

Compressive strength of cement mortar incorporating graphene-based nanosheets in powder and liquid forms

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KEYWORDS

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ABSTRACT

This study investigates the influence of graphene-based nanosheets (GNS) in both liquid (G-L) and powder (G-P) forms on the compressive strength development of cementitious composites. Mortar specimens were prepared with GNS concentrations ranging from 0% to 1% by weight of cement and tested at 1, 3, 7, and 28 days. The results indicate that G-P consistently enhanced compressive strength, with the optimal dosage (0.3-0.5%) improving 1-day strength by up to 24%, and 28-day strength by 17%. Conversely, G-L exhibited inconsistent early-age performance, with some dosages causing strength reductions (e.g., 0.3% G-L decreased 3-day strength by 7%), although higher concentrations (0.8-1.0%) improved 28-day strength by up to 14%. Excessive graphene content ($\geq 0.8\%$) led to agglomeration, negatively impacting early-age strength despite long-term improvements. These findings provide valuable insights into the optimization of GNS dosage to enhance the performance of cement-based materials, or for the reduction of cement without compromising the mechanical strength of cementitious materials.

1. Introduction

Cement-based materials are widely used in modern construction due to their strength, durability and cost-effectiveness. However, their brittle nature, susceptibility to cracking, and limited tensile strength present challenges that can compromise long-term structural performance. In response, nanomaterials have gained significant attention for their ability to enhance cementitious materials, with graphene-based additives emerging as promising candidates due to their exceptional mechanical properties, high surface area, and potential to improve hydration kinetics and microstructural refinement [1]-[3].

Graphene, a two-dimensional carbon nanomaterial, has been explored in various forms for cementitious applications, including graphene oxide (GO), reduced graphene oxide (rGO), graphene nanoplatelets (GNPs), and pristine graphene. These additives contribute to enhanced mechanical properties by promoting better particle packing, increasing nucleation sites for hydration products, and hindering crack propagation [4][5]. However, a key challenge remains in achieving uniform graphene dispersion, as improper distribution can lead to agglomeration, reducing the expected benefits in mechanical properties.

Among these nanomaterials, GO has been extensively studied for its ability to enhance the mechanical properties of cement-based materials even at low dosages. Concrete, a widely used cement-based material, suffers from inherent limitations such as low tensile strength and poor crack resistance, necessitating continuous efforts to improve

its performance. The incorporation of GO into cement paste and mortar has shown significant potential in addressing these issues. Research has demonstrated that GO can significantly improve the morphological and mechanical properties of cement-based composites by accelerating hydration, refining pore structure, and bridging microcracks [6].

To overcome these challenges, researchers have explored different graphene-based nanosheets (GNS), including graphene (Gr) and GO, as additives to cementitious materials. Studies have shown that incorporating GNS (ranging from 0.01 % to 2 % by mass) can significantly enhance mechanical performance. GO, in particular, exhibits higher efficiency than Gr but comes at higher cost. To optimize performance, researchers have developed a Gr-GO solution by dispersing both in water. Advanced characterization techniques such as nano-tomography, scanning electron microscopy (SEM), and confocal Raman imaging have revealed that GNS influences porosity, micropore distribution, and hydration acceleration in cement-based materials. Notably, the addition of 0.03 % Gr-GO resulted in a 28 % increase in compressive strength, indicating its potential for enhancing mechanical performance [7][8].

Beyond traditional graphene additives, researchers have also explored ultra-early strength cementitious materials for modern infrastructure applications. A notable study investigated calcium sulfoaluminate cement (CSA) derived from solid waste, incorporating 3 % GO (G3) and 3 % triethanolamine (TEOA)-modified GO (TG3). The findings showed that TG3 exhibited superior ultra-early strength due to

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improved dispersion, attributed to fewer –COOH groups and more –OH branched chains, enhancing steric hindrance. Compared to the control, the initial setting times of G3 and TG3 decreased by 11.90 % and 26.19 %, respectively, while their 3-hour compressive strengths increased by 253.4 % and 294.9 %. TG3 also improved hydration kinetics, reduced activation energy, and refined pores and microcracks, further enhancing mechanical properties [9].

