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Influence of Limestone Powder on Mechanical and Durability Properties of Alkali Activated Geopolymer Concrete

by

Naeem Babar

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degree of Master of Science

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*I want to dedicate this achievement my parents, teachers and friends who always
encourage and support me in every crucial time*



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Acknowledgement

In the Name of Allah, The Most Gracious, The Most Merciful. Praise be to God, the Cherisher and Sustainer of the worlds. All thanks to Almighty Allah, The Lord of all that exist, who bestowed me with His greatest blessing i.e. knowledge and Wisdom to accomplish my task successfully.

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Naeem Babar

Abstract

Ordinary Portland concrete is the most widely used construction material due its several positive attributes. Such as high compressive strength and readily availability of its raw materials. However, it is turning out to be generating high amounts of CO₂ due to the cement production process. Portland cement concrete can be effectively replaced by geopolymer concrete (GPC), which is manufactured without cement. The naturally available geopolymers which have alumina and silicates (aluminosilicate) as their composition could be used as binders to form this new concrete. The GPC has dense microstructure, it is sustainable and environmental friendly. GPC's reduces greenhouse gas emissions as it does not incorporate cement. In this study, a geopolymer concrete has been manufactured using ground granulated blast furnace slag (GGBS) as major precursor. Alongside GGBS the limestone powder is used as 5%, 10%, 15%, and 20% replacement of GGBS. Flow ability, compressive strength, flexure strength and durability properties are investigated. Porosity, sorptivity, XRD, EDX, SEM, are used to evaluate the microstructural and durability aspects of GPC. The mix design includes GGBS, water, sodium hydroxide, sodium silicate, and a water reducing agents. Hence the study emphasis the use of GGBS as base material instead of commonly used fly ash and investigates the effects of limestone powder on the properties of GPC.

Mechanical testing indicates that as the limestone powder contents are increased, the compressive and flexure strengths are increased. Geopolymer+20% limestone powder shows the maximum compressive strength which is 34.44 MPa. The reduction in compressive strength as compared to Geopolymer+20% limestone powder is 6.62%, 11.76%, 15.68 and 18.21% respectively, 15% gives 32.16 MPa, 10% gives 30.39 MPa, 5% gives 29.04 MPa and pure Geopolymer gives 28.17 MPa. The Flexural strength of GPC+5%, GPC+10%, GPC+15%, GPC+20% increased as compared to the reference specimens (Geopolymer). The increase observed is 0.674%, 32.94% and 93.83% and 205.89% respectively. The GPC with 20% limestone powder has highest compressive and flexural strength, while its durability testing shows a denser structure with decreased porosity and sorptivity compared

to the other mixtures. As, the limestone powder is increased the flowability of the GPC is reduced. Flow ability of Geopolymer decreases with increase limestone percentage. Pure GGBS slag gives 96% flow. Addition of limestone with 5% increment up to 20% gives 93%, 90.4%, 88%, and 86.5% flow. In addition, the unusual chemical compounds as are observed by SEM examination in GPC as are formed with fly ash and other precursor based GPC. The production of size able clouds of tobermorite in the presence of limestone powder are observed. XRD analysis reveals enhanced crystallinity with increasing limestone concentration, whereas EDX reveals a different range of chemical compositions and crystalline behavior. Despite reports of severe efflorescence, the addition of limestone powder enhances the overall mechanical and durability qualities of GPC. Although with 20% limestone powder, higher compressive strength is achieved however flowability is reduced due to higher finer particles. Higher flexural strength and beneficial microstructural properties reported in this thesis could enhances the GPC's reputation as an eco-friendly and effective building material. Further investigation and advancement of geopolymer are recommended so that a promising, long-lasting and eco-friendly building materials in the construction industry could be developed.

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Abbreviations and Symbols

ASTM	American Society of Testing and Materials
CA	Coarse Aggregates
EDX	Energy-dispersive X-ray Spectroscopy
FA	Fly Ash
GGBS	Ground Granulated Blast Furnace Slag
GPC	Geopolymer Concrete
GPC+5% LSP	Geopolymer having 5% Limestone Powder
GPC+10% LSP	Geopolymer having 10% Limestone Powder
GPC+15% LSP	Geopolymer having 15% Limestone Powder
GPC+20% LSP	Geopolymer having 20% Limestone Powder
mm	millimeter
MPa	Mega Pascal
PC	Plain Concrete
PRC	Plain Reinforced Concrete
S	Sand
s	Second
SC	Strength in Compression
SEM	Scanning Electron Microscopy
w/c	Water-cement Ratio
XRD	X-ray Diffraction

Chapter 1

Introduction

1.1 Background

A crucial component of sustainable development is the investigation of environmentally suitable substitutes for traditional materials and technology. We can reduce energy consumption, prevent environmental harm, lessen carbon dioxide emissions, and save industrial costs by choosing these options. Due to the recent major increases in human living standards and population growth, there is an increasing requirement for energy in structures. Adopting sustainable construction practices is strongly recommended due to the depletion of fossil resources, the increase in energy demand, and the negative environmental effects. One example of this kind of cutting-edge technology is the creation of geopolymer concretes. Unlike traditional concrete, geopolymer concrete employs activators such as sodium hydroxide and sodium silicate, which acts as a binder together with precursor components including fly ash, slag, and rice husk ash. The creation of geopolymer concrete is started by the reaction between precursors and activators. As a result, geopolymer concrete which is made of composites made of several materials becomes a feasible way to reduce greenhouse gas emissions.

An eco-friendly substitute for conventional Portland cement-based concrete is geopolymer concrete. It is created by reacting alkaline solutions with aluminosilicate materials including metakaolin, fly ash, and ground granulated blast furnace slag (GGBS). By building a three-dimensional web of interconnected mineral

chains, the geopolymerization process replaces the resource-intensive limestone-based cement used in traditional concrete.

Enhanced compressive strength, longer lifespan, and lower carbon dioxide emissions are just a few benefits of geopolymer concrete. Lower temperatures are needed to produce geopolymer concrete than traditional cement, which saves energy and has a less environmental effect. Additionally, recycling and waste material management are aided by the use of industrial by-products like fly ash or GGBS. The use of geopolymer concrete in building has drawn attention as a sustainable solution that fits with international initiatives to encourage environmentally friendly behavior and lessen the environmental impact of infrastructure development.

GGBS slag when used as replacement of cement in Geopolymer concrete gives compact structure. The size and configuration of the pores have a direct impact on compressive strength, a crucial characteristic that is frequently used to characterize structural concrete. In general, a cementitious matrix with fewer pores has a tendency to have a higher compressive strength, whereas a matrix with more pores frequently has a lower strength. Additionally, it is acknowledged that the water-to-cement ratio (w/c) affects the strength of concrete. It is noteworthy that the strength of the concrete might exhibit significant variation contingent upon the particular constituent materials utilized, even in the presence of a fixed w/c ratio [2].

In Geopolymer, a zone of interface exists between the hydrated cementitious paste and the coarse aggregate particles in concrete, which is a multiphase mixture of coarse aggregate (CA) or fine aggregates (FA) contained in a mortar matrix. As a result, the interfacial area, cement paste performance, and solid aggregate parameters significantly affect the characteristics of concrete. It has exceptional automation qualities and sustainability as opposed to regular concrete with ordinary strength features [3]. Geopolymer concrete mixture design and quality control can be improved by using the right concrete mix chemicals (activators) and adjusting the concrete's compressive strength, which is governed by the mixture proportions [1].

1.2 Problem Statement and Research Motivation

Although concrete is the most suitable and viable construction material and is extensively used around the world. However, there are serious environmental consequences with its increasing use as construction material for infrastructure development around the globe due to high carbon emission and energy consumption in cement manufacturing, and depletion in its raw materials. In order to meet the growing demand for high-performance, eco-friendly concrete, concrete properties must be changed in a way that could eliminate the carbon dioxide emissions during manufacturing. "The utilization of geopolymer concrete presents a viable approach to mitigate carbon dioxide emissions, establishing it as an eco-friendly substitute for conventional building materials. Researchers are trying produce geopolymer concrete with different precursor to achieve standard mix design procedure and ingredients. In this study, the use of ground granulated blast furnace slag (GGBS) to produce cement less concrete. In addition, limestone powder is added to reduce the amount of GGBS and observe the impact on geopolymers concrete properties".

1.2.1 Research Questions

The following inquiries are addressed in the research study:

1. Is it possible to prepare Geopolymer concrete by using GGBS instead of fly ash as precursors?
2. How does the combined influence of alkali activator and limestone powder (mixed ratio) improve compressive strength in GGBS-based geopolymer concrete?
3. How much improvement in the microstructural and durability qualities of geopolymer concrete could be achieved by inclusion of limestone powder?
4. In GGBS-based geopolymer concrete, up to what proportion of limestone powder could be used in place of the binder?

5. What are the possible negative consequences of using limestone powder in geopolymer concrete?

1.3 Overall Goal of the Research Program and Specific Aim of this MS Thesis

The main objective of this study is to develop environmentally friendly and sustainable concrete for construction projects. The primary focus of this master's thesis is to examine how adding limestone powder affects the mechanical, microstructural, and durability characteristics of geopolymer concrete made from alkali-activated slag. The study aims to provide insights into potential enhancements that could arise from adding limestone powder to improve the performance characteristics of this specific concrete composition.

1.4 Scope of Work and Study Limitations

The mechanical, microstructural, and durability properties of specimens composed of geopolymer based on GGBS are investigated in this work. The specimen has been prepared and evaluated in accordance with ASTM standards. On the constructed concrete, tests for flow ability, flexural strength, compressive strength, porosity, sorptivity, X-ray diffraction (XRD) and EDX spectrums, and scanning electron microscopy (SEM) were performed. It's crucial to keep in mind that traditional testing aren't conducted at high temperatures, and this study doesn't analyze concrete corrosion.

1.4.1 Rationale Behind Variable Selection

The use of concrete has gradually started to negatively impact the environment and dramatically depleting the natural resources. The use of slag and limestone powder could reduce the dependency on natural resources and waste while simultaneously enhancing concrete qualities. Sodium hydroxide (activators), Sodium silicate, slag

proportions, and limestone powder are the main rationales. Furthermore, using the waste slag in concrete has a positive effect on sustainability and the economy. All of these components working together might potentially replace of the cement content at the same time.

1.5 Contextual Gap

Following are the identified gaps in literature:

- Manufacturing the geopolymer concrete having GGBS with limestone at 2.5 alkali-activated ratio has not been examined.
- The behavior of limestone in gel formation with a 14M molarity of sodium hydroxide (NaOH) has not been examined.
- Conditions of Ambient Curing. (The behavior of limestone and GGBS when cured in the ambient has not yet been investigated.)

1.6 Novelty of Work, Research Significance and Practical Implementation

To the best of the scholar's knowledge, a significant amount of research is still lacking regarding the combined effects of slag-based geopolymer and limestone as a partial replacement under ambient settings. Producing high-performance, environmentally friendly concretes is a promising area with untapped potential. Few studies have been conducted on the influence of limestone powder on the characteristics of geopolymer concrete. In particular, the Geopolymer, which is built on fly ash instead of GGBS. The building and civil engineering industries may find significant uses for these materials. This novel material not only fills a vacuum in the literature but also has promise for raising the general caliber and environmental friendliness of concrete. Its impacts go beyond convention; it promotes the development of construction materials that combine high performance and environmental sustainability in a balanced manner.

1.7 Brief Methodology

In this investigational work, we looked into the characteristics of geopolymer concrete that was mixed with ground granulated blast furnace slag (GGBS) and limestone which are added as a partial replacement for slag. Tests were performed on cast samples of concrete after it is cured for 28 days. Each batch of geopolymer mixture have three samples cast for mechanical, microstructural and durability testing. Flow ability of each mixture was measured using flow ability test as per ASTM C1437 standard. Durability testing such as porosity and sorptivity were also evaluated. SEM, EDX and XRD were performed to investigate the microstructure of the prepared Geopolymer matrix.

1.8 Thesis Outline

Chapter 1: Overview (Introduction)

The background of the thesis research, the motivation for the research, the problem statement, the overall objective of the research program, the particular goal of the master's thesis, the scope of work, study limitations, research methodology, and an outline of the thesis are all explained in this chapter.

Chapter 2: Literature Review

An extensive literature review is presented in this chapter, with a focus on geopolymer concrete. The focus is on its essential elements and how they are prepared, which is accomplished by activating the chemicals present in slag. The intricacies and subtleties of the chemical processes involved in the complex geopolymerization process are revealed through the thorough investigation. The story aims to provide readers a thorough grasp of this cutting-edge concrete technology by clarifying the interactions between the various components and procedures that characterize the development and use of Geopolymer concrete as a building material. Key facts and thoughts are succinctly summarized at the end of the chapter.

Chapter 3: Experimental Program This chapter elaborates on the experimental program through rigorous research conducted to verify ideas and assess proposed

approaches. How many tests and qualities will be decided is explained in this chapter. This chapter provides informative information about the research and attempts to enhance credibility through experimentation. There is a brief synopsis at the end of the chapter.

Chapter 4: Results and Analysis The results of the tests that were done are revealed in Chapter 4, along with a thorough analysis of the findings. This section covers the mechanical, microstructural, and durability features of ggbs slag concrete with limestone powder, as well as the contextual framework for evaluating reference concrete. The chapter culminates in a succinct synopsis that highlights the principal discoveries and understandings obtained from the thorough analysis of the materials and their characteristics. At the conclusion of the chapter, there is a brief summary.

Chapter 5: The talks on mix optimization and structural performance are found in Chapter 5. Furthermore, a detailed discussion of practical applications in comparison to traditional concrete has been conducted. The chapter goes into great detail about current advancements and developments in the field of geopolymer concrete. A summary of the entire conversation is provided at the end of this chapter.

Chapter 6: The findings drawn from the examination of the test results and the geopolymer performance data are included in Chapter 6. In addition, suggestions have been made in light of the findings.

Chapter 2

Literature Review

2.1 Introduction

In modern architecture, concrete composed of ordinary Portland cement (OPC) is a crucial structural component [4]. When it comes to the environmental impact of construction processes, materials, and products, the construction industry has a several major problems. The development of highly functional, environmentally friendly materials is currently a focus for engineers, scientists, technologists, and material experts worldwide, especially for application in the building sector. Geopolymer concretes offer a strong way to overcome this difficulty. The eco-friendliness and cost-effectiveness of structures could be greatly increased by using this relatively new but efficient technique. The main difference is that these concretes are made without clinker in the binders. By utilising environmentally friendly production methods and integrating waste from industries and other sources, geopolymer concretes open the door for the construction of sustainable and eco-friendly buildings and structures.

It is an extremely beneficial material for many construction projects due to its remarkable strength, accessibility, and affordability. But there are major disadvantages to producing Ordinary Portland Cement (OPC). It depends on the use of fossil fuels and natural resources like limestone and has high energy requirements. Furthermore, there is a significant quantity of greenhouse gas emissions associated with the production of cement clinker, which is an essential part of

OPC. In addition to deforestation and the use of fossil fuels, the manufacturing of cement clinker is estimated to produce 0.8 tons of carbon dioxide emissions, raising worries about global warming [5, 6, 7].

Ordinary Portland Cement (OPC) has an intrinsic defect that makes it unsustainable, despite its great qualities. This emphasises how vital it is to look into cleaner, more ecologically friendly options. The most recent research data from 2022, which shows that the atmospheric concentration of carbon dioxide has above the allowed limit for clean air, which is 300 parts per million (ppm), reaching a level of 421 ppm, adds even more relevance to this endeavor. With observations from prior years surpassing this record-breaking level, 419 ppm in 2021 and 417 ppm in 2020 [8].

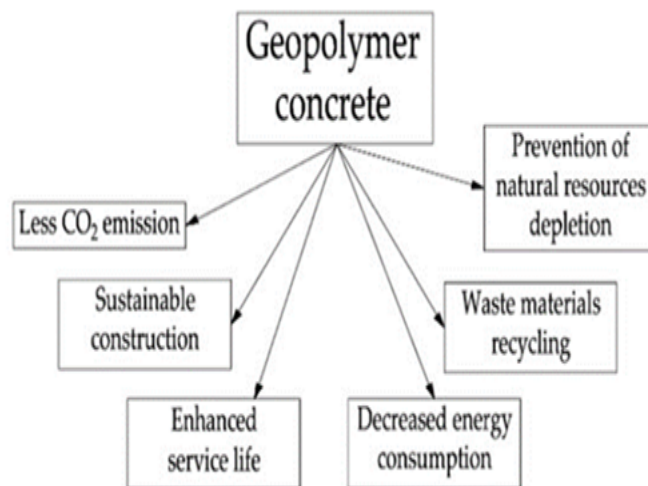


FIGURE 2.1: Special benefits of geopolymer concretes.

French researcher J. Davidovits first introduced the idea of building structures out of geopolymer materials in 1979 [9]. These materials received little attention at first from scientists and researchers. However, the number of studies concentrating on various geopolymer materials and their production processes has increased exponentially during the last 12 years. The growing popularity of geopolymer materials can be attributed to their amazing technological and functional qualities, as illustrated in Figure 1. These materials are chemically resistant to a wide range of solvents and extreme environments. They also have outstanding strength and durability, a long usable life, and low carbon emissions. They also show remarkable tolerance to extreme temperatures. These characteristics make geopolymer

materials very promising for use in the building and medical sectors, among other industrial uses.

2.2 Principle Raw Materials Employed in Production of Geopolymer Concrete

The production of geopolymer concrete, an inorganic polymer-based construction material, involves the heat activation of naturally occurring minerals that are abundant in alumina and silica. When these minerals are combined with alkaline mortars, they polymerize and form the molecular chains that make up the hardened structure that is produced by a solidified adhesive. Table 1 lists the common raw materials used in the manufacturing of geopolymer concrete, including kaolinite, bentonite, fly ash (FA), and a mixture of rice husk ash and wheat straw. These by-products are derived from heavy industry and agriculture. While geopolymer materials and conventional alkaline activation materials are somewhat comparable, the unique structure creation that takes place during the activation process causes a considerable difference in their chemical compositions.

