



# Laboratory investigation of the effect of using metakaolin and clay on the behaviour of recycled glass powder-based geopolymer mortars

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## ABSTRACT

Civil infrastructure relies on cement, a common building material. However, Portland cement production pollutes the world by consuming mineral resources and emitting carbon dioxide. Using modern, cost-effective, and eco-friendly cement materials seems necessary. One such material is geopolymer cement, also known as green cement, composed of aluminosilicate base materials and alkaline-silicate activators. In the present study, waste glass powder was utilised to provide a rich aluminosilicate base material by substituting 10%, 20%, and 30% of the metakaolin or clay with calcium carbide residue as an alkaline activator. The samples were processed under two distinct temperature conditions: natural environment and thermal processing at 80 °C in an autoclave. The mechanical and microstructural properties of the mortars were evaluated regarding compressive strength, bending strength, setting time, volume control, and Scanning Electron Microscope (SEM). The results indicate that the type and amount of glass powder alternatives significantly determine the final sample strength. Substituting 10% metakaolin and 20% clay for glass powder maximises compressive strength. The optimal bending strength of geopolymer samples was obtained by substituting 10% clay with 10% metakaolin. SEM images validate this discovery by exhibiting a more compact and cohesive arrangement. Additionally, the processing conditions of geopolymer samples significantly influence the mechanical properties of geopolymer mortars. The results of the V-cut test indicate that geopolymeric mortars are sluggish and require additional time to reach their primary and secondary strength. This study suggests that geopolymers could produce environmentally friendly concrete by reducing cement use and advancing economic and environmental goals.

## 1. Introduction

Climate change resulting from global warming has become one of the world's most pressing environmental issues. Greenhouse gas emission is the primary cause of global warming. Carbon dioxide (CO<sub>2</sub>), with an emission rate of 65%, plays the most significant role in global warming among greenhouse gases [1,2]. The production of ordinary Portland cement (PC) is one of the sources of greenhouse gas emissions and global warming [3–5]. The production of one ton of Portland cement produces one ton of carbon dioxide [6,7], responsible for 7–10% of global carbon dioxide emissions [3,8]. Furthermore, concrete has a lower energy efficiency and requires a shift in energy inputs from fossil fuels compared to other construction materials. When considering the environment, it is found that the manufacturing of PCCs leads to respiratory diseases in the

cities of these factories [8,9]. Also, when exposed to harsh environments, PCs deteriorate faster than expected service life [10,11]. Therefore, it appears necessary to use an alternative to Portland cement. Geopolymers have recently been proposed as a more environmentally friendly alternative to PC to mitigate the associated environmental impacts [12]. Geopolymers were first introduced in 1771 by David Ovitz [13] as a new binder from the inorganic polymer family. David Ovits suggested naming geopolymers poly (sialate), a polymer prefix and an abbreviation of the silicon-oxo-aluminate chain [14–16].

Geopolymers are a type of inorganic aluminosilicate material obtained through the combination of a geopolymer base material rich in silica (SiO<sub>2</sub>) and alumina (Al<sub>2</sub>O<sub>3</sub>), which may include fly ash, slag, or other industrial by-products [17–19], and an alkaline activating solution [20,21]. Geopolymerization is a rapid chemical reaction between Si and

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Al minerals in alkaline conditions, forming Si–O–Al–O three-dimensional polymer chains [15]. The general formula describing the chemical structure of geopolymer is  $Mn[-(SiO_2)_z-Al_2O_3]_n \cdot wH_2O$ . Here,  $z$  represents the number of aluminate substitutions,  $n$  represents the degree of geopolymerization,  $Mn$  represents the cation, including sodium and potassium, and  $w$  represents the amount of water [22,23]. Geopolymer base material must be amorphous to perform the chemical reaction; the greater the amount of  $SiO_2+Al_2O_3+Fe_2O_3$ , the greater the pozzolanic activity. The smaller the pozzolan particles, the greater the specific surface area and, thus, the faster the reaction rate. In comparison to conventional concrete, geopolymers have superior mechanical and chemical properties, including superior compressive, bending, and tensile strengths [24–26], fire resistance and high temperature [27,28], low permeability and resistance to salt and acid [24] and low creep [29].

The greatest advantage of geopolymer-based building materials is that they are cheaper and more abundant than conventional cement [30, 31]. Another of the main advantages of geopolymers is that they have a very high compressive strength, which means they can withstand much pressure without breaking. The main difference between geopolymers and Portland cement is that the former uses a polymerisation reaction to set rather than hydration. Geopolymers exhibit a significantly faster setting time than traditional cement, leading to a relatively quick process completion [32,33]. This feature makes them particularly suitable for construction applications where time constraints are considered, such as constructing and repairing road surfaces, highways, and airport runways. Also, geopolymers have higher corrosion resistance than traditional Portland cement, and they can be used to make concrete with equal or even higher strength than cement concrete. These properties make geopolymer a suitable alternative to Portland cement in many construction applications [34,35].

As mentioned above, the alkaline activating solution is an important component of geopolymers used in construction materials. It typically contains sodium or potassium hydroxide with sodium or potassium silicate [24]. Calcium carbide ( $CaC_2$ ) is a chemical compound that, when reacted with water, produces acetylene and calcium hydroxide. Calcium Carbide Residue (CCR) is a by-product of the industrial production of acetylene gas and is rich in calcium hydroxide  $Ca(OH)_2$ .

Numerous studies have explored the factors influencing geopolymer concrete's compressive strength (fc). These factors include the type of alkaline activating solution, the composition and percentage of geopolymers, the concentration of the alkaline solution, the water content, particle size, curing conditions, and others [36–40]. Metakaolin is a dehydroxylated form of the clay mineral kaolinite, widely used in ceramics and as a substitute for concrete [41]. Its utilisation has resulted in reduced pollution and decreased energy consumption. Metakaolin, as class N pozzolan considering ASTM classification [42], undergoes a pozzolanic reaction and refines the microstructure of the hydrated cement paste when added to concrete. Due to the small particle size and high surface area, metakaolin reacts rapidly and decreases the diffusion coefficient compared to ordinary Portland cement [43,44]. Also, metakaolin controls the alkaline reactions of aggregates [45]. Metakaolin improves the mechanical strength of conventional concrete [46]. Coal metakaolin (CMK) is an amorphous material produced by calcining coal kaolin at a suitable temperature and has special physical properties such as small grain size and high surface energy [47]. Based on Liu et al.'s study [48], a red coal mud metakaolin geopolymer (RCG) with high mechanical strength was synthesised under ambient conditions. Studies have shown that metakaolin-based geopolymers show better mechanical and thermal properties [46,48]. Remember that curing is one of the parameters studied in this research influencing the compressive strength of geopolymer concretes, which consists of temperature and curing time.

Xie et al. [49] manufactured geopolymeric cement by mixing glass powder and metakaolin. 3%, 5%, and 8% metakaolin were replaced. In addition, an alkaline solution of this type of sodium hydroxide cement was produced with molarities of 5–8–10 and ages of 7–28 and 91 days. At all ages, the compressive strength of samples containing 8% metakaolin

was higher than that of samples containing other replacements. In terms of compressive strength, this amount of replacement was comparable to the control sample, which was comprised entirely of glass powder.

