



OPEN Performance level controlled efficiency of graphene nanoplatelets in cementitious composites

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Graphene nanoplatelets (nGP) have been widely reported as effective nano-reinforcements for cementitious composites; however, their optimal dosage remains inconsistent across existing studies. This variability is commonly attributed to nanomaterial-related parameters, whereas the influence of the cementitious matrix's inherent performance is rarely systematically examined. In this study, cementitious matrices with distinct performance levels were designed by varying water-to-binder ratios (0.55, 0.40, and 0.30) and incorporating silica fume. Their interaction with nGP was experimentally investigated, with contents up to 2.4% being used. A total of 25 mix designs were evaluated for fresh-state behavior, mechanical performance, transport properties, and microstructural characteristics. The results indicate that the efficiency and optimal utilization of nGP are strongly governed by the matrix performance level. High-performance matrices (water-to-binder (w/b) = 0.30, 11% silica fume) achieved peak performance at 1.2% nGP, yielding compressive strength of 118.2 MPa and up to 50% reduction in capillary water transport, whereas low-performance matrices (w/b = 0.55) exhibited limited sensitivity to nGP incorporation. Microstructural observations confirmed that matrix densification improved nGP integration and hierarchical pore refinement by enhancing interfacial bonding, thereby restricting capillary water transport. The findings demonstrate that optimal nGP dosage cannot be treated as an intrinsic material parameter and should instead be defined in relation to matrix performance level, enabling performance-based dosage guidelines for nano-engineered composites in high-durability applications.

Keywords Graphene nanoplatelets, Matrix densification, Pore structure refinement, Performance-based design

The construction industry continuously seeks innovative materials to enhance the performance of cementitious composites¹. In recent years, nanotechnology has emerged as a promising field for developing advanced construction materials with superior properties^{2,3}. Among various nanomaterials, graphene nanoplatelets (nGP) have garnered significant attention due to their exceptional mechanical, thermal, and electrical properties^{4,5}. Recent investigations on hybrid nano-reinforcements for strain hardening cementitious composites have confirmed the potential of carbon nanomaterials in tailoring fresh and hardened behaviors of cementitious matrices⁶. The incorporation of nGP into cement-based materials has shown potential to improve both strength and durability. This development is particularly significant sustainable construction, where the demand for high-performance materials that can reduce environmental impact while maintaining structural integrity is increasing^{4,7}. The unique two-dimensional structure of graphene, combined with its high thermal conductivity (3080–5150 W m⁻¹ K⁻¹) and exceptional mechanical properties including tensile strength of 130 GPa, makes it an ideal candidate for enhancing the performance of cementitious composites in modern construction applications^{8,9}.

The durability benefits of nGP incorporation in cementitious systems have emerged as a critical factor driving research in this field. Studies have demonstrated that nGP can significantly improve resistance to water

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penetration, chloride ingress, and carbonation processes through multiple mechanisms, including pore structure refinement, enhanced interfacial transition zones, and reduced permeability^{10–12}. The nanoscale reinforcement provided by graphene platelets creates physical barriers to the transport of deleterious agents while promoting the formation of denser microstructures through nucleation effects on cement hydration products^{13,14}. These durability enhancements are particularly significant for infrastructure applications where long-term performance under aggressive environmental conditions is paramount, making nGP-modified composites attractive for marine structures, chemical processing facilities, and other demanding service environments^{14,15}.

However, the literature reveals significant contradictions and variations regarding optimum nGP utilization rates, with reported optimal dosages ranging from 0.01% to 1.0% by weight of binder across different studies^{16–18}. This wide variation in optimal dosages has traditionally been attributed to differences in nGP characteristics, dispersion methods, and testing procedures, with most optimization approaches focusing primarily on total surface area considerations for dosage calculations¹⁹. The conventional approach assumes that nanomaterial effectiveness is primarily governed by the available surface area for interaction with cement hydration products, leading to dosage recommendations based on specific surface area measurements and theoretical surface coverage calculations^{20,21}.

The densification of the matrix microstructure is a fundamental factor that can significantly influence the compatibility and performance of nanoscale particles in cementitious systems. Dense matrices with refined pore structures and improved particle packing efficiency can provide enhanced anchorage points and interaction sites for nanomaterials, potentially increasing the effective surface area available for beneficial interactions^{22–24}. While increased matrix density through lower water-to-binder ratios and supplementary cementitious materials may theoretically provide more adsorption sites and interlocking for nGP, studies^{25–27} report that overly dense matrices can hinder nanomaterial dispersion and interfacial bonding due to restricted pore connectivity and nanoparticle mobility. This limitation may reduce the intended nano-scale reinforcement effects.

