# EFFECTS OF NANO SILICA AND BASALT FIBERS ON FLY ASH BASED GEOPOLYMER CONCRETE

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**Title**

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The Supervisory Committee certifies that this ***disquisition*** complies with North Dakota State University's regulations and meets the accepted standards for the degree of

**MASTER OF SCIENCE**

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## ABSTRACT

Emission of carbon dioxide gas has been a source of major concern for the construction industry. To curb this emission, geopolymer concrete has been deemed as a potential alternative in the recent studies. Previous research also indicates that silica and fibers provide strength benefits to ordinary Portland cement concrete OPC. This study was undertaken to recognize the benefits of adding silica and basalt fibers in Class F fly ash based geopolymer concrete and comparing it with OPC concrete. One OPC and four Geopolymer mixtures were prepared. The results show a tremendous potential of using geopolymer concrete in place of OPC concrete with Nano silica proving to be the most advantageous. Nano silica provided 28% increase in compressive strength, 8% increase in resistivity when compared with normal Fly ash based geopolymer concrete. The SEM analysis of geopolymer concrete indicates that nano silica improved the compactness of concrete providing a dense microstructure.

## ACKNOWLEDGMENTS

I would like to first thank my advisor Dr. Todd Sirotiak for his continuous backing and guidance in helping me complete my thesis. He consistently allowed this research to be my own work but steered me in the right direction whenever I needed it. I would also like to thank my committee members Dr. Matthew Stone and Dr. Ying Huang for their inputs and assistance. I thank CM&E department chairman, Dr. Jerry Gao for providing me with valuable suggestions, direction, and support throughout my graduate career. I am grateful to Dr. Achintyamugdha Sharma for providing me help whenever I needed it. Special thanks to my parents and sisters for believing in me and providing unfailing support and continuous encouragement throughout my years of study. Finally, I am extremely thankful and grateful to my friends and all those who helped me throughout my Master's study.

# DEDICATION

I dedicate this dissertation to my parents

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# 1. INTRODUCTION

## 1.1. Background and research significance

The infrastructure plays a great role in the financial growth of a country and improves the quality of life of its people. Concrete plays an important role in this regard as it is the largest manufactured construction material in the World. One of the major ingredients of concrete production is Ordinary Portland cement (OPC). Unfortunately, during the production of OPC large amounts of Carbon dioxide is released into the atmosphere resulting increasing the greenhouse effect (Bondar, 2013). It is posited that the cement industry alone produces 5-7% of global $CO_2$ emissions which is about 1.35 billion tons annually (Allwood, Cullen, & Milford, 2010a). As a result, several alternatives such as alkali-activated cement, calcium sulpho-aluminate cement, magnesium oxy-carbonate cement (carbon negative cement), and super-sulfate cement etc. have been developed (Winnefeld et al., 2015). One alternative binder to portland cement is geopolymer.

Geopolymer has been gaining steady popularity among researchers in recent years due to its early compressive strength, low permeability, good chemical resistance, and fire resistance (Aziz et al., 2016). These features, in addition to the improved sustainability (decreased carbon), makes the product very interesting as a future building material.

Although literature shows that geopolymer concrete provides strength benefits, there is little research on analyzing the benefits of adding supplementary materials like basalt fibers and nano silica in geopolymer concrete. In addition, the Scanning Electron Microscopy (SEM) analysis of this concrete also needs further investigation.

## 1.2. Objectives and scope

This research was undertaken to

1. Investigate the benefits of geopolymer concrete in comparison with ordinary Portland cement concrete.
2. Investigate the benefits of adding Basalt fiber and Nano silica in geopolymer concrete
3. Conduct Scanning Electron Microscopy (SEM) analysis of geopolymer concrete.

Type I/II cement was used for OPC concrete while Class F fly ash was selected to be used as a binder for geopolymer concrete. The concrete properties focused mainly on compressive strength, shrinkage, resistivity, slump, air, hydration.

## 1.3. Research organization and methodology

This dissertation is organized into 5 chapters. Chapter 1 contains the background and objectives of the study. Chapter 2 contains a thorough literature review. In chapter 3 the experimental program gives detailed insight into the materials and equipment to be used in the study. In chapter 4, Four geopolymer mixes (Fly Ash Geopolymer concrete, Fly Ash Geopolymer concrete with Basalt Fibers, Fly Ash Geopolymer concrete with Nano silica, Fly Ash Geopolymer concrete with Nano silica were tested. Chapter 5 provides the conclusion, limitation, and future work. For a more elaborate understanding of the research methodology, a flow-chart is provided in figure 1.1.

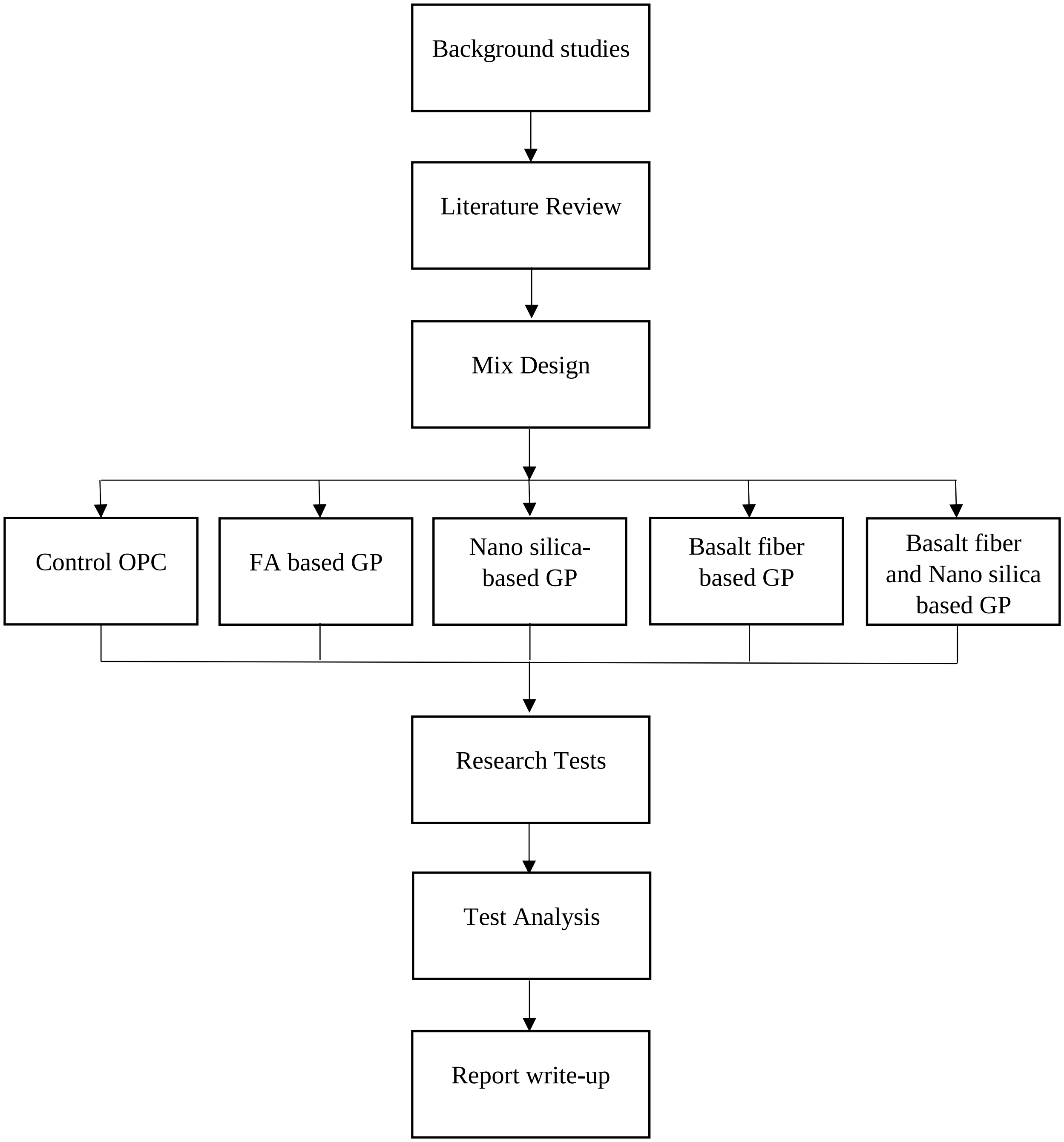


**Figure 1.1.** Research methodology

# 2. LITERATURE REVIEW

## 2.1. Introduction

The infrastructure plays a great role in the financial growth of a country the quality of life of its people. As the World's most widely produced man made product, concrete plays an important role in this regard. One of the major ingredients of concrete production is Ordinary Portland cement (OPC). Unfortunately, the production of cement produces greenhouse gas emissions and is detrimental to the environment around us. (Jawahar & Mounika, 2016)

## 2.2. Environmental issues

There is a reasonable scientific consensus that global warming resulting in climate change will have damaging consequences for all living organisms in this World. The emission of greenhouse gases like carbon dioxide and methane is a major cause of Global warming (Reed, Lokuge, & Karunasena, 2014). The climate change is also attributed to the paradoxical global dimming which is caused by polluted particles in the environment. These polluted particles additionally block the sunlight from reaching the Earth thereby increasing global dimming.

The production of OPC emits large amounts of carbon dioxide into the atmosphere resulting in greenhouse effect (Bondar, 2013). The cement industry alone produces 5-7% of global $CO_2$ emissions which is about 1.35 billion tons annually(Allwood, Cullen, & Milford, 2010b) in 2010. There are various mechanisms encompassing cement manufacturing which lead to the emission of $CO_2$, including calcination of limestone into calcium oxide, and combustion of fossil fuels (Ali, Saidur, & Hossain, 2011)

As parties recognize the harmful effects of cement production to the environment, there has been an upsurge in developing mitigating strategies to overcome this problem (Miller, John, Pacca, & Horvath, 2017). These include fuel substitution, using waste heat as an alternative source, using renewable energy sources (Ali et al., 2011). Using different alternatives to ordinary Portland cement are also being sought in the industry.

As a result, several alternatives such as alkali-activated cement, calcium sulpho-aluminate cement, magnesium oxy-carbonate cement (carbon negative cement), and super-sulfated cement etc. have been developed (Winnefeld, Haha, Le Saout, Costoya, & Ko, 2015). One alternative binder to portland cement is geopolymer.

**2.3. Geopolymer**

Davidovits was an earlier researcher who looked into the chemical process between alkali liquid and Silicon (Si) /Aluminum (Al) material. He proposed that supplementary cementitious materials such as Fly Ash, Slag, Blast furnace, limestone, rice husk, metakaolin, natural pozzolan type materials when combined with mineral and metal resources could be able to produce a binder (Bondar, 2013). He named the term “Geopolymer” since the said chemical reaction is a polymerization process (Davidovits, 1999)

It was suggested that these supplementary cementitious materials should be highly amorphous and have enough glassy content and low water demand in order to develop a stable geopolymer. These cementitious or alumino silicate materials are activated by Alkaline activators i.e. Sodium hydroxide (NaOH), Sodium Silicate ($Na_2CO_3$), Potassium hydroxide (KOH) and Potassium silicate (KCO3) material (Duxson et al., 2007).

The Polymerization process is very much dependent upon the type of alkaline liquid. When the alkaline liquid contains either Sodium or Potassium silicate its reaction occurs at a much higher rate than that as compared to only the alkaline hydroxides (Palomo, Grutzeck, & Blanco, 1999)

The process of synthesis of a typical geopolymer occurs by the reaction of solid alumino silicate powder with alkali hydroxide/alkali silicate. The polymerization occurs when reactive alumino silicates (under high alkaline conditions) dissolve in the solution in order to release $SiO_4$ and $AlO_4$ tetra hedral units. These tetra hedral units form polymeric Si-O-Al-O bonds by linking it to polymeric precursor by sharing oxygen atom. The schematic formation of geopolymer material is given in the following equations. (Komnitsas, 2011) (Singh, Ishwarya, Gupta, & Bhattacharyya, 2015)

$$(Si_2O_5Al_2O_2)n + H_{2O} + OH^- \rightarrow SI(OH)_4 + Al(OH)^{-4} \tag{1}$$

$$Si(OH)_4 + Al(OH)^{-4} \rightarrow (-Si - O - Al - O -)_n + 4H_2O \tag{2}$$

Equation 2 reveals that water is released during the chemical reaction. This water facilitates workability to the mixture and does not play a role in the main chemical reaction. This differs from the chemical reaction of water in an ordinary Portland cement mixture's hydration process that results in the production of calcium hydroxide and calcium silicate hydrate (Komnitsas, 2011).

### 2.4. Classification

Geopolymers can be classified into three forms depending on the ratio of Si/Al (Bondar, 2013)

a) Poly (sialite) having (-Si-O-Al-O-) as the repeating unit

b) Poly (sialite-siloxo) having (-Si-O-Al-O-Si-O) as the repeating unit; or

c) Poly (sialite-disilixo) having (-Si-O-Al-O-Si-O-SI-O-) as the repeating unit

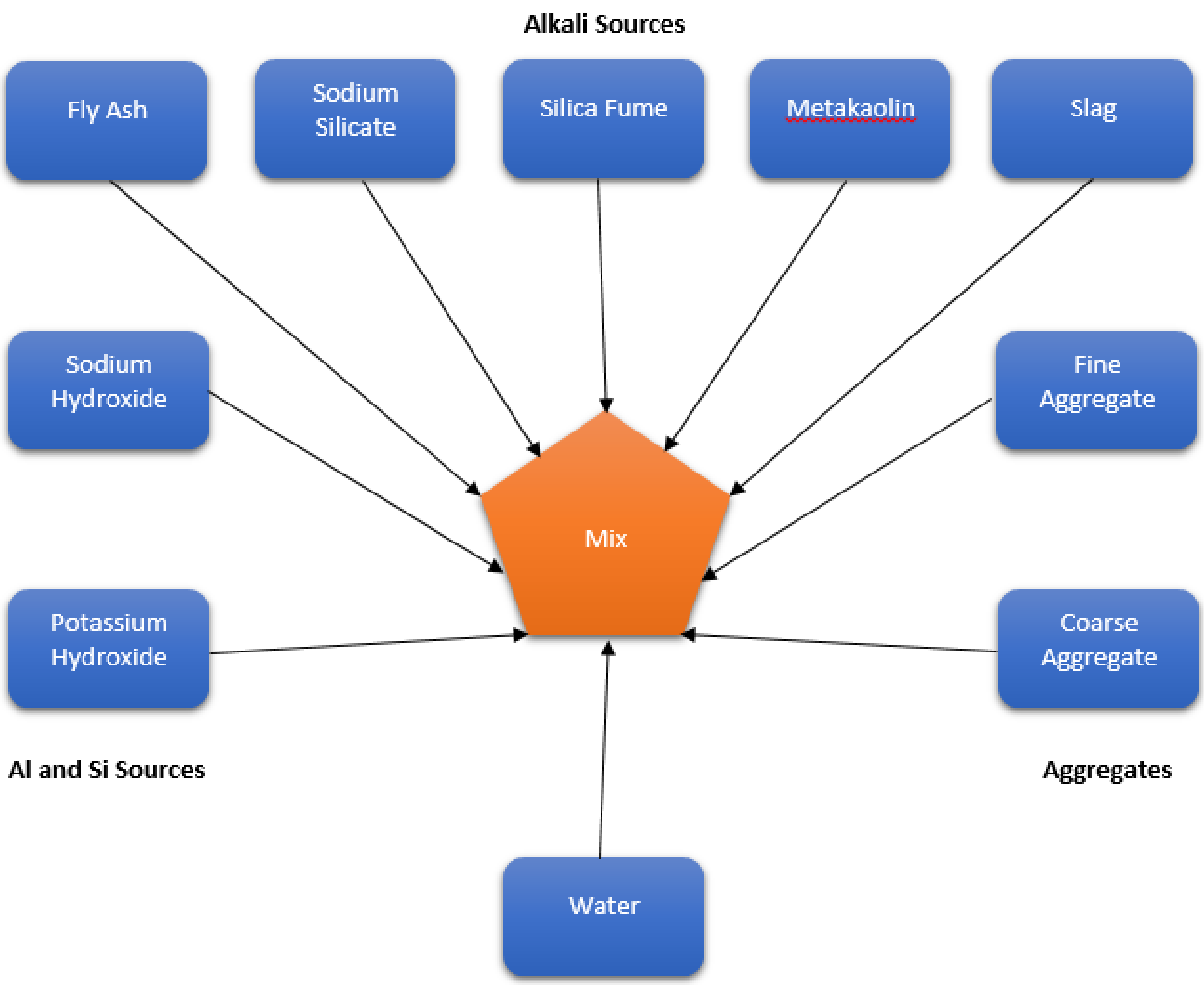


**Figure 2.1.** Production of geopolymer concrete

(McLellan, Williams, Lay, Van Riessen, & Corder, 2011)

