Characterization and Properties of Binder and Nano-Filler in Geopolymer Paste with Graphene Oxide

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ABSTRACT

Globally, current research has developed new cement-based materials to meet increased demands for performance, energy efficiency, and environmental protection. Geopolymer, cost-effective, and high-early strength concrete binder alternative, has grown in popularity. Geopolymer reduces emissions by 80% during production while maintaining strength levels comparable to Ordinary Portland Cement (OPC). In their natural state, geopolymer binders have a microstructure that is cross-linked and significantly more brittle than OPC. To improve the properties of geopolymer, several approaches were adopted. However, recent study suggests that incorporating nanomaterials such as graphene oxide (GO) to geopolymer has shown an improvement in physical and mechanical properties. Graphene oxide is an inorganic nanomaterial that improves the mechanical characteristics of various composite materials by showing substantial filling effects on composite materials that significantly improve composite material integrity. The investigation into the potential of GO to improve the efficacy of geopolymer composite materials in various engineering applications has garnered considerable attention in recent years. The simplified hummer's method was employed to synthesise the GO. Various characterization techniques involving SEM, XRD, and FTIR were utilized to understand the crystalline structure and microstructure of GO nanoparticles. GO powder were used as 0.05%, 0.10%, 0.15%, 0.20%, and 0.25% by weight of binder that includes fly ash class F and steel slag with an optimum binder modification of 40% fly ash and 60% steel slag. The influence of GO on Bulk density, microstructure, and mechanical strength were determined. The compressive strength results indicated an improvement of compressive strength by 21.48% in geopolymer paste with 0.15% GO after 28 days of curing.

Keywords: Graphene oxide; nanoparticles; geopolymer concrete; sustainable construction materials

INTRODUCTION

Ordinary Portland cement (OPC) is the most significant basic material to produce concrete at the present time. However, cement production accounts for 8% of total anthropogenic carbon dioxide emissions and 36% of carbon emissions associated with the construction industry (Revilla-Cuesta et al. 2022). Therefore, it is necessary to develop alternative low-carbon binders. As a novel low-carbon binder, geopolymers are developed (Davidovits 1994). In the sustainable development of building materials, concrete technology provides greater energy efficiency, long-lasting durability, and low life cycle costs. In addition, it is crucial and necessary to expand sustainably

and develop binder resources, as the production of OPC results in high emissions. The possibility of replacing OPC component with geopolymer production to reduce emissions, as well as replacing cement with industrial by-products such as fly ash (FA), metakaolin (MK), ground granulated blast slag (GGBS), and waste agricultures such as sugarcane bagasse, rice husk ash (RHA) as the primary material to provide the aluminosilicate required for products and processes (Pyo & Kim 2017).

Geopolymer (GP) is a novel inorganic polymer and cement replacement made from aluminosilicate-rich waste materials and an alkaline activator solution (Koksal et al. 2022). Early-age strength, good resistance to sulphate attack, and stronger chemical resistance are some of the

technical benefits offered by geopolymer concrete (GPC) technology in comparison to OPC-based concrete. Other advantages include higher chemical resistance. All these factors contribute to the geopolymer technology's increased viability as a building choice (Shi et al. 2021). One of the major goals of researchers is to improve the demand for this material and come up with qualities that can be achieved via the use of a wide variety of additives to evaluate the improved performance of geopolymers. The vast bulk of research efforts have been directed towards revealing geopolymer's mechanical and microstructural characteristics. In addition, a variety of additives such as nanomaterials have been discovered to influence these characteristics.

Since the middle of the 2000s, nanoparticles like nano-silica (NS), carbon nanotubes (CNTs), and graphene oxide (GO) have been used to make progress on improved building materials for the 21st century (Anwar et al. 2020). Nanomaterials offer several benefits that increase the mechanical strengths and long-term durability of geopolymer, as a result, they are widely found in geopolymers and other types of polymers as fillers. This is because nanomaterials have many advantages over other additives that improve these properties (Koksal et al. 2022). According to Arpitha & Parthasarathy (2023) even at lower concentrations, nanoparticles can cause changes in the microstructure of GP, which ultimately leads to enhanced hardened properties. In order to improve the characteristics of geopolymers, nanomaterials are frequently utilised as additives. By modifying the reaction process and optimising the pore structure, nanomaterials, in general, can increase the mechanical strength and durability of geopolymers. Even though several studies have been conducted on the area of nanoparticles in cement-based materials, the most recent application advancement of nanomaterials in geopolymers still needs to be further investigated and examined especially incorporating of graphene oxide (GO).

The nanomaterial known as graphene oxide (GO) is made of carbon. In the year 1859, Benjamin C. Brodie was the first person to produce GO by removing graphite's surface layer by chemical means (Brisebois & Siaj, 2020). GO generally has a structure that is similar to a two-dimensional sheet, and because its thickness is significantly less than its length and breadth, it has a greater aspect ratio (Ranjbar et al. 2015). According to TEM investigations on GO for cement hydration, the wrinkled surface of GO has a larger surface area of due to the exfoliation of graphene oxide, which offers a wider contact area for binding particles (Lin et al. 2016). This is because the surface area is greater since there are more graphene oxide flakes. It has been demonstrated that GO can speed up the process of hydration without causing any changes to the oxygenated

