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# Mechanical Properties of Gypsum-activated Ternary Hybrid Geopolymer Mortar Cured in Ambient Conditions

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**ABSTRACT**

Alkali activation geopolymer material is a type of green inorganic polymer that can effectively reduce carbon footprints and produce viable solutions to industrial waste utilization. Desulphurization Gypsum (DG) was blended with Ground Granulated furnace slag (GGBS) and pulverized Fuel Ash (PFA) to produce ternary blended geopolymer. The ratio of the alkaline activator solution of $NaOH/Na_2SiO_3$ was kept at 1.2. Laboratory tests investigated the engineering properties of GGBS-FA-DG ternary geopolymers. The specimens were cured at ambient temperature for 7 and 28 days and compression strength were determined. With the addition of gypsum, the strength increased first up to a certain limit then decreased with optimal DG content of 10%. The use of DG as secondary activator resulted in creating sustainable building materials has driven progress in geopolymer technology.

## 1. Introduction

One of the industries with the fastest global expansion is construction. The number and scope of infrastructure have risen significantly due to increased population and urbanization. Throughout the world, almost 5 to 10% of employment is provided by the construction industry [1]. The commonly consumed construction material is concrete. Cement is used as a binder for concrete, such as Ordinary Portland Cement. It is considered the most effective binder and has been used for more than 200 years, as it produces reinforced structures that can withstand heavy loads [2]. The manufacturing of cement requires a lot of energy. It consumes 5% of industrial energy and emits 7% of carbon dioxide ($CO_2$) globally [3]. With the development in infrastructure, the need for cement is increasing; by 2050 it will increase to 6000 million tons per year. The primary source material used for the synthesis of cement is limestone, a non-renewable material. An extreme decline in the quantity of limestone is expected after some years. The depletion of natural resources and huge

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carbon dioxide emissions cause serious environmental threats disturbing ecosystems, biodiversity loss, and global warming [4]. Besides, large amounts of industrial waste are produced globally, the disposal of which is a major challenge for industry. The depletion of natural resources, huge emissions of $CO_2$, and large amounts of industrial waste encourage scientists to produce an alternative binder that eliminates the use of cement from the construction industry. Geopolymer concrete has numerous advantages when compared with ordinary Portland cement (OPC) concrete. They can reduce $CO_2$ emissions by about 90% more than OPC [5], resist chemicals, fire, and high temperatures, and provide a viable solution to manage industrial waste [6].

Industrial waste such as GGBS [7,8], silica fume [9], metakaolin [10], gypsum, and fly ash [11] are all sources of aluminosilicates that possess the potential to replace OPC in concrete. All these materials possess pozzolanic properties making them a suitable substitute for OPC in concrete. Most of the gypsum is stockpiled, it not only occupies large volumes of land but also causes several other issues. The sulfate ions ($SO_4^{2-}$) from gypsum undergo a reduction reaction with organic matter to produce $H_2S$ gas causing environmental pollution and odor [12]. The use of waste as raw materials for building materials meets the requirement for green and low-carbon alternatives paving the path toward sustainable development [13] and decreasing the need to consume natural resources [14]. Nowadays Desulphurization gypsum is used to produce cementitious material that proved to be an eco-friendly alternative to ordinary Portland cement in terms of reducing $CO_2$ emissions. Desulphurization gypsum (DG) that contains calcium sulfate dihydrate ($CaSO_4 \cdot 2H_2O$) can be blended with FA and GGBS to make a ternary geopolymer to improve its mechanical properties. The literature review proved that the addition of DG produces a network structure consisting of fibrous ettringite crystals [15]. The sulfate ions enhance the leaching of alumina producing ettringite that can fill pores inside aluminosilicate gel and give rise to the more compact and denser matrix. This dual behavior of gypsum proved it a useful material not only to enhance the strength of geopolymer material but also to decrease the volume of solid waste [16]. The Al-O and Si-O bonds broke and C-A-S-H gel formed as a result of hydration. Previous studies proved that an alkaline medium transformed four coordinated Aluminum (Al) to $[Al(OH)_6]^{-3}$. At the same time, $Ca^{+2}$ and $SO_4^{2-}$ combine with $[Al(OH)_6]^{-3}$ producing ettringite that will accelerate the formation of further ettringite and improve strength [17].