Borciaga Diaz et al. [50] investigated the percentages of replacing metakaolin with glass powder. They created a reference sample of 100% metakaolin and samples containing 15% and 30% glass powder in place of metakaolin. In addition, they considered the ratio of 2–3 times mortar and prepared samples at ages 1, 3, 7, 14, 28, and 90 days. This study utilised 12% and 16% sodium oxide ( $Na_2O$ ) as alkaline solvents. According to the results, samples with 12% sodium oxide had greater compressive strength than samples with 16% sodium oxide, while control samples with 100% metakaolin had greater compressive strength. In contrast, the samples containing 16% sodium oxide had a lower compressive strength than those containing 30% glass powder replacement at all ages and proportions. By increasing the percentage of sodium oxide and the ratio of 0.8 alkaline solvents, it is possible to achieve a high compressive strength at 90 days of age by increasing the amount of substitution with metakaolin to 30%. In addition, according to the two times ratio of aggregate to geopolymer mortar, all samples of varying ages, based on the amount of 12% substitution of sodium oxide with a ratio of 1, obtained a high compressive strength in comparison to the three times ratio of aggregate to geopolymer mortar.

Regarding the fly ash-based geopolymer, Palomo et al. [46] concluded from their research on the effect of curing on the compressive strength of fly ash-based geopolymer concrete that the curing temperature accelerates the geopolymerization process and, in conjunction with the curing time factor, has a direct impact on the mechanical strength of concrete. Higher temperature and curing time, according to these researchers, increased the compressive strength of the concrete. In a similar study, Van Jarsold et al. [16] determined that curing is one of the parameters affecting the geopolymer's compressive strength. These researchers found that curing at extremely high temperatures degrades the properties of concrete due to the formation of cracks. Finally, they recommended medium curing temperatures for geopolymeric concrete.

Hardjito et al. [24] examined the effect of this parameter on the compressive strength of fly ash-based geopolymer concrete in their study. In addition to the efficiency of this parameter, they determined that increasing the curing temperature increases the concrete's compressive strength. Still, the compressive strength of concrete does not change significantly at temperatures above 60 °C. In contrast, Manesh et al. [51] concluded that concrete compressive strength does not increase considerably at temperatures above 120 °C. Palomo and others [46] concluded that the compressive strength of geopolymer concrete increases as the curing time increases. Hardjito et al. [24] concluded that the compressive strength of geopolymeric concrete increases with a 24-h curing time. Still, there is no significant change in compressive strength after more than 24 h, while Memon et al. [52] concluded no substantial change in compressive strength after more than 24 h.

Jongsovathananon et al. [53] attribute the strength decrease with curing time to high molar ratios of  $Na_2O/Al_2O_3$  and  $Na_2O/SiO_2$  and the phase transformation from geopolymer to zeolite. Xie et al. [49] studied the slump, setting time, compressive strength, shrinkage, and Poisson's ratio of slag-metakaolin mixtures with recycled aggregate content. Zhang et al. [54] examined how concrete strength, moisture content, and temperature affect the high-temperature spalling potential for metakaolin-fly ash-based geopolymer mixtures.

Khan et al. [55] investigated the performance of fly ash geopolymers after exposure to 1.5% sulfuric acid solutions for 6, 12, and 24 months, respectively. Most of these tests were performed on metakaolin or fly ash-based geopolymers. The acid strength of the derived geopolymers changes with the participation of red mud (RM) in geopolymerization, so the strength of RCG against sulfuric acid should be clarified. The corrosion of geopolymers in sulfuric acid is a dynamic and time-varying process. In practical engineering applications, the continuous inspection of geopolymer components in corrosive environments has created

technical challenges due to the complexity of the traditional inspection process and damage to the components during sampling. Therefore, a new nondestructive testing method is urgently required.

Exploring and manufacturing geopolymers using inexpensive and readily available waste materials can positively impact the environment and the economy. By utilising materials such as glass powder and calcium carbide, it is possible to create geopolymeric cement. Notably, there is an abundant supply of calcium carbide residue and recycled glass powder in Iran and worldwide, with an annual increase of approximately 5.1%, as projected [56] which presents a promising opportunity for sustainable and cost-effective geopolymer production. Recycled glass powder (GP) alone is high in silica and low in alumina, which limits its use as a base material. The integration of glass powder with higher-alumina substances, such as metakaolin and clay, has been proposed to mitigate this limitation. While this technique is still developing, further research is necessary to optimise its utilisation in the production of geopolymeric cement. No geopolymeric mortar research has used these materials as a base material and alkaline solvent. This article aims to enhance the technical properties of geopolymeric concrete and promote environmental and sustainable development by comparing the impact of GP, CCR, and alkaline activators on geopolymeric mortar. This research examined compressive strength ( $f_c$ ), bending strength (BS), and durability characteristics in an autoclave, normal, setting time, and SEM microstructure images.

## 2. Materials and methods

### 2.1. Laboratory program

2.2 The impact of several parameters on the tensile strength of geopolymer specimens has been extensively studied. These parameters include 1) the quantity of clay/metakaolin, 2) the amount of sand, 3) temperature and curing time, 4) the type and quantity of alkaline solvent, and 5) curing conditions. Fig. A1 depicts the laboratory design for this study. Portland cement.

The PC utilised to produce the samples was type 2 Portland cement. The cement industry in Mashhad supplied it. Table 1 presents the results of an XRF chemical analysis of Portland cement particles.

#### 2.1.1. Waste glass powder

Glass waste recycling is one way we can help reduce pollution and reuse glass waste [57]. One of the most important ways to control glass waste is to reuse it in different industry sections [58]. Each year, 19 thousand tonnes of glass are discarded in Tehran, and sadly, only a small quantity of glass waste (approximately 2%) gets recycled [59,60]. The GP utilised in this investigation was acquired from a glass bead manufacturer in Arak City. The acquired glass powder is micronised, with particle sizes between 1 and 125  $\mu\text{m}$ . Therefore, the obtained glass powder was passed through sieve number 200, and its particles smaller

than 75  $\mu\text{m}$  were used as the basic material of the geopolymer. A laser instrument was used to determine the granulation dispersion of glass powder, as illustrated in Fig. A2. Table 1 also provides the chemical composition of the used glass powder.

#### 2.1.2. Calcium carbide residue

An alkaline environment is required for the geopolymerization procedure to be carried out. Due to its high alkalinity ( $\text{pH} < 12$ ), CCR can facilitate the breakdown of amorphous aluminosilicate particles and glass powder in soil [61]. The execution of the geopolymerization procedure necessitates the presence of a suitable alkaline environment. CCR, due to its elevated alkalinity level, can facilitate the dissolution of amorphous aluminosilicate particles in both soil and glass powder, thus providing a favourable environment for the process. The Calcium carbide residue utilised in this study was sourced from the Toos Acetylene factory. The CCR was subjected to crushing and screened through a 200 mesh sieve before its usage. The chemical composition of the CCR is presented in Table 2. The granulation diagram of CCR, which was determined using a laser device, is shown in Fig. A3. The chemical formula of CCR is also presented as follows (1).

