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Experimental study of the effect of replacing fly ash with glass powder on the mechanical properties of geopolymer mortar

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ABSTRACT Geopolymers are eco-friendly materials emerging recently in the construction industry. These alternative binders are formed from by-products rich in silica and alumina, such as fly ash, slag, or metakaolin, mixed with an activating solution known as the alkaline solution. For this research, fly ash from coal combustion was used with an activating solution composed of sodium hydroxide (NaOH) and sodium silicate (Na₂SiO₃) to produce geopolymer mortar. However, the gradual closure of coal-fired power plants will limit the availability of associated fly ash (FA). Therefore, it is necessary to identify alternative materials that can be used as a source of silica and alumina in geopolymers. In this study, FA was thus partially replaced by glass powder (GP), which also exhibits pozzolanic properties, in order to test its effect on the mechanical properties of GPM. Samples with different percentages of GP (0%, 5%, 10%, and 20% by weight) were tested to determine their impact on the compressive strength of GPM. The effect of the molarity of the alkaline NaOH solution on the compressive strength of GPM was also studied; two molarities were tested (10M and 14M) for this purpose. The results showed that the substitution of 5% of FA by GP in GPM with 14M as the concentration of NaOH gives a compressive strength of 48.3 MPa, very close to that of the GPM without GP which gives 47.7 MPa at 90 days. Similarly, 10% substitution of FA with GP also has no major effect on the compressive strength. On the other hand, a higher molarity of NaOH solution showed a higher compressive strength.

Keywords geopolymer mortar, fly ash, glass powder, alkaline solution, compressive strength

I. INTRODUCTION

The population is growing, and so is the need for concrete that serves several utilities such as shelter, transportation, and water management. Ordinary Portland cement (OPC) is the material used traditionally to create concrete. The production of OPC involves the burning of raw materials, such as limestone and coal, at a very elevated temperature of approximately 1500°C. Therefore, the manufacturing process of OPC is a principal source of CO₂ emission; it is almost responsible for 5-7% of the global total CO₂ dissipation. The fabrication of one ton of OPC liberates between 0.73 and 0.85 tons of CO₂ in the atmosphere (He et al., 2019). Hence, alternative construction materials are grabbing the attention of researchers to provide a sustainable replacement for traditional concrete for some applications. Geopolymer concrete (GPC) is an

environmentally friendly alkali-activated material with the potential to replace or supplement OPC. The term “geopolymer” was first created and applied by the French scientist Joseph Davidovits in 1979 to refer to aluminosilicate binders (Davidovits, 2002). Geopolymers are inorganic mineral polymer materials that can be formed as the result of a chemical reaction (known as the geopolymerization reaction) between natural materials or waste products as source materials and an alkaline activating solution (Cong & Cheng, 2021). The reaction between the precursor and the activator is known as the geopolymerization reaction. This reaction is described as the dissolution of the aluminosilicate materials (source materials) in the alkaline solution (mostly formed by a combination of sodium hydroxide and sodium silicate) to form as final product a three-dimensional aluminosilicate network structure.

The source material (or precursor) can be a natural material such as clay and kaolinite or a waste product such as fly ash, blast furnace slag, rice- husk ash and silica fume (Shehata et al., 2021; Cong & Cheng, 2021). For instance, any material rich in alumina and silica can be used as a precursor for geopolymer since alumina and silica are the two components responsible for the strength development of geopolymers.

Fly ash (FA) is an industrial by-product that results from the burning of coal in power plants (American Coal Ash Association, 2013). The characteristics of fly ash depend on the composition of the burned coal, the accompanying burning conditions and the fineness of its particles (Strydom & Swanepoel, 2002). Fly ash consists mainly of SiO_2 , Al_2O_3 , Fe_2O_3 and CaO which ensure its pozzolanic properties and makes of it a good cementitious material. Also, it contains magnesium, potassium, sodium, titanium, and sulfur in smaller quantities. Fly ash already proved its potential as a precursor material for geopolymer concrete (Ivan et al., 2011; Strydom & Swanepoel, 2002). Nonetheless, the availability of fly ash nowadays is becoming limited because of shutting down coal power plants and shifting towards renewable energy. Hence, promoting the use of other waste products in geopolymers is necessary.