**2.5. Alkaline solutions**

Sodium hydroxide or Potassium hydroxide are combined with Sodium silicate or potassium silicate to produce an alkaline solution (Swanepoel & Strydom, 2002) (Davidovits, 1999)(Barbosa, MacKenzie, & Thaumaturgo, 2000). The reactions in solutions containing silicates are much higher as compared to alkaline hydroxides and enhance the reaction between source material and solution (Palomo et al., 1999). NaOH solution also causes a higher extent of dissolution of materials. (Xu & Van Deventer, 2000)

### 2.6. Properties of Geopolymer concrete

The setting time of geopolymer concrete can either be timed to set fast or slow by adjusting the mixture proportions. Required strength can be achieved in a very short time (Bondar, 2013; Van Jaarsveld, Van Deventer, & Lorenzen, 1997). Curing temperature, source material, and type of alkaline activator plays an important role in the setting time of Geopolymer concrete as studied by (Cheng & Chiu, 2003). They concluded that setting time of Geopolymer paste with Granulated blast furnace slag as a source material was between 15 to 45 minutes at a temperature of 60°C.

Initial setting time of fly ash based geopolymer concrete is faster at high temperatures and the final setting occurred after 15 – 25 minutes after initial setting (Bondar, 2013)
To understand the slump mechanism of fly ash based Geopolymer concrete, experiments were conducted by (Laskar & Bhattacharjee, 2011) that showed varying slumps that ranged from 25mm to flowing having activator strength 1-20M. It was observed that Molar strength of NaOH solution and the ratio of silicate to hydroxide solution affected the yield stress and viscosity of the geopolymer concrete.

Geopolymer concrete hardens swiftly and may gain strength of the order of 100 Mpa in 28 days. Due to their low porosity, it obtains superior mechanical properties as compared to conventional concrete (Komnitsas, 2011; Temuujin, Rickard, Lee, & Van Riessen, 2011). That depends on several following factors:

#### 2.6.1. Curing

Palomo concluded that the mechanical strength of geopolymer concrete was affected by the curing temperatures and the type of alkaline liquid. Higher compressive strengths were achieved as a result of higher curing temperature and longer curing time (Palomo et al., 1999). But it has been reported that too much curing may have a negative impact on finished concrete

(Van Jaarsveld, Van Deventer, & Lukey, 2002). Curing temperature more than 60℃ and duration more than 48 hours may not have a substantial effect on the compressive strength of concrete. Due to the fast polymerization process in geopolymer concrete, the compressive strength does not fluctuate much after it has been oven cured for 24h. This is not the case in OPC concrete where strength increases as a result of extended hydration process. (Djwantoro Hardjito, Dody M. J. Sumajouw, Wallah, & Rangan, 2004)(Bondar, 2013)

**2.6.2. Type of alkaline solution**

Different molarities of NaOH can also have an effect on the strength of geopolymer concrete as studied by (Madheswaran, Gnanasundar, & Gopalakrishnan, 2013) where they prepared geopolymer mixes with NaOH molarities of 3M, 5M and 7M respectively. The compressive strength of the mixes ranged from 15MPa to 52Mpa and it was concluded that the strength of geopolymer concrete increased as the molar concentration of NaOH increased.

Similarly the rate of reaction in alkaline solutions containing sodium silicates, were higher as compared to alkaline solutions having hydroxide (Palomo et al., 1999), but it has been reported that alkali hydroxides especially the NaOH solution resulted in improving mechanical strengths when compared with ordinary Portland Cement (Nagalia et al., 2016). Jaarsveld also studied the influence of higher curing temperatures on geopolymer concrete. They concluded that cracking can occur if the concrete is exposed to high temperatures for curing purpose and suggested to use mild curing (Van Jaarsveld et al., 2002).

The mechanical strength and modulus of elasticity have a significant improvement due to the presence of silicate ions in the alkaline solution (Fernández-Jiménez, A., Palomo, a. and López- Hombrados, 2006). Increase in compressive strength increased the modulus of elasticity of Geopolymer concrete, although it was found to be below the guidelines suggested by ACI for OPC concrete.

## 2.7. Manufacturing process

According to past researches, the production of geopolymer concrete involves a dry mix of aggregates and the binding material like fly ash or slag to ensure homogeneity of the mixture. Activator solution containing NaOH or KOH and $Na_2CO_3$ or $KCO_3$ is added to the mixture followed by more mixing. Any additional water is then added to achieve the desired consistency. (Djwantoro Hardjito, Cheak, & Lee Ing, 2008; Djwantoro Hardjito, Wallah, Sumajouw, & Rangan, 2004; Nagalia et al., 2016; Swanepoel & Strydom, 2002)

## 2.8. Applications of Geopolymer concrete

A review survey of geopolymer concrete indicated that it can be used effectively in pre-cast construction since it provides high early strength (Aleem & Arumairaj, 2012), therefore well suited to build precast structures needed in rehabilitating an existing structure (Lloyd & Rangan, 2010).

Geopolymer concrete can be used in mass concretes like in Dam construction, waste water pipe line, in structural retrofits using geopolymers- fiber composites, in pre tension concrete structures(Aleem & Arumairaj, 2012; Bondar, 2009, 2013). It can also be used in railway sleepers as indicated by (Palomo et al., 1999). It was found that the existing technology can be used to create geopolymer concrete structural members. The characteristics of the products showed limited drying shrinkage (Lleyda, 2004). (Balaguru, 1998; D Hardjito & Rangan, 2005) reported that geopolymer composites can be used to strengthen reinforced concrete beams. The fire resistance as a result of using geopolymers was much higher as compared organic polymers. Sewer pipes reinforced with geopolymer concrete having different diameters ranging from 375mm to 1800mm have been manufactured utilizing the same facilities as a normal portland cement concrete. The results suggested that geopolymer concrete perform much better than OPC and should be considered for future work (Gourley & Johnson, 2005;

Lloyd & Rangan, 2010). On a commercial scale, culverts reinforced with geopolymer concrete have been manufactured that met the specification requirement of such products.(Cheema, Lloyd, & Rangan, 2009). Geopolymer concrete was used to make a precast bridge deck.  The composite bridge structure was made from fiberglass girders which acted as a composite with a geopolymer bridge deck. The bridge is still in service to date and has shown no signs of distress even though continuous truck loading takes place (Aldred & Day, 2012)

**2.9. Economic benefits of Geopolymer concrete**

Geopolymer concrete present economic benefits to the construction industry. The price of fly ash is much cheaper to ordinary portland cement. After incorporating the price of alkaline liquids, geopolymer concrete is estimated to be 10-30% cheaper than OPC.  (Lloyd & Rangan, 2010) Geopolymer concrete also has low risks of repairing costs since it provides good resistance to acid and sulfate attack and minimal drying shrinkage (Lloyd & Rangan, 2010).

The literature review on geopolymer concrete shows several benefits. However, for the purpose of this research, additives such as basalt fibers and nano silica have been utilized to investigate if there are any additional values obtained. Some literature on these materials are given below.

**2.10. Fiber reinforced concrete**

The brittle behavior of cementitious materials and weak resistance to tensile forces makes way for adding reinforcing materials in concrete (Neville & Brooks, 2004). Usually, steel reinforcements are used to increase the tensile strength of concrete. Fibers work in a similar way where they act to transmit tensile forces. Fiber reinforced polymer (FRP) materials, due to their low price and improved cracking and shrinkage resistance have gained popularity in the construction industry (Karahan, Tanyildizi, & Atis, 2009; Reed et al., 2014). Despite its advantages, the FRP, when exposed to high temperatures can emit poisonous fumes (Lee, 2002). To counter this, Basalt fibers are another alternative.

Basalt rocks go through a melting process to produce basalt fibers. These rocks are divided into very small particles that results in the formation of fibers. These fibers do not need any additives during the manufacturing process thereby resulting in extra cost reduction (Sim, Park, & Moon, 2005). Basalt furthermore provides these values:

- Basalt fibers result in better tensile strength when compared with glass fibers.
- It has a greater failure strain when compared with carbon fibers.
- It provides increased resistance to chemical attack and fire with reduced poisonous fumes (Berozashvili, 2001)

**2.11. Nano-silica based concrete**

The small particle size of nano-silica provides a large surface area, increasing the rate of cement hydration and pozzolanic reaction (Belkowitz, 2009). This results in an improved performance of OPC concrete. Dry grains and colloidal suspension are the two main forms in which nano-silica is available.  Unfortunately, a mixing procedure is required for dry nano-silica grains so that it completely disperse in mixing water or other admixtures. Colloidal nano-silica is manufactured as a suspension and is ready to use (Said, Zeidan, Bassuoni, & Tian, 2012).

# 3. EXPERIMENTAL PROGRAM

## 3.1. Introduction

This chapter presents detail description of the process utilized to develop different geopolymer concrete mixes using class F Fly Ash. After review of existing geopolymer concrete studies, it was decided to compare the geopolymer mixes against OPC concrete (Control Mix) and to conduct different mechanical tests in order to determine any positive or negative differences between them. The manufacturing and testing of geopolymer mixes were conducted according to the ASTM standards in OPC manufacturing process. The only difference was the process of curing as the OPC mix require water curing while geopolymer mix needed oven or steam curing. For the purpose of this study, originally only oven curing of the geopolymer mixes was conducted.

## 3.2. Materials used

The materials used for the purpose of this research are discussed below:

### 3.2.1. Ordinary portland cement (OPC)

Type I/II cement was chosen for the OPC concrete mix and was to be used as a control and benchmark for comparative studies. The reason for choosing Type I/II cement was that it is the most commonly used cement in the construction industry for general purposes. Table 3.1 represents the chemical and physical properties of Type I/II cement

**Table 3.1.** Chemical and physical properties of cement used

| **Item** | **Type I/II Cement*** |
|---|---|
| $SiO_2$ (%) | 20.31 |
| $Al_2O_3$ (%) | 3.79 |
| $Fe_2O_3$ (%) | 3.22 |
| CaO (%) | 64.29 |
| MgO (%) | 3.38 |
| $SO_3$ (%) | 2.26 |
| Loss on ignition (%) | 2.73 |
| $Na_2O$ (%) | 0.09 |
| $K_2O$ (%) | 0.20 |
| Insoluble Residue (%) | 0.37 |
| $CO_2$ (%) | 1.85 |
| Limestone (%) | 4.7 |
| $CaCO_3$ in limestone (%) | 90.16 |
| $C_3S$ (%) | 60.00 |
| $C_2S$ (%) | 11.00 |
| $C_3A$ (%) | 4.00 |
| $C_4AF$ (%) | 10.00 |
| Equivalent alkalies (%) | 0.23 |
| Blaine Fineness ($m^2/kg$)*** | 393.00 |

**Limits specified in ASTM C595

***Physical property

### 3.2.2. Fly ash

For the geopolymer mixtures, Class F fly ash was used as a binder. Table 3.2 shows the Chemical and physical properties of Class F fly ash. It is observed that the carbon emission represented by Loss on ignition is very low as compared to OPC which further supports the fact that manufacturing geopolymer concrete reduces carbon emission in the atmosphere.

**Table 3.2.** Chemical and physical properties of fly ash used

| **Item** | **Class F Fly Ash*** |
|---|---|
| $SiO_2$ (%) | 51.65 |
| $Al_2O_3$ (%) | 16.29 |
| $Fe_2O_3$ (%) | 5.63 |
| $SiO_2+Al_2O_3+Fe_2O_3$ | 73.57 |
| $SO_3$ (%) | 0.67 |
| CaO %) | 13.00 |
| MgO (%) | 4.26 |
| $Na_2O$ (%) | 3.23 |
| $K_2O$ (%) | 2.45 |
| Equivalent alkalies (%) | 1.63 |
| Loss on Ignition (%) | 0.10 |
| Fineness (+325 Mesh) (%)** | 21.29 |

*Limits specified in ASTM C618

**Physical property

### 3.2.3. Alkaline solution

The alkaline solution was a combination of sodium hydroxide and sodium silicate solution. They were selected because they were readily available and cheaper as compared to potassium based solutions. The sodium hydroxide (NaOH) of 98% purity was bought in flakes form from a local supplier, while the sodium silicate was bought in liquid form. The NaOH was dissolved in water as per desired Molar (M) concentration of the solution. For the purpose of this study 8M concentration of NaOH was prepared for each geopolymer mix. Therefore 320 grams of NaOH (8x40= 320 grams, where 40 is molecular weight of NaOH) per liter of solution. The two solutions that are NaOH solution and $Na_2SiO_3$ solution were mixed together one day before mixing since this is the time limit extracted from previous literature (D Hardjito & Rangan, 2005). The properties of Sodium silicate and Sodium hydroxide are provided in Table 3.3 and Table 3.4 below.

**Table 3.3.** Properties of Sodium silicate

| **Description** | **Quantity/Property** |
|---|---|
| State | Liquid |
| Sodium Silicate | 37 wt. % suspension in $H_20$<br>(28% Silica + 9% Sodium oxide |
| Water | 63% wt |
| PH | 11.3 approx |
| Specific gravity | 1.39g/cm$^3$ (20℃) |

**Table 3.4.** Properties of Sodium hydroxide

| Description | Quantity/Property |
|---|---|
| State | flake |
| Melting point | 318℃ |
| Boiling point | 1390℃ |
| Density | 2.13 |

### 3.2.4. Nano silica

The properties of Nano silica bought are given below in Table 3.5

**Table 3.5.** Properties of Nano silica

| Description | Quantity |
|---|---|
| State | Colloidal |
| Concentration | 50 wt. % suspension in $H_20$ |
| Surface area | 140 $m^2$/g |
| PH | 9.0 |
| Density | 1.4 g/ml at 25℃ |

### 3.2.5. Basalt fibers

Chopped basalt fibers were used which was 24 mm in length, 13µ in diameter and were treated with silane based coating.