functional groups that are connected to the GO nanosheets, indicating that it can work as a catalyst (Devi & Khan, 2020). An XRD analysis revealed that fly ash and slag-based GP had the highest concentration of at the highest GO content (Maglad et al. 2022a). This is because GO reacts with to produce more C-S-H gel, which speeds up the hydration process. In addition, the increased aspect ratio and filling capacity of GO both contribute to a reduction in the size of the gel pores and an increase in the microstructure's density (Zaid et al. 2022). Numerous research has concluded that the incorporation of nanoparticles into GP may significantly enhance the mechanical characteristics of the material as a result of the improved microstructural features obtained. However, there is limited comprehensive study on the effect of GO on the properties of GP. Two distinct physical forms of GO have been utilized by other researchers: pure GO, also known as powder GO, and liquid GO, which refer to as aqueous solution GO. Considering the additional characteristics of both physical forms of GO, including dispersibility, homogeneity, consistency, workability, and final properties like mechanical properties, influences the composites. The rheological properties of both varieties within the composite determine their flow and dispersibility (Shamsol et al. 2024). As an additive to geopolymer composites, the proportion of pure GO/powder GO ((Ho et al. 2021; Maglad et al. 2022a; Zhang & Lu, 2018) is typically lower compared to the proportion of liquid GO (Bellum et al. 2020; Candamano et al. 2019), which possesses a dispersibility concentration.

In the past two decades, geopolymers have been at the centre of research, and numerous reports and evaluations have been written about them. Typically, the focus of these publications is on the implications of partial or total binder replacement, use of high-density recycled aggregates, etc. According to the authors, there have been few studies to date that provide a comprehensive evaluation of the mix design and properties of lightweight geopolymer concretes (Tale Masoule et al. 2022).

METHODOLOGY

MATERIAL

The main aluminosilicate source material in the production of geopolymer paste are class F fly ash (FA) and steel slag (SL). FA is a residual substance that is generated via the combustion of coal, namely bituminous coal. This specific form of fly ash exhibits a significantly reduced proportion of calcium oxide. While, SL is a byproduct of the blast iron furnace, is commonly utilised as a binder material in several technical applications. Both binders combined with an

alkali activator solution, 12M sodium hydroxide (NaOH) and sodium silicate () to create a mixture of binder formulation. Varying proportions of graphene oxide (GO) is added to the optimum binder formulation to create the mix of binder modification.

SYNTHESIS OF GRAPHENE OXIDE

The graphene powder given has been synthesized to graphene oxide powder using simplified hummer's method which requires the use of chemicals such as sulfuric acid, potassium permanganate, hydrogen peroxide, and hydrochloric acid. The process of obtaining graphene oxide from graphene involved the utilisation of a simplified and environmentally friendly version of the Hummers method. Specifically, 1 g of graphene powder was mixed with 34 ml of 98% concentrated sulfuric acid () and subjected to 30 minutes of stirring on a magnetic stirrer. Subsequently, a quantity of 4.5 g of potassium permanganate () was gradually introduced into the suspension over a period of 30 minutes. This was succeeded by the gradual addition of 200 mL of 5% concentrated sulphuric acid to the mixture, followed by the addition of 200 mL of 6% hydrogen peroxide to terminate the reaction. The mixture is continuously stirred for an additional duration of 2 hours. Subsequently, the combination solution undergoes a triple washing process utilising a 10% hydrochloric acid solution. This is achieved through centrifugation at a speed of 3000 revolutions per minute (rpm) for a duration of 10 minutes. Next, the surplus solution is eliminated, and the resulting supernatant is gathered. The liquid portion is moved to a sterile petri dish and undergoes a drying process in an oven for a duration of 24 hours, kept at a temperature of 80°C. Finally, the production of dried graphene oxide is achieved, followed by a milling procedure to obtain particles of nano size.

PARAMETER

The study's design can be divided into three distinct phases. Phase 1 encompasses the preliminary data pertaining to the characterisation and composition of the raw materials. The main goal of material characterization is to obtain a comprehensive understanding of the properties and behaviour of various materials, including fly ash (FA), steel slag (SL), and graphene oxide (GO). Phase 2 is a comprehensive examination is conducted to evaluate physical geopolymer paste with (binder modification) and without GO (binder formulation). Subsequently, the introduction of phase 3 is anticipated, which encompasses the mechanical properties of geopolymer with and without GO.

The experiment is going to be carried out to investigate the impacts of several different percentages of graphene oxide (GO) on the geopolymer paste. Table 1 shows the parameter of this study. The experimental setup complies to the methods and procedures outlined in the American Standard Testing Method (ASTM) as stated in Table 2. Different proportions of fly ash-based and steel slag-based geopolymer paste and concrete will be prepared and each containing varying percentages of graphene oxide (GO) to investigate the properties stated in every phase of this study.

TABLE 1. Parameter of Study

Material	Parameter	Design
Precursor	Fly Ash: Slag Ratio	100:0, 80:20, 60:40, 40:60, 20:80, 0:100
Graphene Oxide (GO)	Percentage to the weight of binder (wt.%)	0.00, 0.05, 0.10, 015, 0.20, 0.25
Alkaline Activator	Solid: Liquid Ratio	Constant: 2.0:1.0
	Sodium Silicate: Sodium Hydroxide	Constant: 2.5:1.0

