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**Potential Utilization of Rock-based Geopolymers for Cementing of  
Geothermal Wells**

By

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## **Abstract**

Nowadays, the reserves of fossil fuels are running low due to the large consumption. Some few start getting interested in a type of energy that is long-lasting and free of polluting emissions, and that can be found right under our feet: the heat of the Earth. Geothermal energy is shown as one of the most promising and raises a growing interest in the set-off strategies that promote the exploitation of versatile and renewable energy sources. Three conditions must be fulfilled to have a geothermal system economically feasible for energy production: a heat source, permeability, and a waterproof layer. The cementation of geothermal wells aims to create an annular barrier between the casing and formation that can withstand the different conditions upstream, hold the casing in position, and protect the casing from formation fluids. There are several types of cement that can be utilized for geothermal wells applications. Ordinary Portland Cement (OPC) is the traditionally chosen material for zonal isolation on geothermal wells and other industries, such as oil and gas, carbon dioxide sequestration, and gas storage. For the production of OPC, there is high consumption of energy in which large amounts of carbon dioxide are released into the atmosphere. In an attempt to reduce global carbon emissions and to promote large-volume recycling of waste materials into new industrial products that could replace OPC, Geopolymers appear as an attractive option. This project is aiming to examine five different curing temperatures and periods conditions for a rock-based geopolymer recipe. They were investigated to check their applicability to be used under high-temperature conditions. From this study, It was observed that the higher and the longer the curing temperature condition is, the better the mechanical and chemical properties for the examined geopolymer samples are.

## **Acronyms**

OPC: Ordinary Portland Cement

MSR: Moderate sulfate-resistant

HSR: High sulfate-resistant

BHCT: Bottom hole circulating temperature

BHST: Bottom hole static temperature

BTU: British Thermal Unit

GGBFS: Ground granulate blast furnace slag

RM: Red Mud

POFA: Palm oil fuel ash

RHA: Rice husk ash

LOI: Loss in Ignition

XRD: X-ray diffraction

API: American petroleum Institute

UCA: Ultrasonic cement analyzer

UCS: Uniaxial compressive strength

RMP: Revolution per minute

SEM: Scanning electron microscope

Q: Quatz

A: Albite

M: Microcline

EDX: Energy dispersive x-ray spectroscopy

## List of Contents

Abstract .....	iii
Acronyms .....	iv
List of Contents.....	v
List of Figures.....	vii
List of Tables.....	ix
1 Introduction .....	10
1.1 Objective.....	11
2 Literature Review.....	12
2.1 Geothermal energy .....	12
2.1.1 Brief history of geothermal energy.....	12
2.1.2 Types of reservoirs of geothermal energy .....	13
2.1.3 Requirements for geothermal systems.....	14
2.2 Extraction of Geothermal Resources - Geothermal Wells .....	15
2.2.1 Cementation of geothermal Wells.....	16
2.2.2 Typical Slurry of Cement for Geothermal Wells .....	17
2.3 OPC VS Geopolymers Cements .....	18
2.3.1 OPC .....	18
2.3.2 Geopolymers .....	19
3 Methodology.....	21
3.1 Overview.....	21
3.2 Materials .....	21
3.2.1 Slag .....	21
3.2.2 Granite .....	22
3.2.3 Microsilica .....	22
3.2.4 Alkaline activators and Retarders.....	23
3.3 Equipment.....	23

3.3.1 Mettler Toledo Scale.....	23
3.3.2 OFITE Model Commercial Blender .....	24
3.3.3 UCA.....	24
3.4 Mixing Procedures .....	26
3.5 Compositional and Morphological Characterization .....	27
3.5.1 XRD.....	27
3.5.2 SEM.....	27
4 Results and Discussion.....	28
4.1 Results .....	28
4.1.1 UCA.....	28
4.1.2 XRD.....	30
4.1.3 SEM.....	31
4.2 Discussion.....	39
5 Conclusion.....	41
6 References .....	42

## List of Figures

Figure 1: Mettler Toledo Scale.....	24
Figure 2: OFITE Model Commercial Blender. ....	24
Figure 3: Model 120-51 Twin cell UCA was used for 70, 90, 110 and 130C curing samples at 2000 psi.....	25
Figure 4: Chandler model 4265-HT UCA was used for 150C cured sample at 2000 psi. ....	26
Figure 5: Orange line presents sonic strength development geopolymer by an algorithm, red line presents sonic strength development of the neat OPC, and blue line presents transit time for the 70C cured sample.....	28
Figure 6: Orange line presents sonic strength development geopolymer by an algorithm, and blue line presents transit time for the 90C cured sample. ....	29
Figure 7: Orange line presents sonic strength development geopolymer by an algorithm, and blue line presents transit time for the 110C cured sample. ....	29
Figure 8: Orange line presents sonic strength development geopolymer by an algorithm, and blue line presents transit time for the 130C cured sample. ....	30
Figure 9: Orange line presents sonic strength development geopolymer by an algorithm, and blue line presents transit time for the 150C cured sample. ....	30
Figure 10: XRD outputs for the five given curing conditions, Q is quartz, A is Albite, and M is Microcline.....	31
Figure 11: Scanning Electron Microscope (SEM) image for 70C cured sample at 20 micron magnification and orange circle presents the cracks observed. ....	32
Figure 12: Energy-dispersive X-ray spectroscopy image for 70C cured sample. ....	32
Figure 13: Scanning Electron Microscope (SEM) image for 90C cured sample at 20 micron magnification and orange circle presents the cracks observed. ....	33
Figure 14: Energy-dispersive X-ray spectroscopy image for 90C cured sample. ....	34
Figure 15: Scanning Electron Microscope (SEM) image for 110C cured sample at 20 micron magnification and orange circle presents the cracks observed. ....	35
Figure 16: Energy-dispersive X-ray spectroscopy image for 110C cured sample. ....	35
Figure 17: Scanning Electron Microscope (SEM) image for 130C cured sample at 20 micron magnification and orange circle presents the cracks observed. ....	36
Figure 18: Energy-dispersive X-ray spectroscopy image for 130C cured sample. ....	37

Figure 19: Scanning Electron Microscope (SEM) image for 150C cured sample at 20 micron magnification and orange circle presents the cracks observed. ....38

Figure 20: Energy-dispersive X-ray spectroscopy image for 150C cured sample. ....38



## List of Tables

Tabell 1: shows the composition of the GGBFS. ....	21
Tabell 2: shows the chemical composition of the ground granite. ....	22
Tabell 3: shows the chemical composition of Microsilica. ....	23
Tabell 4: Elemental analysis for 70C cured sample outputs. ....	33
Tabell 5: Elemental analysis for 90C cured sample outputs. ....	34
Tabell 6: Elemental analysis for 110C cured sample outputs. ....	36
Tabell 7: Elemental analysis for 130C cured sample outputs. ....	37
Tabell 8: Elemental analysis for 150C cured sample outputs. ....	39

## **1 Introduction**

Nowadays, Several industrial by-products are produced by timber manufacturing, rice milling, iron & steel making, power generation, mining, and others, becoming crucial environmental problems to handle. These industrial by-products are challenging to dispose, and contribute to land pollution and non-environmentally friendly waste materials (1).

For the production of Ordinary Portland Cement (OPC), there is high consumption of energy in which large amounts of carbon dioxide are released into the atmosphere. It has been reported that up to 1.5 billion tons/year of carbon dioxide are emitted by the manufacture of OPC worldwide, accounting for around 5% of total man-made CO<sub>2</sub> emissions. In an attempt to reduce global carbon emissions and to promote large-volume recycling of waste materials into new industrial products that could replace OPC, various studies have been carried out (1,14).

Ordinary Portland Cement (OPC) is the traditionally chosen material for zonal isolation on geothermal wells and other industries, such as oil and gas, carbon dioxide, and gas storage.

The cementation of geothermal wells aims to create an annular barrier between the casing and formation that can withstand the different conditions upstream, hold the casing in position, and protect the casing from formation fluids (26, 27).

Unlike wells in the oil and gas industry, the casing for geothermal wells is usually run back to the surface. The high thermal stresses imposed on the casing demand homogeneous cementation over the full casing length so that the stress will be distributed over the length of the casing as uniformly as possible, and stress concentration is avoided (3).

The cement slurries currently used deteriorate in the geothermal environments, which derives in a shortening of the life expectancy of the geothermal well. Therefore, the economics of geothermal power could be improved significantly if better materials are developed. A successful development of improved materials will lead to a significant advance in the technology required to economically utilize geothermal energy (1, 3).

The consequences from a geothermal well blowout may be much worse than from an oil well due to the large quantity of high-temperature fluid that will be released. Geothermal wells differ from oil wells in that they do not utilize a production string, production packing and a subsurface safety valve. Also, the geothermal fluid is in direct contact with the casing (3).

Thus, greater reliability in components such as the casing and cement is needed. Improved cements will reduce the likelihood of well failures and will have significant positive effects on the economic and environmental aspects of geothermal energy development.

## **1.1 Objective**

The main objective of this project is to study the potential and performance of rock-based geopolymers for zonal isolation in geothermal wells as an alternative to Ordinary Portland Cement. The main objective will be addressed through the following sub-objectives:

- Studying sonic strength of the samples at different temperatures.
- Studying the crystallography of the exposed samples of the exposed samples
- Studying the microstructure of the samples before and after exposure to elevated temperature

## **2 Literature Review**

### **2.1 Geothermal energy**

Geothermal energy is the energy accumulated as heat under the solid surface of the Earth. It encompasses the heat stored in rocks, soil, and groundwater, whatever its temperature, depth, and origin are. It does not include the heat contained in bodies of surface, continental or marine waters, which utilization is also possible through heat exchangers and heat pumps (1, 3).

However, the heat contained in rocks and soils is too diffuse to be extracted directly in an economical way, being necessary to have a fluid, generally water, to transport the concentrated heat to the surface through wells, geothermal probes, horizontal collectors, or through ground-air heat exchangers buried shallowly in the ground. Once at the surface, the geothermal fluid, depending on its heat content, will be used for electrical energy production, if possible, or otherwise, will be used directly by resorting to the use of heat exchangers or heat pumps if necessary (3, 8).

#### **2.1.1 Brief history of geothermal energy**

The volcanic regions have always been poles of attraction for human beings due to the existence of fumaroles and thermal sources that could be used to warm themselves, cook food, or simply bathe. The oldest archaeological remains related to geothermal energy have been found in Niisato, Japan, and are objects carved in volcanic stone that date from the third ice age between 15000 to 20000 years ago. More than 10000 years ago, the Paleo-Indians of North America already used thermal waters to cook food and its minerals for medicinal purposes. The thermal springs were neutral areas where members of the warrior nations were to bathe together in peace (3).

The first civilizations, around 3500 years before Christ, valued the practice of thermal baths and the use of thermo mineral mud, but it was greeks and later Romans who left numerous examples of the application of geothermal energy in district heating and in traditional public hot springs, which became gigantic leisure, health and business centers. The Baths of Caracalla, in Rome, had a capacity for 1600 people. For a long time, man was content using the heat that naturally emerged on the planet's surface. Starting in the 19<sup>th</sup> century technical advances and better knowledge of the subsoil made it possible to search deeper and deeper, and to better exploit the Earth's heat (3, 8).

