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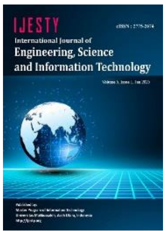

Effect of Carbon Nanotubes and Graphene Oxide on the Hydration Characteristics and Compressive Strength of Cement Mortar with High Volume Fly Ash

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Abstract

High-volume fly ash (HFVA) in cement mortar has gained attention due to its potential to reduce environmental impact and enhance sustainability. However, HVFA mortar often exhibits reduced initial compressive strength compared to conventional mortars. Incorporating carbon nanotubes (CNT) and Graphene oxide (GO) has been shown to enhance the mechanical properties and microstructure of the mortar, which can overcome the shortcomings of HVFA. This study investigates the effect of CNT and GO on compressive strength and cement hydration of HVFA mortar. This study used fly ash as a 60% cement replacement, with 0.01% CNT and GO contents of 0.01%, 0.02%, 0.03%, 0.04%, and 0.05%. Compressive strength tests were performed at 3, 7, and 28 days of curing. The results showed that adding CNT and GO improved the compressive strength by 15.4% over the control mortar at 0.01% CNT and 0.03% GO. Most importantly, incorporating CNT and GO mitigated the typical reduction in initial compressive strength, with around a 15% increase observed at 3 and 7 days compared to the control mortar. The cement hydration characteristics were analyzed using X-ray diffraction (XRD), which revealed the presence of various crystallization phases, including calcium silicate hydrate (C-S-H), calcium aluminate silicate hydrate (C-A-S-H), quartz, portlandite, calcium carbonate, and ettringite. Adding CNT and GO to HVFA mortar significantly enhances its mechanical properties. It promotes the formation of complete chemical bonds in the cement hydration process, improving the mortar's overall performance.

Keywords: *Carbon Nanotube, Graphene Oxide, Hydration Characteristic, Compressive Strength, HVFA Mortar.*

1. Introduction

Cement mortar is commonly used in construction due to its strength, durability, and versatility. However, the production of Portland cement is associated with significant environmental concerns, primarily due to the high carbon dioxide (CO_2) emissions involved in its manufacture [1], [2]. To address these environmental impacts, the incorporation of supplementary cementitious materials, such as fly ash, has gained attention [3]. Fly Ash, a byproduct of coal combustion in power plants, is often used as a partial replacement for cement in mortar and concrete mixtures. High-volume fly ash (HVFA) not only improves the sustainability of cement-based materials but also enhances some properties, such as workability and durability, while reducing the carbon footprint [4], [5].

While using fly ash improves the environmental footprint of cementitious materials, it can also result in slower hydration and reduced early-age strength, which can limit the material's performance, particularly in applications where high early strength is required [6], [7]. To mitigate these limitations, incorporating nanomaterials like carbon nanotubes (CNTs) and graphene oxide (GO) has shown promising results. These nanomaterials have exceptional mechanical, thermal, and electrical properties, which can significantly improve the performance of cement-based composites [8], [9].

Carbon nanotubes (CNTs) are nanomaterials frequently used as additives to enhance cement-based composites' mechanical properties and microstructure. Their unique physical and mechanical characteristics make them promising for developing composites with improved performance and functionality [10]. The mechanical performance of cement mortar is influenced by the structural elements and phenomena occurring at the micro and nano scales [11]. CNTs possess exceptional mechanical strength and thermal conductivity at the atomic level, making them highly effective in enhancing the resistance of cement to extreme pressure and weather conditions [12]. CNTs also improve the material's strength and crack resistance. The silica within CNTs is highly adhesive, making it difficult to disrupt the bonds that typically lead to gaps, thus enabling the binding of atoms at the nanoscale without causing cracking.