Glass powder (GP) is another waste product that shows pozzolanic properties as well. Their disposal in landfills is not favorable because they are not biodegradable; so, their use in geopolymers is valued. Glass is fabricated by melting several materials such as silica, soda ash and calcium carbonate at high temperature pursued by cooling during which solidification occurs (Patil, 2013). Hence, GP consists mainly of SiO_2 and CaO . It contains MgO , Al_2O_3 , and Fe_2O_3 too but in lower quantities.

Toniolo et al. (2017) investigated the possibility of replacing FA by GP in GPC without affecting its strength. They showed that GP can substitute FA in GPC; however, a higher percentage of GP induced a lower compressive strength. After testing FA based geopolymers activated with a combination of NaOH and Na_2SiO_3 , Sasindran C & Jose (2017) proved that up to 15% of substitution of FA by GP causes an increase in the compressive strength of 11.8% at 28 days. Even though, a larger percentage of GP causes an improvement in the strength compared to samples without GP but not as important as the strength improvement obtained with 15% of GP. Hence, in their study, the optimum percentage of GP found was 15%. Moreover, after studying different percentages of GP between 10 and 40% and using the same activating solution, Tho-in et al. (2018) found that the optimum percentage of GP substitution is between 10 and 20%.

Accordingly, the optimum percentage of GP as partial substitution of FA in geopolymer mortar (GPM) is investigated experimentally in this research by testing different percentages of GP (0, 5, 10 and 20%).

On the other hand, the alkaline activators play a crucial role in the production of geopolymers. They are responsible for the dissolution of alumina and silica coming from the precursor and initializing the geopolymerization reaction. The most common activators used are sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) or potassium hydroxide (KOH) and potassium silicate (K_2SiO_3) (Cong & Cheng, 2021). Sodium-based activators are used most often in the literature. The molarity of the NaOH solution has a crucial influence on the compressive strength of the hardened geopolymer concrete. Hardjito et al. (2008) tested the effect of different molarities of the NaOH solution ranging between 8 and 16M on the compressive strength of GPM. They stated that the compressive strength of geopolymer is proportional to the molarity of the NaOH solution. A higher molarity of the NaOH solution increases the dissolution process as well as the geopolymerization reaction leading to a higher strength.

However, those results contradict the results obtained by Mustafa et al. (2011) for fly ash-based geopolymer paste activated with a combination of NaOH and Na_2SiO_3 and cured at a very high temperature for 1 day and then kept at room temperature until testing. In this case, they showed that between different molarities (6, 8, 10, 12, 14 and 16M), 12M gave the highest compressive strength and a higher molarity caused a decrease in the compressive strength.

A study conducted by Abdullah et al. (2021) for different molarities of the NaOH solution ranging between 6 and 14M showed that the compressive strength increases when increasing the molarity from 6 to 12M and then it decreases. The analysis by scanning electron microscopy (SEM) showed that the smallest proportion of unreacted FA was shown in the geopolymer with a NaOH molarity of 12M which explains the highest compressive strength. Besides, geopolymers formed with NaOH solution of molarities 6, 8, 10 and 14M showed large pores and cracks in their SEM analysis which explains the lower compressive strength.

The literature review highlights the diversity of the results obtained regarding the influence of the molarity of the NaOH solution on the compressive strength of GPC and the scarcity of the physical explanation behind this variousness. In this study, a combination of NaOH and Na_2SiO_3 are used as the activating solution. Different concentrations of the NaOH solution (10 and 14M) are investigated experimentally and their impact on the mechanical strength of GPM will be assessed.