DATA ANALYSIS

PHASE 1: CHARACTERIZATION OF RAW MATERIAL

An analysis of the chemical composition, crystalline phase, and microstructure has been conducted to identify and assess the suitability of the primary raw materials, namely fly ash, slag, and graphene oxide. This section presents a discussion of the findings of the raw material characterisation using various types of testing and analysis, which are Scanning Electron Microscopy (SEM), Field Emission Scanning Electron Microscopy (FESEM), and X-ray diffraction (XRD). SEM is used to scan the image of microstruture for binder as binder has larger particles while FESEM is used to scan the image of GO powder for better imaging as GO is nanomaterial as FESEM has high resolution compared to SEM. The correlation between the raw materials composition, physical properties, and crystallographic characteristics was examined concerning the physical, and mechanical properties of geopolymer paste.
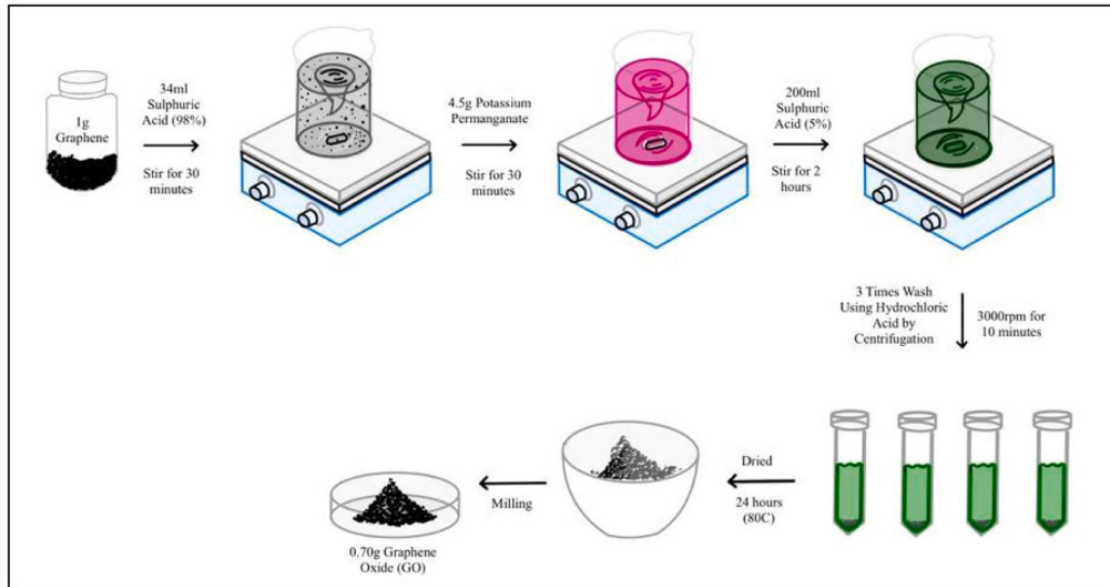
FIGURE 1. Schematic diagram of synthesis process of GO by using modified Hummer's method

SCANNING ELECTRON MICROSCOPY (SEM)

Figure 1 depicts the scanning electron microscopy (SEM) investigation on the class F fly ash and steel slag employed in this research. Based on the data shown in Figure 1, the depicted images exhibit a consistent presence of solid spheres, predominantly characterized by a hollow and glassy composition of fly ash. These spheres are interspersed by particles that have an uneven shape. Alternatively referred to as "miniature ball bearings," fly ash exhibits spherical-shaped particles, as depicted in the accompanying figure. These particles are recognized for their ability to contribute to a lubricating effect in composites.

TABLE 2. Mix Design

Mix Design	Aluminosilicate Binder (kg/m³)			Alkaline Activator		Molarity of NaOH (M)	GO Content (%)
	FA	SL	S/L Ratio	NaOH (kg/m³)	Na$_2$SiO$_3$ (kg/m³)		
Binder Formulation							
100FA	1600	-	2.0	228.57	571.4	12	-
80FA20SL	1280	320	2.0	228.57	571.4	12	-
60FA40SL	960	640	2.0	228.57	571.4	12	-
40FA60SL	640	960	2.0	228.57	571.4	12	-
20FA80SL	320	1280	2.0	228.57	571.4	12	-
100SL	-	1600	2.0	228.57	571.4	12	-
Binder Modification: Selected Binder Formulation of 40FA60SL							
M4			2.0	228.57	571.4	12	0.00
GO0.05			2.0	228.57	571.4	12	0.05
GO0.10	640	960	2.0	228.57	571.4	12	0.10
GO0.15			2.0	228.57	571.4	12	0.15
GO0.20			2.0	228.57	571.4	12	0.20
GO0.25			2.0	228.57	571.4	12	0.25

TABLE 3. Properties and Testing Required.

Phase	Properties	Testing	Standard
1	Characterization	Scanning Electron Microscopy (SEM)	PR
		Field emission scanning electron microscopy (FE-SEM)	PR
		X-Ray Diffraction (XRD)	PR
2 & 3	Physical	Bulk Density	ASTM C138
	Mechanical Properties	Compressive Strength	ASTM C109
		Tensile Strength	ASTM C496
		Flexural Strength	ASTM C78
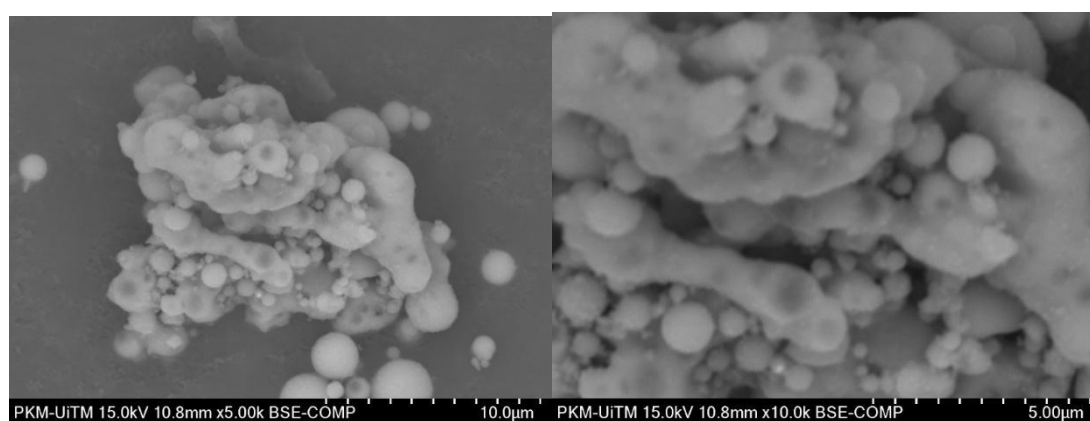
FIGURE 2. SEM Images of Fly Ash