2.3 Principle Raw Materials Employed in Production of Geopolymer Concrete

Thermal activation of naturally existing minerals rich in alumina and silica is required to create geopolymer concrete, a building material based on inorganic polymers. These minerals undergo polymerization when combined with alkaline mortars, which forms the molecular chains that make up the rigid structure of the formed adhesive. Table 1 lists the common raw ingredients used in the production of geopolymer concrete, which include fly ash (FA), bentonite, kaolinite, and a mixture of wheat straw and rice husk ash. These basic resources come from agricultural byproducts and heavy industry. Although there are certain similarities between geopolymer materials and traditional alkaline activation materials, their

chemical compositions differ greatly due to the unique structure that forms during the activation process.

TABLE 2.1: Geopolymer Concrete continents.

Concrete		Binder Material	Activators
Normal Concrete	Conventional	Cement	Cement ingredients react with water & starts hydration process
Geopolymer Concrete		Fly Ash, Metakaolin, Slag, High Calcium wood Ash, Rice Hush Ash, Combinations [44].	Sodium hydroxide, Sodium silicate, Potassium Hydroxide etc. [44].

Three steps are involved in the creation of geopolymers based on fly ash (FA). First, Aluminate and silicate monomers are created during the hydrolysis and dissolution of FA aluminosilicate. The second stage involves the condensation of silicon and aluminium ions which results in the formation of oligomers that help to create a gel with a significant network structure. The gel goes through additional restructuring in the final stage, which triggers the polycondensation process and results in the formation of an amorphous aluminosilicate network. Research results reveal that the content and type of activator have a substantial impact on the strength characteristics of geopolymer composites [10]. Sodium hydroxide and sodium silicate are two prominent activators that are frequently employed in geopolymer composites; hydroxide molarity has been shown to typically range from 6 to 20 M in prior research. Geopolymer composites made entirely of hydroxide have larger porosity, lower strength values, and a greater propensity for shrinkage fractures. On the other hand, the mixture hardens more quickly when a silicate source is added, producing a material with improved properties.

2.4 Classification of Blends Utilize in Composition of Geopolymer Concrete

Like traditional concrete that uses normal ordinary Portland cement (OPC) as a binder, geopolymer concrete formulations call for careful constituent selection

and matching proportions. On the other hand, geopolymer concretes face a more complex problem, whereas the quantities of the elements in OPC-based concretes mainly define their qualities. This intricacy results from the fact that a wide range of factors profoundly affect their characteristics. Unlike OPC-based concretes, where constituent proportions are the primary emphasis, the composition of geopolymer concretes is further complicated by the significant influence of several factors. These factors include temperature and curing times, the water-to-solids ratio, the amount of alkali, the kind and makeup of the aluminosilicate source materials, and the proportions of the various components used in the polymerization process like hydroxides, silicates, and aluminates [11-16].

Just as with conventional concrete that employs Portland cement as a binder, there are numerous approaches to take when selecting compositions for geopolymer concretes, as seen in figure 2.2.

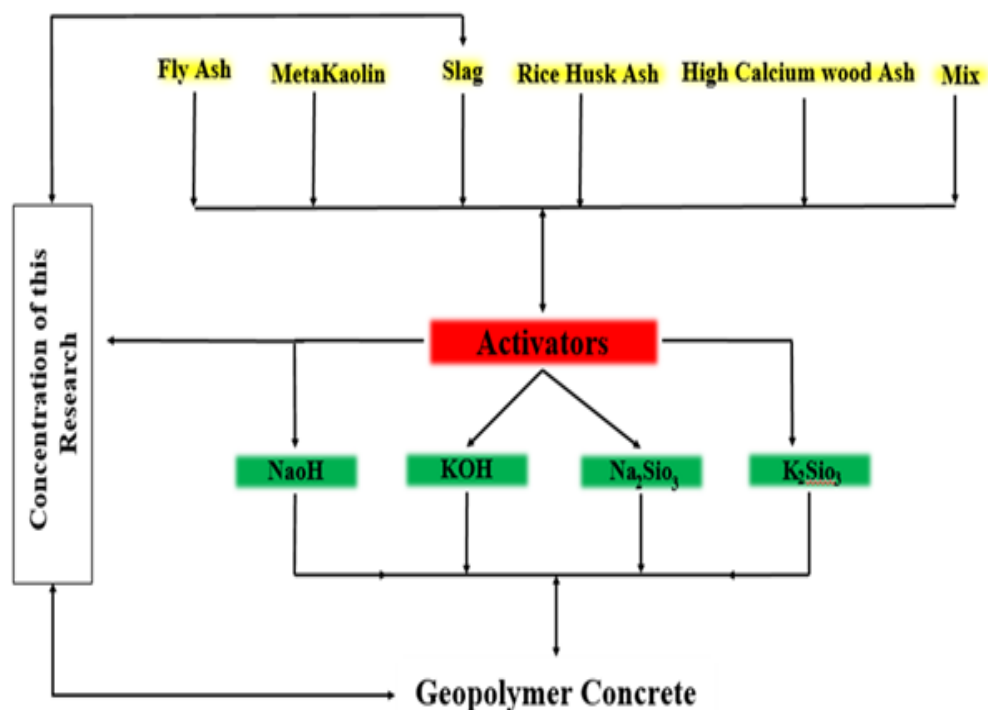


FIGURE 2.2: Categorization of Geopolymer Concrete [44].

The ratio of the activator to the main concentration elements in the geopolymer mixture determines how workable the geopolymer concretes are. Incorporating strategies such as adding hardening retarders and superplasticizers, or varying the water concentration in relation to the binder, can improve workability. It is

noteworthy that the practicality of geopolymer concretes tends to decrease with increasing NaOH concentration. The influence of important parameters on the workability of geopolymer concretes is portrayed in Figure 2.3. The amount of

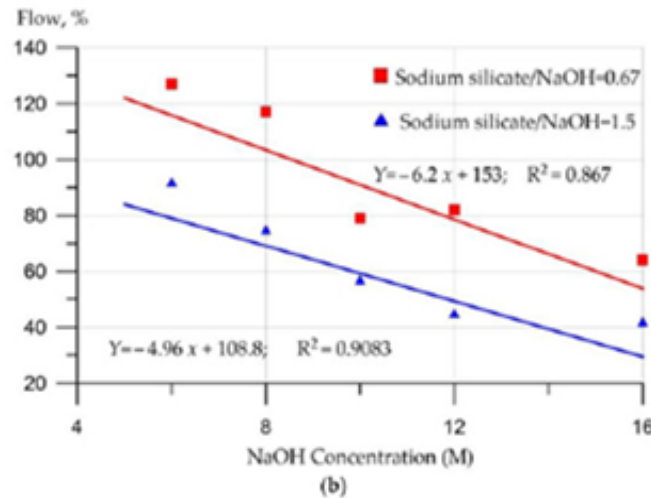


FIGURE 2.3: The primary factors that determine GPC's viability and the strength of each factor's impact.

NaOH in the mixture, the proportion of hydroxides to silicates, the binder's composition, and the addition of superplasticizers all affect how quickly geopolymer concretes solidify. Higher amounts of NaOH quicken the polymerization process in geopolymer compositions, which shortens the hardening time. Nevertheless, the concrete's workability may suffer as a result of this quick hardening. The components and their particle size distribution dictate the strength properties of cured geopolymer concrete. The type of sand and the proportion of binder to sand have an impact on the compressive strength of geopolymer concrete. When the recommended value is exceeded, geopolymer concrete's compressive strength suffers. Studies reveal that the ratio of silicon dioxide to aluminium oxide has a major impact on the polymerization process; a higher ratio results in higher aluminium oxide engagement. As a result, later in the polymerization process, inadequate aluminium oxide is noticed. Additionally, a larger constituent molarity in geopolymer concrete promotes polycondensation, which increases the cured material's compressive strength [37-42]. Research also shows that increasing the ratio of sodium hydroxide to sodium metasilicate as well as the molarity of sodium hydroxide results in improved compressive strength for concrete prepared by using ground-granulated blast furnace slag that has been alkali-activated (GGBS).

Conversely, when the ratio of GGBS to alkali activator is increased, compressive strength often tends to decrease. The main variables and how they affect the compressive strength of geopolymer concretes after 28 days are shown in Figure 4. The mass ratios of aluminium to slag (Al/Slag) and sodium hydroxide to sodium silicate (NaOH/Sodium silicate) are given.

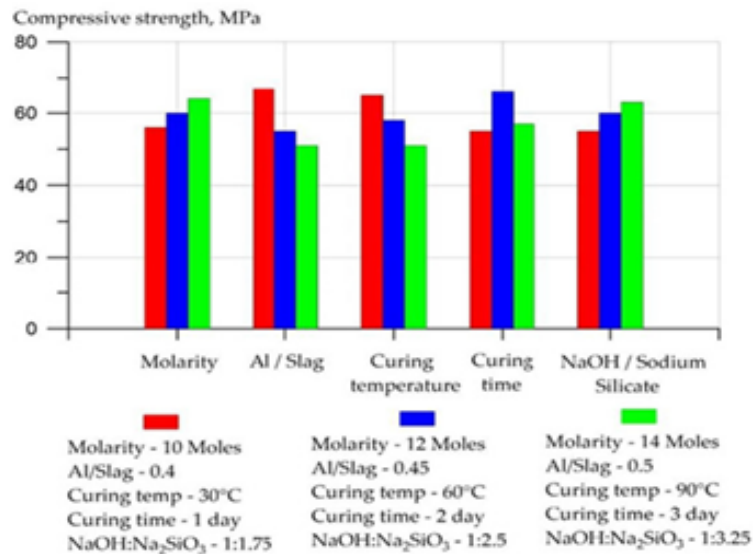


FIGURE 2.4: The primary elements influencing the compressive strength of geopolymer concretes at age 28.

2.5 Chemistry & Micro Structural of Geopolymerization Process

2.5.1 Geopolymer Concrete Polymerization Activities

An alkali-rich activating solution speeds up the polymerization of silica and aluminium oxides in geopolymer compositions, allowing for the development of a polymeric aluminosilicate network in three dimensions. The alkaline activator dissolves silica and aluminum oxide particles, converting them into linked chains of aluminum silicate polymers. The kind of aluminosilicates that are present dictates the particular sort of geopolymer that forms [17].

The interplay between the polymerization process and Ca silicate hydrate saturation binding affects the properties of geopolymeric concrete. The polymerization

of geopolymeric concretes is started by the activation of aluminosilicates when normal Portland cement (OPC) is completely replaced by geopolymeric ingredients. This activation results in the dissolution of aluminosilicate oxides in a very alkaline environment by oxidising the aluminosilicates in an alkaline activator. After dissolution, a gel made of oligomers joined by polymer linkages, such as Si-O-Si and Al-Si-O bonds, is formed. This gel creates the basic structure of the geopolymeric composition when it solidifies, curing into a polymeric matrix that binds and encases any composition particles that have not yet reacted. Geopolymer concretes take the place of conventional calcium silicate hydrate gel formation by substituting aluminosilicates for Ordinary Portland Cement (OPC). By using alumina and silica particles to make polymers, this is accomplished.

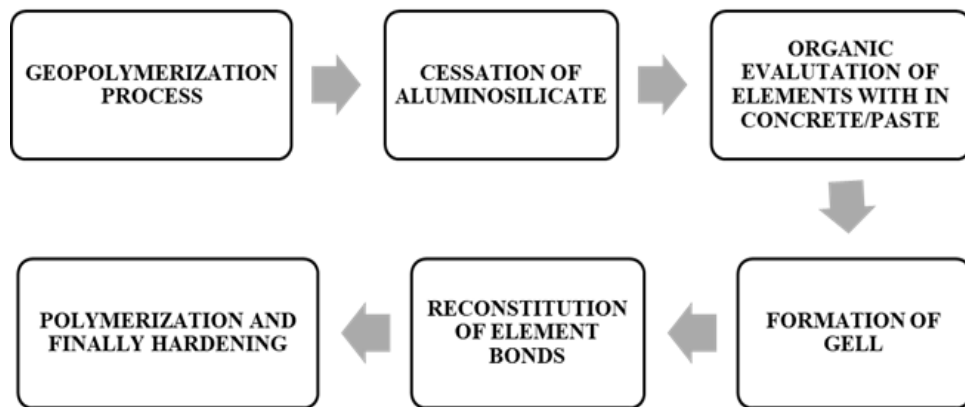


FIGURE 2.5: Main Phases of Geopolymerization Process in Geopolymer concrete production.

The key phases of this transition, as shown in Figure 5, show how the polymerization process in geopolymer concretes advances. Numerous studies have been conducted in the literature on the structural evolution of an alkaline-slag binder utilising ground granulated blast furnace slag, or GGBS. As previously noted, compounds that are exposed to a high concentration of alkaline solution, such as Si-O-Si and Al-Si-O bonds, decompose and become colloidal particles. The colloidal particles gradually condense and harden as a result of this process [36]. The activation mechanism, according to Glukhovsky et al. [19], entails a sequence of densification and disintegration processes. Less stable structural entities are

formed as a result of these reactions, which upset the material's initial structure. These materials then experience compaction as a result of interactions with coagulation structures. Three steps make up the chemical action procedure of geopolymerization, according to Glukhovskiy et al. [19]:

First Stage: In a very alkaline solution, Si-O-Si bond and Al-Si-O bond complexes change from a crystalline to a colloidal state.

Second Stage: More colloidal particles are present, which supports the stages that come after.

Third Stage: Autogenous shrinkage results from the increasing colloidal particles compacting inside the accessible volume.

Different products are formed when calcium hydrosilicate and sodium hydroaluminosilicate combine with a slag-alkaline binder during hydration. When clay minerals are present in the binder, they react with the alkaline activator to form zeolites, or hydroaluminosilicates. Zeolite phases such as tobermorite and hydroxosodalite are examples of those that form with a higher water content than the binder. Moreover, $\text{Na}_2\text{O}-\text{Al}_2\text{O}_3-\text{SiO}_2-\text{H}_2\text{O}$ and $\text{Na}_2\text{O}-\text{CaO}-\text{Al}_2\text{O}_3-\text{SiO}_2-\text{H}_2\text{O}$ are crystalline compounds that can form [20]. X-ray diffraction investigations [19] indicate that during slag hydration, a quick-forming calcium silicate hydroxide gel (CSH gel, $n\text{CaO SiO}_m\text{H}_2\text{O}$) is formed. Then, as the hydration process progresses, hydrotalcite appears. Some specialists have pointed out that xonotlite and calcium hydrosilicates are among the hydrated byproducts of slag binder [21]. X-ray diffraction and scanning electron microscopy have confirmed the existence of calcium hydrosilicate gel. When a geopolymer binder based on GGBS is combined with water, especially at low C/S ratios, the outcome is this gel [22]. Using the same methodology as the authors of [21], scientists found that hydrotalcite, calcite (CaCO_3), and calcium hydrosilicate were among the byproducts of alkali-activated slag hydration one month after hydration began. To sum up, this work explores the complex structural changes of a slag-alkaline binder using GGBS. Dissolution, colloidal transition, compaction, and the creation of various hydration products are all steps in the process. This process is also influenced by elements like the water-binder ratio and the existence of clay minerals. Heat-treated kaolin was used

as the starting material for the geopolymer composition, and it hardened when it came into contact with alkali. A model for the polycondensation process that takes place when geopolymers cure was put out by J. Davidovits [23]. Because of the way they are structured silicon and aluminium atom chains geopolymers can be classified as polymeric materials (Figure 2.6). These theories expand and develop theories from earlier research.

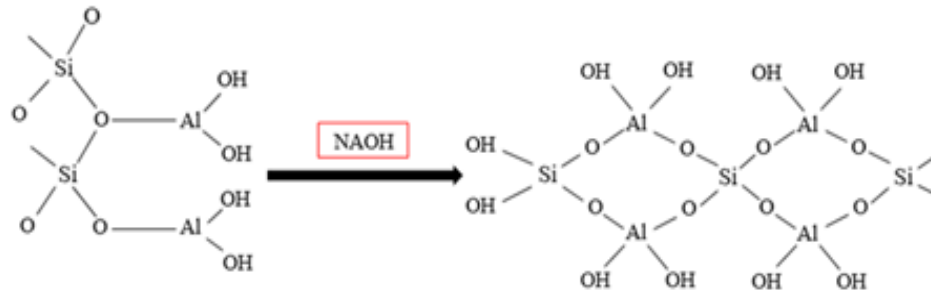


FIGURE 2.6: Kaolinite Polycondensation in alkali surrounding.

Based on data from references [24-29], the configuration of these atoms divides different geopolymer materials into multiple categories. Polysilato-siloxanes, polysilato-siloxo-(disiloxo) compounds, and silicates are a few of them. The several geopolymer complexes are depicted schematically below.

As per Davidovits [24], There are three major steps to the geopolymerization process.

An alkaline solution, usually a concentrated NaOH or KOH solution, is used to dissolve silicon and aluminium oxides during the early part of the process.

Second-stage natural polymer structures are created via disassembled them within their component monomers.

Finally, in the third stage, these monomers change into polymeric components, causing the geopolymer mixture to set and compact.

Complex structures consisting of aluminosilicate utilizing the empirical formula $M-(Si-O)_z-Al-O-n-w-H_2O$ eventually arise during the solidification of geopolymer compositions. In this case, z can be one, two, or three, M stands for K, Na, or Ca atoms or cations, and n indicates the degree of polycondensation. Oxygen bridges are used to link $[SiO_4]$ and $[AlO_4]$ tetrahedra to produce the structure of the

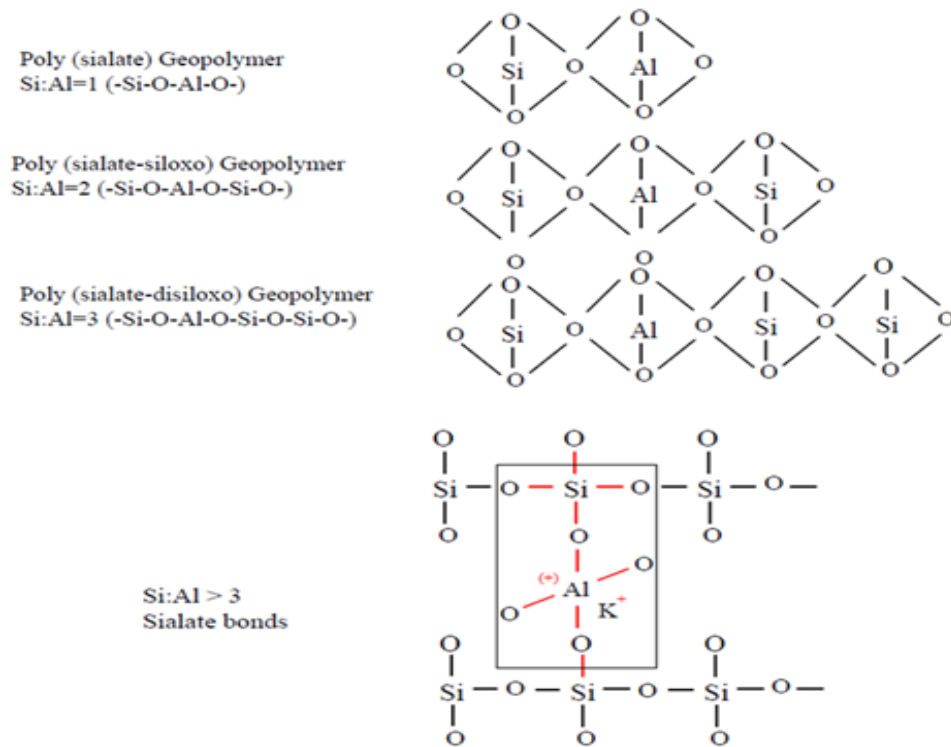


FIGURE 2.7: Structural composition on geopolymer units [24-29].

material. These Si-O-Al compounds occur in rings and chains, where negatively charged four-coordinate Al is balanced by positively charged ions (Na^+ , K^+ , and Ca_2^+).

