional concrete as well as the energy used for its purifying is the use of the large quantity of tap water, which also plays an essential role in hydrating Cement and working fresh concrete.

Geopolymer is a new binding material alternative to Portland cement. Low raw material consumption, low emission of carbon dioxide, low energy consumption, low production costs, high early strength and fast set-up are the common properties of GPC (Davidovits, 1994).

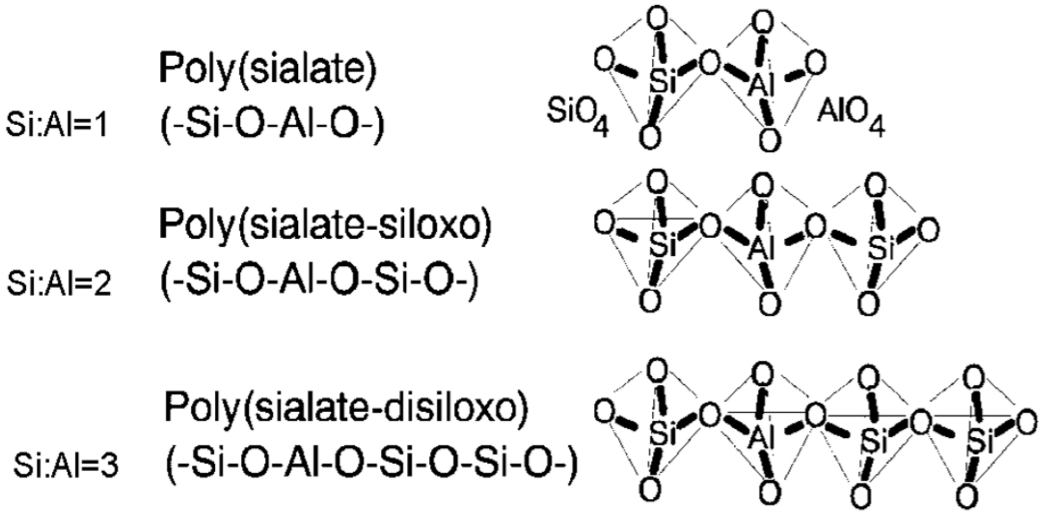
The reports of Davidovits show that 3/5 less energy is needed for the manufacture of geopolymer than Portland cement and around 80-90% less CO2 is produced. Thus, in Davidovits' environment protection (1994), application and development of geopolymer concrete are of great importance. The cement industry produces millions of tonnes, not just emissions of carbon dioxide, but energy from cement and concrete industries play essential roles in the risk of respiratory health and disease every year (Huntzinger and Eatmon, 2009).

2.3. Geopolymer Concrete:

2.3.1. Terminology and Chemistry

In 1978, the French scientist Joseph Davidovits coined the word "geopolymer" as referring to amorphous polymer alumino-silicate polymers that formed in an alkaline environment. Davidovits then developed the concept of geopolymer chemistry following extensive studies and in 1979 defined the properties of the new generation. He proposed term poly(sialate) for silico- aluminate based geopolymers. Sialate is an alkaline silicone oxo-aluminate in which alkali is which alkali might be potassium, sodium, lithium or calcium. Polysialates are Si4 + and Al3 + chain and ring polymers in IV-fold oxygen coordination, from an amorphous to semi-crystalline, as shown in figure 2.2.

The "geopolymers" of the following forms were christened for the amorphous to semi-crystalline silico-aluminate structures (Davidovits, 2002).



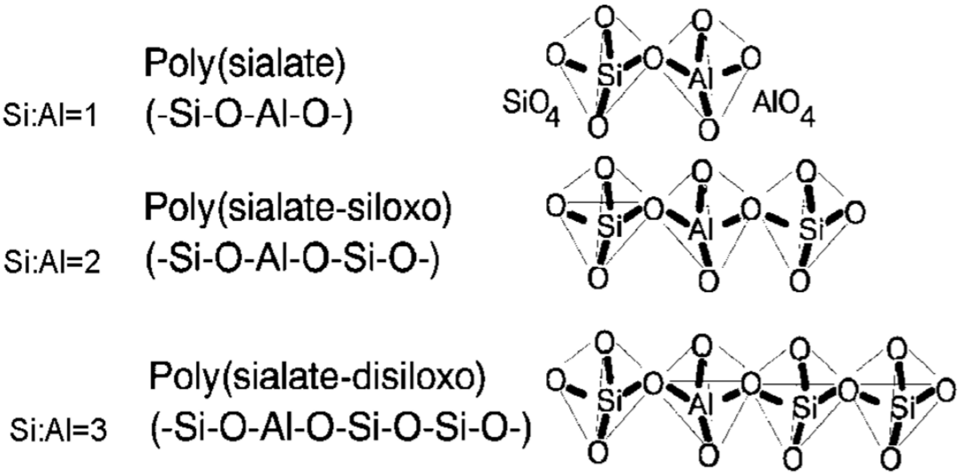


Figure 2.2. Chemical structures of polysialate (Davidovits, 1979)

Davidovits (1999), stated one of the three primary forms above could take the geopolymer. Furthermore, geopolymer's chemical composition is similar to that of minerals like zeolite but it is more likely to be amorphous than crystalline (Palomo et al., 1999a). They are usually hardened by polymerization with OPC-based binders, rather than by the hydration process. To date, most scientists accept that the systematic kinetics of reaction involved in geopolymerization process is still not fixed. In the

1950s, however, Glukhovsky suggested a general alkali activation process for materials, with the key components of silica and reactive alumina that be pointed as directed. This model divides the process of geopolymerisation into different phases like:

- The destruction–coagulation stage
- The coagulation-condensation stage
- The condensation–crystallization stage

Researchers such as Duxson (2007), Fernandez-Jimenez (2006), Provis (2007), have developed various theories for the geopolymer model. After the research, they propose the simple way for the geoploymerisation process which is presented in Figure 2.3 where multiple steps for the process of polymerization and durability are shown.

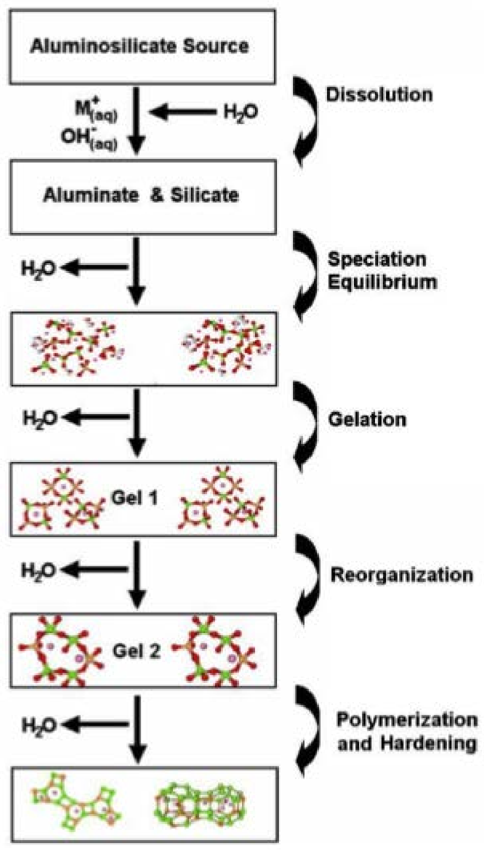


Figure 2.3. Conceptual model for geopolymerization (Fernandez-Jimenez et al.,2006)

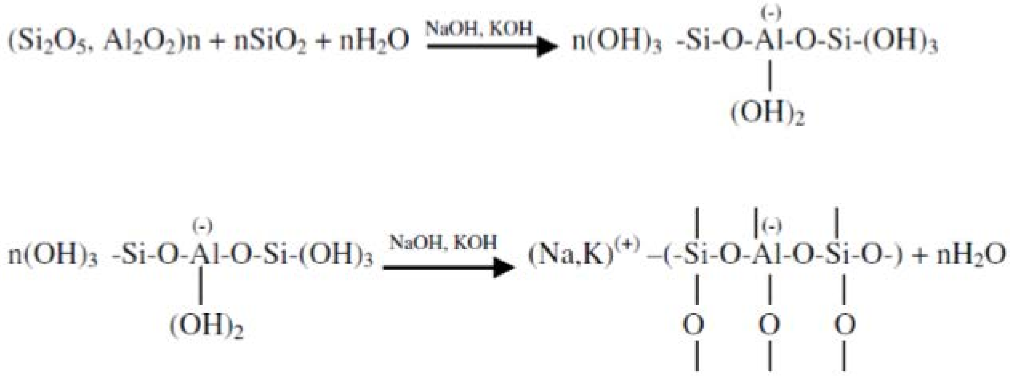
Likewise, the best-known design model for geopolymeric materials is from (Davidovits, 1999; Xu & Van Deventer, 2000). The steps include the following things:

- Firstly, dissolution of Si and Al atoms via the action of hydroxide ions from the source material.
- Then, the phenomenon of transportation or condensation or orientation of precursor ions into monomers was introduced.
- Finally, there involve a process of setting or polymerization of monomers into polymeric structures.

Palomo et al. (1999b) also stated the parallel progression of those stages, making it difficult to distinguish between those stages. In short, the chemical reaction of alumina-silicate oxides with alkali poly silicates leading to polymer Si – O — Al bonds, are necessary for the geopolymerization process. The poly condensation by alkali (Wallah and Rangan, 2006) can be described as the following:

Here, in the reaction, 2nd part was substantially explained by Rangan (2008) and is noted below:

The last term in Equation shows that the chemical reaction in geopolymer formation releases water that benefits geopolymers' performance. It contrasts with the chemical reaction of water during the hydration process in a cement mixture of Portland (Rangan, 2008).



$$(Si_2O_5, Al_2O_2)n + nSiO_2 + nH_2O \xrightarrow{NaOH,\ KOH} n(OH)_3\ \text{-Si-O-}\underset{\substack{|\\ (OH)_2}}{\overset{(-)}{\text{Al}}}\text{-O-Si-}(OH)_3$$

$$n(OH)_3\ \text{-Si-O-}\underset{\substack{|\\ (OH)_2}}{\overset{(-)}{\text{Al}}}\text{-O-Si-}(OH)_3 \xrightarrow{NaOH,\ KOH} (Na,K)^{(+)} -(\text{-}\underset{\substack{|\\ O\\ |}}{\overset{|}{\text{Si}}}\text{-O-}\underset{\substack{|\\ O\\ |}}{\overset{|(-)}{\text{Al}}}\text{-O-}\underset{\substack{|\\ O\\ |}}{\overset{|}{\text{Si}}}\text{-O-}) + nH_2O$$

Rangan (2008), provided a substantial explanation of the second part of the previous equation, and it is reported below:

2.3.2. Source Material

In the range of the source materials previously noted, many of them have been investigated in the making of geopolymer concrete. However, the most popular among them in the technology of geopolymers are clay materials kolinite and metakaolin, and industrial wastes (furnace slag, fly ash).

Different source materials have been used in research for the manufacture of geopolymer binders. However, it is to be noted that the main components of the source materials for alumino-silicate geopolymers should be silicon (Si) and aluminium (Al) in amorphous form. Therefore, natural minerals such as kaolinite, clays, albite, feldspar and stilbite, and by-product materials such as fly ash, silica fume, slag, rice-husk ash, red mud, are suitable to be used as source materials, since they are capable to provide a source of silicon and aluminium. This type of material when dissolved in an alkaline activating solution polymerizes into molecular chains and networks to produce the hardened binder.

2.3.2.1. Fly Ash

Fly ash is usually unwanted product formed by ignition of coal typically at 1500 ° C in the boiler unit oven in electric thermal or steam plants (Mehta and Monteiro, 2006). In 1998, fly ash was generated worldwide at over 390 million tons per year, but only 14.00 % of the ash was used, and the other was thrown out in the dumps. Similarly, the annual production of fly ash was 780 million tons in 2011, but only 53 % of fly ash was used (Heidrich et al., 2013).

In general, Al, Si, Ca, Mg, and Fe are the constitutive elements of fly ash, even though the composition is different from the carbon source. The ASTM C618, reports two types of ash which are given below:

- Class C
  In this generally lignite or sub-bituminous coals falls. They are also known as having high content of calcium and referred as high calcium fly ash.
- Class F
  In this class, usually the bituminous coals fall . They usually have low calcium than the class C and called as low calcium fly ash.

The components summation can be greater than seventy percentages (ACI committee 226 reports) for fly ash classifying as Class C (SiO_2, Al_2O_3 and iron). In this study, low calcium class F is used as shown in Table 2.1. Class F fly ashes have pozzolanic properties, and they are soft in touch and available in powder form depending on the fuel and the iron oxide content. Likewise, class C fly ashes are shaped like a fine greyish polish with physical properties and pozzolanic characteristics that are mainly lime, silica and alumina. In this kind of ash, the quantity of lime (CaO) is high so that they can used without the use of a binder. The criteria for pozzolan for concrete is that fly ash particles should be able to pass through the No. 325 (45 μm).

Table 2.1. Chemical Composition and Physical Properties of Low Calcium Fly Ash (ACI committee 226 report)

Chemical Composition (%)	**Fly Ash**
Silicon dixide (SiO_2)	52.90
Aluminum oxide (Al_2O_3)	28.21
Calcium oxide (CaO)	3.00
Msgnrsium oxide (MgO)	5.21
Ferric oxide (Fe_2O_3)	5.31
Sulfur trioxide (SO3)	0.68
Loss on ignition	3.90
Specific gravity	2.31

The production of fly ash-based GPC, which has a compressive strength range from 30 to 80 MPa and slumps of 100 to 250 mm was investigated by (Hardjito and Rangan, 2014). At about 0.179 water-geopolymer solid ratio, the optimum strength was achieved and healed at 90 °C. The compressive strength decreases if the ratio increases due to the relation between the compressive strength and water-cement ratio for OPC concrete.

Fernandez et al. (2006), agreed that the compressive strength of 45 MPa of fly ash-based geopolymer concrete at 0.555 l/s (liquid/solid) ratio was obtained at 85.5 ° Cover 20.01 hours, as presented in Figure 2.4.

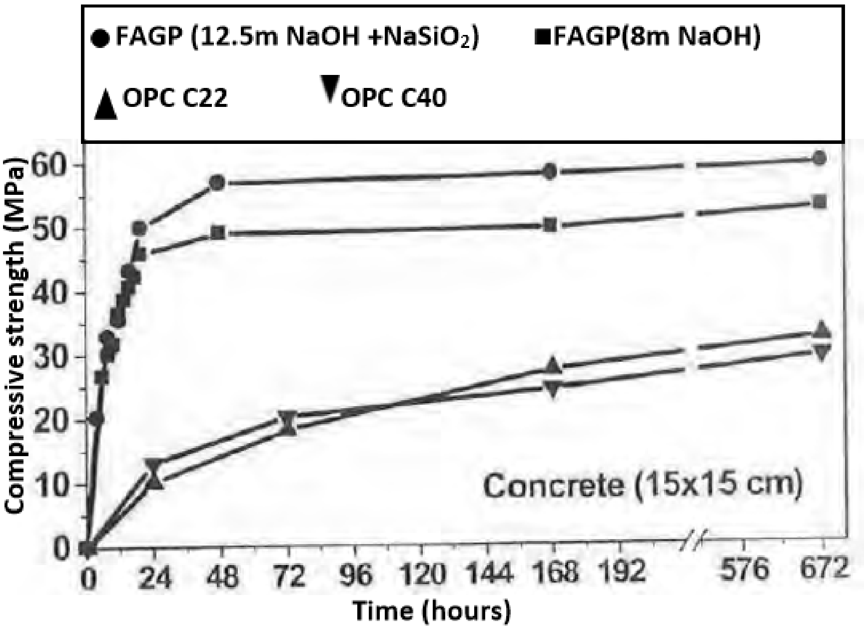


Figure 2.4. Compressive strength of fly ash based geopolymer comparing to OPC concrete (Fernandez et al., 2006)

The conclusion of Olivia and Nikraz (2012) is that the proportionate fly ash-based geopolymer (FAG) concrete can be mixed with 55.00 MPa compressive strength at twenty-eight days and cured in the 60.0–75 C range at various temperatures. This hardened blend had higher tensility and bending strength, less expansion and a 15-29 % less flexibility modulus than the OPC concrete mix. After twelve weeks, the drying decrease of GPC (0.025%) was less than that of OPC concrete (0.09%) which can be due to GPC's high strength, which is similar to the microstructure of zeolite, towards the drying of water incorporated while casting.

Several researchers have reported that the geopolymer containing fly ash has some advantages in term of tensile strength which is higher than OPC concrete, as shown in figure 2.5. Similarly, the increased strength is a thicker interface zone of aggregate and the geopolymer matrix. The elasticity modulus increases when the compressive strength increases the GPC but it has been found to be smaller than the given guidelines by ACI for concrete OPCs (Singh et al., 2015) predicted.

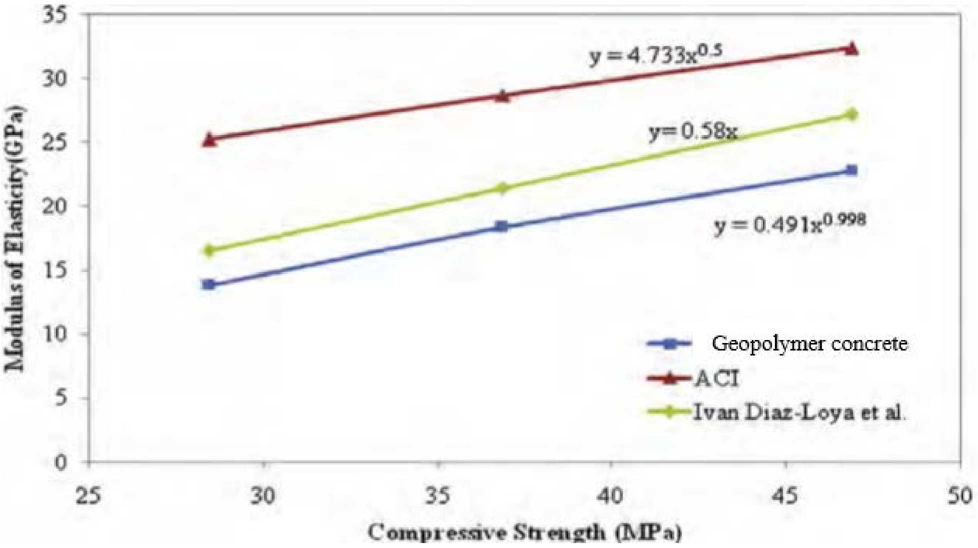


Figure 2.5. Correlations with in the mechanical properties of fly ash-based geopolymer concrete (Singh et al., 2015).

2.3.2.2. Ground Granulated Blast-Furnace Slag (GGBFS)

Ground granulated blast furnace slag (GGBFS), are formed by processes like the manufacture of pig iron from iron ore, coke residue combustion and streams, including calcareous and serpentine ore, and other materials. If high-pressure water cools down the molten slack, a fine-grain glass made of Ca – Al-Mg glass is formed. Granular furnace blast slag is a non-toxic material and can be an excellent raw material for the production of geopolymers.

The strength of GPC with low calcium fly ash and slag have been studied by the Naidu et al. (2012). By substituting the fly ash for slag, the five different mixes presented in Table 2.2 have prepared five different combinations. Table 2.3 provides different cube strengths and divided the tensile strengths of five mixed proportions. It was found that the compressive strength of geopolymer concrete increases when fly ash substitution with GGBS increment is increased for all age groups tested (3, 7, 14 and 28 days). In addition to the fast setting fly ash replaced with GGBS by up to 28.57%.

Table 2.2. Mix proportions (Naidu et al., 2012).

Materials (Kg/m$^{3)}$		Mix 1	Mix 2	Mix 3	Mix 4	Mix 5
Coarse aggregate	20 mm	277	277	277	277	277
	12.5 mm	370	370	370	370	370
	4.75 mm	647	647	647	647	647
Fine aggregate		544	544	544	544	544
Fly ash		408	370	340	313.85	291.43
Slag		0	37	68	94.15	116.57
NaOH		41 (8M)	41 (8M)	41 (8M)	41 (8M)	41 (8M)
Sodium silicate		103	103	103	103	103
Extra added water		22.5	22.5	22.5	22.5	22.5
Final setting time (hr)		6	6	5	3	1

Table 2.3. Compressive and split tensile strength for different ages (Naidu et al., 2012).