Current research on optimum nGP dosages remains limited by the focus on total surface area parameters alone, with insufficient investigation into how matrix density and mortar design characteristics influence the capacity for effective nanomaterial incorporation. Previous studies have not systematically examined how varying mortar performance levels, achieved through different water-to-binder ratios and supplementary cementitious materials, affect the optimum nanomaterial dosage and effectiveness. This knowledge gap represents a significant limitation in developing practical guidelines for nGP utilization across different performance requirements and mix designs. The present study addresses this research gap by conducting pioneering experimental research that systematically investigates the relationship between mortar performance characteristics and optimum nGP utilization, providing fundamental insights into how matrix properties influence nanomaterial integration efficiency and overall composite performance. This research represents the first comprehensive investigation to correlate mortar performance levels with optimal nanomaterial dosages, contributing essential knowledge for the development of performance-based design approaches for nano-modified cementitious composites.

Materials and methods

Materials

CEM I 42.5R type Ordinary Portland cement (PC) and Silica fume (SF) were used as binders in this research. The chemical and physical characteristics of these materials are detailed in Table 1. Potable water from the municipal water supply system was used for concrete mixing and high-temperature curing. Fine aggregate (standard RILEM sand) and quartz sand (QS) (0.5–1.0 mm) were utilized in various proportions to prepare the mortar mixtures. Nano graphene platelets (nGP) supplied by Nanografi Nanotechnology Inc. were incorporated into the mixtures. The technical specifications of the nano graphene material include 99.9% purity, 3 nm thickness, 1.5 μm diameter, 320 m^2/g specific surface area, and 1400–1900 S/m conductivity. Results of X-ray diffraction analysis for nGP and silica fume are presented in Fig. 1. The XRD peak of nGP shows the presence of a (002) crystalline plane at 26.4° for the 2θ angle. This is followed by (100), (101), (004), and (110) planes at 42.2 , 44.3 , 54.3 , and 77.4° , respectively.

Oxide (%)	PC	SF	QS
SiO ₂	20.09	90.7	95.9
Al ₂ O ₃	4.20	0.5	0.2
Fe ₂ O ₃	3.1	0.9	0.3
CaO	60.2	0.6	1.7
MgO	3.1	1.50	0.2
SO ₃	2.65	-	-
Na ₂ O	0.3	0.6	0.1
TiO ₂	0.4	-	-
K ₂ O	0.8	1	0.1
Specific Gravity	3.16	2.69	2.85
Color	Gray	Gray	Light beige

Table 1. Chemical oxides of powder materials.

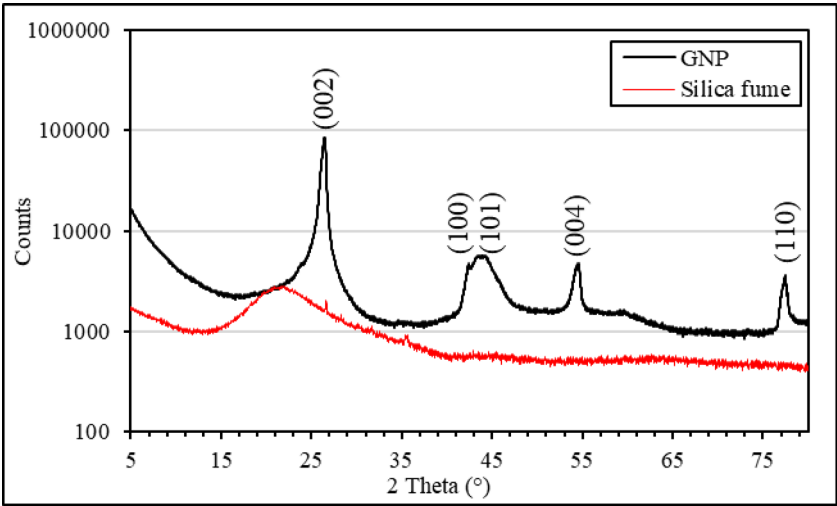


Fig. 1. XRD analysis of nGP and silica fume.

