

## Recycled Ceramic tableware waste in fly ash–slag geopolymer concrete: Microstructure and strength evaluation

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### Abstract

Wise waste management for sustainable construction materials demand development of eco-friendly binders that utilize industrial byproducts and incorporate waste materials such as ceramic tableware waste powder (CTWP) to reduce environmental impact. Geopolymer concrete is a cementless concrete using an alkali-activator to produce low-carbon concrete, and it mainly depends on the alumino-silicate content from the binder. Therefore, this study focuses on the silica effect from the ceramic waste powder (CWP) silica in polymerized aluminosilicate concrete by investigating the mechanical and Microstructural performance in terms of compressive strength, tensile, and flexural properties, as well as using XRD, XRF, FESEM/EDX, FTIR, and AFM. The use of ceramic waste powder (CWP) in geopolymer concrete at a molarity of 12 molarity, with CWP replacement levels of 0%, 3%, 6%, 9%, and 12% of the binder mass. Microstructural analyses confirmed mullite and gismondine formation, increased polymerization, consistent with the mechanical results. Overall, the targeted incorporation of 9% CWP enhanced the mechanical performance and promoted microstructural densification of geopolymer concrete, supporting both resource efficiency and waste reduction in construction.

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## 1. Introduction

Geopolymer concrete (GPC) has been identified as a sustainable and superior-performing substitute to ordinary Portland cement (OPC), providing notable environmental and engineering benefits [1]. However, cement manufacturing, which involves limestone calcination and substantial energy demands, contributes approximately 8% of global CO<sub>2</sub> emissions [3]. With the construction sector identified as one of the biggest CO<sub>2</sub> emissions contributors [2], an environmentally friendly OPC alternative must be made available to reduce the product's negative impact on the environment.

The geopolymer concrete is one of the possible alternatives to OPC and is manufactured with the help of industrial byproducts and waste materials, and is able to decrease carbon emissions. GPC is able to achieve strength faster than ordinary Portland cement, has reduced shrinkage and is able to withstand sulfate attack, chloride penetration and acid exposure better. Also worth mentioning is the fact that because of GPC's greater mechanical strength, it is more resistant to chemical and thermal damage as well as durable, which, in turn, makes it appropriate for the long-term infrastructures [6].

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GPC is an industrial byproduct, such as fly ash, ground granulated blast furnace (GGBS), and is activated by alkaline solutions, particularly in cases where the mixture is held together by ordinary Portland cement (OPC) [7]. Since this reaction was caused by sodium hydroxide (NaOH) and sodium silicate ( $\text{Na}_2\text{SiO}_3$ ) reaction with the binding materials, replacing OPC with GPC can reduce the quantity of carbon released to the atmosphere by an estimated 40-80% of conventional concrete [8].

Geopolymerisation's chemical makeup is formed by a three-dimensional sodium aluminosilicate hydrate (N-A-S-H) network [9], which happens in three key stages: dissolution, gelation, and polymerization [10]. The initial phase involves the alkaline activators, which, before the release of silicate ( $\text{SiO}_2$ ) and aluminate ( $\text{Al}_2\text{O}_3$ ) species into the solution, disintegrate the aluminosilicate precursors [9]. The concentration of the ions at this stage is vital, and the release of the required amount may prove slow, hence negatively affect the reaction, which will subsequently affect the performance of the end product [6]. These released species during gelation then form gels, which are mainly N-A-S-H gels [11], and then the gels are reorganized and cross-linked during the polymerization stage to become hardened with superior strength and long-term durability [12].

Silica (Si) plays a crucial role in geopolymerisation, being one of the primary building blocks of sodium aluminosilicate hydrate (N -A -S -H ). The release of sufficient silicate ions in the dissolution phase aids the gel to grow successfully, and the low concentrations would disrupt the polymerization process and compromise the overall performance of the material [6, 9]. Thus, the addition of silica in the mix contributes to the stronger gelation process by encouraging and forming a denser N -A -S -H network, which decreases pore size, fills the void, and optimizes the microstructure. This means that geopolymer concrete will enjoy superior strength, better survival, and reduced permeability [13, 14]. The evidence of these results is supported by a number of research that indicate that stocked silica rich waste materials have the ability to enhance mechanical properties by 20-30% relative to unmodified mixes, which is mainly because of the establishment of denser matrices with low porosity [15]. On the same note, Gado et al. [16] found that the inclusion of silica-rich waste considerably increased compressive strength, with the highest value of 37.5 Mpa achieved when the waste was included. Collectively, these results demonstrate silica as a key factor in the geopolymerisation process and how it directly affects the mechanical and durability performance of geopolymer concrete.

One of the by-products of construction and demolition processes is ceramic waste powder (CWP), which is usually thrown in dumpsites, contributing to environmental pollution. The production of ceramic tiles in the world amounts to more than 10 million square meters annually, out of which 15-30% cannot be utilized due to the grinding and polishing processes [17, 18]. A major part of this waste is deposited on the landfill sites, which pollute the surrounding soil, water, and air [19, 20]. CWP has great potential as a sustainable feedstock to geopolymer manufacture due to its high silica (Si) and alumina (Al) content and pozzolanic reactivity [21].

Since the management of waste materials in the environment is eco-friendly and thereby helps to reduce the effects of construction and demolition processes on the environment, waste reuse in the construction sector is a bright channel for ensuring ecological sustainability. In this respect, the novelty of this study is the analysis of the mechanical and microstructural properties of geopolymer concrete containing ceramic waste from tablewares. In particular, the study measures the effects of 12 M concentration of the ceramic waste powder, the important mechanical properties, e.g. slump, compressive strength, tensile strength, and flexural strength. Moreover, more detailed microstructural studies, such as XRD, XRF, FESEM/EDX, FTIR, and AFM, are used to obtain more profound information. The application of silica in improving the behaviour of geopolymer concrete can be comprehended more fully through this investigation.

## **2. Methodology**

### **2.1. Materials**

Ground Granulated Blast Furnace Slag (GGBS) is a by-product in the iron and steel sector. In this experiment, GGBS produced in a steel plant in Ipoh, Perak, Malaysia, was considered as an additive

material in the geopolymer mix. It is fine, with low iron oxide (white in colour), mean particle diameter of  $11.7 \mu\text{m}$ , specific surface area of  $4,650 \text{ cm}^2/\text{g}$  and a specific gravity of  $2.88 \text{ g/cm}^3$ . As demonstrated by sieving, virtually all particles were not filtered through a  $45 \mu\text{m}$  sieve, which means high fineness and reactivity in alkaline media that can enhance cohesion in the mixture and decrease intergranular porosity [22].

A geopolymer mix used silica and alumina as sources, with the silica and alumina sources being type F fly ash (FA), which was procured in Port Dickson, Malaysia, in a coal-fired power plant [23]. It weighs an average of  $15.8 \mu\text{m}$  and has a specific surface area of  $3,850 \text{ cm}^2/\text{g}$  and a specific density of  $2.25 \text{ g/cm}^3$ . The distribution of the particles in the mixture can be uniform, as nearly 95% of the particles go through a  $45 \mu\text{m}$  sieve. The material is dark grey because of the presence of iron and minerals, but this could affect the aesthetic, but not the performance [24].

The ceramic that was used in this experiment was locally obtained, it was cleaned to remove any surface dirt, and then 24 hours of drying were done to remove any remaining moisture. The dried materials were then crushed in a Los Angeles abrasion machine fitted with stainless-steel balls (48 mm and 22 mm in diameter) after drying the materials, and then sieved through a  $75 \mu\text{m}$  sieve. The fraction that was retained was further ground in a grinding machine with 56 steel balls of different sizes after 48 hours at 60rev/min to obtain smaller particles. Figure 1 represents how to prepare the ceramic powder.



Fig. 1. Preparation procedure of CWP from discarded ceramic materials

CWP is one of the best structural components, and the average size of the particle is  $0.165 \mu\text{m}$ . This level of fineness, plus the ultra-high specific surface area of  $12,700 \text{ cm}^2/\text{g}$ , the highest of all the materials used, enhances the speed of the alkali activation reaction and the efficiency of the geopolymer gel formation [25]. It is characterised by a sandy beige colour because it contains a unique mineral structure, and it is sieve-analysed that 100% of the particles would pass through a  $45 \mu\text{m}$  sieve, which further allows CWP to penetrate fine structures in the mixture and also leads to a denser and more uniform microstructure [26]. These composite properties are important in enhancing the mechanical performance of the geopolymer concrete and its durability [18]. Table 1 shows the physical properties of the proposed binder.

