Spectrum of Engineering Sciences
ISSN (e) 3007-3138 (p) 3007-312X Volume 3, Issue 6, 2025

# EXPERIMENTAL INVESTIGATION OF THE EFFECTS OF PRESSURE ON THE MECHANICAL, THERMAL AND MICROSTRUCTURAL PROPERTIES OF CLAY-BASED GEOPOLYMERS

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**DOI:** https://doi.org/10.5281/zenodo.15703043

**Keywords**
*Clay-based geopolymer, Compaction pressure, Sodium hydroxide activation, Compressive strength, XRD, SEM, Sustainable construction.*

**Article History**
Received on 08 May 2025
Accepted on 08 June 2025
Published on 17 June 2025


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**Abstract**
*This study reveals that compaction pressure has a strong and positive effect on the mechanical and microstructural properties of clay-based geopolymers. Using naturally available clays from Cherat and Gilgit-Baltistan, activated with a 10M sodium hydroxide solution, geopolymer samples were successfully developed and tested under controlled conditions. The compressive strength results showed a clear trend: as the applied pressure increased from 5000 to 24,000 pounds, the strength of the geopolymer also improved, reaching up to 48.10 N/mm². This strength is almost four times higher than that of traditional fired clay bricks commonly used in local construction. XRD analysis confirmed that pressure promoted the formation of new crystalline compounds such as natrosilite, sodium acetate hydrates, and sodium hydrogen silicate, which are associated with stronger geo-polymeric bonding. SEM images further supported these findings by showing a progressive reduction in porosity and an improvement in particle bonding and matrix formation as pressure increased. The overall performance of the samples prepared under higher pressure was clearly superior in terms of strength and structural integrity. These results show that applying compaction pressure is a practical and effective method to improve the quality of geopolymer materials without the need for high-temperature processing. In the future, research can be extended to study the long-term durability, shrinkage behavior, water resistance, and thermal insulation properties of these materials. Additionally, combining different clays, using alternative alkali activators, or applying pressure in combination with mild heat curing may further enhance their properties and promote their adoption in sustainable construction applications.*

## INTRODUCTION

The most used building material throughout the world is concrete that mainly consists of Portland cement, water, and aggregates. It is often prevalently used in infrastructure, houses and industry, but this prevalence takes a heavy toll on the environment. The manufacturing of Ordinary Portland Cement (OPC) is energy intensive and depends extensively on calcination of limestone which leads to a high intensity of carbon dioxide ($CO_2$), which is a major greenhouse gas causing global warming and climate changes (Das et al., 2018). Production of one ton of cement emits approximately 1 ton of $CO_2$ in the air. The construction industry has become one of the biggest sources of anthropogenic $CO_2$ emissions considering that billions of tons of cement are manufactured every year. Also, the ecological impact of the cement industry is further increased by the damage of the environment through quarrying raw materials to processes and the large amounts of energy used in the process of manufacturing the cement (Rahman et al., 2015).

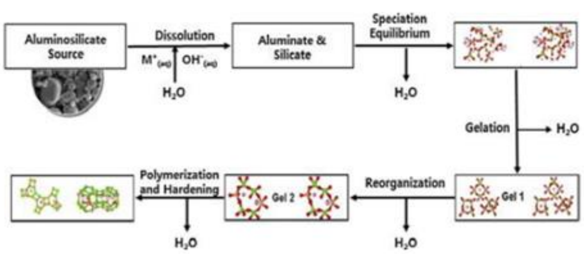


Fig 1: Stages of geopolymerisation process

The environmental emergency push to find some new, sustainable solutions that may fulfill the structural requirements of modern construction without affecting the state of the environment. As an answer to these questions, geopolymers have received some interest as possible alternatives to traditional cementitious materials. Geopolymers are inorganic and ceramic-like types of material that are built through the process of polycondensation of precursors of aluminosilicate material under alkaline environments. This is called geo-polymerization and gives rise to a three-dimensional amorphous (or semi-crystalline) Si-O-Al network (Liew et al., 2016). This structure gives high thermal, chemical and mechanical properties to the resulting material. Markedly, geopolymers have advanced fire resistance, chemical resistance, reduced shrinkage and their carbon emission are much less than OPC concrete (Ettahiri et al., 2023).