The discovery in 1818 of boron salts in Larderello, Tuscany (Italy), marked the beginning of the industrial use of geothermal resources. In 1827 the founder of this industry, the Frenchman Francois Larderel, developed a system to use the heat of the fluids in the evaporation process, instead of burning wood from nearby forests, which were in rapid deforestation. In France, in 1833, in the neighborhood of Grenelle, in Paris, the first deep borehole was started, an artesian well 548 m deep, which took eight years to build and captured drinking water at 30 °C in the Albian sands aquifer. of the Paris Basin. In the United States, the first local district heating network became operational in 1892 in Boise, Idaho (3, 7). In the 20th century, the use of geothermal energy increased, driven by the ever-increasing needs for energy to supply modern civilization. In 1921, in the United States, in the area of The Geysers, in California, two wells were drilled and a small steam engine was installed that, connected to a dynamo, produced electricity for a small thermal establishment. The first modern district heating network powered by geothermal energy was installed in Reykjavik, Iceland, in 1930. Since then, heating networks using geothermal energy have been in operation in France, Italy, Hungary, Romania, Russia, Turkey, Georgia , China, the United States and Iceland itself, where today 95% of the island's inhabitants are heated by a network of 700 km of insulated pipes that carry hot water (1, 3). After World War II, many countries were attracted to geothermal energy as they considered it competitive with other energy sources. Starting in the 1970s, an intense activity of exploration and investigation of geothermal resources began in many parts of the world, to use them for the production of electrical energy or for heating and hot water. It is also from that decade, as a consequence of the increases in crude oil prices, but particularly from the nineties, under the pressure of environmental requirements and, more particularly, of the reduction of CO<sub>2</sub> emissions to the atmosphere, when the use of geothermal heat pumps begins to show a promising development at the international level (3, 7).

### **2.1.2 Types of reservoirs of geothermal energy**

The applications that can be given to a geothermal fluid depend on its heat content, or what is similar, its enthalpy. Enthalpy is the amount of thermal energy that a fluid, or an object, can exchange with its surroundings. It is expressed in kJ / kg, kcal / kg or BTU/lb. As there are no devices that directly determine the enthalpy of a fluid in the subsoil, but there are thermal probes that measure temperature, and as enthalpy and temperature can be considered more or less proportional, the usual practice has generalized the use of the temperatures of geothermal

fluids instead of their heat content, since, after all, it is the temperatures that determine their future industrial application (1).

Different authors establish different temperature limits for these intervals. The division that appears established by the Mining Code, in France, can be applied to geothermal energy, as well as to exploitations, deposits and geothermal resources, is as follows:

- High temperature: more than 150 °C

A temperature higher than 150 °C allows water vapor to be directly transformed into electrical energy.

- Medium temperature: between 90 and 150 °C.

It allows the production of electrical energy using an exchange fluid, which is what feeds the power plants.

- Low temperature: between 30 and 90 °C.

Its heat content is insufficient to produce electrical energy, but it is suitable for heating buildings and in certain industrial and agricultural processes.

- Very low temperature: less than 30 °C.

It can be used for heating and air conditioning, requiring the use of heat pumps.

### **2.1.3 Requirements for geothermal systems**

Three conditions must be fulfilled to have a geothermal system economically feasible for energy production:

- Heat source: a magma, which can be at 700 - 1,000 °C, located 5-10 km from the surface. In certain areas with strong tectonic activity such as graben (sunken blocks limited by normal faults), the magma is closer to the surface and can also rise through faults (8).
- Aquifer: a layer of permeable hot rocks from which circulating water can extract heat that is connected to a surface recharge area, through which meteoric water can replace that escaping through hot springs, or the one that is extracted through the exploitation wells (8).

Water, depending on its temperature and pressure, will be in the liquid phase or in the vapor phase, and may contain dissolved salts and gases such as CO<sub>2</sub>, SH<sub>2</sub>, etc.

- Waterproof layer: located over the aquifer to keep the water trapped and its pressure (8).

## 2.2 Extraction of Geothermal Resources - Geothermal Wells

Except for hot springs that emanate spontaneously, most geothermal operations need, in the first place, a production borehole that allows the hot water and/or steam to be raised to the surface. On hard ground, the simplest, and oldest, technique for drilling is wired percussion. The rock fractures when hit by a heavy tool called a drill bit, which is raised and repeatedly dropped, hanging from a steel cable, on the rock to be fractured. The height of the fall and the frequency of the blows vary according to the hardness of the rocks. The extraction of the rock fragments from the bottom of the borehole is carried out discontinuously by means of a hollow cylindrical spoon, fitted with a check valve at its bottom (7).

A very effective procedure for hard and homogeneous soils, and depths of up to 300 m, is the rotary percussion drilling with hammer in the bottom of the hole. A pneumatic hammer that ends in a mouth with cutting tools, is fixed to the base of a train of rods, and is given a rotational movement from outside the borehole, and percussion, by sending compressed air to high pressure (10 to 25 bar), inside the linkage. Compressed air allows drilling cuttings to rise to the surface. A variant consists of injecting a foam through the compressed air conduit, to favor the stability of the borehole walls, and the rise of the rock fragments (7).

The most widely used method is rotary drilling, either using autonomous equipment mounted on trucks, for shallow drilling, or on huge platforms to drill up to 5,000 m in depth. This drilling technique, also called Rotary, consists of using a bit provided with serrated cones in which pieces of tungsten carbide or industrial diamonds are set, which destroy the rocks by crushing, cutting and abrasion, under the effect of weight. and rotation. A mud, which circulates in a closed circuit, cools the bit, ensures the dragging of the rock fragments to the surface and maintains the stability of the borehole walls until the tubing and injection of cement between the casing and the hole. The cement ensures the stability of the walls, protects the pipes against corrosion and the layers of underground water against contamination, ensuring thermal insulation. If the land is not sufficiently consolidated, the casing and cementing are done as the borehole goes deeper, telescopically, decreasing in diameter, being able to start at 660 mm at the well curb, and end at 200 mm in diameter (3, 7, 8).

To reduce weight, the casing is usually made of light metal alloys, with high tensile strength, smooth, without burrs on the inside of the joints, and, in some cases, lined with corrosion-resistant materials. Not only the casing, but also the pipes that connect the wells, the heat exchangers, the joints and all the materials in contact with the geothermal water, are victims of different types of corrosion. Chemical, because the fluid is loaded with very aggressive mineral

salts; galvanic, caused by the presence of parasitic electric currents; and also bacteriological corrosion, since some bacterial strains, eventually present in the reservoir, can reactivate with the drop in water temperature, and the sulfides, produced by their metabolism, increase the corrosion of the steel (7).

The suspended particles carried by the water can settle in certain places in the pipes forming encrustations that can destroy them or, at least, reduce the useful sections, causing decreases in flow. To protect the facilities, preventive treatments are applied: injections of inhibitor products to obtain a protective layer or prevent the crystallization of salts and the formation of deposits; and bactericidal products to eliminate microorganisms. If the pressure of the geothermal reservoir is higher than atmospheric, the water can flow naturally at the head of the production well, forming an artesian well. If the pressure is not high enough, or if the water does not rise completely to the surface, which is the most common case, it will have to be pumped. If the water that rises from the subsoil is slightly loaded with mineral salts, it can be drained on the surface without damaging the environment. But if the fluid is highly loaded with dissolved salts, or because it is necessary in order not to deplete the resource, it will have to be reinjected back into its original aquifer, after its use, through an injection well. Classic type surface pumps are used for reinjection (7).

The water must be returned to the subsoil at a point relatively far from the extraction point, since, having given up a large part of its heat in the surface exchangers, it is returned to the aquifer at a lower temperature than the extraction temperature. In the Paris Basin it is used about 1 km of distance, according to a technique known as doublet. In urban areas, to avoid the inconvenience of duplicating the works involved in drilling from the surface, the pair of wells, production and reinjection, are usually drilled with a single drilling platform, diverting the trajectory of one or both wells (7).

### **2.2.1 Cementation of geothermal Wells**

After the conclusion of the drilling operation of each section of the well, casing is run in the hole and then it is cemented to surface. Cementation of the casing is a critical point during drilling since:

- It must provide adequate protection to the casing, because it is subjected to thermal expansion (a poor quality in the cementation job can provoke failures)
- There should not exist pockets of water in the annular space since this can occasionate a collapse due to the expansion of water trapped that will not be able to be released.



- The cement should block any infiltration of formation fluid through the annular space.

### **2.2.1.1 Typical Slurry of Cement for Geothermal Wells**

A traditional slurry consist of:

- Cement class H or G
- Silica Flour
- Aditives
  - Retarder for high temperatures
  - Dispersants or friction reducers
  - Filtrate reducers
  - Defoamer

Slurry design is affected by well depth, Bottom Hole Circulating Temperature (BHCT), Bottom Hole Static Temperature (BHST), drilling fluid type previously used (either oil based mud or water based mud), slurry density needed, pumping time, quality of mix water, fluid loss control, flow regime, settling and free water, quality of cement, dry and liquid additives, strength development during the curing period, and the quality of the laboratory testing of the cement and equipment. High temperatures such as 150 - 350°C are found in geothermal wells. Research studies carried out on OPC have shown that it is necessary to add silica flour to the cement to avoid strength retrogression at temperatures above 120°C (3, 7, 8).

Structural damage to the cement sheat can sometimes be in the form of debonding of the casing, formation interfaces, cement envelope cracking and/or compressive shear. Casing collapse in thermal wells can arouse when liquid is trapped in some way in the casing to casing annulus of the well. As this liquid gets heated up, expands enough that it can exceed the collapse resistance of the casing. The thermally induced loads in the cement can be extreme in geothermal wells, given that from the moment the casing is cemented in place to the moment the well is first produced, the casing can be subjected to a temperature increase of 300°C (3, 7, 8).

### **2.2.1.2 Requirements for cementing materials to be used in geothermal wells**

It is estimated that for utilization in geothermal wells, cementing materials with the following characteristics are needed:

- A compressive strength superior to 1000 psi after 24 hours of placement
- Permeability to water lower than 0,1 mD
- Bond strength to steel casing above to 10 psi
- To be stable, no significant reduction in the strength or increase on permeability after prolonged exposure at a temperature of 400°C, to 25% solutions of brine, flashing brine

or dry steam

- Placeability, capable of 3 to 4 hours of retardation at the expected temperatures of placement
- The cement has to be compatible with the drilling mud previously used
- Non- corrosive to steel well casing

### 2.3 OPC VS Geopolymers Cements

The main objective of casing cementation is to ensure that the entire length of the annulus is completely filled with cement, resistant to long term exposure to geothermal fluids and temperatures, and provide mechanical integrity to the wellbore. The most important functions of the cement sheath between the casing and the formation are (2):

- Preventing the migration of geothermal fluids from one zone to another, and from the drilled formations to surface through the annular space.
- Holding casing string in the well, avoiding buckling.
- Protecting the casing from corrosive formation fluids.
- Sustaining the wellbore walls to avoid any possibility for collapse of the formations.
- Avoiding any likelihood of blowouts by forming the desired seal for the annulus.
- Preserving the casing from shock loads when drilling a deep well.

Cementing is also an operation carried out for workover and intervention operations of the well, when it is necessary to seal loss of circulation zones, stabilize weak zones (washouts, collapses), plug the well for abandonment, help in kick off side tracking in an open hole, and plugging a well temporarily (2, 5, 6).

#### 2.3.1 OPC

Ordinary Portland cement (OPC), is the most used binder for civil and petroleum engineering operations. However, it has been criticized for its environmental impact on air pollution. OPC is a calcium silicate material, which main components are tricalcium silicate ( $C_3S$ ), dicalcium silicate ( $C_2S$ ) and tricalcium aluminate ( $C_3A$ ). In petroleum industry, API Spec 10A classification of cement employed for well cementing is as follows (2, 12):

- **Class A:** Intended for use when special properties are not required. It is available only in ordinary (O) grade.
- **Class B:** Intended for use when conditions require moderate or high sulfate-resistance. It is available in both, moderate sulfate-resistant (MSR) and high sulfate-resistant (HSR)

grades.