Chemical compositions of Fly Ash, GGBS and CWP were also identified by X-ray fluorescence (XRF), and the findings are shown in Table 2. The obtained results revealed that the GGBS was dominated by a high-calcium oxide content of 43.00%, accompanied by  $\text{SiO}_2$  content of 35.00% and  $\text{Al}_2\text{O}_3$  content of 13.20%. This chemical composition consists of high proportions of calcium and silica, which makes GGBS have a high ability to react with alkali and C-(A)-S-H gel forming compounds,

which is one of the reasons why it has become a pivotal compound in enhancing early compressive strength in geopolymer systems [23].

Table 1. Physical properties of the fly ash, GGBS, and CWP

| Physical Property                                | Fly Ash   | GGBS  | CWP         |
|--------------------------------------------------|-----------|-------|-------------|
| Average particle size ( $\mu\text{m}$ )          | 15.8      | 11.7  | 0.165       |
| Surface area (Blaine) ( $\text{cm}^2/\text{g}$ ) | 3850      | 4650  | 12700       |
| Specific gravity ( $\text{g}/\text{cm}^3$ )      | 2.25      | 2.88  | 2.70        |
| Colour                                           | Dark grey | White | Sandy beige |
| % Passing through 45 $\mu\text{m}$ sieve         | 95%       | 98%   | 100%        |

$\text{SiO}_2$  (52.40%) and  $\text{Al}_2\text{O}_3$  (23.10%) were the main components of Fly Ash, with  $\text{Fe}_2\text{O}_3$  (7.50%) coming next. It also had calcium oxide in 9.80% besides other minor percentages of magnesium oxide and potassium oxide. The value of loss on ignition was 5.30% showing the availability of volatile or carbonaceous materials [23]. According to the calculations we have, the  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ , and  $\text{Fe}_2\text{O}_3$  make up more than 83% of the total fly ash content, which is in line with the ASTM C618 requirements of effective pozzolanic materials, thus resulting in the conclusion that fly ash is suitable to react with geopolymer and form N-A-S-H gels. The XRF indicated that the CWP contains mainly  $\text{SiO}_2$ , then  $\text{Al}_2\text{O}_3$ , with small traces of calcium oxide and iron oxide.

The GGBS can be described as low-intensity with few peaks, which is the pattern of diffraction that reflects an almost completely glassy nature with minor crystalline residues (quartz or feldspar phases). Under alkaline conditions, this amorphous structure is very reactive and offers calcium, silicon and aluminium ions required in the formation of C-(A)-S-H or N-(C)-A-S-H gel that enhances early compressive strength. The XRD pattern of fly ash identifies clear peaks with quartz (Q) and mullite (M) phases, which are indicators of a combination of glassy and crystalline phases. The glassy fraction is used to supply both the pozzolanic activity required to form N-A-S-H gel, and the crystalline phases serve as fillers and nucleation sites (increasing the microstructure of the material). Figure 3 represents the FESEM micrograph of the ceramic powder. The findings denoted the shape of ground ceramic powder once milled, which diminished the median particle size. The particles were mainly round and spherical in shape, with an inclination towards forming agglomerated clusters.

Table 2. Chemical compositions of the fly ash, GGBS, and CWP

| Chemical Compositions                       | Mass content (%) |        |        |
|---------------------------------------------|------------------|--------|--------|
|                                             | Fly Ash          | GGBS   | CWP    |
| Silicon dioxide ( $\text{SiO}_2$ )          | 52.40%           | 35.00% | 59.80% |
| Aluminium oxide ( $\text{Al}_2\text{O}_3$ ) | 23.10%           | 13.20% | 19.60% |
| Iron oxide ( $\text{Fe}_2\text{O}_3$ )      | 7.50%            | 1.00%  | 2.10%  |
| Calcium oxide (CaO)                         | 9.80%            | 43.00% | 6.20%  |
| Magnesium oxide (MgO)                       | 1.40%            | 6.30%  | 1.80%  |
| Potassium oxide ( $\text{K}_2\text{O}$ )    | 0.50%            | 0.30%  | 0.40%  |
| Loss on Ignition (LOI)                      | 5.30%            | 1.20%  | 0.10%  |

Figure 2 displays the XRD patterns of CWP with crystalline sharp peaks with a large range of diffraction angles and with distinguishable peaks at around  $20.8^\circ$ ,  $26.6^\circ$ ,  $27.9^\circ$  and  $40.8^\circ$  ( $2\theta$ ), which are attributed to the existence of quartz ( $\text{SiO}_2$ ) as a primary phase. Other reflections of about  $22.0^\circ$  and  $28.0^\circ$  are of the albite ( $\text{NaAlSi}_3\text{O}_8$ ), and  $16.4^\circ$  and  $33.2^\circ$  reflect the mullite ( $\text{Al}_6\text{Si}_2\text{O}_{13}$ ), and  $29.4^\circ$  and  $50.1^\circ$  indicate gesmondine ( $\text{CaAl}_2\text{Si}_2\text{O}_8 \cdot 4\text{H}_2\text{O}$ ). This spectrum is a sign that the material is highly crystalline in nature and has a small percentage of amorphous components, giving it a relatively low early reactivity in the geopolymer system, but which offers a supporting mineral structure and nucleation sites in growing geopolymer gels, which increases micro-density and long-term durability.

A characteristic of the GGBS is a low-intensity diffraction pattern with few peaks that indicate an almost pure glassy nature with small crystalline fragments (quartz or feldspar phases). This amorphous structure can be very reactive in an alkaline environment, which supplies the calcium, silicon, and aluminium ions, which are required to form C-(A)-S-H or N-(C)-A-S-H gel to enhance the early compressive strength. The fly ash XRD pattern indicates clear peaks, which are a mixture of crystalline and glassy phases with quartz (Q) and mullite (M) as phases. The glassy fraction is what gives the pozzolanic properties required to form N-A-S-H gel, whereas the crystalline phases are used as filler and nucleation sites that contribute to the enhancement of the microstructure of the material. In Figure 3, the FESEM micrograph of ceramic powder is presented. The findings showed the shape of ground ceramic powder following the milling process, which decreased the median size of particles. The particles were mainly rounded and spherical shapes with a tendency to develop agglomerated clusters.

Figure 2 reveals that XRD patterns of CWP presented sharp crystalline peaks that were spread across a large range of diffraction angle with noticeable peaks at approximately  $0.8^\circ$ ,  $26.6^\circ$ ,  $27.9^\circ$ , and  $40.8^\circ$  ( $2\theta$ ), attributed to the presence of quartz ( $\text{SiO}_2$ ) as the main phase. Other reflections at approximately  $22.0^\circ$  and  $28.0^\circ$  are associated with albite ( $\text{NaAlSi}_3\text{O}_8$ ), peaks at approximately  $16.4^\circ$  and  $33.2^\circ$  reflect mullite ( $\text{Al}_6\text{Si}_2\text{O}_{13}$ ), and peaks at approximately  $29.4^\circ$  and  $50.1^\circ$  indicate gismondine ( $\text{CaAl}_2\text{Si}_2\text{O}_8 \cdot 4\text{H}_2\text{O}$ ). This spectrum indicates that the material is highly crystalline in nature with a limited proportion of amorphous components, making its early reactivity in the geopolymer system relatively low, but providing a supporting mineral framework and nucleation sites for geopolymer gel growth, which enhances micro-density and long-term durability.