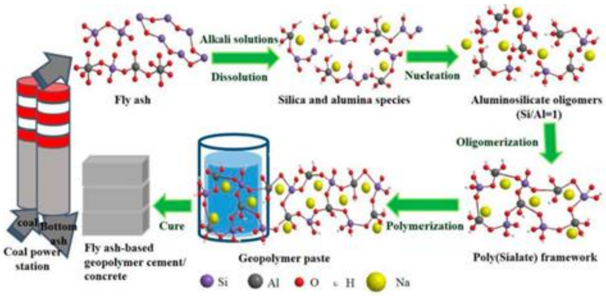


Fig: 2: Fly Ash Geopolymers

Their production does not involve the calcination step, and it uses a significantly lesser amount of energy and leads to a significant $CO_2$ emission saving too. Therefore, the technology of geopolymers implies a paradigm shift in sustainable material science, which coincides with the green infrastructure goals pursued at the global level and carbon neutrality. Geopolymer raw materials may be natural, synthetic or by-products of industry(Liew et al., 2016). Clay is a freely available, low-cost and high-performing precursor among the natural sources. Geo-polymeric frameworks are dependent on silica ($SiO_2$) and alumina ($Al_2O_3$) materials, both prevalent components of clays, including kaolinite and illite. When they are mixed with an alkaline activator like sodium hydroxide (NaOH) or sodium silicate, these minerals dissolve and again form a cohesive cementitious matrix. The self-sustaining characteristics of geopolymer formation can be applied in geopolymer synthesis with local clay, therefore, in areas where clay has been found in significant quantities and is freely available such as the case of Pakistan, the dependency on imported components may be shelved, and the cost of construction reduced since the raw materials are in the area itself. Precisely, this research has used clay found in Cherat and Gilgit-Baltistan regions, which too contain aluminosilicates of superior grade in geological hills (Malkani et al., 2016). Through use of such native materials, not only does the study give prominence to the concept of stewardship of the environment but also throws light on the possibility of locally based, sustainable building materials to developing economies.

Although the positive features of geopolymers are known, there are plenty of gaps in the knowledge about the effect of the mechanical processing parameters on the final properties. This is one parameter; the pressure that is used to compact the samples. The studies are concentrated around chemical composition, curing temperature and the molar concentration of the alkaline activator (Benhelal et al., 2012).

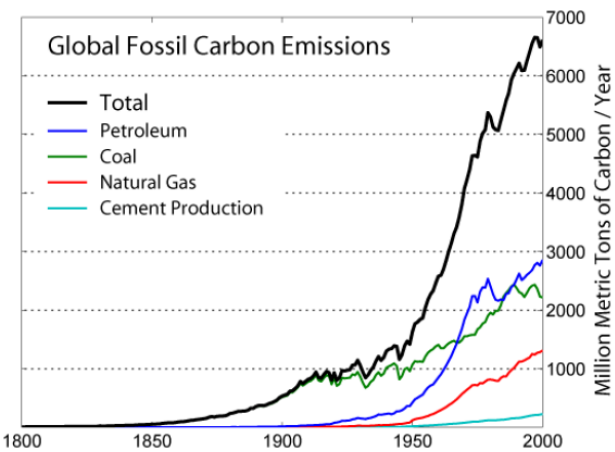


Fig. 3: Global Fossil Carbon emissions

Nevertheless, the effect of pressure on densification of geopolymer matrix enhancing the particle packing and encouraging interfacial bonding has not been well investigated. This study is based on the hypothesis that higher compaction pressure that is used to mold clay based geopolymer samples will result in better mechanical characteristics, especially compressive strength. By applying pressure throughout forming stage, the void spaces are reduced, the orientation of the particulate is improved and the contact surface through which geo-polymerization reactions occur is maximized. These microstructural refinements can greatly increase the compressive strength of the material thus allowing it to be considered a material capable of high-performance structural application.