- **Class C:** Intended for use when conditions require high early strength. Available in O, MSR and HSR grades.
- **Class D:** Intended for use under conditions of moderately high temperatures and pressures. Available in MSR and HSR grades.
- **Class E:** Intended for use under conditions of high temperatures and pressures. Available in MSR and HSR grades.
- **Class F:** Intended for use under conditions of extremely high temperatures and pressures. Available in MSR and HSR grades.
- **Class G:** Intended for use as basic well cement. Available in MSR and HSR grades. No additives other than calcium sulfate or water, or both, shall be inter-ground or blended with the clinker during the manufacture of class G well cement.
- **Class H:** Intended for use as basic well cement. Available in MSR grade. No additives other than calcium sulfate or water, or both, shall be inter-ground or blended with the clinker during the manufacture of class H well cement.

### 2.3.2 Geopolymers

The problem of how to dispose industrial by-products, together with the greenhouse gas emissions in the production of OPC, and the disadvantages of using it in geothermal conditions, (such as autogenous shrinking, permeability to gas influx, instability in corrosive environments and high temperatures) could be addressed by green cementitious materials, with a high potential of replacing OPC as the main binder in the future of concrete elaboration (5, 6).

At this moment in time, one of the waste recycling options that has become more important in research and development is geopolymer binders. In 1970s, Joseph Davidovits introduced geopolymer binders as a green alternative cementitious material to OPC. Since then, researchers have investigated and identified several methods and reaction products between alkaline solutions, as activators (such as sodium hydroxide [NaOH], potassium hydroxide [KOH], sodium silicate [SiO<sub>2</sub>Na<sub>2</sub>O] or potassium silicate [SiO<sub>2</sub>K<sub>2</sub>O]) and source materials of geological origin or by-product materials rich in silica and alumina, as precursors (such as fly ash, rice-husk ash, aplite, ground granulated blast furnace slag [GGBFS], metakaolin, etc.) (2, 5, 6, 9, 12).

Other research studies have discussed geopolymers possess excellent mechanical properties, fire resistance, and anticorrosive properties. In consequence, GGBFS, an industrial by-product

of iron or steel manufacturing, has been used significantly in the production of a geopolymer slurry with superior mechanical properties. Fly ash, is a by-product of coal burning power plants that has also been found to yield a geopolymer concrete with excellent strength compared to OPC concrete. In addition, industrial by-products such as red mud (RM) from the aluminum refining industry, palm oil fuel ash (POFA) from the palm oil industry, rice husk ash (RHA) from the rice milling industry, etc., have also a capability to be utilized into the production of geopolymer concrete (5, 6, 9, 12).

## 3 Methodology

### 3.1 Overview

This chapter presents a detailed overview of all the experimental methods used for the development of rock-based geopolymer samples. The properties of these materials are investigated, as well as the mix-designs, are described too. Moreover, the experimental design and the test parameters were also used to examine and analyze the obtained results.

### 3.2 Materials

The following materials have been used in this thesis for the geopolymeric formation:

#### 3.2.1 Slag

The slag used to produce the rock-based geopolymer was a commercial ground granulated blast furnace slag (GGBFS) (25). The GGBFS was used as an additive to compensate the low aluminum content of the rock-based geopolymer. Table 1 shows the composition of the GGBFS used, as provided by the supplier.

*Tabell 1: shows the composition of the GGBFS.*

Compound	Chemical content (weight %)
SiO <sub>2</sub>	34,0
Al <sub>2</sub> O <sub>3</sub>	13,0
CaO	31,0
MgO	17,0
Na <sub>2</sub> O	0,9
TiO <sub>2</sub>	2,4
MnO	0,6
S <sup>-2</sup>	1,1
LOI	-

Note: LOI: Loss in Ignition

### 3.2.2 Granite

In this work, granite powders were used as a starting material for geopolymerization. It is rich in large amount of SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> (13-22). It was used without any processing other than grinding. Table 2 shows the chemical composition of the ground granite.

Tabell 2: shows the chemical composition of the ground granite.

Compound	Chemical content (weight %)
SiO <sub>2</sub>	60.02
Al <sub>2</sub> O <sub>3</sub>	10.88
Fe <sub>2</sub> O <sub>3</sub>	0.58
CaO	5.04
MgO	8.07
Na <sub>2</sub> O	1.84
K <sub>2</sub> O	1.62
TiO <sub>2</sub>	1.15
MnO	0.01
SrO	0.01
BaO	0.01
LOI	0.15

Recently, (13 – 22) and other researches have studied the utilization of different rock-based materials as source materials for geopolymers, and with the use of X-ray power diffraction (XRD), quartz has been found to be the most abundant of its crystalline phases, while biotite, albite and microcline are found as minor crystalline phases.

### 3.2.3 Microsilica

Microsilica is a mineral admixture made of very fine, solid, glassy spheres of amorphous silicon dioxide (SiO<sub>2</sub>) in a micro-powder form. It is a by-product of the industrial manufacturing of silicon metal or ferrosilicon alloy. Microsilica is mainly used as a pozzolanic material for high strength development and reduction of permeability in concrete. For this thesis, microsilica was supplied by the Elkem Company of Oslo, Norway. It is composed of ultra-fine amorphous spheres of silicon dioxide, and it was used as a microfiller to make a high-performance geopolymer by decreasing average pore size in the geopolymer paste (13 – 22). Its Chemical composition is given in Table 3.

Tabell 3: shows the chemical composition of Microsilica.

Compound	Chemical content (weight %)
SiO <sub>2</sub>	95,50
Al <sub>2</sub> O <sub>3</sub>	0,70
Fe <sub>2</sub> O <sub>3</sub>	0,30
CaO	0,40
MgO	0,50
Na <sub>2</sub> O	0,40
K <sub>2</sub> O	1,00
C	1,00
LOI	2,00

### 3.2.4 Alkaline activators and Retarders

The activator was a solution containing a combination of two potassium silicate (K<sub>2</sub>SiO<sub>3</sub>) solutions. The two potassium silicate solution were supplied by Univ AS, Norway. Their chemical contents were reported as the following; the first solution was containing 35 wt.% of K<sub>2</sub>SiO<sub>3</sub> and 65 wt.% of H<sub>2</sub>O however the second one was containing 57 wt.% of K<sub>2</sub>SiO<sub>3</sub> and 43 wt.% of H<sub>2</sub>O. The final activator solution was with a modular ratio of 2.49 that was used as a hardener and mixed with the solid phase prior to pre-conditioning. Furthermore, Zinc nitrate was used as the only retarder in this study with 0.5 wt.% of the whole system.

### 3.3 Equipment

The tools used during the preparation of the geopolymer are mentioned and shortly described below. Each of them has its function and procedures to use them appropriately.

#### 3.3.1 Mettler Toledo Scale

It is used to weight the components of the mixture. The accuracy of this scale is about +/-0.01 g. Figure 1 shows the scale used for the project.



*Figure 1: Mettler Toledo Scale.*

### **3.3.2 OFITE Model Commercial Blender**

It is used to prepare the liquid phase and mix it with the solid phase to prepare the slurry according to API procedures. The constant speed blender has a high rotating speed, which results in a high-level of mixing energy as shown in Figure 2.



*Figure 2: OFITE Model Commercial Blender.*

### **3.3.3 UCA**

There are two different ways to estimate the mechanical properties of the slurry, destructive test, and non-destructive one. Each of them has its benefits and shortcomings. An



ultrasonic cement analyzer (UCA) is a non-destructive test and is applied to estimate the compressive strength through measuring the velocity of an ultrasonic signal under pressure and temperature conditions. The sonic strength is correlated to the transit time. An empirical relationship between the compressive strength and the transit time is used, and then the collected data is sent to a computer to highlight the results (Khalifeh et al., 2014). This method has pros and cons. Its benefits are non-destructive, realistic (simulate a downhole condition), and accurate. Establishing the desired temperature and pressure of downhole condition through an internal oven is integrated with UCA, and A compressor is connected with it. Its drawback is that the measurements are just mathematical estimations and correlations of the measurements that could be obtained by Uniaxial Compressive Strength (UCS) data (19, 20, 28). Figures 3 and 4 show the used UCA equipment that were used for the five given curing conditions.



Figure 3: Model 120-51 Twin cell UCA was used for 70, 90, 110 and 130°C curing samples at 2000 psi.

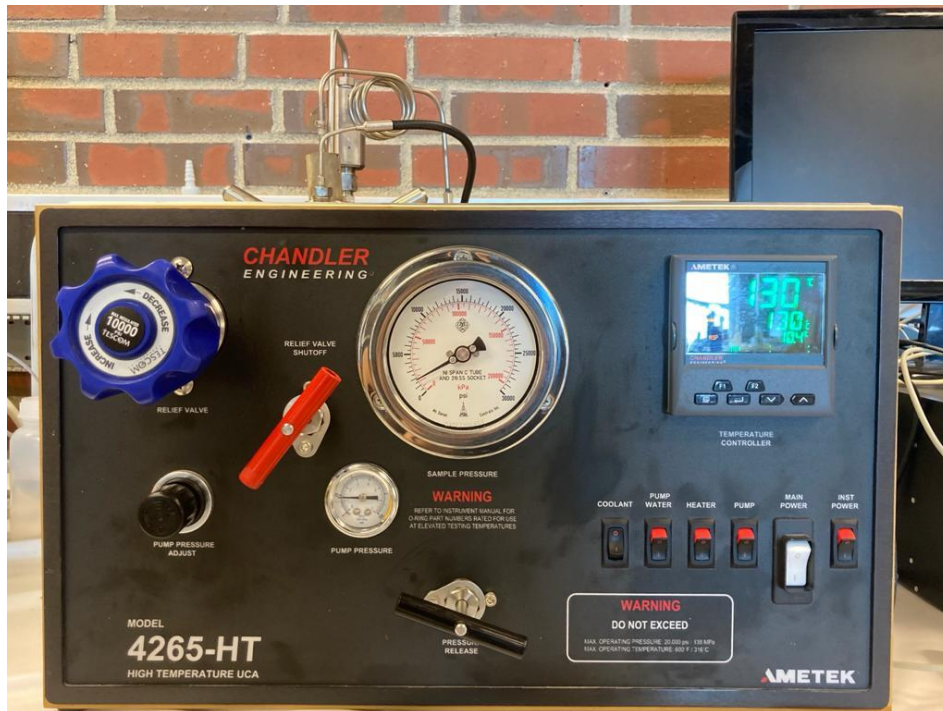


Figure 4: Chandler model 4265-HT UCA was used for 150°C cured sample at 2000 psi.

### 3.4 Mixing Procedures

There are many different ways to mix the solid and liquid phases. In this project, the following steps are applied to prepare the rock-based geopolymer paste:

1. Preparing each element of the slurry according to its dosage.
2. Mixing the components of solid-phase at dry conditions.
3. Adding zinc nitrate powder as a retarded to the required amount of distilled water and then adding this solution to the activator.
4. Adding and mixing elements of the activator hardener including the zinc nitrate solution for a few seconds.
5. Mixing these mixture of solutions together at 4000 RPM for 15 seconds
6. Turning the commercial blender on an automatic position. So, it works for 50 sec, 15 sec at low speed 4000 RPM and 35 sec at high speed 12000 RPM according to API RP 10B-2 standards.
7. Adding the solid to the liquid during 15 secs and then the mixing process is continued for the remaining 35 seconds (Petroleum and natural gas industries. Cement and materials for well cementing, n.d.).