In addition to CNTs, graphene oxide (GO) is another promising nanomaterial, which contains oxygen-functional groups bound to its carbon atoms. This functionality makes GO hydrophilic, allowing it to disperse quickly in aqueous solutions. Each carbon atom in Graphene is covalently bonded to three adjacent carbon atoms through strong bonds, giving the material remarkable strength. When an external force is applied to GO, the surface atoms bend slightly to accommodate the force without causing any rearrangement of the carbon bonds. This allows GO to maintain a stable and robust structure under stress [13]. Studies have shown that adding GO to cement mortars can significantly improve their mechanical properties. For example, incorporating 0.05% by weight of GO into magnesium potassium phosphate cement paste resulted in a 6.8% increase in compressive strength and an 8.3% improvement in flexural strength [14]. In another study, adding just 0.03% of GO by weight of cement to mortar increased its compressive strength by 52.4% and its flexural strength by 34.3% [15].

The microstructure of a material is crucial to its performance, as the structure directly influences its physical, mechanical, and thermal characteristics. By examining the material's structure, one can also assess its workability. A common technique for this analysis is X-ray diffraction (XRD), which helps characterize the material's crystal structure. Diffraction methods are beneficial for identifying compounds with unknown molecular bonds in solids [16], [17].

This study investigates the combined effects of CNTs and GO on the hydration characteristics and compressive strength of cement mortar containing high-volume fly ash (HVFA). By studying these effects, it is possible to understand better how these nanomaterials can optimize the performance of fly ash-based cement mortars, making them more sustainable without sacrificing strength or durability. This exploration aims to develop more efficient, eco-friendly alternatives for construction materials with enhanced properties and lower environmental impact.

2. Literature Review

2.1. Mortar

Mortar is a mixture of sand, adhesive, and water [18]. The adhesive can be clay, lime, Portland cement, or water. If the soil is used as an adhesive, it is called mud mortar; if Portland cement is used, it is called cement mortar. The primary role of mortar is to increase adhesion and bonding strength with the construction components [19]. The strength of mortar depends on the cohesion of the cement paste to the fine aggregate particles; generally, people use lime powder for mortar mixtures because it is cheaper than cement; therefore, making mortar with HVFA is more efficient due to the utilization of coal waste. A typical mortar mix comprises approximately 68% aggregate, 11% cement, 17% water, and 4% air. The properties of mortar include compressive strength, water absorption, modulus of elasticity, high adhesion, and good workability. Mortar can be applied to a wide range of building materials and is designed to resist rain penetration, provide adequate water retention, and dry quickly. PT Semen Andalas Indonesia produced the OPC type I used in this study. The physical properties and chemical composition of cement have satisfied the requirements of ASTM standards C150. Fine aggregates were obtained from local quarry mines. It has specific gravity and water absorption of 2.64 and 0.5 %. The fineness modulus of fine sand is 3.82.

2.2. Carbon Nanotubes

CNTs is a carbon structure with a cylindrical shape and a nano-sized structure and consist of carbon atoms. It is a derivative of the carbon structure that can be described as a 1-atom thick graphite sheet that is rolled like a cylinder and has a diameter of the order of nanometers. This sheet has a honeycomb structure consisting of carbon atom bonds. The use of CNTs in cement composites has been carried out in the past because of the desired mechanical properties of CNTs [20], [21].

2.3. Graphene Oxide

Carbon has allotropic forms from 0-D to 3-D; Graphene is classified as a 2-D carbon nanostructure based on its structure. Graphene is a material composed of a hexagonal lattice with a thickness of one atom. Graphene consists of a single layer of carbon atoms arranged in a hexagonal lattice, with a thickness of just one atom. When multiple graphene layers are stacked on top of each other, the layers are held together by Van der Waals forces, forming materials such as graphite, carbon nanotubes, and fullerenes. These structures exhibit unique properties, making them highly valuable for various advanced technological applications [22], [23].

2.4. Fly Ash

Fly ash used in this study was obtained from PLTU Nagan Raya, located in Aceh, Indonesia. The fly ash is classified as Class F, as defined in standard classification systems [24]. The detailed chemical composition of the fly ash is provided in Table 1, which highlights the key components contributing to its properties and suitability for various applications.