II. EXPERIMENTAL MATERIALS AND METHODS

A. Materials

In this study, low calcium fly ash (type F) was used to create geopolymer mortar. Glass powder (GP) was added as a partial substitution of FA. The chemical composition of FA and GP are given in Table 1. Standard sand (certified according to EN 196-1) having a maximum moisture content of 0.2% was added as fine aggregates to the mix. Its specific grain size distribution ranges between 0.08 and 2 mm. For the activation of the source materials, a sodium-based activator was

used formed by combining NaOH and Na₂SiO₃. NaOH was purchased in the form of pellets with a purity of 99%. NaOH pellets were dissolved in distilled water to form a solution with the adequate molarity needed. The sodium silicate Na₂SiO₃ is already found in liquid form and has a chemical composition of 7.5-8.5% of Na₂O and 25.5- 28.5% of SiO₂ by mass.

TABLE 1. Chemical composition (mass %)

<i>Component</i>	<i>Fly ash</i>	<i>Glass powder</i>
SiO ₂	48- 52	69- 74
Fe ₂ O ₃	7- 9	0- 1
Al ₂ O ₃	27- 31	0- 3
CaO	0- 3	5- 14
MgO	2.5	0- 6
K ₂ O	2- 5	-
Na ₂ O	0.5- 1	10- 16

B. Alkaline liquid preparation

In order to investigate the effect of the molarity of the NaOH solution on the mechanical properties of GPM, two different solutions of sodium hydroxide were prepared, each with different molarity (10M and 14M). For the solution with a molarity of 10M, 400g of NaOH pellets were dissolved in 1l of distilled water. For the solution with a molarity of 14M, 560g of NaOH pellets were dissolved in 1l of distilled water. Since the dissolution of NaOH in distilled water is an exothermic reaction, the solution should be left for 24 hours at rest before being used. Then, each NaOH solution was mixed with the sodium silicate with a ratio of NaOH/ Na₂SiO₃ = 1:4 to create two different types of the alkaline solution.

C. Geopolymer mortar preparation

To prepare the geopolymer mortar, an alkaline liquid to precursor ratio of 0.51 and a sand to precursor ratio of 2 were fixed.

GP was used as a partial substitution of FA with percentages of 0, 5, 10, and 20% (by weight) to observe the possibility of using GP in geopolymers. Thus, for each type of the alkaline solution, four different types of mixes were performed. Table 2. shows the mix design proportions for each mix.

Dry components (FA, GP, and sand) were mixed first in a mixer for 30 seconds at low speed followed by 30 seconds of mixing at high speed. Then, the alkaline solution was added, and the combination was mixed for further 60 seconds at high speed.

TABLE 2. Mix design proportions

Mix notation	Sand/ precursor	Alkaline liquid/ precursor	NaOH/ Na ₂ SiO ₃	NaOH molarity	% of FA	% of GP
M10-0	2	0.51	0.25	10	100	0
M10-5	2	0.51	0.25	10	95	5
M10-10	2	0.51	0.25	10	90	10
M10-20	2	0.51	0.25	10	80	20
M14-0	2	0.51	0.25	14	100	0
M14-5	2	0.51	0.25	14	95	5
M14-10	2	0.51	0.25	14	90	10
M14-20	2	0.51	0.25	14	80	20

D. Samples preparation

After mixing, the geopolymer mortar was cast in prisms of 4x4x16 cm in two layers. After pouring each layer, molds were vibrated on a jolting table for 60 cycles. It was observed that GPM reacted very slowly to start developing strength at ambient temperature, therefore, prisms were left at rest in humid conditions for 7 days before demolding. Then, geopolymer samples were kept at room temperature for curing until the day of testing.