### **3.5 Compositional and Morphological Characterization**

#### **3.5.1 XRD**

All crystalline minerals of the examined geopolymers were analysed by a Bruker-AXS Micro-diffractometer D8 Advance which uses a  $\text{CuK}\alpha$  radiation (40.0 kV, 25.0 mA) with a  $2\theta$  range from  $5^\circ$  to  $92^\circ$  with  $1^\circ$  /mins step and  $0.010^\circ$  increment. These samples were kept in a vacuum-dryer overnight to remove any water residue. These crystalline minerals were identified using the intensity of the observed diffraction as a function of the angle. As the chemical composition of the mixtures is complex and minor differences can occur due to sample preparation and random distribution of minerals, only the main peaks of the XRD patterns have been considered in comparison with the solid precursor “Granite” (16-22).

#### **3.5.2 SEM**

A Scanning Electron Microscope (SEM) was used to study the microstructure and for qualitative composition analysis of the geopolymers. A SEM Hitachi TM3000 was used in this study. This microscope operates at 5 kV and 15 kV in backscattered electron mode (BSE).

## 4 Results and Discussion

### 4.1 Results

#### 4.1.1 UCA

An ultrasonic cement analyzer (UCA) was used as a non-destructive test to estimate the compressive strength through measuring the velocity of an ultrasonic signal under pressure and temperature conditions. After establishing the desired temperature and pressure of downhole conditions for each experiment to be done as discussed in the methodology part starting from 70°C for one-week up to 150°C for five-weeks as shown in Figures 5, 6, 7, 8 and 9.

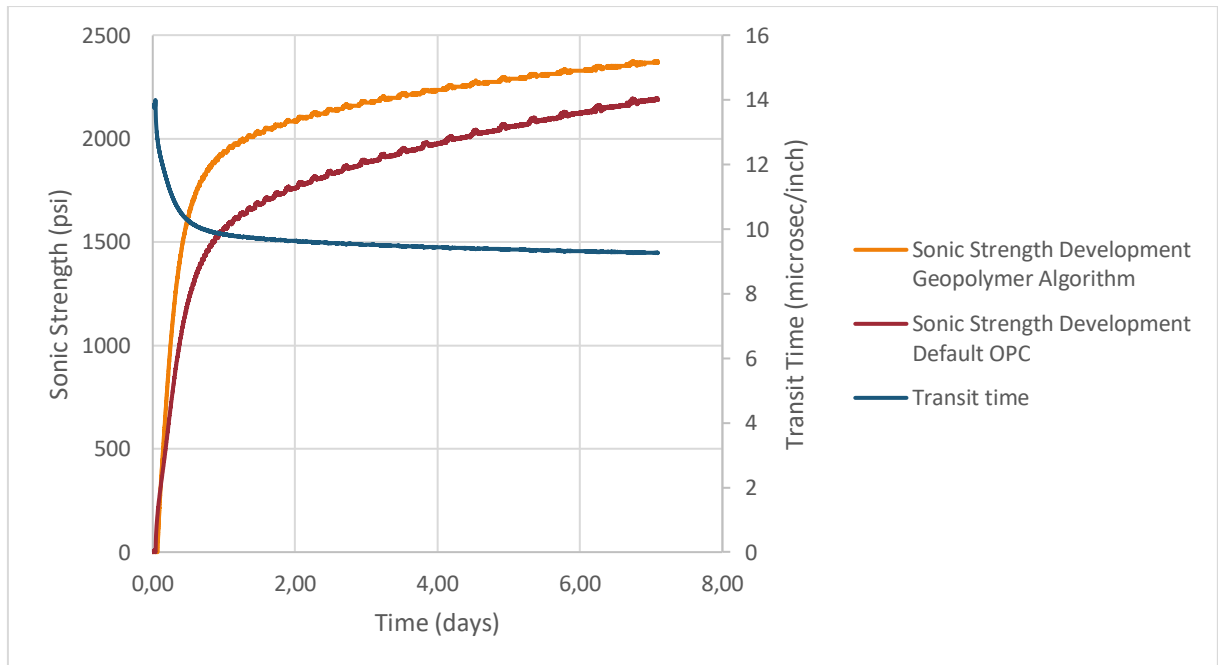


Figure 5: Orange line presents sonic strength development geopolymer by an algorithm, red line presents sonic strength development of the neat OPC, and blue line presents transit time for the 70C cured sample.

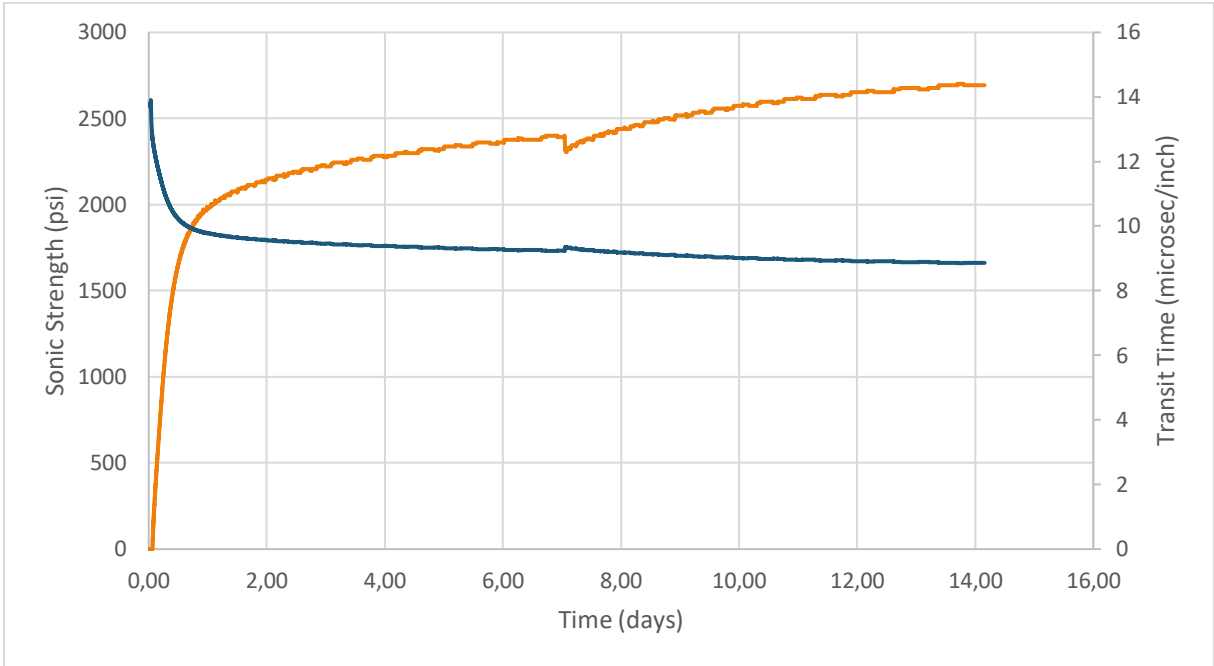


Figure 6: Orange line presents sonic strength development geopolymer by an algorithm, and blue line presents transit time for the 90C cured sample.

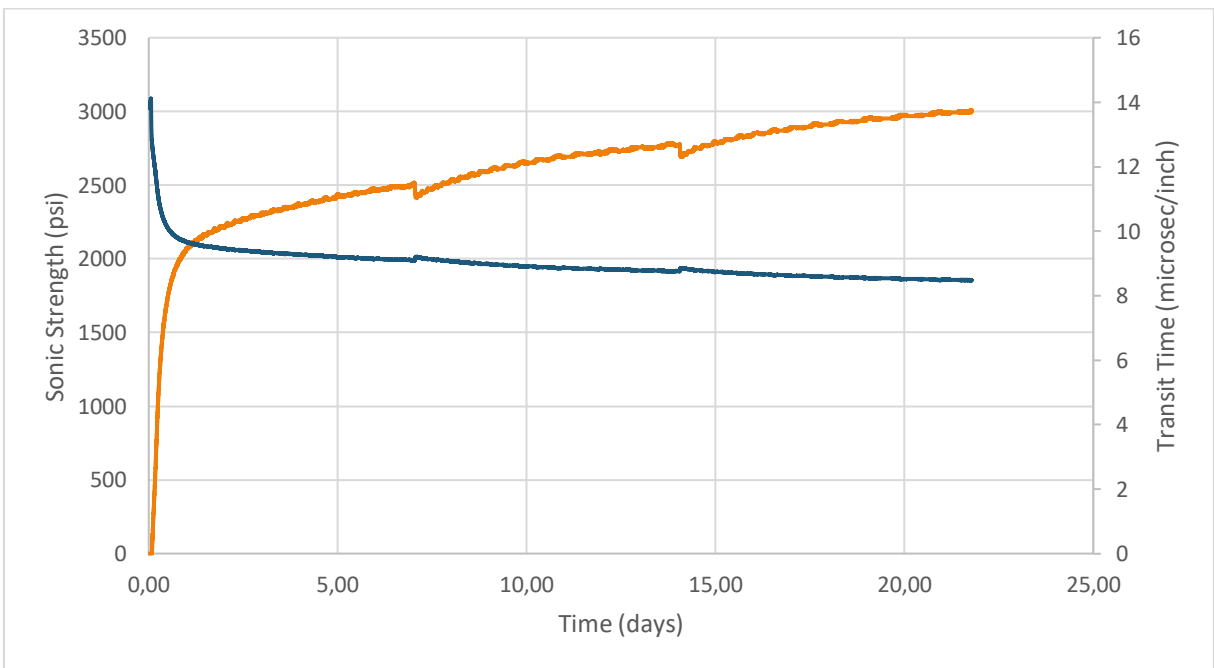


Figure 7: Orange line presents sonic strength development geopolymer by an algorithm, and blue line presents transit time for the 110C cured sample.

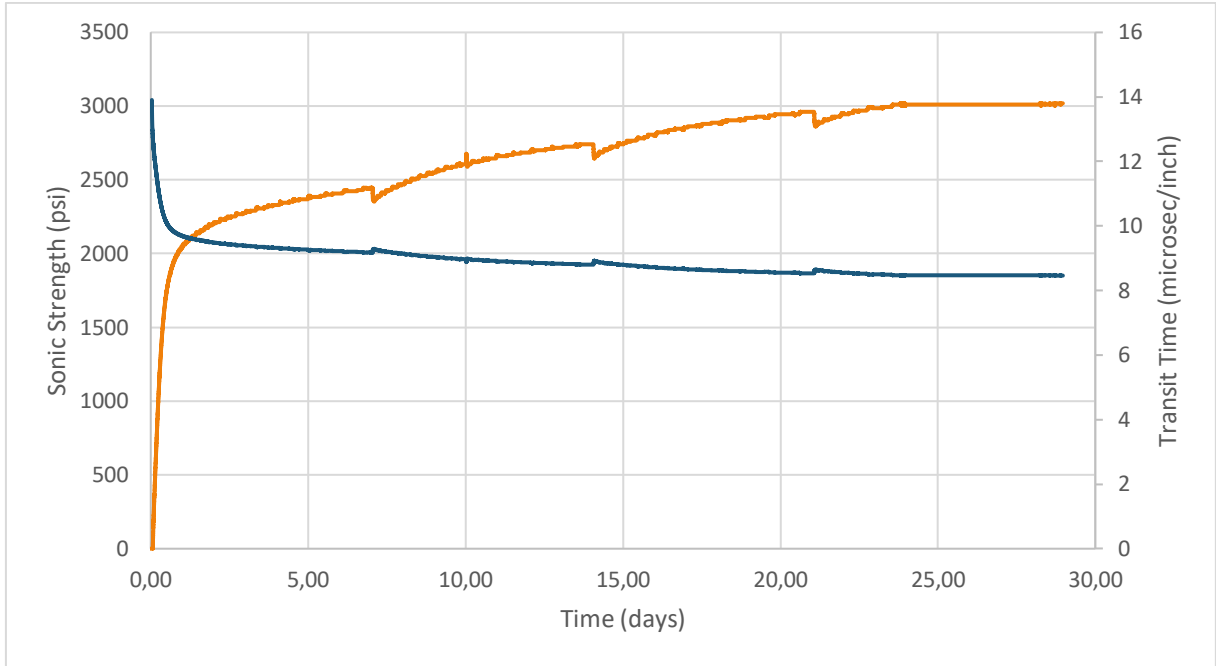


Figure 8: Orange line presents sonic strength development geopolymer by an algorithm, and blue line presents transit time for the 130C cured sample.