Mix ID	Cement	w/b*	SF	Sand	QS	SP** (%)	nGP** (%)
M_W0.55_N0	1	0.55	0	2.75	0	0	0
M_W0.55_N0.3						0	0.3
M_W0.55_N0.6						0	0.6
M_W0.55_N1.2						0	1.2
M_W0.55_N2.4						0	2.4
M_W0.40_N0	1	0.40	0	2.00	0	0	0
M_W0.40_N0.3						0.55	0.3
M_W0.40_N0.6						0.58	0.6
M_W0.40_N1.2						0.65	1.2
M_W0.40_N2.4						1.63	2.4
MS_W0.40_N0	0.89	0.40	0.11	2.00	0	0	0
MS_W0.40_N0.3						0.49	0.3
MS_W0.40_N0.6						0.61	0.6
MS_W0.40_N1.2						0.66	1.2
MS_W0.40_N2.4						1.37	2.4
M_W0.30_N0	1	0.30	0	1.12	0.13	0.33	0
M_W0.30_N0.3						0.55	0.3
M_W0.30_N0.6						0.62	0.6
M_W0.30_N1.2						0.69	1.2
M_W0.30_N2.4						1.21	2.4
MS_W0.30_N0	0.89	0.30	0.11	1.12	0.13	0.49	0
MS_W0.30_N0.3						0.64	0.3
MS_W0.30_N0.6						0.70	0.6
MS_W0.30_N1.2						0.76	1.2
MS_W0.30_N2.4						1.47	2.4

Table 2. Mixing properties. *: water/binder, binder=(PC + SF), ** by weight of binder.

The superplasticizer used in the study is a polycarboxylate-based chemical produced by Polisan Chemical Company. This concrete admixture provides high workability without causing segregation in fresh concrete and enables the design of concrete mixtures with low water/cement ratios.

Methods

The experimental matrix was designed to investigate the influence of water-to-binder (w/b) ratio, SF content, and nGP dosage on the composite performance. As presented in Table 2, a systematic mix design methodology was employed with three distinct w/b ratios (0.55, 0.40, and 0.30) and five nGP dosages (0, 0.3, 0.6, 1.2, and 2.4% by total binder weight) for each w/b ratio.

Two primary mix series were developed: conventional mortar mixes (M) containing only Portland cement as the binder, and quaternary mixes (MS) where 11% of the cement was replaced with silica fume. The cement content was normalized to 1 for the M series, while the MQ series utilized 0.89 parts cement with 0.11 parts silica fume. Sand content was proportionally adjusted according to the w/b ratio, with values of 2.75, 2.00, and 1.12 for w/b ratios of 0.55, 0.40, and 0.30, respectively. For the mixes with w/b ratio of 0.30, quartz sand (QS) was incorporated at 0.13 parts to maintain adequate workability and particle packing density. Each mix was designated with a unique identifier indicating the mix type (M or MS), w/b ratio (W0.55, W0.40, or W0.30), and nGP dosage (N0 to N2.4). A superplasticizer (SP) additive was added to the mixture to achieve ideal flowability.

For the purposes of this study, the term “matrix performance level” is used in a quantitative sense, on the basis of three observable identifiers: the water-to-binder ratio, the silica fume replacement level, and the 90-day compressive strength of the reference (graphene-nanoplatelet-free) mix. Three classes are defined accordingly and are summarized in Table 3: low-performance matrices (water-to-binder ratio of 0.55, no silica fume, reference compressive strength ≤ 65 MPa), intermediate performance matrices (water-to-binder ratio of 0.40 with or without 11% silica fume replacement, $65 \text{ MPa} < \text{reference compressive strength} \leq 95 \text{ MPa}$), and high-performance matrices (water-to-binder ratio of 0.30 with or without 11% silica fume replacement, reference compressive strength $> 95 \text{ MPa}$).

Mixture proportioning

A comprehensive experimental program comprising twenty-five distinct cementitious composites was systematically designed and fabricated. The primary variable parameters included w/b ratio, silica fume incorporation, and nGP dosage. For the conventional mortar formulations, a planetary mixer operating at precisely controlled rotational velocities was employed for homogenization. The protocol involved initial blending of cementitious materials with water at 140 rpm for 120 s, followed by an intensification phase at 280 rpm for 60 s to ensure complete particle dispersion. Subsequently, fine aggregates were introduced incrementally while maintaining continuous agitation for an additional 120 s. For the nano-modified cementitious composites, a specialized two-step dispersion methodology was implemented to facilitate optimal nGP distribution. Initially, the predetermined nGP concentration (ranging from 0.30% to 2.4% by weight of binder) was incorporated into 50% of the designated mixing water, and the mixture was stirred magnetically under controlled conditions to achieve uniform suspension. This nGP-water dispersion was subsequently integrated into the primary cementitious matrix and subjected to sequential mechanical homogenization at 140 rpm (120 s) and 280 rpm (120 s). A polycarboxylate-based high-range water-reducing admixture was judiciously incorporated when necessary to maintain rheological consistency across varying w/b ratios. The prepared mixtures were placed in $50 \times 50 \times 50 \text{ mm}$ cube molds and cured in lime-saturated water for 90 days. All tests were carried out at 90 days. This age was selected because low water-to-binder silica fume matrices develop their pore structure slowly. The pozzolanic reaction of silica fume at low water-to-binder ratios continues past 28 days and refines the pore network at later ages^{28,29}. The 90 days of age, therefore, provides a mature and stable reference state for comparing matrices of different performance levels.