Using methods like mercury porosimetry, nuclear magnetic resonance, and thermal analysis, researchers have discovered the existence of voids inside geopolymers that contain unreacted water and sodium or potassium cations within the binder. These elements move to the dried material's surface where they are carbonised by the environment. A certain process is followed when efflorescence appears on the surfaces of items constructed of geopolymer materials. In order to create geopolymer structures, Ca and Na^+ ions must first take the place of H^+ ions. Aluminosilicate compounds then experience hydrolysis, which causes the depolymerized glassy structure to disintegrate. Si and Al compounds consequently break down into unstable monomeric forms [30].

A step-by-step approach in the work by Elyamany et al. [31] allows for a systematic understanding of the geopolymerization process. We must give the dissolving process top priority. Initially, the heat-treated, finely powdered raw materials for

aluminosilicates need to be slowly dissolved in an acidic medium. It is imperative to guarantee that these aluminosilicate raw materials are properly dispersed, taking into account their reactivity to the included aluminium as well as their particle dispersion. These variables have a big impact on the dissolution rate. In tandem with the continuous dissolving, silicon and aluminium compounds break down if there is an adequate supply of water. Once in solution, these substance-soften in monomeric form accumulate on the surface as separate particles. This sequential model makes the geopolymerization process easy to understand and emphasises the significance of carefully regulating the raw material's dissolution and dispersion.

As solid particles continue to accumulate in the fluid, a process known as geopolymerization takes place. This explains how the solution polymerizes as a result of interactions between the solid particles that are collecting. However, a higher-level model has been proposed by researchers to explain the material processes involved in geopolymer solidification. This more complex model, which explains how the chemical reactions proceed through several phases, ultimately yields the zeolite phase and amorphous aluminosilicate gel as the two basic components. The solubility of aluminum in an alkaline solution increases with respect to potassium throughout the hydration process, resulting in a decrease in the silicon to aluminum ratio. The way the composition shifts during the geopolymerization process is typical of this change in ratio [32] as describe in figure 2.8.

The processed aluminosilicate raw materials dissolve in an acidic solution to start the geopolymerization process. Following this disintegration, the accumulating constituents proceed through polymerization. A more thorough model highlights the shifting ratios of important components while showing the simultaneous formation of an amorphous aluminosilicate gel and zeolite phase. When creating geopolymer materials, aluminium oxide nanoparticle-enriched aluminosilicate raw materials are activated by alkaline hydroxides. By using this technique, the usual induction phase seen in binders with similar activators is avoided [33]. The geopolymer gel outweighs the silica gel in the early phases of the process. A zeolite phase, distinguished by the presence of crystalline faujasite, forms as the

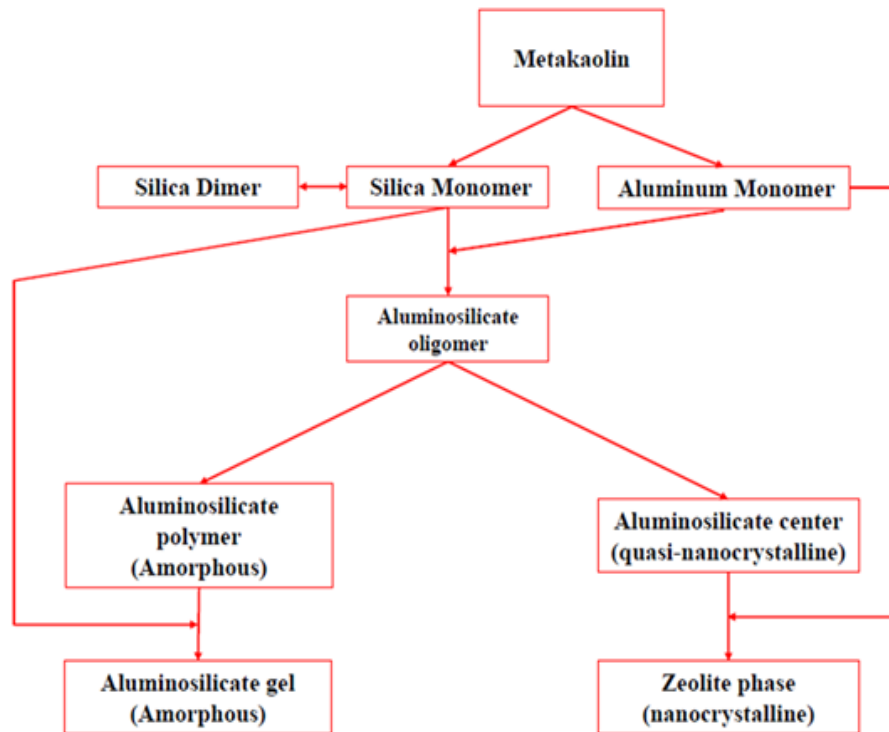


FIGURE 2.8: Steps covered in geopolymerization mechanism according to the model [32].

mixture solidifies. It is interesting to note that the resulting Na-F geopolymer changes in structure to resemble edingtonite.

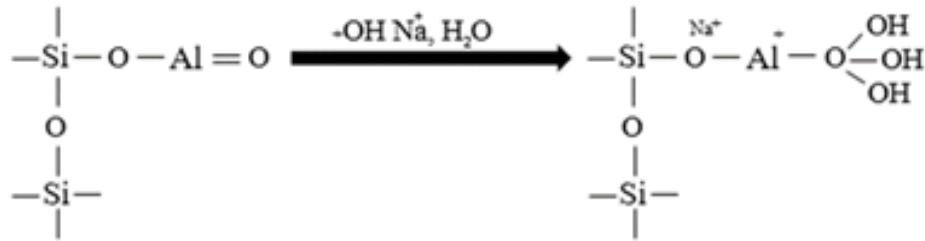
The scientific literature has provided a comprehensive explanation of the mechanism underlying alkaline dissolution-driven geopolymerization. The synthesis of geopolymers actually happens in conditions of low solvation, in contrast to the widely accepted belief that it occurs in a strongly alkaline environment. The majority of early geopolymer research was concerned with polymers made from metakaolin. Similar solidification techniques can be applied to components derived from GGBS, FA, heat-treated feldspar rocks, and different aluminosilicate compositions, such as natural rocks and technological wastes, according to the results of further research.

2.5.2 Chemistry of Geopolymer Concrete

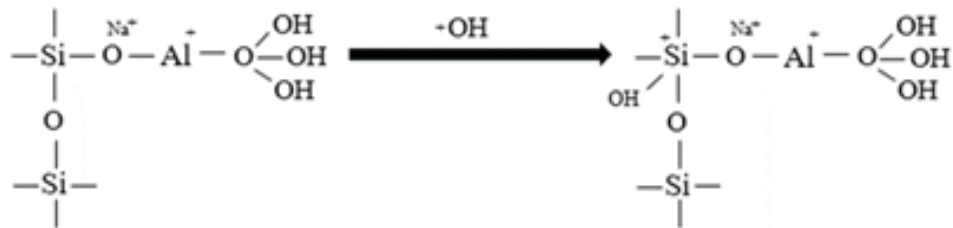
A thorough understanding of the mechanism controlling the structural evolution of geopolymer materials has been made possible by research on the hardening

process in these materials. This procedure, which is supported by interactions between a number of chemical reactions with NaOH or KOH, can be divided into discrete phases.

Phase 1: of the geopolymerization process, involves the formation of tetravalent aluminium (Al) as $-\text{Si}-\text{O}-\text{Al}(\text{OH})\text{Na}$ in the silicate side group in response to an alkaline activator.



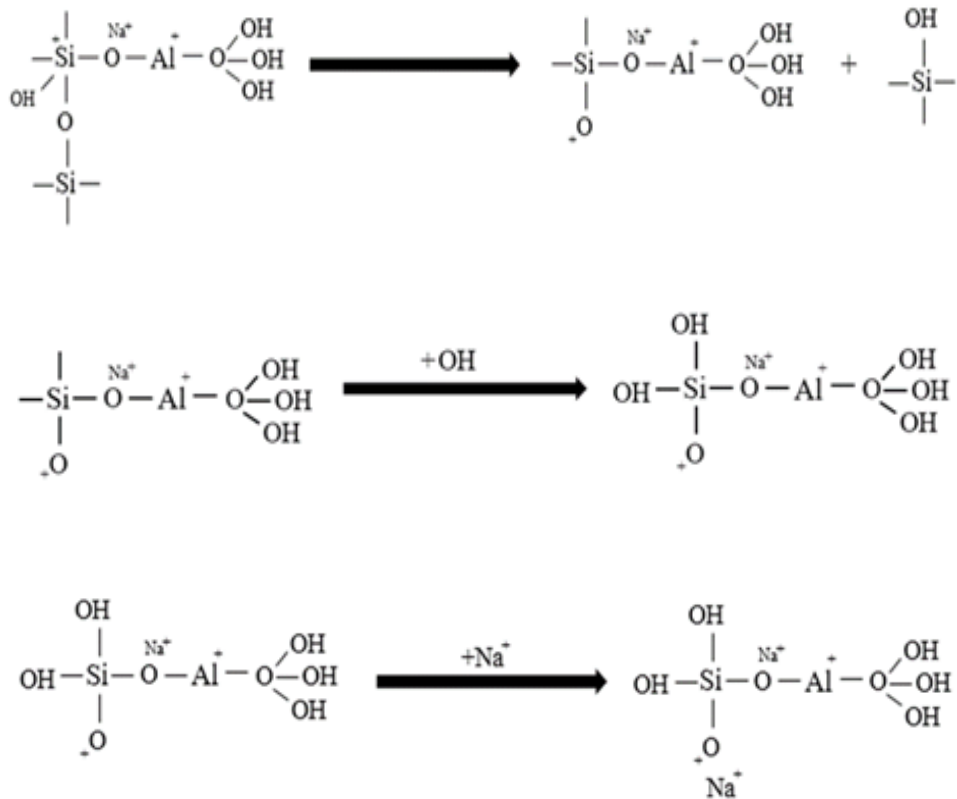
Phase 2: As hydroxyl (OH) groups are added to silicon (Si) atoms, alkali dissolution begins. The penta-covalent state of the valence electrons is the result of this procedure.



Phase 3: Third phase is the electron transport from silicon to oxygen. The creation of the foundational siloxo groups $\text{Si}-\text{O}-$ and intermediate silane groups $\text{Si}-\text{JH}$ is the outcome of the oxygen in siloxane $-\text{Si}-\text{O}-\text{Si}$ disconnecting during this process.

Phase 4: During this phase $\text{Si}-\text{OH}$ silane groups continue to form and mature into orthosilicates, which are the main constituents of geopolymers.

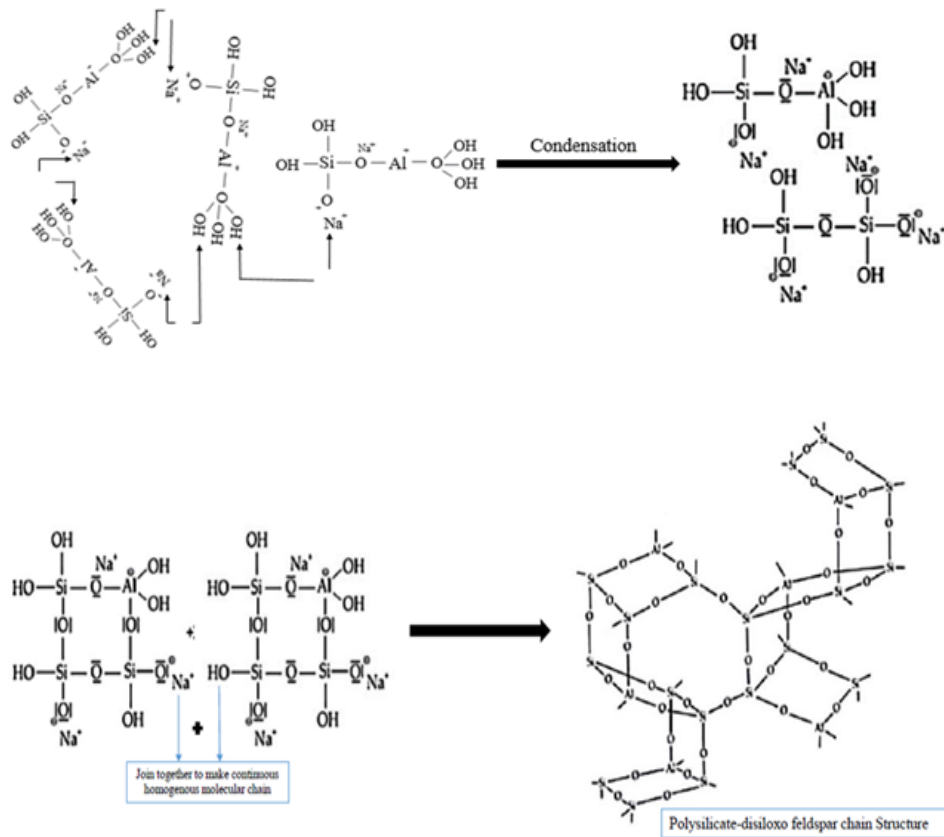
Phase 5: This phase involves the production of simple (terminal) $\text{Si}-\text{O}-\text{Na}$ bonds as a result of interactions between the initial $\text{Si}-\text{O}$ -molecules and sodium cations (Na).



Phase 6: In Phase 6, when NaOH serves as the activator, condensation takes place between molecules, reactive Si-O-Na groups, and orthosilicate OH-Al-hydroxoaluminate groups. NaOH is produced as a result, which promotes the creation of cyclotrisilicate structures. Afterwards, alkali NaOH is released and participates in polycondensation processes, which results in the formation of sodium polysilicate nepheline structures. When Si-OH and Si-O-Na groups are reacting with orthosilicate and disilicate molecules, a cyclic structure of orthosilicate-disiloxane is created. This occurs when liquid glass, which is a soluble form of sodium polysilicate, is used as the activator in this process. After that, alkali NaOH is freed from the reaction and re-enters the cycle.

Phase 7: Phase 7 sees the creation of unique feldspar chain topologies as a result of the polycondensation of the sodium polysilicate-disiloxane albite structure [34, 35].

Understanding the molecular mechanisms involved in the formation of structure forms the basis for investigating geopolymer materials. This entails a thorough analysis of their traits, the processes underlying structural growth, different kinds



of alkaline binders, and formulation combinations. Developing new composites, creating products and buildings out of geopolymer materials, and designing geopolymer materials all require a thorough understanding of the basic ideas and procedures of geopolymerization. A significant influence on the field of geopolymeric concretes is the Si to Al ratio, including their composition and properties [34, 35].

2.6 Summary

Because of its dense and durable aluminosilicate spatial microstructure, geopolymer concrete outperforms conventional Portland cement concrete in terms of adaptability, sustainability, and environmental friendliness. It is positioned as a good substitute due to its improved strength and microstructural characteristics. The ratios of the mixture's component parts and composition have a major impact on the properties and durability of geopolymer concretes. A more compact and dense microstructure is formed when some Ordinary Portland Cement (OPC) is

substituted with an aluminosilicate binder. This leads to increased strength, durability, resistance to shrinkage, decreased porosity, and decreased water absorption. Because geopolymer concrete has the potential to significantly cut greenhouse gas emissions in contrast to Portland cement (OPC) regular, its use is environmentally benign.

Moreover, the activation of silicate groups by activators starts the polymerization process that results in the creation of geopolymer concrete. The silicate compounds are then exposed to hydroxyl groups. The next step involves the transfer of electrons from oxygen-containing silicates to orthosilicates, which act as the main building blocks for the geopolymerization process. Si-O-Na connections are later formed by these orthosilicates. Orthosilicate-disiloxic cyclic structures are formed by the condensation of orthosilicate molecules containing Si-O-Na and OH-Al- groups. These cyclic structures go through polycondensation in the presence of sodium (the activator), which forms solid structures and the geopolymer in its solid state.

Chapter 3

Experimental Program

3.1 Background

Slag-infused geopolymer concrete is a significant advancement in environmentally friendly building materials and a worthy replacement for Portland cement-based traditional concrete. This innovative material is made from recycling industrial by-products, specifically slag, which is a leftover generated during the fabrication of metals, particularly during the creation of iron and steel. GGBS integration into geopolymer concrete addresses issues with the environment related to the disposal of industrial waste and is essential to reducing carbon emissions, a major concern in the building industry.

The aluminosilicate source found in slag is the key ingredient in slag-based geopolymer concrete. This component interacts with an alkaline activator to form an inorganic polymer matrix that is three-dimensional. By using industrial by-products that would otherwise wind up in landfills, geopolymer concrete reduces its carbon footprint significantly in comparison to regular concrete, which relies on Portland cement and produces a lot of carbon dioxide during the manufacturing process. This feature makes slag-based geopolymer concrete an eco-friendly and sustainable substitute that fits well with global efforts to reduce the environmental impact of construction techniques.

The final material has excellent mechanical properties, including remarkable compressive strength and longevity, making it suitable for a variety of building uses.

In addition, slag-based geopolymer concrete has proven to be more resilient than traditional concrete in a variety of harsh chemical conditions, offering greater longevity. This increased resilience is particularly helpful for infrastructure projects where long-term resilience and resistance to deterioration are important factors.