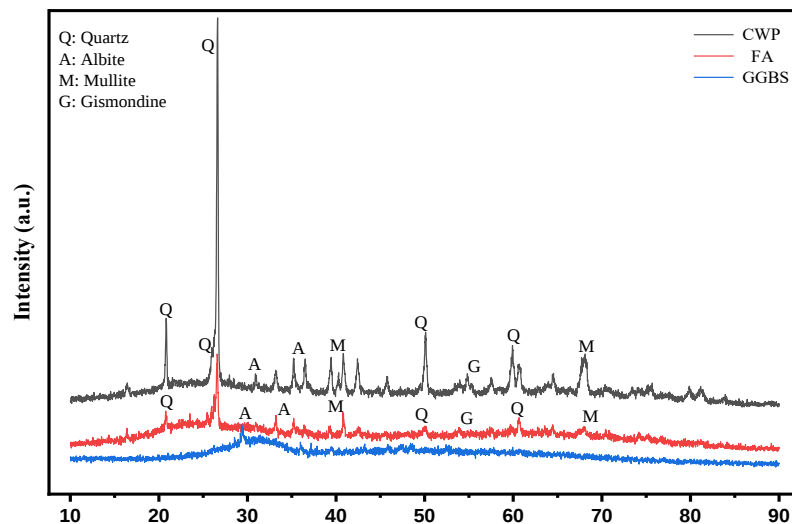


Fig. 2. The XRD patterns of Fly Ash, GGBS, and CWP

The alkaline activator, which consisted of sodium silicate (NS) and sodium hydroxide (NH, 98% purity), was used to activate the alumina and silica in CWP and GGBS. The NS solution contained 29.5%  $\text{SiO}_2$ , 14.7%  $\text{Na}_2\text{O}$ , and 55.8%  $\text{H}_2\text{O}$  by mass. Sodium hydroxide pellets were later dissolved in water to prepare a 12 M solution, which was cooled for 24 hours before being mixed with NS. The reactivity of the NH is high at high molarity of 12M, which requires a time (24 hours) for cooling the solution and remains chemically stable until the casting date. At a rate of 20 mL per minute, the  $\text{Na}_2\text{SiO}_3$  was added to NaOH and mixed at a rotation speed of 500 rpm for 10 minutes by a mechanical stirrer to confirm the uniformity of the alkalinity solution. The final alkaline solution was prepared with varying  $\text{SiO}_2$ :  $\text{Na}_2\text{O}$  mass ratios, as shown in Table 2, while the NS: NH ratio was kept constant for all mixtures.

Physical testing was conducted on natural aggregate to assess its suitability for concrete production. For coarse aggregate, sieve analysis revealed that 0.4 % of the total mass passed through the  $75 \mu\text{m}$  sieve, which is well within the maximum allowable limit of 1.0 % as defined by ASTM C33/C33M-18, the standard specification for concrete aggregates. The apparent specific gravity obtained was 2.65, which is within the normal range of 2.4 to 3.0 as recorded in the C127-

15 of ASTM. The ratio of water absorption, in the meantime, was 1.0% which also falls within the range of 0.5 to 4.0% which is accepted as per the same standard. Such a degree of absorption implies that the aggregate has a relatively low porosity, and this fact can help to increase the durability of concrete and reduce the level of permeability. This is in line with ASTM C33/C33M-18 requirements on coarse aggregate that is employed in structural concrete.

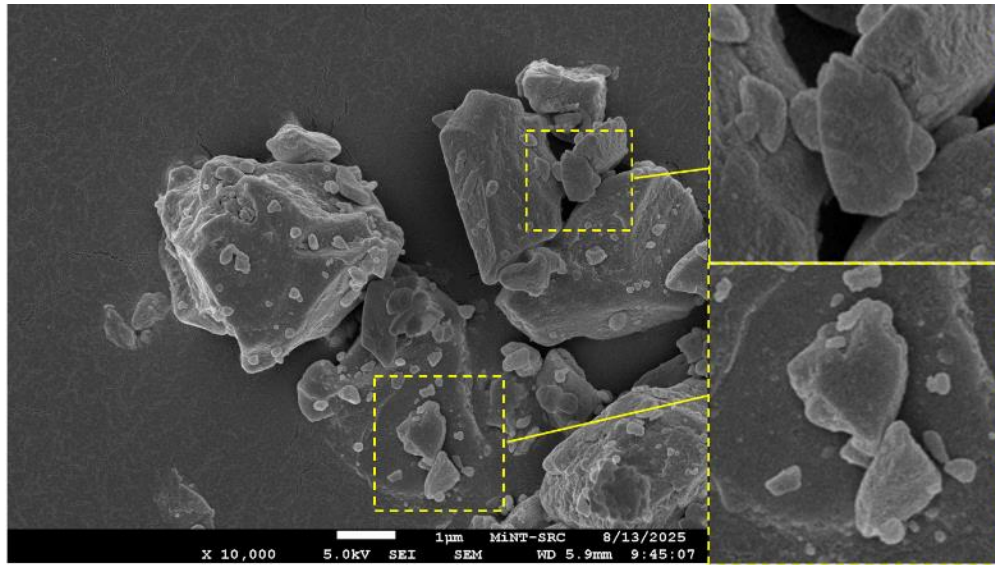


Fig. 3. The FESEM micrograph of CWP particles



Fig. 4. Procedure for the alkaline activator solution preparation

Table 3. Mix proportions and chemical composition of alkaline solutions

| Alkaline Solution | NaOH Solution (NH) |                   |                  | Na <sub>2</sub> SiO <sub>3</sub> Solution (NS) |                   |                  | NS: NH Mass% | SiO <sub>2</sub> :Na <sub>2</sub> O Mass% |
|-------------------|--------------------|-------------------|------------------|------------------------------------------------|-------------------|------------------|--------------|-------------------------------------------|
|                   | Molarity           | Na <sub>2</sub> O | H <sub>2</sub> O | SiO <sub>2</sub>                               | Na <sub>2</sub> O | H <sub>2</sub> O |              |                                           |
|                   | M                  | Mass%             | Mass%            | Mass%                                          | Mass%             | Mass%            |              |                                           |
| S1                | 12                 | 26.98             | 65.17            | 29.5                                           | 14.7              | 55.8             | 0.75         | 1.02                                      |

A set of physical tests was done on the fine aggregate to determine its suitability for use in the concrete mixtures. Sieve analysis disclosed that the majority of the particles were in the range of 150 µm to 4.75 mm. This distribution matches the requirements of the gradation of particles in the ASTM C33/C33M-18, which focuses on a well balanced aggregate profile in order to facilitate cohesive behaviour within the fresh concrete mix. Specific gravity of the fine aggregate was measured at 2.61, which is within the acceptable range of 2.4 and 2.8, as indicated in the ASTM

C128-15 directive. The rate of water absorption achieved was 1.8%, which also fell within the acceptable value of 0.5 to 4.0% stipulated in ASTM C127-15.

## 2.2 Mix Design

In this study, the mix design was calculated using methods for normal concrete mix design. Subsequently, a mix design for geopolymer concrete was developed by replacing ordinary Portland cement with a combination of 70% fly ash and 30% GGBS. The alkaline activator solution was formulated using a mixture of sodium hydroxide (NaOH) and sodium silicate ( $\text{Na}_2\text{SiO}_3$ ), with NaOH prepared at a concentration of 12 molarity (12M). Specifically, the NaOH solution was created by dissolving 480 grams of NaOH pellets in 1 litre of water. The  $\text{Na}_2\text{SiO}_3$  solution was prepared by combining equal parts of water and sodium silicate. The CWP content was added as a replacement at levels of 0%, 3%, 6%, 9%, and 12% by weight of fly ash and GGBS. The mix proportions for a 1  $\text{m}^3$  volume of geopolymer concrete were calculated and are presented in Table 4.

Table 4. Mixed design of the proposed concrete

| Mix Code | Geopolymer Binder (%) |      |      | Geopolymer Binder ( $\text{kg}/\text{m}^3$ ) |          |          | Natural Aggregate ( $\text{kg}/\text{m}^3$ ) |        |
|----------|-----------------------|------|------|----------------------------------------------|----------|----------|----------------------------------------------|--------|
|          | CWP                   | GGBS | FA   | CWP                                          | GGBS     | FA       | River                                        | Gravel |
| CG0      | 0                     | 30   | 70   | 0                                            | 118.53   | 276.57   | 810.71                                       | 990.86 |
| CG3      | 3                     | 28.5 | 68.5 | 11.853                                       | 112.6035 | 270.6435 | 810.71                                       | 990.86 |
| CG6      | 6                     | 27   | 67   | 23.706                                       | 106.677  | 264.717  | 810.71                                       | 990.86 |
| CG9      | 9                     | 25.5 | 65.5 | 35.559                                       | 100.7505 | 258.7905 | 810.71                                       | 990.86 |
| CG12     | 12                    | 24   | 64   | 47.412                                       | 94.824   | 252.864  | 810.71                                       | 990.86 |

\*CG0 (Geopolymer concrete with 0% of CWP) \*\* CG9 (Geopolymer concrete with 9% of CWP)

## 2.3 Test Procedure

The geopolymer concrete's slump test was conducted in accordance with ASTM C143, which warrants the use of a cone-shaped frustum (300 mm height) filled with three layers of concrete, each compacted by 25 blows of a steel rod. Once the mould is filled and levelled, it is gently lifted vertically, allowing the unsupported concrete to slump, with the height reduction measured using a tape to determine the slump value. For this purpose, cubic moulds of size 100 mm, a cylinder of dimension (100 mm x 200 mm), and a prism of dimension (100 mm x 100 mm x 500 mm) were cast for compressive, tensile and flexural strength samples, and were measured using BS EN 12390-3:2019, BS EN 12390-6:2023 and BS 12390-5:2019 standards, respectively. The samples were evaluated at 7 and 28 days under ambient curing conditions at a temperature of 25-30 °C.