Rheological characterization

Flow behavior assessment The rheological properties of the fresh mixtures were quantitatively evaluated using the standardized flow table methodology in accordance with ASTM C1437³⁰. The assessment involved placing the fresh composite in a truncated conical mold using a two-layer placement technique with appropriate consolidation. Following mold removal, the specimen was subjected to 25 drops at a rate of 1 drop every 15 s. The resultant flow diameter was determined by averaging two perpendicular measurements across the spread material, providing a quantitative indication of workability.

Evaluation of mechanical and physical properties

Density and water absorption characteristics Prismatic specimens ($50 \times 50 \times 50 \text{ mm}$) were subjected to hydrostatic weighing following their prescribed curing regime. Saturated surface-dry mass was determined after careful removal of surface moisture, while submerged mass was recorded using a high-precision analytical balance ($\pm 0.05 \text{ g}$). The specimens were subsequently subjected to thermal treatment at $105 \pm 5 \text{ }^\circ\text{C}$ in a forced-convection oven until a constant mass was achieved to determine the oven-dry condition. Apparent density, absolute density, and effective porosity were calculated in accordance with ASTM C642³¹ standard.

Compressive strength determination Uniaxial compressive strength evaluations were conducted on prismatic specimens in full compliance with ASTM C109³² standard. After 90 days of controlled water curing, specimens were desiccated at $105 \pm 5 \text{ }^\circ\text{C}$ for 24 h, then equilibrated to ambient temperature. The specimens were then positioned in a calibrated compression testing apparatus, ensuring precise axial alignment, and subjected to

Performance class	w/b ratio	Silica fume (%)	Reference compressive strength f_c^* (MPa)	Mix IDs
Low	0.55	0	≤ 65	M_W0.55 series
Intermediate	0.40	0 or 11	$65 < f_c(\text{ref}) \leq 95$	M_W0.40, MS_W0.40 series
High	0.30	0 or 11	> 95	M_W0.30, MS_W0.30 series

Table 3. Quantitative classification of matrix performance level adopted in study. f_c^* : 90-day compressive strength results for each series.

monotonic loading until failure. The ultimate load was recorded and utilized to calculate the characteristic compressive strength. Compressive strength values were calculated by averaging the results of three samples for each mixture. Standard deviation values are shown with error bars in the relevant results.

Capillary water absorption kinetics Unidirectional water absorption characteristics were investigated following the methodology prescribed by ASTM C1585³³. Specimens were conditioned to constant mass in a ventilated oven maintained at 55 ± 2 °C. To ensure unidirectional moisture migration, lateral surfaces were meticulously sealed with hydrophobic silicone sealant, leaving only one face exposed as the absorption surface. The specimens were positioned on non-absorbent supports in a controlled water bath with the test surface immersed to a depth of 2–3 mm. Mass evolution was monitored at precisely defined intervals (0.1, 5, 10, 20, and 30 min; 1, 2, 3, 4, 5, and 6 h; and 1, 2, 3, 5, 6, 7, and 8 days). Prior to each gravimetric measurement, excess surface moisture was removed using a standardized blotting procedure to ensure measurement accuracy.

Microstructural analysis

Scanning electron microscopy (SEM) Morphological characterization of the composite microstructure was performed utilizing scanning electron microscopy (SEM). Specimens were analyzed after 90 days of hydration using a ZEISS EVO LS 10 instrument at the Central Research Laboratory of Karadeniz Technical University. Freshly fractured surfaces were examined under high vacuum conditions to elucidate the micro-architectural features, with particular emphasis on the interfacial transition zone and the dispersion characteristics of nGP within the cementitious matrix.