Slag-based geopolymer concrete is becoming a more attractive alternative to conventional concrete, providing the building industry with an environmentally friendly solution. This innovative approach addresses environmental concerns and promotes the construction of strong, eco-friendly structures by transforming industrial waste into an excellent building material. The growing popularity of slag-based geopolymer concrete has the potential to revolutionize construction methods and promote a more environmentally friendly and sustainable way of constructing, especially as more research and developments in this field take place.

3.2 GGBS Slag

Ground Granulated Blast Furnace Slag is the main ingredient used as a guide while preparing concrete (GGBS). This iron-making industrial byproduct is highly valued for its adaptable and sustainable use in the manufacturing of concrete. Silicate and aluminate compounds, which result from quickly cooling molten blast furnace slag with water during the manufacturing process, dominate its composition. Important components include phases that are rich in aluminium and calcium as well as tricalcium silicate (3CaOSiO_2) and dicalcium silicate (2CaOSiO_2). Additionally, glassy granules are frequently added to GGBS, which enhances its pozzolanic properties. When added to concrete as a supplement, GGBS reacts with the calcium hydroxide that is produced during the cement hydration process. As a result of this interaction, more calcium silicate hydrates are produced, which increases the concrete's overall strength, durability, and chemical resistance. By reducing environmental impact and improving performance, GGBS inclusion into concrete mixtures embodies a more sustainable approach to infrastructure development in construction practices. Ground Granulated Blast Furnace Slag (GGBS) with a fineness of 365 m/Kg was purchased for this research project from a nearby store.

Among other ingredients, the GGBS contains 7.5% magnesium oxide, 0.10% manganese oxide, and 0.22% sulfate.



FIGURE 3.1: Ground Granulated Blast Furnace Slag.

3.3 Limestone Powder

Limestone powder is an essential additional element in the field of slag-based geopolymers, adding to the complex chemistry of the geopolymerization process. Limestone powder, which is mostly composed of calcium carbonate (CaCO_3), is an essential activator that works in concert with slag to enhance the geopolymeric matrix. CaO , or CaCO_3 when broken down to content, makes up the majority of the mixture (84.85%wt). Aluminosilicates, which occur as feldspars, clay minerals, and quartz, are the carriers of SiO_2 (7.06 percent weight) and Al_2O_3 (2.38 percent weight). This finely powdered powder is used as a filler and has a substantial effect on the mix's reactivity, which helps to generate geopolymer gels. Because of the additional calcium ions brought into the system by its chemical makeup, the material's mechanical strength is increased through geopolymerization reactions. Incorporating limestone powder into slag-based geopolymers not only improves the material's overall durability but also helps to lower the likelihood of alkali-silica

reactions. Moreover, limestone powder is essential for improving the geopolymer's microstructure, controlling the pore network, and enhancing the material's durability and functionality. Limestone powder's dual role as a catalyst and modifier highlights how important it is to maximising the performance and characteristics of slag-based geopolymers. It is an important step forward in the development of long-lasting and ecological building materials.



FIGURE 3.2: Limestone Powder.

3.4 Activators

3.4.1 Sodium Hydroxide/Sodium Silicate Ratio (Primary Activators)

Sodium hydroxide, sometimes known as caustic soda, is a strong alkaline substance made up of hydrogen (H), oxygen (O), and sodium (Na). Its chemical formula is NaOH. This extremely reactive material is used in many different industrial processes. When it comes to the manufacture of geopolymer concrete, sodium hydroxide plays a vital function as an alkaline activator. Sodium hydroxide combines with aluminosilicate minerals, such as fly ash or slag, to start a chemical reaction

that forms a geopolymer binder. The conventional Portland cement activator ratio is no longer necessary with this method. The molarity (M) of sodium hydroxide is set at 14M, and the ratio of sodium hydroxide to sodium silicate is kept at 2.5 in the particular research environment.

Sodium silicate, also called water glass, is a chemical compound with the formula Na_2SiO_3 , which is made up of sodium, silicon, and oxygen. It functions as an additional crucial component in the creation of the three-dimensional inorganic polymer matrix seen in geopolymer concrete compositions. By interacting with the aluminosilicate source, the sodium silicate solution starts the geopolymerization process and gives the concrete mix cohesiveness. Through the geopolymerization process, sodium hydroxide and sodium silicate both play crucial roles in creating a long-lasting and sustainable substitute for conventional concrete.

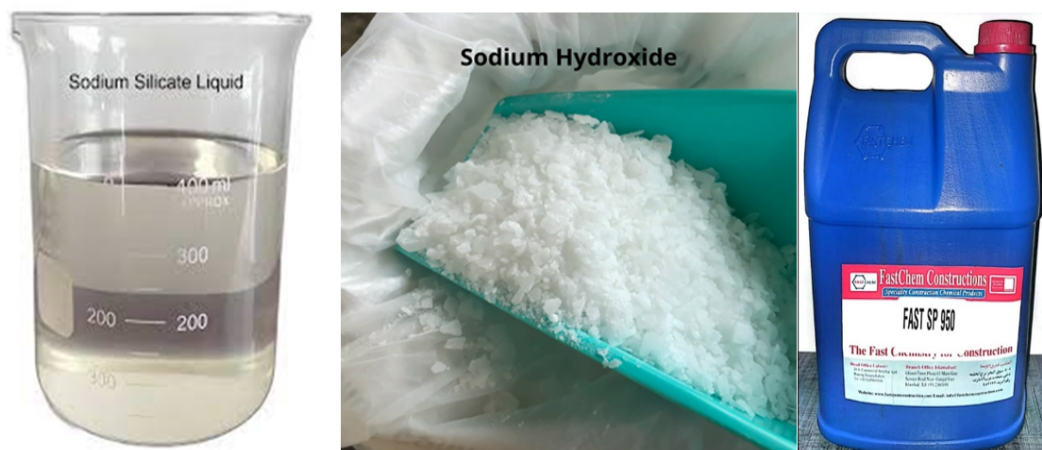


FIGURE 3.3: Sodium Silicate, Sodium hydroxide & FastChem SP-950.

3.4.2 FastChem SP-950 (Secondary Activator)

A specialised chemical solution called Fast Chem 950 is intended to operate as an accelerator in the field of activating geopolymer concrete. Fast Chem 950, which is a combination of sodium hydroxide and sodium silicate, is an alkaline activator that works well for aluminosilicate materials including fly ash and slag. Because of its special formulation, concrete sets quickly and develops strength early on by accelerating the geopolymerization process. Fast Chem 950 improves the productivity of geopolymer concrete production by speeding up the chemical interactions

between the activator and the aluminosilicate source, offering a time-saving option for building projects. This accelerator helps make geopolymer technology more sustainable while also making it easier to use geopolymer concretes in situations where quick curing and early strength increase are required.

3.5 Mix Design, Casting Procedure, Specimens

In order to prepare the geopolymer, a precise mix design must be used, with different amounts of slag starting at 550 grammes and progressively decreasing from zero to twenty percent in 5% increments. The first sample is a geopolymer matrix made entirely of GGBS slag; limestone powder is not included as shown in Table 3.1 (Geopolymer design mix with limestone percentage). With a constant sodium hydroxide molarity of 14M, the ratio of sodium hydroxide to sodium silicate is consistently maintained at 2.5. Because this is an exothermic process that generates a lot of heat, a 14 molar solution of sodium hydroxide is made and left for 24 hours. This solution is cooled to room temperature (24 degrees) after 24 hours so that it can be used in geopolymer concrete. A water/powder ratio of 0.23 is obtained by adding more water, fixed at a unit quantity of 128 mg. The high-range water reducer Fastchem SP-950 is regularly added to the mixture at a weight percentage of 3% of the unit mix powder in order to improve it.









Important computations also determine a constant Binder/Fine aggregate unit of 1.5. In particular, the study focuses on assessing how limestone powder affects the slag geopolymer cement matrix. The investigation yields significant findings that demonstrate the efficacy of the designed mix design. Careful slag proportion modifications combined with a thorough examination into the effects of limestone powder on the geopolymer matrix yield important insights for optimizing the geopolymerization process. These results make a substantial contribution to the understanding and investigation of possible uses of slag-based geopolymer systems in the material science and construction domains. Thirty cubes and thirty prisms were carefully poured, and each set was carefully inspected using average test results from three specimens at each percentage point in the mix proportion spectrum.

TABLE 3.1: Geopolymer design mix with limestone percentage

Ingredients, Kg/m ³										
ID	Slag (Ground Granulated Blast Slag)	(Ground -Furance)	Limestone	NAOH (M)	(14 Sodium Silicate)	HRWR	Fine Aggregate	Ag-	Extra wa-	ter
Geopoylmer (GPC)	550		0	60	150	16.5	825		128	
GPC+5% LSP	522		28	60	150	16.5	825		128	
GPC+10% LSP	495		55	60	150	16.5	825		128	
GPC+15% LSP	467		83	60	150	16.5	825		128	
GPC+20% LSP	440		110	60	150	16.5	825		128	
	Decreasing 5%	by	Increasing by 5%	Alkali activator ratio 2.5		3% dosage	Binder/Fine Aggre- gates ratio 1.5	Fine extra wa- ter/pow- der ratio 0.23		

This methodical methodology ensures a thorough evaluation of the mix’s performance in a variety of compositions. In addition, ten cylindrical specimens with dimensions of 100 x 200 mm were made especially for testing water porosity and sorptivity, providing important information about the permeability and porosity properties of the material.

TABLE 3.2: Experimental Plan

Sr. No	Test	ASTM Code	Dimensions	Sample Cast	Shape
1	Compression Test	ASTM C109/C109-20	50mmx50mmx50mm	3 For each mix Total Mix = 5 Total Cylinders = 30 (15@14 days +15@28 days)	 Cube
2	Flexural Test	ASTM C348-14	40mmx40mmx160mm	3 For each mix Total Mix = 5 Total Cylinders = 30 (15@14 days +15@28 days)	 Prisms
3	Flow ability Test	ASTM C1437	Fresh matrix	No Casting	 Flow Cone
4	Scanning Electron Microscopy (SEM)	ASTM C1723-16	Broken Sample	No Casting	 Piece from Broken sample
5	X-Rays Diffraction (XRD)	ASTM E3294-22	Broken sample	No Casting	 Piece from Broken sample
6	Energy Dispersive X-rays (EDX)	ASTM C1723-16	Broken sample	No Casting	 Piece from Broken sample
7	Sorptivity	ASTM C1585-13	100mm x 50mm	1 Cylinder for each mix (3 disks from same cylinder)= 5 cylinders	 Disk Samples
8	Porosity	ASTM C1754/C1754 M-12	100mm x 50mm	1 Cylinder for each mix (3 disk from same cylinder)=5 cylinders	 Disk Samples

After a critical 28-day curing period-a pivotal moment in the material’s development the cylindrical specimens were subjected to further processing for a comprehensive examination. Each of the fifteen cylinders was precisely sliced into

50 x 100 mm discs by skilled artisans. These discs allowed sorptivity and water porosity tests to continue while maintaining the original material's integrity. By converting the cylindrical specimens into discs, researchers were able to examine the material's behavior in greater depth and gain a more nuanced understanding of its microstructural performance. The extensive testing plan, which includes cubes, prisms, and cylindrical specimens with more refining, highlights how careful the research is. The goal of the research is to provide a comprehensive and in-depth evaluation of the material's properties by employing a wide variety of testing techniques and sample preparations. The objective of this technique is to make a significant contribution to the wider comprehension of the material's applicability for various applications.



FIGURE 3.4: Casting Process of Geopolymer matrix.

To guarantee the creation of a long-lasting and high-performing material, a methodical set of procedures is followed during the casting process for a geopolymer matrix based on slag. First, the exact ratios of the mixture are established, with slag acting as the main binder. Slag content is adjusted carefully to determine the ideal composition, usually working in percentages between 0% and 20%. Both the sodium hydroxide molarity of 14M and the constant sodium hydroxide to sodium

silicate ratio of 2.5 are maintained. More water is added to the mixture, resulting in a water/powder ratio of 0.23. Moreover, a consistent 3% weight-based addition of a high-range water reducer, like Fastchem SP-950, is made to the unit mix powder.

Then the carefully combined material is placed into moulds shaped like cubes or prisms for easier testing later on. Thirty cubes and thirty prisms are usually cast for each mix proportion, and three specimens are used to derive the average test findings. Furthermore, fifteen 100x200 mm cylindrical specimens have been specially produced for measuring water porosity and sorptivity. After a critical 28-day curing period, these cylindrical specimens are precisely sliced into 50 mm by 100 mm discs, allowing for a more thorough analysis of the material's properties related to water porosity and sorptivity. The production of slag-based geopolymer matrices with a variety of compositions is ensured by this methodical casting process, which makes thorough research and optimisation for construction applications possible.

3.6 Experimental Plan

The goal of the experiment is to produce geopolymer specimens by combining different activators with different aluminosilicate sources, including slag. Both compressive and flexural strength tests will be used to thoroughly investigate the mechanical properties. Scanning electron microscopy (SEM), X-ray diffraction (XRD), and energy-dispersive X-ray spectroscopy (EDX) will be used in the microstructural study process to examine the fine details of the geopolymer matrix. Additionally, porosity and sorptivity tests will be used to carefully evaluate the durability properties. The goal of this thorough method is to produce a sophisticated understanding of the mechanical strength, microstructural makeup, and environmental resilience of geopolymer concrete. Through the assessment of the material's porosity and sorptivity, the research will provide significant understanding of its durability and improve our understanding of its overall performance attributes.

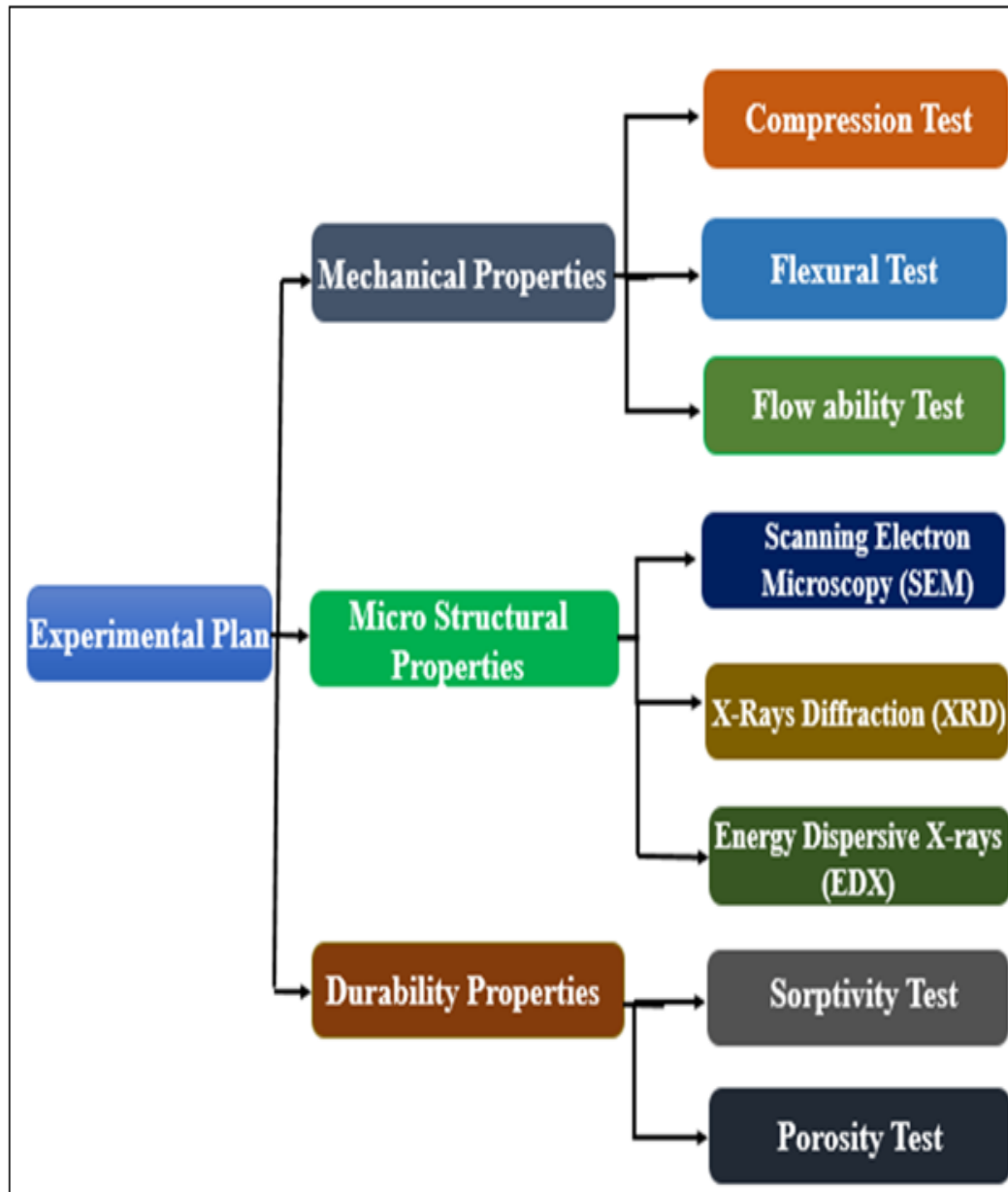


FIGURE 3.5: Experimental Plan.

3.6.1 Mechanical Properties Tests

The mechanical properties of the geopolymers were comprehensively evaluated by the use of ASTM standard tests in the inquiry. While bending qualities were measured using flexural tests (ASTM C348-14), the axial strength was assessed using compression tests (ASTM C109/C109-20). Conversely, flowability tests were used to gauge workability (ASTM C1437). These standardised testing procedures guaranteed a thorough analysis of the geopolymer's performance in accordance with accepted industry standards. These ASTM-compliant test findings supported the

material's potential in the engineering and construction sectors by providing insightful information about the material's appropriateness for a range of applications.

3.6.2 Microstructural Properties

The geopolymer's microstructural analysis complied with ASTM guidelines. Scanning electron microscopy (SEM) was utilised to acquire high-resolution pictures that illustrate surface morphology in compliance with ASTM C1723-16. Using X-ray Diffraction (XRD) (ASTM E3294-22), the crystalline phases in the geopolymer structure were revealed. Energy-Dispersive X-ray Spectroscopy (EDX) in accordance with ASTM C1723-16 enabled elemental mapping enabling insights into material composition. By ensuring a methodical and uniform approach to microstructural examination, the application of these ASTM standards improved the dependability and comparability of outcomes. Through a strict and proven testing process, this approach offered a thorough understanding of the properties of the geopolymer.