The equation for the formation of ettringite is as follows [17]:

$$AlO_2^{\,-} + OH^- + H_2O \rightarrow [Al(OH)_6\,]^{3-} \tag{1}$$

$$2[Al(OH)_6\,]^{3-} + 6Ca^{2+} + +3SO_4^{2-} + 26H_2O \rightarrow Ca_6Al_2(SO_4)_3(OH)_{12} \cdot 26H_2O \text{ (AFt)} \tag{2}$$

Scanning electron microscopy (SEM) and X-ray diffraction also revealed the presence of needle-like ettringite (AFt) crystals in the matrix that fill the pores reducing porosity and increasing the density of the matrix. In addition, AFt combines with 32 water molecules resulting in a beneficial expansion of volume that offsets the dry shrinkage of hybrid geopolymer with limited DG content. The optimized value for DG is 6% and the excessive amount will degrade the overall performance of the geopolymer. As logically anticipated, the increase in DG content led to higher production of AFt. The silicates and aluminosilicates wrapped the AFt on the surface through dihydroxylation, preventing its further polycondensation that inhibits the formation of a three-dimensional geopolymer network [18]. Thus, it is proved that the use of DG as an additive to geopolymer is a viable and promising way to boost the engineering properties of geopolymer as well as the effective utilization of DG.

Geopolymers reliance on highly alkaline activators and the need for post-heat treatment poses significant challenges, including safety risks during handling, increased production costs, and environmental concerns due to their energy-intensive manufacturing processes. By incorporating DG into geopolymer formulations it can reduce the dependence on alkaline activators and eliminate the need for post heat treatment. Several research gaps need to be addressed, specifically, limited studies exist on the synergistic reactions between gypsum and geopolymer precursors and their impact on the mechanical and durability characteristics. Additionally, the optimal proportions of gypsum to precursors remain unclear, particularly in maintaining mechanical strength and controlling setting time. Further quantitative research is needed to determine how effectively gypsum can reduce the dosage of alkaline activators without compromising performance. Addressing these gaps can pave the way for eco-friendly and more cost-effective geopolymer products in construction.

## 2. Methodology

The materials used in this study were GGBS, PFA, DG, fine aggregate, water, and alkali activator of $Na_2SiO_3$, NaOH. The physical properties and chemical properties of the materials will be explained in this section.

The raw materials GGBS and PFA used in this study followed the standards specified in MS EN 15167-1 and BS EN 450, respectively. The GGBS contained 45.83% of CaO and 32.52% of $SiO_2$, and an $Al_2O_3$ content of 13.71%. The specific gravity of GGBS is 2.9g/cm³ and 0.50mm soundness. Table 1 includes the physical properties of GGBS.

**Table 1**
Physical properties of GGBS

| Testing Parameter | UOM | MS EN 15167-1-2010 Specifications | Test Method | Test Results |
|---|---|---|---|---|
| Fineness | $m^2/kg$ | ≥275 | MS EN 196-6:2007 | 425 |
| Soundness | mm | - | MS EN 196-3:2007 | 0.50 |
| Initial Setting | | | | |
| Initial | mins | - | MS EN 196-3:2007 | 223 |
| GGBS/test cement | - | ≤2 | | 1.31 |
| Compressive Strength | | | | |
| 7 days | MPa | - | MS EN 196-1:2007 | 26.91 |
| 28 days | MPa | - | | 46.26 |
| Ref. Cement Type CEM1 | | | | |
| 7 days | MPa | - | | 41.72 |
| 28 days | MPa | 42.5MPa 28days 62.5 MPa | | 51.38 |
| Activity Index | | | | |
| 7 days | % | ≥45 | Calculated | 64.54 |
| 28 days | % | ≥70 | | 90.06 |
| Moisture Content | % | ≤1.0 | MS EN 15167-1:2010 | 0.15 |

The pulverized fly ash (PFA) of class F (CaO > 10 %) was a grey color powder, with a specific surface area of 341m²/kg and a specific gravity of 2.288 g/cm$^3$. The PFA was composed primarily of 54.72% silicon dioxide and 27.28% aluminum oxide. The physical properties of PFA are represented in Table 2.