Mercury intrusion porosimetry (MIP) Pore structure characteristics of the hardened specimens were comprehensively analyzed using mercury intrusion porosimetry (MIP). Representative fragments measuring approximately 1–2 cm³ were carefully extracted from the core regions of the specimens and thoroughly dried to eliminate residual moisture. The analyses were conducted using an AutoPore V series instrument capable of generating pressures up to 33,000 or 60,000 psi, depending on the targeted pore diameter range. The MIP technique enabled quantitative assessment of pore volume, size distribution, and interconnectivity within the cementitious matrix. During the analytical procedure, mercury was incrementally forced into the specimen under progressively increasing pressure, beginning with larger pores and systematically penetrating smaller capillaries as the applied pressure intensified. This methodological approach facilitated the differentiation between macropores and micropores, providing detailed information regarding the hierarchical pore structure. The high-resolution pore analysis afforded by this technique served as a fundamental data source for evaluating the influence of nGP incorporation on the microstructural development of the cementitious composites, particularly with respect to permeability characteristics and potential refinement of the pore network. The derived parameters were critical in establishing correlations between microstructural modifications induced by nanomaterial addition and the corresponding macroscopic performance attributes, notably durability and mechanical properties.

Results and discussion

Flow behavior assessment

The flow table test results, as visualized in Fig. 2, provide critical insights into the rheological behavior of cementitious composites modified with varying dosages of graphene nanoplatelets (nGPs) and different binder compositions. A consistent trend is observed across all series: incorporating nGPs reduces flowability, with the effect of becoming more pronounced at higher nGP dosages and lower w/b ratios. This phenomenon can be attributed to the exceptionally high specific surface area and strong van der Waals interactions of nGPs, which

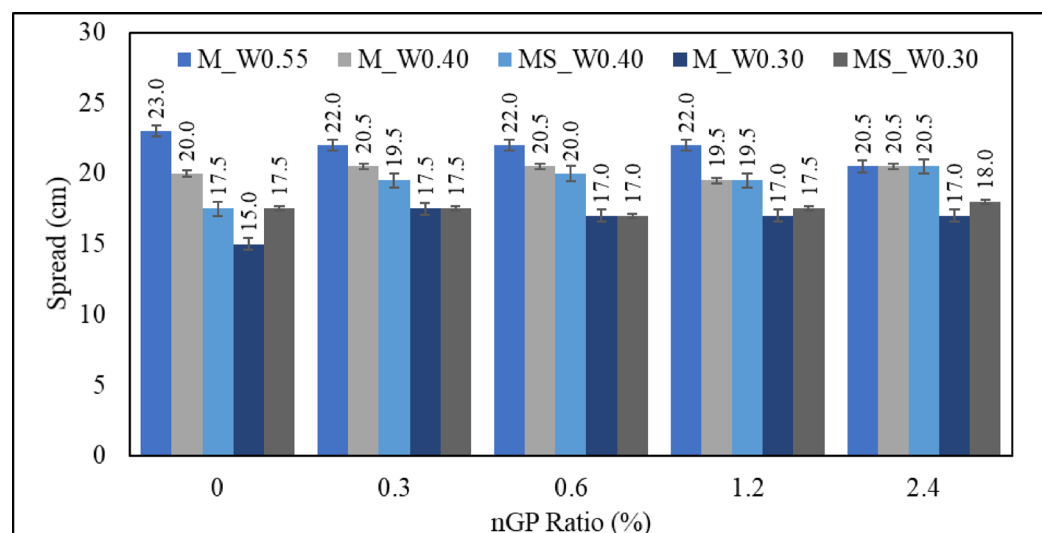


Fig. 2. Flow table test results of mixes.

increase the system's water demand and promote agglomeration, thereby impeding the mobility of the fresh mix. These findings are in strong agreement with previous studies^{34,35}, which have reported similar reductions in workability upon the addition of carbon-based nanomaterials. The superplasticizer dosage in Table 2 was not held constant. It was tuned for each mix to reach a workable flow. For this reason, flow and dosage cannot be compared directly across the mixes. M_W0.40_N2.4 needed 1.63% superplasticizer, but it flowed less than MS_W0.40_N2.4 at 1.37%. The silica fume mix flowed more for two reasons. Its spherical particles reduce inter-particle friction through a ball-bearing action^{36,37}. The polycarboxylate also adsorbs preferentially on silica fume, and the finer packing improves dispersion at a lower dosage^{38,39}. Silica fume adds further surface area and water demand, so it competes with nGP for free water. This competition is strongest at low water-to-binder ratios^{36,39}. The plain mix needed a higher dosage because dispersing 2.4% nGP without silica fume is difficult⁴⁰.