3.6.3 Durability Properties Tests

The ASTM guidelines were followed in the evaluation of the geopolymer's durability characteristics. Important information on the material's durability features was provided by moisture absorption, which was evaluated by measuring sorptivity using ASTM C1585-13, and quantification of void spaces using porosity measurements in accordance with ASTM C1754/C1754 M-12. The ability of the geopolymer to endure environmental conditions was fully understood thanks in large part to these standardized tests.

3.7 Summary

The experimental design for the production of geopolymer specimens was carried out using a range of activators and aluminosilicate sources. Testing methods to be



FIGURE 3.6: Geopolymer matrix Disks.

used to assess mechanical, microstructural, and durability qualities include compressive and flexural strength, SEM, XRD, EDX, porosity, and sorptivity. With this thorough approach, we hope to learn more about the microstructure, strength, and resistance to the environment of geopolymer concrete, which will help us use it in a variety of construction settings. The mix design includes different amounts of slag, a 2.5 ratio of sodium hydroxide to sodium silicate, 14M sodium hydroxide, 128 mg of water, and 3% of Fastchem SP-950 as a water reducer. Specifically, the impact of limestone powder and the optimisation of the geopolymerization process are the priorities.

Chapter 4

Results and Analysis

4.1 Background

All mechanical, microstructural and durability testing is carried out in accordance with ASTM standards. In the pursuit of creating Geopolymer in replacement of conventional concrete mix with enhanced durability properties, understanding the role of mix proportions is paramount. These proportions dictate the quantity of each component - slag, fine aggregates, water, and any supplementary chemicals - in a geopolymer mix. The performance characteristics of geopolymer, particularly strength and durability, are deeply influenced by these proportions. In this study, two distinct geopolymer mixes were investigated, each with different mix proportions with limestone powder.

4.2 Mechanical Properties

4.2.1 Flow Ability Properties

The flow ability test, conducted in accordance with ASTM standards, produced exceptionally positive outcomes. The results indicate a high level of performance and suitability, showcasing the excellent flow characteristics of the material being tested. This adherence to ASTM standards ensures a standardized and reliable assessment of flow ability, offering valuable insights into the material's behavior

under specific conditions. The excellent results obtained underscore the material's favorable attributes, affirming its capability to flow smoothly and consistently as per established industry standards, enhancing its applicability and performance in various contexts.

TABLE 4.1: Flow ability Results

ID	Flow Test for Geopolymer mortar (ASTM C1437)					Flow (%)
	Slag (GGBS)	Limestone powder (LS)	Dia of flow mould (mm) Do	Average dia of flow (mm) Davg		
LS0	550	0	100	196		96
LS5	522	28	100	193		93
LS10	495	55	100	188		90.4
LS15	467	83	100	184		88
LS20	440	110	100	181		86.5

Pure Geopolymer with 14M of sodium hydroxide and with sodium silicate activator ratio 2.5 produces flow of 96% which is 11.12% less than the conventional cement mix. Percentage of limestone increases with 5% increment up to 20% gives 93% 90.4% 88% and 86.5% flow respectively as shown in Table 4.1. According to Qadir et. al.[43] presented same study with same limestone increment percentage up to 20%. As compared to cement mixed study with 20% limestone powder Geopolymer limestone mix matrix produces 13.94% less flow due to GGBS density mix as shown in Figure 4.1.

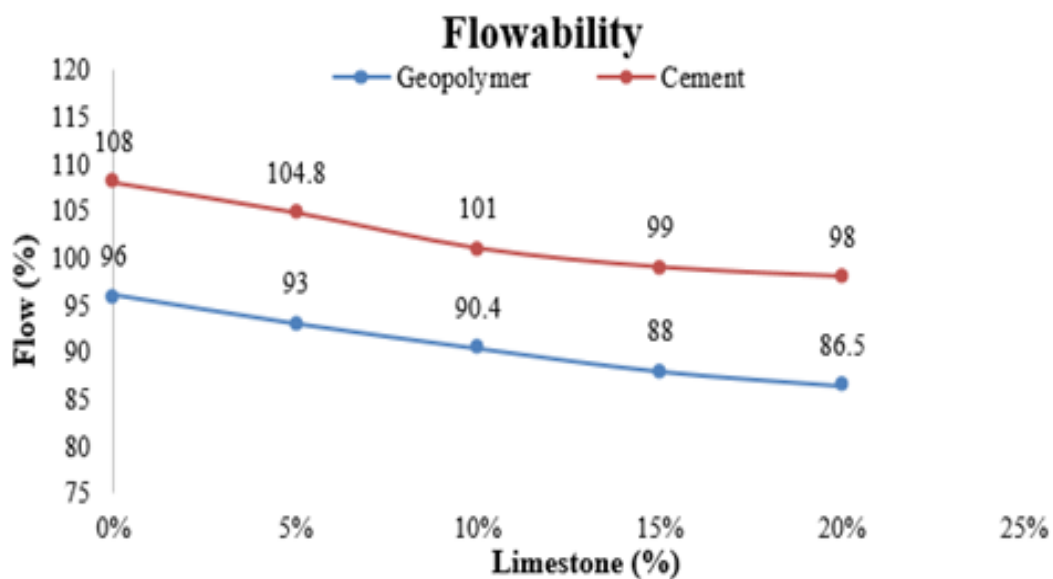


FIGURE 4.1: Flow ability of geopolymer with addition of limestones powder

4.2.2 Compressive Strength

In the process of studying the effect of different geopolymer mix proportions on its compressive strength, three unique geopolymer mixes were tested under compressive loading. The first mix, a pure GGBS slag based Geopolymer mix matrix, achieved a compressive strength of 23.58Mpa in 14 days of curing and after 28 days of curing it gives 28.17 MPa as shown in Table 4.3. This strength is within the typical range for standard concrete mixes and is indicative of its performance under compressive forces. The second mix varied from the conventional composition by incorporating smaller Limestone powder by 5% by weight of Slag. This modification resulted in a considerably enhanced compressive strength of 29.04 MPa under loading which is 3.09% more than GGBS Geopolymer concrete.

This increase in strength signifies the effective role of limestone powder and activators in improving the properties of Geopolymer matrix under compressive loads, while the use of smaller limestone powder also contributed to the enhancement in compressive strength. The third mix, which was made by adding 10% of limestone powder in replacement of GGBS slag, exhibited the high compressive strength of 30.39 MPa which is 7.88% more than GGBS slag Geopolymer. This increase in compressive strength can be attributed to the combined effects of the slag and limestone powder along with activators, which potentially led to a denser and more uniform Geopolymer matrix, hence providing higher resistance to compressive forces.

The fourth and fifth mix, which was made by adding 15% and 20% of limestone powder in replacement of GGBS slag, provides the highest compressive strength of 32.16 MPa & 34.44 MPa which is 14.16% and 22.26% high from GGBS slag based Geopolymer respectively. Figure 4.2 clearly shows the Results, confirm that the mix proportions significantly influence the compressive strength of the Geopolymer mix matrices. Compression results in comparison with Rashad et al. (2023), GGBS slag gives relatively low compressive strength when mixed with limestone powder as compared to GGBS used with feldspar [45].

TABLE 4.2: Compressive Strength results @ 14 days

ASTM C109/C109- 20 @14 days	Samples (Qty)	Compressive Strength Fc' @14 days			Avg of 3 Spec- imens (Mpa)	Avg of 3 Speci- men (Psi)
		Ultimate Load (KN)	Compressive Strength (Mpa)	Compressive Strength (Psi)		
Geopolymer Concrete (GPC)	1st	66.67	25.83	3746.94	23.58	3420.79
	2nd	56.47	21.88	3173.69		
	3rd	59.46	23.04	3341.73		
GPC+5% LSP	1st	66.25	25.67	3723.34	24.15	3503.03
	2nd	61.4	23.79	3450.76		
	3rd	59.34	22.99	3334.99		
GPC+10% LSP	1st	56.24	21.79	3160.76	25.73	3732.33
	2nd	79.04	30.63	4442.15		
	3rd	63.95	24.78	3594.07		
GPC+15% LSP	1st	74.31	28.79	4176.32	27.89	4045.93
	2nd	77.17	29.9	4337.06		
	3rd	64.49	24.99	3624.42		
GPC+20% LSP	1st	67.26	26.06	3780.1	28.12	4079.46
	2nd	71.05	27.53	3993.1		
	3rd	79.45	30.79	4465.19		

TABLE 4.3: Compressive Strength results @ 28 days

ASTM C109/C109- 20 @28 days	Compressive Strength F_c' @28 days					
	Samples (Qty)	Ultimate Load (KN)	Compressive Strength (Mpa)	Compressive Strength (Psi)	Avg of 3 Spec- imens (Mpa)	Avg of 3 Speci- men (Psi)
Geopolymer Concrete (GPC)	1st	71.45	27.69	4015.58	28.17	4085.72
	2nd	76.71	29.72	4311.2		
	3rd	69.95	27.11	3931.28		
GPC+5% LSP	1st	68.32	26.47	3839.67	29.04	4211.9
	2nd	77.4	29.99	4349.98		
	3rd	79.14	30.67	4447.77		
GPC+10% LSP	1st	85.29	33.05	4793.41	30.39	4407.7
	2nd	79.04	30.63	4442.15		
	3rd	70.95	27.49	3987.48		
GPC+15% LSP	1st	86.31	33.44	4850.74	32.16	4664.42
	2nd	73.17	28.35	4112.25		
	3rd	89.49	34.68	5029.46		
GPC+20% LSP	1st	93.26	36.14	5241.33	34.44	4995.11
	2nd	89.05	34.51	5004.73		
	3rd	84.34	32.68	4740.02		

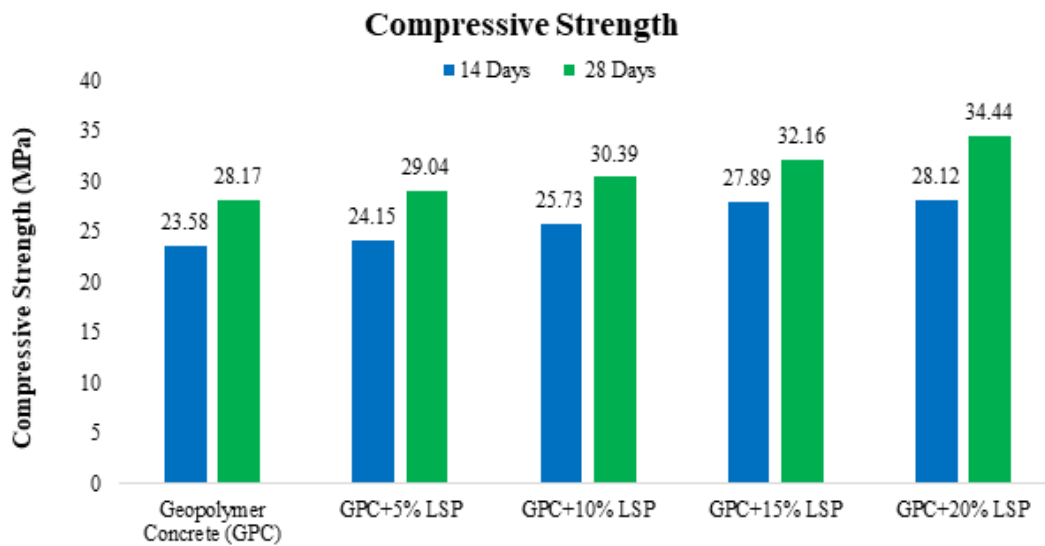


FIGURE 4.2: Compressive Strength cumulative Results

4.2.3 Properties under Flexural Strength

This section delves into the impact of various geopolymer mix proportions on the flexural strength. Flexural strength, often considered a measure of tensile strength in bending, is an essential property in many structural applications of concrete. Table 4.5 first mix was a pure slag based concrete mix. This mix demonstrated a flexural strength of 4.71 MPa under loading, which aligns with the typical range for standard concrete mixes. The second mix was modified by including smaller amount of limestone powder (5%). This mix proportion resulted in a superior flexural strength of 4.75 MPa under loading. This improvement can be attributed to the synergistic effects of limestone powder, makes denseness of the concrete mix. Additionally, the use of limestone may have improved the packing density of the mix, contributing to the increase in flexural strength. The third mix, which employed a higher quantity of limestone, exhibited the high flexural strength of 6.27 MPa. The significant increase suggests that the higher proportion of limestone powder may have led to a denser and more homogenous matrix, thus providing improved resistance to bending forces.

The fourth and fifth mix, which was made by adding 15% and 20% of limestone powder in replacement of GGBS slag, provides the highest flexural strength of



FIGURE 4.3: Flexural Strength Testing

8.42 MPa & 9.71 MPa respectively among the tested mixes. Comparison between standard 14days curing strength and 28days curing strength is given in figure 4.4. The creation of a tobermorite needle-like structure, which provides more resistance in flexure than assistance in compression, is responsible for these incredible results in flexure specially [46].

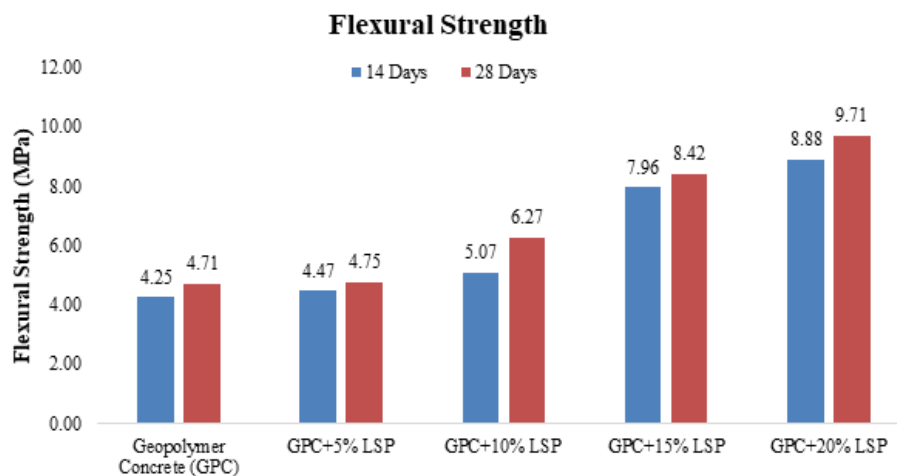


FIGURE 4.4: Flexural Strength cumulative Results

The outcomes of the study conclusively validate the pivotal role played by mix proportions in determining the flexural strength of Geopolymer mix matrices. The outcomes highlight how much different mix proportions can affect these matrices' mechanical characteristics, especially when it comes to flexural strength. This confirmation highlights the importance of precision and careful consideration in formulating Geopolymer mixes, as the resultant matrix's ability to resist bending forces is directly tied to the specific ratios of its constituent materials. These findings contribute valuable insights to the field, emphasizing the need for a nuanced approach in mix design to achieve desired flexural strength characteristics in Geopolymer-based materials. Ultimately, this knowledge aids in optimizing the

TABLE 4.4: Flexural Strength results @ 14 days

ASTM 348-14 @14 days	Flexural Strength @14days						
	Samples (Qty)	Ultimate Load (KN)	Flexural Strength (Mpa)	Flexural Strength (Psi)	Avg of 3 Specimens (Mpa)	Avg of 3 Specimens (Psi)	Flexural strength in %Fc'
Geopolymer Concrete (GPC)	1st	1.435	4.018	582.76	615.93	4.247	18.01
	2nd	1.523	4.264	618.5			
	3rd	1.592	4.458	646.52			
GPC+5% LSP	1st	1.598	4.474	648.96	647.6	4.465	18.49
	2nd	1.345	3.766	546.21			
	3rd	1.841	5.155	747.64			
GPC+10% LSP	1st	1.671	4.679	678.6	734.92	5.067	19.69
	2nd	1.811	5.071	735.46			
	3rd	1.947	5.452	790.69			
GPC+15% LSP	1st	2.952	8.266	1198.83	1154.29	7.959	28.54
	2nd	2.891	8.095	1174.05			
	3rd	2.684	7.515	1089.99			
GPC+20% LSP	1st	2.879	8.061	1169.18	1287.36	8.876	31.56
	2nd	3.218	9.01	1306.85			
	3rd	3.413	9.556	1386.04			

TABLE 4.5: Flexural Strength results @ 28 days

ASTM 348-14 @28 days	Samples (Qty)	Flexural Strength @28days			Avg of 3 Specimens (Mpa)	Avg of 3 Specimens (Psi)	Flexural strength in %Fc'
		Ultimate Load (KN)	Flexural Strength (Mpa)	Flexural Strength (Psi)			
Geopolymer Concrete (GPC)	1st	1.536	4.301	623.78	4.714	683.71	16.73
	2nd	1.922	5.382	780.54			
	3rd	1.592	4.458	646.52			
GPC+5% LSP	1st	1.591	4.455	646.12	4.746	688.35	16.34
	2nd	1.755	4.914	712.72			
	3rd	1.739	4.869	706.22			
GPC+10% LSP	1st	2.034	5.695	826.02	6.267	908.95	20.62
	2nd	2.684	7.515	1089.99			
	3rd	1.997	5.592	810.99			
GPC+15% LSP	1st	3.252	9.106	1320.66	8.423	1221.66	26.19
	2nd	2.891	8.095	1174.05			
	3rd	2.881	8.067	1169.99			
GPC+20% LSP	1st	3.68	10.304	1494.47	9.706	1407.74	28.18
	2nd	3.742	10.478	1519.65			
	3rd	2.977	8.336	1208.98			

4.3 Analysis of Microstructural Properties

4.3.1 X-Ray Diffraction

The X-ray diffraction (XRD) analysis of the materials, including Quartz (Silica), Magnesite, Syn, Wollastonite, Potassium Manganese Oxide, Sodium Oxide, Torilite, Zeolite X, Aluminum Silicon, Calcium Silicate, and Diopside Sodium Silicon, exhibited pronounced crystalline behavior which eventually plays part in strength. The XRD patterns revealed sharp peaks, indicating well-defined crystal structures within the specimens. Remarkably, the addition of limestone powder resulted in an augmentation of crystalline behavior, evidenced by the increasing intensity and sharpness of the XRD peaks. This trend was consistent with prior studies on similar systems. In comparison, the XRD analysis of specimens containing slag demonstrated diminished crystalline behavior as shown in figure 4.5. The XRD patterns for slag-containing samples exhibited broader and less defined peaks, suggesting a reduction in overall crystallinity compared to their limestone-containing counterparts. This finding aligns with existing literature highlighting the influence of slag content on crystalline characteristics. 1) Quartz (Silica) 2) Maganosite, Syn 3) Wollastonite 4) Pottasium Maganese Oxide 5) Sodium Oxide 6) Torilite 7) Zeolite X 8) Alumanium Silicon 9) Calcium silicate 10) Diopside 11) Sodium Silicon