Table 1. Chemical composition of fly ash

Oxide Compounds	Percentage (wt. %)
CaO	6.67
SiO_2	46.4
Al_2O_3	28.0
Fe_2O_3	7.17
MgO	3.12

Na_2O	1.93
K_2O	1.85
TiO_2	0.743
P_2O_5	0.455
SO_3	2.90
Cl	0.415

2.5. Compressive Strength

Compressive strength is one of the most important characteristics determining cement-based composites' strength. It is used to assess the ability of the material to withstand axial loads without fail. The compressive strength test is performed by subjecting the specimens to a compressive force applied uniformly per unit area using a Universal Testing Machine (UTM). The force gradually increases until the specimen fails and the maximum sustained load is recorded. This value is then used to calculate the compressive strength, a crucial indicator of the material's performance and suitability for construction applications.

2.6. Cement Hydration Characteristics

Mortar is a mixture of cement, sand, and water, where the combination of these components triggers a chemical reaction known as hydration. This exothermic reaction releases heat as essential compounds like Tricalcium Aluminate (C_3A) and Tricalcium Silicate react with water. The heat generated during this process leads to an increase in the temperature of the mortar or concrete. Due to the low thermal conductivity of concrete, the heat becomes trapped within the material, while the outer surface loses heat through convection with the surrounding environment. This results in a temperature gradient between the concrete's core and surface. The trapped heat causes the concrete to expand while the surface contracts, creating internal stress. If the tensile stress on the surface exceeds the material's strength, cracking can occur. Understanding cement hydration is crucial in constructing large structures such as dams and buildings, where temperature control is essential. Further investigation into the chemical reactions occurring in these mixtures—mainly when additives like fly ash, CNT, and GO are included—requires techniques such as X-ray diffraction (XRD) to analyze the specific phases and products formed during hydration.

3. Research Method

3.1. Mix Design

The mix proportion of materials used in this study was one part cement to two parts graded sand by volume. Fly ash was incorporated as a 60% replacement by weight of cement. Given the significant role of GO in enhancing the synergistic effects of hybrid nanomaterials in cementitious materials, GO was selected as a variable for this experiment. Mortar specimens were prepared with a constant water-to-cement ratio of 0.46, incorporating CNTs at a dosage of 0.01% by weight of cement. In comparison, GO was added in varying amounts (ranging from 0.01% to 0.05% by weight of cement). A polycarboxylate superplasticizer (SP) was added at 1.5% by weight of the mix to optimize workability.

Cement and fine sand were first mixed to obtain a homogenous mix. Then, the dispersed nano solutions of CNTs and GO were added to the mixer and stirred for 1 minute at 140 rpm to ensure proper dispersion. The mixture was then blended for an additional minute at a higher speed of 285 rpm to achieve a consistent and smooth mortar mix. After mixing, all specimens were left in the laboratory at room temperature for one day to set. The specimens were carefully demolded and subsequently cured in water for proper hydration and strength development. The mixed proportion of materials can be seen in Table 2.

Table 2. Proportion of materials for specimen preparation

Variation sample	Cement (g)	Sand (g)	*Fly Ash* (g)	Nanomaterials (g)		Water (g)	Superplasticizers (g)
				CNTs	GO		
MF	86,3	532,16	129,44	-	-	99,24	3,88
MFCG-1	86,3	532,16	129,44	0,022	0,022	99,24	3,88
MFCG-2	86,3	532,16	129,44	0,022	0,043	99,24	3,88
MFCG-3	86,3	532,16	129,44	0,022	0,065	99,24	3,88
MFCG-4	86,3	532,16	129,44	0,022	0,086	99,24	3,88
MFCG-5	86,3	532,16	129,44	0,022	0,108	99,24	3,88

Where:

MF = Control mortar (cement + sand + FA + water + SP)
MFCG = HVFA Mortar with CNTs and GO (cement+sand+ FA + water + SP + CNTs + GO)

3.2. Slump Flow Test

Based on SNI 03-6825-2002, the slump test is carried out in several stages. First, the ring is placed on the slump table. Then, the mortar is filled to half the height of the ring and compacted with 20 taps using a tamper. Next, the mortar is added to fill the ring and compacted with another 20 taps. The upper surface of the ring is leveled, and any mortar that spills over the top is cleaned off. The ring is then carefully lifted, leaving the conical mound of mortar on the slump table. The table is activated to perform 25 taps over 15 seconds. Finally, the diameter of the mortar spread is measured at least at four different points on the slump table to calculate the average diameter.

3.3. Cement Hydration Characteristics Test

The cement hydration test is designed to analyze the mixture's mineral composition and crystal structure. By using X-rays to identify the diffraction patterns of the crystals present in the mortar samples, this testing provides valuable information about the types and quantities of minerals involved in the mortar's formation. XRD testing of mortars is a crucial analytical method in the construction and materials industry, as it helps ensure the quality and consistency of the final product. It also allows for a deeper understanding of the compounds in mortar mixtures that incorporate nanomaterials.

3.4. Compressive Strength Test

A compressive strength test was conducted following SNI 03-6825-2002. The test involves preparing mortar cubes with dimensions of 5 cm x 5 cm x 5 cm, with a minimum of three test specimens for each sample. In total, 54 samples were used for compressive strength testing, including control samples (MF) and samples with GO variations (MFCG), with testing performed at 3, 7, and 28 days. The molds used for the mortar must be made of metal to prevent water absorption during the curing process. The compressive strength is determined by dividing the maximum load applied by the cross-sectional area of the specimen. The compressive strength of the mortar can be calculated using the following equation,

$$f'_c = \frac{P}{A} \tag{1}$$

Where:
f'$_c$ = Compressive strength obtained from the test specimen (MPa)
P = Maximum compressive load (N)
A = Cross-sectional area (mm^2)

4. Result and Discussion

4.1. Slump Flow Test

The mortar slump test is carried out using a mini-slump cone to determine the workability of the mortar. When CNT and GO amounts vary, a decrease in the slump value is observed, as both CNT and GO tend to absorb water in the mortar. The results of this test are presented in Table 3.

Table 3. Slump flow test results

Mortar sample	Slump flow (cm)
MF	16,5
MFCG-1	15,2
MFCG-2	14,7
MFCG-3	14,0
MFCG-4	13,6
MFCG-5	13,0

Based on the slump flow test, the slump flow value in the control mortar is higher than that of mortar with additional CNT and GO variations. In this test, if MWCT and GO are added, there will be a decrease in the slump value because MWCT absorbs more water in the mortar. This follows research conducted by [3], which showed that the increasing percentage of MWCT and use decreases, and the workability of mortar decreases. This decrease in workability is caused by the properties of MWCT, which has a high water absorption capacity and reduces water availability in the mortar mixture. As a result, the fluidity of the mortar decreases, causing the slump flow value to decrease significantly. Thus, adjusting the proportion of materials and adding additives accordingly is necessary to offset the effect of water absorption by MWCT and GO and maintain good workability. The results of the slump flow test are presented in the form of a graph, as shown in Figure 1.