### 3.3. Mixing and curing

Before the actual mixing of geopolymer concrete, several trial mixtures were conducted using the results from literature review. The purpose of trial mixes was to identify the behavior of fresh geopolymer concrete, to develop basic mix proportion, and to understand curing technique. It was found that geopolymer concrete was not suitable for water curing, since, the preliminary test results were below par and therefore was decided to oven cure them.

#### 3.3.1. Control mix

The standard procedure for making OPC concrete mix was followed per ASTMC192. The coarse aggregate was placed in the mixer followed by 2/3 parts water. Fine aggregates and cement were then added and the mixer was switched on for three minutes. 1/3 parts water was added periodically. After mixing for three minutes the mix was rested for a period of three minutes and the mixer was covered with a polythene sheet so that water doesn’t evaporate. The machine was finally switched on again for two more minutes. The concrete was poured into molds and compacted by applying 25 manual strokes per layer in three equal layers. After a period of 24h, the molds were demolded, and the hardened concrete was immersed in the water tank for curing and subsequently removed after 7 and 28 days to conduct tests.

#### 3.3.2. Geopolymer mix

Coarse aggregates, fine aggregates, fly ash and other additives (Basalt fibers and silica, these were added depending on the type of mix) were poured into the mixer as shown in Figure 3.1 and dry mixed for about two minutes. The alkaline solution, i.e. the sodium silicate solution and sodium hydroxide solution, which was prepared 24h before mixing was then added followed by water (if any). The wet mix continued again for three more minutes.

The fresh geopolymer concrete had a glossy appearance and had a stiff consistency, therefore it was needed to pour it in molds quickly. Before pouring slump flow test was conducted to measure workability. Compaction of the geopolymer concrete during pouring was done again using 25 manual strokes. Ten samples for each mix was prepared to perform different test on them.

The molded concrete was left at room temperature for 24 h and then demolded and placed inside an oven for 24 h for elevated curing as shown in Figure 3.2. After oven curing, the hardened concretes were stored at room temperature before being taken to conduct mechanical tests.

**Figure 3.1.** Concrete mixing in lab

**Figure 3.2.** Oven curing of Geopolymer concrete samples

### 3.4. Mixture proportions

After conducting several trial mixes to get familiarize with the properties of geopolymer concrete, the mixture proportions for the concrete manufacturing was developed. Type I/II cement was selected as the binder for OPC concrete mix and was to serve as a control against Class F Fly Ash geopolymer concrete. To investigate the effect of geopolymer concrete with other additives, it was decided to prepare five mixes which are as follows:

1. Control Type I/II Cement (Control)
2. Fly Ash Geopolymer concrete (FA)
3. Fly Ash Geopolymer concrete with 0.25% v/v Basalt Fibers (Addition) (FA + Fiber)
4. Fly Ash Geopolymer concrete with 5% Nano silica (Replacement) (FA + Silica)
5. Fly Ash Geopolymer concrete with 5% Nano silica (Replacement) and 0.25% v/v basalt fibers (addition) (FA+ Fiber + Silica)

Apart from these five mixtures, a sixth mix was prepared to test whether fly ash based geopolymer concrete provide same benefits in ambient curing conditions.

### 3.5. Test methods

It was decided to conduct seven different test on the manufactured OPC and Geopolymer concrete. Three tests were conducted on fresh concrete while the remaining four on hardened concrete. These tests are discussed below.

#### 3.5.1. Slump/slump flow test

To measure the workability of fresh concrete, slump test for OPC concrete and slump flow test for geopolymer concretes (ASTM C143) were conducted. This was performed using standard cone test apparatus. For control mix, the difference in height between the top of the cone and top of concrete was measured. For geopolymer mixes, the cone apparatus was placed on top of a wooden platform. When the cone was pulled, the concrete spread towards the side. Once the concrete comes to the rest position, the diameter of the spread was measured and recorded as shown in Figure 3.3

**Figure 3.3.** Spread measurement of geopolymer concrete

### 3.5.2. Air content

To measure the air content of freshly mixed concrete (ASTM C231) an air meter was used consisting of a measuring bowl, pressure gauge, and cover assembly as shown in Figure 3.4. The fresh concrete was placed in the bowl in three equal layers. Each layer was rodded 25 times for compaction. After covering the bowl and closing the petcocks, the main air valve between the air chamber and the measuring bowl was opened. To relieve restraints, the bowl was tapped lightly with a mallet and the air percentage was recorded from the pressure gauge.


**Figure 3.4.** Concrete air meter

### 3.5.3. Semi-Adiabatic calorimetry

Semi-adiabatic calorimetry tests were used to investigate the hydration characteristics of the concrete mixtures (ASTM C1753). A calorimetric container was used which connected to the laptop using a USB cable. A data logger software called Calcommander was used for data acquisition. The fresh concrete sample was poured in cylindrical molds and placed inside the calorimetric container as shown in Figure 3.5. The samples were left inside the container for 48 hours to investigate the development of temperature in concrete.

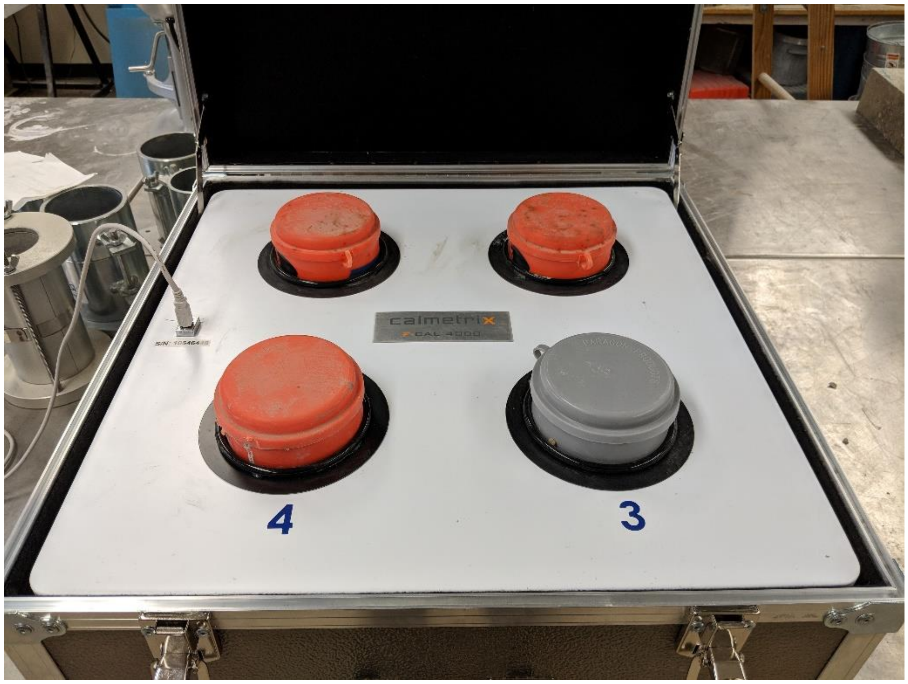


**Figure 3.5.** Temperature testing using Calorimeter

### 3.5.4. Compressive strength

The compressive strength of the control and geopolymer cylinders were conducted through a universal testing machine in accordance with ASTM C39 standard as shown in Figure 3.6. The compressive load was axially applied continuously within the prescribed range until failure occurred. The cylinders were tested on 1, 7 and 28 days. In total, nine cylinders from each mix design were tested.


**Figure 3.6.** Compressive strength test equipment

### 3.5.5. Drying shrinkage

The moisture loss in concrete's fine pores results in shrinkage. Drying shrinkage was tested according to ASTM C157 standard using a Humboldt's digital length comparator as shown in Figure 3.7. The concrete specimens were molded in 12"x4" prism, placed in two equal layers and rodded manually. The specimens were tested on 1, 4, 7, 14 and 28 days and the difference in the lengths was recorded.

**Figure 3.7.** Shrinkage test

### 3.5.6. Resistivity

The resistivity of the cylinders was tested according to ASTM C1760 standard using a concrete resistivity meter as shown in Figure 3.8. Two samples from each mix design were tested on 1, 7 and 28 days.


**Figure 3.8.** Resistivity meter

### 3.5.7. Scanning electron microscopy (SEM)

SEM tests were conducted to ascertain the microstructure properties of the concrete and to understand the development of geopolymer concrete on a microstructural level. The collected SEM images were very detailed which helped in understanding the chemistry behind geopolymer concrete for all the different mixes.

# 4. RESULT AND DISCUSSION

## 4.1. Introduction

This chapter provides a detailed analysis of the tests performed and provides comparative examination between control and geopolymer concrete through seven (7) different parameters: Slump/slump flow Test, Air content, Compressive strength, Drying Shrinkage, Resistivity, Semi-Adiabatic Calorimetry, Scanning Electron Microscopy (SEM). Only hardened properties of room temperature mixture were studied since it had the same mix design as the normal fly ash based geopolymer concrete with oven curing and would most probably result in same fresh properties.

## 4.2. Fresh properties

### 4.2.1. Slump/slump flow test

The slump results of the fresh concrete are given in Table 4.1. It was observed that the initial slump of geopolymer concrete was extremely high and was measured in terms of diameter spread. This is in line with previous literature (D Hardjito & Rangan, 2005). It was observed that among the geopolymer mixes, Fly Ash Geopolymer concrete with 0.25% basalt Fiber had the least spread. This is in lines with prior literature. Previous studies  have reported the decrease in workability of concrete with the addition of fiber (Topçu & Canbaz, 2007). This is likely due to a physical binding effect of aggregates by the fibers.

**Table 4.1.** Slump measurement of concrete samples

| Mix | Spread (cm) |
|---|---|
| Control | 11.43 |
| FA | 48.26 |
| FA + Fiber | 35.56 |
| FA+ silica | 40.34 |
| FA + Fiber + Silica | 38.26 |

#### 4.2.2. Air content

Table 4.2 shows the percentage of air content in the fresh concrete. It is observed that geopolymer concrete had a relatively low content of air as compared to control mix which can be verified through previous literature (Kong & Sanjayan, 2010).

**Table 4.2.** Air content of concrete samples

| Mix | Air percentage % |
|---|---|
| Control | 5.2 |
| FA | 3.2 |
| FA + Fiber | 3.0 |
| FA+ silica | 3.2 |
| FA + Fiber + Silica | 3.4 |

#### 4.2.3. Semi-Adiabatic calorimetry

The purpose of Semi-adiabatic calorimetry was to record variation in temperature of concrete with respect to time. The temperature was recorded over a 48 hour period and plotted against time as shown in Figure 4.1. There was a stark difference in the readings of control concrete and geopolymer concrete. The temperature of control concrete accelerated after mixing (indicating hydration of aluminates) followed by a decrease during the deceleration period (formation of ettringite) and a plateau formation (silicate hydration) and near constancy thereafter. This is typical of ordinary portland cement concrete. Whereas, geopolymer samples peaked fairly quickly and then followed a relatively constant temperature. Geopolymer mix incorporating nano silica had the highest temperature reaching a peak of 78.98F at 15 hours. The incorporation of nano silica, due to its increased surface area, enhances the rate of reaction material (Khaloo, Mobini, & Hosseini, 2016). This is reflected in the temperature readings of nano silica-based geopolymer concrete.

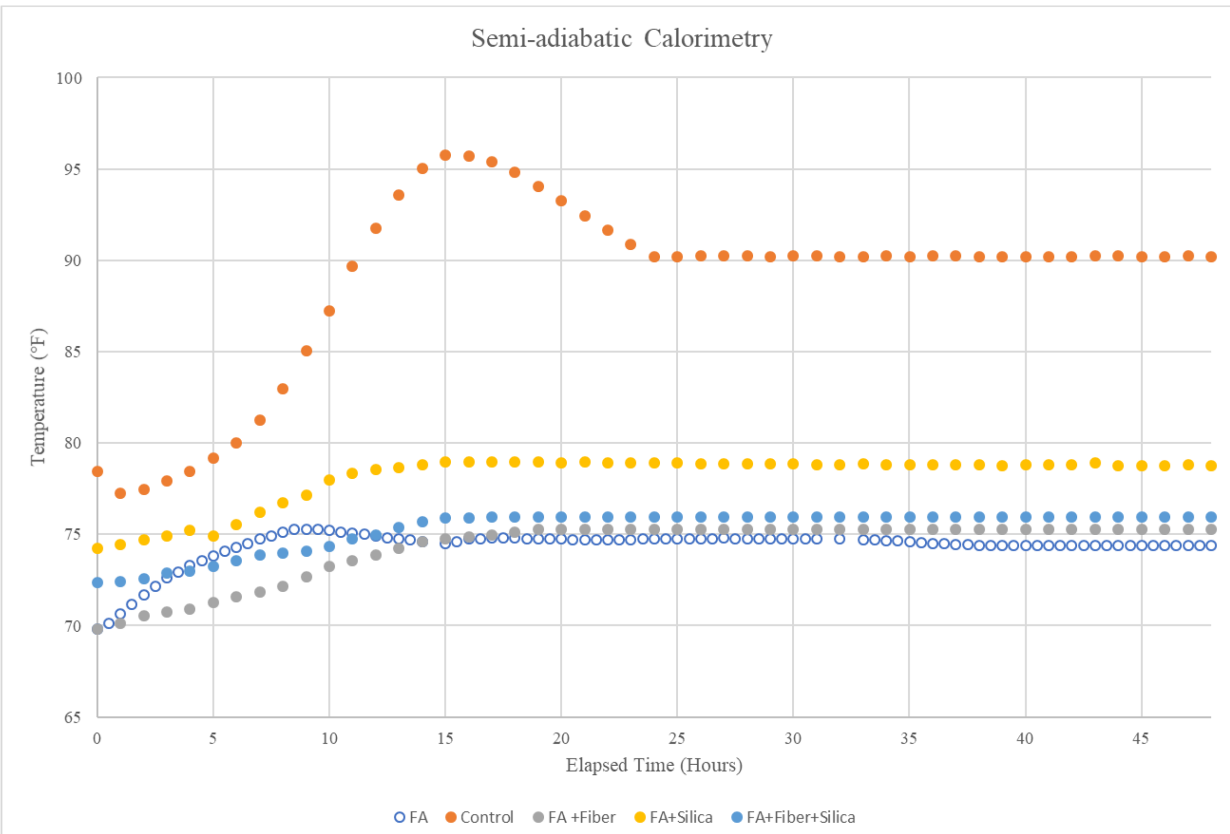


**Figure 4.1.** Calorimetry curves of OPC and Geopolymer concrete samples

## 4.3. Hardened properties

### 4.3.1. Compressive strength

Table 4.3 shows the compressive strengths of control and geopolymer mixes on 1, 7 and 28 days measured in terms of MPa. It is observed that geopolymer concretes perform much better than plain cement concrete counterpart as evident in Figure 4.2. Among all the geopolymer mixes, Fly Ash Geopolymer concrete with 5% Nano silica (replacement) has the highest average strength on all three days followed by fly ash geopolymer concrete with 0.25% v/v basalt fibers. It is also important to mention that normal fly ash based geopolymer concrete and geopolymer concrete with 5% Nano silica had similar compressive strength on the 1st day, but FA+ silica had a much higher increase in strength on 7 and 28 days.