4.3.2 Scanning Electron Microscopy (SEM) Analysis

The scanning electron microscopy (SEM) results revealed distinctive features in the microstructure of the samples. Notably, the SEM images exhibited the cracks in samples these cracks are due to mechanical test loading (due to external loading). The CSH (calcium silicate hydrate) gel and ettringite phases are observed suggesting potential of composition in the material structure. Additionally, the presence of well-defined crystalline behavior and micro GGBS (Ground Granulated Blast Furnace Slag) crystals was evident. The SEM analysis also identified the existence of pores within the material, indicating variations in density and potential implications for permeability. Interestingly, the introduction of limestone powder yielded a notable transformation in the microstructure, generating a

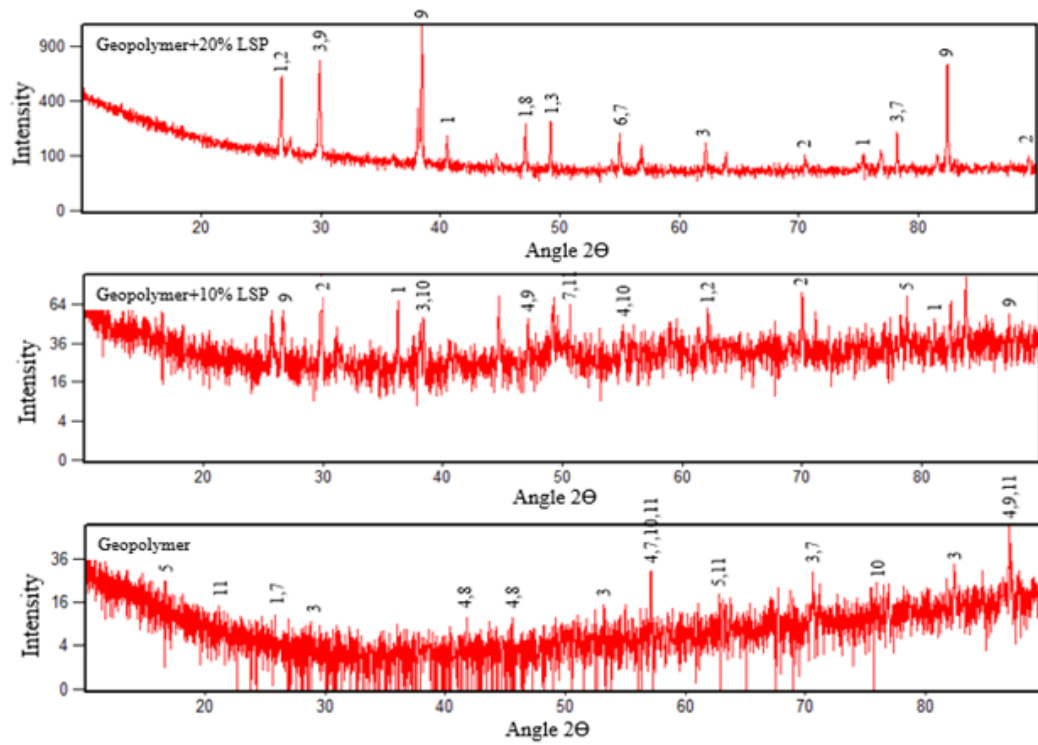


FIGURE 4.5: XRD analysis

dense cloud of tobermorite. This observation aligns with prior research indicating the propensity of limestone to influence the formation of tobermorite phases in cementitious systems. The dense cloud of tobermorite signifies a unique and desirable microstructural development, suggesting improved mechanical properties and durability.

These SEM findings described in figure 4.6 provide valuable insights into the intricate details of the material's microstructure, shedding light on phenomena such as crack formation, crystalline behavior, and the impact of limestone powder addition. Understanding these microstructural characteristics is crucial for tailoring material formulations and optimizing performance in various applications, ranging from construction to materials engineering

4.3.3 EDX SPECTRUMS

The significant presence of atoms Na 2.1%, Mg 0.5%, Al 1.3%, Si 3.7%, S 0.6% (in free drawing 1), K0.1%, Ca 10.7%, and Fe 0.9% in the EDX results indicates

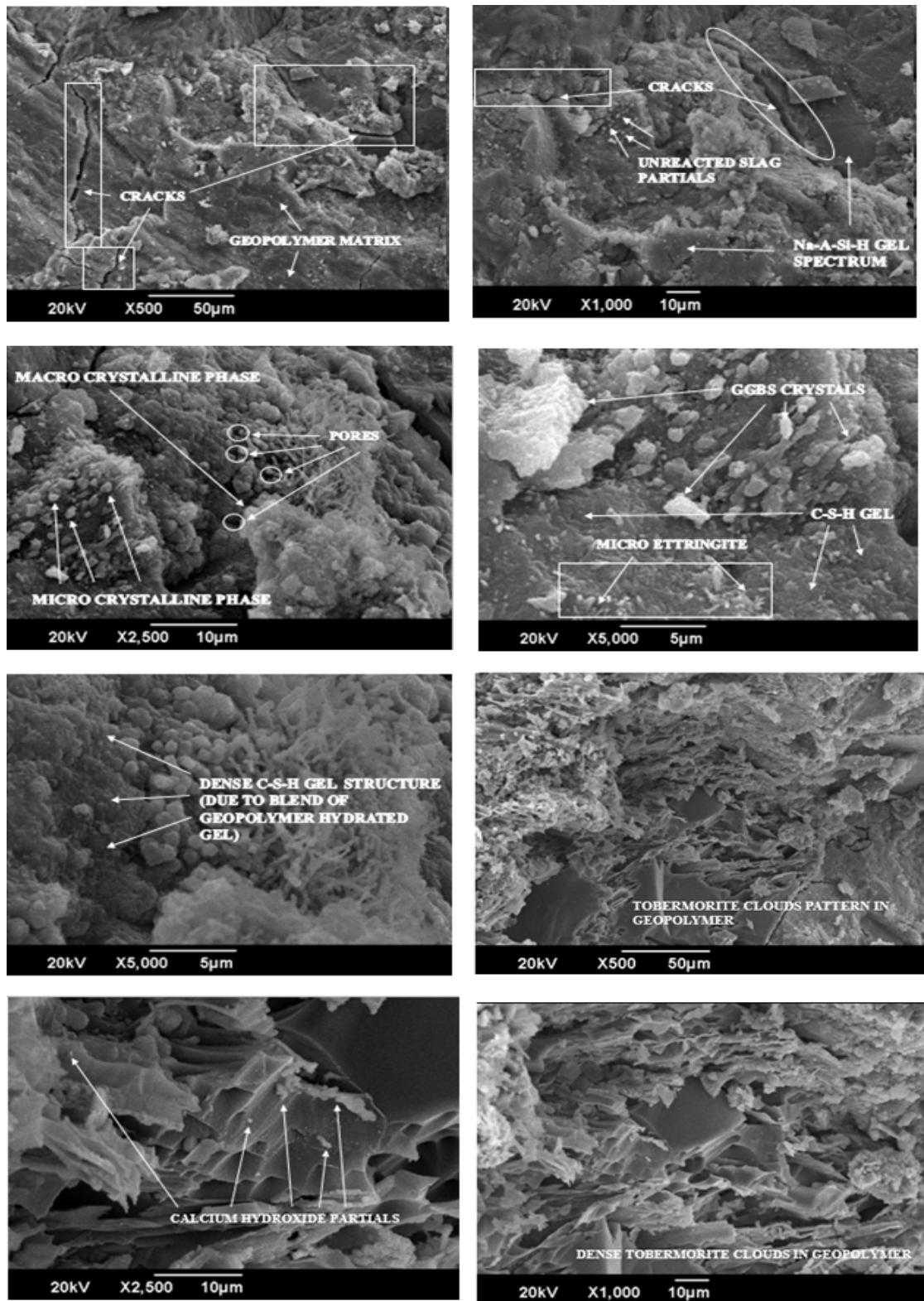


FIGURE 4.6: SEM Damaged surfaces & material description

a diverse elemental composition within the material. Sodium (Na), magnesium (Mg), aluminum (Al), silicon (Si), sulfur (S), potassium (K), calcium (Ca), and

iron (Fe) are common elements found in various minerals and construction materials. Different drawing areas as shown in figure 4.5 found different chemical atoms this is due to non-uniformity mix (ideal mix is rarely possible).

Their notable concentrations suggest a complex mixture, potentially encompassing silicate minerals, salts, and metal oxides. Table 4.6 shows material weight percentage. The EDX analysis underscores the material's diverse elemental makeup, providing essential insights into its composition, which is crucial for understanding its properties and potential applications in fields such as geology, materials science, or construction.

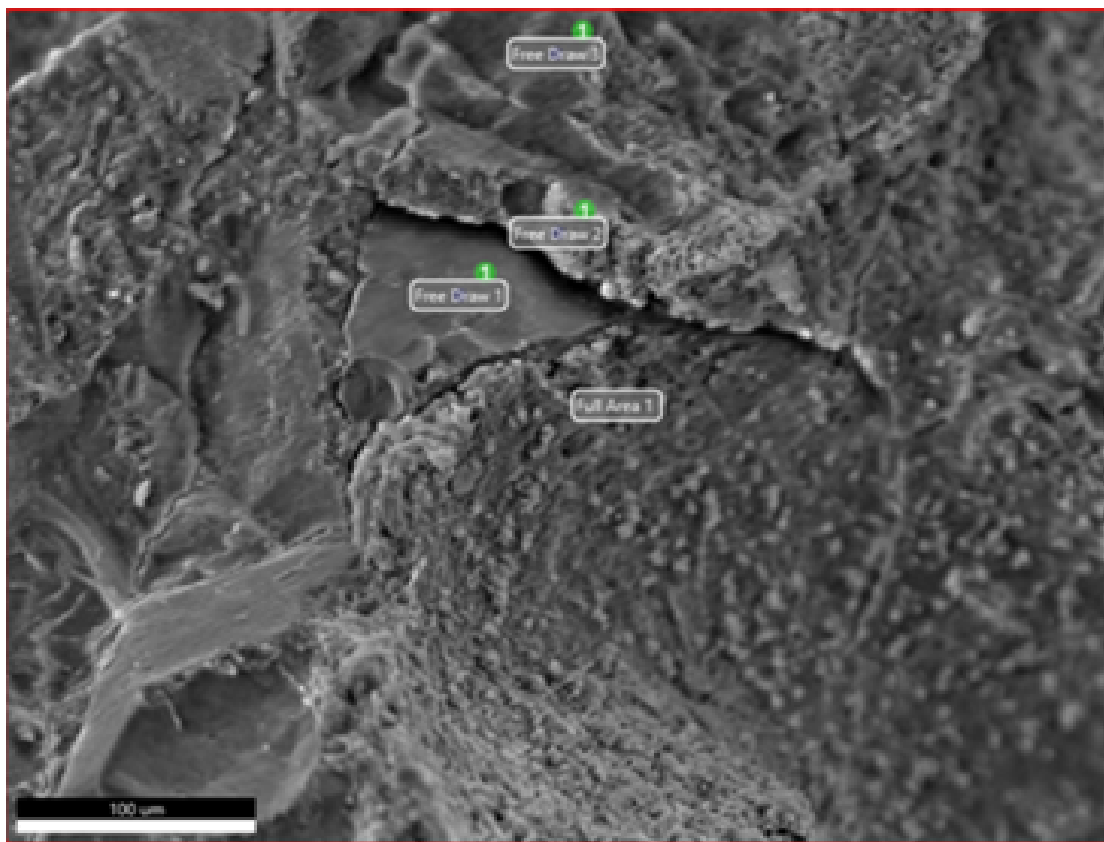


FIGURE 4.7: EDX surfaces Area evaluation (Pure GPC)

The spectrum overlay analysis in figure 4.8, geopolymer matrix indicates the intensity of materials present in the mix. Oxygen (O), calcium (Ca), silicon (Si), aluminum (Al), gold (Au), sulfur (S), and iron (Fe) are prominent, with additional elements dominating compared to the limestone-mixed counterpart. This suggests a diverse composition with an increased variety of elements, potentially influencing the geopolymer matrix's properties.

TABLE 4.6: EDX surface material Evaluation

Element	Weight %	MDL	Atomic %	Error %
S2 - Full Area 1				
C	12.7	0.55	28.1	11.6
O	29	0.16	48.1	10.4
N a	1.8	0.06	2.1	10
M g	0.5	0.04	0.5	10.6
A l	1.3	0.03	1.3	7.5
S i	3.9	0.03	3.7	5.8
K	0.2	0.05	0.1	17
C a	16.2	0.06	10.7	3.8
F e	1.9	0.08	0.9	5.1
A u	32.6	0.68	4.4	4.9
S2 - Free Draw 1				
O	19.8	0.24	56	11
N a	1.1	0.09	2.2	11.6
A l	0.6	0.04	1.1	9.3
S i	2.1	0.04	3.3	6.9
S	0.4	0.06	0.6	13.6
K	0.2	0.07	0.3	22.9
C a	19.8	0.08	22.5	4.8
F e	2	0.18	1.6	7.6
A u	53.9	1.66	12.4	6

Element	Weight %	MDL	Atomic %	Error %
S2 - Free Draw 2				
O	18.9	0.12	45.9	10.4
N a	1.4	0.07	2.3	10.4
A l	1.7	0.04	2.7	8.5
S i	8.8	0.04	12.6	6.9
S	8.5	0.04	11.8	6.3
K	0.6	0.06	0.6	12.7
C a	11.6	0.06	11.2	4.6
F e	6.5	0.1	4.5	3.7
A u	42.1	0.69	8.3	4.5
S2 - Free Draw 3				
O	7.4	0.18	41.5	12
N a	0.4	0.1	1.6	17
A l	0.5	0.05	1.5	11.3
S i	1.1	0.04	3.6	8.3
S	0.1	0.07	0.2	45.3
K	3.9	0.09	8.7	7
C a	1.7	0.21	2.8	9.9
F e	1.1	0.23	1.7	13
A u	83.8	2.17	38.3	5.4

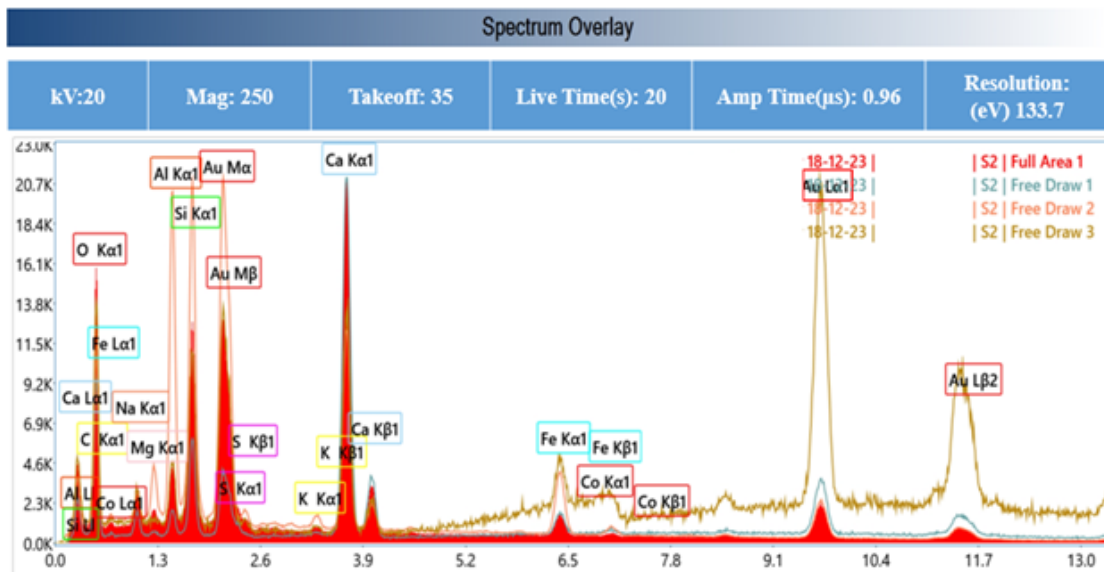


FIGURE 4.8: EDX Spectrum Overlay Graph of pure Geopolymer

The higher prevalence of certain elements, including Au, indicates specific additives or reactions, highlighting the complexity of the material's chemical makeup and emphasizing the importance of spectrum analysis in understanding and optimizing geopolymer formulations.