To investigate the surface morphology of the geopolymer matrix, Field Emission Scanning Electron Microscopy (FESEM) was followed as per the ASTM E1508. The samples were washed, placed and coated to allow imaging. Following the stabilization of the chamber and changing the voltage (0.5-30 kV), high-resolution images were taken at various magnifications. The integrated Energy Dispersive X-Ray Spectroscopy (EDX) detector was employed to ascertain the elemental composition to allow the microstructure and chemicals to be uniformly distributed. Further, to have a better insight into the mineralogical structure and the stage of development of the material, the crystalline phases of the geopolymer matrix were examined by the requirements of ASTM E2570 in X-ray Diffraction (XRD).

Besides EDX and XRD to perform a fine analysis, the spectrum was also tested by means of software and reference databases to check the bonding and phasing of the chemicals. To do this, Fourier Transform Infrared (FTIR) spectroscopy was employed to measure and compare the recorded peaks' positions and intensities. According to the requirements of ASTM E1252, the tool was applied in the identification of the functional groups of the geopolymer matrix. Besides employing EDX, FTIR, and XRD, the Atomic Force Microscopy (AFM), in compliance with the ASTM E2543, was also employed in the analysis of the surface topography of the geopolymer matrix. The reason why AFM was preferred was because it was capable of determining external irregularity of the matrix, the sizes of features and structural features of the matrix.

### 3. Results and Discussion

#### 3.1 Slump Test

Workability of the geopolymer concrete was determined through a slump test with varying replacement of fly ash/GGBS binder with ceramic powder. Unmixed reference with no ceramic powder (CG0 %) registered an estimated value of 110mm slump, which is of good workability to work across all general applications of concrete. When the content of ceramic powder went up to 3 %, 6 %, 9 %, and 12 %, the slump values went down to 106.5 mm, 102.5 mm, 98.6 mm and 94.0 mm, respectively. It is explained by the large surface area and absorption of the ceramic powder that decreases the percentage of free water by absorbing a portion of the alkaline solution, raising the viscosity and hindering the flow. The reduced values of slump could have a minor implication on the ease of placing and distributing the concrete, particularly at higher levels of replacement, and it might entail more compaction effort. This is, however, in the acceptable range of workability and helps to enhance the mix uniformity and minimize bleeding and segregation, which is a favorable sign of mix quality and uniformity [17]. This decreasing tendency in the slump values as the ceramic content increases is shown in Figure 5. High surface area, leading to higher water and internal friction requirements, due to the shape of the particles, explains the drop in workability caused by the incorporation of ceramic.

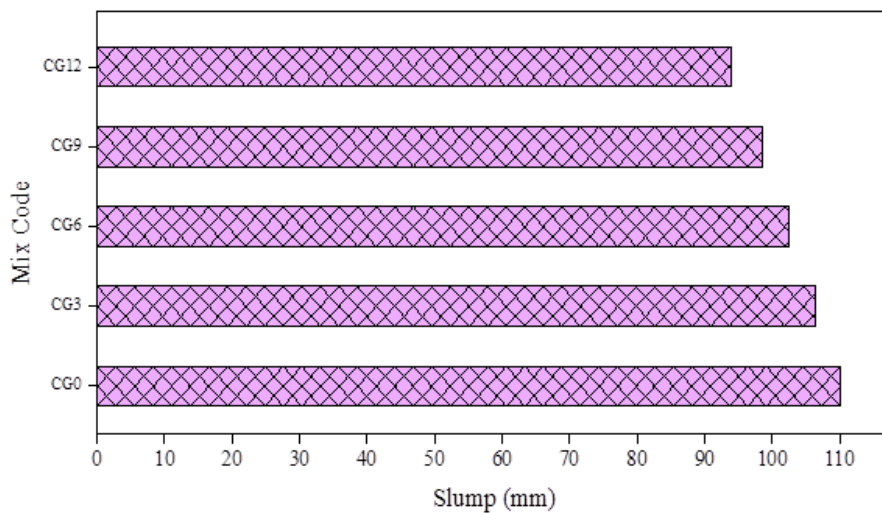


Fig. 5. Slump values of geopolymer concrete with varying CWP content

#### 3.2 Compressive Strength

The binder (which was 0%, 3%, 6%, 9%, and 12%) was subsequently substituted with the ceramic powder in small steps of 3%. This progressive increase was to establish the optimum ratio that produces the maximum compressive strength by assessing the variation in the mechanical properties, especially the compressive strength, of the resultant mixes at both 7 and 28 days of ambient curing. The results of compressive strength, as shown in Figure 6, indicate that the development of concrete strength is not linearly related to CWP content. The control mix (0% CWP) had a compressive strength of 24.37 Mpa, which has been used as the starting point of assessing the influence of CWP inclusion. On substituting 3% of the binder with CWP, the strength plummeted to 10.25 Mpa. This significant drop can be attributed to either a poor dispersion of the powder in lower dosages or possible interference of the geopolymerization reaction by insufficient supply of reactive aluminosilicates and alkaline activators, low level of ceramic powder, with insufficient dosage of aluminosilicate to fully develop the geopolymerization reaction to increase the gel formation. The CWP of this level can also cause particle agglomeration, which leaves weak areas in the matrix and causes the loss of strength.

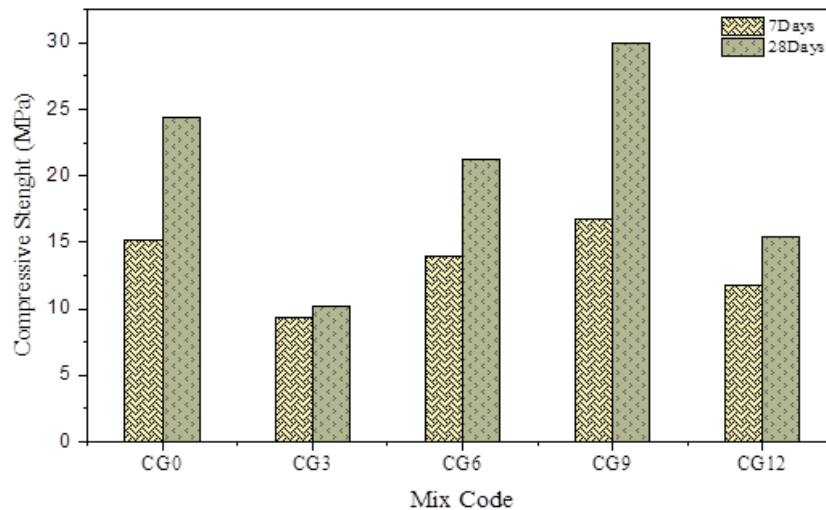


Fig. 6. CWP Optimising level based on compressive strength

This study found that strength rose rapidly at a 6% CWP replacement to 21.2 MPa, indicating a better CWP incorporation into the geopolymer matrix at this dosage, possibly because of a better distribution of particles and increased pozzolanic activity. The CWP starts to play a productive role at this stage as a site of nucleation of further development of geopolymer gels, and hence makes the matrix dense, enhancing the mechanical interconnection between the components. Nevertheless, the greatest strength was recorded to be at 9% CWP replacement, and the compressive strength was highest at 29.96 Mpa, which is much higher than the control mix. This finding shows that 9% is the most ideal amount of CWP replacement in the tested conditions because it gives the most favorable proportion between the particle packing, gel formation and microstructural refinement. Since the CWP was increased by 9%, the percentage could be due to the filler effect that increased the pozzolanic reactivity and the densification of the matrix. CWP might also be the cause of a finer pore structure that seals the small holes and enhances bonding between the binder matrix due to these interactions. Its greater strength implies that CWP is not purely a filler, but rather an active component of the geopolymerization process and in particular, when applied in the appropriate dose. CWP contributes to a finer and refined pore structure, which is well graded, and the voids between the large particles are filled.