Mix	Strength (Mpa)							
	3 days		3 days		3 days		3 days	
	Comp.	Tensile	Comp.	Tensile	Comp.	Tensile	Comp.	Tensile
Mix 1	4.25	0	6.2	0.63	7.14	1.2	8.27	1.77
Mix 2	9.48	2.3	17.89	3.7	19.55	4	24.29	5.35
Mix 3	15.26	3.45	31.26	5.39	36.0	8.98	41.04	10.32
Mix 4	25.55	5.17	39.17	7.59	40.63	9.20	45.76	12.43
Mix 5	31.85	9.05	46.52	10.56	53.63	10.94	57.33	11.40

Venkatesan and Pazhani (2016), have been studying specific properties of geopolymers made of GGBFS and other black rice husk ash (BRHA), respectively. As shown in Table 2.4. four mixes have been prepared, which are at 60 °C and 90 °C in the ambient and in the oven. Experimental findings show that the compressive strength for the oven-cured was different as it was higher than that for the ambient cured samples. Further, increase in heating temperature to 90 °C as shown in figure 2.6 a, b and c respectively showed a moderate increase in compression strength.

Table 2.4. Mix Proportions (Naidu et al., 2012).

Quantities (kg/m^3)	GP	GPR1	GPR2	GPR3
GGBS	394	355	315	276
BRHA	0	39	79	118
Coarse aggregate	1201	1201	1201	1201
Fine aggregate	647	647	647	647
NaOH	45	45	45	45
Sodium Silicate	113	113	113	113
Super plasticizer % of binder	1.5	1.5	1.5	1.5
Extra water	59	59	59	59

Note : GP- Control Concrete GPR1-10% BRHA- 20% BRHA GPR3- 30% BRHA

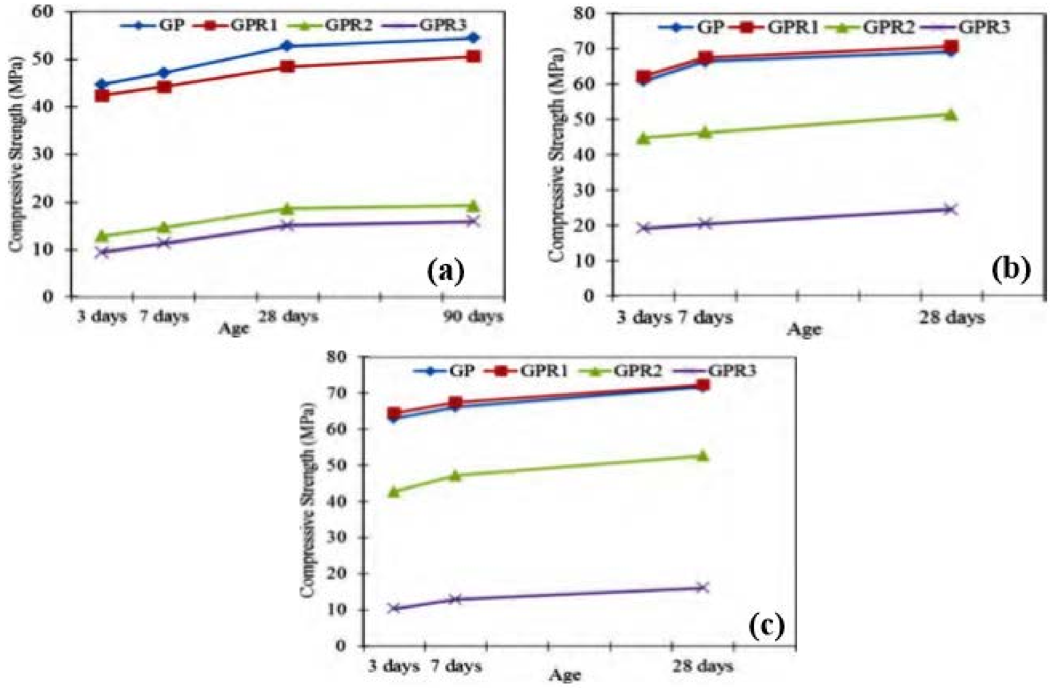


Figure 2.6. Compressive Strength of Geopolymer Concrete cured at (a)Ambient Temperature, (b)60°C, and (c) 90°C (Naidu et al., 2012).

2.3.2.3. Metakaolin

Metakaolin production depends on the output of Kaolin clay under controlled conditions to produce an amorphous aluminium silicate material (Kakali et al., 2001; Tironi et al., 2012; Marín-Lópeza et al., 2009) compare Metakaolin Geopolymer concrete properties to Portland concrete through research in which Metakaolin Geopolymer concrete has a compressive strength higher than Portland concrete. The characteristic increase is due to the increased resilience of the binding material, which synthesizes geopolymer with calcined source materials. The compressive strength comparison is shown in Figure 2.7.

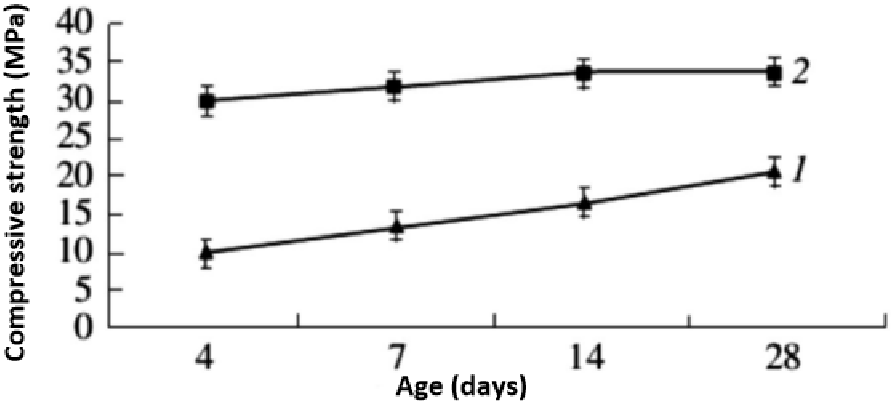


Figure 2.7. Compressive strength development with age (1)Portland cement concrete, (2)Metakaolin geopolymer concrete (Marín-Lópeza et al., 2009)

2.3.3. alkali Solution

In this scenario, as noted earlier the alkaline liquid is one of geopolymer binders' main constituents. The geopolymer containing fly ash requires high-alkaline solutions to dissolve SiO_2 and Al_2O_3 ions present in fly ash (Davidovits, 1994). Many study studies have demonstrated in this context that silicone and aluminium atoms in the sources of materials may be dissolved and gel formed by a combination of NaOH with Na_2SiO_3 or KOH with K_2SiO_3. The principle of alkaline activation of burning furnace slag, as set out in (Shi and Qian, 1999). It was, however, in the 1960s, (Gluhovsky and Pakhovmov, 1960), that systemic exploratory reviews on this subject were carried out.

In the latest decades, a lot of research has been carried out about the different use of alkaline fluids in the production of geopolymer binders and concrete. It should be noted that NaOH with Na_2SiO_3 solution or KOH with K_2SiO_3 solution is the most frequently used alkaline activator by researchers. Geopolymeric fly ash based cement was developed by Silverstrim et al. (1997) and van Jaarsveld et al. (1997) using a combination of NaOH and sodium silicate (Davidovits 2008). The use of a NaOH solution in order to achieve kaolinite polymerization was reported by (Davidovits et al., 1999). In addition, these types of alkaline activators have been used for various research in the various studies which were previously reported on source material carried out by Palomo et al. (1999); Grutzeck et al. (1999); Xu and van Deventer (2001); Swanepoel and Strydom, (2002); Xu and van Deventer, (2002). It was concluded that the type of alkali solution used to activate fly ash plays an important role in the process of geopolymerisation.

Likewise, improved solution-to-raw material-reaction section of the sodium silicate solution mixture as an alkaline activator. The other geopolymers such as potassium polysialate (K-PS) and potassium sialate disiloxo (K-PSDS) were made by Barbosa and. They found that there was good thermal stability of potassium geopolymers. The process of producing geopolymer containing fly ash concrete was also studied by Hardjito (2005), in which the fly ash was activated with a combination of NaOH solution and Na_2SiO_3 solution. As a consequence, it was reported that when the SiO_3^{2-} to NaOH ratio increases by mass, the compressive strength of fly ash-based geopolymer concrete increases. Similarly, after that the reseach step ahead for the good design with good properties for the manufacture of low-calcium fly ash geopolymer concretes, using a combination of NaOH and Na_2SiO_3 was carried out by (Rangan et al., 2008).

2.3.4. Curing of Geopolymer Concrete

Various tests were conducted for studying the effect on characteristics of geopolymer pastes of different curing conditions. The temperature for a total geopolymerisation reaction was reported in the range of 40- 85 C.

Similarly, Palomo et al. (1999) studied fly ash curing at 65.01 ° C and 84.99° C. The compressive strength of the geopolymers (8.001–12.01 M) cured at 84.9 ° C for

24 hours as shown in Table 2.5. which was much greater than those cured at 65.9 ° where the strength increase was lower after 24 hours.

Table 2.5. Compressive strength (MPa) of prisms made within activator/fly ash ratio of 0.25 (Palomo et al., 1999)

Activator	Curing temperature	Activator/fly ash ratio 0.25 (time of curing)			Activator/fly ash ratio 0.3 (time of curing)		
	(C°)	2h	5h	24h	2h	5h	24h
Solution 1	65	0.0	0.0	21.2	0.0	1.8	17.3
	85	9.3	22.0	34.6	9.2	9.6	23.4
Solution 2	65	0.0	0.0	8.7	0.0	0.0	3.9
	85	1.4	9.4	23.3	2.6	16.4	27.3
Solution 3	65	4.3	31.7	52.7	0.00	30.0	62.6
	85	39.8	48.2	54.5	31.6	57.4	68.7
Solution 4	65	0.0	9.5	38.7	0.0	10.2	39
	85	7.7	34.3	63.0	16.0	31.6	35.9

2.4. Properties of Geopolymer Concrete

For determine the properties of geopolymer concrete in terms of chemical and physical, different research was carried out. Also, studied the sustainability of geopolymer materials during development phase of it. Finally, the division of physical properties such as behaviour and chemical properties such as heat, temperature, electric or magnetic field or due to rays of light was done. The mechanical properties reflect the performance of materials deformed by force systems. Of course, not all of the characteristics, but only those that are addressed in this thesis will be reviewed.

Davidovits et al. (1988), carried out different physical and chemical tests on the mechanical properties of geopolymers that had shown various exceptional properties, such as high strength, high resistance to freezing and thawing, sulfate attack, corrosion and low shrinkage. He also mentioned that geopolymer bindings hardened rapidly at a room temperature, while after 4 hours at 20 ° C the strength of the compressive increases up to 20 MPa and after 28 days about 70.5-100 MPa.

Comrie et al. (1988) also showed that they had a compressive strength of 40 MPa over 28 days, following unconfined cubes made from sand and geopolymer composite mixtures. In addition, strengths of 30 MPa during the initial two days of curing (representing 75% of the last strength) was achieve successfully. Thus, from this it was finalized that strengths were able to acquire quickly when compared with concrete mortars made of OPC with geopolymer composites.

Likewise, the temperature is a speed-up of reactions in geopolymeric binders, according to Palomo et al. (1999) and also gain the mechanical strength with the rise in temperature. Normally, there are other important factors such as temperature and the kind of activator affecting geopolymer materials mechanical strength. Also, it was cleared that if the curing time was longer, it had the higher strength (Palomo et al., 1999).

2.4.1. Fresh Geopolymer Concrete

Nanavati et al. (2107) indicating that the geopolymer mixtures as defined in IS: 1199-1959 were performed to evaluate the operability. Similarly, it was found that when the amount of water was increased, it helped to increase the working capacity of the concrete. But the compressive strength seemed to be reduced. Some compacting factor test IS: 1199-1959 was used for evaluating some mixtures. The compositions showed a growing factor of slump and compaction with a decreased angular content of 7 mm (0.273 inches) in relation to the behaviour of fresh cement in Portland cement.

2.4.2. Hardened Geopolymer Concrete

2.4.2.1. Compressive Strength

Experimental research has shown that the aggregate binder interfaces are better than for Portland concrete in geopolymers. This can result in better mechanical and long-lasting properties of the concrete geopolymers (Provis et al., 2007).

2.4.2.2 Tensile Strength

Nanavati et al. (2017) reported that, after a period unlike that OPC, GPC is more effective in tensile strength at an early age. The Splitting Tensile Test or Indirect Tensile Test is the test used for the tensile strength of concrete. The tensile strength objective is similar to the compressive strength objective. In comparison with control OPC mixes, higher strengths were observed at long ages for the blends of geopolymer concretes based on activated natural pozzolan. The research showed that the long-term tensile strengths of geopolymer concrete mixtures are higher than those of OPC mixtures. Geopolymer concrete's tensile strength is more sensitive to inappropriate cure than its compressive strength, as is the case with OPC concrete. The maximum heating temperature was 40 °C (104 °F), which was the same as for compressive strength.

2.4.2.3 Unit Weight

Unit weight is another criterion for the hardening geopolymer concrete. It depends primarily on the unit weight of combinations. Experiments have shown that the unit weight of geopolymer containing fly ash concrete with the low-calcium fly is linked to Portland cement. Also, it was seen that unit weight between lied between 2330.00 and 2430.01 kg /m^3 if coarse granite-types were used. The methods used in PC Hardjito, D. and Rangan, B. V., (2005), are likely to create geopolymer light-weight concrete and geopolymer foamed concrete.

2.5 Mixture proportion and Mixing of Geopolymer Concrete

The literature analysis on geopolymeric development shows that very little research on geopolymer concrete was reported from the initiation by Davidovits (1978) until Rangan et al. (2005, 2006, 2008) conducted intensive investigations and tests in Curtin University of Technology (Australia) (2005, 2006, and 2008). In the majority of the research, the component and properties of geopolymer paste have been determined; thus, no exact mixing proportions have been proposed for concrete.

Moreover, it was actually focused on attracting the compression strength of the geopolymer containing fly ash. He reported using four concentrations of NaOH and various fly ash/water ratios.

The previously described studies by Palomo et al. (1999) aimed at analysing the geopolymerisation of low calcium ASTM Class F fly ash with a Si / Al proportion of 1.810. Furthermore, the paper reported that four of the different activator solutions had been applied with a mass proportion of alkaline liquids to ash fluctuating between 0.250 and 0.3001.

Geopolymer concrete preparation mix is connected to cement concrete. Two sorts of ground aggregates were blended in a dry state, sand and fly ash. Subsequently added a mixed solution of NaOH and Na_2SiO_3 with more water-based binder proportions and mixed well for three to four minutes to ensure a uniform mixture. The cohesive geopolymer concrete from fresh fly ash was discovered, which was vicious and dark in colour. The working capacity of fresh geopolymer concrete was calculated after making the homogeneous mix in accordance with IS 5512-1983 and IS 1727-1967. The 150 * 150 * 150 mm concrete cubes are cast in 3 layers. The 16 mm diameter tamping poles are fully compacted in each layer. For conventional concrete compaction, all cubes were located on the vibrator and vibrated for 2 min. The top surface was leveraged by applying a trowel after the compaction of concrete. All cubes were de-moulded after twenty-four hours of long casting and subsequently placed in the heating (thermal) oven. Then, to reduce the unexpected change in temperature, the such concrete cubs were allowed to cool down to the room temperature in an oven. Three cubes have been casted and tested Mr Kamble V.S. et al. (2016), for compressive strength during each curing period.

Similarly, a range of 8 molars to 16 molars for NaOH solution concentrations was recommended. The proportion between two things solution to fly ash alkaline activator solution by mass was proposed to be 0.30 and 0.40 respectively. Finally, the fine and ground aggregates used in the fabrication of ordinary Portland concrete were suggested to suit the geopolymer concrete as well. The amount of mass can be used for the concrete mixture is from 75 to 80%. Ragan and Wallah (2006) had eventually published an article that provides a further area in the case of lower calcium based geopolymer containing fly ash as shown in Tables 2.6. The alkaline liquid used for this study was a combination of Na_2SiO_3 solution and NaOH solids dissolved in water in flakes or pellets.

Table 2.6. Geopolymer Concrete Mix Proportions (Rangan and wallah, 2006)

Materials		Weight (Ib/ft^3)	
		Mix 1	Mix 2
Caorse Aggregate	¾ in	17	17
	½ in	23	23
	¼ in	40	40
Fine sand		34	34
Fly ash		25	25
Sodium silicate solution		6.42	6.42
Sodium hydroxide solution		2.55 (8M)	2.55 (12M)
Super plasticizer		0.374	0.374
Extra water		None	1.4

2.6 Durability Requirements

Concrete durability is the capacity to withstand climate changes, chemical invasion and abrasion. It is also affected by the strength of concrete to fluid penetration and has a direct correlation with the way it works, design life and safety. As part of the durability studies for concrete, the conditions of ACI 318R-14 have recognized types and categories of concrete which is given below:

- Exposure Category F: It applies to external concrete that is exposed to moisture and cycles of freezing and thawing with or without dicing chemicals.
- Exposure Category S: It applies to concrete that contains deleterious quantities of water-soluble ions in contact with soil or water.
- Exposure Category W: It applies to concrete in water contact but not subject to freezing and thawing, chlorides or sulfates.

- Exposure Category C: It is for non-prestressed and prestressed concrete exposed to conditions requiring additional protection against reinforcement corrosion.

The effect of these components should be lowered by combining or individually in order to increase the concrete durability. ACI has recognized two key aspects for determining the lifespan of this concrete in cement-based concrete (OPC) i.e the ratio of water to cement (w/c) and its compressive strength (f'c). The scenario ought to be distinct for geopolymer concrete because there is no (w/c) ratio and the compressive strength is influenced by multiple components such as source material, the concentration of NaOH, the ratio of Na_2SiO_3 to NaOH etc.

2.6.1 Exposure to Freezing and Thawing

In some countries, the long-lasting and resistance of concrete against freezing and thawing might not be relevant. But this seems to be the significant thing in cold countries. The freezing begins with broad areas and moves into smaller spaces (Shetty M.S., 2006).

It is known that if water freezes its volume increase up to 9%, which lead to internal tensile stresses if further increases. The increase in pore size will continue during the thawing phase, which will lead to increased water absorption so that the concrete exposure to frequent freezing and thawing cycles will cause damage (Neville and Brooks, 2010).

The major factors affecting concrete resistance to freezing and thawing are (Harrison et al., 2001):

- The saturation degree prior to freezing.
- The quality of the concrete will reduce the damage due to freezing and thawing while increasing the strength.
- The greater the water available, the greater the water volume available.
- Lowest freezing temperature
- The freezing period.
- The presence of icing agents like salt or urea increases the severity of any damages to the freeze-thaw.

The delayed exposure to freezing and thawing of the concrete is shown in figure 2.8, finally reducing the risk of concrete damage.

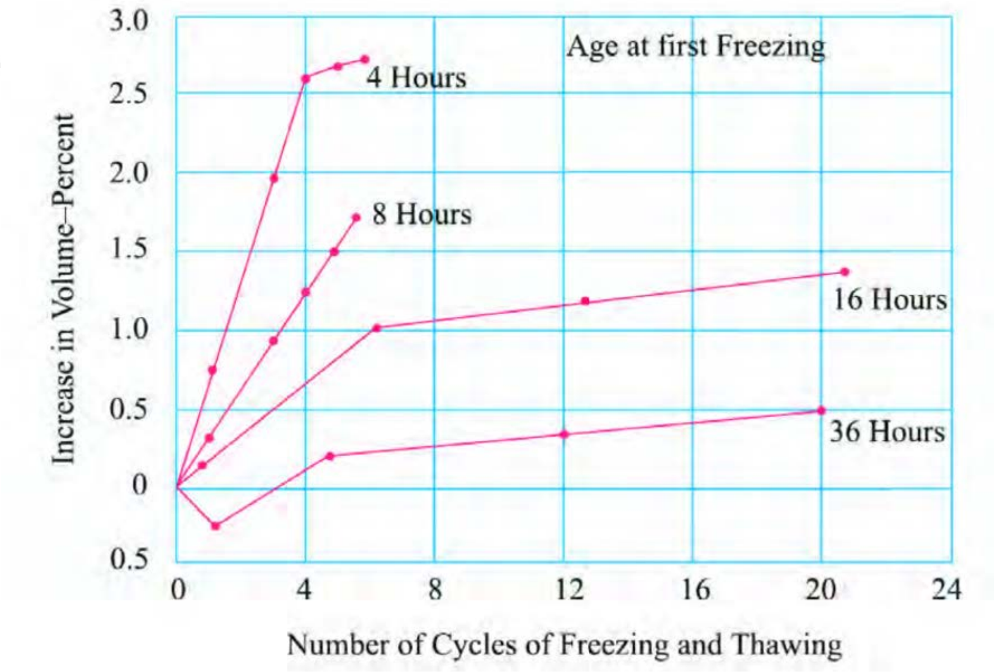


Figure 2.8. Effect of first freezing on volume of concrete (Shetty M. S., 2006)

An analysis of freezing and thawing, according to ASTM C666, is appropriate for determining concrete resistance to freezing and thawing, which includes two methods:

- Procedure A - In which the water surrounding samples is contained, i.e. the freezing and thawing process.
- Procedure B - The air surrounding samples are in the freezing phase, but the water is thawing.