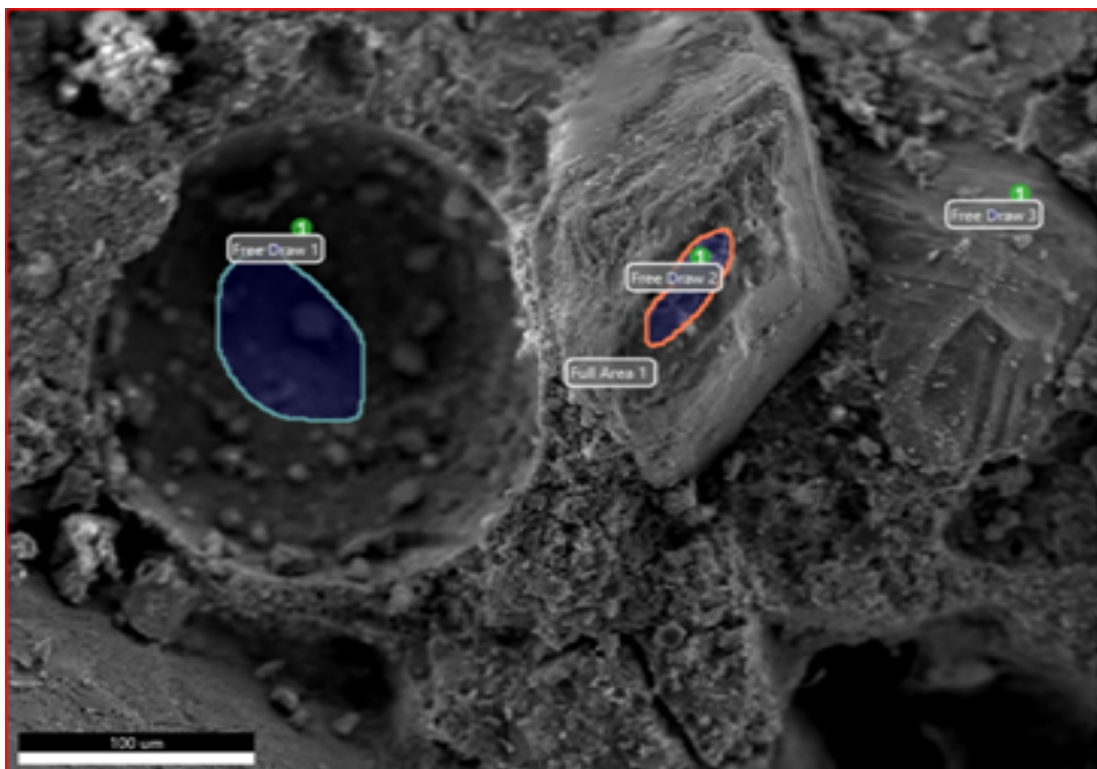


FIGURE 4.9: EDX surfaces Area evaluation (+15% LSP)

TABLE 4.7: EDX surface Evaluation (+15% LSP)

Element	Weight %	MDL	Atomic %	Error %
S2 - Full Area 1				
N a	5.3	0.15	8	9.4
M g	0.9	0.1	1.3	10.5
A l	3.5	0.08	4.5	7.1
S i	16	0.08	19.6	5.5
S	17	0.09	18.3	4.7
K	0.6	0.11	0.5	15
C a	52.3	0.18	45.1	2.9
F e	4.4	0.25	2.7	5.6
S2 - Free Draw 1				
N a	1.6	0.09	2	8.6
M g	0.5	0.06	0.6	8.5
A l	1	0.06	1.1	6.4
S i	70.6	0.06	73.4	3.5
S	21	0.13	19.1	6.6
K	0	0	0	100
C a	4.8	0.15	3.5	6.3
F e	0.5	0.21	0.3	24.8

Element	Weight %	MDL	Atomic %	Error %
S2 - Free Draw 2				
N a	8.5	0.15	12.2	8.8
M g	1.3	0.1	1.8	9.4
A l	2.7	0.08	3.4	7.1
S i	19.2	0.07	22.6	5.3
S	21.4	0.09	22.1	4.8
K	0.5	0.1	0.4	15
C a	43.3	0.13	35.7	3.1
F e	3	0.2	1.8	6.3
S2 - Free Draw 3				
N a	8	0.14	11.7	8.9
M g	1	0.09	1.4	10.2
A l	3.8	0.08	4.8	7.1
S i	16.1	0.07	19.4	5.5
S	16	0.09	16.9	4.7
K	0.8	0.1	0.7	12.4
C a	51.2	0.14	43.2	2.9
F e	3.1	0.22	1.8	6.2

The EDX results show notable atomic concentrations, with Na at 8%, Mg at 1.3%, Al at 14.5%, Si at 19.6%, S at 18.3%, K at 0.5%, Ca at 45.1%, and Fe at 2.7%. These significant atomic percentages signify a varied elemental makeup in the material. The prevalence of calcium and silicon, at 45.1% and 19.6% respectively as shown in table 4.7 suggests a substantial presence of cementitious or silicate components, influencing the material's properties. The diverse elemental composition, including trace amounts of other elements, provides valuable insights into the material's chemical content, aiding in understanding its potential applications in construction industry.

The spectrum overlay analysis in figure 4.10 reveals the elemental intensity distribution within the mix matrix. Calcium (Ca), silicon (Si), aluminum (Al), iron (Fe), and other associated elements emerge as dominant contributors. Their prevalence suggests a substantial presence of mineral phases, oxides, or compounds containing these elements in the material. Calcium and silicon often signify the influence of cementitious or silicate components, while aluminum and iron may indicate the presence of aluminate and iron oxide phases. The spectrum overlay's emphasis on these elements implies their significant roles in shaping the material's composition and likely influences its structural and chemical properties.

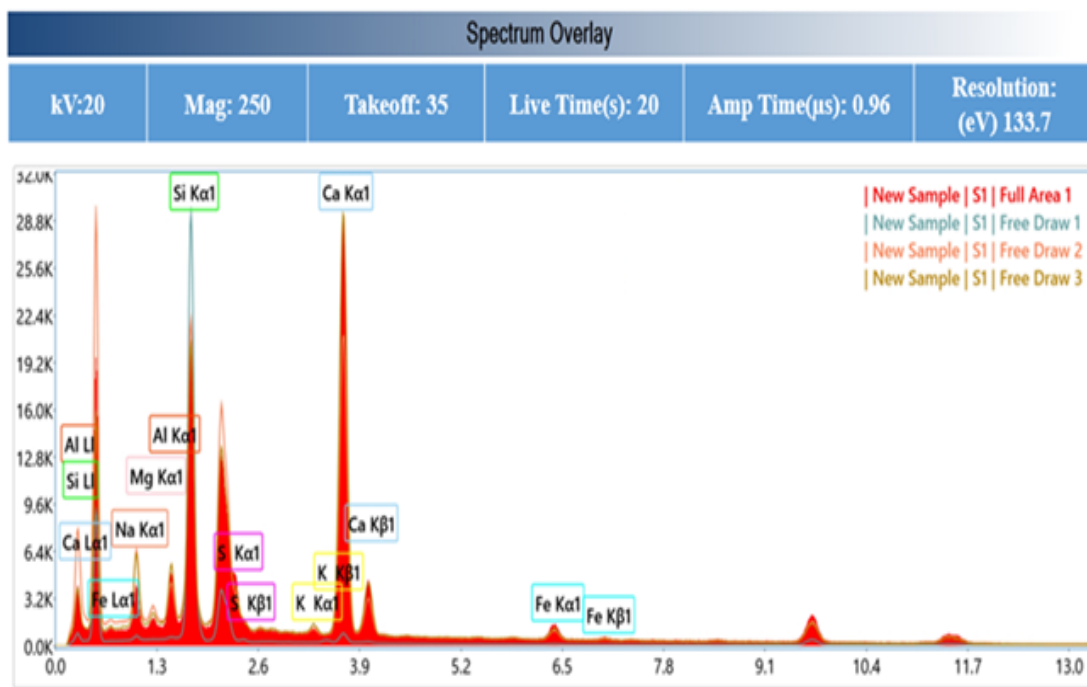


FIGURE 4.10: EDX Spectrum Overlay Graph of Geopolymer+15%LSP

4.4 Analysis of Durability Properties

The durability properties of the five distinct geopolymer mix proportions were thoroughly evaluated to ascertain their performance over time and under various environmental conditions. These properties, specifically sorptivity and porosity are instrumental in predicting the long-term performance and durability of the geopolymer. The result shows that as strength heavily depends upon durability properties.

4.4.1 Sorptivity Analysis

Sorptivity is an important property that measures a substance's ability to take in and transfer water through capillarity, it is a major factor in deciding how long concrete will last. The five Geopolymer concrete mixes in our study underwent sorptivity tests according to the ASTM C1585 standard method. The first mix, a Geopolymer mix, provided baseline data for sorptivity. This mix exhibited a certain level of sorptivity, determined by its constituent materials and their proportions. The second mix included 5% of limestone powder. Prior research suggests that limestone powder can contribute to reducing the sorptivity of concrete, improving its resistance to water absorption. The third mix, containing 10% proportion of limestone powder, presented the more reduction of sorptivity. Fourth and fifth mix proportion contains 15% and 20% limestone powder gives lowest sorptivity as shown in figure 4.11. The higher usage of limestone might have resulted in a more homogenous and denser matrix, thereby further reducing the sorptivity of these mixes.

In figure 4.12 graph's y-axis shows the initial sorptivity values ($\text{mm}/\text{sec}^{0.5}$), while the x-axis shows the concrete strength in MPa. When comparing the initial sorptivity values with the strength, we can observe the trend. As the concrete strength increases, there is a general trend of decreasing initial sorptivity. This means that higher strength concrete mixes tend to have lower initial sorptivity values. The 1st point corresponding to the 28.17 MPa compressive strength will have the highest initial sorptivity value, indicating a relatively higher rate of water absorption compared to the other mixes. The points 29.04 MPa, 30.39 MPa, 32.16 MPa, 34.44

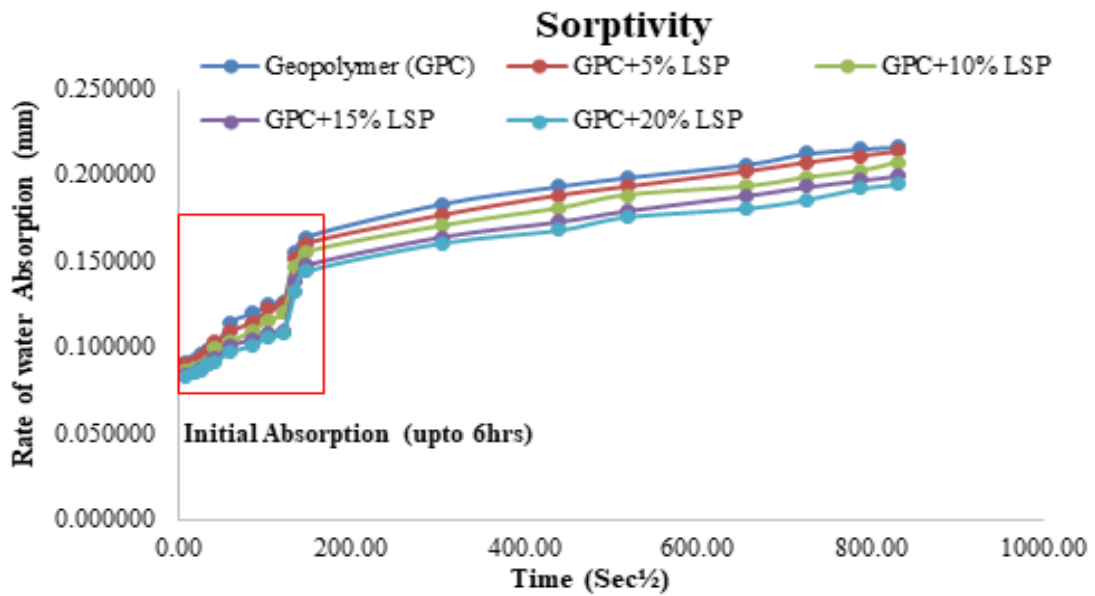


FIGURE 4.11: Sorptivity Analysis results

MPa will show progressively lower initial sorptivity values, suggesting a decreasing rate of water absorption as the concrete strength increases. Overall, the graph demonstrates that there is an inverse relationship between concrete strength and initial sorptivity. As the strength of the concrete increases, the initial sorptivity tends to decrease, indicating a reduced rate of water absorption.

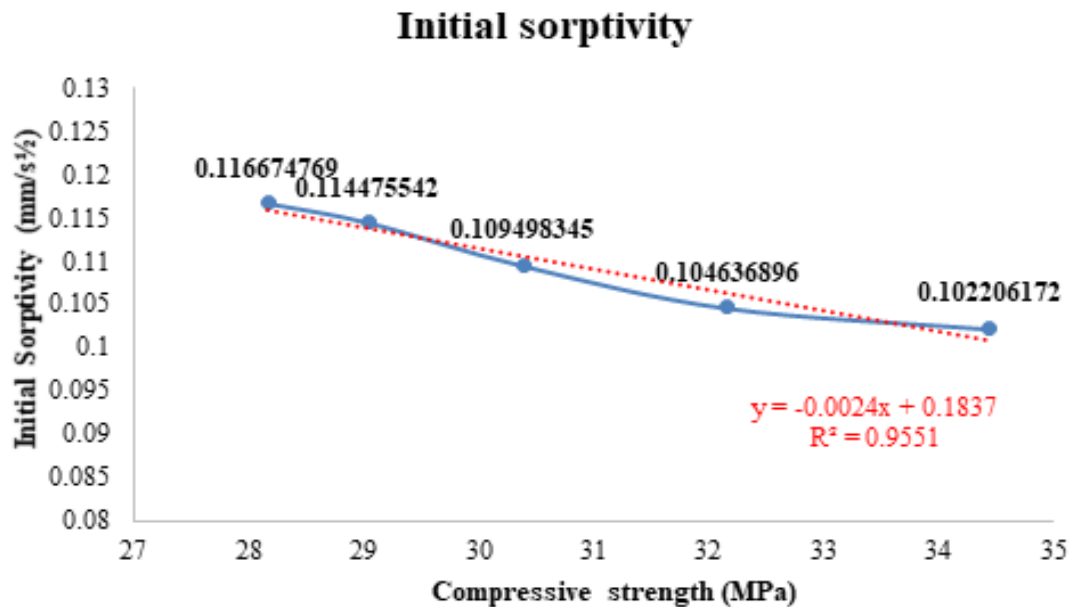


FIGURE 4.12: Sorptivity comparison with strength

These findings align with expectations, as higher strength concrete is typically denser, with lower porosity and better resistance to water penetration. The lower initial sorptivity values for higher strength mixes indicate their potential for improved durability and reduced vulnerability to moisture-related issues. It is important to note that other factors, such as mix design, curing conditions, and aggregate properties, can also influence sorptivity.

Based on our experimental research, linear regression line equation generated is:

$$Y = -0.0024x + 0.1837 \quad (4.1)$$

where x represents the compressive strength in MPa, the equation represents a linear regression line generated from the initial sorptivity data. In the graph, the compressive strength (MPa) is represented by the x -axis, and the initial sorptivity value is represented by the y -axis ($\text{mm}/\text{sec}^{0.5}$). The regression line represents the trend observed in the initial sorptivity values as the compressive strength increases. The negative coefficient (-0.0024) indicates an inverse relationship between compressive strength and initial sorptivity. As the compressive strength increases (x -axis), the initial sorptivity decreases (y -axis). The line equation $y = -0.0024x + 0.1837$ can be used to estimate the initial sorptivity values for different compressive strength values within the tested range. By substituting the desired compressive strength value for x in the equation, you can obtain an estimated initial sorptivity value.

4.4.2 Porosity Analysis

Porosity is an intrinsic property of concrete that significantly impacts its durability. It consists of the proportion of a material's void volume to its entire volume. In the context of concrete, these voids are the spaces between the cement paste and aggregates that are not filled with solid material. High porosity can increase the concrete's permeability, enabling harmful agents such as water, chlorides, and sulfates to penetrate the concrete, which can lead to a reduction in durability and lifespan. In this study, the porosity of the five different Geopolymer mixes was evaluated to assess the effect of mix proportions on this crucial property. It

is essential to note that each mix, due to its unique composition, was expected to display varying degrees of porosity. The first mix, a GGBS slag Geopolymer mix, represented a standard porosity level, acting as a control or benchmark for comparing the results of the other two mixes. Result shows high porosity as compared to other mixtures. The second mix, which incorporated small amount of limestone powder was expected to present a different porosity profile, given the alterations in its composition. Limestone powder have been reported to influence the pore structure of concrete, potentially leading to changes in porosity. Result in table 4.8 shows moderate porosity.

TABLE 4.8: Porosity Results

Sample Name	Dry weight (wd)	Submerged weight (ws)	Volume of sample (cm)	Porosity (%)	Average porosity (%)
Geopolymer (GPC)	804	863	392700	7.338	7.988
	799	861	392700	7.759	
	733	798	392700	8.867	
GPC+5% LSP	754	811	392700	7.559	7.392
	739	795	392700	7.58	
	739	791	392700	7.037	
GPC+10% LSP	783	835	392700	6.641	6.569
	810	859	392700	6.049	
	798	854	392700	7.018	
GPC+15% LSP	825	873	392700	5.818	5.739
	773	824	392700	6.598	
	812	851	392700	4.803	
GPC+20% LSP	770	807	392700	4.805	4.931
	740	778	392700	5.135	
	783	821	392700	4.853	

The third mix, with a high proportion of Limestone powder offered an even more distinctive case for porosity examination. Result shows low porosity as compared to other. Fourth and fifth mix, contains more high 15% and 20% limestone powder shows lowest porosity which is acceptable in any type of concrete. The bar chart

in figure 4.13 shows a decreasing trend in porosity as the strength and limestone percentage of the Geopolymer mix increases.