On the other hand, a further increase of CWP to 12% decreased the compressive strength to 15.41 Mpa, therefore, showing that a high level of ceramic content above 9% would potentially be a factor in influencing the concrete strength and diminishing its mechanical strength. The surplus CWP at large replacement levels may serve as a non-reactive diluent, decreasing the concentration of reactive aluminosilicates required to form an effective gel of geopolymers. Additionally, excessive levels of powder may result in excessively large particle matrix, which may inhibit ionic mobility and thereby slow down or prevent the polycondensation reactions which underlie the development of strength. Increased water requirement at elevated CWP levels may also cause a lack of adequate availability of activators per unit mass, which could result in incomplete polymerization.

Comprehensively, the compressive strength measurements indicate that there is a point where CWP replacement results are more harmful than beneficial. The best fitting level, according to the present outcomes, is clearly 9% wherein both a filler effect and chemical contribution of CWP are maximized. The discovery is of significance, especially when it comes to sustainable concrete design, since it shows the possibility of using materials derived out of ceramic waste not only to improve the environment but also to improve mechanical performance.

### 3.3 Splitting Tensile Strength

Splitting tensile strength test gives a vital understanding of the behaviour of ceramic-modified geopolymer concrete under tension. The tensile strengths of the unmodified mixture (CG0) were

1.82 Mpa at 7 days and 2.31 Mpa at 28 days, and were used as a control group. When using CWP, significant tensile performance increase was also performed, which was credited with increased matrix cohesion and stress transfer mechanisms. In the case of the CG3 mixture with 3 percent CWP, tensile strengths were recorded to be 1.20 MPa (7 days) and 1.54 MPa (28 days). These findings indicated that the control had a small but significant improvement and that low concentrations of additives of ceramic may have a beneficial influence on the integrity of the microstructure.

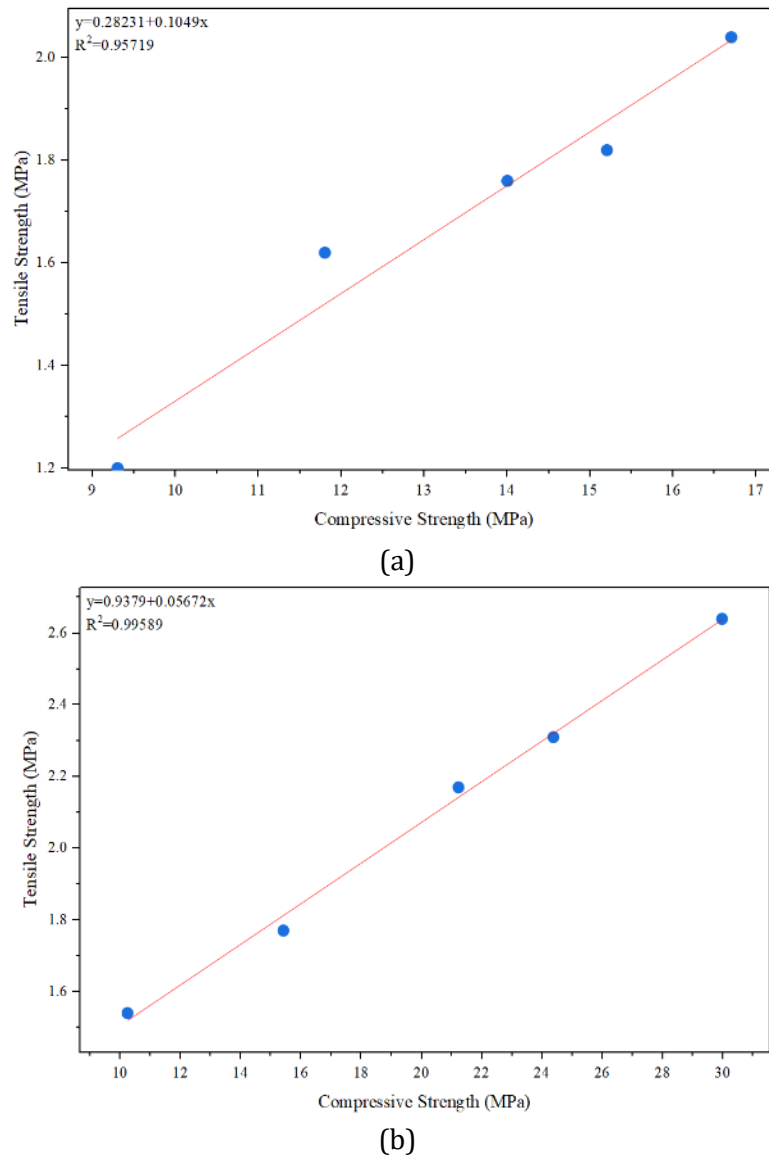


Fig. 7. The relationship between tensile and compressive strength (a) 7 days and (b) 28 days

Moreover, the CG6 mixture was seen to be more improved after 7 days at 1.76 MPa and 2.17 MPa, 28 days onwards. These findings showed that the effects were more outstanding as the ceramic content increased. It was also found that when CWP was contained in the CG9 mixture (CG9-R0) at 9% the tensile strengths increased (2.04 MPa) after 7 days and 2.64 MPa after 28 days, and thus proved that it is possible to increase the tensile strengths when the particle dispersion and matrix densification are well balanced. It should be mentioned that the composition and formula of the mixture play a pivotal role in determining the capability of the material to scatter stress load and, at the same time, reduce microcracking and therefore improve tensile resistance of the ceramic-only mixes. Precision of the formula is relevant in improving the sustainability and durability of materials since the performance of the CG12, which had 12 percent CWP, dropped a little with tensile strengths of 1.62 MPa after 7 days and 1.77 MPa after 28 days. Although the ceramic content recorded higher scores than in the control, unlike CG9, the performance of the ceramic content was

lower when it contained large amounts of ceramic content, and therefore, indicates that the ceramic content has an impact on the tensile strength and workability of the material.

The findings in general demonstrate that ceramic waste powder improves the tensile behaviour of geopolymer concrete up to a certain optimum. The optimal replacement level of 9% (CG9) performed best, and increasing dosage produced a negative effect. The above findings justify the establishment of ceramic additives as an effective approach to enhancing tensile strength in geopolymer systems without rubber reinforcement. The compressive strength and splitting tensile strength in the current study were directly related and had a very good correlation of 0.95, as shown in Figure 7.

### 3.4 Flexural Strength

The flexural strength test is used to assess the ability of the concrete to withstand stresses caused by bending and fractures, which is especially applicable in structural components (beams, slabs, and overlays) that experience tensile stress under the effect of external loads. This test is used to supplement compressive and tensile strength tests that provide a more comprehensive picture of concrete performance during flexural loads. Figure 5.11 demonstrates the flexural strength development of ceramic-only geopolymer concrete mixtures of 7 and 28 days. Control mixture (CG0) recorded the strength of 3.5 Mpa and 4.8 Mpa in 7 days and 28 days, respectively and was used as a reference to compare against others.

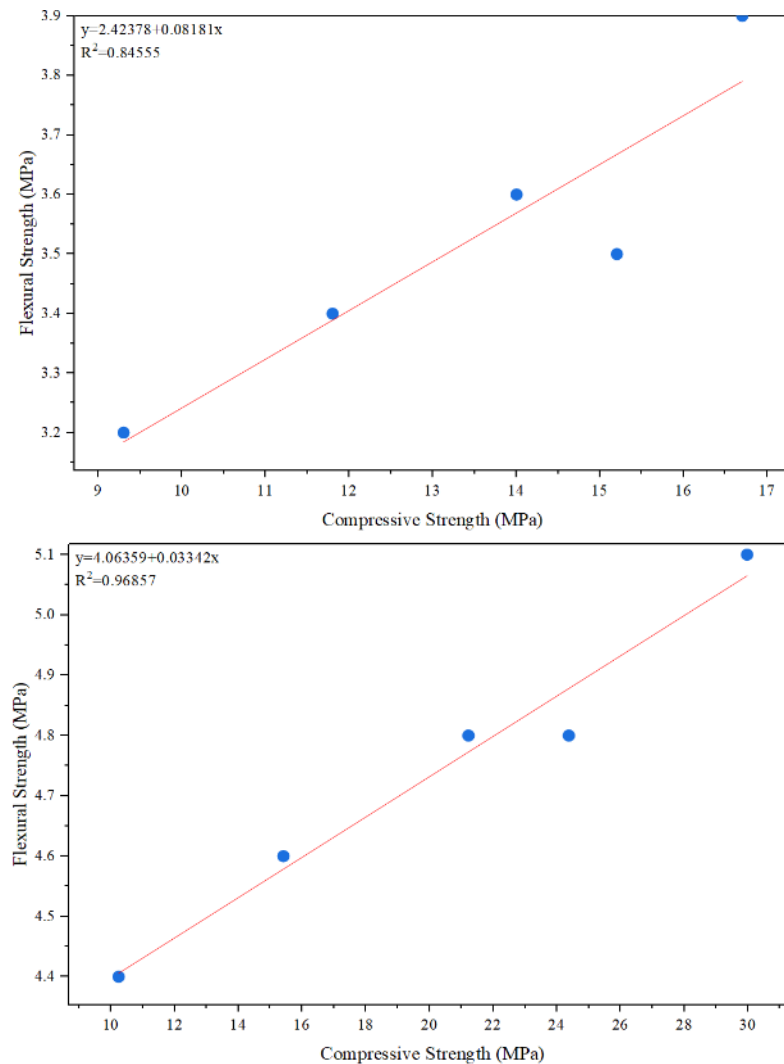


Fig. 8: The relationship between flexural and compressive strength (a) 7 days and (b) 28 days