Method B is employed for testing the geopolymeric mortar, which has 150 cycles. In comparison to non-exposed samples, compression strength decreased to 70%.

Cylindrical geopolymer paste was made of metakaolin, and fluidized bed combustion bottom ash and samples were processed at laboratory temperature as part or entire of metakaolin substitution (22 C). The exposure of these samples in accordance with the European Standard EN 14617-5:2005 was investigated by Slavik et al. (2008), into 50 freezing cycles and found that the compressive strength fell to 80%.

The effect of freezing and thawing on metakaolin based geopolymer composite using quartz sand was studied by Steinerova (2011) and was cured for 3 weeks on lab temperature and pressures of sealed plastic foils. Samples 40 * 40 * 160 mm was subjected to 25 freezing and thawing cycles. The relationship between sand and freezing resistance in geopolymer composite was established, and the minimum weight was 34 %. The small pores of the samples which contain less sand do not have 25 freezing and thawing cycles. On the other hand, if the sand content was increased in geopolymer composite, the large pores were produced, which were able to bear freezing and thawing cycles as illustrated in figure 2.9.

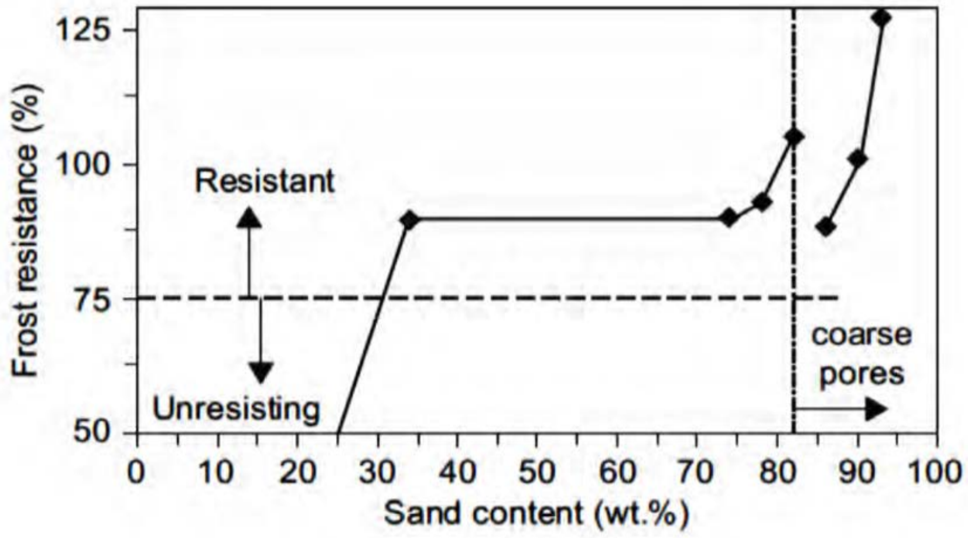


Figure 2.9. The relationship between Geopolymer composite resistance to freezing and Thawing and sand content (Steinerova, 2011).

Temuujin et al. (2014), approved using two types of calcium oxide fly ash C class of 14% and 30% where six various mixtures in terms of alkaline solution composition and percentage were used. Further, the samples were subjected to 40 freezing and thawing cycles by Method B, but the samples were immersed in water at a temperature for 6 hours before each cycle which are displayed in table 2.8.

The comparison between ordinary cement mortar and geopolymer slag and fly ash, with several percentages of alkali solution with quartz sand ranging from 0-2 mm and involvement of glass fiber, was examined by Funke et al. (2016). At the lab temperature, the samples were cured and after 28 frozen and cooled cycles, 96% compared with the conventional concrete mix of 91% were relatively dynamic modulus elasticity for the geopolymer mix.

2.6.2. Exposure to Sulfate attack

One of the major intense environmental factors affecting the long-lasting of concrete structures is the sulfate attack. It may cause scratches to expand and concrete structures to deteriorate.

Generally, the sulfate attack is the process of concrete deterioration due to the reaction between the cement and sulfates. The formation of calcium-sulfate (gypsum) or sulfuric calcium-aluminate (ettringite) as shown in equations (2 -3) and (2 – 4) will result in the creation and cracking of concrete expansion by the formation of concrete due to sulfate (Neville and Brooks, 2010).

Sulfate reactions are due to calcium hydroxide or hydrated C3A so that low C3A content like cement type V and Portland blast-furnace or Portland pozzolana is preferable to use.

$$Ca(OH)_2 + Na_2SO_4.H_2O \longrightarrow CaSO_4.2H_2O + NaOH + H_2O \qquad (2\text{-}3)$$

$$(3CaO.Al_2O_3.H_2O) + Na_2SO_4.H_2O \longrightarrow 3CaO.Al_2O_3.3CaSO_4.H_2O + Al(OH)_3 + NaOH + H_2O \qquad (2\text{-}4)$$

2.7. Fiber Reinforced Geopolymer Concrete (FRGC)

In order to increase flexural strength and energy absorption, fibers in the different forms of threads, filaments, whiskers and nanoparticles were used for reinforcing in geopolymeric composites. In presumption, the criteria of fiber as reinforcement for cement and geopolymer composites should take into consideration of three main requirements, including:

- ❖ Compatibility of with applications of materials properties.
- ❖ Sufficient fiber-matrix interaction to transmit stresses.
- ❖ Optimum look ratio for effective behaviour after cracking.

It is necessary to give an insight into the components and geometric properties of fibers in cement material before addressing the composite action of fibers and geopolymer.

2.7.1. Basalt Fibers

Nayan Rathod et al. (2013) revealed Basalt Fiber as high-quality non-metallic fiber made of high-temperature basalt stone fiber. Basalt stones, chipped basalt fiber, basalt fabrics and endless filament wire can be developed from basalt stone.

The basalt fiber is generally produced from magma and volcano coming beneath the earth crust, and they become hard when exposed to air. Basalt refers to a kind of rock that is dark grey in colour which is inserted into small nozzles to produce filament of basalt fibre. In one single production process, the basalt fibers contain no other supplements that provide an additional advantage in costs. Rock basalt fibres are non-combustible and explosion resistant to air or water. They do not cause a chemical reaction that could damage the health or the environment if in contact with other chemicals.

2.7.1.1. Physical Properties Color

Color that is available in golden brown. It can found in different diameters like 5.8 microns and three different lengths as mention 6mm,8mm,12mm, etc. The density of basalt fiber is about 2.75 g/cm^3. The coefficient of friction may be between 0.42 to 0.50 (Nayan Rathod et. al., 2013).

2.7.1.2. Chemical Properties

Basalts are more durable in strong alkalis, also significantly lower Weight loss in heating water, acid and alkali. high protection to UV- Light & biologic and fungal poisoning. Are agreeable with phenolic resins. Consumption of humidity comes to less (Nayan Rathod et. al., 2013).

2.7.1.3. Thermal Properties

The thermal properties of the basalt fiber are given below: (Nayan Rathod et al., 2013).

- Heat range from –260 ◦C to 980 ◦C and m.p of 1450◦C
- Low thermal conductivity with 0.031- 0.038w/mk and good for fire protection and insulation use.
- Cost-effective in comparison to other materials like e-glass, ceramics, stainless steel, silica
- Provides three times more thermal conductivity than asbestos with no heat issues
- When exposition to less temperature around 400 C, basalt fibers loss initial strength but it low in comparison to E-glass, which loss about 50 % more.

2.7.2. Steel Fibers

The use of steel fiber in concrete was first advocated by Porter in 1910 (Naaman, 1985). The first academic studies on fiber reinforced concrete (FRC) was carried out in the United States. SFRC is supplied with conventional hydraulic cement, raw and fine aggregates, water and steel fibres. Steel fibres are defined by an American concrete institution (ACI 544.IR 1996) as discrete, short steel lengths with an aspect ratio between L/D of 20. Superplasticizers can also be added to the concrete mix to improve the workability and stability of SFRC. The specifications for steel fibres, such as their shape, diameter, material, length and cross-section types in different way and shapes.

2.7.2.1. Steel Fibers Benefits

The benefit of the steel fiber attraction in the concrete depends on a number of factors, such as form, length, strength, category, fiber content, cross-section, S.F.s bonding strength, matrix strength, mixing design and concrete mixture. Traditional load-deflection curves for smooth concrete and the FRC. Adding S.F.s to the current R.C. members have a number of benefits, such as:

- S.F.s improve matrix tensile strength by enhancing concrete bending strength.
- The cracking mechanism of S.F.s and their tendency to uniformly distribute stress throughout the matrix ensure that cracking strength and cracks in the concrete are restrained.
- Enhance concrete ductility.

The SFRC is longer sustainable and operational than conventional (Rapoport, 2001; Grzybowski and Shah, 1990; Grzybowski, 1989).

2.7.3. Glass Fibers

Glass fiber is defined as a material with embedded reinforcing glass filaments that are produced in different types for a variety of specific applications. Glass fiber is more useful because it contains high percentages of surface area by weight, so it is highly susceptible to chemical adhesion. Due to the air trapping in glass particles, the glass-fibre blocks produce good thermal isolation with the specific temperature conductivity of 0.05 W/Mk. Glass fiber contains more than 50% of silica, and the resulting product has distinguishing properties as a compound with different metal oxides (Kasım MERMERDAS et al. 2019).

Glass fibers are manufactured from silicon oxide by adding a small number of other oxides which have good properties such as high strength, excellent resistance to temperature and corrosion at a low price. Alkali resistance 12mm glass fibers and 14 microns in nominal diameter with a density of 2680 kg / m3 Bhalchandra and Bhosle (2010) was studied in the investigation.

2.7.4. Carbon Fibers

The carbon fiber reinforces geopolymer composites that were produced with met kaolin, water glass, and short carbon fibers was mentioned by Le-ping Liu et al. (2014) which are strongly constructed, strengthened with different mass portions of short carbon fibers. In the geopolymer matrix, the external treatment consists of shorter carbon fibers. These composites exhibit outstanding mechanical characteristics and electrical conductivity. With an electrical conductivity of 10-5 to 10-1 sm-1, C.F. / geopolymer combinations reached flexural strength of 27 MPa and a carbon fiber contents of 2.78 %. But while the fiber content of carbon fiber increased by 4.15%, the composite conductivity remained constant.

3. MATERIAL and METHOD

3.1. Introduction

The chapter here is split into two parts. The first section covers primary materials used in the production. It also further deals with the physical and chemical properties of concrete, mixing ratios, method of preparing geopolymer concrete and the curing condition of the source material used.

The second part covers the types of aggressive conditions which the geopolymer concrete is exposed in accordance with the ACI 318-14 freezing and thawing requirements. It was also studied the impact on the durability of geopolymer concrete under the influence of the harsh environment of different kinds of fiber, their proportion in the mixture, the conc. of the alkaline solution and the period of exposure. The experimental study is shown in Figure (3.1.).

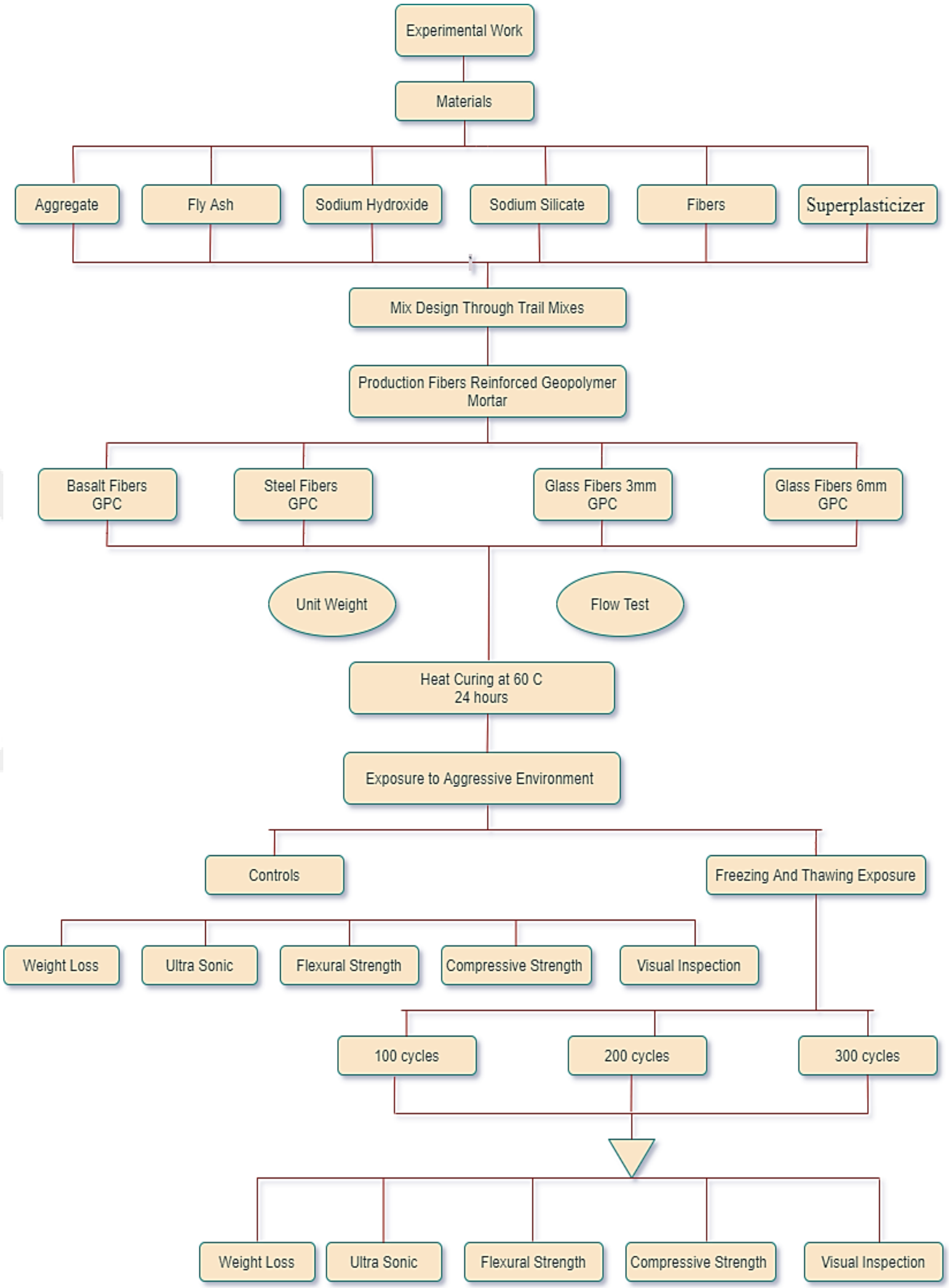


Figure 3.1. Schematic diagram of the experimental work

3.2. Materials and Their Properties

3.2.1. Fly ash

Turkish hard coal F.A. of the Catalagzi power station, Zonguldak, was used in this study. Figure 3.2. in accordance with ASTM C618-05 and ACI 226, low-calcium dry ash (ASTM Class F) reports. Table 3.1 shows the chemical composition and physical characteristics of that material as provided by the supplier.

Table 3.1. Chemical compositions and physical properties of class F fly ash

Chemical composition, %									Specific gravity
CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	SO_3	Na_2O	K_2O	LOI*	2.0
1.69	55.46	26.33	6.71	2.42	0.05	1.08	4.22	1.2	

*Loss on ignition

Figure 3.2. Fly Ash

3.2.2. Alkali solution

The second essential component after source material in Geopolymer production is the alkaline solution or alkaline activator. Two basic compounds, silicate sodium and sodium hydroxide, consist of the alkaline solution. Sodium silicate concentration should range from (38% to 55%) with SiO2 molar ratios: Na2O should be at a minimum of 97% pure within the range of 3:1 sodium hydroxide.

The NaOH solution has been selected because it is cheaper than KOH. TEKKIM, Bursa, Turkey was used for the sodium silicate solution used in this study with a chemically-based composition of Silica (SiO2) -27.1%, Na2O - 9% and water - 63.9% with modulus ratios (SiO2 / Na2O) of 3.01 with a specific gravity of 1,367 g / m^3, as illustrated in Table 3.2. NaOH flakes were used to make sodium hydroxide solution with 98 % purity dissolving in water. The NaOH solutions have been prepared with distilled water that is available in the laboratory, which released a considerable amount of warmth during the dissolution of the NaOH.

Table. 3.2. Chemical compositions and physical properties of sodium silicate (Na_2SiO_3)

Chemical composition, %				Density, g/ml
NaOH (sodium hydroxide)	Na_2CO_3 (sodium carbonate)	NaCl (sodium chloride)	Fe (iron)	2.13
≥ 98.0	≤ 0.5	≤ 0.02	≤ 0.001	

Table 3.3. Chemical compositions and physical properties of sodium hydroxide (NaOH)

Chemical composition, %		Density, g/ml	Module	Bome, °B
Na_2O (sodium oxide)	SiO_2 (silica)	1.367	2.93	38.68
9.03	27.08			

The weight of NaOH flakes in solution varies according to the desired molarity (8, 10, 12, 14, 16 and 18 M) concentration of the solution with NaOH. For example, the 12 molarity commonly used NaOH solution consisted of 480 g NaOH per litre of the solution. For all mixtures, the ratio of silicate and sodium hydroxide mass was 1:3. The alkaline fluid mixture was subjected to a 24-hour waiting period after mixing with sodium silicate and NaOH solutions using a stirrer and mixing equipment to be used in the geopolymer paste preparation.

3.2.3 Aggregate

The natural river sand with particle sizes of less than 4 mm and a specific gravity of 2.65 that was determined concerning ASTM C127 was utilized as fine aggregate in the geopolymer composite manufacturing. Table 3.4. depicts the sieve analysis results of the natural river sand used in this study.

Table 3.4. Gradation and specific gravity of natural river sand

Fine aggregate						Specific gravity
Sieve size, mm	0.25	0.50	1.0	2.0	4.0	2.65
Percent passing, %	15	28	48	79	95	

3.2.4 High- Range Water Reduce

Polycarboxylic ether-based superplasticizer having the commercial name of MGlenium 51 with the specific gravity value of 1.07 was used to achieve the desired consistency for the geopolymer composite batches in the fresh state. A fixed superplasticizer content of 5% of fly ash dosage by mass was considered in the manufacturing of all geopolymer composite batches.

Figure 3.3. Superplasticizer

3.2.5 Fibers

Apart from the binding material, alkaline activator, aggregate, and superplasticizer, in this study, four different fibers were used in the geopolymer composite manufacturing. The fibers employed in the current study were the basalt fiber (BF) with a length of 3 mm, the brass coated steel fiber (SF) with a length of 6 mm, two glass fiber types with lengths of 3 (GF-3) and 6 (GF-6) mm. The length, diameter and specific gravity values of the fibers are presented in Table 5 and their photographic views are shown in Figure 3.4.