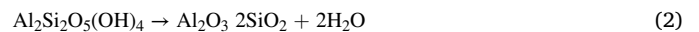


#### 2.1.3. Clay

Clays, or aluminium hydro silicates, contain small natural minerals of soil origin. From the mineralogical point of view, clays have the smallest size among mineral compounds, with dimensions less than 4  $\mu\text{m}$ . For a long time, clays have been considered harmful to concrete due to their high water absorption, which increases the need for mixing water and their considerable swelling. The clay used for part of the base material of geopolymer was prepared from the north of Mashhad. It was sieved through a 200-mesh screen to utilise C as an Alumina ( $\text{Al}_2\text{O}_3$ ) source. Also, the chemical composition of the clay used is presented by XRF analysis in Table 1.

#### 2.1.4. Metakaolin

Following the calcination process, during which the clay is subjected to high temperatures, it transforms into a new substance known as metakaolin. Due to its elevated alumina content, this product is employed as an active pozzolan in the production of geopolymeric cement [62,63]. The chemical formula of metakaolin is as follows (2):



The metakaolin was purchased from the Tehran-based Asan Seram Company. The chemical composition of utilised metakaolin was also determined using XRF analysis, as shown in Table 1. Metakaolin was granulated using a Laser-equipped particle size analysis instrument. According to prior research, the particle size of metakaolin has a clear correlation with its reactivity, and it is also a highly effective method for assessing the sample's strength.

#### 2.1.5. Sand

Mortar mixtures typically necessitate the inclusion of cement and regular sand. Standard sand, water, and cement are essential when creating cement mortar. The sand utilised in this study was produced from silica and Ottawa sand, adhering to the ASTM C349-18 standard [64].

## 2.2. Preparation of samples

Geopolymeric cement is comprised of basic ingredients and alkaline-silicate activator solutions. These substances are used to make geopolymer in this study.

**Table 1**

XRF analysis's GP, C, MK, CCR, OPC chemical compositions. The abbreviation includes ND: None Detected and OPC: Ordinary Portland Cement. Other abbreviations are explained in Figure A1's caption.

CHEMICAL COMPOSITION (%)	GP	C	MK	CCR	OPC
SiO <sub>2</sub>	75.21	50.07	51.65	6.48	19.53
Al <sub>2</sub> O <sub>3</sub>	0.88	11.04	42.84	2.56	3.66
CaO	8.84	13.00	0.12	70.79	58.33
Fe <sub>2</sub> O <sub>3</sub>	0.46	4.86	1.54	3.24	2.54
Na <sub>2</sub> O	10.83	1.56	0.01	N.D	4.91
MgO	3.38	3.39	0.01	0.68	4.31
K <sub>2</sub> O	0.13	2.20	0.12	7.92	1.18
SO <sub>3</sub>	0.01	0.01	0.01	0.66	2.95
TiO <sub>2</sub>	0.06	0.61	2.99	–	0.18
MnO	0.01	0.07	0.03	–	0.02
P <sub>2</sub> O <sub>5</sub>	0.02	0.10	0.04	–	0.27
LOI	0.00	12.94	0.26	–	2.06

**Table 2**

Mixing scheme of geopolymeric mortars. The abbreviation description is W/S: Water Solid ratio. The other abbreviations are explained in Table 1 and Figure A1's captions. The alphabet letters represent the particle type, whereas the numerical values represent the percentage of each particle ratio used.

GROUP	SAMPLE	GP %	C %	MK %	CCR %	PC %	W/S	SAND	F <sub>C</sub> CURING TIMES (DAYS)	BS CURING TIMES (DAYS)	CURING TEMPERATURE (°C)
1	PC100	–	–	–	–	100	0.6	2.75	7–28	28	25–80
1	GP100	100	–	–	50	–	0.6	2.75	7–28	28	25–80
1	GP9C1	90	10	–	50	–	0.6	2.75	7–28	28	25–80
1	GP8C2	80	20	–	50	–	0.6	2.75	7–28	28	25–80
1	GP7C3	70	30	–	50	–	0.6	2.75	7–28	28	25–80
2	MK100	–	–	100	50	–	0.6	2.75	7–28	28	25–80
2	GP9MK1	90	–	10	50	–	0.6	2.75	7–28	28	25–80
2	GP8MK2	80	–	20	50	–	0.6	2.75	7–28	28	25–80
2	GP7MK3	70	–	30	50	–	0.6	2.75	7–28	28	25–80

- A) Basic materials: glass powder - clay - metakaolin  
b) Alkaline activator solution: CCR

RGP, as a source of silica, C, and MK, as a rich source of alumina, is utilised to make an aluminosilicate cement foundation material. CCR was also utilised as an alkaline activator. This study's primary objective is to evaluate the mechanical properties of geopolymeric mortar made from the ingredients above. Also tested were the effects of uncalcined and unbaked clay and calcined or baked clay (metakaolin) on compressive strength, bending strength, setting time, and curing conditions. In earlier research, the appropriate amount of sodium hydroxide and sodium silicate utilised in the production of geopolymeric cement ranged between 35% and 55% based on the properties of the raw ingredients. The pH of the sodium hydroxide solution is pH = 14, but the pH of CCR is 12. Therefore, for the geopolymer mortar used in this investigation, the ratio of CCR to base material was 50% (CCR/BM = 50%). In general, the compounds are divided into two groups:

a. Group 1: This group's samples are GP, C, and an alkaline solvent. In addition to using samples containing 100% glass powder (GP100) to test the rich silica source, Portland cement (PC100) was utilised as a control sample for comparison with geopolymer samples (Table 2)

b. Group 2 consists of GP, MK, and alkaline solvent as its constituent ingredients. As a rich source of alumina, using calcined clay (metakaolin) as a class N natural pozzolan is anticipated to result in enhanced geopolymerization reactivity (Table 2).

10%–20% and 30% of recycled glass powder are replaced by each alumina material (clay and metakaolin) in group 1 and group 2 compounds. In addition, glass powder control samples (GP100) and Portland cement control samples (PC100) are classified in Group 1, while metakaolin control samples (MK100) are categorised in Group 2. The sample naming is determined by the initial letter of each substance's Latin name and the replacement amount of the consumables. For instance, sample GP7MK3 consists of 70% glass powder and 30% metakaolin as its basic material.

### 2.2.1. Material preparation

Before creating the samples, all raw materials were dried in an oven at 60 °C for 72 h. This action was taken to balance the moisture content of all source materials. For drying raw materials, it is essential to establish the oven temperature. The samples were dried at a temperature of 60 °C to maintain the features of each geopolymer substance and the nature of all raw components. The templates used to make the samples are in the following two forms.

- 50 mm × 50 mm × 50 mm cube-shaped iron casting was used to test the compressive strength of mortar.
- 160x40 × 40 mm rectangular-shaped to make Prism mould for checking the bending properties of the mortar.

### 2.2.2. Sample-making method

Fig. A4 illustrates the step-by-step process of creating geopolymer

cement samples. To begin with, all the measured dry components, namely sand, glass powder, clay, and metakaolin, were mixed uniformly for 5 min in a mixer to achieve a homogeneous blend. Subsequently, the components were moistened with water from the mixing plan and thoroughly mixed for 5 min. This step facilitated the combination of ingredients after introducing the alkaline solvent.