E. Testing

Geopolymer prisms were tested for compressive strength using the UTEST Material Testing Equipment after a curing time of 28 and 90 days to represent respectively the short and long term mechanical behavior of GPM. No testing at earlier stages was performed because of the fact that FA-based GPM gained strength very slowly at room temperature and earlier stages were expected to show very low strength development. The rate of loading applied is 0.5 kN/s. Each test was performed on three samples prepared in similar conditions to verify the reproducibility of the experiment.

III. EXPERIMENTAL RESULTS AND DISCUSSION

A. Effect of NaOH molarity on the compressive strength of fly-ash based Geopolymer mortar

First, the impact of the molarities of the alkaline solution on the compressive strength development of pure fly ash-based geopolymers (i.e. without any substitution with GP) is investigated. Figure 1 shows the evolution of the average compressive strength (error bars provide minimal and maximal strength observed experimentally) of fly ash based geopolymer with different molarities (10M and 14M) of the alkaline solution over time. First, Figure 1 highlights the very good reproducibility of the experimental results. Overall, a higher molarity of the NaOH solution gives higher compressive strength in both the short and long term. Fly ash-based geopolymer with NaOH molarity of 10M (M10-0) reached a compressive strength of 20.4

MPa at 28 days while samples with a NaOH molarity of 14M (M14-0) reached a compressive strength of 25.4 MPa. On the other hand, M10-0 and M14-0 samples reached a compressive strength of 36.3 MPa and 47.7 MPa respectively at 90 days. Thus, increasing the molarity of the NaOH solution from 10M to 14M increased the compressive strength by around 20% in the short term and around 25% in the long term. In fact, those results are in conformity with the results obtained by Hardjito et al. (2008) who tested the effect of different molarities of the NaOH solution ranging between 8 and 16M on the compressive strength of GPM. They stated that the compressive strength of geopolymer is proportional to the molarity of the NaOH solution. This can be explained by the presence of a higher amount of OH⁻ ions and hence induce a better dissolution reaction leading to an improvement in the strength.

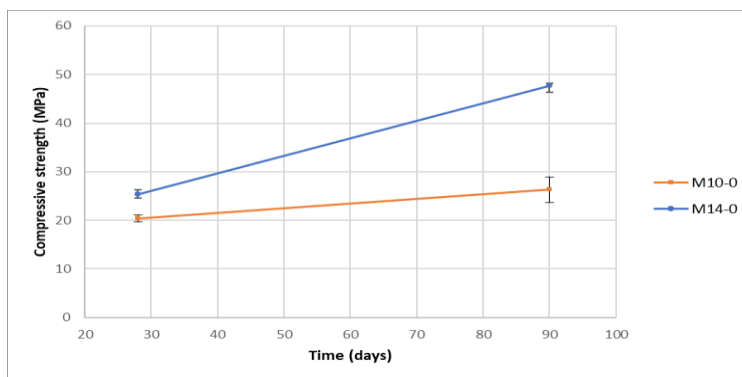


FIGURE 1. Effect of the NaOH molarity on the average compressive strength of FA-based geopolymer

B. Effect of FA substitution with GP on the compressive strength of Geopolymer mortar

Then the impact of a FA substitution with GP on the mechanical strength is investigated. Figure 2 shows the average compressive strength of both M10 and M14 samples with different percentages of GP in the short and long term (error bars provide the minimum and maximum experimental values obtained experimentally). Results showed first that M14 provides again the highest compressive strength whatever is the rate of substitution of FA by GP. In addition, up to 10% of substitution of FA by GP might be an option without having to compromise the strength of the GPM. In fact, a 5% substitution of FA by GP increased the compressive strength of FA-based GPM between 10 and 15% compared to samples without GP at both ages. For samples with 10% of GP, the compressive strength is almost equal to the compressive strength of samples without GP (the difference is less than 4%). For samples with 20% of GP, the compressive strength decreased around 6% for M10 samples and 10% for M14 samples compared to samples without GP at long term. Although, for the same samples, the decrease in strength is more important at short term. A strength reduction of around 25% occurred with the addition of 20% of GP at 28 days.