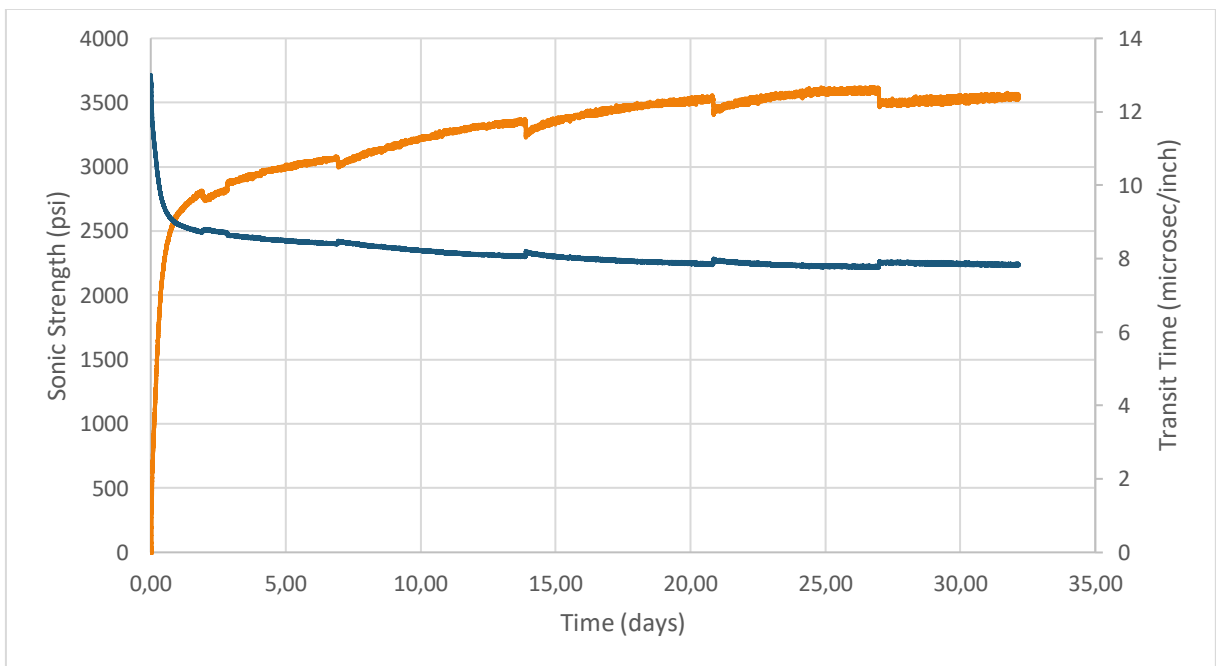


Figure 9: Orange line presents sonic strength development geopolymer by an algorithm, and blue line presents transit time for the 150C cured sample.

#### 4.1.2 XRD

A XRD Bruker-AXS Micro-diffractometer D8 Advance which uses a  $\text{CuK}\alpha$  radiation (40.0 kV, 25.0 mA) with a  $2\theta$  range from  $5^\circ$  to  $92^\circ$  with  $1^\circ$  /mins step and  $0.010^\circ$  increment was

used to investigate the five given conditions and to conduct the compositional analysis to these geopolymer samples as shown in figure 10.

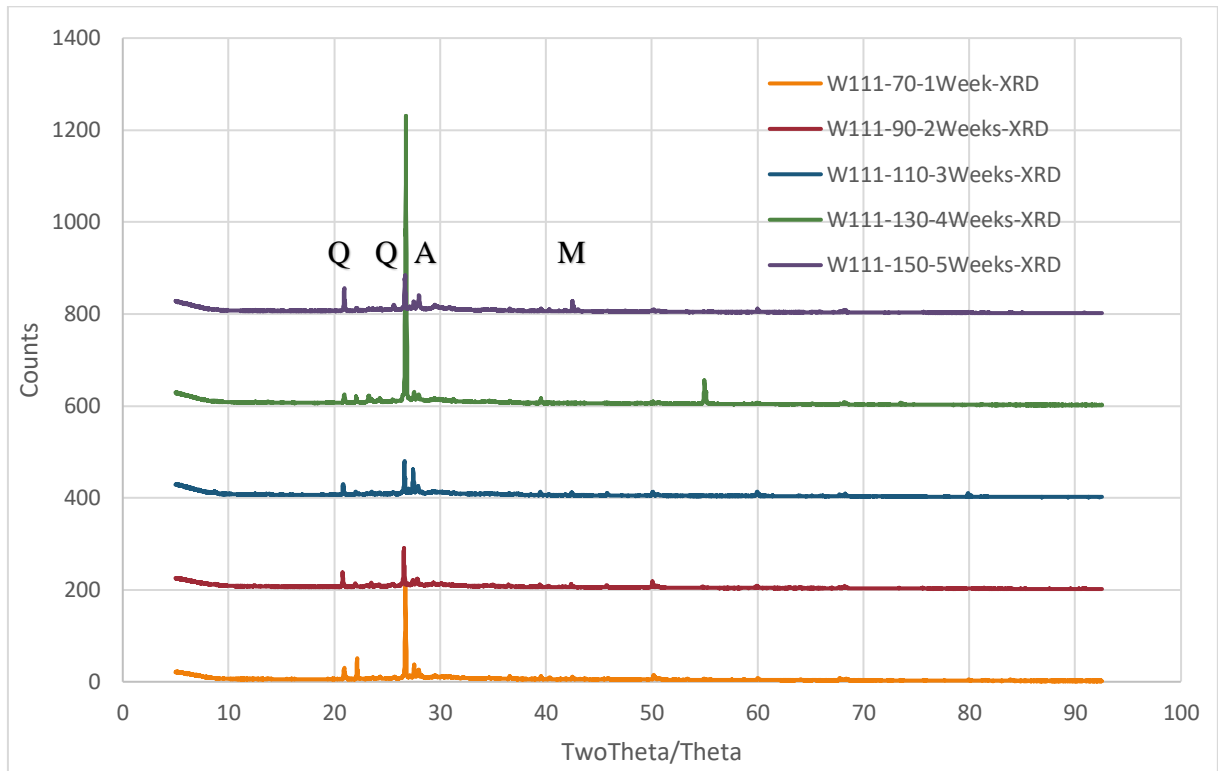


Figure 10: XRD outputs for the five given curing conditions, *Q* is quartz, *A* is Albite, and *M* is Microcline.

#### 4.1.3 SEM

A Scanning Electron Microscope (SEM) was used to study the microstructure and for qualitative composition analysis of the geopolymers starting from 70°C up to 150°C respectively, as shown in figures 11, 13, 15, 17 and 19. In addition, Elemental analysis investigations were conducted for these five conditions as given in figures 12, 14, 16, 18 and 20, and in tables 4, 5, 6, 7 and 8 respectively.

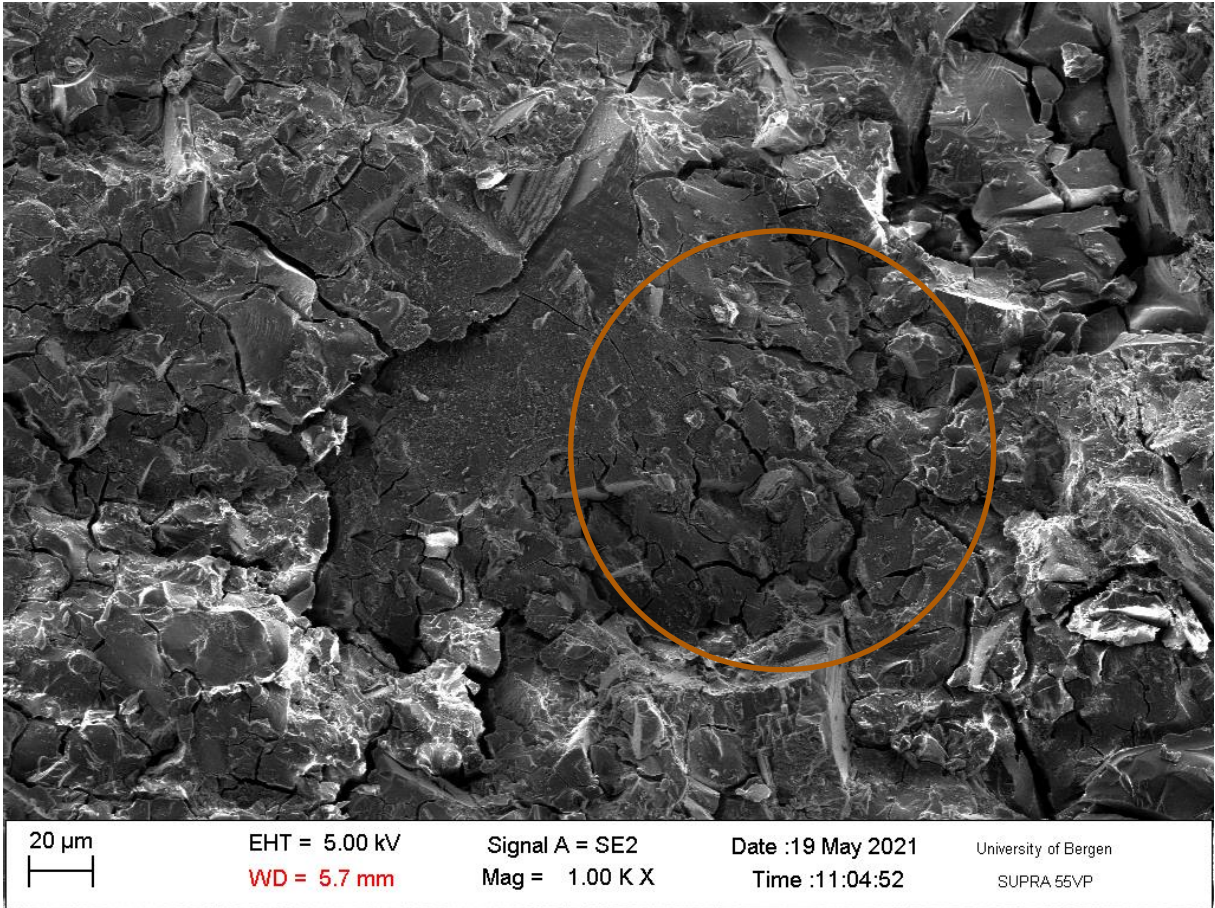


Figure 11: Scanning Electron Microscope (SEM) image for 70C cured sample at 20 micron magnification and orange circle presents the cracks observed.