Table 3.5. Physical properties of fibers

Fiber type	Length, mm	Diameter, mm	Specific gravity
Basalt fiber	3	0.014	2.78
Steel fiber	6	0.160	7.85
Glass fiber (3 mm)	3	0.016	2.67
Glass fiber (6 mm)	6	0.016	2.60

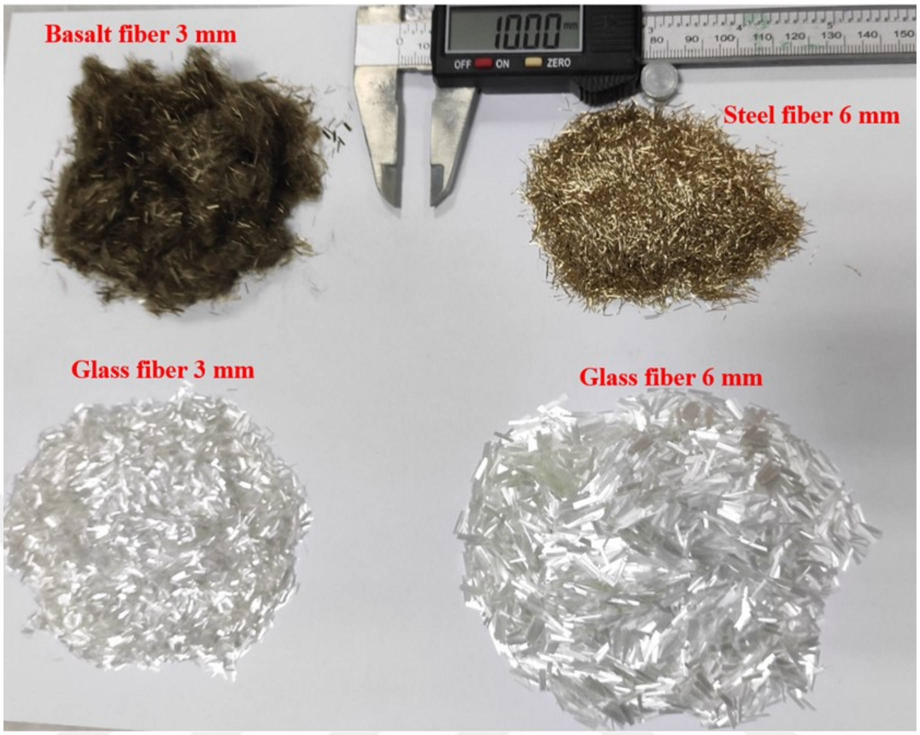


Figure 3.4. Fibers employed in the current study: basalt fiber, steel fiber, 3-mm glass fiber, and mm glass fiber

3.3 Details of mix design and production process

The geopolymer composite batches were designed and produced at the constant alkaline activator-to-binding material (solid binding component) ratio of 0.5. Here, the solid binding component was the fly ash with a fixed dosage of 550 kg/m^3 while the alkaline activator consisted of prefabricated Na_2SiO_3 and 12 M NaOH solutions. As also mentioned before, the Na_2SiO_3-to-NaOH ratio was designated as 3:1, with a total alkaline activator content of 275 kg/m^3. Besides, four fiber types were incorporated into the geopolymer composite batches with the volume fractions of 0, 0.4, 0.8, and 1.2% by the total volume of mortar. Thereby, a total of 13 different fly ash-based geopolymer mixtures of which one is the control (fiberless) batch and the remaining were fibrous batches were designed. Table 3.6. thoroughly depicts the proportions of the ingredients of the fly ash-based fibrous geopolymer composite batches.

Table 3.6. Batch proportions for the fibrous geopolymer composites (kg/m^3)

Batch ID	Fly ash	NaOH	Na_2SiO_3	Natural sand	Fiber				SP*
					Basalt	Steel	Glass		
							3 mm	6 mm	
Control	550	68.75	206.25	1423.7	0	0	0	0	27.5
BF|0.4	550	68.75	206.25	1413.2	11.12	0	0	0	27.5
BF|0.8	550	68.75	206.25	1402.6	22.24	0	0	0	27.5
BF|1.2	550	68.75	206.25	1392.0	33.36	0	0	0	27.5
SF|0.4	550	68.75	206.25	1413.2	0	31.40	0	0	27.5
SF|0.8	550	68.75	206.25	1402.6	0	62.80	0	0	27.5
SF|1.2	550	68.75	206.25	1392.0	0	94.20	0	0	27.5
GF-3|0.4	550	68.75	206.25	1413.2	0	0	10.68	0	27.5
GF-3|0.8	550	68.75	206.25	1402.6	0	0	21.36	0	27.5
GF-3|1.2	550	68.75	206.25	1392.0	0	0	32.04	0	27.5
GF-6|0.4	550	68.75	206.25	1413.2	0	0	0	10.40	27.5
GF-6|0.8	550	68.75	206.25	1402.6	0	0	0	20.80	27.5
GF-6|1.2	550	68.75	206.25	1392.0	0	0	0	31.20	27.5

*SP: superplasticizer

3.3.1 Mixing process

The manufacturing process of the geopolymer composite begins with mixing the fine aggregate and fly ash in the mixer pan by rotation for about half a minute in order to achieve a homogeneous aggregate-fly ash mixture. Once this step ends, almost half of the alkaline activator solution is poured into the mixer pan where is the homogeneously mixed aggregate-fly ash materials, immediately afterwards, the pan is let to revolve for another one minute. In the meanwhile, the remain alkaline activator solution was blended with the superplasticizer and after the mixer pan has stopped, this liquid mixture is added to the mixer. Thereafter, the mixer pan is rotated for about extra three minutes, subsequently, the freshly mixed ingredients are left to rest for two minutes. Then, the manufacturing process for the plain batch is completed by stirring the rested mixture for two more minutes to achieve the fresh geopolymer composite batch. However, for the fibrous batches, the fiber is strewn on the freshly mixed ingredients prior to resting the mixture, following this, the mixture is left to rest for two minutes and then the manufacturing is completed by mixing the mixture for two more minutes to obtain the fresh fibrous geopolymer composite batch.

3.3.2 Molded and Curing process

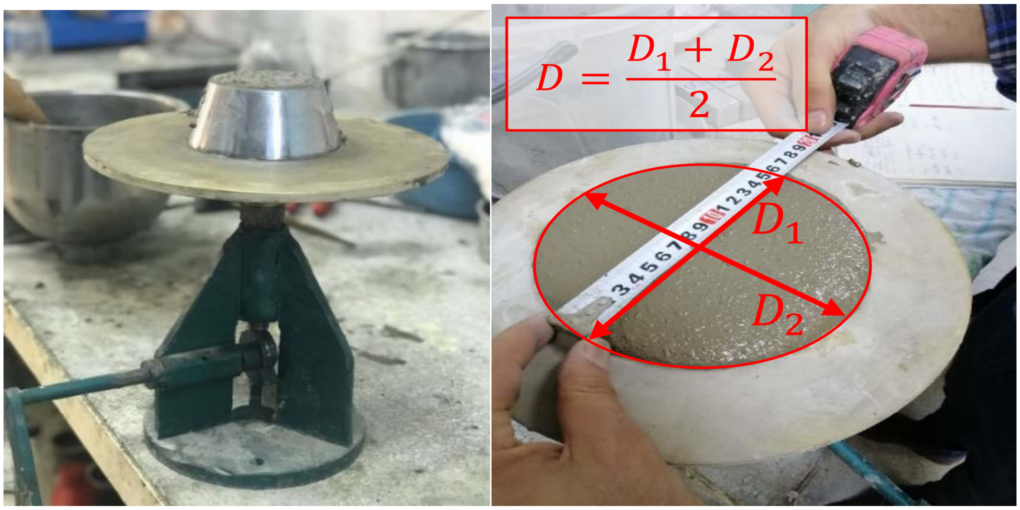
Immediately after the manufacturing process was completed, the workability test was conducted, and then, the fly ash-based fibrous geopolymer composite was poured into the molds as two layers which were individually compacted by rodding and vibration table. In total, twelve prismatic samples with dimensions of 40x40x160 mm were prepared for the hardened state tests. The samples covered with a plastic sheet were put into the oven having a temperature of 65 °C and kept there for one day. After the samples were taken out from the oven, they were demoulded and for 7 days, left in the laboratory having a temperature of 23±2 °C.

Figure 3.5. The mix preparation process

3.4 Test methods

3.4.1 Flow test and fresh unit weight

In this study, the flow table test was carried out to determine the flowability of the fibrous geopolymer composite batches by following the ASTM C1437. The flow table test was performed by employing a conical mould having the top and bottom opening diameters and the height of 70, 100, and 50 mm, respectively. The mould was filled with the fresh fibrous geopolymer composite as two equal layers that were separately compacted by 20 tamping. Thereafter, the top surface of the mortar in the mould was finished by using a trowel, as shown in Figure 3.6 a. One minute later after filling the conical mould, it was removed, and then in order to spread the fresh fibrous mortar as indicated in Figure 3.6 b, the table was rammed 25 times within 15 seconds. In the end, the flow diameter value was determined by taking the average of two perpendicular diameters measured on the sprawled fibrous geopolymer composite. Also, the fresh unit weight of the fibrous geopolymer composites was computed according to the ASTM C138.



(a) (b)

Figure 3.6. (a) flow table testing instrument (filled with fibrous geopolymer composite), and (b)measurement of the flow diameter of the fibrous geopolymer composite

3.4.2 Flexural and compressive strength tests before and after exposure freezing and thawing

Three 40-mm cubic samples were used to conduct the compressive strength test by following the ASTM C109 and three 40x40x160-mm prismatic samples were used to perform the flexural strength test in accordance with the ASTM C348. The ASTM C597 was followed to carry out the ultrasonic pulse velocity test of the fibrous geopolymer composite and on the other hand, the freeze-thaw resistance of the fibrous geopolymer composite was determined by following the ASTM C666.

The 40x40x160-mm prismatic samples were subjected to the freeze-thaw cycles, in which one cycle was between the temperature of -17.7 and 4.4 °C. The machine applying the freeze-thaw cycles is presented in Figure 3a and the samples subjected to these cycles are demonstrated in Figure 3b. Besides, the influence of the freeze-thaw cycles on the appearance of the fibrous geopolymer composite was designated pursuant to the visual inspection that constitutes from a series of professionally taken photographs.

(a) (b)

Figure 3.7. Photographic display of freeze-thaw testing: (a) cabin and (b) sampl

4. RESULTS and DISCUSSIONS

4.1. Introduction

In this chapter, the results obtained from experimental work were analyzed. The chapter is divided into three parts as follows:

1- The first part is the results of the GPCs during mix preparation for all mixes.
2- The second part compares the resistance of different types of GPCs exposed to different levels of freezing and thawing cycles.

Last part includes visual inspection.

4.2. Effect of fibres on fresh properties

The workability of the fly ash-based geopolymer composite was determined through the flow diameter attained from performing the flow table test. The variation in the flow diameters of the geopolymer composites in regard to the fiber type and volume fraction is demonstrated in Table 4.1. and Figure 4.1. The flow diameter for the control batch was measured as 185 mm that implies 85% flowing. A reduction in flow ability of the geopolymer composite was observed when the fiber was added. Putting the BF and SF in the geopolymer composite at 0.4% volume fraction caused an 8 and 10% reduction in the flow diameter, respectively, whereas adding GF-3 and GF-6 decreased the flow diameter values about the rates of 24 and 22%, respectively. Hence, increasing the fiber volume fraction systematically decreased the flow diameter of the geopolymer composites. At all volume fraction levels, the highest reductions were observed in the geopolymer composite batches manufactured with both types of glass fiber. The flowing diameter values for the GF-3-1.2 and GF-6-1.2 named batches were 110 and 115 mm, respectively, while that for the BF-1.2 and SF-1.2 named batches were 140 and 150 mm, respectively. So, it can be stated that the geopolymer composites produced at a 1.2% volume fraction of GF have stiff and adherent consistency than those produced with BF and SF. On the other hand, in all batches, a glossy and cohesive appearance that was because of the existence of the sodium silicate in the alkaline solution was observed.

Table 4.1. Effect of fiber type and volume fraction on the workability in terms of flow diameter value

Fibres Types	Flow %			
	0%	0.40%	0.80%	1.20%
Basalt fibres	85	70	55	40
Steel fibres	85	65	60	50
Glass fibres 3mm	85	40	20	10
Glass fibres 6mm	85	45	20	15

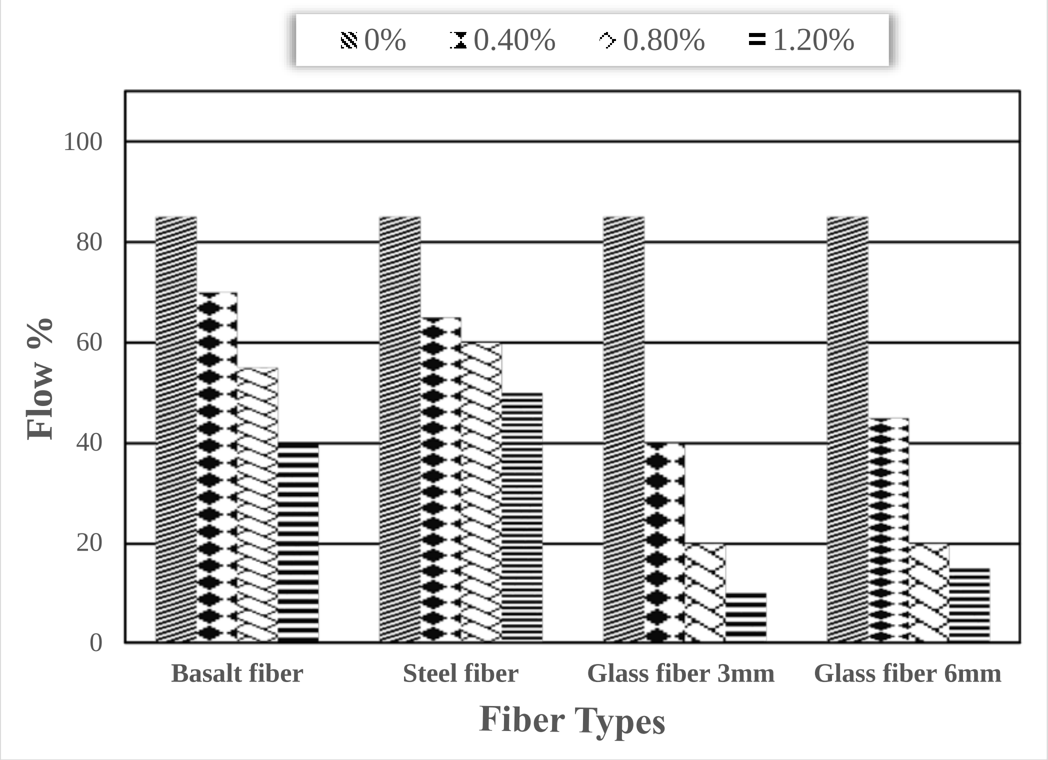


Figure 4.1. Effect of fiber type and volume fraction on the workability in terms of flow diameter value

The effect of fiber content on the workability of the geopolymer composites was also examined by Ganes and Muthukannan (2020). They used 6-mm GF in the manufacturing of the geopolymer concrete at the volume fractions of 0.25, 0.50, 0.75, 1.0, and 1.25%. They determined the influence of fiber on the workability feature of the geopolymer concrete by performing the compaction factor test. In the study conducted by Ganes and Muthukannan (2020), it was stated that there is a strong relationship between the fiber volume fraction and the workability of the geopolymer concrete which increasing the fiber volume fraction from 0 to 1.25% caused a decrease in the workability of more than 10%. Besides, they reported that adding GF more than a volume fraction of 1% causes an agglomeration of fibers, thus inclining a diminish in the workability. Another reason lying under this effect of fiber addition is the high surface area that the fiber has. In other words, some part of the alkaline solutions may assign to provide wetting of the fibers, thus decreasing the processability and flowability. A similar effect of fiber addition was reported by other researchers. Mulapeer et al. (2019) studied the effect of BF and SF addition on the workability characteristics of geopolymer composite by means of the flow table test. They reported that adding both BF and SF adversely exerts an influence on the flow diameter of the geopolymer composites. At the fiber volume fractions of 0.2 and 0.4%, both fiber types have almost the same reducing rate on the flowability, however, at higher fiber volume fraction levels, BF addition much more negatively affected the flow diameter than the SF addition.

4.3. Effect of fiber on strength and ultrasonic pulse velocity

Figure 4.2. depicts the influence of fiber type and volume fraction on the compressive strength of the fly ash-based geopolymer composite. The results indicated that the control geopolymer composite batch, namely the fibreless, has a compressive strength of 23.3 MPa. Adding fibers at the volume fraction of 0.4% provided a partial enhancement in the compressive strength. The best enhancement was observed when SF was added while a slight increase was seen in the case of adding BF and GF-3 types. SF and GF-6 type fibers incorporation at the volume fraction of 0.4% provided about 22 and

18% enhancement in the compressive strength while the enhancement rates were about 5 and 3% when BF and GF-3 type fibers were added. The best results for all volume fractions were achieved in the fibrous geopolymer composites manufactured with SF. Increasing SF volume fraction from 0 to 1.2% led to a remarkable increase in the compressive strength. The compressive strength for the mortar batch manufactured with SF at the volume fraction of 1.2% was 33.4 MPa. A similar trend with a lower impact was observed when the BF was used in the manufacturing of the fibrous geopolymer composite. Increasing the volume fraction of BF resulted in a systematical augmentation of the compressive strength of the geopolymer composite, as shown in Figure 4.2. On the other hand, incorporating GF-6 type fiber more than 0.4% volume fraction caused the decreasing of the compressive strength whereas adding GF-3 type fiber reasoned the compressive strength diminishing after 0.8% volume fraction. The compressive strength values of the fibrous geopolymer composites involving the GF-6 type fiber at the volume fractions of 0.8 and 1.2% and the GF-3 type fiber at the volume fraction of 1.2% were less than that of the control geopolymer composite.

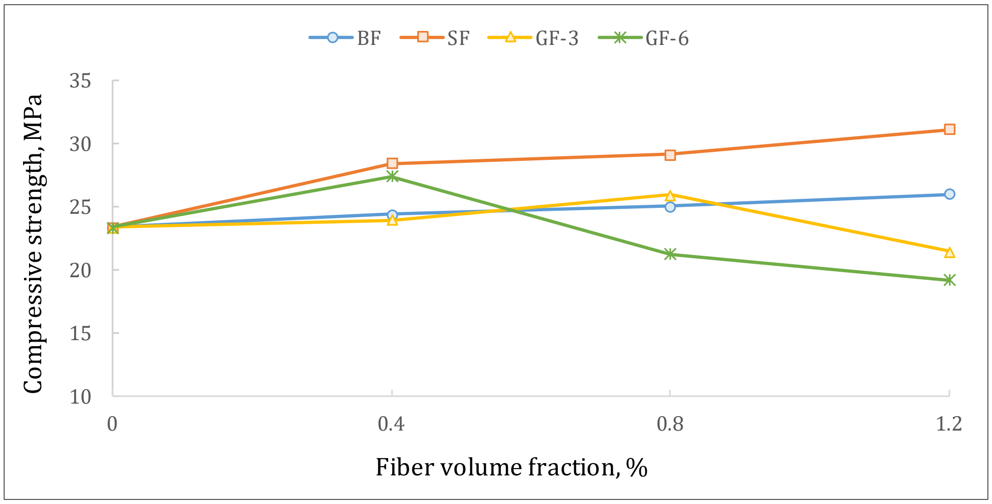


Figure 4.2. Variation in the compressive strength of fly ash-based geopolymer composite in regard to the fiber volume fraction

Ali et al. (2020) investigated the influence of the BF type fiber with two different lengths of 12 and 24 mm on the compressive strength and reported that adding BF to the geopolymer composites increases the compressive strength. Besides, in the study of Ganesh and Muthukannan (2020), it was stated that adding GF type fiber more than a 1.0% volume fraction causes a decrease in the compressive strength. On the other hand, Çelik et al. (2018) incorporated 12-mm BF in the production of the metakaolin-based geopolymer composites at the volume fractions of 0.4, 0.8, and 1.2%. They observed a continuous, but slight increase in the compressive strength of the mortar mixtures by augmenting the BF volume fraction from 0 to 1.2%. Ganesan et al. (2015) investigated the effect of hooked-end SF on the compressive strength of the geopolymer concrete. They designated the volume fractions of 0.25, 0.5, 0.75, and 1% and observed a continuous compressive strength gain by increasing the fiber volume fraction. However, Islam et al. (2017) also employed hooked-end SF at the volume fraction of 0.5% in the production of lightweight geopolymer concrete and observed a slight reduction in the compressive strength of the geopolymer concretes. Moreover, Mulapeer et al. (2019) also studied the effect of BF and brass coated SF on the geopolymer composite. They designated five different fiber volume fractions of 0.2, 0.4, 0.6, 0.8, and 1.0%, and their compressive strength results revealed that BF and SF addition first decreased the compressive strength and then increased it. The decrease due to SF incorporation was more than that owing to BF addition. After the BF and SF volume fractions of 0.4%, an increment in the compressive strength of the geopolymer composite was observed and the greatest compressive strength values were achieved in the case of fiber addition at a volume fraction of 1.2%.