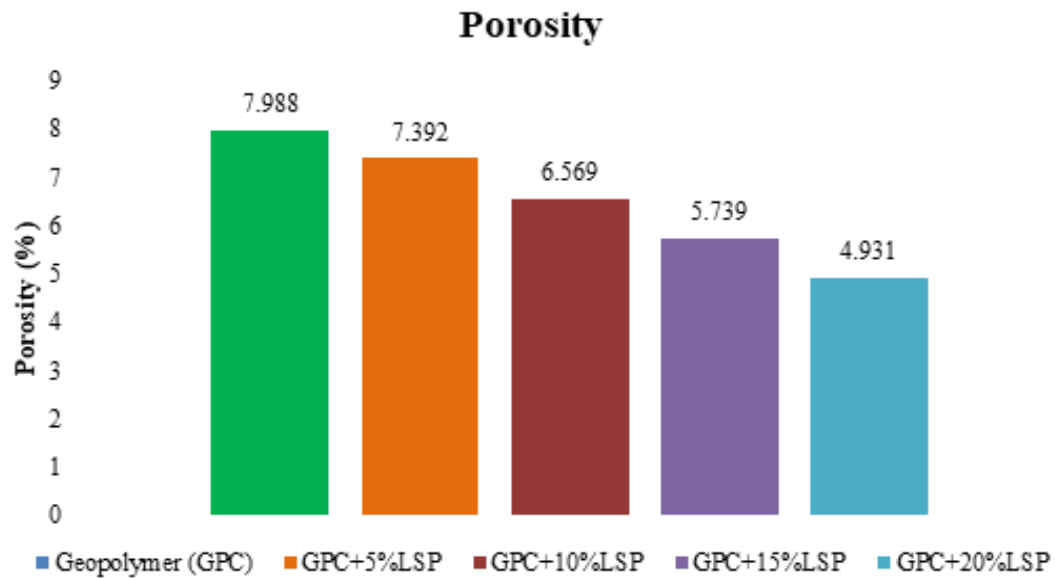


FIGURE 4.13: Porosity test results

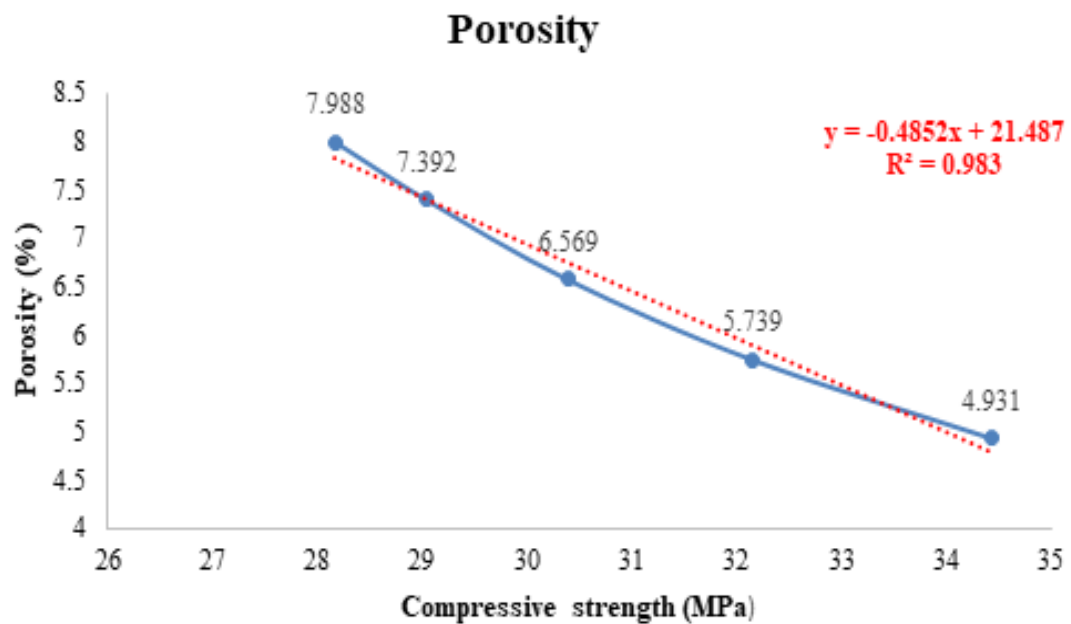


FIGURE 4.14: Porosity comparison with strength

Figure 4.14 indicates an inverse relationship between strength and porosity, which is generally expected. As the geopolymer strength increases, it implies that the material has a higher ability to withstand compressive forces without failure. This

is often achieved through better aggregate packing, improved cementitious paste, and reduced void spaces within the concrete matrix. Consequently, the lower porosity values for higher strength mixes indicate a more compact and less porous microstructure. The graph will illustrate a downward, indicating the reduction in porosity with increasing geopolymer strength.

Based on our experimental studies the regression line equation generated is:

$$Y = -0.4852x + 21.487 \quad (4.2)$$

This line equation can be used to estimate the porosity values for different compressive strength values within the tested range. Simply substitute the desired compressive strength value for x in the equation to obtain an estimated porosity value. Important notable thing is that the line equation and regression line are based on the specific data provided and the assumption of a linear relationship between compressive strength and porosity.

4.5 Correlation Analysis between Geo-polymer Strength and Durability Properties

It is significant to remember that the quality of the constituent materials and the proportions in the mix of concrete control both its strength and durability. However, the way these factors interact to produce the final properties can be complex and multi-faceted. For example, while higher strength often suggests a denser and less porous concrete matrix, which could imply improve durability, this is not always the case due to various factors such as the type and proportion of cementitious materials, aggregate size, and curing conditions.

In this analysis, the correlation between the strength (compressive) of the five different Geopolymer mixes and their respective sorptivity, and porosity values were explored. The aim was to determine how changes in strength relate to changes in these durability properties. This involved statistical analyses to identify any significant trends or patterns in the data. In this analysis, we examined the relationship

between concrete strength and two important durability properties: porosity and sorptivity.

Porosity refers to the amount of void space or open pores in geopolymer, which can impact its durability. The provided data indicated that as the concrete strength increased, the porosity decreased. Specifically, the 28.17 MPa mix had a higher porosity of 7.988%, while the 29.04 MPa mix had a lower porosity of 7.392%. This suggests a negative correlation between concrete strength and porosity, indicating that higher strength concrete generally exhibits lower porosity. Sorptivity measures the rate at which water is absorbed by concrete. The sorptivity data demonstrated that as the geopolymer strength increased, the initial sorptivity values decreased as well as final sorptivity. The 28.17 MPa mix had an initial sorptivity of 0.1167 mm/sec^{1/2}, while the 29.04 MPa mix had a lower initial sorptivity of 0.1144 mm/sec^{1/2}. This indicates a negative correlation between concrete strength and sorptivity, indicating that higher strength concrete generally exhibits lower sorptivity.

Overall, the correlation analysis between geopolymer strength and durability properties reveals a consistent trend. There is a negative correlation between concrete strength and porosity as well as sorptivity. This implies that as the geopolymer strength increases, there is a tendency for improved durability properties. Higher strength Geopolymer typically possesses a denser microstructure, reduced pore connectivity, and enhanced resistance to water ingress. These characteristics contribute to lower porosity and diminished sorptivity, which are desirable qualities for increased durability and long-term performance of concrete structures. It is crucial to remember that while the provided data supports a correlation between geopolymer strength and the durability properties mentioned, other factors, such as mix design, curing conditions, and aggregate characteristics, type of chemicals, type of binder etc can also influence these properties.

4.6 Abnormal Efflorescence

Excessive efflorescence is a noticeable sign of the geopolymer matrix hardening process, and this phenomenon is linked to the activators sodium hydroxide and

sodium silicate. The appearance of efflorescence, which is defined as the production of unattractive white deposits, is associated with salts. A thorough review of the formulation is required, as it appears that the addition of sodium silicate and sodium hydroxide as activators is a contributing factor to this phenomena.



FIGURE 4.15: Abnormal Efflorescence

Optimizing the performance and look of geopolymers during hardening necessitates addressing the unique salt-related problems, which has prompted more research into substitute activators and formulations.

4.7 Summary

The properties under mechanical testing, microstructural analysis and durability testing have been determined. The mechanical testing reveals that the compressive strength & flexural strength increases with increase in limestone powder till 20%. Similarly, durability testing shows the dense and compact structure with addition of limestone powder. (sorptivity and porosity decreases with increase in strength and limestone percentage). The SEM analysis shows pores, formation for CSH gel as well as formation of excessive tobermorite clouds with addition of limestone powder causes different chemical reactions within the geopolymer mix matrix. XRD analysis shows increase in crystallinity as limestone percentage increases and EDX shows availability of different chemical atoms percentages as well as crystalline behavior with increase in limestone powder as shown in XRD analysis. Excessive Efflorescence is observed.

Chapter 5

Practical Applications

The practical application of sustainable concrete in the building of environmentally friendly structures is based on the test results from the generated samples. To find the ideal combination, a thorough examination of several concrete mixtures is required. The structural design is based on a mechanistic empirical method and uses the ACI (Allowable Stress Design or Load and Resistance Factor Design) technique. In order to create a relationship between these values and the overall structural performance, the values obtained from each combination are essential. In this chapter, the use of Geopolymer in structural design is examined, along with a comparison between it and conventional concrete and an assessment of the structural performance that results.

5.1 Comparison for Optimized Mix

The performance of several specimens, including Geopolymer (GPC), GPC with 5% limestone powder, GPC with 10% limestone powder, GPC with 15% limestone powder, and GPC with 20% limestone powder, shows better results than the reference concrete. In particular, GPC containing 20% limestone powder exhibits a noteworthy enhancement in compressive strength, recording an 18.98% rise in comparison to pure slag-based Geopolymer. Lower porosity and sorptivity readings result from this, demonstrating the improved performance of the concrete mix that contains 20% limestone powder in the Geopolymer composition. In

terms of mechanical and durability properties, the performance of several specimens, including Geopolymer (GPC), GPC with 5% limestone powder, GPC with 10% limestone powder, GPC with 15% limestone powder, and GPC with 20% limestone powder, shows better results than the reference concrete. In particular, GPC containing 20% limestone powder exhibits a noteworthy enhancement in compressive strength, recording an 18.98% rise in comparison to pure slag-based Geopolymer. Lower porosity and sorptivity readings result from this, demonstrating the improved performance of the concrete mix that contains 20% limestone powder in the Geopolymer composition. Similarly, the microstructural characteristics show outstanding results. Aqueous aluminates and silicates are produced as a result of chemical reactions triggered by the widespread development of tobermorite in the Geopolymer+20% limestone powder. The material hardens as a result of this process, giving it remarkable mechanical and durability qualities. Moreover, Geopolymer+20% limestone powder exhibits exceptionally good durability properties, as seen by its decreased porosity and sorptivity.

5.2 Practical Application as Compare To Conventional Concrete

With a plethora of useful uses that could revolutionise the building sector, geopolymers provide a strong substitute for conventional concrete. These novel materials are the result of a chemical interaction between alkaline activators and aluminosilicate minerals, which forms a cementitious matrix. Since of their unique qualities, geopolymers are a desirable choice for many construction applications since they are strong, long-lasting, and environmentally friendly. The development of customised geopolymers for particular uses is a growing trend. In order to meet the unique needs of precast components, high-performance concrete, and even 3D-printed structures, researchers are tailoring geopolymer concretes. This change represents a departure from a general strategy and a move towards a more customised and application-specific use of geopolymers.

Furthermore, investigating the blending of geopolymers with other cutting-edge materials, such as fibres or nanoparticles, is becoming more and more important. By adding these additives to geopolymers, it may be possible to produce composites with improved mechanical and functional qualities, expanding the range of applications for structural engineering and infrastructure projects.

Current developments and trends in the field of geopolymer concrete research point to a dynamic and changing landscape. The advancement of sustainable and high-performing construction solutions is facilitated by the emphasis on optimisation, the use of alternative raw materials, formulas tailored to specific applications, and partnerships with advanced materials. These developments, together with more study, should make it easier for geopolymer concretes to become widely used in conventional construction methods. Geopolymers have a far less carbon footprint than traditional concrete, which is one of its main advantages. Carbon dioxide emissions from the production of typical Portland cement, an essential component of conventional concrete, are increased. On the other hand, industrial waste materials like fly ash or slag can be used to create geopolymers. This reduces the negative effects on the environment and provides an environmentally friendly way to deal with industrial waste. This environmentally beneficial feature is in line with the growing emphasis on sustainable building methods and minimising the environmental impact of infrastructure projects worldwide. Superior mechanical qualities are demonstrated by geopolymers in comparison to traditional concrete. A three-dimensional, amorphous structure with stronger intermolecular connections is created during the geopolymerization process, which increases the material's compressive and flexural strength. Because of its increased strength, lighter and thinner structural components may be made, which encourages better material use and lowers building costs. Geopolymers also have improved chemical corrosion resistance, which makes them ideal for use in harsh environments like chemical processing plants and wastewater treatment facilities. One significant benefit of geopolymers is their quick setting and early strength development. When it comes to achieving sufficient strength, ordinary concrete can take weeks, but geopolymers can do it in a matter of days. This expedited curing method can speed up construction schedules, reduce downtime, and ultimately save money and

increase project efficiency. The capacity to achieve early strength is very useful in situations like urgent repairs or deadline-driven infrastructure projects. When it comes to fire resistance, geopolymers outperform regular concrete. The enhanced insulation against heat transmission provided by the amorphous structure formed during geopolymerization slows down the material's quick deterioration at high temperatures. Due to this property, geopolymers are highly suitable for use in high-rise buildings, tunnels, bridges, and other vital constructions where fire resistance is essential. In addition, geopolymers contribute to a reduction in landfill waste by incorporating industrial by-products into building materials. By using fly ash, slag, or other waste materials in the process of making geopolymers, these materials can be kept out of landfills and used with less natural resources. The construction sector is encouraged to use resources sustainably and recycle them by adhering to the principles of a circular economy. The widespread use of geopolymers is hampered by some issues, despite their many benefits. Since the technology is still developing, more effort needs to be done on standardising and developing regulatory frameworks for geopolymer-based building materials. Furthermore, switching to geopolymer technology could have higher upfront expenses than using conventional concrete. Investments in education, research, and development within the building industry are necessary for this. To sum up, geopolymers offer a variety of benefits over standard concrete in practical applications that are both flexible and promising. With advantages including quick setting, fire resistance, improved mechanical qualities, and environmental sustainability, geopolymers have the potential to completely change the building industry. Growing awareness and technological advancements will allow geopolymers to have a significant impact on sustainable and effective building methods in the future.

5.2.1 Modern Trends and Innovations in the Field of Research of Geopolymer Concretes

The quest for eco-friendly and high-performing building materials has led to a boom in the field of geopolymer concrete research and invention. In order to enable broad adoption, current trends point to a growing emphasis on improving

geopolymer formulations, looking into novel raw materials, and addressing logistical issues.

To enhance the mechanical characteristics and durability of geopolymer mixes, researchers are hard at work improving their designs. The focus of optimisation efforts is to achieve the ideal ratio of precursors, activators, and curing conditions for aluminosilicate in order to optimise the material's flexural strength, compressive strength, and resistance to environmental influences. The expansion of geopolymers' potential applications in traditional construction projects is greatly dependent on this trend.

Investigating advances in raw materials is an important area of research. Although fly ash and slag are still commonly used, researchers are looking at other waste materials and industrial by-products to expand the feedstock options. This addresses concerns about the availability and consistency of conventional precursors while also improving the sustainability of geopolymer synthesis.

5.3 Summary

The mechanical and durability features of Geopolymer concrete (GPC) are enhanced by the addition of limestone powder. Specifically, GPC+20% limestone powder has much higher compressive strength in addition to lower porosity and sorptivity. Its great durability and performance are further boosted by its improved flow ability, remarkable flexural strength, and favorable microstructural alterations. With its many benefits such as excellent mechanical qualities, quick setting times, fire resistance, and environmental sustainability, geopolymers are revolutionising the building industry and paving the way for a more sustainable future. Trends in geopolymer research continue to highlight the need for these novel materials to be widely adopted, optimized, and sustainable.

Chapter 6

Conclusions and Recommendations

6.1 Conclusions

For the development of a sustainable concrete in term of geopolymer and then a sustainable for structures, GGBS slag is used as replacement of cement and limestone powder is used in five various ratios, such as 0%, 5%, 10%, 15%, and 20%. For each of the five mix design proportions, the molarity of sodium hydroxide is fixed at 14 molar, and the activator ratio of sodium hydroxide to sodium silicate is kept constant at 2.5. Fixed 3% high range water reducer is mixed in all mix design proportions. Mechanical and durability tests are performed along with the micro-structural analysis, resulting following conclusions are drawn:

1. Mechanical testing of the specimens reveals the strength in compression, flexural strength and flow ability. The conclusions are discussed below:
 - Geopolymer + 20% limestone powder shows the maximum compressive strength which is 34.44 MPa. The reduction in compressive strength as compared to Geopolymer+20% limestone powder is 6.62%, 11.76%, 15.68 and 18.21% respectively(20% limestone with ggbs slag gives 34.44 MPa, 15% gives 32.16 MPa, 10% gives 30.39 MPa, 5% gives 29.04 MPa and pure Geopolymer gives 28.17 MPa).

- The Flexural strength of GPC+5%, GPC+10%, GPC+15%, GPC+20% increased as compared to the reference specimens (Geopolymer). The increase observed is 0.674%, 32.94% and 93.83% and 205.89% respectively. (20% limestone with ggbs slag gives 9.706 MPa, 15% gives 8.423 MPa, 10% gives 6.267 MPa, 5% gives 4.746 MPa and pure Geopolymer gives 4.714 MPa)
- Flow ability of Geopolymer decreases with increase limestone percentage. Pure GGBS slag gives 96% flow. Addition of limestone with 5% increment upto 20% gives 93%, 90.4%, 88%, and 86.5% flow.

2. Microstructural properties reveal following conclusions:

- The SEM imaging of fractured surface reveals that pore are available in Geopolymer. Due to use of GGBS slag, macro and micro crystals are getting prepared which is in favor of matrix. Moreover excessive tobermorite clouds are prepared due to addition of limestone powder force different chemical reactions within the geopolymer mix matrix.
- XRD analysis shows formation of quartz, maganosite, wollastonite,, pottasium maganese oxide, alumanium silicon, Calcium silicate in Geopolymer as well as in limestone mix matrix. Key indications are found in XRD analysis are pure GGBS slag Geopolymer shows amorphous behavior but as the percentage of limestone increases, matrix starts showing crystalline behavior with maximum crystallinity behavior at 20% limestone powder.
- EDX analysis shows chemical material composition with their notable concentrations suggest a complex mixture, potentially encompassing silicate minerals, salts, and metal oxides. With addition of limestone Ca and Si dominates maximum over the other materials.

3. Durability testing shows the dense and compact structure with addition of limestone powder:

- Porosity decreases as percentage of limestone increases. This indicates an inverse relationship between porosity and limestone addition up to

20%. Geopolymer+20% limestone gives minimum porosity which is 4.931%.

- Sorptivity also decreases as the percentage of limestone increases. When comparing the sorptivity values with the limestone percentage, it is observed that the trend is inverse. The sorptivity tends to decrease, indicating a reduced rate of water absorption with addition of limestone powder. Geopolymer+20% limestone powder gives minimum sorptivity value which is $0.102206172 \text{ mm/sec}^{0.5}$.

Hence, based on the above results, alkali activated geopolymer having limestone has great potential to be used in structures in replacement of conventional concrete for more sustainability and better performance. The partial replacement can save the overall cost of structure. Moreover, utilizing alkali-activated geopolymer with limestone yields structures outperforming traditional concrete in terms of performance and durability.

6.2 Future Work

Based on the conclusions deduced from this research work, following are the recommendations for future work:

- Limestone powder should be used in different proportions by reducing the content of slag, in order to read out an optimized dosage of limestone powder for better compressive strength.
- More natural geopolymer should be explored which have a similar nature to limestone powder with slag in order to develop sustainable concrete.
- Properties of Geopolymer having slag and limestone should be evaluated for higher age i.e. 90 days of curing.
- Properties of Geopolymer having slag and limestone should be evaluated for higher and lower activator ratios i.e. 16M, 12M with change in activator ratio as well.

- The same combination should be used but different samples should be cast for mechanical testing and water absorption as well as for other durability tests like acidic attack etc.

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