An accurately weighed amount of CCR was then added to a specified quantity of water in a separate container to prepare the alkaline solvent. The solution was stirred until the CCR particles were entirely dissolved. This alkaline solvent solution was then added to the mixture of wet solids obtained in the previous stage. After adding the alkaline solvent, the resulting mortar was thoroughly stirred for another 10 min. Next, the remaining alkaline solvent was added to the geopolymer mortar and mixed in a mixer for 5 min. After mixing and achieving a homogenous mixture in compliance with ASTM C349-18 and C305-20 requirements [42,64], the geopolymer mortar was poured into a 50x50 × 50 mm cube mould in three layers. Each layer was compacted by applying 25 blows from a special hammer. To further enhance the density of the samples, the moulds were subjected to vibrations on a vibrating table for 5 s. Lastly, the surface of the samples was sanded, and the moulds were placed in their designated location for further processing.

After 24 h, the samples were removed from the moulds and stored in a plastic container to process and maintain proper humidity until the desired test time. Due to the high sensitivity of geopolymer mixtures to the temperature when mixing the raw materials and the curing temperature, the mixing time and the temperature of the samples were kept constant at around 23–25 °C.

### 2.3. Sample curing

After the initial curing of the mortar in the moulds for 24 h, the samples were divided into two groups and treated separately for seven or twenty-eight days. Following are separate discussions of each group.

#### 2.3.1. Normal environment

Within this experimental group, the samples were extracted from the moulds and carefully stored in plastic containers (zip bags) to retain moisture. Subsequently, these containers were placed inside a monolithic storage unit, ensuring a consistent temperature throughout the 7- or 28-day curing period. The ideal temperature range chosen for preserving these specimens until the completion of the curing process was maintained at 23–25 °C.

#### 2.3.2. In the autoclave

In this set of experiments, the samples were taken out from the mould and subjected to autoclaving at 80 °C for 4 h. The autoclave's controlled environment and the surrounding steam ensured that the water content within the samples remained constant, preventing evaporation throughout the temperature synthesis process. Immediately after the samples were removed from the autoclave, they were quickly relocated to a holding container. A moist cloth was placed over each sample to



maintain a consistent humidity level. Subsequently, the samples underwent a curing process at a temperature range of 23–25 °C. Compressive strength test.

Numerous researchers have utilised the measurement of compressive strength to study the performance of chemical stabilisers. Standard ASTM C349-18 [64] was utilised to examine the compressive strength of geopolymer mortar and mortar manufactured from Portland cement (control sample). Based on this information, 50x50 × 50 mm samples were created, aged 7 and 28 days following construction. They have undergone compressive strength testing.

#### 2.4. Bending strength test

By performing a three-point bending test on a small prismatic sample, the bending strength test for mortar indirectly evaluates the tensile strength of the mortar and the tension created by bending. During this test, at least three loading points impart force to a 160 mm × 40 mm × 40 mm cement beam. ASTM C348-21 tests were conducted on geopolymer samples and 28-day controls [65]. The aim of conducting the bending strength test of the mortar is to indirectly check the tensile strength of the mortar and the tension caused by bending, which is done by performing a three-point bending test on a small prismatic sample.

### 3. Results and discussion

This study divides the geopolymer samples into two categories: those with a glass powder and clay base and those with a glass powder and metakaolin base. The findings from the tests conducted on each category are presented below.

#### 3.1. Group 1: samples containing clay

##### 3.1.1. The effect of replacing the amount of clay on compressive strength

The results for compressive strength values shown are based on the average of the three samples tested. Group 1 samples used clay as a replacement for 10%, 20%, and 30% of 100% glass powder and the amount of alkaline activator used was fixed at 50% of the total base raw material. The strength values of the group of samples, which were processed under normal environmental conditions and autoclave conditions (at 7 and 28 days), are presented in Fig. A5 and Fig. A6, respectively.

The results shown in Fig. A5 reveal that among the geopolymer samples made by replacing clay and processed under normal environmental conditions, the GP9C1 sample had the highest strength at seven days. Additionally, the GP8C2 sample achieved its maximum strength at 28 days, with a compressive strength of 7.87 MPa, slightly lower than

that of other clay samples. On the other hand, the MK100 sample, made using metakaolin, showed the highest strength at 28 days with a compressive strength of 8.96 MPa. It's worth noting that the production process of metakaolin requires high temperatures of 600–850 °C. Given the slight difference in strength between the GP8C2 and MK100 samples, clay may be more economically and environmentally viable.

The control sample made with Portland cement (PC100) had the highest strength compared to other geopolymer control samples (MK100 and GP100) in both normal and autoclave curing conditions and at 7 and 28 days. Furthermore, a significant difference in strength can be observed between the control samples PC100 and MK100 at both 7 and 28 days under normal environmental conditions and in the autoclave environment. In Fig. A6, based on the compressive strength of geopolymer clay samples in the autoclave environment, the GP8C2 sample with 20% glass powder replacement has a higher strength than other clay samples at 7 and 28 days. This diagram shows that this sample can achieve higher strength than the samples containing clay replacements through temperature synthesis and autoclave storage.

Fig. 1 compares the effects of curing conditions in two normal and autoclave environments. This graph demonstrates that the compressive strength of all clay-containing samples processed in an autoclave for seven days was greater than that of geopolymer samples processed in a normal environment. In addition, during the 28-day curing period in the normal environment, the compressive strength of geopolymer samples increased compared to the autoclave environment.

It should be noted that the 7-day samples placed in an autoclave at 80 °C have a higher strength than the normal environment samples. However, the 28-day samples in a normal environment were stronger than those in an autoclave, demonstrating that applying the heat and humidity synthesis process increases the strong growth rate at a young age (7 days) and cannot increase the samples' final strength at 28 days. Some geopolymer research has yielded similar findings, suggesting that using a high-temperature thermal synthesis process may harm the final strength of geopolymers [48,61,66]. As a result, from a technical and economic standpoint, normal environment curing is preferred over autoclave curing. Furthermore, the samples placed in a normal environment have a higher final strength at 28 days than the autoclave environment for all samples. This is because subjecting the material to high temperatures has minimal impact on the final strength measurements, only influencing the speed at which the strength increases. This finding is also consistent with the results by Abbas et al. [61]. Remember that the effect of replacing the amount of clay on compressive strength is presented in Figs. A5 and A6.

##### 3.1.2. The effect of curing time on the compressive strength of clay samples

The research conducted by Abbas et al. [61] determined that the

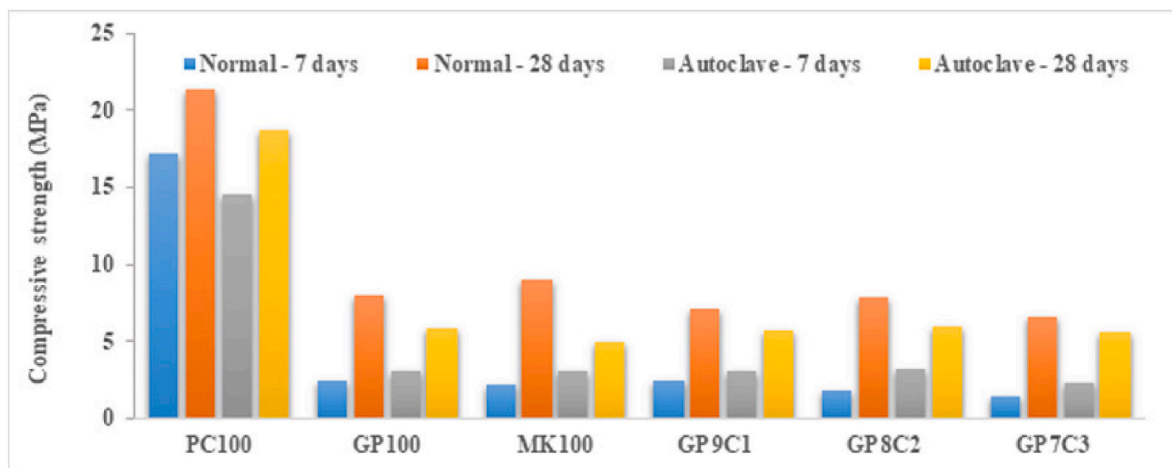


Fig. 1. Compressive strength of samples containing clay (group 1) placed in the normal and autoclave environments.