In this regard, pressure is no longer a shape maker but an important variable that determines the performance of the final product. Indicatively, traditional clay bricks are normally molded in line with the extrusion processes and high temperatures, both which use up energy and pollute the air with emission of greenhouse gases and other toxic fumes. Geopolymer bricks, by contrast, can be formed at ambient or slightly elevated temperatures, and have cures that do not need combustion (Ettahiri et al., 2023). Manufacturing process could be enhanced to produce bricks and the components of the structures having increased strength, durability, and environmental compatibility by incorporating the pressure assisted molding processes.

This paper carries out a rational study on how the different levels of exerted pressure (5000-24000 pounds) affect the compressive strength and microstrutural properties of the geopolymer samples that are made of clay. The raw materials include Cherat clay and Gilgit-Baltistan-clay in equal proportions and 10 molar sodium hydroxide solutions as alkaline activators. The samples are then compacted with the help of hydraulic press and these are then cured between 35$^0$C and 42$^0$C. A complete set of test is applied in order to characterize the mechanical and structural development of the samples, the compressive strength is determined using an Universal Testing Machine (UTM), X-ray Diffraction (XRD) is used to confirm the phases present in the sample, and finally the microstructure is imaged using Scanning Electron Microscopy (SEM). The main aim of the study is to determine the direct relationship between the sensible amount of compaction pressure and the performance of the material in geopolymer composite (Liew et al., 2016).


Fig. 4: Environmental effects of geo-polymer

ISSN (e) 3007-3138 (p) 3007-312X Volume 3, Issue 6, 2025

Through the explanation of this relationship, the study would contribute fundamental information that would be feasible to optimize manufacturing processes of geopolymer bricks, tiles and other structural components. Moreover, the results are likely to contribute to the design of industrial-scale manufacturing procedures capable of maximizing on the mechanical functionality and the eco-friendliness. This study has a great importance outside academia. In third world countries such as Pakistan, there is an even greater need of inexpensive, long lasting and environmentally friendly materials of construction. Fast urbanization, short supply of resources along with vulnerability to climate change require incorporation of innovative technologies that can achieve high performance results without compromising on the environment (Sukmak et al., 2013).

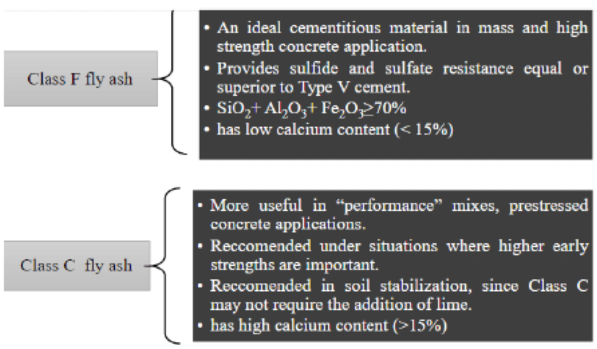


Fig.5: Difference between Class F and Class C fly ash

A possible solution to these problems is clay-based geopolymers that can be engineered under controlled conditions of pressure. They are made using rich resources found locally; they do not require high temperatures processing, and their strength is much greater than regular bricks: reportedly up to four-fold in the optimal case. This strength gain is not only able to diminish the amounts of materials that are used with each unit of construction, but also it boosts the durability and even safety of the structure especially in the earthquake prone areas or even flood prone areas.

## 2. Materials and Methods

The purpose of carrying out this experimental study was to determine the influence of different concentrations of the compaction pressure in reducing the mechanical and micro structural characteristics of the geopolymers (clay based). All the experiments were conducted under Material Research Laboratory (MRL) and Centralized Resource Laboratory (CRL), University of Peshawar.