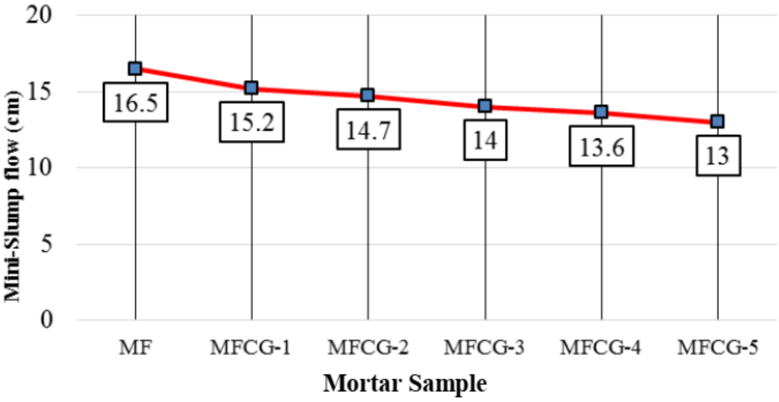

Fig 1. Slump flow of mortar samples

The incorporation of 0.01% CNT with GO variation of 0.01% - 0.05% exhibits a significant decrease in slump flow due to the addition of CNT-GO absorbing more water in the mortar mixture. As the GO content increases, the slump flow decreases [2].

4.2. Cement Hydration Characteristics

The cement hydration test is used to analyze the mixture's mineral composition and crystal structure. Using XRD to identify the diffraction patterns of the crystals in the mortar samples provides valuable information about the types and quantities of minerals involved in mortar formation. XRD testing is a crucial analytical technique for ensuring the quality and consistency of mortar products and identifying the compounds present in mortar mixtures containing nanomaterials. The XRD results from this analysis are presented in Figure 2.