growth rate of geopolymer samples manufactured utilising recycled glass powder is slower than that of Portland cement. It requires a longer duration to attain desirable resistance and growth of the geopolymer gels in these samples, as depicted in the data presented in Fig. A7 and Fig. A8. These figures showcase the influence of curing time on the increase of the compressive strength of the samples in normal and autoclave curing conditions, respectively. For example, the compressive strength of the Portland cement sample (PC100) after seven days of curing in the normal environment is 17.20 MPa. As the curing period increases, the compressive strength increases with less growth, and its value at 28 days equals 21.4 MPa. The growth rate of the cement sample was fast during the period of 1–7 days and slowed down during the period of 7–28 days.

As shown in Fig. A7, the geopolymer samples displayed different behaviour a normal environment. The process of obtaining the compressive strength of geopolymer samples is much slower than cement samples when the curing time is increased to 7–28 days. As a result, the strength of the PC100 sample increased from 17.20 MPa to 21.4 MPa, representing a growth of about 1.2 times. On the other hand, the strength of the optimal clay-based geopolymer sample (GP8C2) increased from 1.78 MPa to 7.87 MPa over the same period of 7–28 days in the normal environment. It represents a growth of about 4.4 times, indicating a relatively slower rate of gaining strength in geopolymer samples compared to Portland cement samples.

As shown in Fig. A8, the compressive strength of samples containing glass powder and clay in an autoclave environment increased over 7–28 days. The graph also reveals that applying high-temperature conditions has a negligible effect on the final strength values, only affecting the rate of strength gain. This is because increased temperatures can only speed up the geopolymerization process and gel formation but cannot alter the gels' volume or quantity. Here, however, the rate of strength in 7 days was also acceptable.

### 3.1.3. The effect of replacing the amount of clay on bending strength

As with the preceding samples, the geopolymer samples prepared for bending strength tests in group 1 contain clay and glass powder as aluminosilicate components. The percentages of clay replacement are 10%, 20%, and 30%. The samples were produced in 160x40 × 40 mm moulds made of cast iron. The investigation of the bending strength of geopolymer samples with clay replacement (group 1) after 28 days of curing is depicted in Fig. 2. As shown, sample GP7C3, with 30% clay replacement and treated in an autoclave environment, and sample GP9C1, with 10% clay replacement in normal environmental conditions, have greater strength than other geopolymer samples in group 1. Additionally, the sample containing 20% clay replacement, i.e. sample GP8C2, has the lowest bending strength. The bending strength was more significant in the normal environment than in the autoclave environment.

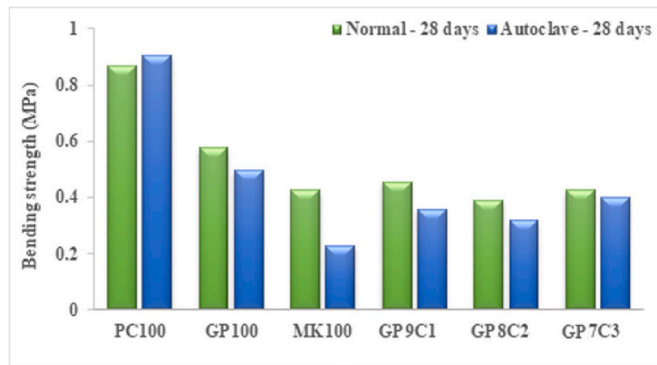


Fig. 2. Bending strength of geopolymer samples with clay replacement (group 1).

As depicted in the image below, however, the bending strength of all clay samples in a normal environment is comparable, with slight variation. GP9C1 has the highest bending strength in a normal setting, while GP7C3 and GP8C2 have slightly different bending strengths. The bending strength was more significant in the normal environment than the autoclave environment. Among the geopolymer samples containing clay, the GP9C1 sample has the highest bending strength, 0.45 MPa, in a normal environment. The difference in bending strength between the geopolymer control sample (GP100) in a normal environment and autoclave and the cement control sample (PC100) at 28 days is 66.4% and 54.7%, respectively. Additionally, this sample's bending strength values exceeded those of clay samples. The effect of heat treatment for 4 h in an autoclave environment on the bending strength of geopolymer samples was accompanied by a decrease in the growth of these samples' strength compared to samples treated in a normal environment. In addition, applying primary heat treatment in an autoclave environment has no impact on cement samples' bending strength.

### 3.2. Group 2: samples containing metakaolin

#### 3.2.1. The effect of replacing metakaolin on compressive strength

In this section, we present results that are consistent with previous findings. We used metakaolin in 10%, 20%, and 30% concentrations, as opposed to 100% glass powder, and a constant ratio of 50% alkaline activator to the base material. The strength of the samples is depicted in Fig. A9 and Fig. A10, which present the data collected at 7 and 28 days under both normal and autoclave conditions.

As shown in Fig. A9, among the samples containing metakaolin that were processed in a normal environment, samples GP7MK3 and GP9MK1 had the highest compressive strength values at 7 and 28 days, respectively. The 7-day compressive strength of the control sample containing 100% metakaolin (MK100) was similar, and the 10% and 20% samples containing metakaolin had the lowest 7-day strength. The difference in compressive strength between the MK100 sample and the other samples containing metakaolin increased from 7 to 28 days over time. Using smaller amounts of metakaolin and larger amounts of glass powder is more effective for achieving higher 28-day strength values. Therefore, 90% glass powder and 10% metakaolin (GP9MK1) can achieve higher strength than 100% metakaolin (MK100). Since metakaolin production requires a thermal process and energy, using it in smaller amounts is more cost-effective and environmentally friendly. Fig. A10 illustrates the compressive strength of geopolymer samples with different percentages of metakaolin replacement and initial heat treatment conditions in an autoclave environment. According to this graph, the GP7MK3 sample has the highest 7-day compressive strength. By curing in a normal environment with 6.12 MPa and in an autoclave environment with 5.51 MPa, the GP7MK3 sample achieved the greatest 7-day compressive strength among metakaolin samples. In addition, sample GP7MK3 with 9.7 MPa had the highest compressive strength after 28 days among samples containing metakaolin, slightly higher than sample GP8MK2 with 9.67 MPa. In the autoclave environment at 7 and 28 days, samples containing both glass powder and metakaolin are stronger than samples containing only metakaolin (MK100) or glass powder (GP100). Combining glass powder as the primary source of silica particles and metakaolin as the primary source of alumina in geopolymer can be effective and suitable.