### 2.1 Raw Materials and Preparation

The main source of aluminosilicate was chosen as two natural clay samples. This was procured in Cherat and Gilgit-Baltistan regions of Pakistan where there have been geologically rich deposits of clay. All the clays were initially air-dried at room temperature for 48 hours to prepare moisture free clays. The clays were dried and grinded before sieve through a 150-mesh (aperture 0.105 mm or105 microns) (Wiley, 1911). This was involved in obtaining a fine homogenized particle size distribution that was used in achieving uniform mixing and better reaction kinetics. Cherat and Gilgit-Baltistan clays were thoroughly mixed (50:50 weight ratio) in a mechanical mixer and the process continued to 15 minutes to obtain the uniform mixed up composite. Physical and chemical compatibility of two types of clay was visually inspected and presumed sufficient due to color, texture, and the earlier studies, which claimed the similar alumina -silicate structure.

### 2.2 Alkali Activator Solution

An alkaline activator was used, sodium hydroxide (NaOH). The 10 molar (10M) solution of NaOH was realized by adding 400 grams of pellets of NaOH (1 mol = 40 g/mol) in 1 liter of distilled water. To remove the possibility of thermal damage when blending with the clay it was necessary to stabilize the solution and cool it, and therefore it was placed under ambient conditions for 24 hours to rest. The reason as to why the high concentration was selected was because there was the desire to dissolve aluminosilicate minerals thoroughly that enhance the effective geo-polymerization process.

### 2.3. Mixing and Sample Molding

A liquid-to-solid (L/S) ratio of about 0.4 by weight of dry mixture of clay in sodium hydroxide solution was used. The dispersion was hand stirred during the initial 5 minutes to avoid early setting and after which it was placed in a mechanical stirrer and stirred at the same rate for about the next 10 minutes. The outcome was a thick paste like geopolymer slurry that could be molded. The pouring of fresh geopolymer paste into steel cylindrical molds with an inner diameter of 13 mm and a height of 5.2 mm was done. Molds were cleaned and lightly oiled so that it could be easy to remove the hardened sample. Four groups of samples were prepared with the help of a hydraulic pressing machine to investigate the impact of pressure. Each group had a different pressure as table 1 below indicates.

**Table 1: Compaction Force Applied to Sample Groups**

| Group | Applied Pressure (lbs) | Approx. Pressure (MPa) | Sample Dimensions (mm) |
|---|---|---|---|
| A | 5,000 | ˜15 | 13 (D) × 5.2 (H) |
| B | 10,000 | ˜30 | 13 × 5.2 |
| C | 15,000 | ˜45 | 13 × 5.2 |
| D | 24,000 | ˜72 | 13 × 5.2 |

Every sample was pressed for 2 minutes and was removed carefully out of the mold. The pressure increased slowly in order to prevent the crack or uneven compaction. Each of the groups was sampled in triplets to obtain repeatability and statistical value.

### 2.4 Curing Conditions

The samples were cured in ambient conditions (35°C to 42°C) over a period of 48 hours after they have been molded. Dried took place in a cool place under proper ventilation so that the drying is slow and uniform. The temperature range was chosen to represent the field conditions as will be normally found in Northern areas such as Pakistan. There was no high-temperature firing in the process as compared to conventional bricks, and this increases the sustainability of the process. In order to improve the level of geo-polymerization, some post activation process was made during curing; extra NaOH solution was injected to the samples at intervals of 12 hours by using a syringe. This has managed to provide hydroxide ions that can react with unreacted alumina-silicate particles with a possible outcome of keeping the mechanical properties high and lower porosity.

### 2.5 Compressive Strength Testing

The compressive strength of individual samples in cured condition was determined on a Universal Testing Machine (UTM) (Make: ZwickRoell, Model: Z030). The sample was laid vertically, and a load pushing down on the sample was made until the sample broke. It used cross-sectional area to determine the compressive strength of:

$$\mathbf{6c} = \frac{\mathbf{F}}{\mathbf{A}}$$

The yield load and strain were also recorded to observe the deformation behavior of the samples under load.