**Table 4.3.** Compressive strength measurements of concrete samples

| Mix | Solution-to-cementitious materials ratio | Compressive Strength (Mpa) | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | 1 Day | | | Average | 7 Days | | | Average | 28 Days | | | Average |
| **Control** | 0.42 | 15.7 | 14.8 | 13.6 | **14.7** | 27.1 | 23.1 | 26.7 | **25.6** | 31.7 | 28.7 | 29.1 | **29.8** |
| **FA** | 0.35 | 17.5 | 18.3 | 16.3 | **17.4** | 31.5 | 32.7 | 32.6 | **32.3** | 35.8 | 41.6 | 33.7 | **37.0** |
| **FA + Fiber** | 0.35 | 16.4 | 15.4 | 17.4 | **16.4** | 31.4 | 37.6 | 38.1 | **35.7** | 40.5 | 38.2 | 45.8 | **41.5** |
| **FA+ silica** | 0.35 | 17.7 | 18.2 | 17.2 | **17.7** | 33.4 | 36.1 | 41.5 | **37.0** | 45.6 | 43.6 | 53.7 | **47.6** |
| **FA + Fiber + Silica** | 0.35 | 18.2 | 16.3 | 16.1 | **16.9** | 29.6 | 31.4 | 37.6 | **32.9** | 41.3 | 33.7 | 38.9 | **38.0** |
| **FA + RT** | 0.35 | 5.5 | 6.3 | 6.1 | **6** | 13.7 | 13.2 | 14 | **13.6** | 19.8 | 20.2 | 20 | **20** |

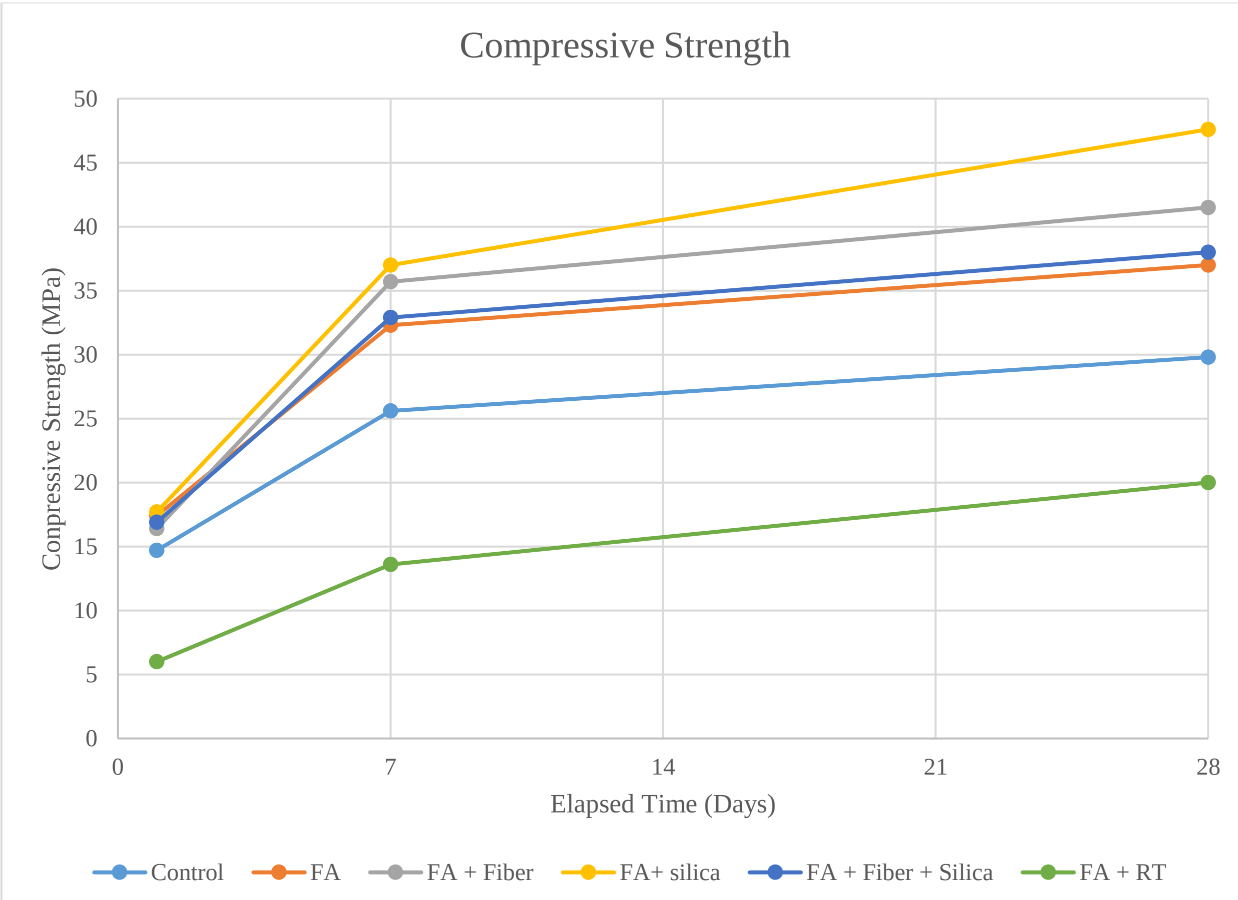


**Figure 4.2.** Compressive strength of concrete samples

### 4.3.2. Drying shrinkage

Table 4.4 shows the results for shrinkage of the concrete mixtures measured in terms of negative microstrain. The shrinkage readings were taken at 1, 4, 7, 14 and 28 days. The geopolymer concrete beams provide the lowest shrinkage readings after 28 days with FA based geopolymer concrete having the lowest reading of 257 followed closely by nano silica-based geopolymer concrete which has a reading of 265 as seen in Figure 4.3

**Table 4.4.** Free shrinkage readings of concrete samples

| Mix \ Elapsed time(days) | 0 | 1 | 4 | 7 | 14 | 28 |
|---|---|---|---|---|---|---|
| Control | 0 | 87 | 131 | 195 | 243 | 320 |
| FA | 0 | 30 | 97 | 156 | 197 | 257 |
| FA + Fiber | 0 | 42 | 101 | 171 | 211 | 296 |
| FA+ silica | 0 | 54 | 117 | 164 | 201 | 265 |
| FA + Fiber + Silica | 0 | 57 | 120 | 178 | 216 | 301 |
| FA + RT | 0 | 58 | 115 | 175 | 220 | 310 |

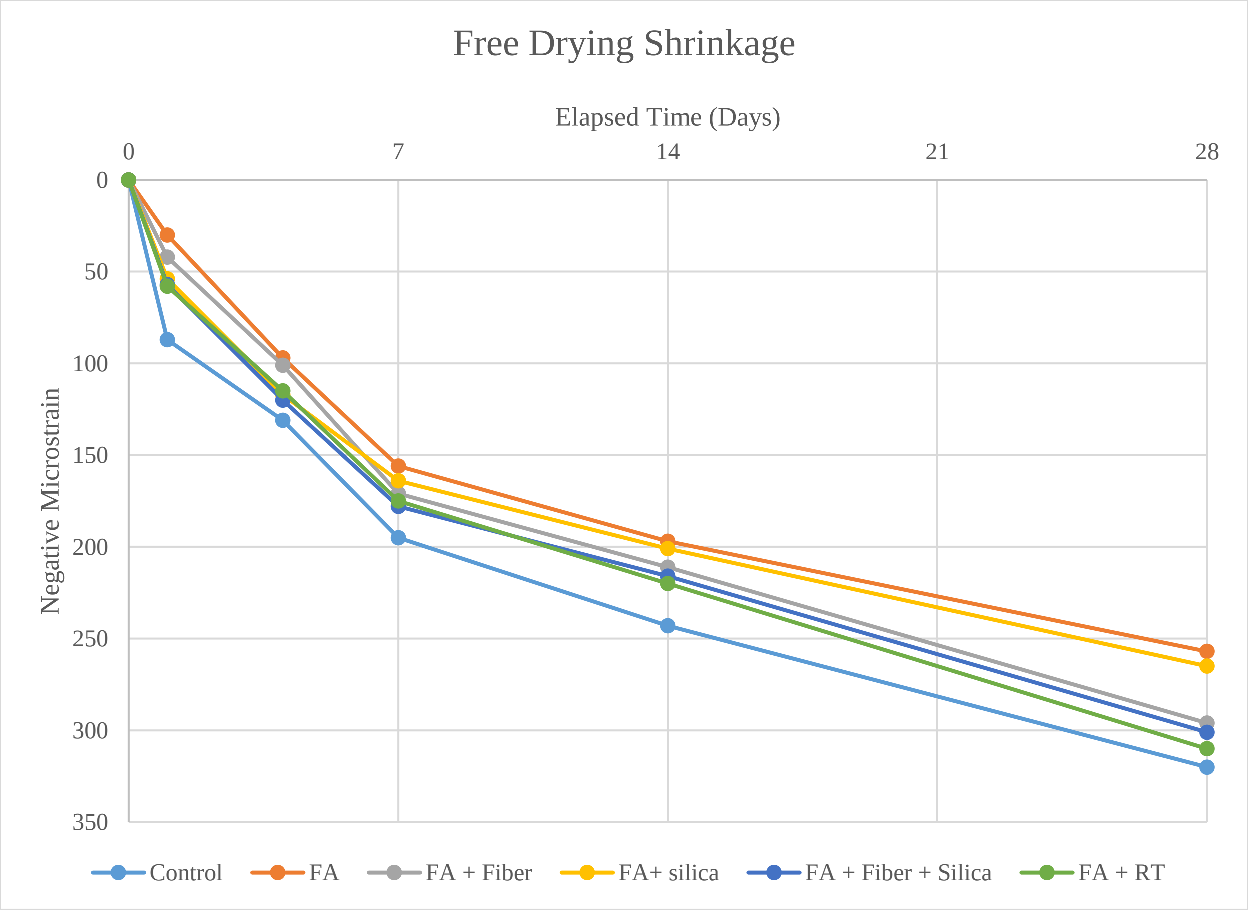


**Figure 4.3**. Shrinkage readings of concrete samples

#### 4.3.3. Resistivity

Table 4.5 shows the results for the electrical resistivity of the concrete mixtures measured in terms of kΩcm. It is observed that geopolymer mixes have a higher electrical resistivity compared to OPC concrete with FA + silica having the highest resistivity on 28 days. It is noted again that FA + silica concrete didn’t have the highest resistivity at 1 day but it got considerably higher on 7 and 28 days as evident in Figure 4.4

**Table 4.5.** Resistivity readings of concrete samples

| Mix | Solution-to-cementitious materials ratio | Resistivity (kΩcm) | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | 1 Day | | Average | 7 Days | | Average | 28 Days | | Average |
| Control | 0.42 | 6.6 | 7.1 | 6.9 | 7.9 | 8.4 | 8.2 | 11.4 | 11.1 | 11.3 |
| FA | 0.35 | 7.2 | 7.7 | 7.5 | 9.5 | 9.1 | 9.3 | 13.1 | 13.2 | 13.2 |
| FA + Fiber | 0.35 | 8.1 | 8.9 | 8.5 | 9.1 | 9.8 | 9.5 | 11.7 | 12.5 | 12.1 |
| FA+ silica | 0.35 | 7.7 | 8.1 | 7.9 | 10.3 | 9.7 | 10.0 | 13.7 | 14.8 | 14.3 |
| FA + Fiber + Silica | 0.35 | 8.1 | 6.8 | 7.5 | 10.7 | 9.1 | 9.9 | 13.1 | 13.8 | 13.5 |
| FA + RT | 0.35 | 6.8 | 7.2 | 7.0 | 8.2 | 8.8 | 8.5 | 11.6 | 11.9 | 11.8 |

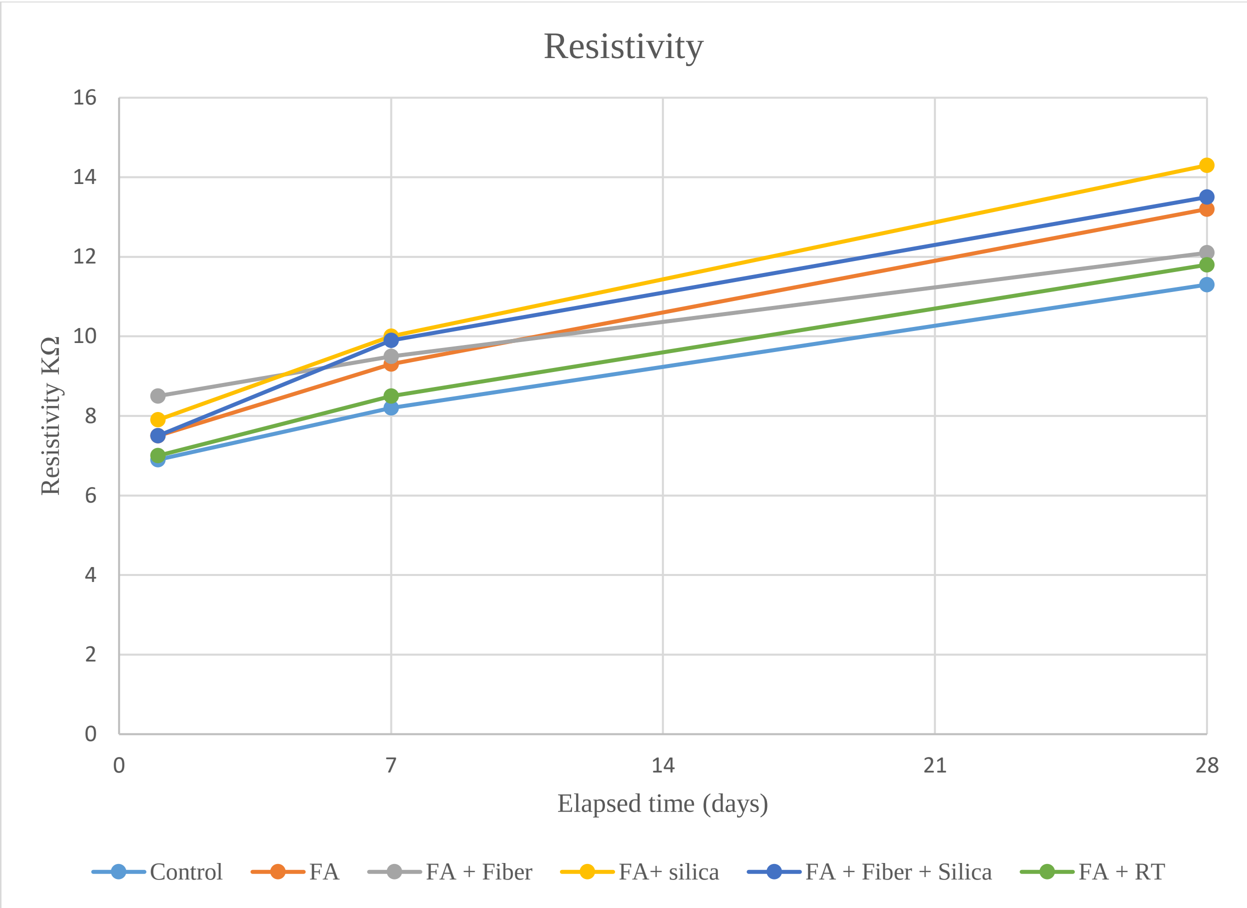


**Figure 4.4.** Resistivity curve of concrete samples

### 4.3.4. Scanning electron microscopy (SEM) and EDS analysis

#### 4.3.4.1. Fly ash Geopolymer concrete (FA)

Figure 4.5 illustrates the microstructural analysis of FA based geopolymer concrete. The microstructure of Geopolymer concrete consists of a sphere like the structure of different sizes. These spheres are usually hollow and may contain other particles in their interiors (Guo, Shi, & Dick, 2010). Since the experimental work is based on low calcium fly ash, it is observed that there is less content of Calcium as compared to control concrete as observed in EDS analysis. The fly ash appears to be gray in color which makes the geopolymer concrete darker as compared to OPC.