The compressive strength values of Group 2 compounds at two times and two different curing conditions are shown in Fig. 3. This group of samples was made by replacing 10%, 20%, and 30% metakaolin with glass powder. Alkaline solvent with a constant ratio of activator to the base material of 50% has been used in making all the samples. It can be seen that the 7-day strength of geopolymer cement containing different percentages of metakaolin in the autoclave environment is higher than curing in normal environmental conditions. However, the GP7MK3 sample in the normal environment obtained a higher compressive strength than the autoclave environment at 7 days, with a tiny

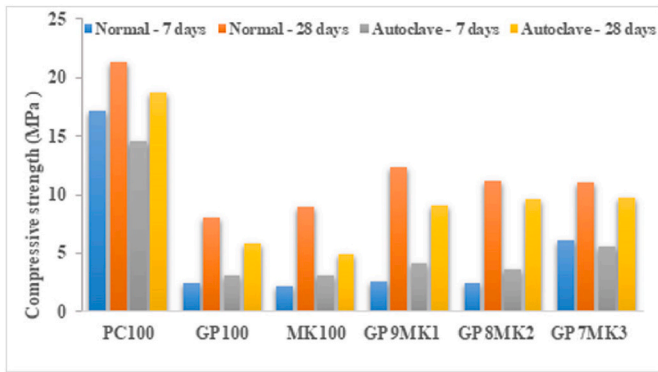


Fig. 3. Compressive strength of samples containing metakaolin (group 2) placed in the normal and autoclave environments.

difference. Also, this procedure is entirely different from the 28-day strength of geopolymer samples in such a way that the compressive strength in the curing conditions of the normal environment has a completely upward trend and has higher values than in the autoclave environment. It shows that the effect of heat treatment is only on the speed of growth of strength in 7-day samples, and heat treatment in an autoclave environment not only did not have a positive effect on obtaining the final 28-day strength of geopolymer samples but also caused a decrease in the 28-day strength growth of the sample. Similar studies [21,52,67] on the effect of geopolymer sample initial curing temperature show that increasing the curing temperature causes a relative increase in the geopolymerization phenomenon's speed and a significant increase in the formation speed of geopolymer gels. However, the increasing temperature is much more noticeable at younger ages because heat accelerates the geopolymerization process, and geopolymeric concrete gains a higher percentage of its resistance at younger ages [68]. As a result, the amount of premature resistance increases in geopolymer concrete. Indeed, increasing the curing temperature improves compressive strength, which is more noticeable and significant in the 3 and 7-day resistance.

### 3.2.2. The effect of curing time on the compressive strength of metakaolin samples

Fig. A11 depicts the greatest growth rate of metakaolin samples during curing between 7 and 28 days and at normal temperatures. As with group 1, in geopolymer samples containing metakaolin, the rate and speed of solid growth in the 7 to 28 per cent range was comparable to that of group 1. Due to metakaolin's high pozzolanic activity and sufficient alumina [69], the second seven days are more productive than the first. It demonstrates that the geopolymer samples in this group are slower than a standard cement sample. In this group, the strength of the samples at seven days of age was comparable (except for the GP7MK3 sample). In a normal environment, the compressive strength of the optimal sample of metakaolin (GP9MK1) increased from 2.6 MPa at 7 days to 12.37 MPa at 28 days. In addition, the cement sample's (PC100) growth rate has increased from approximately 17 MPa–21 MPa. The sample (GP7MK3) with 30% metakaolin substituted for glass powder after 7 days in a normal environment has greater strength than other geopolymer samples in its category. However, in the 28-day sample, the obtained strength is lower than in the other two samples in the metakaolin group. The reason behind the decline in strength at the age of 28 days in sample GP7MK3 was the excessive consumption of alumina (metakaolin) despite the initial increase in compressive strength with higher amounts of consumed alumina (metakaolin) in the early stages.

In other words, to obtain suitable values of long-term strength, consuming an optimal amount of metakaolin (with alumina particles) is necessary. In contrast, if the objective is to obtain higher values of strength at younger ages, it will be appropriate to increase the amount of

alumina consumed (above the optimal amount). In Fig. A12, the strong growth for sample GP9MK1 decreased from the age of 7–28 days compared to the other two metakaolin samples, so it can be seen that if the percentage of metakaolin used in the geopolymer samples is increased, the suitable environment for growth and better performance for the creation of aluminosilicate gels, which increases in compressive strength, must be processed in the initial conditions of an autoclave. Curing at an ambient temperature of  $23 \pm 2^\circ\text{C}$  is advised if this material is decreased. Comparing Fig. A11 and Fig. A12, it is evident that the behaviour and growth rate of cement samples (PC100) in two normal-temperature environments and those treated in an autoclave are extremely similar and are unaffected by the synthesis temperature.

### 3.2.3. The effect of replacing metakaolin on bending strength

According to Fig. 4, sample GP9MK1, with a bending strength of 0.458 MPa, has the highest bending strength among the samples containing a glass powder mixture and metakaolin processed in an autoclave. Meanwhile, the GP8MK2 and GP7MK3 samples had a strength of 0.41 MPa and 0.38 MPa, respectively. The bending strength values of samples containing glass powder and metakaolin were comparable in a typical environment. However, Sun et al. [70] found that the bending and tensile strengths of geopolymers can decrease at high temperatures. The GP9MK1 sample possessed the highest bending strength of 0.49 MPa. As with the samples of group 1, the thermal synthesis process of the primary elements was ineffective in the 28-day bending strength of the samples to the extent that in all samples of this group, the strength values for the samples processed at room temperature were greater than the strength values for the samples subjected to primary thermal synthesis. As stated in the preceding sections, the effect of high temperatures on the final amount of formation of geopolymeric gels and obtaining the long-term strength of geopolymeric samples will be ineffective or detrimental.

Comparing Figs. 2 and 4, it is clear that the combination of metakaolin and glass powder is a more effective base material for the geopolymerization process than the combination of clay and glass powder, and the bending strength values obtained from the combinations containing metakaolin and glass powder are compared to the soil samples. There was more clay and glass powder.

### 3.3. The results of taking time (V-cut test)

Since the mixture's water-to-cement ratio affects the cement's setting rate [71,72], the initial setting time test should be conducted with a specific quantity of water (normal concentration). The initial and final setting time tests followed ASTM C0191-2 specifications [73]. Table 3 displays the test results for the initial and final setting times of Portland cement and two optimal samples of geopolymeric mortar composed of clay and metakaolin. The initial and final setting times of

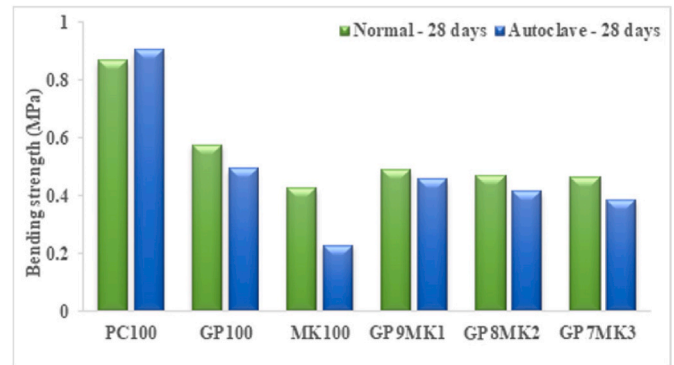


Fig. 4. Bending strength of geopolymer samples with metakaolin replacement (group 2).