**Table 2: Compressive Strength Results**

| Pressure Force (lbs) | Yield Load (N) | Strength (N/mm²) | Yield Strain (%) |
|---|---|---|---|
| 5,000 | 4413 | 33.247 | 20.615 |
| 10,000 | 4527 | 34.106 | 18.712 |
| 15,000 | 6111 | 46.040 | 13.654 |
| 24,000 | 6385 | 48.104 | 23.019 |

### 2.6 X-Ray Diffraction (XRD) Analysis

To investigate phase composition and transformation of minerals, the raw clay came and some of the geopolymers were subjected to XRD. The applied instrument was a JEOL JDX-3532 X-ray Diffractometer with Cu-K alpha radiation (l = 1.5418

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A) modified to run at 40 kV and 30 mA. The scanning was carried out between 10 to 80 at a speed of 2degrees per minute. The raw Clay exhibited a major peak as that of quartz ($SiO_2$) with JCPDS Card No. 46-1045. The 15,000 lbs. pressure sample exhibited other peaks of Sodium silicate ($Na_2Si_2O_5$) a geopolymer related crystalline phase (JCPDS 241123), as well as sodium acetate trihydrate ($C_2H_3NaO_23H_2O$, JCPDS 281030). Within the sample of 24,000 lbs, the other compounds like $Na_2HSiO_4H_2O$ (JCPDS 200579) and $C_2HNaO_4H_2O$ (JCPDS 321087) were also identified. Such structures indicate continuous acidification of sodium ions in contact with clay minerals, which agrees with an increased level of polymerization in incremental pressures of compaction.

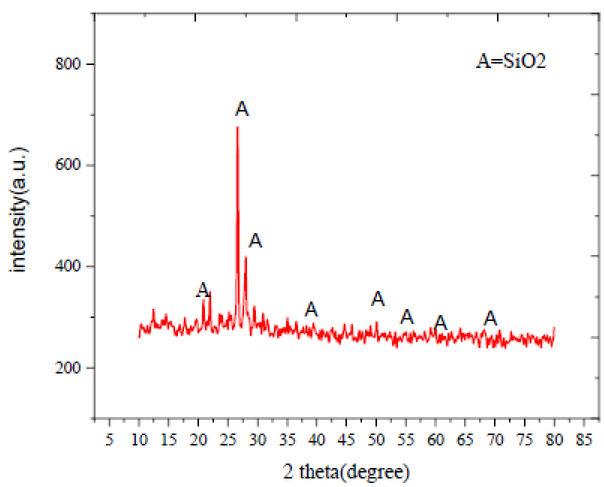


Fig. 5: XRD Analysis of Clay without NaOH solution

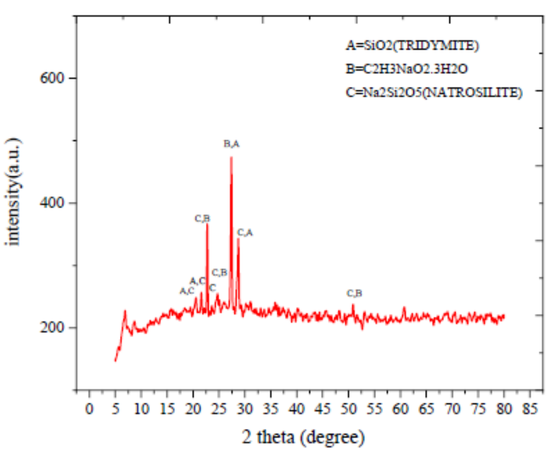


Fig. 6: XRD Analysis of sample made with 15000 pounds force and 10 molar NaOH solution

## 2.7 Scanning Electron Microscopy (SEM)

The samples were scanned using SEM to see microstructural changes. JSM-5910 SEM system manufactured by JEOL with the resolution of 2.3 nanometers and a magnification of 300,000 times was used. Photographs at 20, 000, 25,000, 30,000 and 35,000 magnifications were taken of all the sample groups.