The SEM images of steel slag (SL) in Figure 2 shows the outside of SL has a roughness that can be described as granular. The size of the grains can differ from one another, and the forms of the grains themselves may be varied across the board. Sharp edges in steel slag, which may be attributable to the crushing that occurs during the milling process that is part of the manufacture of steel slag. The morphology of slag particles nearly always looks the same in terms of size distribution when viewed at a higher magnification, which is evidence of the heterogeneous nature of the particle.

FIELD EMISSION SCANNING ELECTRON MICROSCOPY (FESEM) OF GRAPHENE OXIDE

Figure 3 displays the field emission scanning electron microscopy (FESEM) images of the acquired graphene oxide (GO). The figure presented provides evidence that the morphology of the GO is planar. As anticipated, graphene oxide (GO) exhibits a stratified arrangement, characterised by plane thicknesses ranging from 40 to 50 nanometers. In better view, Figure 4 shows that GO exhibited a layered structure and formed an ultrathin film that exhibited folding in space. The structural characteristics of graphene oxide (GO) have been observed to consist of planar flakes or layers. The Field Emission Scanning Electron Microscopy (FESEM) image reveals that graphene oxide (GO) tends towards a disordered solid state, characterized by aggressive aggregation and thin, closely spaced layers. This phenomenon can alternatively be interpreted as a collection of closely interconnected layers.

Additionally, it was shown that the wrinkled appearance of graphene oxide (GO) sheets contributed to enhanced adsorption capabilities. The existence of oxygen functional group interaction during synthesis, particularly in the oxidation process, is indicated by the layered structure and wrinkled appearance of GO. The wrinkled morphology of graphene oxide provides extensive surface areas and promotes interfacial solid interactions with the geopolymer composite mixture. The wrinkled appearance of GO enhances the mechanism for transferring loads throughout the concrete, leading to improved mechanical properties (X. Liu et al. 2020).
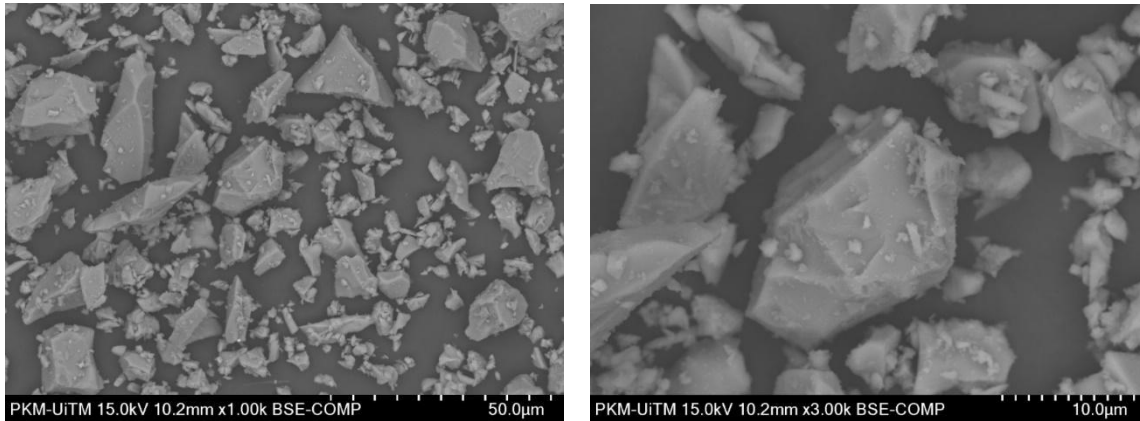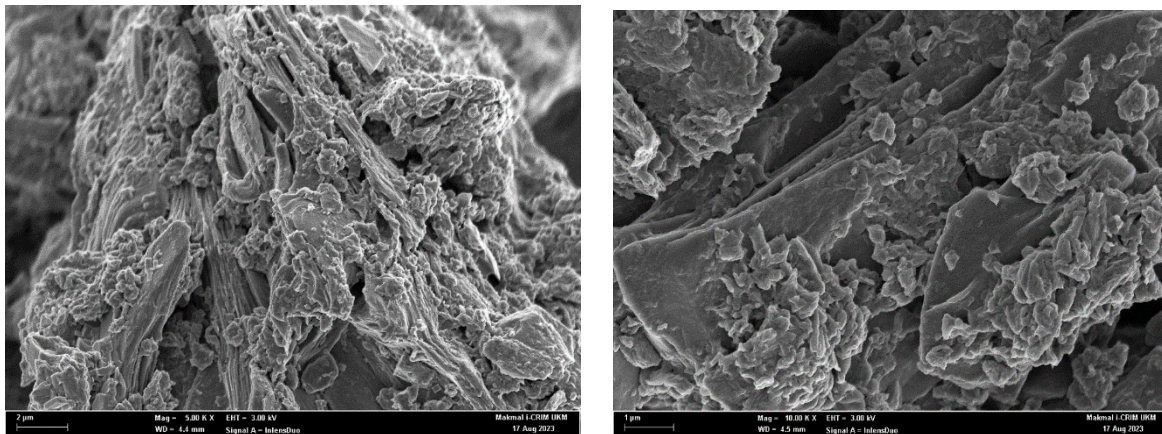
FIGURE 3. SEM Images of Slag