The reduction in flowability is particularly significant in mixtures with lower w/b ratios and in those containing silica fume (MS series). The synergistic effect of silica fume and nGPs, both characterized by fine particle sizes and high surface energies, exacerbates the demand for mixing water and further restricts the flow of the fresh mortar. This observation is consistent with the literature, where the combined use of supplementary cementitious materials and nanomaterials has been shown to intensify the competition for available water, leading to a denser particle packing but reduced workability³⁴.

Despite the general trend of reduced flowability, it is noteworthy that moderate nGP dosages (up to 0.6% by total binder weight) do not drastically impair the workability of the mixtures. This suggests the existence of a practical threshold for nGP incorporation, beyond which the negative impact on rheology outweighs the potential benefits in terms of mechanical and durability performance. Such a threshold is particularly important for the design of advanced cementitious composites, as it enables optimization of both fresh and hardened properties. Furthermore, the variability in flow table spread is greater in mixes with lower w/b ratios and in the MS series, indicating a heightened sensitivity of these systems to changes in mix composition and nanomaterial dosage. This underscores the necessity for precise control over mix design parameters when working with nano-modified cementitious materials. In summary, the present findings corroborate the consensus in the literature that incorporating nanomaterials such as nGPs requires careful optimization of mix proportions to balance workability and performance. The observed trends highlight the complex interplay between particle size distribution, surface chemistry, and water demand in determining the rheological behavior of advanced cementitious systems.

Density and water absorption

The scatter plot matrix in Fig. 3 provides a comprehensive visualization of the relationships among the key physical properties of the investigated cementitious composites: dry density (g/dm^3), water absorption (%), and porosity (%). The diagonal elements display kernel density estimates for each variable, offering insights into their distribution patterns, while the off-diagonal plots illustrate the pairwise correlations between variables. A pronounced negative correlation is observed between dry density and both water absorption and porosity. As the dry density increases, both water absorption and porosity tend to decrease, indicating that denser composites exhibit a more compact microstructure with reduced pore connectivity and lower water-uptake capacity. This trend is consistent with the fundamental understanding that higher density in cementitious materials is typically associated with improved packing of solid particles and reduced volume and connectivity of capillary pores. Conversely, a strong positive correlation between water absorption and porosity is evident. Samples with higher porosity also demonstrate increased water absorption, which can be attributed to the greater availability of interconnected voids that facilitate water ingress⁴¹. This relationship underscores the critical role of pore structure in governing the transport properties of cement-based materials. The investigation of porosity, water absorption, and density characteristics in cementitious composites modified with nGP and SF revealed significant insights into the microstructural evolution of these advanced materials. The experimental results demonstrated a complex interplay among the w/b ratio, nGP dosage, and SF incorporation on the physical properties of the composites^{35,42}, which aligns with previous studies in the field.

The conventional mortar series (M series) exhibited a clear correlation between the w/b ratio and the physical properties of the composites, consistent with the findings of Li et al.⁴³, who reported similar trends in graphene-modified cementitious systems. For the M_W0.55 series, the incorporation of nGP showed a nonlinear relationship with porosity and water absorption, with the initial addition of 0.3% nGP resulting in a slight increase in porosity from 18.08% to 18.19%. This initial increase in porosity at low nGP dosages has been previously observed by Tziviloglou et al.⁴⁴, who attributed it to temporary disruption of the cement matrix during the early stages of nGP incorporation. The M_W0.40 series demonstrated more pronounced improvements in physical properties compared to the M_W0.55 series, with the control mix (M_W0.40_N0) exhibiting a porosity of 15.14%, which was reduced to 14.02% with the addition of 1.2% nGP. This substantial reduction in porosity resulted in a decrease in water absorption from 7.09% to 6.46%. Optimal performance at a 1.2% nGP dosage suggests a threshold effect, beyond which the benefits of nGP addition diminish. The most remarkable improvements were observed in the M_W0.30 series, where the incorporation of 1.2% nGP resulted in an exceptional reduction in porosity from 12.26% to 5.92%. This dramatic improvement, accompanied by a reduction in water absorption from 5.55% to 2.64%, demonstrates the synergistic effect of low w/b ratio and optimal nGP dosage. These findings are consistent with the work of Arslan et al.⁴, who reported similar improvements in high-performance cementitious composites modified with graphene-based materials. The density values increased from $2.455 \text{ g}/\text{cm}^3$ to $2.516 \text{ g}/\text{cm}^3$, indicating enhanced particle packing and reduced void content, which aligns with the density-porosity relationships reported by Arslan et al.⁴.