Here, another significant feature of the geopolymer composite is the flexural strength. Figure 4.3. indicates the variation in the flexural strength of fly ash-based geopolymer composite in regard to the fiber volume fraction. The flexural strength of the control geopolymer composite was about 6.9 MPa and a similar influence of the fiber addition was also seen in the flexural strength test. Incorporating fiber at the volume fraction of 0.4% enhanced the flexural strength with various rates depending on the fiber type. The highest increase in the flexural strength was observed in the case of SF addition. After 0.4% volume fraction level, a reduction in the flexural strength was seen for the fibrous geopolymer composite produced with the GF-3 and GF-6 type fibers, whereas a continuous strength gain was achieved when the geopolymer composite manufactured with the BF type fiber. Although the best enhancement in the flexural strength was attained from the fibrous geopolymer composite produced with the SF type fiber, there was a slight reduction in the flexural strength when the SF was added more than the volume fraction of 0.8%, but the flexural strength value after this volume fraction level was still higher than the flexural strength of the control geopolymer composite. SF incorporation conduced to increase the flexural strength of more than 20% while BF augmented the flexural strength at the fiber volume fractions of 0.4, 0.8, and 1.2% as about 5%, 8%, and 14%, respectively.

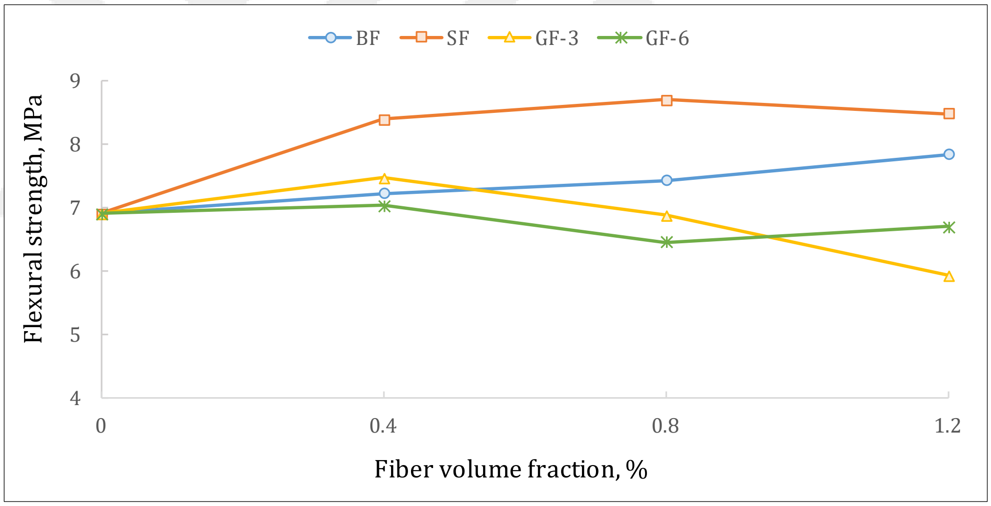


Figure 4.3. Variation in the flexural strength of fly ash-based geopolymer composite in regard to the fiber volume fraction

Mulapeer et al. (2019) achieved a gradual and continuous flexural strength gain by increasing the BF and SF volume fraction. Ali et al. (2020) reported that utilizing BF in geopolymer composite manufacturing increased flexural strength. Guo and Xiong (2020) also found that the geopolymer composite containing SF type fiber indicates better flexural performance than that involving BF type fiber. On the other hand, Al-mashhadani (2018) expressed that adding and increasing the volume fraction of polypropylene and polyvinyl alcohol type fibers do not significantly exert an influence on the flexural strength as much as the SF type fiber. Besides, Ganesh and Muthukannan (2020) observed that increasing the GF volume fraction enhances the flexural strength of the geopolymer composite till 1.0% volume fraction level, however, after this level, there is a diminishing in the flexural strength.

As a consequence, in light of the aforementioned finding and comparison, it can be concluded that in literature, there is a common decision about the impact of fiber type and volume fraction on the compressive strength, but more studies are required to generalize the fiber effect on the geopolymer composites.

The ultrasonic pulse velocity values achieved in this study are indicated in Figure 4.4. The ultrasonic pulse velocity value of the fibreless geopolymer composite was about 2840 m/s and there is an increasing tendency of ultrasonic pulse velocity values by increasing the fiber volume fraction, as can be seen in Figure 4.4. The most remarkable effect was observed when the fiber was incorporated at the volume fraction of 0.4%. Increasing the volume fraction of the fibers after this level has an inconsiderable influence on the ultrasonic pulse velocity values of the geopolymer composites, except GF-6 type fiber. The main objective of applying the ultrasonic pulse velocity test was to determine the continuity and homogeneity of the fibrous geopolymer composites. When the results presented in Figure 4.4. was observed, it can be comprehended that the continuity, compactness, and homogeneity of the geopolymer composite batches were not particularly affected by the fiber incorporation. In the literature, many studies announced similar findings of the effect of fiber on the ultrasonic pulse velocity (Ali et al., 2020; Guo and Giong et al., 2020; Çelik et al., 2018; Al-Mashhadani et al., 2018).

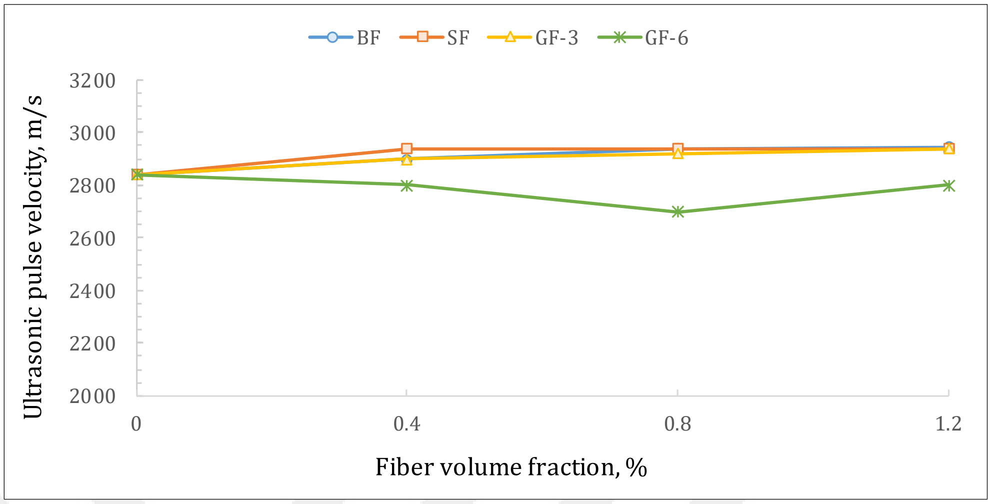


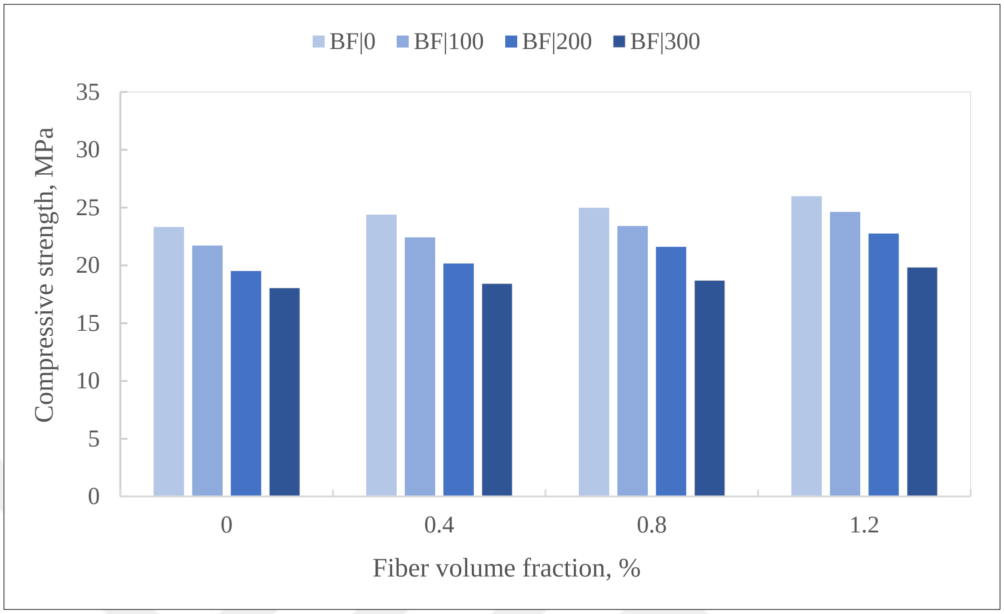
Figure 4.4. Variation in the ultrasonic pulse velocity of fly ash-based geopolymer composite regarding the fiber volume fraction

4.4. Effect of the freeze-thaw cycle on strength and ultrasonic pulse velocity

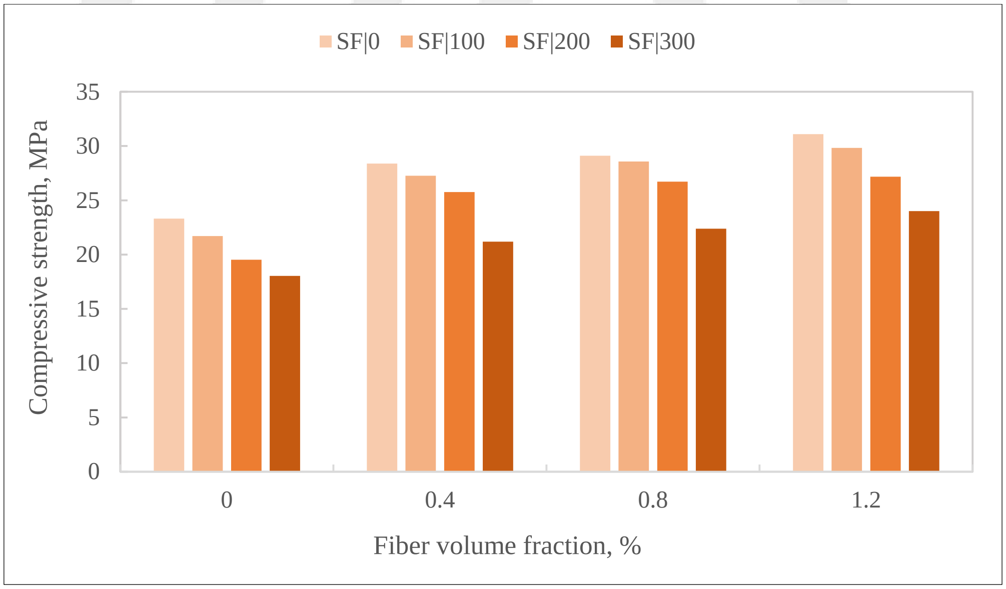
To display the influence of freeze-thaw cycles on the compressive strength of fibrous geopolymer composite, Table 4.2. and Figures 4.5. (a-d) are presented. The results revealed that applying freeze-thaw cycles to the fibrous geopolymer composites decreases the compressive strength. The compressive strength of the control geopolymer composite was about 23.3 MPa, applying 100 freeze-thaw cycles to this mortar batch resulted in a 7% decrease in the compressive strength while about 16 and 23% reduction in the compressive strength was observed when the mortar subjected to 200 and 300 freeze-thaw cycles. Adding the SF with the volume fraction of 0.4% significantly decreased the compressive strength reduction associated with applying the freeze-thaw cycles, whereas, in this manner, the benefit of the other type of fiber was observed when they were added at the volume fraction of more than 0.4%. In fact, adding BS, GF-3, and GF-6 type fibers at the volume fraction of 0.4% increased the compressive strength reduction caused by freeze-thaw cycles. Ali et al. (2020) employed the BF at the volume fraction of 1% in geopolymer composite manufacturing and stated that adding BF decreased the strength loss due to the freeze-thaw effect. Yunsheng et al. (2008) reported that incorporating the fiber in the geopolymer production is an effective way for enhancing the freeze-thaw durability. The main reason behind this scenario is that the fiber particles counterbalances the internal stresses that are caused by the freezing of the water. The freeze-thaw resistance of the coal bottom ash-based geopolymers and observed about a 7% reduction in the strength when it was subjected to the 30 freeze-thaw cycles.

Table 4.2. Influence of freeze-thaw cycle on the compressive strength of the fibrous geopolymer composites

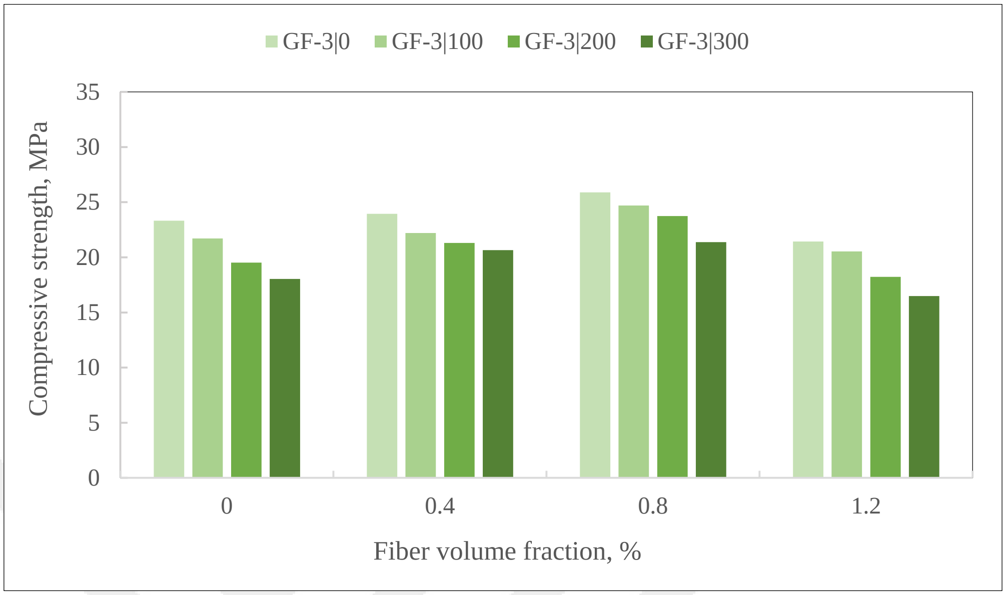
Fibres Content %	Freeze and thaw Cycles	BF	SF	GF-3	GF-6
0	0	23.32	23.32	23.32	23.32
	100	21.71	21.71	21.71	21.71
	200	19.52	19.52	19.52	19.52
	300	18.04	18.04	18.04	18.04
0.4	0	24.39	28.39	23.95	27.41
	100	22.42	27.27	22.2	24.4
	200	20.16	25.76	21.3	20.41
	300	18.42	21.19	20.65	19.13
0.8	0	24.98	29.11	25.89	21.26
	100	23.41	28.57	24.7	20.01
	200	21.6	26.72	23.74	19.28
	300	18.69	22.39	21.38	18.4
1.2	0	25.98	31.1	21.43	19.15
	100	24.63	29.83	20.53	18.7
	200	22.75	27.18	18.23	17.12
	300	19.83	24	16.49	16.9


BF|0
BF|100
BF|200
BF|300
Compressive strength, MPa
35
30
25
20
15
10
5
0
0
0.4
0.8
1.2
Fiber volume fraction, %

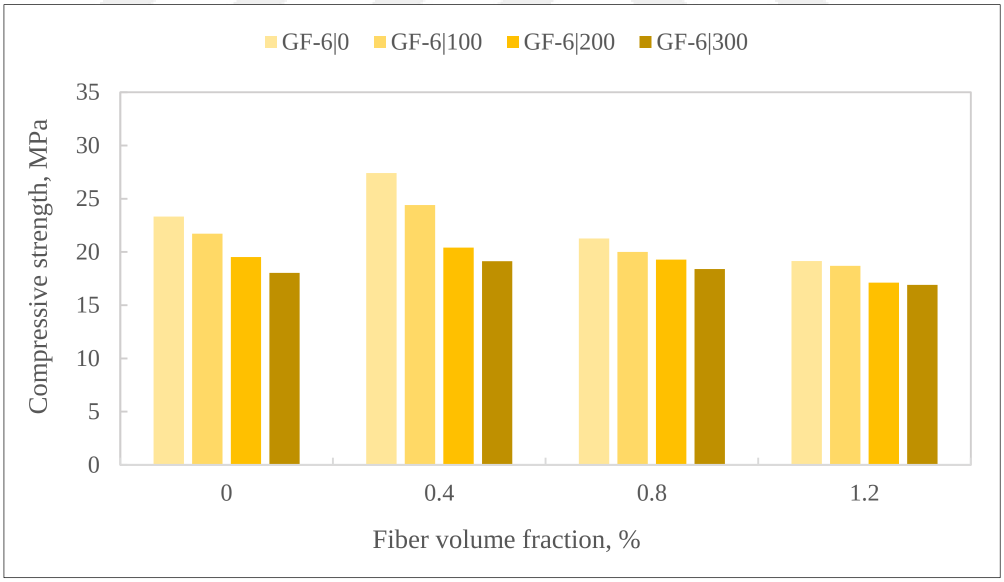
(a) BF


SF|0
SF|100
SF|200
SF|300
Compressive strength, MPa
35
30
25
20
15
10
5
0
0
0.4
0.8
1.2
Fiber volume fraction, %

(b) SF



(c) GF-3mm



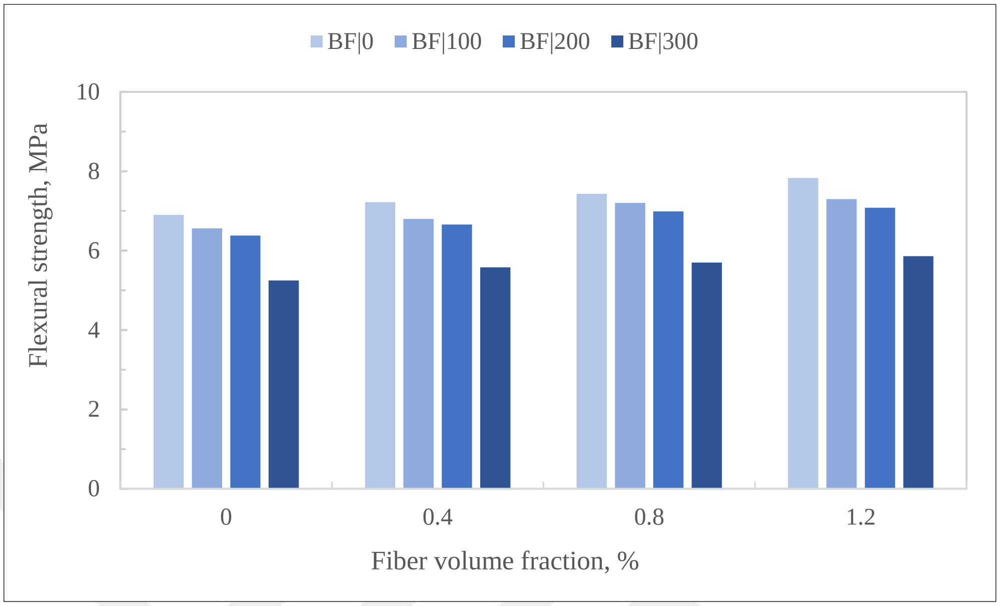
(d) GF-6mm

Figure 4.5. Influence of freeze-thaw cycle on the compressive strength of the fibrous geopolymer Mortars (a) basalt fiber. (b) steel fiber. (c) glass fiber-3mm. (d) glass fiber-6mm.