In other words, the optimum percentage of GP to be added to a FA-based GPM is 5% since it causes an increase in the compressive strength of the mix. Also, up to 10% of GP substitution can be considered without largely affecting the compressive strength of the GPM.

Results obtained by Sasindran C & Jose (2017) and Tho-in et al. (2018) acclaim those findings; although in their studies the optimum percentage of GP as partial substitution of FA was found to be 15%. A possible physical explanation of those experimental observations was proposed by Sasindran C & Jose (2017). They explained that the increase in the compressive strength is due to the very fine structure of GP that allows them to fill the pores in the geopolymer mix and provides hence a denser structure. The microstructure of the geopolymer studied by Tho-in et al. (2018) and Sethi et al. (2019), using scanning electron microscopy (SEM) and energy dispersive X-ray spectrometry (EDS), proved this statement. Decreasing the amount of FA and increasing the amount of GP reduces the amount of alumina in the mix causing the decrease in strength with higher percentages of GP. The smaller amount of alumina in the mix produces smaller amounts of NASH hydrates responsible for the strength development of geopolymers (Sethi et al., 2019).

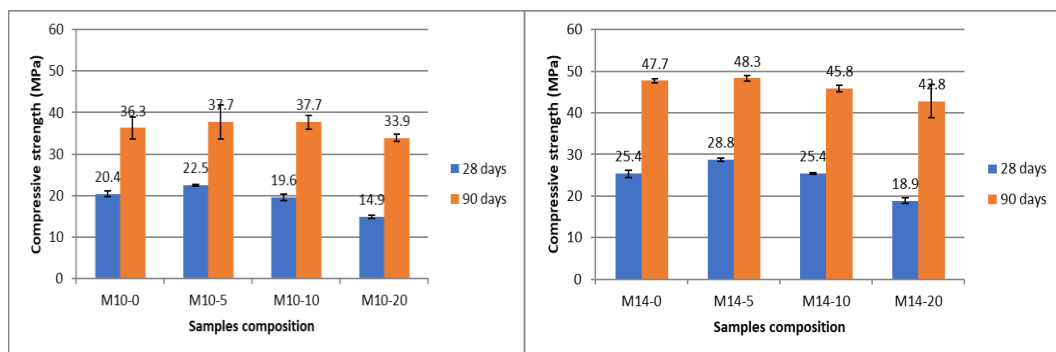


FIGURE 2. Effect of FA substitution by GP on the average compressive strength of geopolymer for samples with (a) 10M as concentration of NaOH (b) 14M as concentration of NaOH

IV. CONCLUSION

The purpose of this study is to investigate the effect of the molarity of the NaOH solution and the substitution of FA by GP on the mechanical properties of GPM. To serve those objectives, an experimental study was performed on GPM samples with two different molarities of the NaOH solution (10M and 14M) and with different percentages of GP (0, 5, 10, and 20% by weight of FA). Results showed that:

- Increasing the molarity of the NaOH solution from 10M to 14M increased the compressive strength of GPM around 20% at 28 days and 25% at 90 days. However, a trade-off between the required strength and the cost of the GPM should be considered here because the NaOH is the most expensive component in the GPM.
- A 5% substitution of FA by GP caused an increase in strength between 10 and 15% compared to 100% FA geopolymer.
- Up to 10% substitution of FA by GP is possible without major influence on the compressive strength of FA based geopolymer.

To sum up, this research, in agreement with other experimental results and microstructural observations, proved that GP can replace FA in GPM up to a certain percentage without affecting the mechanical performance of the GPM. Although, the optimum percentage of GP as partial substitution of FA should be more investigated on both macro and microstructural levels.

Alternatively, a higher molarity proved to give higher compressive strength. While some studies suggest that the optimum molarity for FA-based geopolymer is 12M (Abdullah et al., 2021; Mustafa et al., 2011), further microstructural investigations are needed to explain the reduction in strength at higher molarities.

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