**Table 2**
Physical and Chemical properties of PFA

| Item | Unit | Result | MS EN 450-1 |
|---|---|---|---|
| Loss on Ignition, LOI | % | 2.79 | 5.0 Max. |
| Chloride | % | 0.065 | 0.1 Max. |
| Sulphur Trioxide, $SO_3$ | % | 1.03 | 3.0 Max. |
| Free CaO | % | 0.64 | 1.5 Max. |
| Calcium Oxide, CaO | % | 19.55 | 10.0 Max. |
| Silicon Dioxide, $SiO_2$ | % | 49.81 | 25 Max. |
| $SiO_2$ + $Fe_2O_3$ + $AI_2O_3$ | % | 73.65 | 70 Max. |
| Total Alkalies ($Na_2O_{eq}$) | % | 1.11 | 5.0 Max. |
| Magnesium Oxide, MgO | % | 2.60 | 4.0 Max. |
| Fineness by Residue at 45µm | % | 19.90 | 40.0 Max. |

The DG is a greyish-yellow color powder, and the main composition of DG is calcium sulfate hemihydrate ($CaSO_4 \cdot ½H_2O$).

Alkaline activators play a crucial role in the synthesis of geopolymer. They cause the dissolution of aluminosilicates and initiate the geopolymerization process. The hydroxides of Sodium and Potassium or a mixture of both [19] or silicates and carbonates of Sodium and Potassium can be used as alkaline activators [20]. The alkali activator solution used in this study is the combination of $Na_2SiO_3$ and NaOH from LST Trading. The concentration of NaOH was fixed at 12 M with the constant $Na_2SiO_3$/NaOH ratio of 1.2 and AA/B ratio at 11 %. The alkaline solution was prepared one day before the production of geopolymer mortar.

The fine aggregates used in this research conform to the BSI specification standard in BS EN 932-1. The bulk density of fine aggregates under saturated surface dry conditions is between 17 and 25 g/cm$^3$, with size normally < 4.75mm and a specific gravity of 2.70.

The mixture design aimed to study the mechanical properties and workability of geopolymer mortar with various compositions of GGBS, PFA, and DG when subjected to ambient curing. In the study, six categories of mix design were prepared where P represents the percentage of PFA, G represents the percentage of GGBS, and Y represents the percentage of DG with a sodium silicate/sodium hydroxide (SS/SH) ratio of 1.2, and alkaline activator/binder (AA/B) ratio 0.35. The ratio of fine aggregate/binder (FA/B) was kept at 2.25.

For accessing the mechanical properties and workability of geopolymer 50mm x 50mm cube molds were prepared. First of all, fine aggregate and binders such as GGBS, PFA, and DG were weighted precisely to ensure the accuracy of the mixing proportion. The dry ingredients were mixed with an electric hand cement blender for 3 minutes. The alkaline mixture and water were poured and mixed to prepare fresh mortar. The mortar was then placed in molds and kept at room temperature for hardening. The specimens were demolded after one day and cured with a moist curing method. The specimens were wrapped in cling wrap and kept at room temperature for curing. Figure 1 represents the specimens demolded, wrapped, and placed at room temperature for moist curing.

**Fig. 1.** Casted ternary blended geopolymer specimen

The composition of all the mix designs of geopolymer composites is given in Table 3.

**Table 3**
Mixture proportion of ternary hybrid geopolymer mortar composites

| Mix designation | PFA (kg/m3) | GGBS (kg/m3) | Gypsum (kg/m3) | Sand (kg/m3) | Water (kg/m3) | SS (kg/m3) | SH (kg/m3) |
|---|---|---|---|---|---|---|---|
| P60G40Y0 | 306 | 204 | 0 | 1147 | 280 | 97 | 82 |
| P55G35Y10 | 284 | 181 | 52 | 1161 | 284 | 85 | 70 |
| P50G30Y20 | 261 | 157 | 104 | 1175 | 287 | 71 | 60 |
| P45G25Y30 | 237 | 131 | 158 | 1183 | 289 | 63 | 52 |
| P40G20Y40 | 214 | 107 | 214 | 1205 | 295 | 44 | 36 |

## 3. Results

### *3.1 Flow Table Test*

The flow table test is the standard test method to determine the workability of the geopolymer mortar. The workability of ternary blended geopolymer with the combination of GGBS, PFA, and DG was determined by utilizing the standardized flow table apparatus according to ASTM C230. In this experiment, the fresh mix mortar of every mix designation will spread over a distance after the vibration. The spread diameter of every mix designation will be measured and recorded whereas the optimum spread diameter is 150mm. In the experiment six mix designations with different contents of PFA, GGBS, and DG show their flowability of fresh mix mortar by measuring their spread diameter in millimeters (mm). Figure 2 shows a graphical representation of the spread diameter of the six mix designations.