With the addition of ceramic waste powder (CWP), the flexural performance was enhanced gradually with the increase of the replacement level. It was observed that CG3 with 3% CWP had

recorded 3.2 MPa flexural strengths on the 7<sup>th</sup> day and 4.4 MPa on the 28<sup>th</sup> day. These values were evidence that the mix is slightly improved in terms of its resistance to bends, which means that the particles of the mix were well packed, therefore making the matrix more cohesive. CG6 was the next mix that contained more data with 3.6 MPa on the 7<sup>th</sup> day and 4.8 MPa on the 28<sup>th</sup> day. These values also demonstrated that the mix was superior in spreading out the stress and minimizing the possible microcracking. In the case of the CG9 mixture, the flexural strengths were observed to be 3.9 MPa on the 7<sup>th</sup> day and 5.1 Mpa on the 28<sup>th</sup> day. The mix, as compared to the two previous mixtures, revealed that CWP at 0.09% is the prescribed concentration that enhances flexural performance since the CWP enhances matrix densification and sectional load transfer, which reduces the bending resistance.

Nevertheless, to gain a more appropriate understanding of the effect of CWP on the concrete mix, a different mix containing 12% CWP was subjected to the same conditions and time (7 and 28 days). The researchers determined that this mixture was reported to record lower values of 3.4 MPa on the 7<sup>th</sup> day and 4.6 MPa on the 28<sup>th</sup> day, as compared to the three past mixes. These figures implied that increasing CWP beyond the recommended amounts (0.09%) may impact the particle agglomeration and reduce the workability of the concrete, therefore, diminishing the flexural strength of the material.

Although the performance remains higher than the control, the deterioration compared to CG9 suggests that there would be a breaking point beyond which extra ceramic content would not be able to add more benefits. Concisely, the findings affirm the presence of ceramic waste powder, which improves and maximizes the flexural behaviour of geopolymer concrete. CG9 blend is the most balanced blend with better strength without the degradation of workability or structural integrity. The results justify the inclusion of ceramic additives in the mix design strategies to enhance the resistance to bending in geopolymer systems. A positive correlation existed between compressive strength and flexural strength, and the correlation was found to be very high, with an  $R^2$  of more than 0.84, as in Figure 8.

### **3.5 XRD Test**

In order to get a clear picture of the chemical reactivity of raw materials such as fly ash, GGBS, and CWP on the effect of undertaking the geopolymerization process, the XRD test enabled the researchers to identify and quantify the crystalline phases of GPC. The experiment revealed the appearance of new binding phases, e.g. N-A-S-H and C-A-S-H gels, which revealed the degree of the extent of the reaction, not to mention the presence of unreacted components left in the matrix. Its result also assisted in developing the understanding of the development of microstructure, its mechanical behavior, durability, and stability of the concrete and its capacity to resist external and internal stressors. According to the necessity to receive this input to form an appropriate OPC alternative, the role of XRD cannot be compromised, being the connector between the inner structure of the material and its physical and mechanical performance. XRD in this study context was charged with the responsibility of crystallographic analysis of solids, and it gave crucial details about those materials that had partial crystallographic or disordered phases. To obtain the accuracy of the test, the X-ray beam was directed at a sample surface, and the pattern of reflection was measured with reference to the scattering patterns and the arrangement of the rays emitted by the atoms of the crystals.

Such a pattern analysis enabled the correct identification of the crystalline phases, the lattice distances between the atoms (d-spacings) were determined and the correct determination of the amorphous matter crystallinity and crystalline ratios. This analysis is an important aspect of the investigation since the information gives an insight into the effect the additives have on the newly formed phases, how they alter the network structure of the geopolymer or enhance the structural composition and mechanical stability of the final product. As Figure 9 demonstrates, the XRD curves of both the GPC sample (base, that is, 0% CWP) and the corresponding mix (9% CWP) contained certain diffraction peaks. Such peaks represent the signs of different crystalline phases there and how much it is substantially connected with the primary components and side reactions of the products.

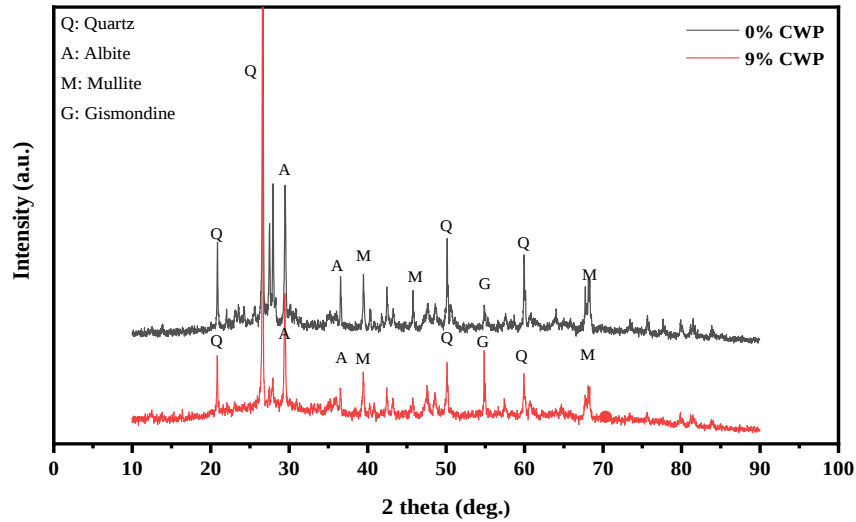


Fig. 9. XRD images of concretes containing 0% and 9% CWP

### 3.6 Fourier Transform Infrared Spectroscopy (FTIR) Test

Fourier Transform Infrared Spectroscopy (FTIR) is a well-known method of analysis applied to the identification of functional groups in solid substances. The technique is based on the fact that infrared energy taken up by chemical bonds in the material at certain wavelengths can be used to determine the quality of the bond and the extent of chemical reactivity or stability of the material. FTIR spectra provide a useful piece of information on N-A-S-H gel formation in geopolymer concrete as well as the character of chemical interactions between the constituents of the composite. As Figure 10 demonstrates, 0% base mix design, which consisted of no ceramic powder, had major peaks at particular positions that indicate the basic chemical structure of the material.

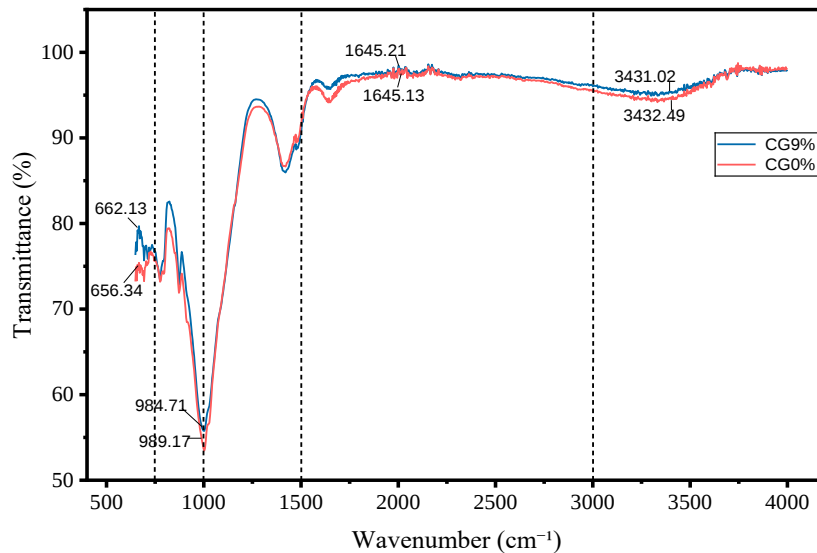


Fig. 10. FTIR spectra 0% ceramic powder and 9% ceramic powder

The analysis of the FTIR spectrum showed that it has a broad peak at  $3432.49 \text{ cm}^{-1}$ , which is associated with O-H vibrations of the adsorbed water or surface hydroxyl groups. The other sharp peak at  $1645.13 \text{ cm}^{-1}$  is associated with the H-O-H bending, which is a confirmation that crystalline water is also present in the network. The tallest peak was noticed at  $989.17 \text{ cm}^{-1}$ , which is Si-O-Al vibrations, which characterize the primary formation of geopolymer gel. Moreover, there exists a peak of  $456.34 \text{ cm}^{-1}$  indicating that the structure of the silicate-based geopolymer is composed of Si-O bending. Similar spectral features with only minor changes in the location of the peaks were