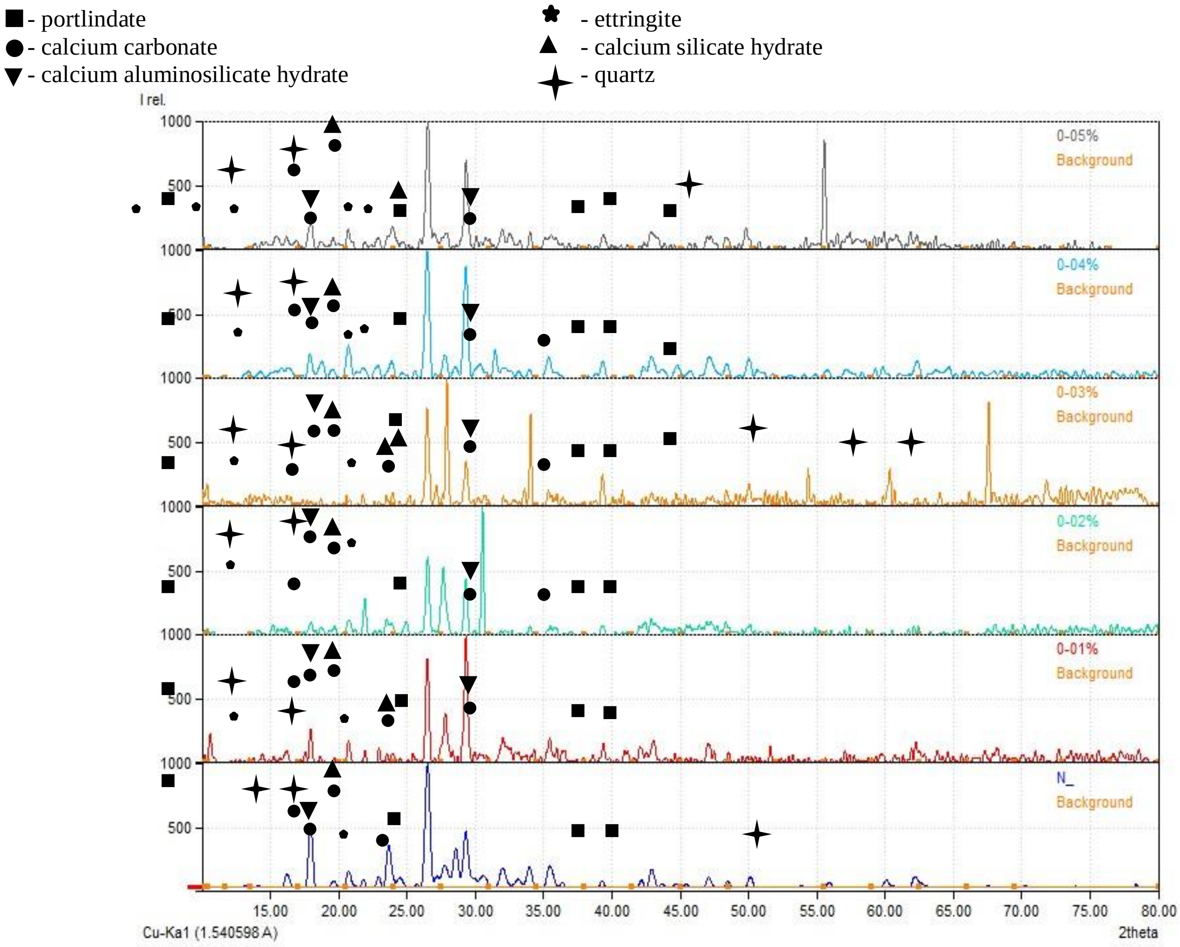

Fig 2. XRD results of mortar samples

Each mixture variation contains several key ingredients, including ettringite, portlandite, calcium carbonate, calcium aluminosilicate hydrate, calcium silicate hydrate, and quartz. Ettringite is a compound with the chemical formula $Ca_6Al_2(SO_4)_3(OH)_{12}\cdot 26H_2O$, formed during the early stages of hydration of Portland cement. It forms when tricalcium aluminate (C_3A) reacts with gypsum ($CaSO_4\cdot 2H_2O$), a reaction that occurs rapidly in the initial hydration phase. Portlandite ($Ca(OH)_2$) is another essential compound formed during the hydration of tricalcium aluminate (C_3A) and dicalcium silicate (C_2S) in Portland cement. This reaction produces calcium hydroxide and calcium silicate hydrate (C-S-H), which contributes to the strength of the mortar.

Calcium carbonate ($CaCO_3$) typically reacts with portlandite to enhance cement hydration, improving the overall quality of the cement paste. Calcium aluminosilicate hydrate (C-A-S-H) is a hydration product formed from the reaction between calcium, alumina, and silica in the cement mortar system. C-A-S-H is similar to calcium silicate hydrate (C-S-H), the main compound responsible for concrete strength, but contains alumina. C-A-S-H is particularly prevalent in concrete containing pozzolanic materials such as fly ash, where it forms from reactions between portlandite and aluminosilicates. Calcium silicate hydrate (C-S-H) is the primary product of the hydration of Portland cement, mainly from the responses of tricalcium silicate (C_3S) and dicalcium silicate (C_2S). This reaction produces C-S-H and portlandite ($Ca(OH)_2$), contributing to the strength of the cement. Quartz (SiO_2) is a widely distributed mineral commonly found in fly ash and can influence the properties of the mortar. The XRD results show the formation of complex crystallization phases at a GO variation of 0.03%. The peaks corresponding to calcium silicate hydrate (C-S-H) are observed at 2θ values of 29.5º, 32.0º, and 35.0º (corresponding to 505, 210, and 770 intensities, respectively). For portlandite ($Ca(OH)_2$), peaks are observed at 18.1º, 34.1º, 47.1º, 50.8º, and 54.35º (intensities of 90, 770, 85, 70, and 335, respectively). Ettringite ($Ca_6Al_2(SO_4)_3(OH)_{12}\cdot 26H_2O$) shows peaks at 22.92º, 24.24º, and 31.58º (intensities of 118, 95, and 67, respectively). Calcium carbonate ($CaCO_3$) peaks appear at 26.5º, 28.1º, 29.4º, 39.34º, and 43.9º (intensities of 744, 1000, 505, 309, and 105, respectively). Calcium aluminosilicate hydrate (C-A-S-H) exhibits peaks at 27.9º and 39.4º (intensities of 1000 and 309, respectively). Finally, quartz (SiO_2) peaks at 26.5º, 60.0º, 67.6º, and 71.8º (intensities of 745, 325, 749, and 234, respectively). These results suggest that a GO variation of 0.03% promotes the formation of desirable C-S-H compounds, supporting adequate cement hydration.