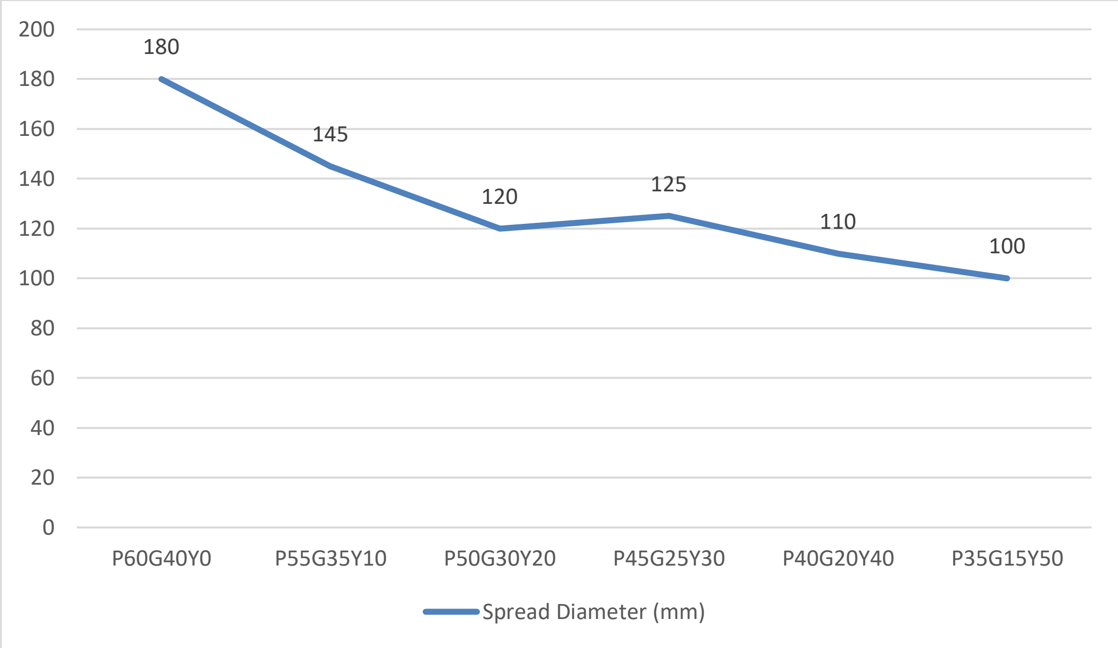


**Fig. 2.** Flow table test

It can be seen clearly that P55G35Y10 is the optimum mix designation among the 6 mix designations as its spread diameter was 145mm, which is close to the most optimum spread diameter, 150mm. P50G30Y20, P45G25Y30, P40G15Y40, and P35G15Y50 were less workable compared to P55G35Y10 as they have moderate workability with spread diameters of 120mm, 125mm, 110mm, and 100mm respectively. However, P45G25Y30 possessed more workability compared to P50G30Y20 as it has a 5mm wider spread diameter. It means that a widespread diameter indicates higher workability while a small spread diameter indicates less workability. The apparatus used for the flow table test is shown in Figure 3.

**Fig. 3.** Flow table apparatus

Zhaofeng *et al.,* [21] conducted research in 2020, indicating that the spread diameter of geopolymer mortar decreases upon the addition of gypsum into the composites. The results conclude that increasing the content of gypsum in the specimen reduces the spread diameter of fresh mix mortar. This is because gypsum is monoclinic with a hexagonal plate-like structure, thus the increase in gypsum content in a medium with water will reduce its fluidity.

### *3.2 Compression Strength Test*

The compressive strength test was conducted to determine the strength of ternary geopolymer mortar comprising GGBS, PFA, DG, and alkaline activator solution. The curing condition, binder content, and alkaline activator content all influence the compression strength of the blended geopolymer. For the compression strength test, six cube mortars with dimensions of 50mm x 50mm x 50mm were cast and cured in 7 and 28 days under ambient conditions. The maximum forces that the cube mortar can withstand were recorded in megapascal (MPa) and the average compressive strength was calculated for each designation. A graphical representation of the compressive strength of mortars is shown in Figure 4. In every mix designation, it is shown that the 28 days compressive strength is higher than 7 days compressive strength as the extending curing time and ambient temperature curing method can help achieve the expected strength. The addition of gypsum showed a significant increase in compressive strength as it has been reported that needle-like ettringite crystals fill the pores, reducing porosity and improving the mechanical properties of geopolymer. The $SO_4^{2-}$ ions in DG activate aluminate in Fly ash, increasing Al ions dissolution, intensifying polymerization, and improving strength [22]. It is clearly shown that the P55G35Y10 has the highest average compressive strength in both 7 days and 28 days of curing and compressive strength decreases upon further addition of DG. It may be because increased Aft crystals may cause volume expansion and destroy the gel structure thus resulting in a negative effect on strength improvement [23].