In recent years, the integration of nanomaterials and polymers has been proposed as a novel approach to improving cementitious materials. However, challenges such as high graphene costs, low production yield, and poor dispersion in the cement matrix have limited its practical applications. To address these issues, a one-step modification method using graphene and polyvinyl alcohol (PVA) has been developed. This method utilizes shear exfoliation to create a stable graphene/PVA aqueous dispersion, which can directly replace water in cement casting. Molecular dynamics simulations have revealed the dispersion mechanisms, highlighting the synergistic effects of graphene and PVA. Compared to plain cement, this modified material demonstrates substantial improvements, with a 71.1 % increase in compressive strength, 106.5 % in flexural strength, and 75.8 % in thermal conductivity, significantly outperforming PVA-only modification. This innovative technique offers a cost-effective and scalable solution for improving cement-based composites [10].

Furthermore, ongoing research continues to investigate the effects of different graphene forms on cementitious materials. Graphene powder (G-P) and graphene liquid (G-L) are two commonly used forms, each with distinct dispersion characteristics [11][12]. G-P provides a dry, stable form of graphene that can be mixed into cementitious materials, but its dispersion in aqueous systems remains a challenge. In contrast, G-L offers better initial dispersion but may introduce variability in hydration kinetics and strength development. The comparative effectiveness of these two forms, particularly regarding their optimal dosages and long-term performance in cementitious composites, remains an area of active research.

This study aims to evaluate the impact of G-P and G-L on the compressive strength development of cementitious materials at dosages ranging from 0.1 % to 1.0 % by weight of cement. Compressive strength was assessed at 1, 3, 7, and 28 days to evaluate both early-age and long-term performance. The findings provide critical insights into the potential of graphene as a high-performance additive in concrete applications, emphasizing the importance of proper dispersion techniques to fully harness its benefits. Furthermore, the graphene used in this study is bio-derived and developed through a sustainable production process. Compared to conventional graphene oxide, this material offers significantly lower production cost and reduced environmental impact while maintaining effective performance in cementitious systems. The material is also associated with an industrial production route with validated technical specifications, supporting its potential for practical applications.

2. Experimental program

2.1. Materials and specimen preparation

The cement used in this study was a PCB 40 (Portland cement blended), a commonly used type of cement known for its balanced strength and durability. To enhance the properties of the cement mortar, GNS was introduced in two different forms: powder (G-P) and liquid (G-L), at concentrations of 0 %, 0.1 %, 0.5 %, 0.8 %, and 1 % by weight of cement.



Figure 1. Graphene-based nanosheets under powder (a) and liquid (b) forms.

Maintaining consistency across all samples, the reference cement mortar was prepared following the TCVN 6016:2011 standard [13][13], with a mix ratio of cement : sand : water = 1 : 3 : 0.5 (equivalent to 450 g : 1350 g : 225 g). For the sample having GNS added, these ratios were kept constant to ensure uniformity and reliable comparative analysis across all samples.

To investigate the effects of GNS on cementitious materials, GNS-enhanced specimens were labeled as G-X-Y, where G represents graphene-enhanced samples, X denotes the form of GNS: P (powder) or L (liquid), and Y indicates the percentage by weight of cement. For example, G-P-0.1 refers to a specimen with 0.1 % graphene powder in mass (0.45 g) by cement weight, while G-L-0.5 refers to a sample containing 0.5 % GNS liquid in mass. The control specimen G-0 contained no GNS. The selected dosage range (0.1–1.0 % by weight of cement) was chosen based on previous studies, which indicate that graphene contents within this range are sufficient to enhance hydration and mechanical properties while minimizing agglomeration effects.