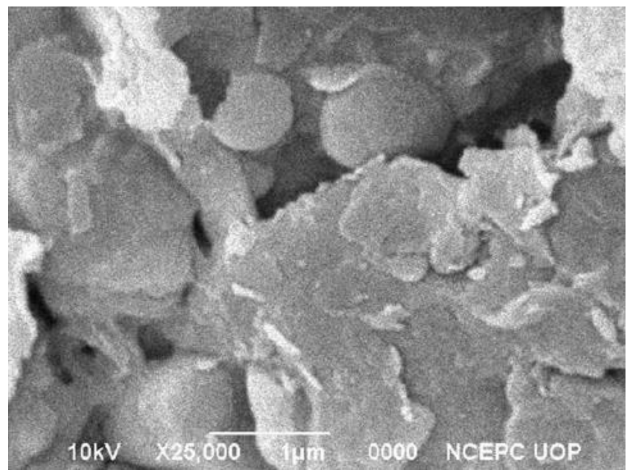


Fig. 7: SEM results of simple Clay Sample with 25000 magnification

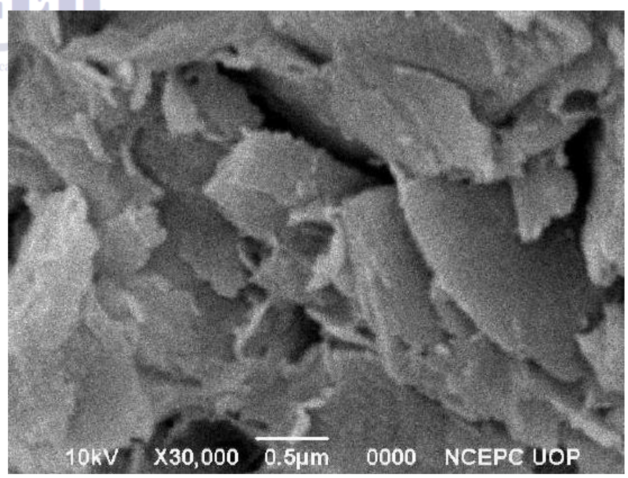


Fig. 8: SEM results of simple Clay Sample with 30000 magnification

When in a simple form (non-pressured), the clay sample had irregular shapes of loosely bonded particles that were highly porous. It was found that the sample 15,000 lbs presented much denser microstructure and less voids and improved connection between particles. The sample of 24,000 lbs sample had a compact, gel-like phase and the continuous matrix of pores with minimal pores and microcracks. These results justify why the compressive strength improved with an increment in compaction pressures.

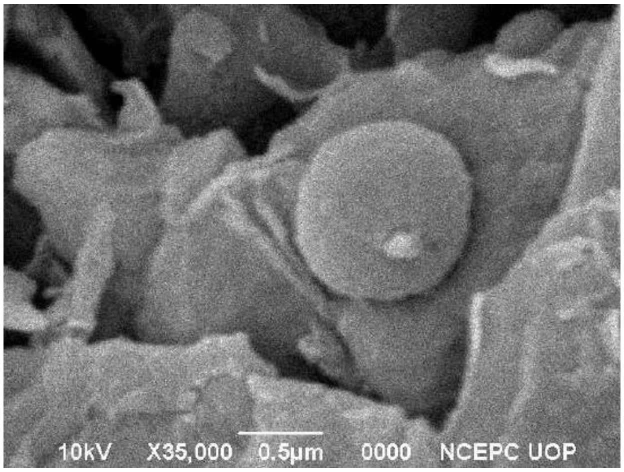

Fig. 9: SEM results of simple Clay Sample with 35000 magnification

The experiments were carried out keeping all other factors uniform except the pressure, by maintaining the type and source of clay, activator type and concentration, the time of curing and the sample size. Such control permitted an intentional examination pressure as the basic driving component in the geopolymer performance.