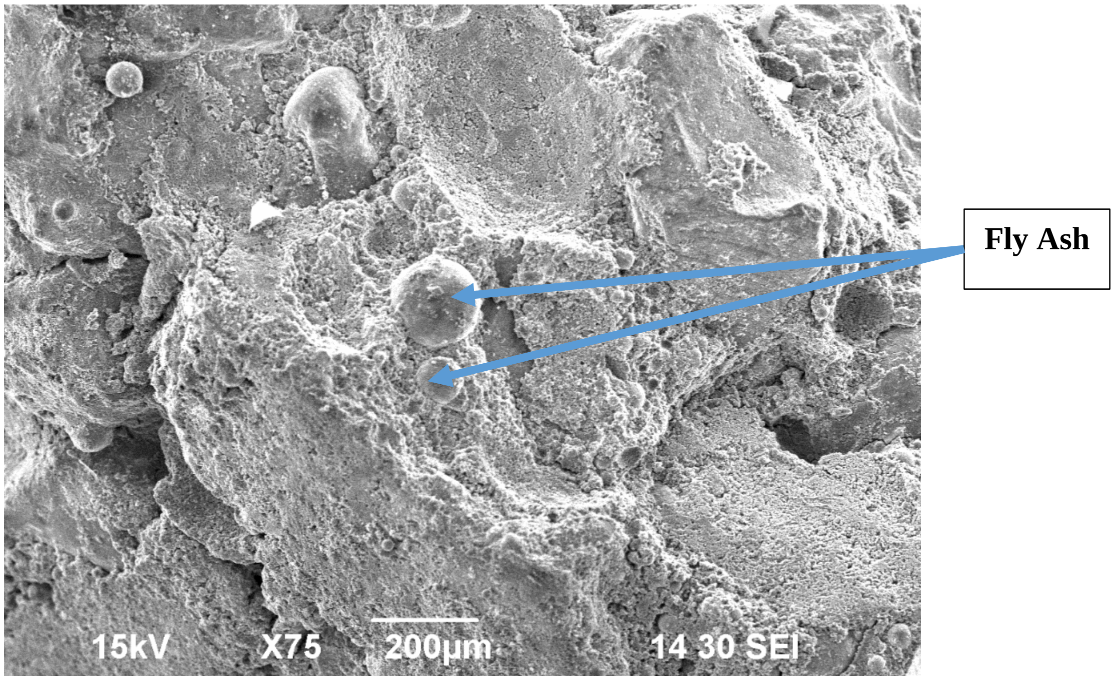


**Figure 4.5.** SEM image showing fly ash identification

Figure 4.6 shows the microstructure of geopolymer concrete taken at 250x magnification. The dense deposits on the surface of fly ash is a gel-like substance and are known as geopolymer binder(S. Alehyen, M. EL Achouri, 2017), while Figure 4.7 shows that FA based geopolymer concrete has a porous, heterogeneous structure with some amount of non-reacted fly ash.

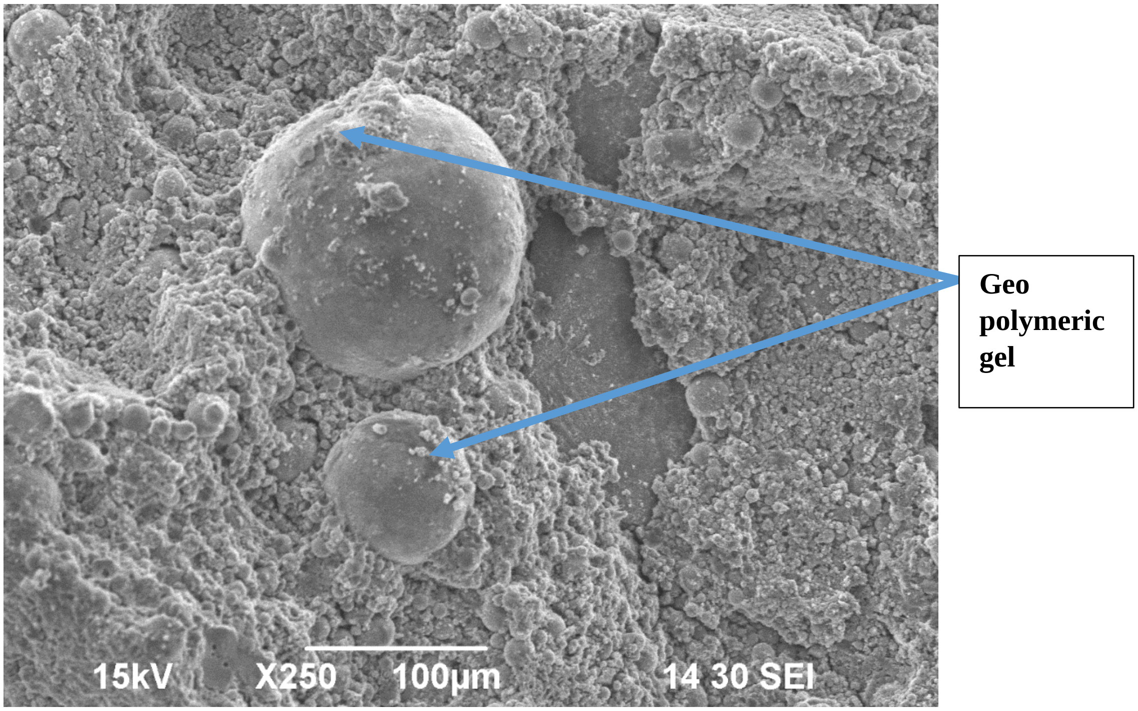


**Figure 4.6.** SEM image showing geopolymeric gel

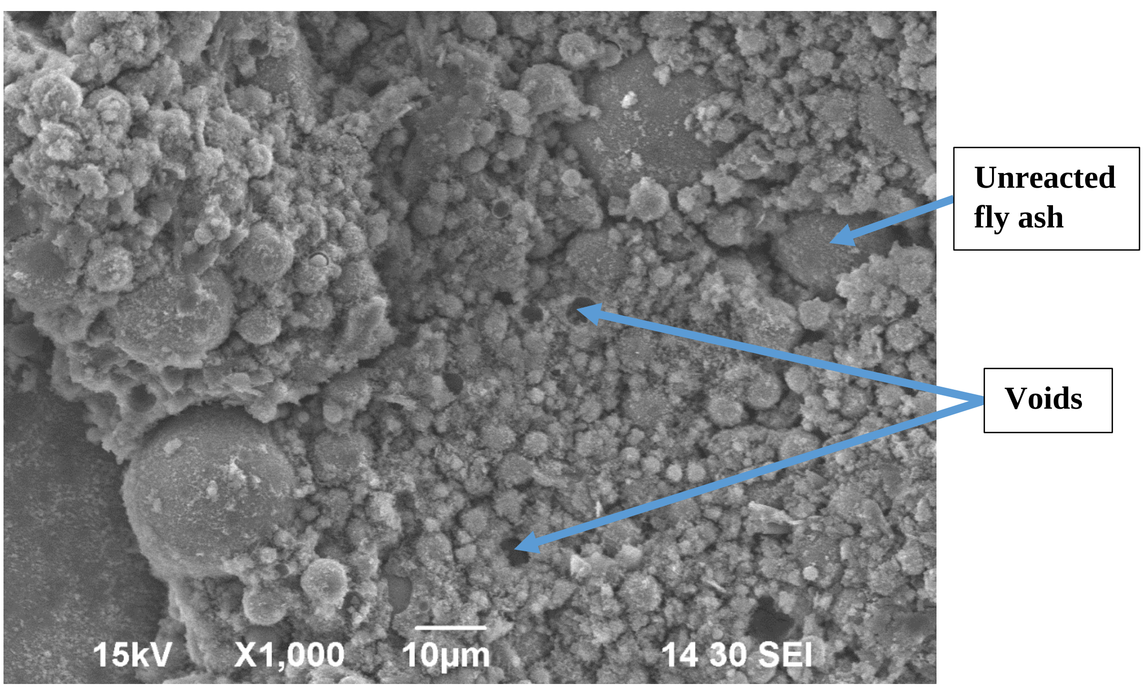


**Figure 4.7.** SEM image showing voids and unreacted fly ash

EDS analysis was conducted at four different spots of fly ash based geopolymer concrete sample as shown in Figure 4.8. The major elements observed through EDS analysis were Si, Al and Ca, but some quantities of Fe, K, Mg were also recorded as shown in Figure 4.9. The fine aggregates and Fly ash may be responsible for the presence of silica and Fe, K and Mg respectively (Nagalia et al., 2016)

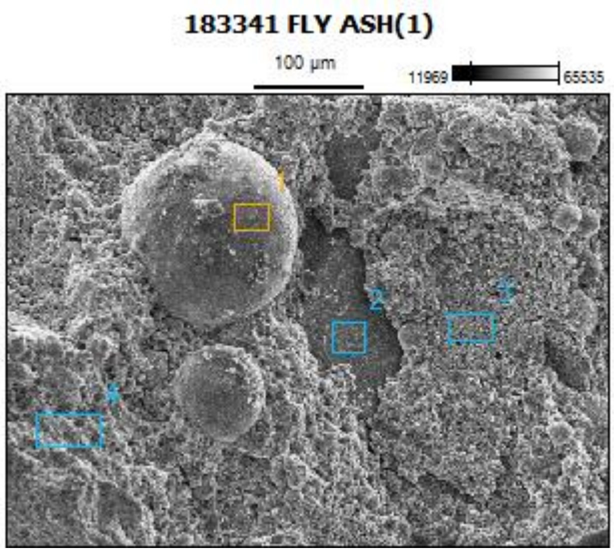


**Figure 4.8.** SEM image for EDS analysis for FA based geopolymer concrete

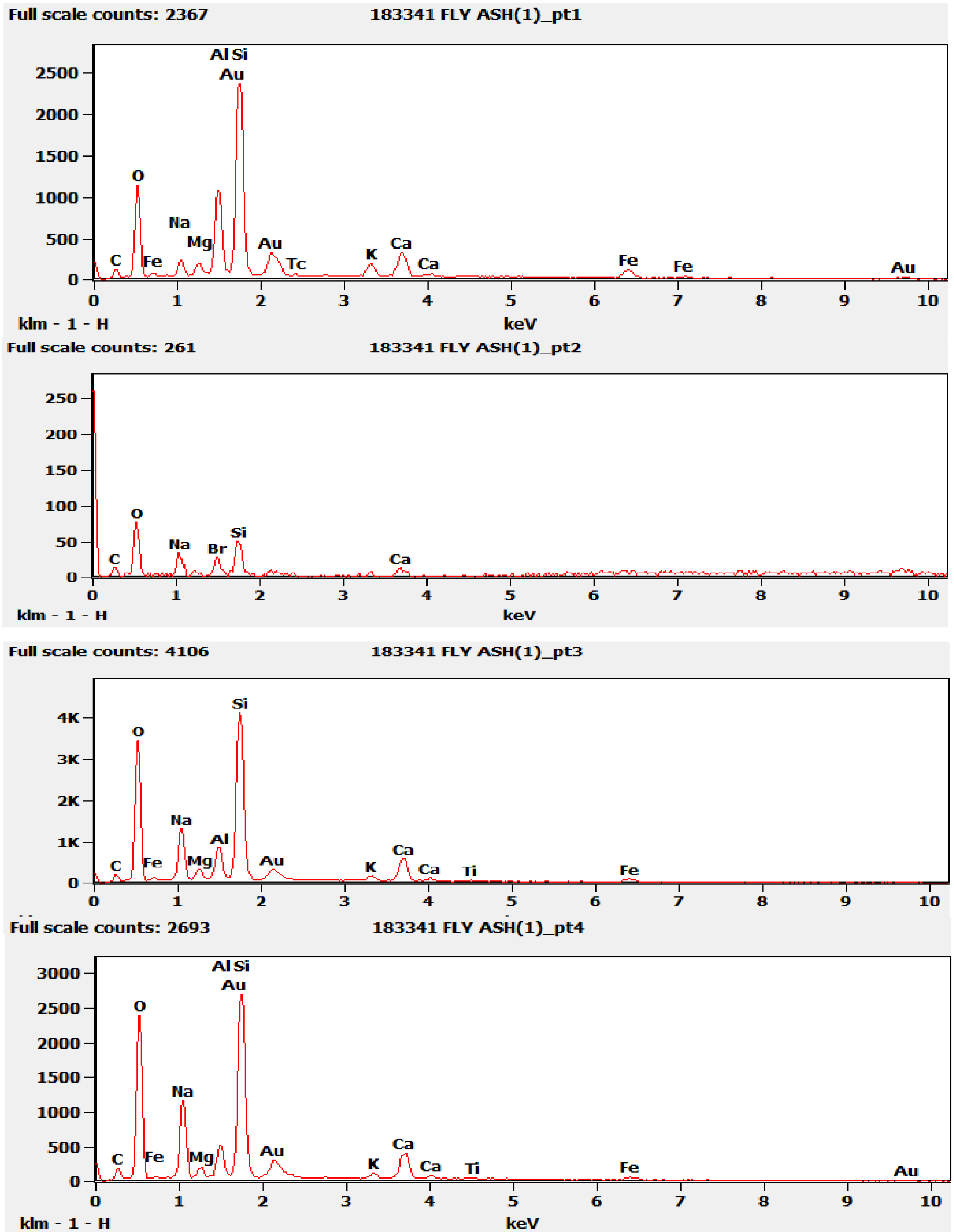


**Figure 4.9.** EDS graphs for FA based geopolymer concrete

#### 4.3.4.2. Fly ash Geopolymer concrete with 5% Nano silica

Figure 4.10 and Figure 4.11 shows the SEM images of geopolymer concrete incorporated with 5% nano-silica. Unreacted fly ash particles can be observed but the surface generally looks compact, although few micro cracks can be detected. There appear to be fewer voids as compared to FA based geopolymer concrete which may indicate that nano silica increased the compactness of geopolymer concrete by filling the pores (Deb, Sarker, & Barbhuiya, 2015). The formation of secondary C-S-H gels might also lead to fewer pores (Shaikh, Supit, & Sarker, 2014). A magnification of 5000x shows that this concrete seems to have a crystalline structure as shown in Figure 4.12.