In addition, the GNS used in this study are based on a bio-derived production process and were supplied in both powder and liquid forms. According to the technical datasheet and independent characterization reports, the graphene exhibits a platelet morphology with lateral particle sizes ranging from approximately 5 to 50 μm in powder form, while the dispersed nanosheets in liquid form present a characteristic size in the nanometer range (approximately 16 nm). The material shows a specific surface area of approximately 5.37 m^2/g , as determined by BET analysis [14].

2.2. Experimental procedure

To ensure uniform graphene dispersion in the cement matrix, different dispersion techniques were applied. For GNS powder (G-P), the required amount of GNS was pre-mixed with cement using a mechanical mixer at 200 rpm for 5 minutes to improve distribution. For graphene liquid (G-L), graphene was first sonicated for 30 minutes in deionized water before being added to the mix, following recommendations from previous studies [3]. Cement, sand and water were then mixed in a mixer for 3 minutes to achieve homogeneity before being poured into molds. The compositions of GNS-enhanced cementitious specimens are presented in Table 1.

Table 1. Mix compositions of mortar specimens investigated.

Specimens	Cement (g)	GNS (%)	Sand (g)	Water (g)
G-0	450	0.0	1350	225
G-P-0.1	450	0.1	1350	225
G-P-0.3	450	0.3	1350	225
G-P-0.5	450	0.5	1350	225
G-P-0.8	450	0.8	1350	225
G-P-1.0	450	1.0	1350	225
G-L-0.1	450	0.1	1350	225
G-L-0.3	450	0.3	1350	225
G-L-0.5	450	0.5	1350	225
G-L-0.8	450	0.8	1350	225
G-L-1.0	450	1.0	1350	225

Mortar specimens were cast in 50 mm cubic molds and subjected to vibration for 30 seconds to remove air voids. After casting, specimens were covered with plastic sheets to prevent moisture loss and were demolded after 24 hours. The curing process was carried out in water bath at an ambient controlled temperature until testing. The specimens were tested at 1, 3, 7, and 28 days to evaluate compressive strength development.

Compressive strength tests were conducted according to ASTM C109/C109M standards [15] using a universal testing machine (UTM) with a loading rate of 1.0 MPa/s. Each result represents the average of three specimens per condition.

3. Experimental results and discussion

3.1. Compressive strength development

The compressive strength development of cement-based materials incorporating GNS powder (G-P) and GNS liquid (G-L) over 1, 3, 7 and 28 days is summarized in Figure 2. The results illustrate the influence of varying graphene concentrations on early-age (1-7 days) and long-term (28 days) strength trends.

3.1.1. Early-age strength enhancement

GNS powder (G-P) significantly influenced early-age strength, particularly at 0.3 % concentration (G-P-0.3). At 1 day, G-P-0.3 achieved the highest early strength (12.6 ± 2.5 MPa), representing a 24 % increase over the control (10.2 ± 1.8 MPa). This enhancement suggests that a 0.3 % GNS powder addition accelerates early hydration, improves particle packing, and enhances mechanical interlocking. However, at higher concentrations (≥ 0.8 %), graphene agglomeration negatively impacted strength, as seen in G-P-0.8 (6.9 ± 1.9 MPa) and G-P-1.0 (4.7 ± 1.6 MPa).

At 3 days, G-P-0.3 (30.7 ± 2.3 MPa), and G-P-0.5 (28.4 ± 0.6 MPa) significantly outperformed the control (24.4 ± 2.9) by 26 % and 16 %, respectively, confirming the accelerating effect of graphene powder on hydration. By 7 days, graphene-enhanced specimens continued to exhibit superior strength. G-P-1.0 (42.1 ± 3.0 MPa) and G-P-0.8 (41.7 ± 1.5 MPa), and G-P-0.3 (41.3 ± 2.6 MPa) exceeded the control G-0 (34.5 ± 2.5 MPa) by over 20 %, confirming the long-term hydration benefits of graphene powder at optimal dosages.