FIGURE 4. FESEM Images of Graphene Oxide

X-RAY DIFFRACTION (XRD)

The X-ray diffraction (XRD) data presented in Figure 4, Figure 5, and Figure 6 indicate the peak composition of fly ash (FA), steel slag (SL), and graphene oxide (GO) respectively.

Figure 4 shows the XRD of FA that primarily consists of both amorphous and crystalline phases, including quartz () and sillimanite (). Diffraction analysis of FA samples indicated that the reaction products were mainly amorphous and crystalline phases where the presence of both of the phases confirms that FA can be used as a raw material for geopolymer synthesis, which was confirmed by a broad and diffuse reflection in the range of 10 to 90 2 In addition, from the FA spectrums, the hump corresponding to the amorphous phase increases in the FA spectrum which can be attributed to the formation of the quartz phase.

According to the findings shown in Figure 5, it is evident that the structure of SL can be characterised by two distinct crystalline perspectives, specifically referred to as the amorphous and crystalline phases. The slag material contains significant mineralogical phases, including calcium silicate (, with a 2θ value of 29.433) and epidote (with 2θ values of 32.306). Consequently, the presence of a substantial quantity of Calcium Silicate facilitates the formation of an optimal conditions for significant hydration compounds, ultimately resulting in enhanced binder strength. When comparing FA and cement, it is evident that both materials exhibit similar characteristics and behaviour, making them potential substitutes for one another. Nevertheless, SL exhibits a higher degree of amorphousness and has a greater amount of amorphous substance in comparison to FA. This study indirectly assesses the disparity in the effects of these two precursor advantages on the mechanical robustness of the geopolymer matrix.
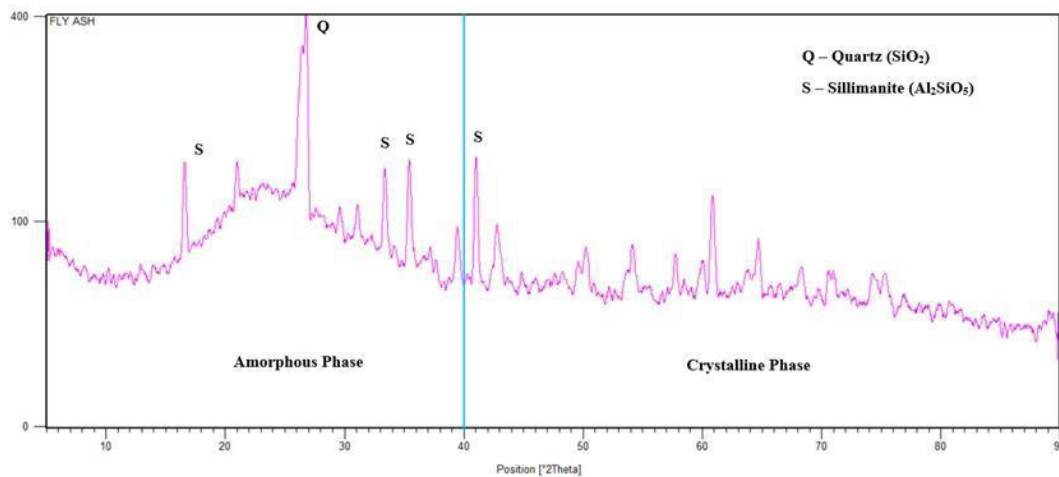
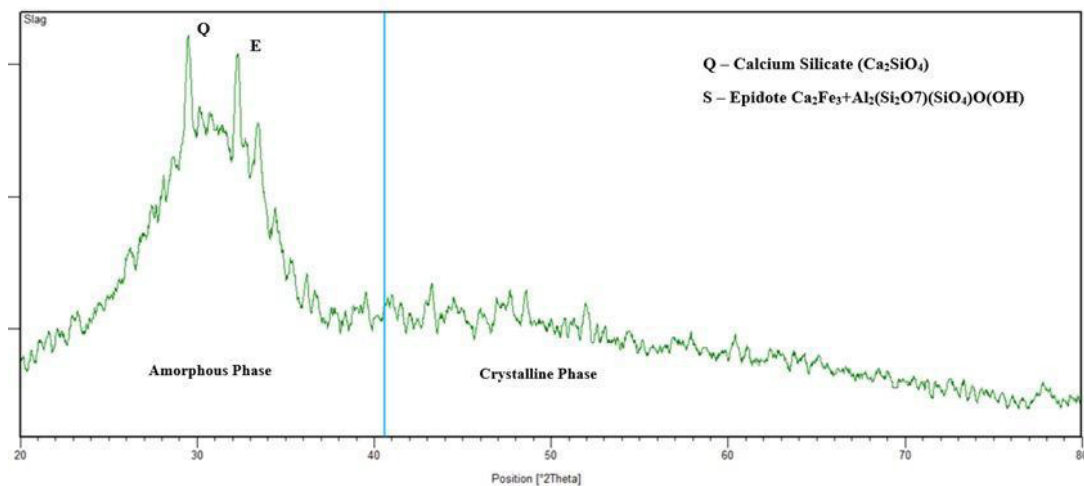FIGURE 5. Phase characterization of Fly Ash