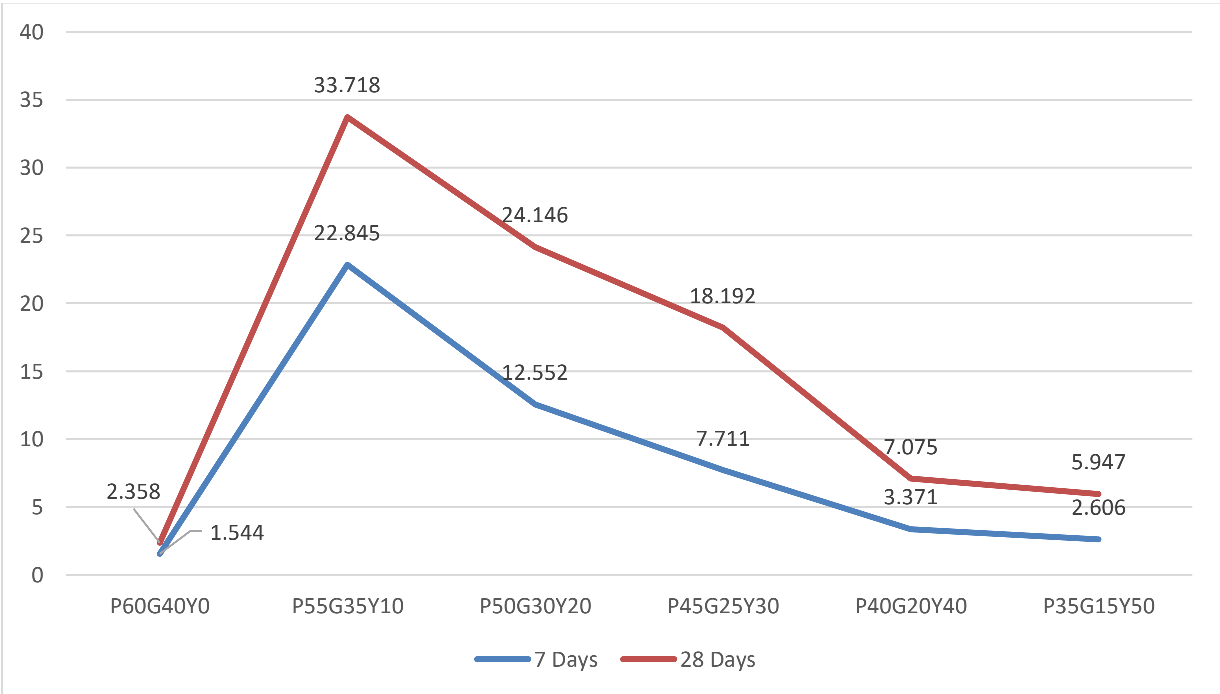


**Fig. 4.** Compression strength test

## 4. Conclusions

The above research investigated the effect of the addition of DG into the mix design of GGBS and PFA on the mechanical properties such as workability and compression strength of ternary blended geopolymer. The following conclusions were drawn from this investigation.

i) The higher the DG content will improve the workability and mechanical properties of the geopolymer mortar up to a certain limit after this strength reduction occurs.
ii) The incorporation of 10% of DG into geopolymer mortar containing GGBS and PFA has the optimum spread diameter, 145mm, more workable compared to geopolymer mortar without DG.
iii) The addition of 10% of DG has the greatest compression strength of 22.845Mpa, and 33.718Mpa, in 7 days and 28 days curing period.

In conclusion, mixture P55G35Y10 has the potential to be studied further in combination with various kinds of industrial waste ash to improve their mechanical and durability characteristics. In other words, utilizing industrial by-products such as GGBS, PFA, and DG in the production of sustainable building materials resulted in advancement in geopolymer technology.

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