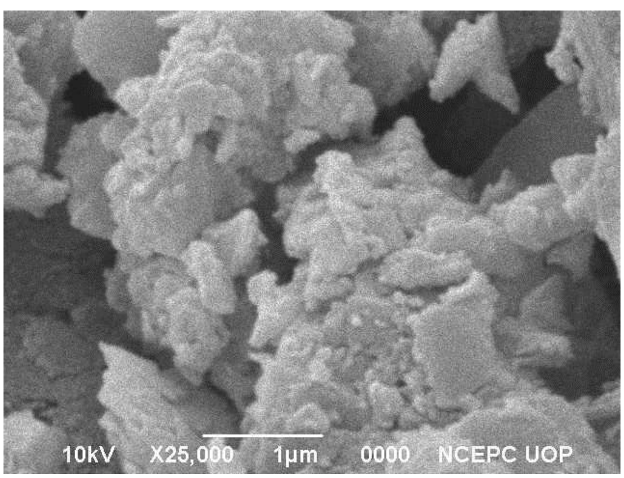

Fig. 10: SEM result of Clay Sample made with 15000 pounds pressure force with 25000 magnificatiom

### 3. Results and Discussion

The findings of this experiment demonstrate that using pressure in the process of making clay-based geopolymers influences the strength and the inner structure of this material significantly. After testing sample with Universal Testing Machine, it was also discovered that compressive strength improved with the increase in pressure applied in molding the samples (Douiri et al., 2017). The compressive strength of 33.25 N/mm$^2$was at 5000 pounds pressure. At 10,000 pounds, there was a bit increment in the strength, to 34.11 N/mm$^2$. The strength was found to take a far greater steady rise at the pressure of 15,000 pounds when it went up to 46.04 N/mm$^2$. The sample prepared with 24,000 pounds of pressure was used to come up with the highest strength of 48.10 N/mm.

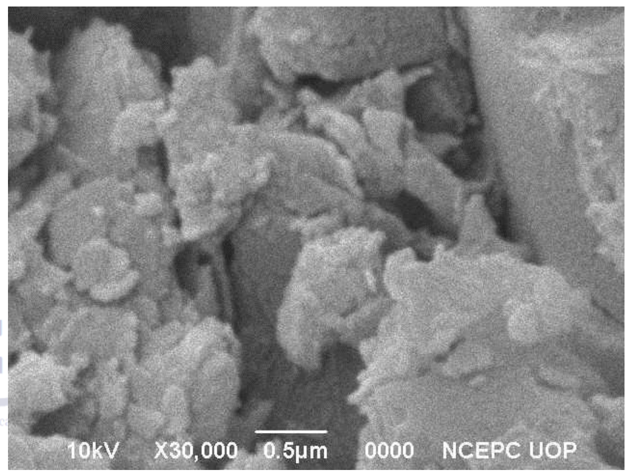

Fig. 11: SEM result of Clay Sample made with 15000 pounds pressure force with 30000 magnification

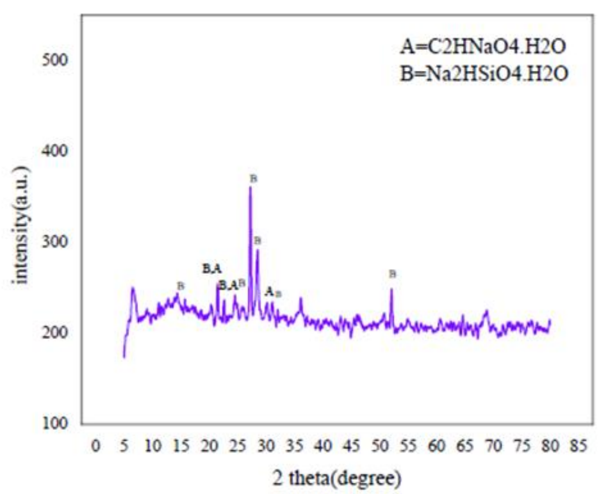

Fig. 12: XRD Analysis of sample made with 24000 pounds force and 10 molar NaOH solution