The incorporation of silica fume (11% replacement) in the MS series resulted in superior physical properties compared to the conventional mortar series, with the MS_W0.30 series exhibiting exceptional performance, showing porosity values ranging from 2.96% to 3.42%. These results are in excellent agreement with the findings

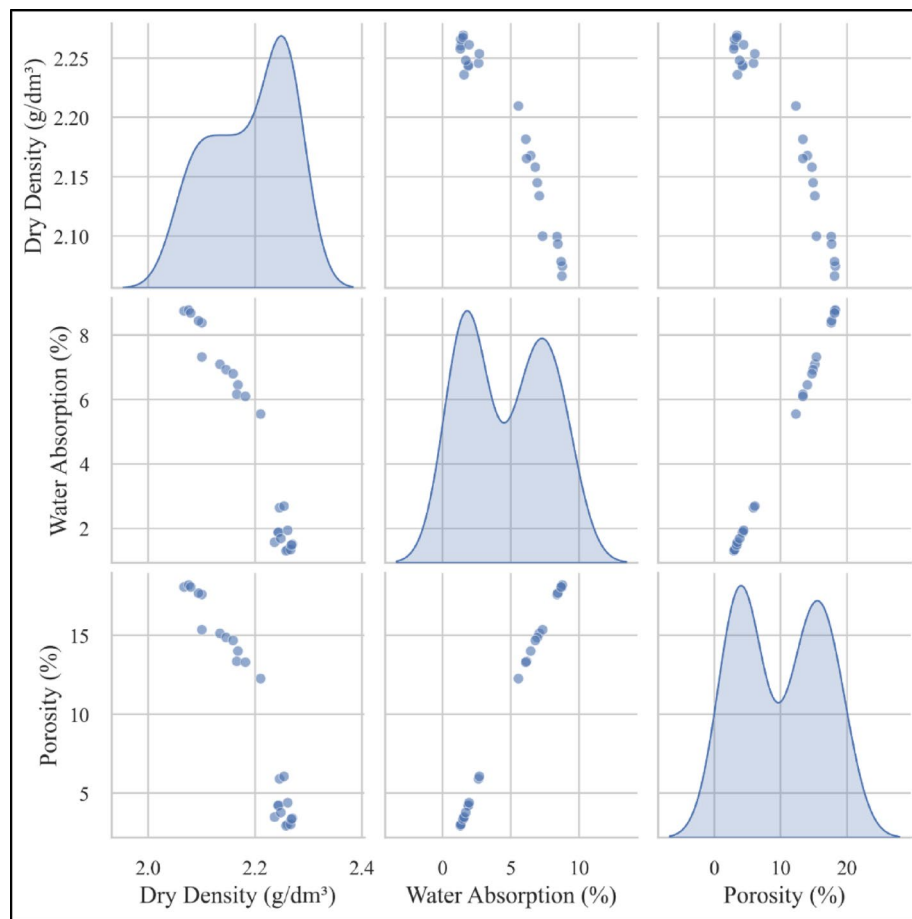


Fig. 3. Scatter plot of dry density, water absorption, and porosity results.

of Li et al.⁴⁵, who reported similar improvements in silica fume-graphene hybrid systems. The MS_W0.40 series demonstrated consistent improvements in physical properties, with porosity values ranging from 18.67% to 25.67%, supporting the conclusions of Bai et al.⁴⁶ regarding the effectiveness of combined SF and graphene modifications in cementitious systems. These findings demonstrate the potential of nGP and SF in enhancing the physical properties of cementitious composites, with particular emphasis on the importance of optimal dosage selection and the synergistic effects of multiple modifications. The results are supported by a substantial body of literature, indicating the reliability and significance of the observed improvements in physical properties. The combination of experimental data and literature review provides a comprehensive understanding of the mechanisms underlying the modification of cementitious materials with graphene nanoplatelets and silica fume, advancing knowledge in this field.

Mechanical performance analysis

The mechanical performance of cementitious composites incorporating nGP was systematically investigated through comprehensive compressive strength testing, and test results are given in Fig. 4. Compressive strength values are the mean of three specimens per mix, and error bars showing the standard deviation have been added to Fig. 4. The standard deviation ranged from 0.2 to 4.9 MPa, and the coefficient of variation remained below 6% (1 to 5%), indicating good repeatability. The experimental matrix encompassed a range of w/b ratios (0.55, 0.40, and 0.30), nGP dosages (0–2.4% by binder weight), and binder compositions. The results revealed significant variations in compressive strength, ranging from 61.8 MPa to 118.2 MPa, demonstrating the substantial influence of mix design parameters on mechanical performance.