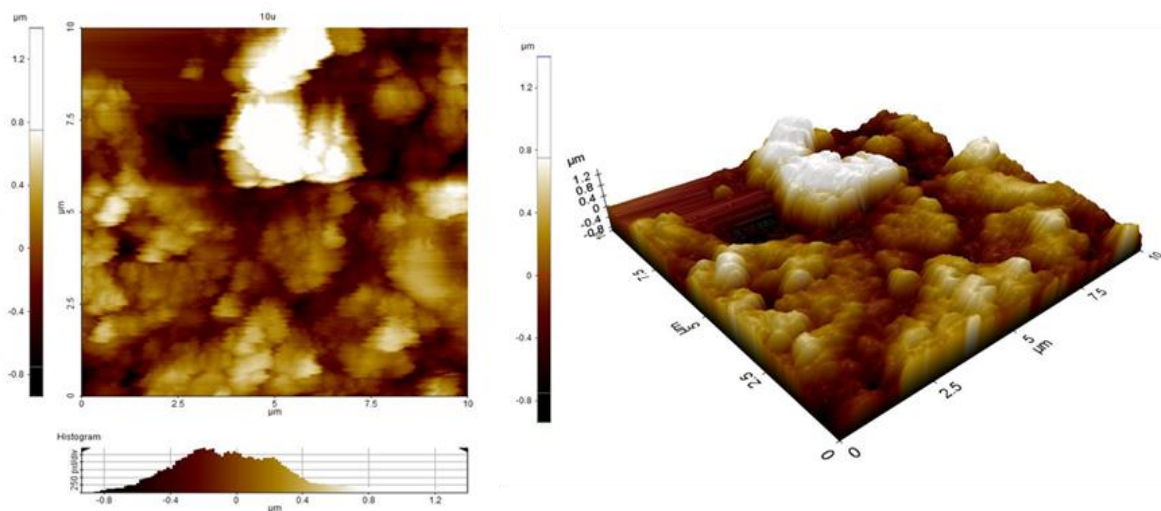
observed in the 9% CG sample that contains 9% CWP, which showed the effect caused by the additive on the gel formation.

The recorded O-H peak at  $3431.02\text{ cm}^{-1}$  was close to that of the reference sample, which showed that the moisture levels remained constant. Conversely, the H-O-H peak of  $1645.21\text{ cm}^{-1}$ , was consistent, which suggests the consistency in the content of crystalline water. Although these varied, the peak changed to  $984.71\text{ cm}^{-1}$ , and this variation revealed that there might have been an increase in the polymerization of the geopolymer matrix and thus enhanced its organization structure. The observation procedure also found that the difference in the Si-O bending peak was also found to be  $462.13\text{ cm}^{-1}$ , which means that the internal bonding was much stronger. Interestingly, in the comparative analysis of these samples, the researchers felt that the addition of 0.09% assisted in the improvement of the formation of geopolymer gel. The shift of the Si -O -Al peak into lower wavenumbers is an indication of the enhanced progress of the polymerization process, and thus forming more resilient, even bonds. Besides, the refined structure enhanced the strength of the concrete matrix, which enhanced the mechanical performance, including the high compressive and flexural strength. Comprehensively, it can be concluded that the internal crystallinity and molecular connectivity are increased in the ceramic powder in the concrete mix as opposed to the unmodified (0%) sample.

### 3.7 Atomic Force Microscopy (AFM) Test

The surface of the geopolymer concrete with varying concentrations of CWP is studied using an Atomic Force Microscopy (AFM) test. This test was utilized so as to see the microstructural change that had occurred due to the addition of these materials. The surface of the concrete without CWP (the control) was extremely rough and irregular. There were numerous high and low spots in the AFM images, indicating that the material was not highly compact. This kind of surface can make the concrete weaker and less durable. Figure 11 shows the base mix design without ceramic powder (CG0) and (CG9). The CG0 surface appears with sharp peaks and deep valleys. The average roughness (Ra) measures around 190.5 nm, while the root mean square roughness (Rq) reaches approximately 237.8 nm. Peak to valley height (Rz) is about  $1.20\text{ }\mu\text{m}$ , and valley depth (Rv) is near  $1.00\text{ }\mu\text{m}$ . This uneven topography reflects localized accumulation of geopolymer gel and fine pores, which concentrate stress at the surface peaks and make crack initiation under mechanical loading or thermal cycling more likely.

Figure 11 shows the geopolymer concrete containing 9 % CWP; the surface becomes noticeably smoother and more uniform. The Ra drops to 145.2 nm, and Rq decreases to 180.3 nm. Rz falls to  $0.90\text{ }\mu\text{m}$ , and Rv to  $-0.70\text{ }\mu\text{m}$ . This smoothing is due to the filling of the microscopic openings in the geopolymer structure by ceramic particles, which serve as nucleation and inhibit the development of large crystals and favor a finer and more coherent microstructure.



(a)

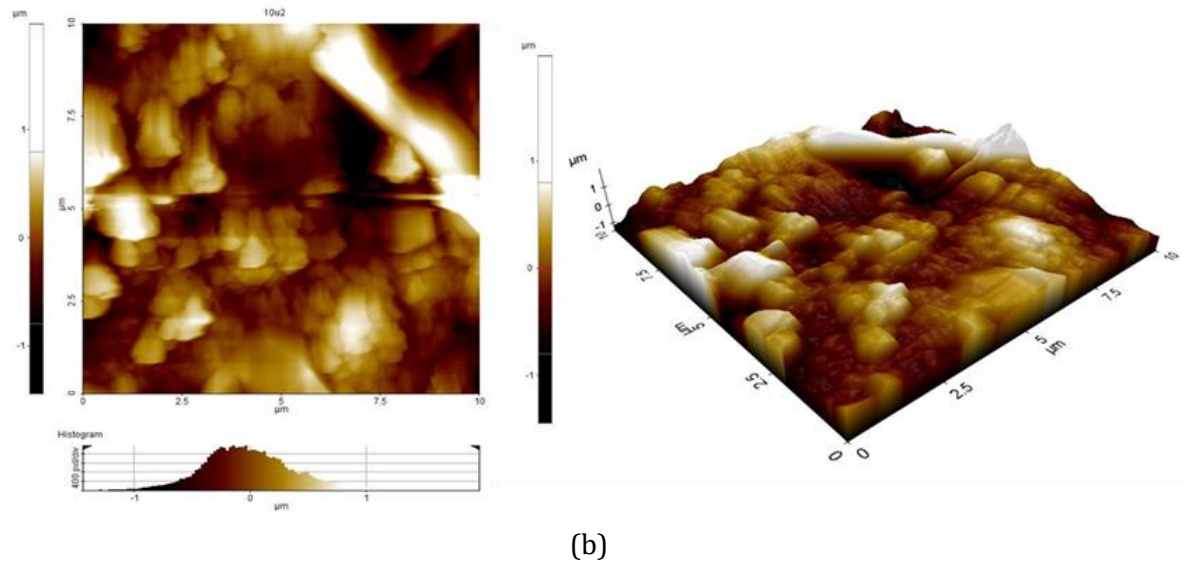
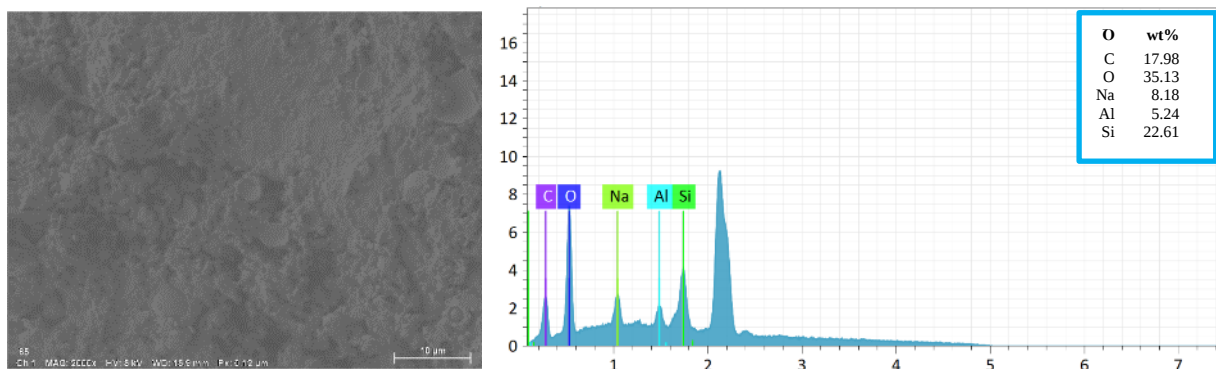