This is a clear demonstration of one fact: that more compaction pressure results in improved mechanical performance. This is because higher pressure eliminates the air bits, gets the particles closer together and forms stronger, denser material. Other than strength values, values of strain were obtained. The sample that was pressed at 15,000 pounds had the lowest strain and hence it was stiffer and not flexible. Conversely, the largest strain was on the sample with 24,000 pounds which means that it may bear more stress until it breaks. It denotes that the high-pressure samples are strong and load enduring. X-ray Diffraction (XRD) tests were conducted to determine what transformations occurred within the material. Before a reaction could occur, the raw clay contained predominantly quartz, which is a common mineral. Following the geopolymer reaction, in particular samples prepared in higher pressure, new compounds emerged.

The sample prepared at 15,000 pounds lead to the formation of natrosilite and sodium acetate trihydrate, which are formed during alkali reaction. More complicated materials were discovered in the sample, which is 24,000 pounds, which include sodium hydrogen silicate hydrate and sodium acetate monohydrate (Abdullah et al., 2019). The raw clay did not contain these new phases, so they obviously formed during the reaction between the clay and sodium hydroxide and with the help of the pressure. This aids the notion that pressure enhances the process of chemical reaction in the geo-polymerization process. These findings were supported by the Scanning Electron Microscopy (SEM) pictures. The structure of the simple clay sample, in which no pressure was applied, appeared gravely and porous (Silva et al., 2019). Particles were loose and quite a few cracks could be seen. This was the reason why the strength of that sample was weak. The sample prepared at 15000 pounds had a much cleaner and tighter microstructure. There were less pores, bonding between particles was higher. On the sample of 24,000 pounds the surface appeared very compact and smooth (Abdullah et al., 2020). There was a close structure and minute pores could hardly be visible. This image helped us to understand the fact that the stronger the pressure, the denser the material will be. The outcome of compressive tests, the XRD, and SEM all seem to suggest one thing, pressure enhances the behavior of clay-based geopolymer materials. It becomes stronger, the inner structure denser and its chemical reactions more thorough (Tahmasebi Yamchelou et al., 2021). This implies that pressure can be effectively and easily used in the manufacturing process to enhance the quality of geopolymers (Ogundiran & Kumar, 2015). In real practice, such technique might be applied to produce bricks or other building elements, not only stronger than usual clay bricks, but also eco-friendlier, in the sense that they would not require baking in ovens. This will make it cleaner, at a low cost and sustainable in the future.

## 4. Conclusion and Recommendations

As results of this experiment indicate very clearly, the pressure of compaction is important to the performance of clay based geopolymer substances. By mixing natural clay sampled in Cherat and Gilgit-Baltistan and treating the geopolymer with a solution of sodium hydroxide in the concentration of 10M, it was possible to create the samples of the geopolymer with different mechanical and structural parameters in accordance with the pressure applied during racemic molding. As the pressure of compaction increased, the compress strength rose substantially to a high of 48.10N/mm$^2$ with 24,000 pounds of pressure and was close to four times the strength of the conventional clay bricks. XRD analysis specifically determined that increased pressures resulted in more of those complex crystalline phases being produced, including natrolite, sodium acetate monohydrate as well as sodium hydrogen silicate hydrate, which is suggestive of a more complete geo-polymerization reaction. SEM imaging was used to confirm this since with more pressure a denser and more compact microstructure was formed that contained fewer pores and a stronger adherence of particles. The findings indicate that pressure not only enhances the strength of the mechanical geopolymer samples but also assists in the development of superior internal structure. An environmental benefit of using geopolymer material is also noted in the study, where such material is not kiln fired and less carbon emission is required. According to these findings, it is advisable that in the future high-pressure geopolymer composites studies focus on long-term durability, thermal insulation and water absorption properties. It is also implied that varieties of alkali activators and mixtures of clay can also be done to improve performance further and expand the usage of geopolymers in construction industry especially in a part of the world where the conventional brick-making techniques create hazards to the environment.

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