GNS liquid (G-L), in contrast, showed less consistent early-age performance. At 1 day, G-L-1.0 (11.2 ± 1.8 MPa) and G-L-0.8 (10.8 ± 1.7 MPa) demonstrated moderate improvement over the control. However, at 3 days, graphene liquid exhibited variable performance, with G-L-0.5 (19.5 ± 2.2 MPa) being 20 % lower than the control, indicating potential dispersion challenges. By 7 days, G-L-1.0 (37.2 ± 2.6 MPa) marginally outperformed the control, while lower concentrations showed minimal improvement. This enhancement is attributed to the nucleation effect of graphene nanosheets, which accelerates the formation of C-S-H gel and refines the pore structure.

3.1.2. Compressive strength at 28 days

At 28 days, higher graphene powder concentrations contributed strength enhancement. G-P-1.0 achieved the highest compressive strength (52.8 ± 2.0 MPa), surpassing the control (44.2 ± 2.4 MPa) by 19 %, followed by G-P-0.5 (51.5 ± 1.1 MPa) and G-P-0.3 (50.1 ± 2.5 MPa). These improvements suggest that GNS powder supports continued hydration and improves load transfer efficiency without reducing mechanical strength.

However, G-P-0.8 (44.2 ± 1.4 MPa) matched the control, indicating that excess GNS powder (> 0.5 %) does not always provide benefits. The diminishing strength gain at higher concentrations is likely due to agglomeration, which disrupts matrix continuity.

Among GNS liquid added specimens, G-L-1.0 (50.4 ± 2.5 MPa) and G-L-0.1 (50.1 ± 2.5 MPa) showed moderate improvements, while G-L-0.5 (40.9 ± 2.4 MPa) remained lower than the control, reinforcing the importance of dispersion efficiency in graphene-modified cementitious materials.

3.2. Comparative analysis of GNS powder (G-P) and GNS liquid (G-L)

A comparative analysis of GNS powder (G-P) and GNS liquid (G-L) is summarized in Figure 2, which presents the percentage increase in compressive strength relative to the control specimen (G-P-0). The percentage increase is computed as the ratio of the strength difference between each graphene-enhanced specimen and the control, normalized by the control specimen's strength. This analysis highlights the influence of different graphene concentrations on strength development at various curing ages.

As evident from Figure 2, GNS powder (G-P) consistently enhanced compressive strength, with optimal dosages between 0.3-0.5 % offering the best balance of early-age and long-term improvements. These concentrations may lead to a densified microstructure, improved C-S-H gel formation, and reduced microcrack formation, all of which contributed to high mechanical performance. However, excessive GNS content (≥ 0.8 %) resulted in strength reductions at early ages, likely due to particle agglomeration limiting its dispersion and reinforcement efficiency.

Conversely, GNS liquid (G-L) exhibited more variable early-age performance, likely due to dispersion challenges. While lower concentrations (≤ 0.5 %) had minimal effect on strength, higher dosages of 0.8-1.0 % led to notable strength gains at 28 days, suggesting a delayed reinforcement mechanism. Despite these late-stage gains, the overall effectiveness of graphene liquid was less consistent than that of graphene powder, indicating potential limitations in its interaction with the cementitious matrix. Although G-P-1.0 exhibited the highest 28-day strength, lower dosages (0.3–0.5 %) are considered optimal due to their more stable performance and reduced risk of agglomeration.

From a practical perspective, the results suggest that GNS powder (0.3-0.5 %) is the most effective additive, offering superior dispersion, enhanced hydration, and improved interfacial bonding within the cement matrix. In contrast, graphene liquid (0.8-1.0 %) may still be beneficial in some applications but requires optimized dispersion techniques to maximize its reinforcing potential.