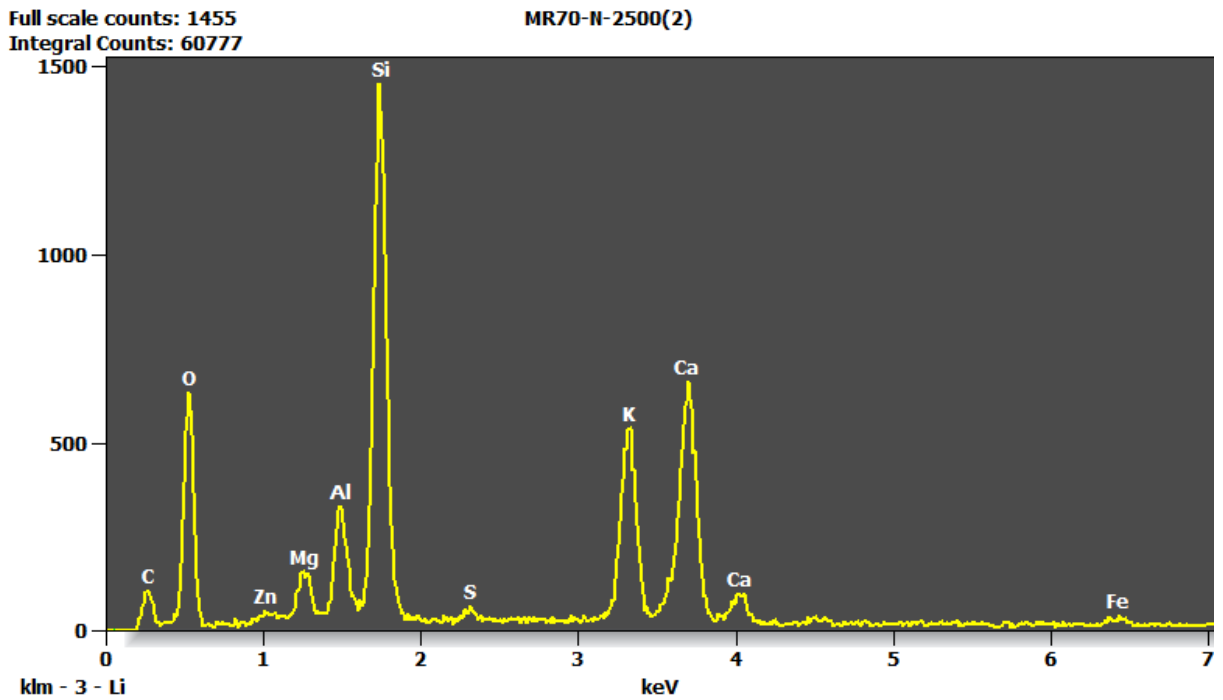


Figure 12: Energy-dispersive X-ray spectroscopy image for 70C cured sample.



Tabell 4: Elemental analysis for 70C cured sample outputs.

Element	Weight %	Atom %	Formula
C	5.82	10.21	C
O	43.66	57.54	O
Mg	2.08	1.81	Mg
Al	3.73	2.92	Al
Si	18.28	13.72	Si
S	0.45	0.30	S
K	9.85	5.31	K
Ca	14.33	7.54	Ca
Fe	1.33	0.50	Fe
Zn	0.47	0.15	Zn
Total	100.00	100.00	

- 90

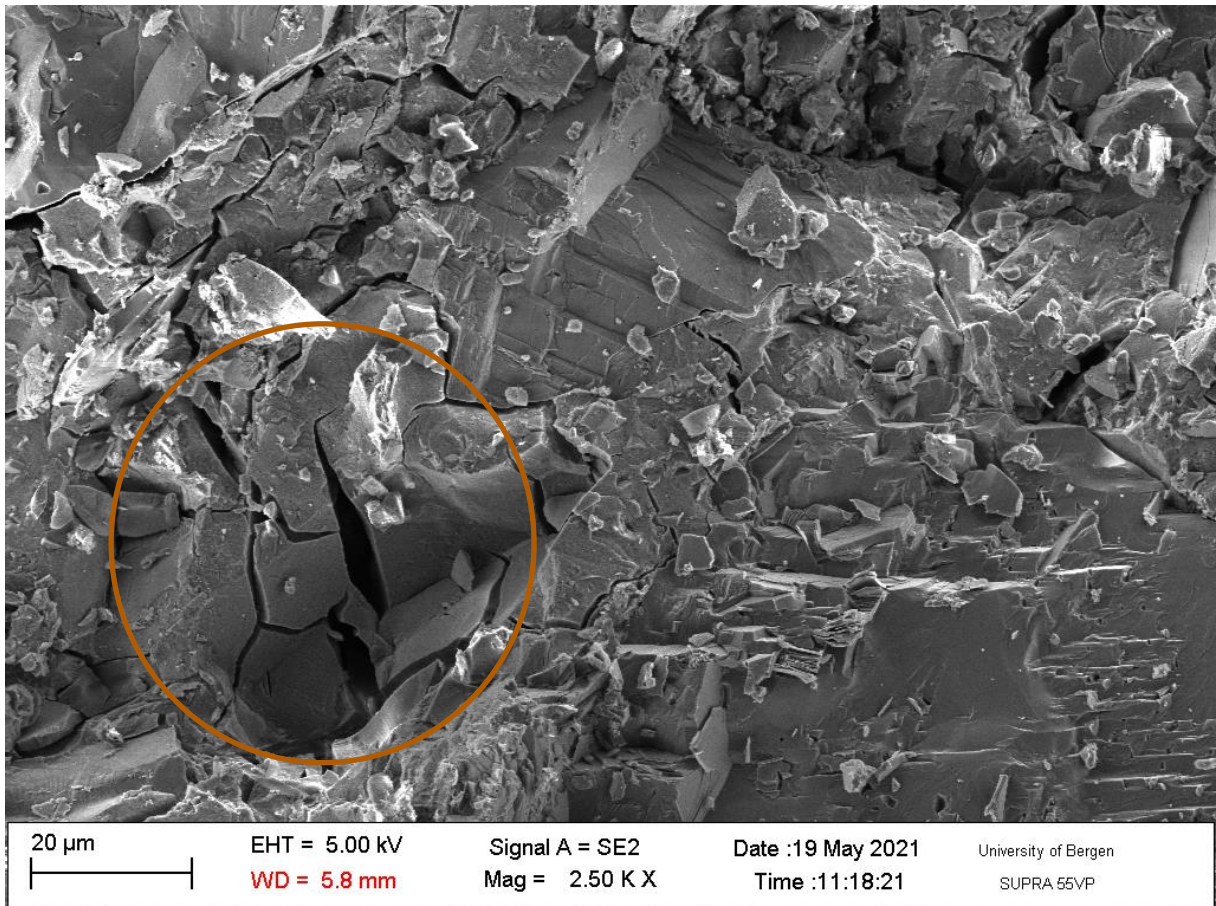


Figure 13: Scanning Electron Microscope (SEM) image for 90C cured sample at 20 micron magnification and orange circle presents the cracks observed.

Full scale counts: 2388  
Integral Counts: 96860

MR90-II-2500(5)

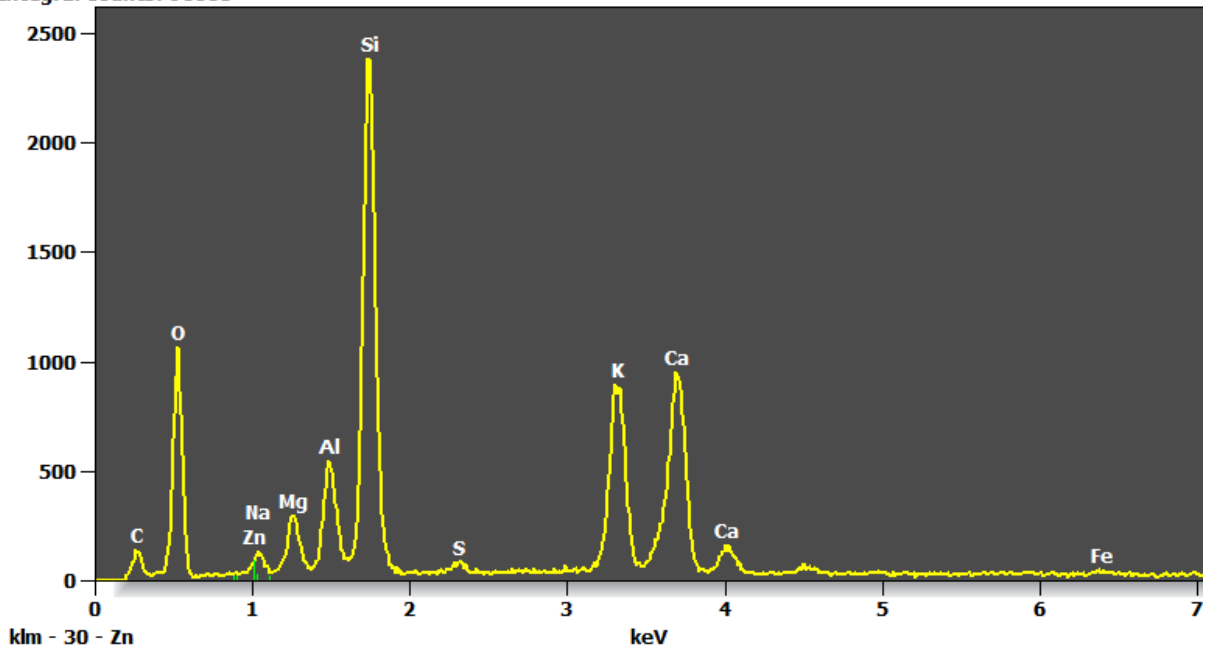


Figure 14: Energy-dispersive X-ray spectroscopy image for 90C cured sample.

Tabell 5: Elemental analysis for 90C cured sample outputs.

Element	Weight %	Atom %	Formula
C	4.71	8.39	C
O	42.38	56.63	O
Na	1.14	1.06	Na
Mg	2.53	2.23	Mg
Al	4.07	3.23	Al
Si	19.20	14.62	Si
S	0.44	0.30	S
K	10.78	5.89	K
Ca	13.58	7.24	Ca
Fe	0.67	0.26	Fe
Zn	0.50	0.16	Zn
Total	100.00	100.00	

- 110

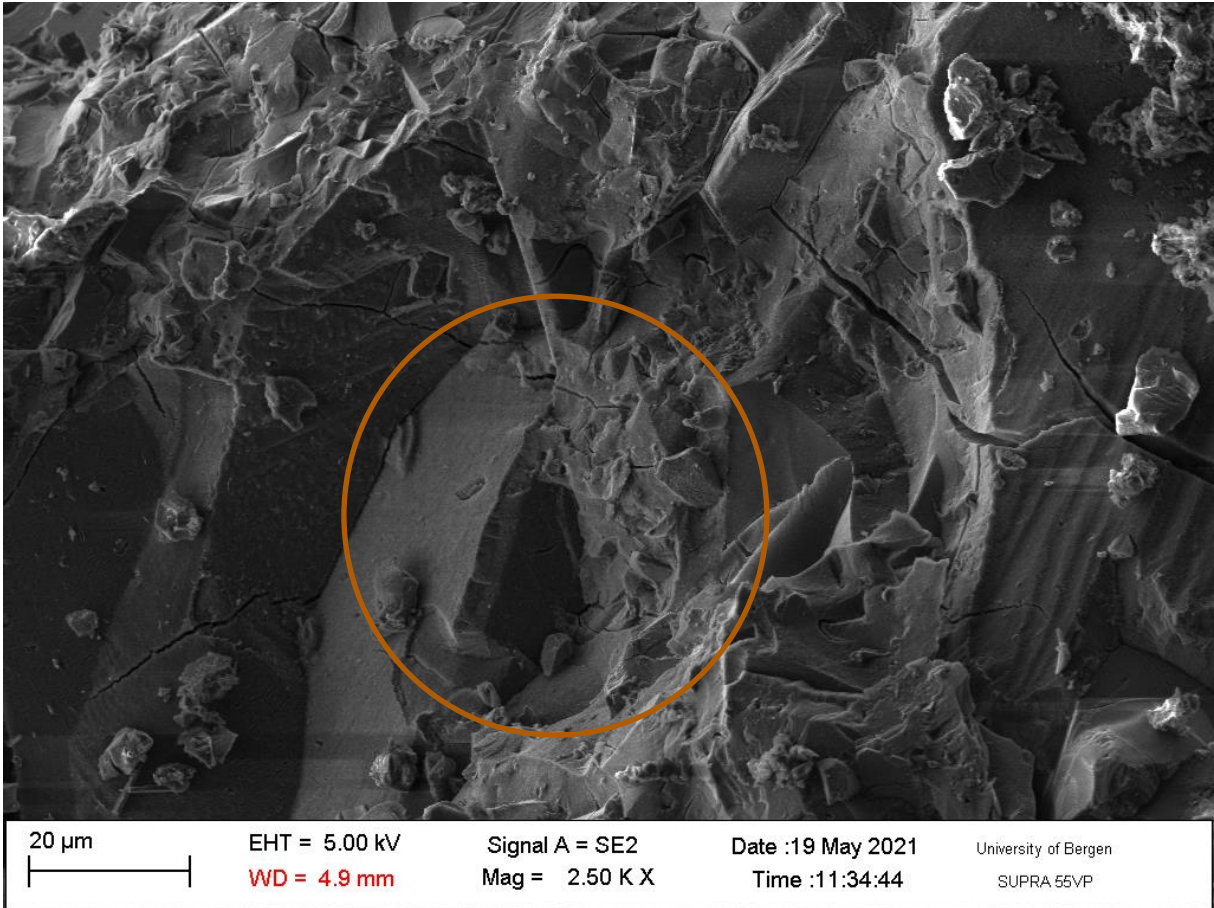


Figure 15: Scanning Electron Microscope (SEM) image for 110C cured sample at 20 micron magnification and orange circle presents the cracks observed.