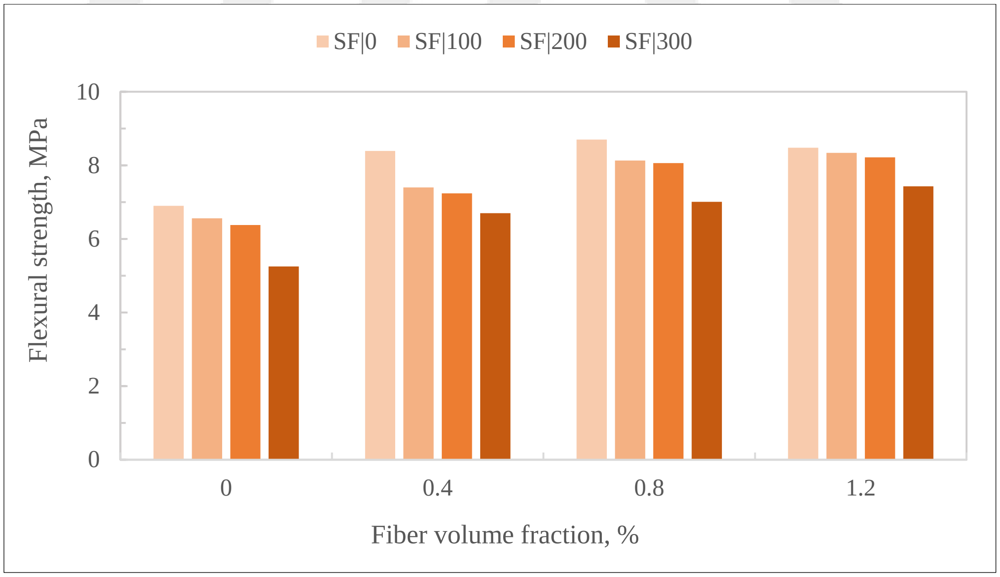
Table 4.3. and Figures 4.6. (a-d) depicts the effect of the freeze-thaw cycles on the flexural strength of the geopolymer composite. The control geopolymer composite had a flexural strength of 6.9 MPa. A gradual loss in the flexural strength was seen when 100, 200, and 300 freeze-thaw cycles were subjected to the samples of the control geopolymer composite. When 100 freeze-thaw cycles were applied to the control batch, a 5% decrease in the flexural strength was observed while the decrement rates were, respectively, about 8 and 24% in the case of the 200 and 300 freeze-thaw cycles application. For all geopolymer composite batches, the highest damage of the freeze-thaw cycles in terms of the strength loss was observed when the samples were exposed to 300 cycles. However, the results revealed that reinforcing the geopolymer composite by incorporating the fiber increases the resistance to freeze-thaw. Although a positive influence of the fiber addition was observed, no systematical relationship between the fiber volume fraction and freeze-thaw damage was achieved. In the literature, a number of studies have reported similar findings of the effect of fiber utilization in geopolymer composite production on freeze-thaw durability (Ali et al., 2020; Çelik et al., 2018; Zhao et al., 2019; Yunsheng et al., 2008).

Table 4.3. Influence of freeze-thaw cycle on the flexural strength of the fibrous geopolymer composites

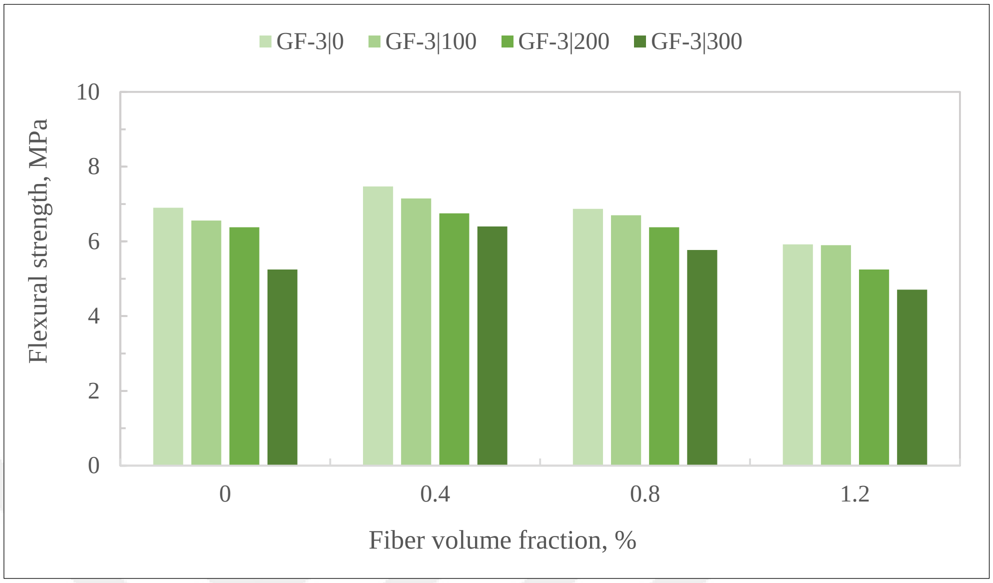
Fibres Content %	Freeze and thaw Cycles	BF	SF	GF-3	GF-6
0	0	6.9	6.9	6.9	6.9
	100	6.56	6.56	6.56	6.56
	200	6.38	6.38	6.38	6.38
	300	5.25	5.25	5.25	5.25
0.4	0	7.22	8.39	7.47	7.03
	100	6.8	7.4	7.15	6.59
	200	6.66	7.24	6.75	6.05
	300	5.58	6.7	6.4	5.84
0.8	0	7.43	8.7	6.87	6.45
	100	7.2	8.13	6.7	6.21
	200	6.99	8.06	6.38	5.55
	300	5.7	7.01	5.77	5.41
1.2	0	7.83	8.48	5.92	6.7
	100	7.3	8.34	5.9	6.38
	200	7.08	8.22	5.25	6.05
	300	5.86	7.43	4.71	4.95


BF|0
BF|100
BF|200
BF|300
Flexural strength, MPa
10
8
6
4
2
0
0
0.4
0.8
1.2
Fiber volume fraction, %

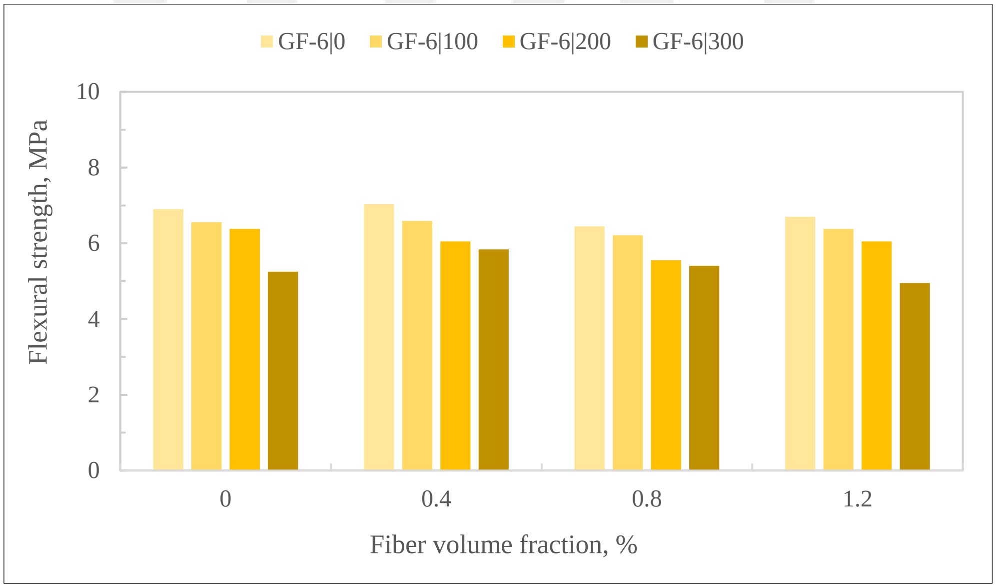
(a) BF


SF|0
SF|100
SF|200
SF|300
Flexural strength, MPa
10
8
6
4
2
0
0
0.4
0.8
1.2
Fiber volume fraction, %

(b) SF



(c) GF-3mm



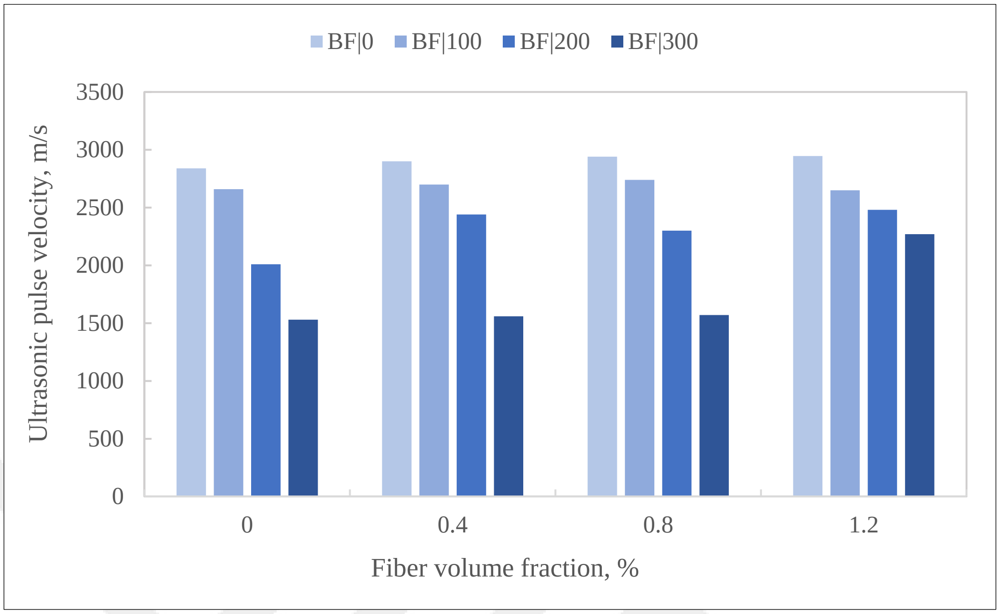
(d) GF-6mm

Figure 4.6. Influence of freeze-thaw cycle on the flexural strength of the fibrous geopolymer composites (a) basalt fiber. (b) steel fiber. (c) glass fiber-3mm. (d) glass fiber-6mm.

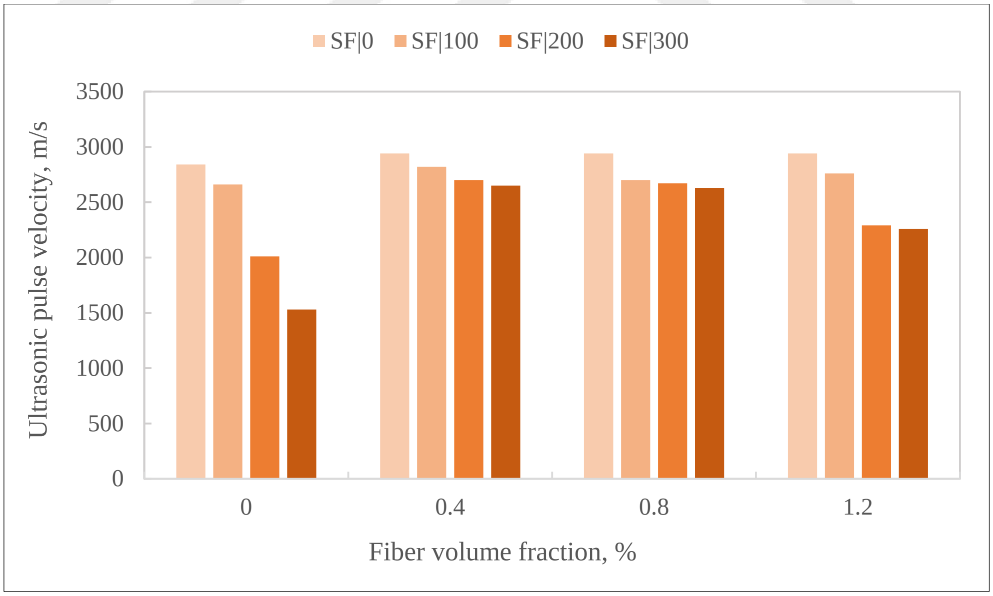
The results of ultrasonic pulse velocity values of the fibrous geopolymer composites exposed to the freeze-thaw cycles are indicated in Table 4.4. and Figures 4.7. (a-d). The results indicated that in the control geopolymer composite, applying 100 freeze-thaw cycles results in a slight diminish in the ultrasonic pulse velocity, however, there is a remarkable decrease in the ultrasonic pulse velocity values after 200 freeze-thaw cycles. Almost a 45% reduction in the ultrasonic pulse velocity was observed when the control geopolymer composite batch was subjected to 300 freeze-thaw cycles. Figure 10a depicts that the BF addition is effective to decrease the reduction in the ultrasonic pulse velocity at the 1.2% volume fraction for all freeze-thaw cycle levels while incorporating SF has a significantly positive effect on the ultrasonic pulse velocity of the geopolymer composite at even 0.4% volume fraction, as indicated in Figure 10b. On the other hand, the GF-3 and GF-6 addition also reduced the decrease in the ultrasonic pulse velocity of the geopolymer composite exposed to freeze-thaw cycles, as can be seen in Figures 10c and 10d, respectively. However, using the SF at the volume fraction of 1.2% has not positive effect to reduce the decrease in ultrasonic pulse velocity due to the applying freeze-thaw cycles as much as using it at the volume fraction of 0.4 and 0.8%, as demonstrated in Figure 10b. Ali et al. (2020) attributed the reduction in both strengths and ultrasonic pulse velocity to the micro-crack occurrences in the geopolymer matrix that was seen from analyzing the SEM images.

Table 4.4. Influence of freeze-thaw cycle on the compressive strength of the fibrous geopolymer composites

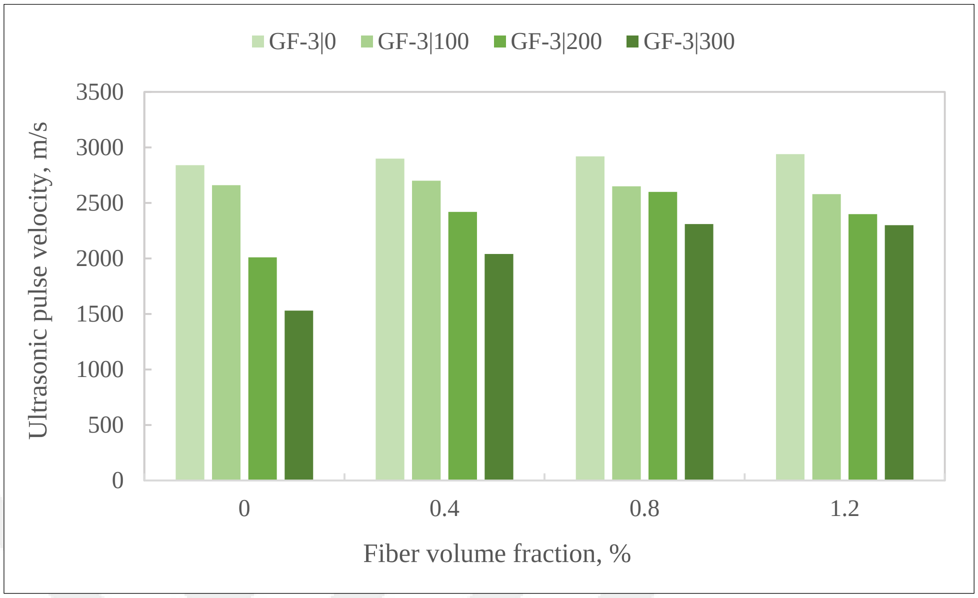
Fibres Content %	Freeze and thaw Cycles	BF	SF	GF-3	GF-6
0	0	2840	2840	2840	2840
	100	2660	2660	2660	2660
	200	2010	2010	2010	2010
	300	1530	1530	1530	1530
0.4	0	2900	2940	2900	2800
	100	2700	2820	2700	2600
	200	2440	2700	2420	2450
	300	1560	2650	2040	2310
0.8	0	2940	2940	2920	2700
	100	2740	2700	2650	2560
	200	2300	2670	2600	2420
	300	1570	2630	2310	2380
1.2	0	2946	2940	2940	2800
	100	2650	2760	2580	2530
	200	2480	2290	2400	2310
	300	2270	2260	2300	2260


BF|0
BF|100
BF|200
BF|300
Ultrasonic pulse velocity, m/s
3500
3000
2500
2000
1500
1000
500
0
0
0.4
0.8
1.2
Fiber volume fraction, %

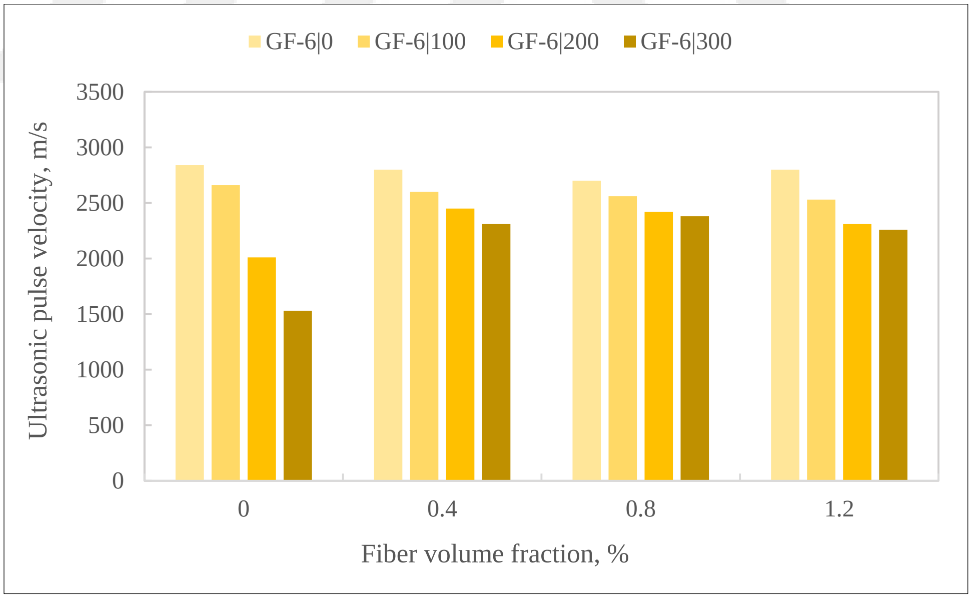
(a) BF


SF|0
SF|100
SF|200
SF|300
Ultrasonic pulse velocity, m/s
3500
3000
2500
2000
1500
1000
500
0
0
0.4
0.8
1.2
Fiber volume fraction, %

(b) SF



(c) GF-3mm



(d) GF-6mm

Figure 4.7. Influence of freeze-thaw cycle on the compressive strength of the fibrous geopolymer composites (a) basalt fiber. (b) steel fiber. (c) glass fiber-3mm. (d) glass fiber-6mm.

4.5. Effect of fiber on freeze-thaw durability

The geopolymer composites indicate a good performance against the freeze-thaw cycles since its matrix has a compact structure creating a good adhesion level. The freeze-thaw cycles directly exert an influence on the matrix by exerting hydraulic pressure which occurs due to the freezing of water that expands about 9% by volume during the frozen (Pilehvar et al., 2019). The irregular and nonhomogeneous pores in the matrix can compensate for these hydraulic pressures to certain levels, however, when, these pores are filled up, the matrix may suffer from the internal pressures. As soon as the pressure level exceeds the stress of the matrix, the micro-cracks may occur and the propagation of these micro-cracks and connecting to each other may result in deterioration (Allahverdi et al., 2014; Basheer et al., 2001). As a consequence, the spalling of the geopolymer composite starts. Here, Figure 4.8. exhibits the variation of the percent weight loss in the fibrous geopolymer composites after subjecting the designated freeze-thaw cycles. The results exhibited that when the fibrous geopolymer composites are subjected to 100 freeze-thaw cycles, weight losses are less than 0.5% almost in all mortar batches. The addition of the SF at the volume fractions of 0.4 and 0.8% decreased the weight losses of the geopolymer composites, however, no remarkable impact of adding the SF at the volume fraction of 1.2% was observed. Besides, employing the BF and GF type fibers in geopolymer composite manufacturing had a tendency to increase weight loss. This may be caused by the filling ability of the BS and GF type fibers the micropores of the geopolymeric matrix. Thus the compensation of the hydraulic pressures that occurred due to freezing of the water is being prevented. Despite the negative effect of BS and GF types of the fibers on the weight loss in the geopolymer composite, it can be stated that the fibrous geopolymer composites manufactured in this study are the mortars durable against the freeze-thaw since the total weight loss in these mortars after subjecting the 300 freeze-thaw cycles are less than 2.5%, as can be seen from Figures 4.8. and 4.9. Here, Figure 12 depicts the influence of the fiber type and volume fraction on the normalized weight loss values that were determined by dividing the weight measured after the 300 cycles by the original weight. It can be seen that the weight loss in the control geopolymer composite is a little more than 1%, whereas the weight losses in the geopolymer composites containing the SF at the volume fractions of 0.4 and 0.8% are less than that of the control samples. Meanwhile, adding the BF and GF type fibers at the volume fraction of 0.4% results in almost the same weight loss values as the control samples, however, employing these fibers more than this fraction systematically causes more weight losses.