FIGURE 6. Phase characterization of slag

Figure 6 shows the GO pattern suggested a wide diffraction peak based on the data, and it started at 7.099° 2 with the separation between the plane equal to 12.54 A. It is possible to explain this by noting that an increase in the spacing between fields in the GO pattern denotes the existence of oxygen-functional groups in the substance being examined. This can be made into a more detailed illustration, such as the one seen in Figure 6. In addition, the phase of the material, which might be either crystalline or amorphous, can be established with the use of these measurements. In general, the crystalline phase signifies an increase in the durability of the concrete since it decreases cracks of any size, from the smallest to the largest. This suggests that the use of GO as a nanofiller material is sufficient to meet the requirements necessary to close the gap in the study. In addition to that, GO encourages an effect known as the amorphous phase effect, which contributes to the creation of C-A-S-H having more strength. This invariably results in an increase not just in the mechanical but also the physical strength of the concrete. In addition, the XRD pattern revealed the presence of a broad diffraction band with a crystalline peak composed of carbon element.

X-Ray Diffraction (XRD) test conducted by Hulagabali et al. (2023) shows the diffraction peak of graphene oxide (GO) can be attributed to a specific crystallographic plane as it exhibited a pronounced and well-defined intensity at an angle of 11.68∘. This observation suggests the presence of a GO sample that has undergone significant oxidation. The presence of intercalated water molecules and the inclusion of oxygen function groups are present as the interlayer spacing of the GO is increased.
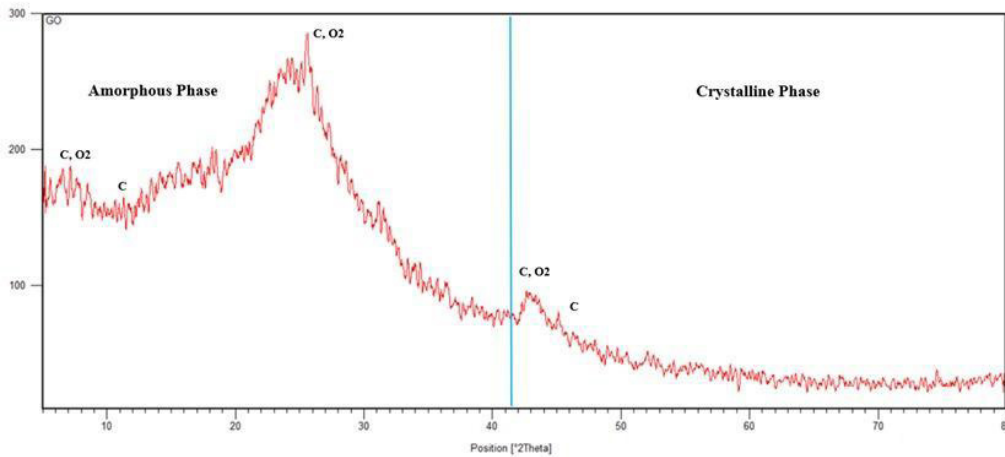
FIGURE 7. Phase characterization of Graphene Oxide

BULK DENSITY

Figure 7 shows graphs that reflect the bulk density of binder formulation, which includes the replacement of fly ash (FA) and steel slag (SL) in the geopolymer paste. In addition, the data gathered refers to the mean bulk density of samples with various percentage ratios of FA:SL, including 100:0, 80:20, 60:40, 40:60, 20:80, and 0:100, with recorded mean data of 1.566 , 1.591 , 1.610 , 1.673 , 1.69 , and 1.71 , respectively . In a scientific sense, the trend of the bulk density rose in a linear fashion with each successive addition of SL binder. When compared to FA, SL has a higher density and a higher specific gravity than FA, which may help to explain this phenomenon. The result shown in the Figure 8 depicted the bulk density of modification formulation of 60FA40SL with various GO replacement content of 0.05%, 0.10%, 0.15%, 0.20% and 0.25%. On the overview data, it can be concluded that the average reading for both materials is nuanced or varied. This can be explained geopolymer paste with GO tends to achieve higher readings compared to without GO. There is no significance different in bulk density as the additive or GO is nanomaterial and has low in density and its incorporation in geopolymer paste is low.
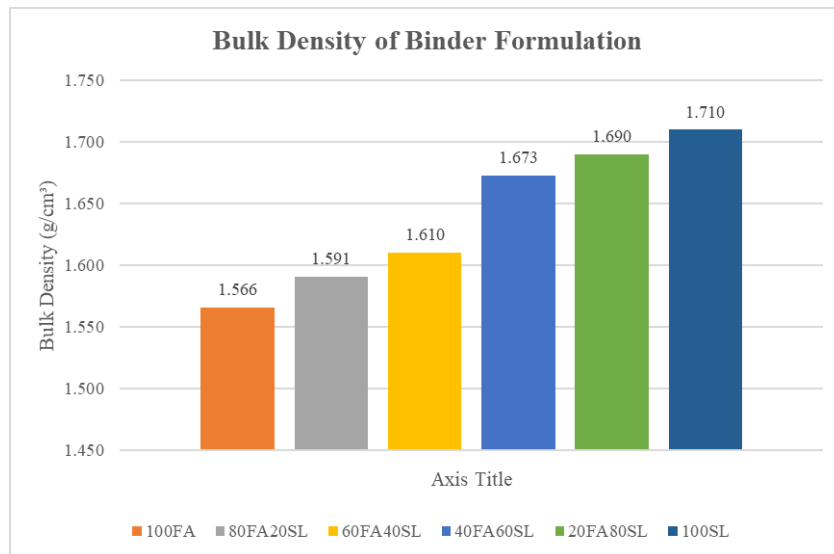