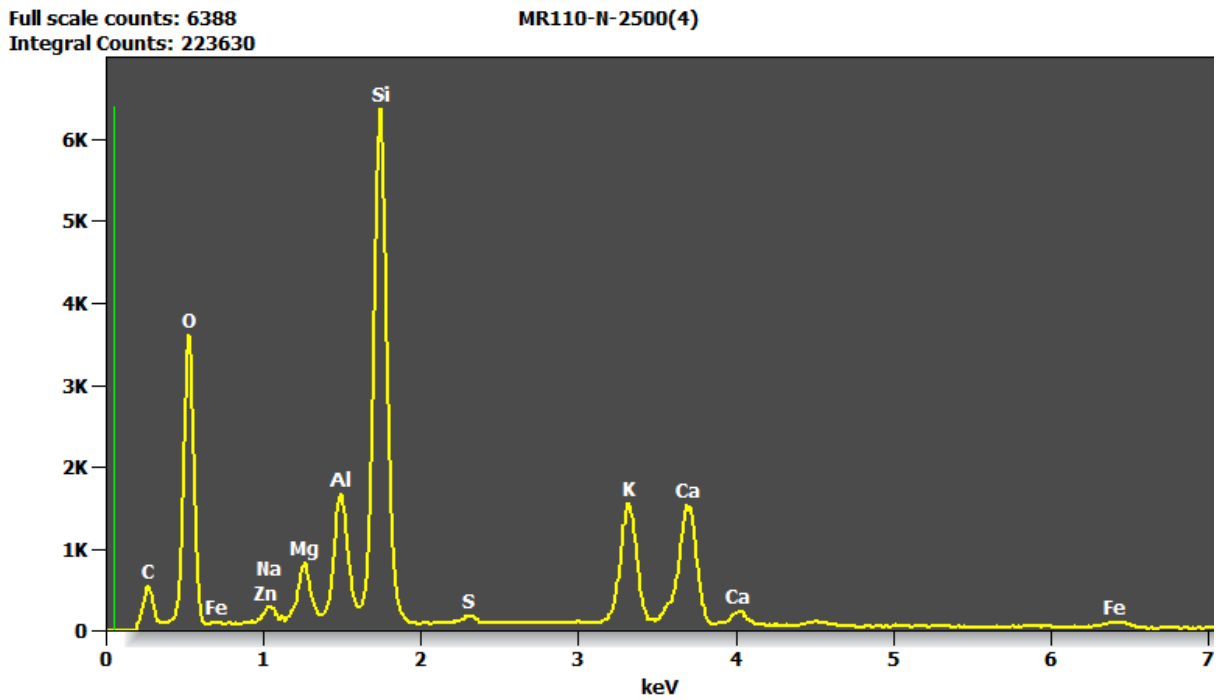


Figure 16: Energy-dispersive X-ray spectroscopy image for 110C cured sample.

Tabell 6: Elemental analysis for 110C cured sample outputs.

Element	Weight %	Atom %	Formula
C	7.19	11.89	C
O	48.10	59.69	O
Na	0.71	0.61	Na
Mg	2.41	1.97	Mg
Al	4.75	3.50	Al
Si	19.83	14.02	Si
S	0.28	0.17	S
K	7.04	3.58	K
Ca	8.15	4.04	Ca
Fe	1.39	0.49	Fe
Zn	0.14	0.04	Zn
Total	100.00	100.00	

- 130

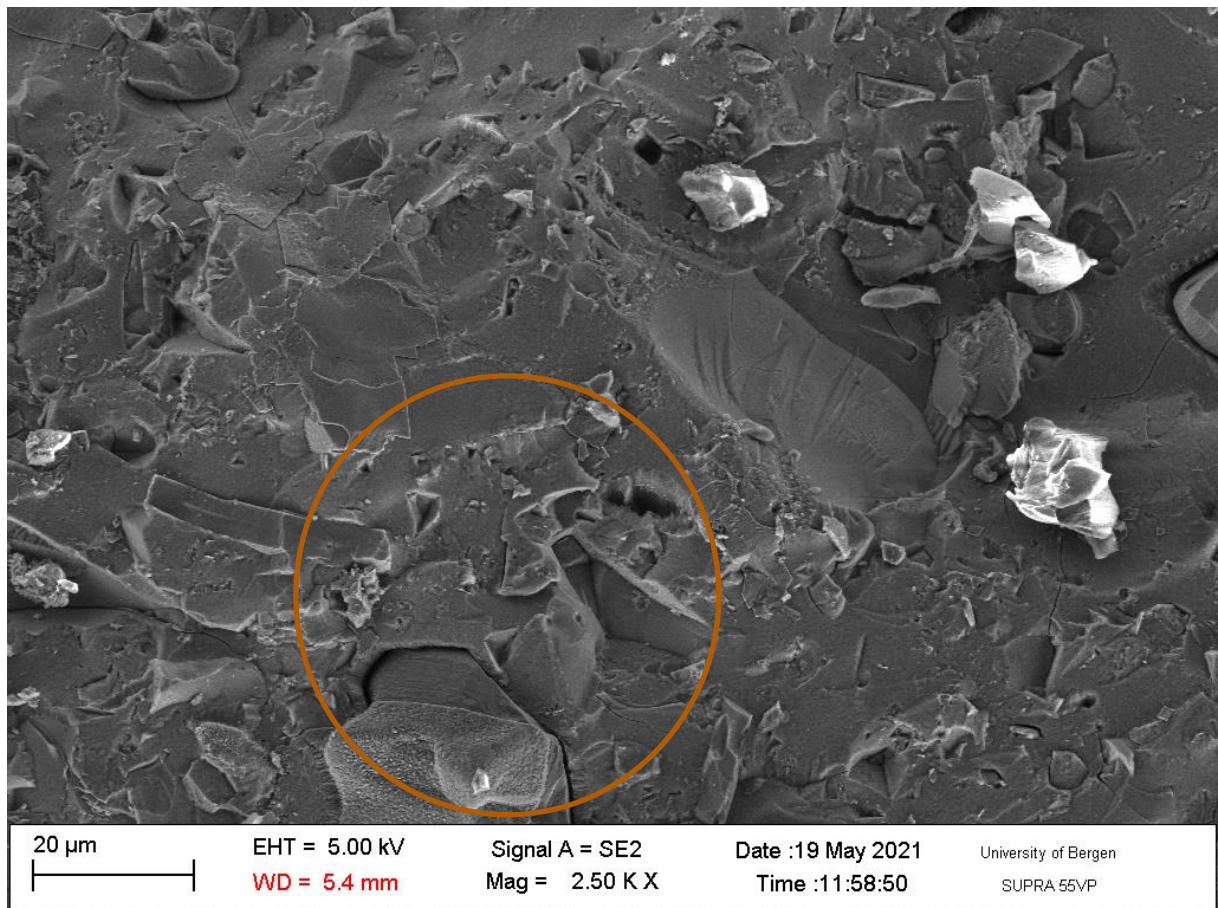


Figure 17: Scanning Electron Microscope (SEM) image for 130C cured sample at 20 micron magnification and orange circle presents the cracks observed.

Full scale counts: 7782  
Integral Counts: 264289

MR130-N-2500(1)

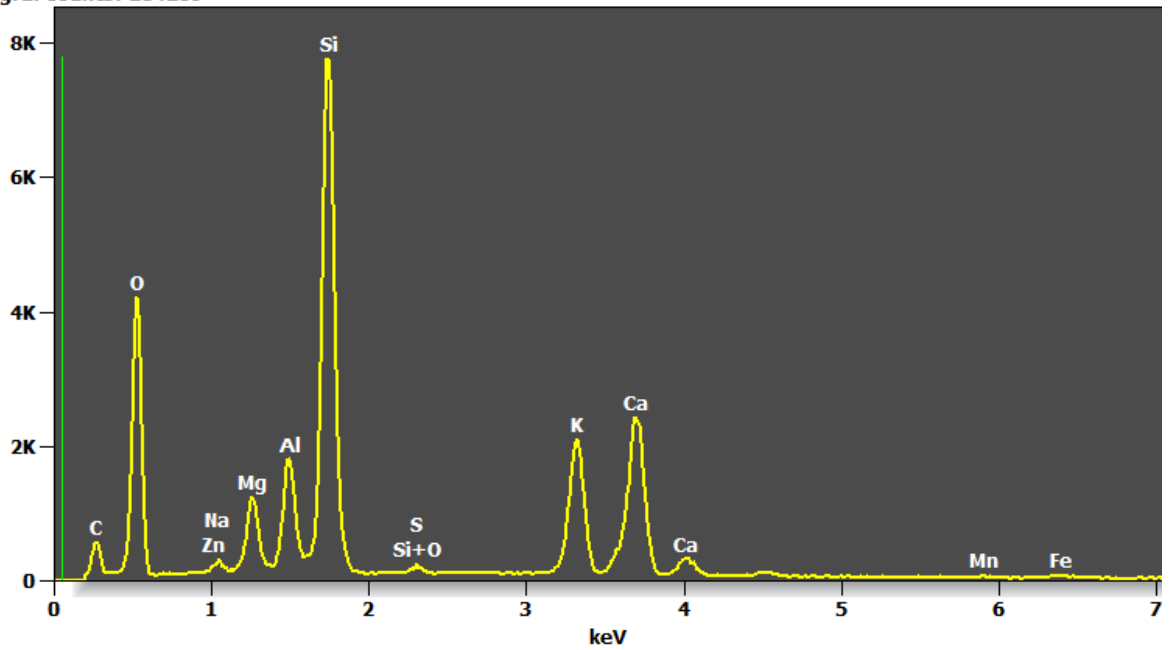


Figure 18: Energy-dispersive X-ray spectroscopy image for 130C cured sample.

Tabell 7: Elemental analysis for 130C cured sample outputs.