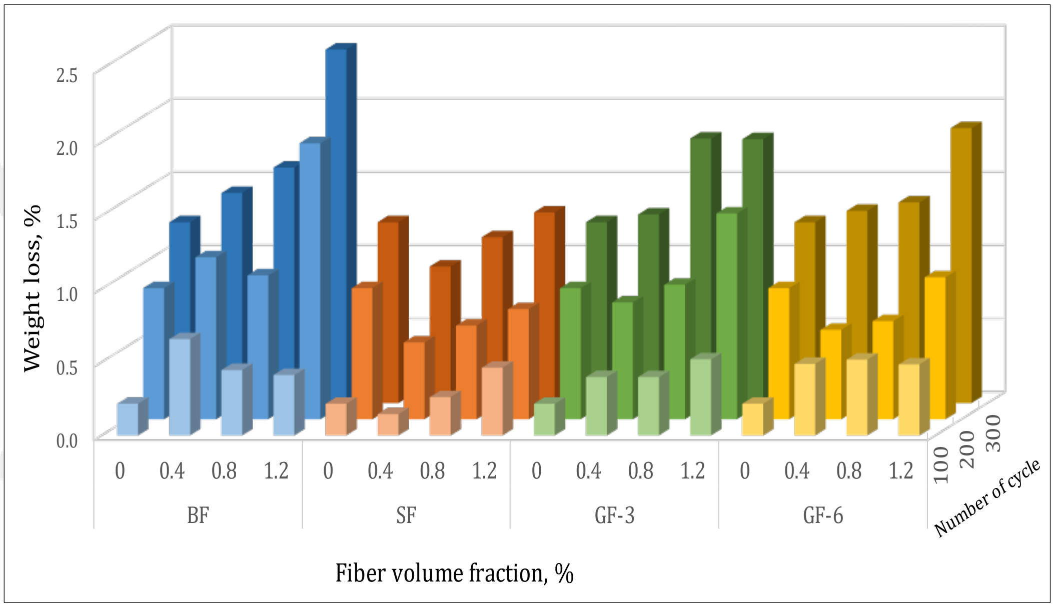


Figure 4.8. Variation of the weight loss in the fibrous geopolymer composites subjected to certain freeze-thaw cycles

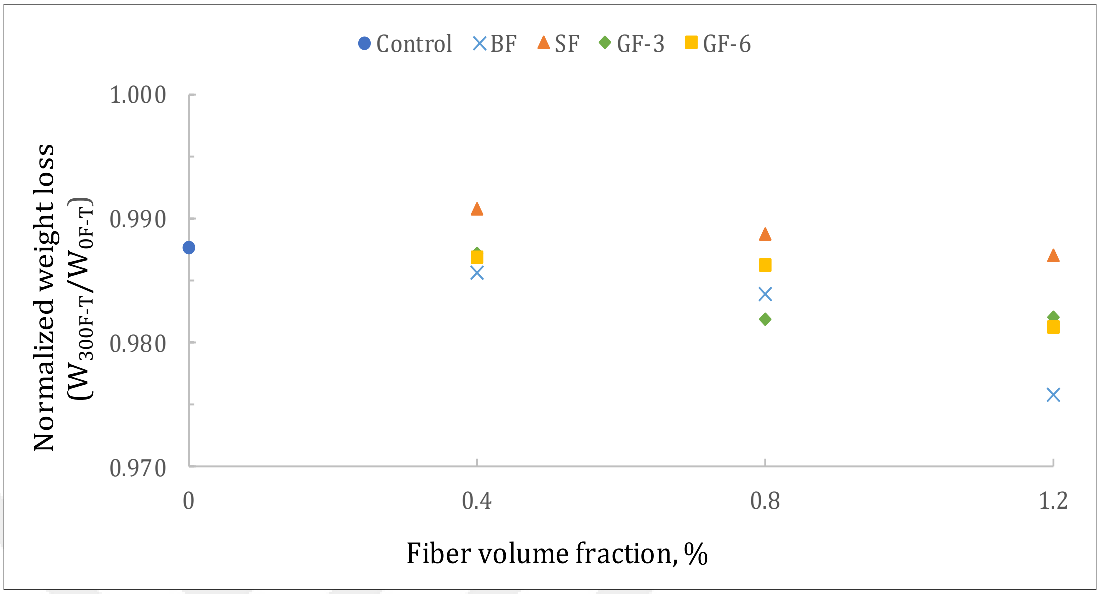
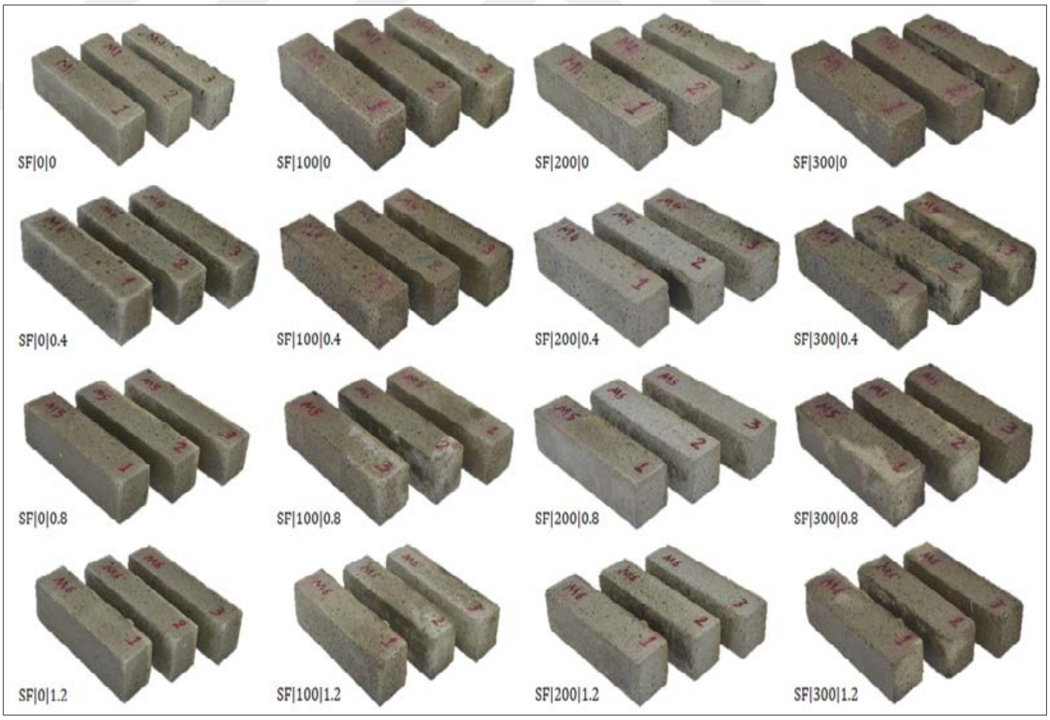


Figure 4.9. Effect of fiber type and fiber volume fraction on the normalized weight loss of the geopolymer composite subjected to the 300 freeze-thaw cycles

4.6.Visual inspection after freeze-thaw exposure

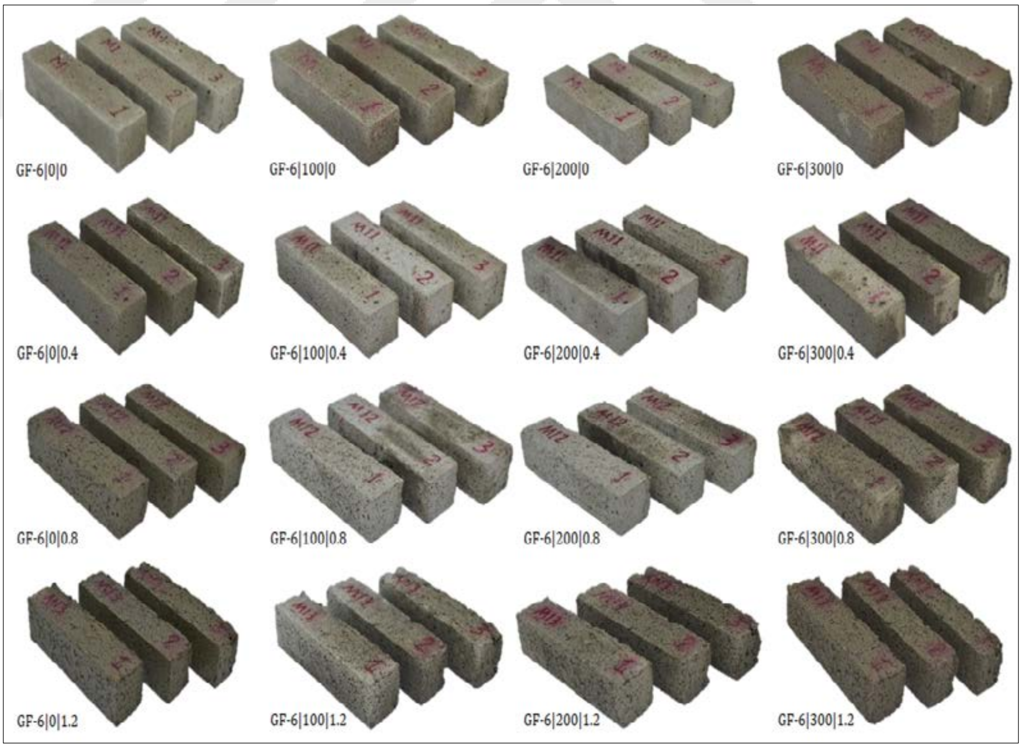
The change in the appearance of the fibrous geopolymer composite with respect to the freeze-thaw cycle is photographically presented in Figures 10 (a-d). The photos shown in these figures revealed that there is almost no change in the appearance of the fibrous geopolymer composite samples because of applying the freeze-thaw cycles. In another saying, the visual inspection indicated that the specimens preserve their stability and integrity after subjecting the 300 freeze-thaw cycles. Although no remarkable change in the external look of the fibrous geopolymer composite samples was seen, it can be stated that there is a deterioration in the microscale when the variation in the strength and ultrasonic pulse velocity was taken into consideration. As a consequence, it can be stated that applying the freeze-thaw cycles to the fibrous geopolymer composites did not significantly damage the semblance of the samples. The samples maintained the visual integrity even they were subjected to the 300 freeze-thaw cycles.

(a) BF mortar series



(b) SF mortar series

(b) GF-3mm mortar series



(d) GF-6mm mortar series

Figure 4.10. Visual inspection of the fibrous geopolymer composite subjected to freeze-thaw cycles (a) basalt fiber. (b) steel fiber. (c) glass fiber-3mm. (d) glass fiber-6mm.

4.7. Statistical assessment

The previous section involves the graphically and photographically presentation of the experimental findings of the fibrous geopolymer composites. In this section, the findings would be statistically assessed by handling the statistical analyzing method named general linear model analysis of variance (GLM-ANOVA). The analysis variance is an effective and beneficial tool for revealing and comprehending the impact of the independent parameters on the dependent parameters. Herein, firstly, the impact of fiber type and fiber volume fraction on the flowability of the mortar batches was analyzed. In this evaluation, the fiber type and fiber volume fraction were chosen as the independent parameters while the flowability in terms of flow diameter was designated as the dependent parameter. Then, in addition to the impact of the fiber type and fiber volume fraction as well as the freeze-thaw cycle (independent parameters) on the compressive and flexural strengths, ultrasonic pulse velocity, and weight loss (dependent parameters) was analyzed. The analyses were performed on a MINITAB-named software that includes the GLM-ANOVA method. The significance test of the independent parameters was performed at the level of 0.05 (P-value).

Table 4.5. depicts the results of the statistical analysis of the experimental findings. When the statistical results submitted for the flow diameter are considered, it can be seen that both fiber type and fiber volume fraction are statistically important parameters on the flowability of the geopolymer composites. This conclusion was arrived at by taking into consideration the P-values of the fiber type and fiber volume fraction. Because the P-value of less than 0.05 incontrovertibly approves the significance of the independent parameter while the P-value of greater than 0.05 means that this independent parameter has no accounting impact on the dependent parameter. Although both fiber type and fiber volume fraction are statistically meaningful parameters on the flowability of the geopolymer composite, it can be expressed pursuant to the percent contribution values that the fiber volume fraction has almost three-fold more impact on the flowability than the fiber type. Besides, the statistical results revealed that in addition to fiber type and fiber volume fraction, the freeze-thaw cycles have also a significant effect on both compressive and flexural strengths in the

case of considering P-values. However, it can be obviously seen from the last column of Table 4.5. that the fiber volume fraction has a fewer impact on the strengths of the geopolymer composite than the fiber type and freeze-thaw cycles. On the other hand, the percent contribution-based effectiveness of fiber type and freeze-thaw cycles indicated that these two independent parameters have a close impact on the strength of the geopolymer composite. An important finding has been achieved from the statistical assessment of the experimental results of the ultrasonic pulse velocity. In this regard, it was seen that the fiber type is not a statistically significant parameter on the ultrasonic pulse velocity of the geopolymer composite since its P-value is more than 0.05. Nevertheless, although the fiber volume fraction and freeze-thaw cycles can be considered statistically important parameters in accordance with their P-values, the effectiveness of the freeze-thaw cycles is much more than that of the fiber volume fraction in regard to percent contribution values. The statistical evaluation also showed that all independent parameters have a meaningful impact on the weight loss, however, the effectiveness of the freeze-thaw cycles is remarkably greater than the others.

Table 4.5. Statistical analysis of independent variables

Dependent Variable	Independent variable	Sequential Sum of Squares	Computed F	P-value	Significance	Contribution (%)
Flow diameter	Fiber type	2281.3	7.55	0.008	YES	21.8
	FVF*	7256.3	24.02	0.000	YES	69.5
	Error	906.2	-	-	-	8.7
	Total	10443.8	-	-	-	-
Compressive strength	Fiber type	218.317	18.73	0.000	YES	28.8
	FVF*	61.973	5.32	0.000	YES	8.2
	# of cycle	266.758	18.73	0.003	YES	35.2
	Error	209.760	-	-	-	27.7
	Total	756.808	-	-	-	-
Flexural strength	Fiber type	16.5960	22.09	0.000	YES	31.7
	FVF*	3.0706	4.09	0.000	YES	5.9
	# of cycle	19.1654	25.51	0.011	YES	36.6
	Error	13.5238	-	-	-	25.8
	Total	52.3558	-	-	-	-
Ultrasonic pulse velocity	Fiber type	271611	2.10	0.111	NO	2.7
	FVF*	1052833	49.69	0.000	YES	10.4
	# of cycle	6432303	8.13	0.000	YES	63.8
	Error	2330299	-	-	-	23.1
	Total	10087046	-	-	-	-
Weight loss	Fiber type	0.7550	5.96	0.001	YES	3.2
	FVF*	0.9456	7.46	0.000	YES	4.1
	# of cycle	19.2988	152.31	0.000	YES	82.9
	Error	2.2807	-	-	-	9.8
	Total	23.2801	-	-	-	-

*FVF: fiber volume fraction

5. CONCLUSION and SUGGESTIONS

The following conclusions have been arrived in reference to the experimental findings and statistical analysis mentioned above.

- The fibrous geopolymer composite at different fiber volume fractions can be manufactured with basalt, steel, and glass fibers.
- The fiber addition and fiber volume fraction negatively influenced the workability of the geopolymer composites.
- Incorporating fiber at the volume fraction of 0.4% increased the compressive strength, however, after this fraction, the compressive strength of the geopolymer composite decreased when GF-6 type fiber was employed. The decrease in the compressive strength was also observed when the GF-3 type fiber was added to the geopolymer composite at a volume fraction of more than 0.8%, whereas a continuous increase in the compressive strength was achieved when the volume fraction of the BF and SF type fibers were increased.
- Fiber type and fiber volume fraction had a similar influence on the flexural strength. A continuous increase in the flexural strength was achieved in the case of BF and SF type fibers while a decrease in the flexural strength was observed after a certain volume fraction in the case of GF-3 and GF-6 type fibers.
- In both compressive and flexural strengths, the highest strengths were attained when the SF type fiber was employed in the geopolymer composite manufacturing, while the lowest values were observed in the geopolymer composite batches involving GF-6 type fiber.
- Fiber addition slightly increased the ultrasonic pulse velocity values of the geopolymer composites, except the GF-6 type fiber addition.
- The use of fiber made the geopolymer composite more durable against the freeze-thaw. Namely, the fiber addition reduced the decreases in the compressive and flexural strength due to applying the freeze-thaw cycles.
- A sudden drop in the ultrasonic pulse velocity values was observed by augmenting the freeze-thaw cycles. The fiber addition led to reducing the

difference in the ultrasonic pulse velocity of the geopolymer composites caused by subjecting the freeze-thaw cycles. Namely, the fibers decreased the damage of the freeze-thaw cycles on the ultrasonic pulse velocity of the geopolymer composites.

- The results revealed that the weight loss occurring in the geopolymer composites was not remarkably affected by the use of fiber and increasing the volume fraction. But, the geopolymer composite produced in this study performed a good resistance to weight loss. A weight loss of less than 2.5% was observed in all the mortar samples after the 300 freeze-thaw cycles. The best impact was achieved from the SF type fiber.
- The visual inspection indicated that applying the freeze-thaw cycles did not destroy the integrity of the fibrous geopolymer composite samples even subjecting the 300 freeze-thaw cycles.
- The statistical analysis showed that both fiber type and fiber volume fraction are significant independent parameters on the workability of the geopolymer composite but the effectiveness of the volume fraction on the workability is higher than that of the fiber type. Besides, it was seen that both compressive and flexural strengths are statistically importantly affected by the fiber type, fiber volume fraction, and freeze-thaw cycles. However, the fiber volume fraction had the fewest impact on the strengths, while the other two independent parameters had a close impact.
- Moreover, it was seen that the fiber type is not a statistically significant parameter on the ultrasonic pulse velocity of the geopolymer composite. Nonetheless, although the other two independent parameters are statistically important, the effectiveness of the freeze-thaw cycle is much more than that of the fiber volume fraction. Finally, it was seen that all independent parameters have a meaningful influence on the weight loss of the geopolymer composite but the impact of the freeze-thaw is incommensurably more than the other two.

REFERENCES

ABDUL RAHIM, R. H., RAHMIATI, T., AZIZLI, K. A., MAN, Z., NURUDDIN, M. F., ISMAIL, L., 2014. Comparison of using NaOH and KOH activated fly ash-based geopolymer on the mechanical properties. Materials Science Forum, 803:179-184.

ACI Committee 318, 2014. Building Code Requirements for Structural Concrete.

ACI Committee 445-IR, 1982. State - of - the art report in fiber reinforced concrete.

ACI Committee 544-IR, 1996. Fiber reinforced concrete.

ADAM A. A, HORİANTO X. X. X., 2014 The effect of temperature and duration of curing on the strength of fly ash based geopolymer composite. Procedia Engineering, 95:410-414.

ADAK, D., SARKAR, M., MANDAL, S., 2014. Effect of Nano-silica on strength and durability of fly ash based geopolymer composite. Construction and Building Materials, 70:453-459.

ALDIN, Z. 3D printing of geopolymer concrete. MSc Thesis, the Technical University of Delft, The Netherlands.

ALLAHVERDİ, A., ABADİ, M. M .B. R., ANWAR, HOSSAIN, K. M., LACHEMI, M., 2014. Resistance of chemically-activated high phosphorous slag content cement against freeze–thaw cycles. Cold Regions Science and Technology, 103:107-114.

AL-MASHHADANİ, M. M., CANPOLAT, O., AYGÖRMEZ, Y., UYSAL, M., ERDEM, S., 2018. Mechanical and microstructural characterization of fiber reinforced fly ash based geopolymer composites. Construction and Building Materials, 167:505-513.

ALI, W., CANPOLAT, O., AYGÖRMEZ, Y., AL-MASHHADANI, M. M., 2020. Evaluation of the 12–24 mm basalt fibers and boron waste on reinforced metakaolin-based geopolymer. Construction and Building Materials, 251:118976.

AYDIN, S., BARADAN, B., 2012. Mechanical and microstructural properties of heat cured alkali-activated slag mortars. Materials and Design, 35:374-383.

AYGÖRMEZ, Y., Canpolat, O., Al-mashhadani, M., Uysal, M., 2020. Elevated temperature, freezing-thawing and wetting-drying effects on polypropylene fiber reinforced metakaolin based geopolymer composites. Construction and Building Materials, 235:117502.

ASTM C311/C311M, 2018. Standard Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use in Portland-Cement Concrete.

ASTM C618-19, Standard Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use in Concrete, ASTM International, West Conshohocken, PA, 2019, www.astm.org.

ASTM C618-19, 2019. Standard Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use in Concrete, ASTM International, West Conshohocken, PA.