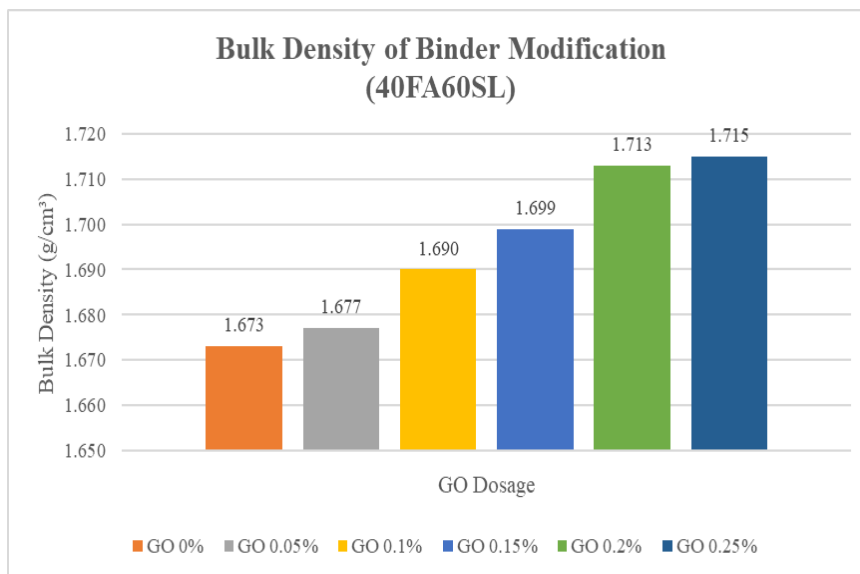FIGURE 8. Bulk Density of Binder Formulation

FIGURE 9. Bulk Density of Binder Modification

PHASE 3: MECHANICAL PROPERTIES

COMPRESSIVE STRENGTH

Figure 9 depicts the compressive strength of fly ash-slag geopolymer binder with difference proportion of fly ash to slag ratio (FA:SL) of 100:0, 80:20, 60:40, 40:60, 20:80, and 0:100. The strength of geopolymer binder has increased at an early and later ages with the addition of slag. The strength of geopolymer binder samples was investigated at 7, 14, and 28 days and shown in Figure 9. The geopolymer binder mixes were evident for continuous strength development over the age from the date of ambient curing. Mainly the compressive strength of geopolymer binder samples is dependent on physical and chemical interaction between alkaline activator and the binder matrixes. The addition of slag has shown enhanced strength properties of the matrix. At 28 days, replacing 20% of FA with SL (80FA20SL) increased compressive strength by 7.26%. However excessive amount of SL (100% SL) has shown reduced the compressive strength by 8.25% at 28 days, but it has a better result compared to fly ash geopolymer binder. The compressive strength of 60% addition of the slag (40FA60SL) has attained an optimum binder formulation of 27.80MPa (+58%), 35.70MPa (+40%), and 42.20MPa (+16.83%) for 3, 7, and 28 days of ambient curing, respectively as shown in Figure 9. The presence of a higher amount of the calcium in slag and the connective to the additional of C-A-S-H gel formation could be a further reason for the enhanced compressive strengths for the mix 60FA40SL. Decreases in the percentage of compressive strength by the curing days for every type of binder combination are caused by the mixture's microstructure exhibiting early strength development (Liang & Zhang 2020).

Figure 10 displays the compressive strength of fly ash-slag geopolymer binder mixed with 0%, 0.05 wt.%, 0.10 wt.%, 0.15 wt.%, 0.20% and 0.25 wt.% inclusions of graphene oxide (GO) powder at different curing days (3, 7, and 28 days). The geopolymer binder strength is enhanced at yearly and later ages with graphene oxide addition. The compressive strength of graphene oxide-geopolymer binder specimens mostly relies on the chemical and physical interaction between the geopolymer matrixes and GO. A mechanical interlacement between the geopolymer binder and the wrinkled surface structure of the nanosheets of GO can possibly explain the enhancement in the strength characteristics obtained. The GO can possibly also behave like filler material in geopolymer matrix and developing a dense structure, which paves the way to the improvement of compressive strength in the initial days. The replacement of 60% FA with SL into specimens of geopolymer binder displayed improved strength characteristics with GO of less than 0.15% and when GO exceeds 0.15% the compressive strength slightly decreases when compared to the matrix with 0.15% of GO. However, the strength remains high when compared to sample without GO. The little amount of GO inclusion in geopolymer binder the compressive strength attainment increased drastically in all curing ages. Geopolymer binder with 0.15% GO obtained 38.90MPa (+39.92%), 45.30MPa (+26.89%) and 51.10MPa (+21.48%) for ambient curing of 3, 7, and 28 days. A 0.15% addition of GO has shown better performance than all other levels. The higher