These findings highlight the potential of GNS for high-performance concrete applications, particularly in scenarios demanding superior mechanical properties and durability; or for the reduction of cement without reducing the mechanical strengths. Future research may focus on advanced dispersion techniques, hybrid graphene-cement systems, and long-term durability assessments to further enhance graphene's effectiveness as a reinforcing agent in cement-based composites.

Improving graphene dispersion techniques is crucial to ensuring its effectiveness in concrete. Studies have shown that using polyvinyl pyrrolidone (PVP) as a dispersant, in combination with ultrasonication and high-shear mixing, can significantly enhance the dispersion of graphene in cement mortar, leading to improved compressive and flexural strength of the material [16]. This behavior can also be attributed to the high surface area and nanoscale dispersion capability of the graphene nanosheets, which promote nucleation of hydration

products and improve the compactness of the microstructure.

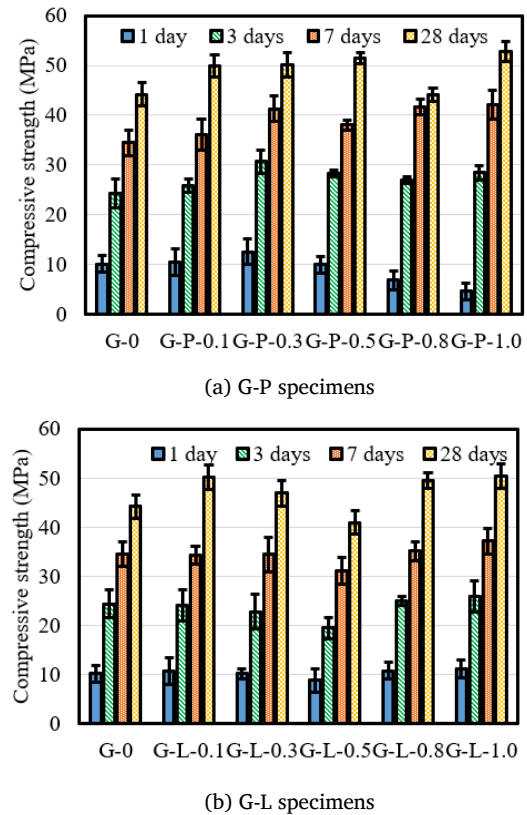


Figure 2. Compressive strength of mortar specimens without and with GNS: (a) powder; (b) liquid.

4. Conclusions

This study investigated the effects of GNS in the forms of powder (G-P) and liquid (G-L) on the compressive strength development of cementitious materials. The experimental results demonstrated that G-P consistently enhanced both early-age and long-term strength, whereas G-L exhibited variable, particularly at early ages. The conclusions can be drawn:

(1) GNS powder (G-P) at optimal dosages (0.3-0.5 %) significantly enhanced hydration, particle packing, and microstructural refinement. At 0.3 % G-P, compressive strength increased by 24 % at 1 day, and 13 % at 28 days compared to the control specimen. At 0.5 % G-P, the highest 28-days strength gain (17 %) was observed. However, excessive graphene dosages (≥ 0.8 %) led to agglomeration, reducing early-age strength but still yielding long-term improvements.

(2) GNS liquid (G-L) exhibited inconsistent early-age performance, with some reductions in strength (e.g., a 7 % decrease in 3-days strength at 0.3 % G-L). While higher dosages (0.8 - 1.0 % G-L) contribute to moderate 28-days strength improvements (12-14 %), its overall effectiveness was less consistent than G-P, likely due to dispersion challenges.

Comparative analysis indicates that GNS powder (G-P) is the superior additive, providing better dispersion, interfacial bonding, and sustained strength gains. However, optimizing dispersion techniques remains critical to mitigating agglomeration effects at higher dosages and optimize the potential of GNS in cementitious applications. The use of bio-derived graphene nanosheets further highlights the potential for sustainable and scalable application of nanomaterials in cementitious composites.

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