Figure 4.13 Figure 4.14 shows elements identified through EDS analysis. A typical practice in EDS analysis is to observe the difference in brightness along the subject area to decode the chemical composition. High quantities of calcium, sodium, aluminum, and silica. Some amount of titanium is also recorded which may have been extracted from silica

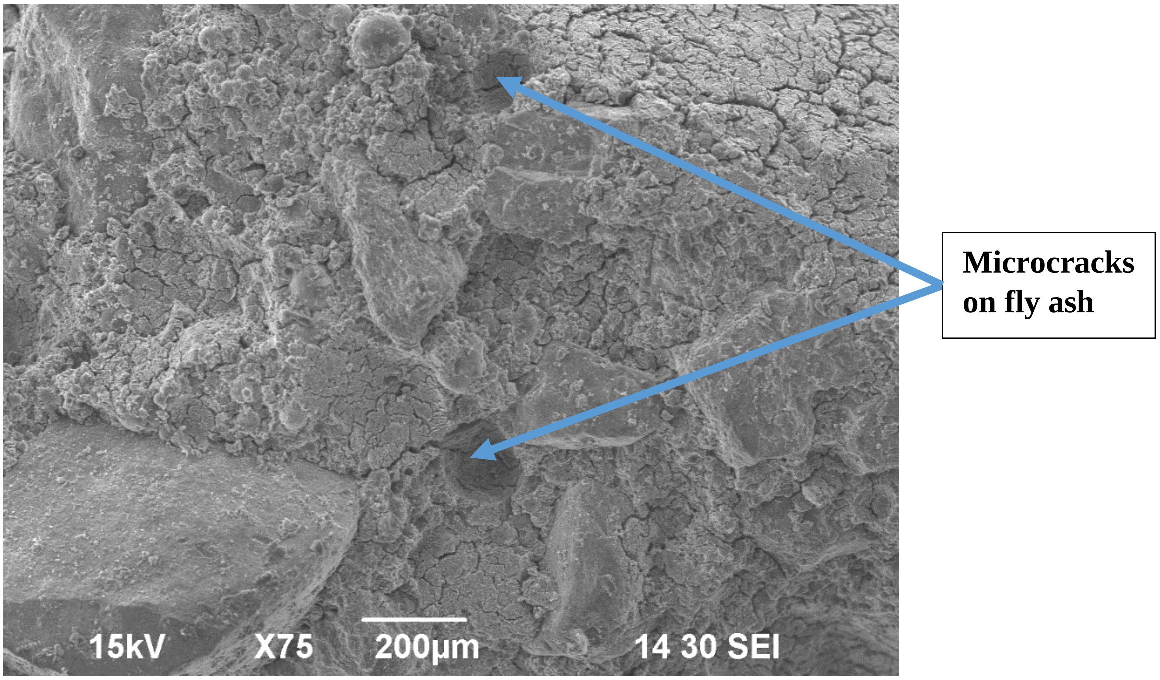


**Figure 4.10.** Cracks on Geopolymer concrete (5% nano Silica)

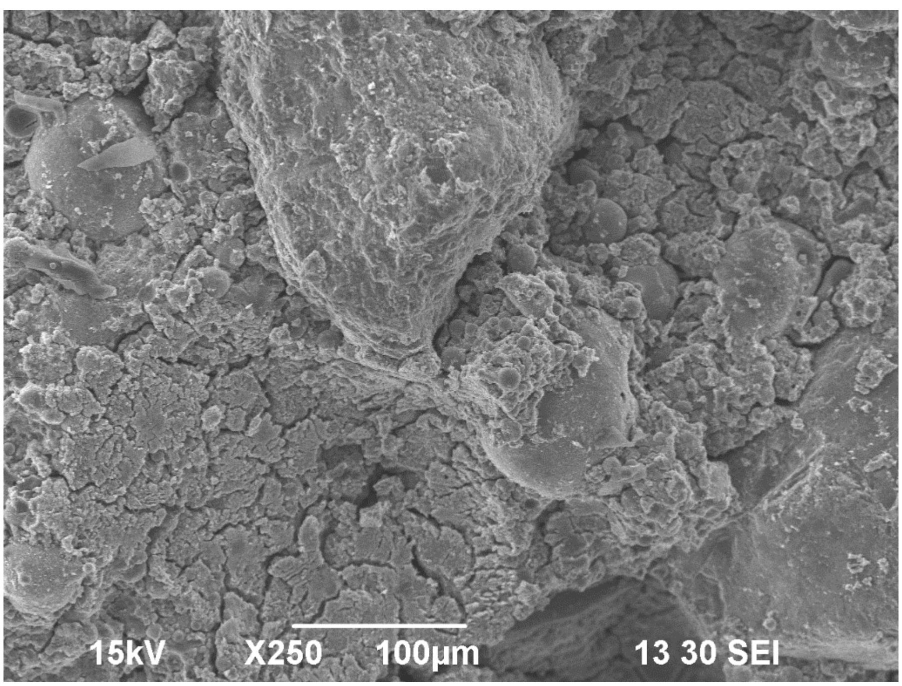


**Figure 4.11.** x 250 magnification of Geopolymer concrete (5% nano silica)

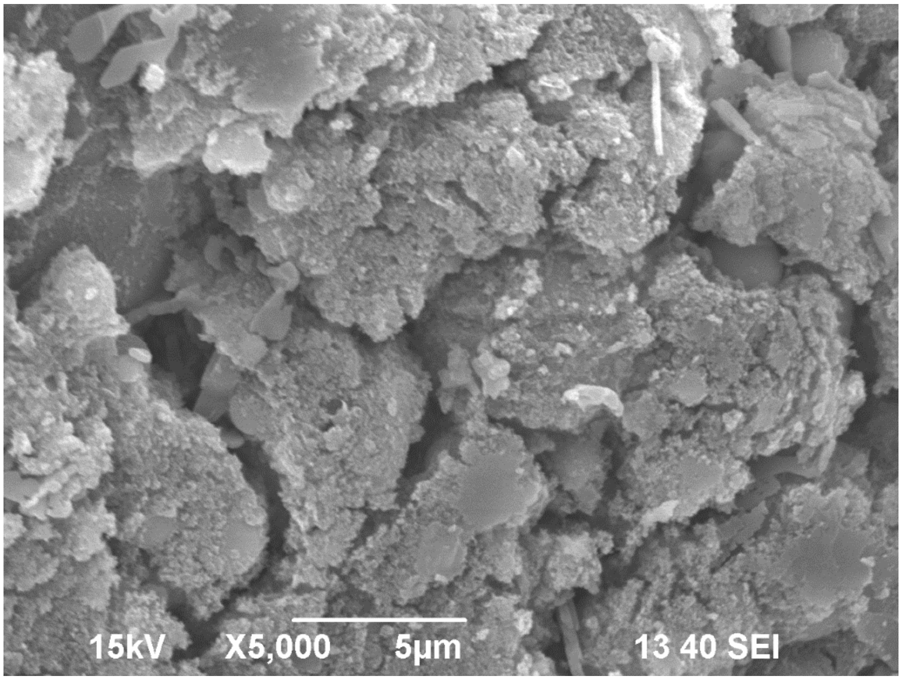


**Figure 4.12.** Crystalline structure in Geopolymer concrete (5% nano silica)

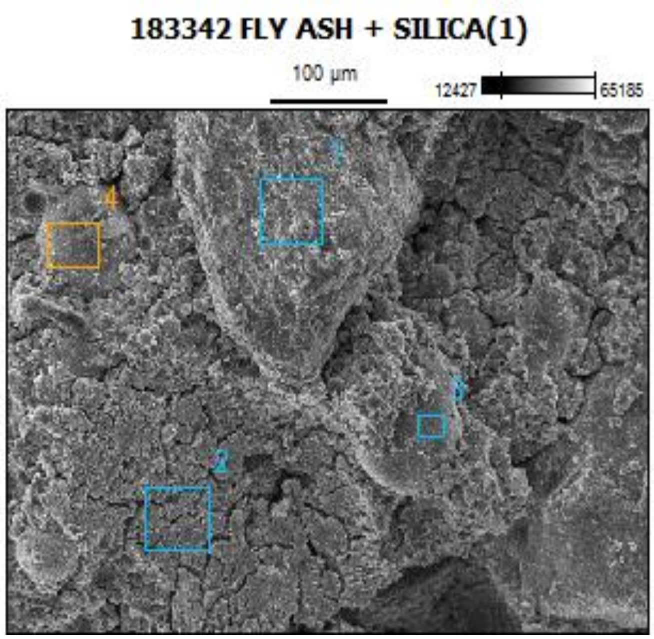


**Figure 4.13.** SEM image for EDS analysis for geopolymer concrete (5% nano silica)

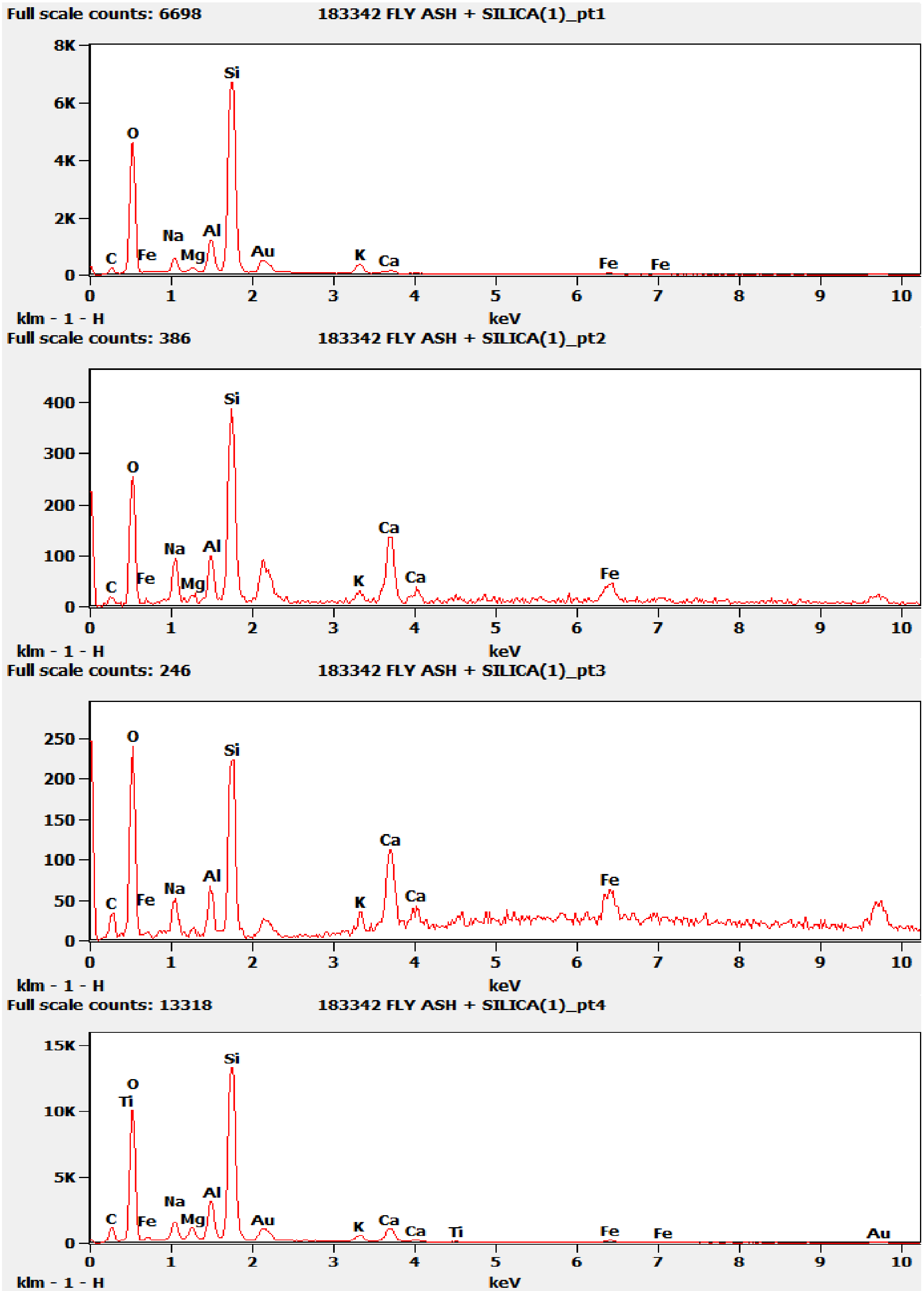


**Figure 4.14.** EDS graphs for geopolymer concrete (5% nano silica)

#### 4.3.4.3. Fly ash Geopolymer concrete with 0.25% v/v Basalt fibers

The SEM images of geopolymer concrete with Basalt fibers is shown in Figure 4.15. The microstructure of the GP concrete looks a heterogeneous, compact and dense structure with basalt fibers embedded as shown in Figure 4.16. The reaction product was seen deposited on fly ash and basalt fibers as shown in Figure 4.17. The main chemical compositions of basalt fibers are $SiO_2$ and CaO (Timakul, Rattanaprasit, & Aungkavattana, 2016).