strengths were attained due to the greater surface area and wrinkled. However, a 0.10% inclusion of graphene oxide seems to be an inflection point as displayed improved performance compared to other levels. The increase strength was obtained because of the wrinkled morphology and increased surface area of graphene oxide nanosheets. Because of the surface structure, the interconnected mechanism inside the mixtures of geopolymers was enhanced. The maximum strength was obtained at the inclusion of 0.15% graphene oxide by the binder weight utilized in the geopolymer binder. According to (Maglad et al. (2022b); (Bellum et al. (2020a), The addition of GO results in a significant increase in strength due to the enhanced formation of calcium aluminosilicate (C-A-S-H) gel. The hydration process was accelerated by the reaction of GO with calcium hydroxide content in alkaline activator, which produced an amorphous gel of calcium silicate hydrate.
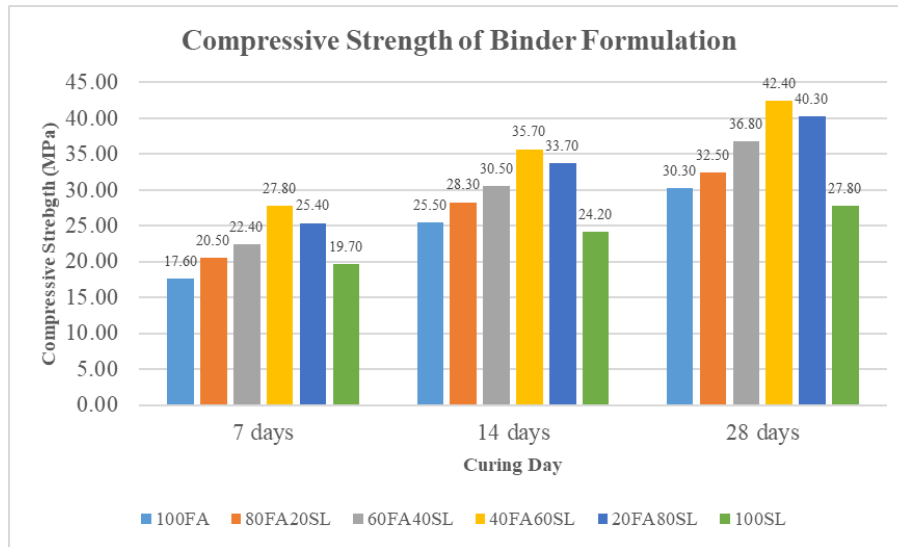

FIGURE 10. Compressive Strength of Binder Formulation
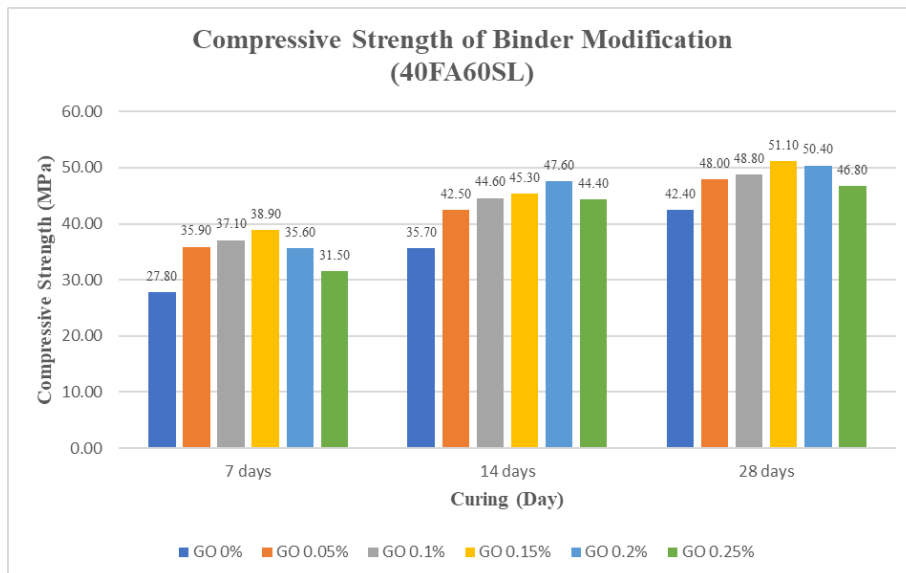

FIGURE 11. Compressive Strength of Binder Modification

CONCLUSION

This article establishes an insight into utilizing GO as an additive in geopolymer composites. Critical analysis of the literature and the data revealed the research outcomes as follows:

1. Admixing an appropriate dosage of GO has been proven to be beneficial to the mechanical properties of geopolymer paste especially in high percentage of slag precursor, given that the GO are dispersed reasonably well.

2. GO functions as a catalyst, accelerating the hydration process while leaving the oxygenated functional groups attached to the GO nanosheets unmodified.

3. The study emphasizes the promising potential of GO as a carbon-based material for improving the properties of geopolymers, thereby contributing to the development of more sustainable building materials.

4. At 28 days, replacing 20% of FA with SL (80FA20SL) increased compressive strength by 7.26%. However excessive amount of SL (100% SL) has shown reduced the compressive strength by 8.25% at 28 days, but it has a better result compared to fly ash geopolymer binder.

5. Geopolymer binder (40FA60SL) with 0.15% GO obtained 38.90MPa (+39.92%), 45.30MPa (+26.89%) and 51.10MPa (+21.48%) for ambient curing of 3, 7, and 28 days. A 0.15% addition of GO has shown better performance than all other levels.

6. Decreases in the percentage of compressive strength by the curing days for every type of binder combination are caused by the mixture's microstructure exhibiting early strength development.

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DECLARATION OF COMPETING INTEREST

None

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