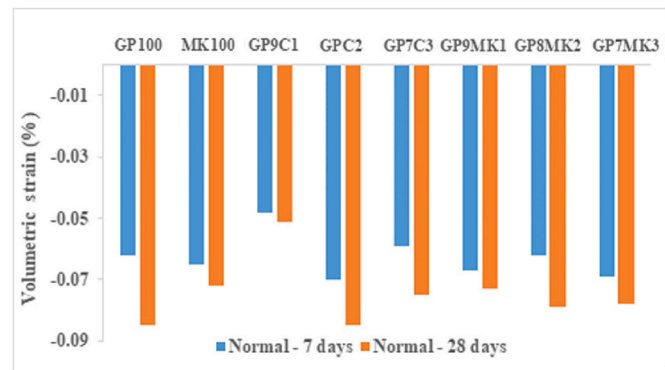
**Table 3**  
initial and final setting time test (V-cut).

Row	Sample name	Initial setting time (min)	Secondary setting time (min)
1	PC100	110	380
2	G9MK1	140	420
3	G8C2	125	400

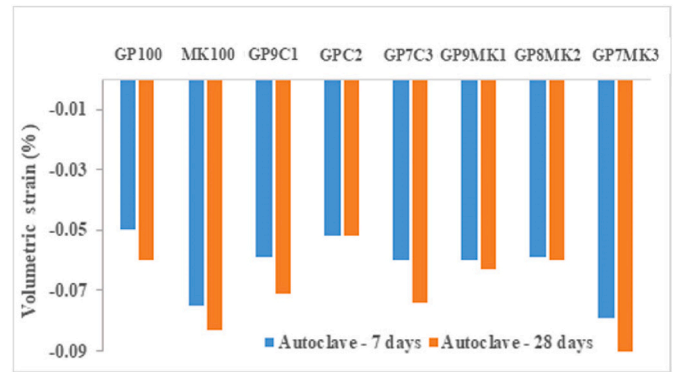
both geopolymer samples are longer than those of the cement sample, as is well known. Additionally, the initial and final setting times for the GP9MK1 sample were longer than those for the GP8C2 sample. The time allotted for transporting and finishing mortar and concrete will increase as the duration of the process grows. Based on the below-listed setting times, it can be concluded that to initiate the geopolymerization reaction in geopolymeric cement and the beginning of the dissolution phase of  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  ions and the formation of aluminosilicate gels through the creation of an alkaline environment using CaO present in calcium carbide residue, more time is required than to initiate the hydration process through the combination of water and cement in cement mortars.

### 3.4. Volumetric changes of geopolymeric samples

Throughout the curing process, volume strains of both the control and geopolymer samples were meticulously measured and recorded to monitor and study any volume changes. After a 24-h curing period, all geopolymeric mortar samples were extracted from the moulds and stored in two different environments, namely a zip bag and an autoclave. Figs. 5 and 6 illustrate the volume changes of  $50 \times 50 \times 50$  mm cubic geopolymer samples cured for 7 and 28 days in a normal environment and autoclave, respectively. Before laboratory analyses, the samples' dimensions (length, width, and height) were measured at 1, 7, and 28 days. As a result, all geopolymer samples experienced some degree of volume loss during the curing process. Among the samples, the one containing 30% clay replacement (GP7C3) and processed in a normal environment for 7–28 days exhibited the most substantial volume loss. However, the GP7MK3 and PC100 samples remained stable in the normal environment from 7 to 28 days with no significant volume change. Overall, the volume behaviour and shrinkage of geopolymer samples were comparable to that of Portland cement samples. The behaviour of volume changes in geopolymer samples differed when subjected to autoclave curing conditions. For instance, the GP8MK2 and GP8C2 samples, substituting 20% metakaolin and 20% clay, experienced no volume change between 7 and 28 days of age. On the other hand, the GP7MK3 and PC100 samples encountered the most significant volume change. Based on the examination of volume changes, it can be inferred that initial heat treatment in autoclave conditions resulted in smaller volume changes for most samples compared to the normal environment, attributed to the accelerated formation of geopolymeric



**Fig. 5.** volume changes of geopolymer cubic samples in a normal environment - 7 and 28 days.



**Fig. 6.** volume changes of geopolymer cubic samples in an autoclave - 7 and 28 days.

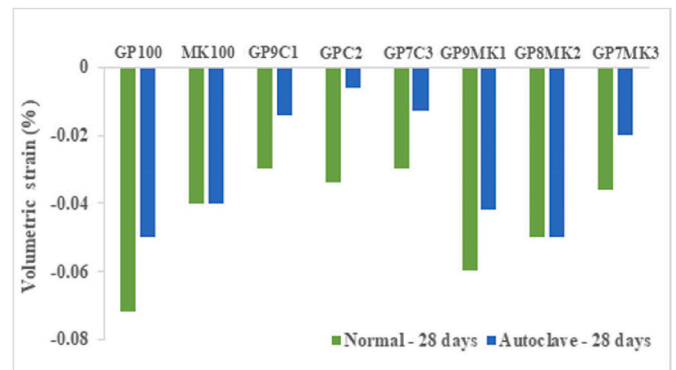
gels and moisture preservation in samples placed in the autoclave environment. The presence of water and reduced evaporation rates significantly influenced the volume stability and shrinkage of the mortar samples. Additionally, Fig. 6 demonstrates that samples processed in an autoclave environment showed fewer volume changes between 7 and 28 days.

Fig. 7 illustrates the volume changes of rectangular cube samples with dimensions of  $160 \times 40 \times 40$  mm in a normal environment and an autoclave after 28 days. Based on this diagram, the volume changes of the samples placed in the autoclave environment are more stable, and the shrinkage values in these samples are greater than those in the natural environment at  $25^\circ\text{C}$ . Also, the shrinkage of geopolymer samples containing metakaolin appears less than that of clay samples. Based on the study's findings, the optimal geopolymer samples have a lower volume change potential and greater volume stability. Due to the complete formation of geopolymer gels in the optimal samples, the internal structure of the samples has become more compact, thereby reducing the likelihood of water evaporation from the sample structure. As a result, the volume stability of geopolymer samples has improved, and the drop has decreased.

### 3.5. Discussion about microstructural studies

Geopolymer cement is a composite material composed of a gel matrix phase with unreacted crystalline phases, which may be derived from raw material impurities or due to the matrix phase's recrystallisation process [74], and which increases the bonding of clay particles [61]. Fig. 8(a–d) depicts scanning electron micrographs (SEM) of PC100, GP9MK1, GP100, and GP9C1 samples with a 28-day curing period in a normal environment and a 500x magnification.