4.3. Compressive Strength

The compressive strength test determines the mortar's compressive strength until failure occurs. The tests are performed at 3, 7, and 28 days of curing. The average compressive strength results of cement mortars are presented in Table 4. The results showed the following percentage increases in compressive strength compared to the control mortar: at 3 days, the increases were 5.92%, 10.53%, 15.79%, 13.82%, and 10.53%, respectively. At 7 days, the compressive strength increases were 5.82%, 10.58%, 15.87%, 13.76%, and 10.58%. At 28 days, the increases were 5.29%, 10.13%, 14.54%, 13.22%, and 10.13%. The compressive strength results are illustrated in Figure 3.

Figure 3 shows the maximum mortar compressive strength at GO variation of 0.03% compared to the control sample. Similar results were reported by Gong et al. (2020), who found that mortars with CNT and GO contents exhibited an increase in compressive strength across all test samples. However, they also noted that excessive amounts of CNT and GO could decrease the mortar's compressive strength.

Table 4. Compressive strength test results of cement mortars

Sample Type	Compressive Strength (MPa)		
	3 days	7 days	28 days
MF	15,2	18,9	22,7
MFCG 1	16,1	20,0	23,9
MFCG 2	16,8	20,9	25,0
MFCG 3	17,6	21,9	26,0
MFCG 4	17,3	21,5	25,7
MFCG 5	16,8	20,9	25,0

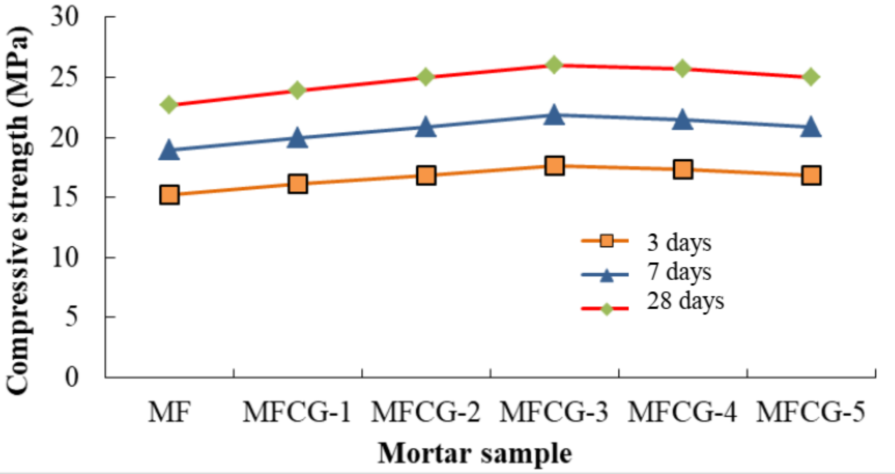

Fig 3. Compressive strength test results of mortars

5. Conclusion

Based on the results and discussions, it can be concluded that adding CNT and GO enhances the compressive strength of HVFA mortar in all variations. The optimal GO content of 0.03% resulted in a significant increase of 15.4% in compressive strength compared to the control mortar. Although HVFA generally reduces initial compressive strength, incorporating nanomaterials mitigates this effect, with a 15.7% increase in compressive strength observed at 3 days for the HVFA mortar with 0.03% GO compared to the HVFA control. The XRD analysis showed the formation of various crystallization phases, including C-S-H, C-A-S-H, quartz, portlandite, calcium carbonate, and ettringite, in each variation. The optimum combination of 0.01% CNT and 0.03% GO resulted in the most prominent crystallization phase, with the highest peak intensities observed compared to other variations.

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