Fig. 11. 2D & 3D AFM images of (a) base mix design (CG0) and (b) concrete containing 9% CWP (CG9)

On the microlevel, the increased connection of ceramic particles to the geopolymer gel spreads mechanical loads more evenly over the surface, eliminating concentrations of stress. The fine microstructure also alleviates the development and propagation of microcracks under dynamic or thermal stresses. Furthermore, the overall porosity and water permeability are decreased by the void-filling effect of the ceramic, lowering deterioration due to the entry of moisture, varying temperatures or mechanical abrasion. Combined, these enhancements increase the service life of the geopolymer concrete and its performance in hostile or aggressive environments.

### 3.8 FESEM/EDX Test

The EDX spectra and map of modified geopolymer concrete with (a) 0% and (b) 9% of WCP are shown in Figure 12. The addition of WCP to the geopolymer concrete was found to have a positive effect on its surface morphologies. The geopolymer concrete generated using GBFS and fly ash exhibited a Si/Al ratio of 5.08, while the incorporation of 9% CWP increased the ratio to 7.17. This increase is the result of higher CWP silica content, which raises the  $\text{SiO}_2$  contribution in the matrix and differentiates the EDX outcomes between the two specimens. A Si/Al ratio of approximately 5.0 favours the coexistence of N-A-S-H and C-A-S-H gels, as the balanced availability of Si and Al is complemented by the reactive calcium present in GGBS [26]. However, the higher ratio of 7.17 obtained with CWP addition drives the system toward a silica-rich gel with a higher degree of polymerization but reduced Al cross-linking [27]. Such gels may enhance long-term densification and durability but may also reduce the efficiency of early-age binding and alter the mechanical response [28].



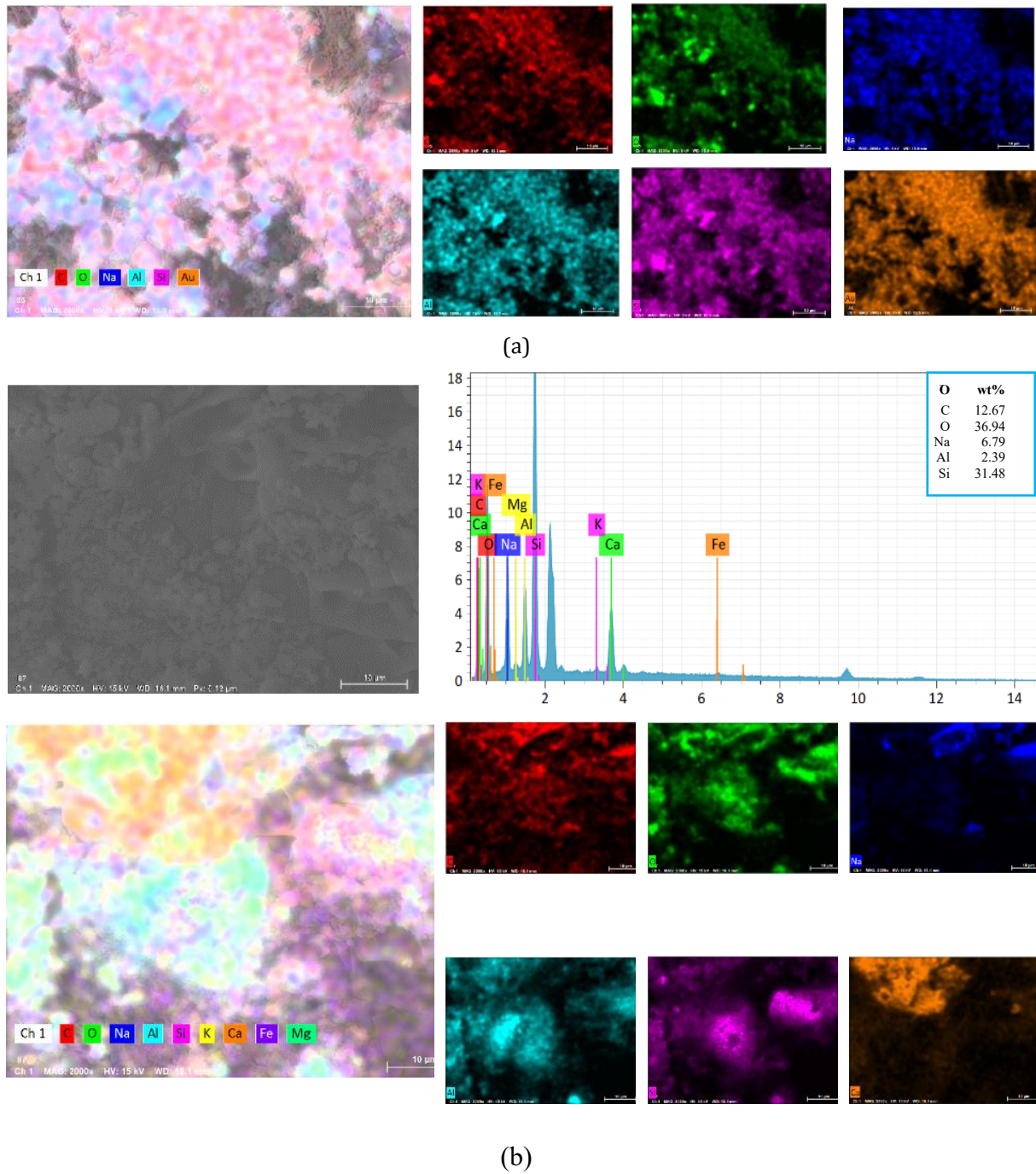


Fig.12. EDX spectra and elemental distribution maps of geopolymer concrete with (a) 0% and (b) 9% WCP

#### 4. Conclusions

The results of this present research can be summarised as follows:

- The results confirmed that the raw materials fly ash, GGBS, and CWP possess a chemical composition conducive to forming effective geopolymer binders. A high level of silicon and aluminium oxides in CWP was a contributing factor to the growth of sodium aluminosilicate hydrate (N-A-S-H) gels in the matrix.
- The optimum CWP replacement ratio was found to be 9% according to the experimental findings. This percentage provided the best compressive strength (29.96 0 Mpa) as well as improvements in the density of materials and internal structural bonding.

- The compressive strength results indicated that the CG9-R0 mix with 9% CWP had better performance in comparison with the other mixes, and it was approximately 23% above the reference mix CG0.
- The same trend and direct relationship was found between compressive and tensile/flexural strength, and that between the two was strongly correlated at above 0.84. XRD and FTIR analysis indicated that the internal structure of CWP modified blends was improved and that the presence of crystalline phases such as Mullite and Gismondine was responsible for the increase in hardness and reduction in permeability.
- The analysis of AFM also showed that CWP is effective in surface roughness reduction and homogeneous distribution within the structure, especially in the CG9 blend, which influences the cracking and the corrosion resistance positively.
- The incorporation of CWP increases the Si/Al ratio, shifting the binder toward a silica-rich gel that may enhance long-term durability but compromise early-age reactivity due to excess silica that delays the formation of the binding gel (N-A-S-H) by making dissolution of aluminosilicate less rapid with the alkaline solution at early age.

In conclusion, the addition of 9% of ceramic waste powder (CWP) to fly ash-GGBS-based geopolymer mixtures remarkably increases the mechanical strength, structural integrity, and durability of the product. The synergistic chemical and microstructural improvements, which are driven by increased Si/Al ratios and the formation of stable crystalline phases, confirm CWP's potential as a sustainable and effective supplementary material for geopolymer production.

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