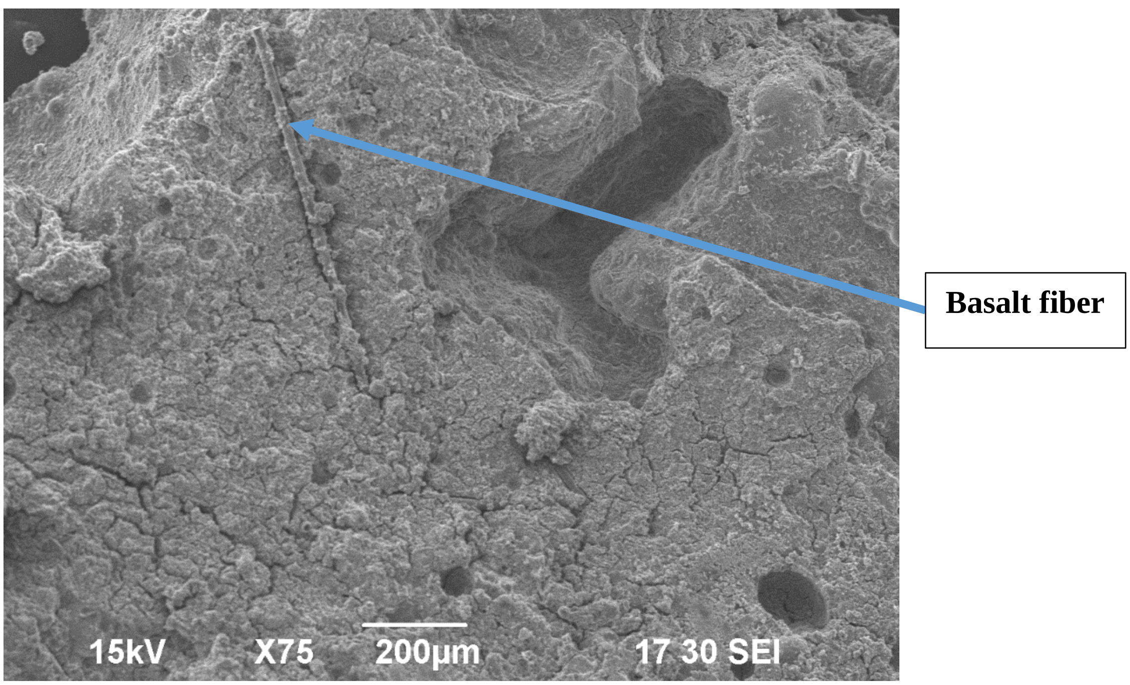


**Figure 4.15.** SEM image of geopolymer concrete (0.25% Basalt fibers)

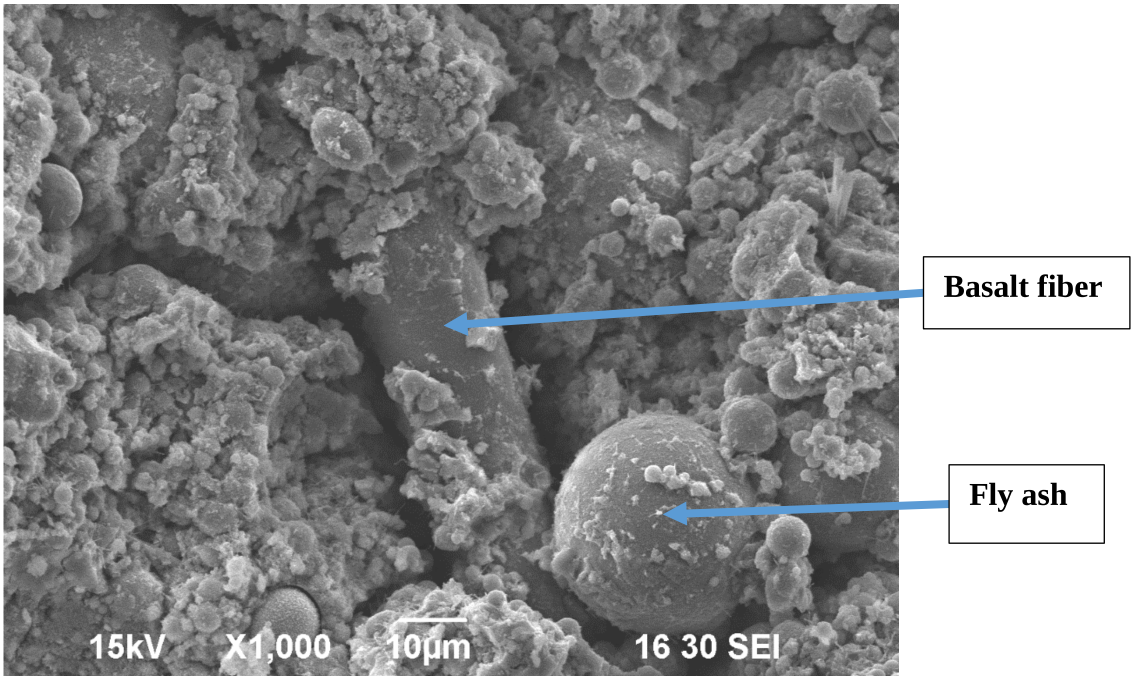


**Figure 4.16.** SEM image of geopolymer concrete with Basalt fiber embedded

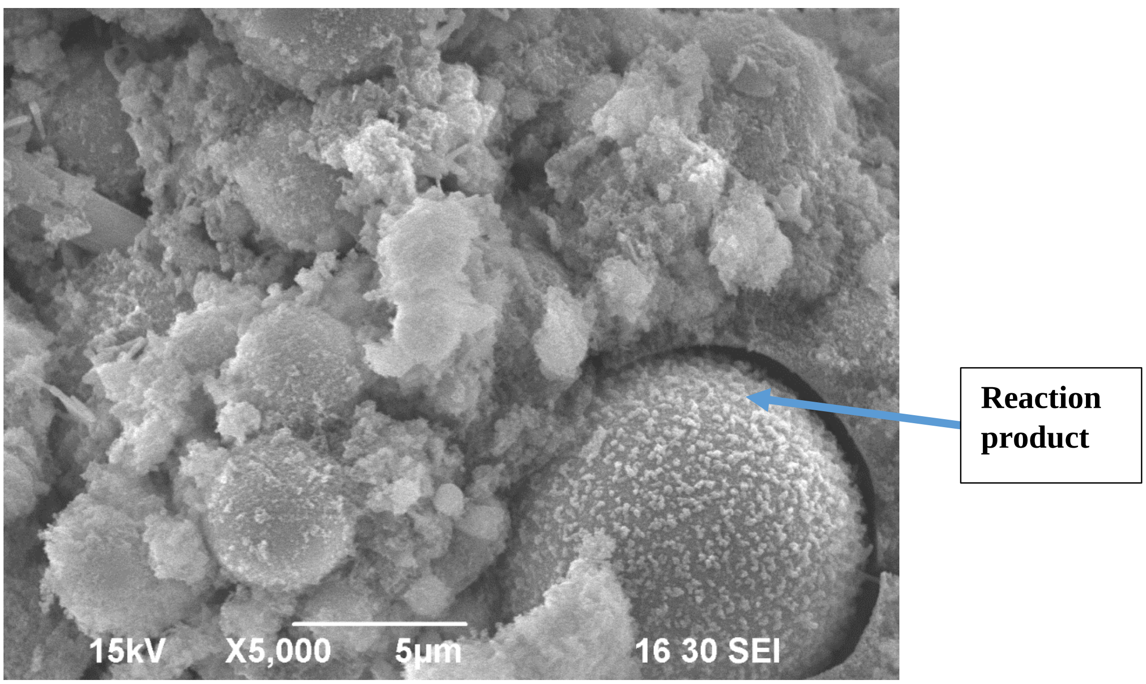


**Figure 4.17.** Reaction product on geopolymer concrete (0.25% Basalt fiber)

The image in Figure 4.18 shows the geopolymer concrete incorporating basalt fiber analyzed for chemical deposits at four different places on its surface. The EDS analysis shows higher presence of Ca, Na, Si, Al in the system as shown in Figure 4.19, which helped in the development of reaction product calcium silicate hydrate CSH, calcium alumino silicate hydrate CASH (Punurai, Kroehong, Saptamongkol, & Chindaprasirt, 2018)

The increasing ratios of Si/Al and Ca/Si could imply the formation of calcium silicate compound. This compound helped in filling small voids resulting in increased compressive strength. (Punurai et al., 2018)

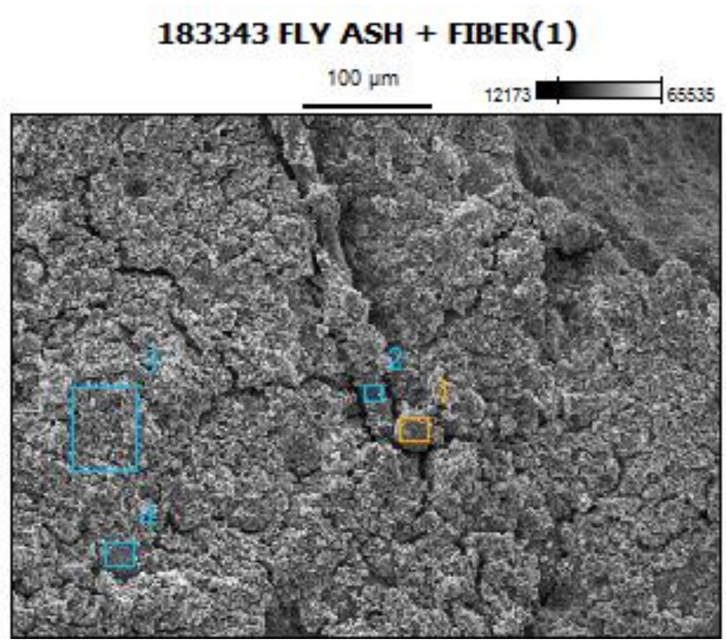


**Figure 4.18.** SEM image for EDS analysis for geopolymer concrete (0.25% Basalt fibers)

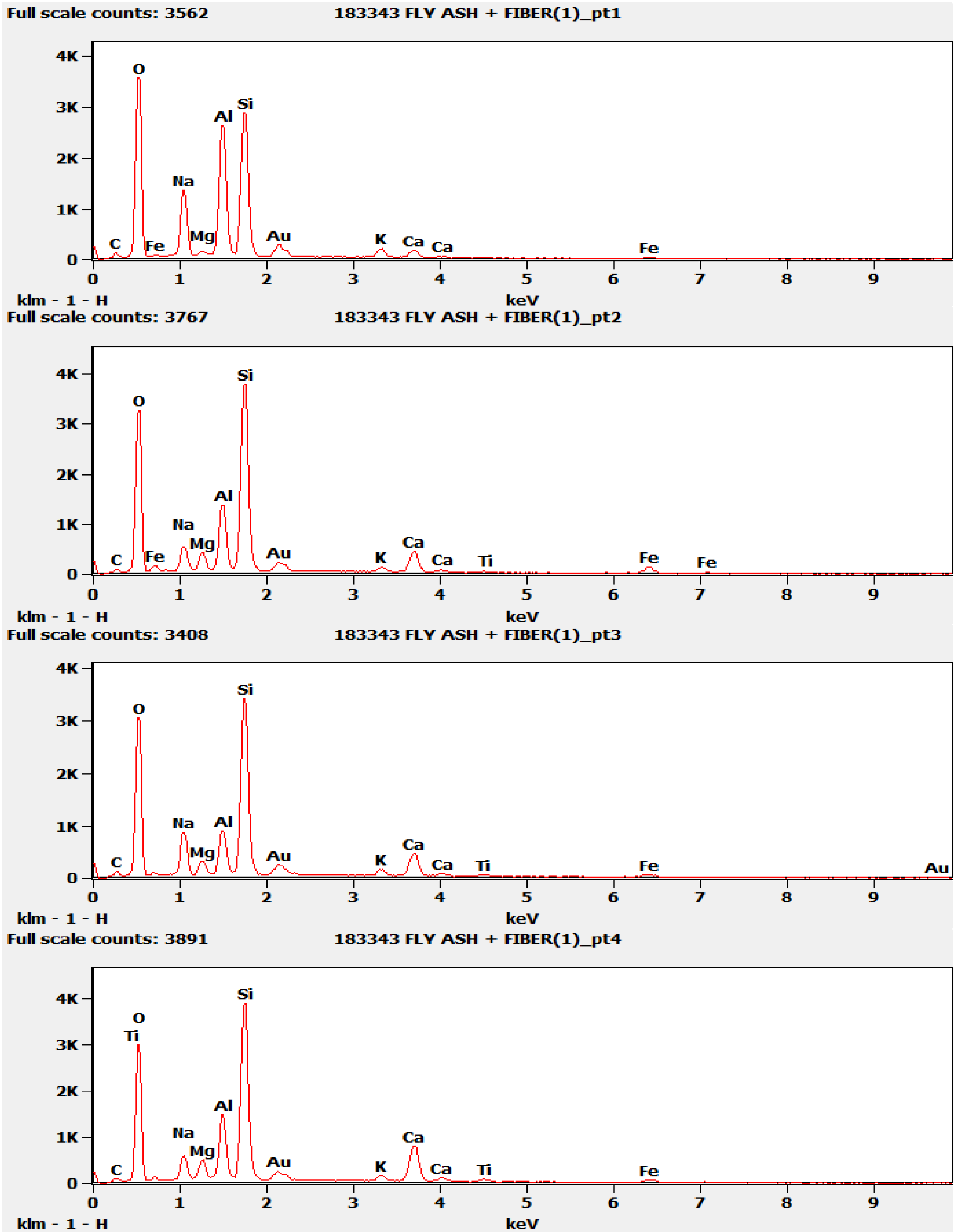


**Figure 4.19.** EDS graphs for geopolymer concrete (0.25% Basalt fibers)

#### 4.3.4.4. Fly ash Geopolymer concrete with 5% Nano silica and 0.25% v/v Basalt fibers

Figure 4.20 Figure 4.21 provides SEM images for geopolymer concrete incorporating basalt fiber and nano silica. Basalt fibers can be seen embedded in concrete and few microcracks are also observed on fly ash. Calcium silicate deposits can be observed as was seen in geopolymer concrete incorporating just the basalt fibers. This sample also seems to have a crystalline structure as evident in Figure 4.22.