The image of sample GP9MK1 has a denser and more integrated



**Fig. 7.** volume changes of geopolymer prismatic samples in a normal environment and an autoclave - 28 days.



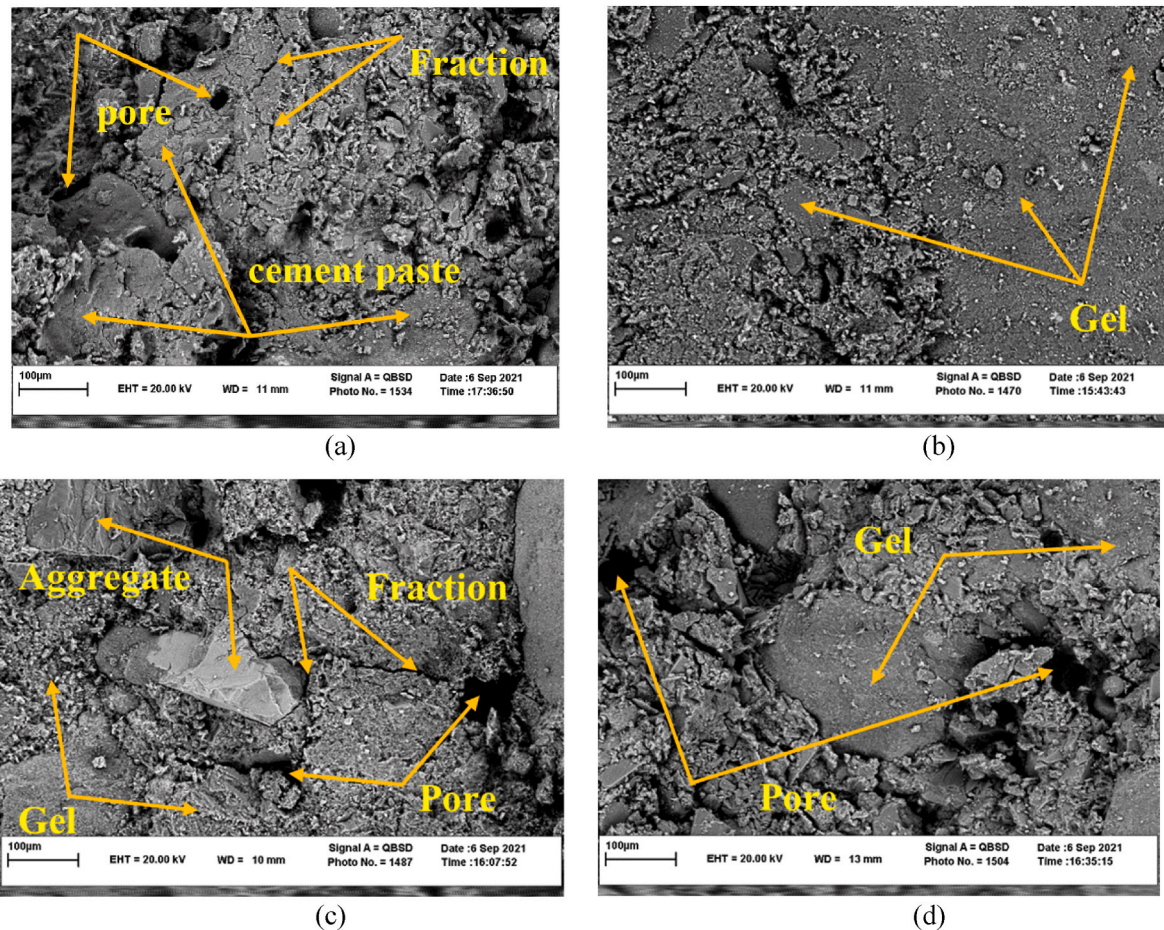


Fig. 8. the SEM images of: a) PC100 sample b) GP9MK1 sample c) GP100 Sample d) GP9C1 Sample.

structure than the images of samples without metakaolin, as shown in Fig. 8-b. The GP9MK1 sample has greater density, porosity, and far fewer hairline cracks than the other samples because metakaolin's chemical structure creates better porosity (homogeneity and density) in cement paste [69]. This difference in the surface microstructure of the samples may be the primary reason for the variation in compressive strength between the samples. As the number of pores on a surface increases, the stress concentration on a homogeneous and continuous component also increases, resulting in a decrease in strength. The fineness of metakaolin and the composition of this material has resulted in a greater reactivity than that of glass powder. In addition, calcium carbide residue as a more appropriate alkaline solvent has formed more aluminosilicate gels in this sample.

Fig. 8-b demonstrates the integrity of the surface created by forming an aluminosilicate gel. Fig. 8-c displays the lack of surface integrity and heterogeneous mixing between aggregate and aluminosilicate gel in the GP100 sample, which is composed of 100% glass powder and does not contain clay or metakaolin (as alumina sources). In this sample, the raw material is abundant in silica compounds, while the alumina source is minimal, preventing geopolymeric gels from forming in this sample. In terms of appearance, sample GP100 exhibits greater porosity and microcracks than samples GP9MK1 and GP9C1. Notably, 10% of metakaolin and clay have been replaced as alumina sources in two samples, GP9MK1 and GP9C1. This has resulted in the enhancement of geopolymerization reactions, the formation of geopolymeric gels with stronger bonds, and increased surface integrity of these samples. According to the data, the number of cracks and pores in the surface structure of the GP9C1 sample is greater than that of the GP9MK1 sample, which may account for the GP9MK1 sample's higher

compressive and bending strength values. Fig. 8-a demonstrates that the surface structure of the PC100 sample contains a relatively high number of microcracks and pores. This sample's sensitivity to destructive environmental conditions can be attributed to its significance.

#### 4. Conclusion

The study examined geopolymer mortar samples' compressive and flexural strengths, incorporating recycled glass powder and two alumina sources: clay and metakaolin. Both formulations utilised a 50% calcium carbide residue alkaline activator mixed with the geopolymer base material. Results indicate that geopolymer samples exhibited lower compressive strength compared to cement samples. Nevertheless, considering environmental sustainability and using recycled materials, geopolymer cement offers a viable option for specific applications while maintaining comparable strength levels. Additionally, waste calcium carbide proved effective as an alkaline activator, facilitating the dissolution of aluminosilicate particles and the formation of geopolymeric gels. Tests conducted under normal curing conditions (23–25 °C) for up to 28 days demonstrated an optimal compressive strength of 12.37 MPa for the GP9MK1 geopolymer sample, with a flexural strength of 0.6 MPa for the GP100 sample in the same environment. Notably, alumina sources derived from metakaolin and clay exhibited higher compressive strengths. Autoclave curing for seven days enhanced initial strength, particularly in geopolymer samples, although specimens cured in a normal environment showed more remarkable volume changes. Metakaolin proved more effective than clay in reducing volume changes due to its finer particle size and enhanced geopolymer gel formation in alkaline environments.

Metakaolin's smaller particle size facilitates geopolymerization reactions and gel formation, contributing to greater stability and denser microstructures. This is evident in the superior surface homogeneity of the GP9MK1 sample, attributed to higher proportions of metakaolin relative to glass powder. Despite its advantages, the high-heat production process associated with metakaolin renders clay a more environmentally friendly alumina source. Maintaining an appropriate silica-to-alumina ratio is crucial for optimizing geopolymerization. The GP8C2 and GP9MK1 samples consistently demonstrated optimal compressive and flexural strengths across normal and autoclave curing environments. Further research is warranted to investigate the effects of varying alkaline activator concentrations on durability testing, along with a comprehensive analysis of specimen behaviour under corrosive, sub-zero, and high-temperature conditions. Additionally, exploring the potential incorporation of fibres to enhance tensile strength is recommended.

### CRedit authorship contribution statement

**Zahra Safarzadeh:** Writing – original draft, Visualization, Validation, Resources, Methodology, Investigation, Formal analysis. **Meysam Pourabbas Bilondi:** Writing – original draft, Project administration, Methodology, Investigation, Data curation, Conceptualization. **Mojtaba Zaresefat:** Writing – review & editing, Visualization, Data curation.

### Declaration of competing interest

The authors declared that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

### Data availability

Data will be made available on request.

### Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.rineng.2024.101974>.

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