ASTM C666-97, 1997. Standard Test Method for Resistance of Concrete to Rapid Freezing and Thawing,” ASTM Int.

ASTM C127-15, 2015. Standard Test Method for Relative Density (Specific Gravity) and Absorption of Coarse Aggregate, ASTM International, West Conshohocken, PA.

ASTM C1437-20, 2020. Standard Test Method for Flow of Hydraulic Cement Mortar, ASTM International, West Conshohocken, PA.

ASTM C138/C138M-17a, 2017. Standard Test Method for Density (Unit Weight), Yield, and Air Content (Gravimetric) of Concrete, ASTM International, West Conshohocken, PA.

ASTM C109/C109M-20b, 2020. Standard Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or [50 mm] Cube Specimens), ASTM International, West Conshohocken, PA.

ASTM C348-20, 2020. Standard Test Method for Flexural Strength of Hydraulic-Cement Mortars, ASTM International, West Conshohocken, PA, 2020.

ASTM C597-16, 2016. Standard Test Method for Pulse Velocity Through Concrete, ASTM International, West Conshohocken, PA.

ASTM C666/C666M-15, 2015. Standard Test Method for Resistance of Concrete to Rapid Freezing and Thawing, ASTM International, West Conshohocken, PA.

BASHEER, L., KROPP, J., CLELAND, D.J., 2001. Assessment of the durability of concrete from its permeation properties: a review. Construction and Building Materials, 15(2):93-103.

BATSON, G. B., 1962. Mechanics of Crack Arrest in Concrete with Closely Spaced Wire Reinforcement.

BHALCHANDRA, S. A. and BHOSLE, A. Y., 2013. Properties of glass fibre reinforced geopolymer concrete. International Journal of Modern Engineering Research (IJMER), 3(4), pp.2007-2010.

BOSOAGA, A., MASEK, O. and OAKEY, J. E., 2009. CO2 capture technologies for cement industry. Energy procedia, 1(1):133-140.

ÇELİK, A., YILMAZ, K., CANPOLAT, O., AL-MASHHADANI, MM., AYGÖRMEZ, Y., UYSAL, M., 2018. High-temperature behaviour and mechanical characteristics of boron waste additive metakaolin based geopolymer composites reinforced with synthetic fibers. Construction and Building Materials, 187:1190-1203.

CHOWDHURY, S., MOHAPATRA, S., GAUR, A., DWIVEDI, G., SONI, A., 2020. Study of various properties of geopolymer concrete- A review. Materials Today: Proceedings: 2020.

COMRİE, D. C., PATERSON, J. H. AND RİTCEY, D. J., 1988. Geopolymer technologies in toxic waste management. In Proceedings of Geopolymer: 107-123.

DAVIDOVITS J., 1979. Polymère Minéral, French Patent Application FR 79.22041

(FR 2,464,227) and FR 80.18970 (FR 2,489,290); US Patent 4,349,386, Mineral polymer.

DAVIDOVITS, J., 1991a. Geopolymers. Journal of Thermal Analysis, 37(8): 1633–1656.

DAVIDOVITS, J., 1991b. Geopolymers - Inorganic polymeric new materials. Journal of Thermal Analysis, 37(8): 1633–1656.

DAVIDOVITS, J., 1994, October. Properties of geopolymer cements. In First international conference on alkaline cements and concretes (Vol. 1, pp. 131-

149). Kiev State Technical University, Ukraine: Scientific Research Institute on Binders and Materials.

DEB, P. S., SARKER, P. K., BARBHUIYA, S., 2016. Sorptivity and acid resistance of ambient cured geopolymer composites containing nano-silica. Cement and Concrete Composites, 72:235-245.

DUAN, P., YAN, C., ZHOU, W., LUO, W., 2015. Thermal behaviour of Portland cement and fly ash–metakaolin-based geopolymer cement pastes. Arabian Journal for Science and Engineering, 40(8):2261-2269.

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é

DUXSON, P., FERNNEZ, A., PROVIS, J. L., LUKEY, G. C., PALOMO, VAN DEVENTER, J. S. 2007. Geopolymer technology: The current state of the art. J. Mater. Sci., 42:2917–2933.

FERNANDEZ-JIMENEZ, A.M., PALOMO, A. AND LOPEZ-HOMBRADOS, C., 2006. Engineering properties of alkali-activated fly ash concrete. ACI Materials Journal, 103(2);106.

FUNKE, H. L., GELBRİCH, S. AND KROLL, L., 2016. An Alkali Activated Binder for High Chemical Resistant Self-Levelling Mortar. Open Journal of Composite Materials, 6(4);132-142.

GANESAN, N., ABRAHAM, R., RAJ, S. D., 2015. Durability characteristics of steel fibre reinforced geopolymer concrete. Construction and Building Materials, 93:471-476.

GANESH, A. C., MUTHUKANNAN, M., 2020. Development of high performance sustainable optimized fiber reinforced geopolymer concrete and prediction of compressive strength. Journal of Cleaner Production, 124543.

GORIPARTHI, M. R., RAO, G. T., 2017. Effect of fly ash and GGBS combination on mechanical and durability properties of GPC. Advances in Concrete Construction, 5(4):313-330.

GRZYBOWSKI, M., 1991. Determination of crack-arresting properties of fiber-reinforced cementitious composites.

GRZYBOWSKI, M., AND, SHAH, S. P., 1990. Shrinkage cracking of fiber reinforced concrete. Materials journal, 87(2):138-148.

GUO, X., XIONG, G., 2020. Resistance of fiber-reinforced fly ash-steel slag based geopolymer composite to sulfate attack and drying-wetting cycles. Construction and Building Materials, 121326.

HABERT, G., BİLLARD, C., ROSSİ, P., CHEN, C., ROUSSEL, N., 2010. Cement production technology improvement compared to factor 4 objectives. Cement and Concrete Research, 40(5):820-826.

RANGAN, B. V., 2014. Geopolymer concrete for environmental protection. The Indian Concrete Journal, 88(4):41-59.

HABERT, G., BILLARD, C., ROSSI, P., CHEN, C., ROUSSEL, N., 2010. Cement production technology improvement compared to factor 4 objectives. Cement and Concrete Research, 40(5):820-826.

HAKE, S. L., DAMGIR, R. M., PATANKAR, S. V., 2018. Temperature effect on lime powder-added geopolymer concrete. Advances in Civil Engineering, 11:1-5.

HARDJITO, D., WALLAH, S. E., RANGAN, B. V., 2002. Research into engineering properties of geopolymer concrete. International Conference on Geopolymer, 2002. Melbourne, Australia.

HARRISON, T. A., BROWN, B. V. and DEWAR, J. D., 2001. Freeze-thaw resisting concrete: its achievement in the UK. Construction Industry Research and Information Association.

HASANBEIGI, A., 2013. Emerging energy-efficiency and carbon dioxide emissions-reduction technologies for the iron and steel industry.

HEIDRICH, C., FEUERBORN, H. J. AND WEIR, A., 2013, April. Coal combustion products: a global perspective. In World of coal ash conference: 22-25.

HOU, Y., WANG, D., ZHOU, W., LU, H. O., WANG, L., 2009. Effect of activator and curing mode on fly ash-based geopolymers. Wuhan University Journal of Natural Sciences Ed. 24(5):711-5.

HUNTZINGER, D. N. and EATMON, T.D., 2009. A life-cycle assessment of Portland cement manufacturing: comparing the traditional process with alternative technologies. Journal of Cleaner Production, 17(7): 668-675.

ISLAM, A., ALENGARAM, U. J., JUMAAT, M. Z., GHAZALI, N. B., YUSOFF, S., BASHAR, I.I., 2017. Influence of steel fibers on the mechanical properties and impact resistance of lightweight geopolymer concrete. Construction and Building Materials, 152: 964-977.

JEAN, P. J., 2009. Sustainable Benefits of Concrete Structure. Published by the European Concrete Platform ASBL.

JINDAL., B. B., SİNGHAL, D., SHARMA, S. K., ASHISH, D. K., PARVEEN, J., 2017. Improving compressive strength of low calcium fly ash geopolymer concrete with alccofine. Advances in Concrete Construction, 5(1):17–29.

JINDAL, B. B., 2019. Investigations on the properties of geopolymer composite and concrete with mineral admixtures: A review. Construction and Building Materials; 227:116644.

KAKALI, G., PERRAKİ, T. H., TSİVİLİS, S. and BADOGİANNİS, E., 2001. Thermal treatment of kaolin: the effect of mineralogy on the pozzolanic activity. Applied clay science, 20(1-2):73-80.

KANI., E. N., ALLAHVERDI, A., 2009. Effects of curing time and temperature on strength development of inorganic polymeric binder based on natural pozzolan. Journal of Materials Science, 44(12):3088-3097.

KONG, D. L. Y., SANJAYAN, J. G., 2010. Effect of elevated temperatures on geopolymer paste, mortar and concrete. Cement and Concrete Research, 40(2):334-339.

LIU, L. P., ZHU, H., HE, Y. and CUI, X. M., 2015. Preparation of Carbon Fiber Reinforced Geopolymer Composites. In Advanced Materials Research Vol. 1081:275-278.

LI, Z., LIU, S., 2007. Influence of slag as additive on compressive strength of fly ash-based geopolymer. Journal of Materials in Civil Engineering, 19(6):470-474.

LÄMMLEIN, T. D., MESSINA, F., WYRZYKOWSKI, M., TERRASI, G. P., LURA, P., 2019. Low clinker high performance concretes and their potential in CFRP-prestressed structural elements. Cement and Concrete Composite, 100:130-138.

MA, C. K., AWANG, A. Z., OMAR, W., 2018. Structural and material performance of geopolymer concrete: A review. Construction and Building Materials, 186: 90-102.

MARÍN-LÓPEZ, C., ARAİZA, J. R., MANZANO-RAMÍREZ, A., AVALOS, J. R., PEREZ-BUENO, J. J., MUÑİZ-VİLLAREAL, M. S., VENTURA-RAMOS, E.

and VOROBİEV, Y., 2009. Synthesis and characterization of a concrete based on metakaolin geopolymer. Inorganic Materials, 45(12):1429.

MERMERDAŞ, K., MULAPEER, E. S., OLEIWI, S. M., 2019. Effect of glass fiber addition on the strength properties and pore structure of fly ash based geopolymer composites. Eskişehir Technical University Journal of Science and Technology A – Applied Sciences and Engineering, 20(4):427-435.

MCCAFFREY, R., 2013. Climate change and the cement industry. Global Cement and Lime Magazine.

MEHTA, K. P., 2001. Reducing the environmental impact of concrete. Concrete international, 23(10):61-66.

MEHTA, P. K. and MONTEIRO, P. J., 2017. Concrete microstructure, properties and materials.

MULAPEER, E. S., MERMERDAŞ, K., ALGIN, Z., SOR, N. H., EKMEN, Ş., 2019. Engineering properties of steel and basalt fiber reinforced geopolymer composite. 6th International Scientific Research Congress, Şanlıurfa, Turkey.

NAAMAN, A. E., 1985. Fiber reinforcement for concrete. Concrete International, 7(3):21-25.

NANAVATI, S.C., LULLA, S. J., SINGH, A. R., MEHTA, D. B., PATEL, A. M. and LADE, A. D., 2017. A Review on Fly Ash-based Geopolymer Concrete. IOSR Journal of Mechanical and Civil Engineering (IOSR-JMCE) Vol 14:12-16.

NAIDU, P. G., ADISESHU, S. and SATAYANARAYANA, P. V. V., 2012. A study on strength properties of geopolymer concrete with addition of GGBS. International Journal of Engineering Research and Development ISSN:19-28.

NAYAN, R., MUKUND, G., MALLIKARJUN, P., 2013. “Basalt Fiber Reinforced Concrete” ISSN (Online): 2319-7064.

NEMATOLLAHI, B., SANJAYAN, J., CHAI, J. X. H., LU, T. M., 2014. Properties of fresh and hardened glass fiber reinforced fly ash based geopolymer concrete. Key Engineering Materials, 594-595:629-633.

NEVILLE, A. M. and BROOKS, J. J., 1987. Concrete technology. Longman Scientific & Technical: 242-246.

NOUSHINI, A., BABAEE, M., CASTEL, A., 2016. Suitability of heat-cured low-calcium fly ash-based geopolymer concrete for precast applications. Magazine of Concrete Research, 68(4):163-177.

OKOYE, F. N., DURGAPRASAD, J., SINGH, N. B., 2015. Mechanical properties of alkali activated fly ash/Kaolin based geopolymer concrete. Construction and Building Materials, 98:685-691.

OLIVIA, M. and NIKRAZ, H., 2012. Properties of fly ash geopolymer concrete designed by Taguchi method. Materials & Design (1980-2015), 36:191-198.

PENG, J. X., HUANG, L., ZHAO, Y. B., CHEN, P., ZENG, L., 2012. Modelling of carbon dioxide measurement on cement plants. Advanced Materials Research, 610-613:2120-2128.

PALOMO, A., BLANCO-VARELA, M. T., GRANIZO, M. L., PUERTAS, F., VAZQUEZ, T., GRUTZECK, M. W., 1999. Chemical stability of cementitious materials based on metakaolin. Cement and Concrete Research, 29(7):997-1004.

PALOMO, A., GRUTZECK, M. W., and BLANCO, M. T., 1999. Alkali-activated fly ashes: a cement for the future. Cement and concrete research, 29(8):1323-1329.

PILEHVAR, S., SZCZOTOK, A. M., RODRÍGUEZ J. F., VALENTINI, L., LANZÓN, M., PAMIES, R., KJØNIKSEN, A.L., 2019. Effect of freeze-thaw cycles on the mechanical behavior of geopolymer concrete and Portland cement concrete containing micro-encapsulated phase change materials. Construction and Building Materials. 200:94-103

PROVIS, J. L., VAN DEVENTER, J. S. J., 2007. Direct measurement of the kinetics of geopolymerisation by in-situ energy dispersive X-ray diffractometry. Journal of Materials Science 42 (9): 2974–2981.

PUERTAS, F., PALACIOS, M., MANZANO, H., DOLADO, J., RICO, A., RODRÍGUEZ, J., 2019. A model for the CASH gel formed in alkali-activated slag cements. Journal of European Ceramics Society; 31:2043-2056.

PUERTAS, F., FERNÁNDEZ-JIMÉNEZ, A., 2003. Mineralogical and microstructural characterisation of alkali-activated fly ash/slag pastes. Cement and Concrete Composite, 25(3):287-292.

PURNELL, P., 2012. Material nature versus structural nurture: the embodied carbon of fundamental structural elements. Environmental science & technology, 46(1):454-461.

RAPOPORT, J., ALDEA, C.M., SHAH, S. P., ANKENMAN, B. and KARR, A., 2002. Permeability of cracked steel fiber-reinforced concrete. Journal of materials in civil engineering, 14(4):355-358.

RANGAN, B. V., 2008. Fly ash-based geopolymer concrete.

ROVNANÍK, P., ŠAFRÁNKOVÁ, K., 2016. Thermal Behaviour of Metakaolin/Fly Ash Geopolymers with Chamotte Aggregate. Materials, 9(7):535.

ŠKVÁRA, F., JÍLEK, T. AND KOPECKÝ, L., 2005. Geopolymer materials based on fly ash. Ceram. -Salic, 49(3):195-204.

SHETTY, M. S., 2005. Concrete Technology Theory and Practice, Chand S. and Company LTD.

SINGH, N. B., SAXENA, S., KUMAR, M., 2018. Effect of nanomaterials on the properties of geopolymer composites and concrete. Materials Today Proceedings, 5(3):9035-9040.

SLAVIK, R., BEDNARIK, V., VONDRUSKA, M. and NEMEC, A., 2008. Preparation of geopolymer from fluidized bed combustion bottom ash. Journal of Materials Processing Technology, 200(1-3):265-270.

STEINEROVA, M., 2011. Mechanical properties of geopolymer composites in relation to their porous structure. Ceramics-Silikáty, 55(4):362-372.

SWAMY, R. N., 2008. Sustainable concrete for the 21st century concept of strength through durability. Japan Society of Civil Engineers Concrete Committee Newsletter, 13.

TEMUUJIN, J., MINJIGMAA, A., DAVAABAL, B., BAYARZUL, U., ANKHTUYA, A., JADAMBAA, T. and MACKENZIE, K.J.D., 2014. Utilization of radioactive high-calcium Mongolian fly ash for the preparation of alkali-activated geopolymers for safe use as construction materials. Ceramics International, 40(10):16475-16483.

TEMUUJIN, J., VAN, RIESSEN, A., WILLIAMS, R., 2009. Influence of calcium compounds on the mechanical properties of fly ash geopolymer pastes. Journal of Hazardous Materials, 167(1):82-88.

TERZANO, R., SPAGNUOLO, M., MEDİCİ, L., TATEO, F., RUGGİERO, P., 2005. Characterization of different coal fly ashes for their application in the synthesis of zeolite X as cation exchanger for soil remediation. Fresenius Environmental Bulletin, 14(4):263-267.

TIRONI, A., TREZZA, M. A., SCIAN, A. N. and IRASSAR, E.F., 2012. Kaolinitic calcined clays: Factors affecting its performance as pozzolans. Construction and Building Materials, 28(1):276-281.

TURNER, L. K., COLLINS, F. G., 2013. Carbon dioxide equivalent (CO2-e) emissions: A comparison between geopolymer and OPC cement concrete. Construction and Building materials, 43:125-130.

YUNSHENG, Z., WEI, S., ZONGJIN, L., XIANGMING, Z., EDDIE, CHUNGKONG, C., 2008. Impact properties of geopolymer based extradites incorporated with fly ash and PVA short fiber. Construction and Building Materials, 22(3):370-383.

VAN, JAARSVELD, J., VAN, DEVENTER, J. and LORENZEN, L., 1997. The potential use of geopolymeric materials to immobilise toxic metals: Part I. Theory and applications. Minerals engineering, 10(7):659-669.

VAN, JAARSVELD, J., VAN, DEVENTER, J, LUKEY, G., 2002. The effect of composition and temperature on the properties of fly ash-and kaolinite-based geopolymers. Chemical Engineering Journal, 89(1):63-73.

VAN, CHANH, N., 2004. Steel fiber reinforced concrete. In Faculty of Civil Engineering Ho chi minh City university of Technology. Seminar Material: 108-116.

VENKATESWARA, J., SRINIVASA, K., RAMBABU, K., 2017. Performance of heat and ambient cured geopolymer concrete exposed to acid attack. Proceedings of the Institution of Civil Engineers - Construction Materials, 172(4):192-200.

VENKATESAN, R. P. and PAZHANI, K. C., 2016. Strength and durability properties of geopolymer concrete made with ground granulated blast furnace slag and black rice husk ash. KSCE Journal of Civil Engineering: 2384-2391.

VS, M. K., NAGENDRA, M. V. and SHINDE, D. N., 2016. Fly ash based geopolymer Concrete-Material/parameters Guidelines.

WALLAH, S. and RANGAN, B. V., 2006. Low-calcium fly ash-based geopolymer concrete.

XIE, N., DANG, Y., SHI, X., 2014. New insights into how MgCl2 deteriorates Portland cement concrete. Cement and Concrete Research, 120:244-255.

XIE, T., OZBAKKALOGLU, T., 2019. Behaviour of low-calcium fly ash bottom ash based geopolymer concrete cured at ambient temperature. Ceramics International; 85:5945-5958.

ZHANG, P., GAO, Z., WANG, J., GUO, J., HU, S., LING, Y., 2020. Properties of fresh and hardened fly ash/slag based geopolymer concrete: A review. Journal of Cleaner Production, 270:122389.

ZHAO, M., ZHANG, G., HTET, K. W., KWON, M., LIU, C., XU, Y., TAO, M., 2019. Freeze-thaw durability of red mud slurry-class F fly ash-based geopolymer: Effect of curing conditions. Construction and Building Materials, 215:381-390.

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B) International Conference, Symposium, Congress Proceedings

B1. Zana Mahmood, Kasım Mermerdaş, Ahmed Anwer (2020) “Resistance of basalt fiber reinforced geopolymer based mortars against freezing and thawing cycles” *International Conference on Technology, Engineering and Science (IConTES 2020)*

B2. Ahmed Anwer, Kasım Mermerdaş, **Zana Mahmood** (2020) Effect of Hybrid Reinforcement On Mechanical And Sorption Characteristics Of Cement Based Composites. *International Conference on Technology, Engineering and Science (IConTES 2020)*