Element	Weight %	Atom %	Formula
C	5.85	9.88	C
O	47.14	59.81	O
Na	0.55	0.49	Na
Mg	3.20	2.67	Mg
Al	3.89	2.93	Al
Si	19.74	14.27	Si
S	0.27	0.17	S
K	7.65	3.97	K
Ca	10.93	5.54	Ca
Mn	0.22	0.08	Mn
Fe	0.35	0.13	Fe
Zn	0.21	0.06	Zn
Total	100.00	100.00	

- 150

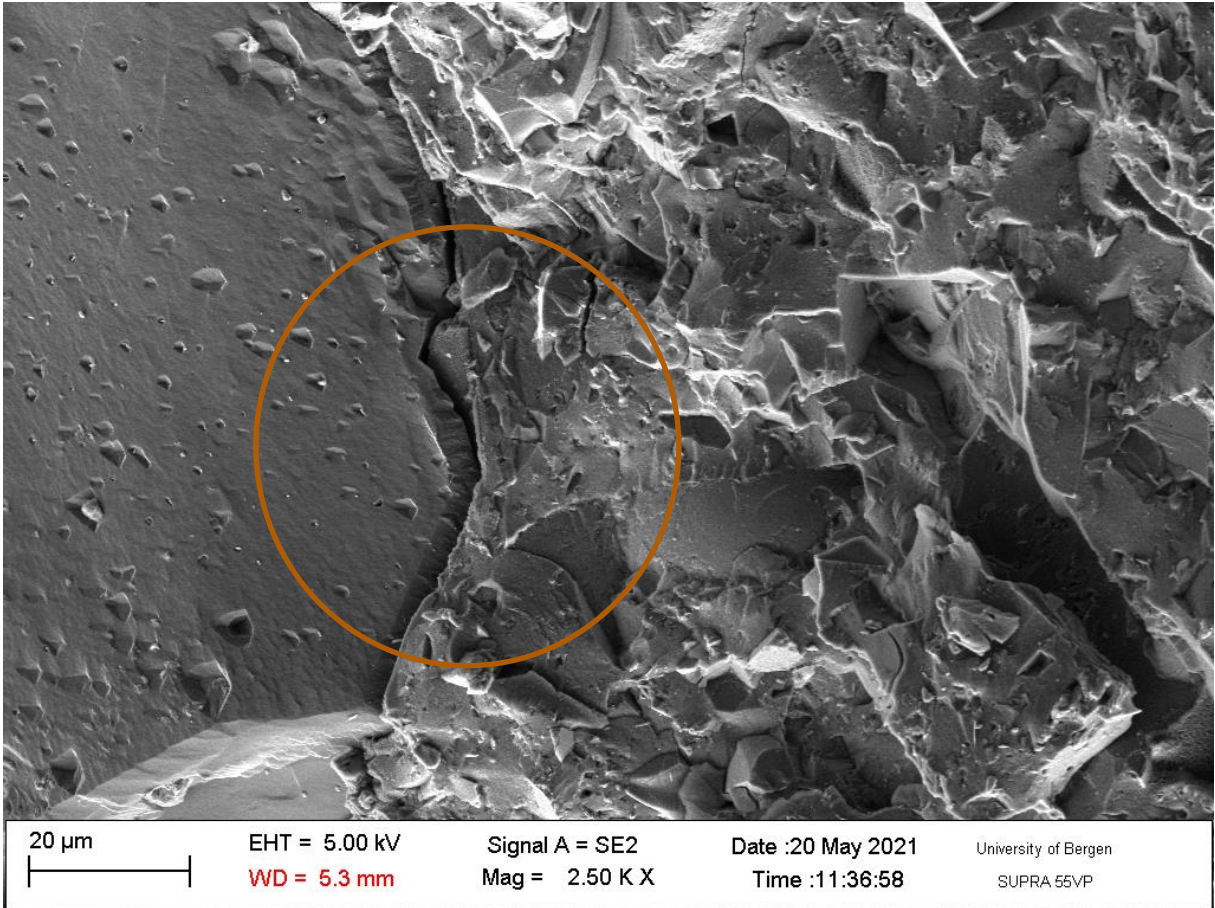


Figure 19: Scanning Electron Microscope (SEM) image for 150C cured sample at 20 micron magnification and orange circle presents the cracks observed.

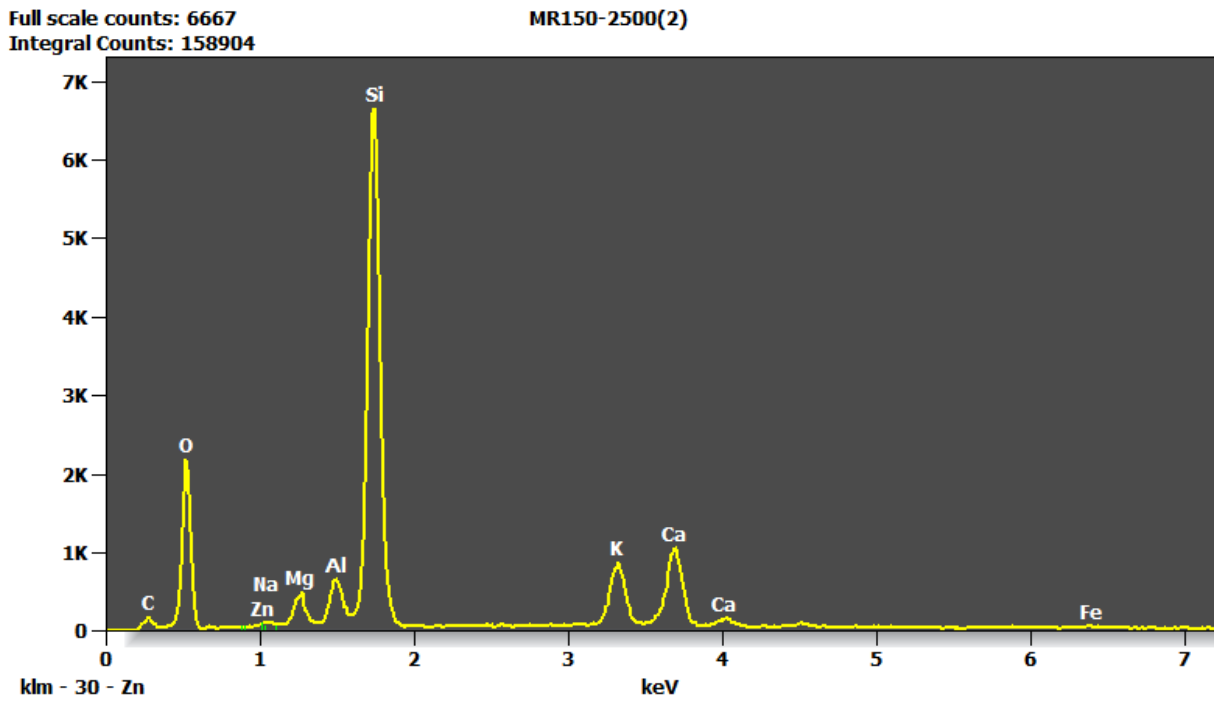


Figure 20: Energy-dispersive X-ray spectroscopy image for 150C cured sample.

Tabell 8: Elemental analysis for 150C cured sample outputs.

Element	Weight %	Atom %	Formula
C	3.97	6.84	C
O	45.62	59.07	O
Na	0.46	0.42	Na
Mg	2.11	1.80	Mg
Al	2.37	1.82	Al
Si	29.92	22.07	Si
K	5.97	3.16	K
Ca	8.81	4.55	Ca
Fe	0.46	0.17	Fe
Zn	0.31	0.10h	Zn
Total	100.00	100.00	

## 4.2 Discussion

From UCA outputs for the given five conditions to prepare a geopolymer cement for geothermal wells application, it is observed that the higher the temperature and the longer the time, the higher the compressive strength to be achieved as shown in figures 8 and 9. As in figure 9, presents the highest achievable compressive strength up to 3600 psia within 32-days as a step-temperature increase experiment. Thus, the lower the curing temperature and curing period the lower the compressive strength down to 2400 psi within 7-days as shown in figure 5.

XRD analysis presents similar patterns for the original composition of Granite precursor. Granite precursor composition was as expected dominated by Quartz, Albite, Microcline and Biotite (Khalifeh et al., Kamali et al... ). No major changes can be observed over 7-days of thermal curing at 70°, 14-days of thermal curing at 70° and 90°, 21-days of thermal curing, 35-days of thermal curing at 70°, 90°, 110°, and 150° respectively as a step-temperature increase experiments rather than the absence of biotite from all geopolymer pastes. Except for the 28-days of thermal curing at 130°C geopolymer sample, there is no any considerable or significant minerals changes were found between these mentioned curing temperatures and duration rather than the amorphosity hump was bigger as the temperature and time goes higher as shown in figure 10 which was also discussed by 19-24.

These outputs of XRD data are matching what have been observed by 19 - 24 in which studied the same mineralogy and chemical compositions of rock-based geopolymers formed in presence of potassium silicate solutions such meta silicate, tri silicate and their mixtures as

activators. They found that quartz as the major phase, and albite, and microcline as minor phases. The results also indicate that different types of feldspar such as Albite and Microcline react differently with the variation in temperature and time put into the geopolymers, however, more data is needed to fully understand these complex chemical processes.

By correlating SEM and Energy-dispersive X-ray spectroscopy outputs, it was observed that the higher the curing temperature and curing period, the higher the silicon content in the geopolymer samples. As shown from 1500 counts with 18.28 wt.% at 70°C curing condition up to 7000-8000 counts with 19.74 wt.% and 29.92 wt.% for 130°C and 150°C curing conditions respectively. Silicon content was important to be investigated due to its contribution on the compressive strength of the geopolymer cements. However, the oxygen counts and content were varies with the applied conditions that started by 600 at 70°C curing condition and then increase to 1100 counts at 90°C, 3600 at 110°C, and 4500 at 130°C. And then oxygen content was decreased to 2200 counts at 150°C. However, the utilized voltage for SEM experiments was low, so it not enough to be sure about crystallization phenomenon for our geopolymer samples.

From figure 11, SEM image for 70C cured sample shows an amorphous structure in addition to the presence of cracks that might be from shrinkage or expansion if the sample. Orange circles on figures 11, 13, 15, 17 and 19, present the observed cracks on the five investigated conditions, where the higher the heat curing temperature and the longer curing period, the less cracks were observed from the SEM images as mentioned.

Therefore, by integrating all outputs of UCA, XRD, and SEM, it is observed that the minerals were reacting differently under the applied conditions through the step-temperature increase tests and curing period. From these observations, UCA data might be correlated with the mineral consumptions through the observed phase changes in XRD and elemental analysis data in which increased the compressive strength by decreasing the transient time for the UCA machine through the given samples. In addition, the higher the silicon content was observed by the EDX which means the higher the silicon content was released to achieve higher compressive strength was observed by the UCA as presented in the results section.



## 5 Conclusion

Therefore, it can be concluded as geopolymers can be considered as a good cementing material candidate in geothermal wells. Geopolymers as cementing materials have the characteristics which are needed for geothermal wells applications. After integrating and interpreting XRD, UCA and SEM outputs, geopolymer minerals were reacting differently under the applied conditions through the step-temperature increase tests and curing period. UCA measurements can be correlated with these minerals consumptions in which led to increase the compressive strength. However, the low voltage SEM experiments still not enough to be sure about crystallization phenomenon for our cured samples. So, more studies are needed to fully understand these complex chemical processes and their effect on compressive strength.

Further work is recommended to be done and conduct more studies in the future, to explain other variables and to be analyzed such as expansions and shrinkages phenomena in for the five given conditions, investigating the expected well life in different type fields, the expected characteristics of geothermal fluids (brine or steam), studying other static and circulating temperatures expected for the various type of fields, the needed weight range for cementing slurries, the type of drilling muds expected to be used in geothermal wells, economic limitations on geothermal completion systems, minimum strength desired for cement and acceptable strength, Porosity and permeability, desired and accepted, the desired bonding strength to pipe and formation, etc...

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