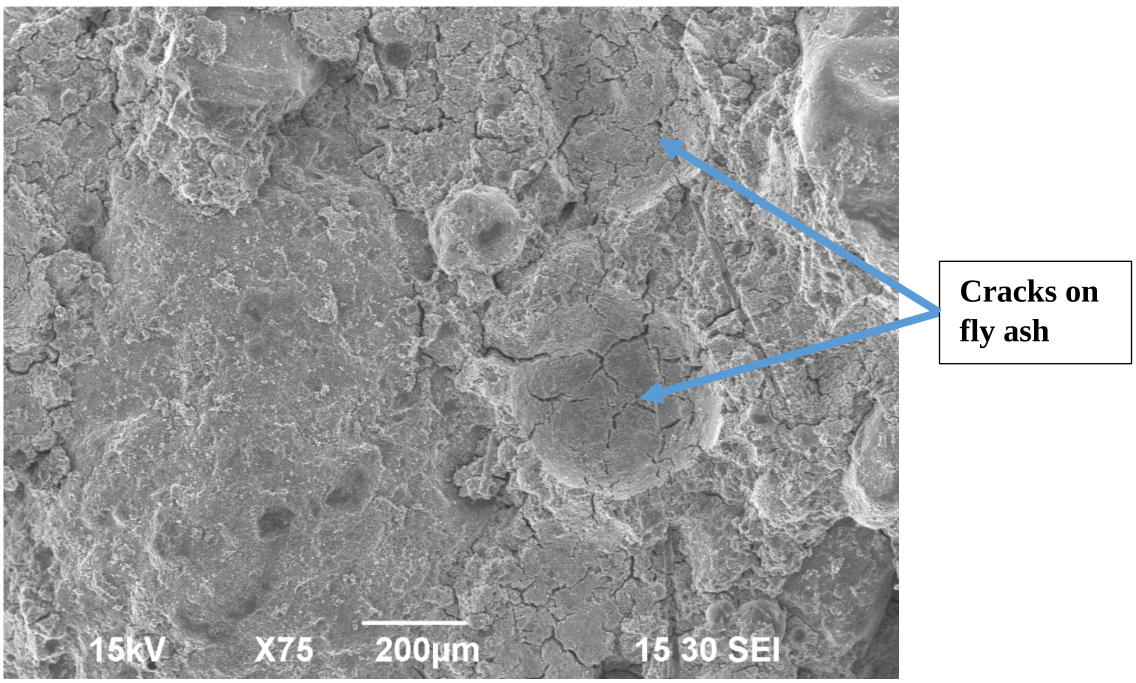


**Figure 4.20.** SEM image of geopolymer concrete (5% nano-silica and 0.25% Basalt fibers)

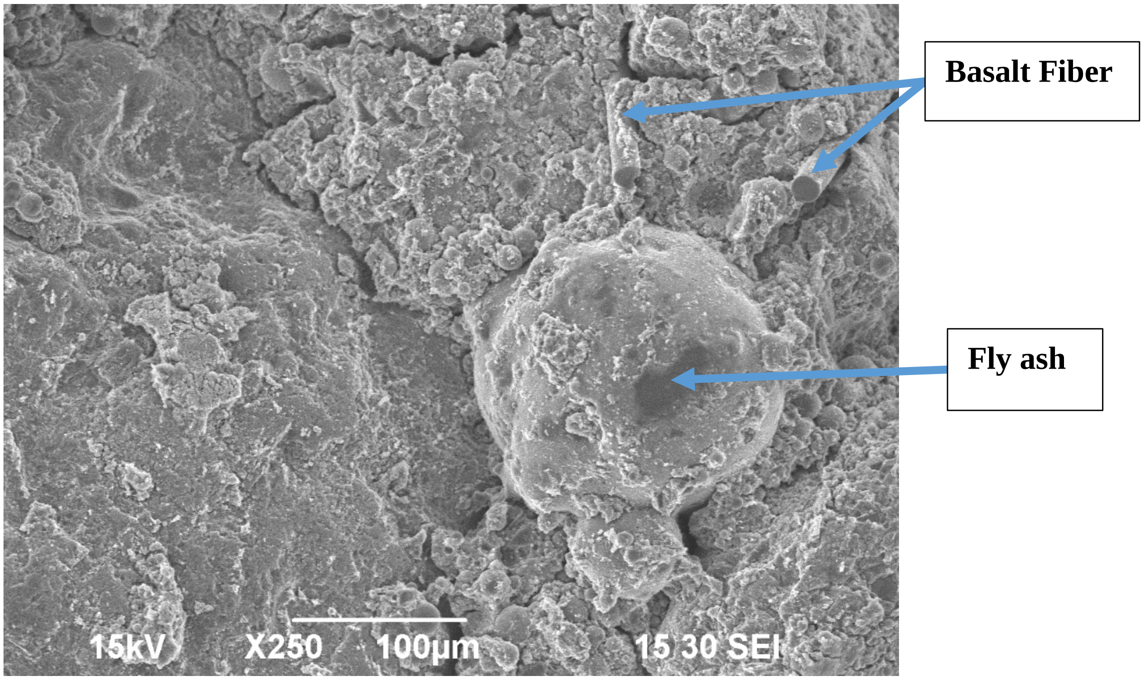


**Figure 4.21.** SEM image showing basalt fiber and fly ash

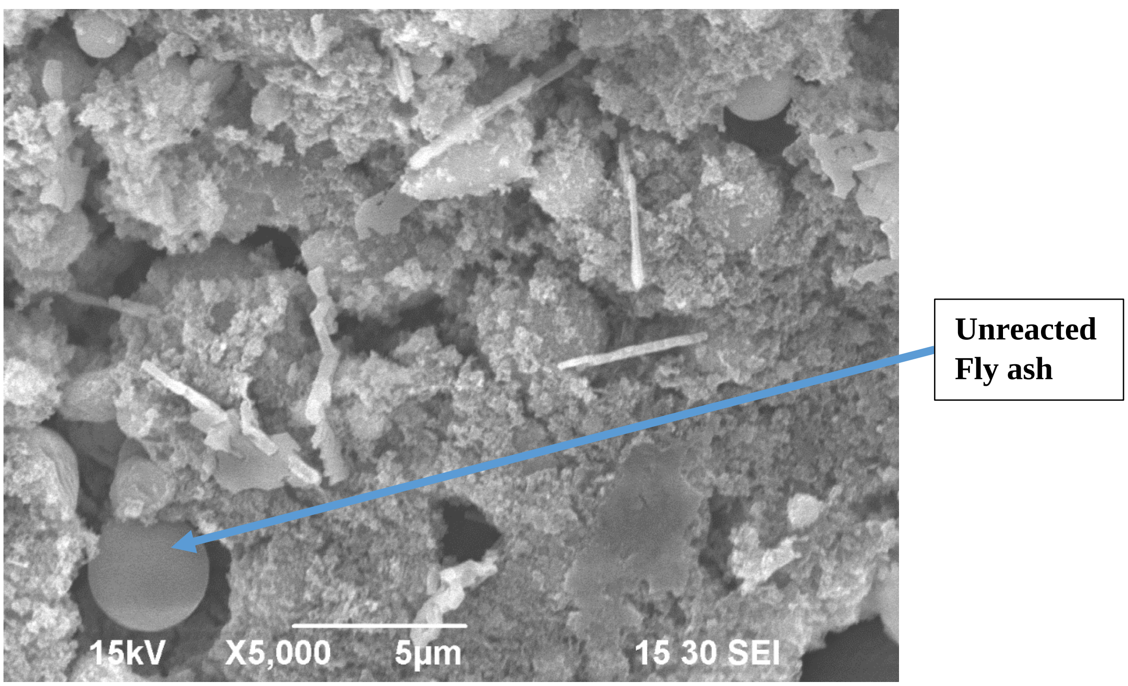


**Figure 4.22.** SEM image showing unreacted fly ash in geopolymer concrete (5% nano-silica and 0.25% Basalt fibers

Figure 4.23 Figure 4.24 and provides chemical deposits on geo polymer concrete incorporating Basalt fibers and nano silica through EDS analysis. Similar to geo polymer concrete incorporating basalt fibers and nano silica, this sample had relatively the same chemical composition with Na, Al Si being major compounds.

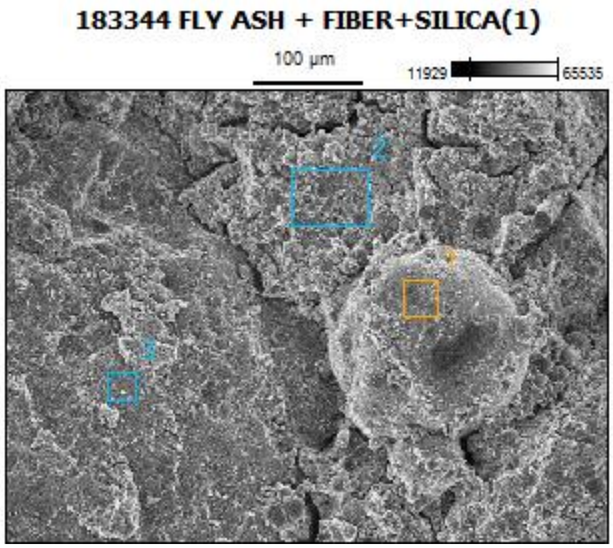


**Figure 4.23.** SEM image for EDS analysis of geopolymer concrete (5% nano-silica and 0.25% Basalt fibers)

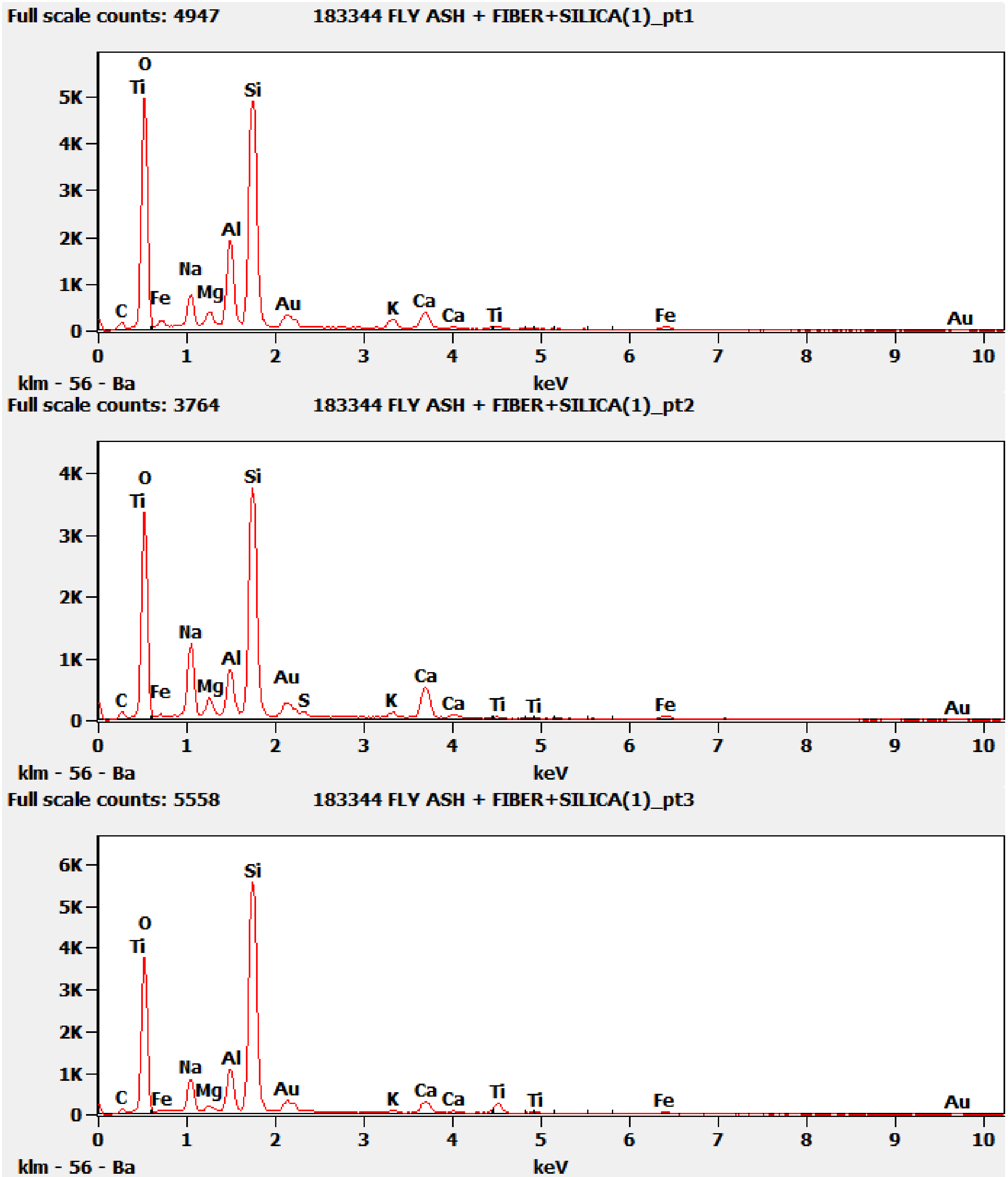


**Figure 4.24**. EDS graphs of geopolymer concrete (5% nano silica and 0.25% Basalt fibers)

# 5. CONCLUSION

This project provided a detailed analysis on geopolymer concrete while also incorporating basalt fibers and nano silica and comparing it with OPC concrete to see any difference among them. Four geopolymer mixes (fly ash geopolymer concrete, fly ash geopolymer concrete with basalt fibers, fly ash geopolymer concrete with nano silica, fly ash geopolymer concrete with nano silica and basalt fibers) and one ordinary portland cement concrete were manufactured and tested. Six quantitative tests were conducted to analyze the slump, air content, strength, resistivity, hydration, shrinkage of the manufactured concrete samples. Qualitative analysis of the samples was conducted using SEM images. Based on the results of this study, a performance matrix was developed to conclude the best mixes for further research.

## 5.1. Performance matrix

Table 5.1 shows the results for spread test and 28 day results for compressive strength, shrinkage and resistivity of all the five mixtures prepared. Some limits are set on the values based on industry standards and prior literature review (D Hardjito & Rangan, 2005). Based on the results of this performance matrix, nano silica based geopolymer concrete provides the best results in all parameters and therefore recommended for further research.

**Table 5.1.** Performance Matrix

| Mix code | Parameter and Performance limits | 28 day compressive strength (Mpa) | 28 day resistivity (kΩcm | 28 day shrinkage (μ-Strain | Spread Test (cm) |
|---|---|---|---|---|---|
| | | **>40 Mpa** | **> 13 kΩcm** | **> -280 μ-Strain** | **< 42 cm** |
| Control | | 29.8 | 11.3 | 320 | NA |
| FA | | 37 | 13.2 | 257 | 48.26 |
| FA + Fiber | | 41.5 | 12.1 | 296 | 35.56 |
| FA + Silica | | 47.6 | 14.3 | 265 | 40.34 |
| FA+ Fiber + Silica | | 38 | 13.5 | 301 | 38.26 |
| FA + RT | | 20 | 11.8 | 310 | NA |

## 5.2. Conclusion

The key conclusions are as follows:

1. The slump of geopolymer concrete is very high and is measured in terms of spread or slump flow.
2. The initial texture of geopolymer concrete shows a high spread and almost appear to be segregated, however the plasticity was lost very quickly in a matter of minutes, thereby decreasing its workability.

3. The compressive strength of all the different geopolymer concrete samples were higher than control OPC concrete which shows the potential of using it in place of OPC concrete.
4. A remarkable 59% increase in compressive strength was noticed in geopolymer concrete incorporating nano silica when compared with OPC control concrete.
5. Nano silica also provided 28% increase in compressive strength compared to normal geopolymer concrete
6. A 26.5% increase in resistivity was recorded in geopolymer concrete incorporating nano silica when compared with OPC control concrete while 8% increase was recorded when compared with normal geopolymer concrete.
7. Basalt fibers helped in enhancing the workability among the geopolymer concrete samples
8. The SEM analysis of geopolymer concrete shows that fly ash in concrete has a spherical like structure.
9. Geopolymer concrete having nano silica appears to have less voids which may be the reason behind higher compressive strength. The formation of secondary C-S-H gels may be partly the reason behind less pores.
10. Over all it appears from the results that nano silica enhances the properties of geopolymer concrete.

**5.3. Pros and cons**

The results indicate that geopolymer concrete provides strength, shrinkage and resistivity benefits, but there are some limitations in its usage. The geopolymer concrete needs to be oven or steam cured which is a little impractical on site but can be utilized for precast structures. The cohesive nature of geopolymer concrete makes it difficult to handle fresh mix.

**5.4. Future work**

A preliminary study was conduct to analyze whether fly ash based geopolymer concrete provides the same results in ambient curing conditions. The results indicate a 46% decrease in compressive strength, but the shrinkage and resistivity reading showed minimal reduction. Future researches can look to enhance the geopolymer concrete by tweaking the mix design or increase the Molar concentration of NaOH solution to see if it provides the same benefits in ambient curing. The cohesive nature of geopolymer concrete entices future researchers to come up with some kind of composition that helps in its workability.

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