The influence of w/b ratio on compressive strength exhibited a well-established inverse relationship, consistent with previous studies on high-performance cementitious composites^{47,48}. For conventional mortar mixes (M series), the compressive strength increased substantially as the w/b ratio decreased from 0.55 to 0.30, with average values of 63.3 MPa, 81.7 MPa, and 107.4 MPa, respectively. This enhancement can be attributed to the improved particle packing density and reduced porosity at lower w/b ratios, as evidenced by the porosity measurements ranging from 18.19% to 12.26% for the M series. The incorporation of nGP demonstrated a complex, dosage-dependent relationship with compressive strength, consistent with recent studies on nanomaterial-reinforced cementitious systems⁷. For the w/b ratio of 0.55, the addition of nGP did not significantly enhance compressive strength, with values remaining relatively constant around 63 MPa across different dosages. This behavior suggests that, at higher w/b ratios, the beneficial effects of nGP may be offset by the increased porosity and

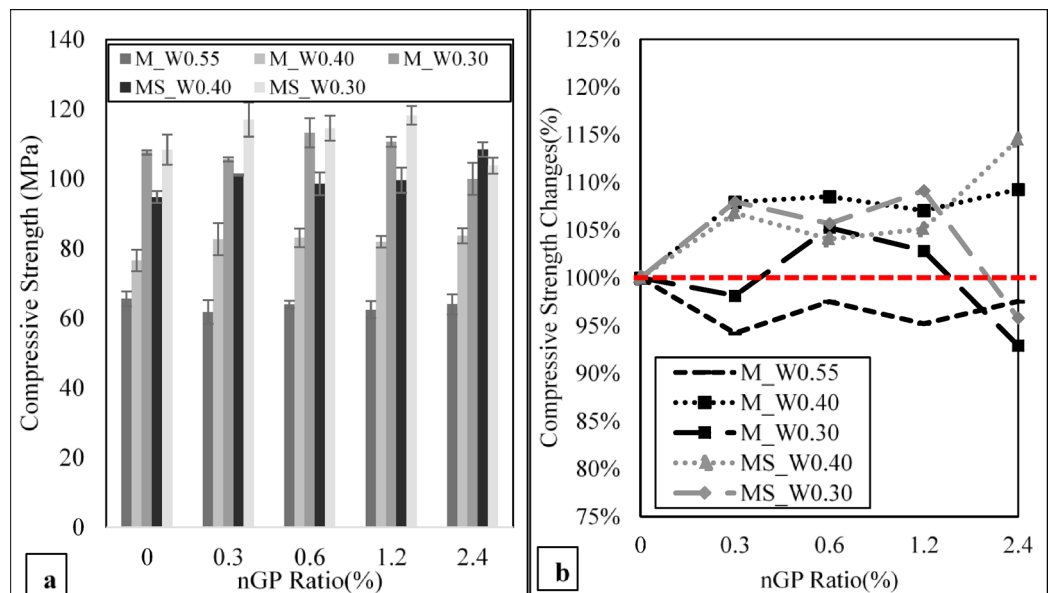


Fig. 4. Compressive strength test results of mixes: (a) test results, (b) relative changes according to reference mixes.

reduced particle packing efficiency. However, for w/b ratios of 0.40 and 0.30, optimal nGP dosages were observed at 0.6% and 1.2%, respectively, resulting in maximum compressive strengths of 83.1 MPa and 113.2 MPa.

The quaternary mixes (MS series) incorporating silica fume demonstrated superior mechanical performance compared to conventional mortars, particularly at lower w/b ratios. For the w/b ratio of 0.30, the MS series achieved compressive strengths ranging from 108.3 MPa to 118.2 MPa, representing an improvement of up to 10% over the corresponding M series mixes. This enhancement can be attributed to the synergistic effects of silica fume, where silica fume contributes to the formation of additional C-S-H gel through pozzolanic^{49,50}. The interfacial transition zone (ITZ) characteristics were significantly improved, as evidenced by reduced porosity (2.96–4.41%) and water absorption (1.31–1.95%) in the MS series compared to the M series (2.64–8.77% water absorption). The maximum compressive strength of 118.2 MPa was achieved in the MS_W0.30_N1.2 mix, representing a substantial improvement over conventional mortar mix. This enhancement can be attributed to several microstructural mechanisms: (1) the pozzolanic reaction of silica fume, which contributes to the formation of additional C-S-H gel and improved ITZ characteristics^{49,50}; (2) the nucleation effect of nGP, which promotes the formation of hydration products and enhances the microstructure^{51,52}; and (3) the improved particle packing density at lower w/b ratios, which reduces porosity and enhances the overall composite integrity³⁴.

The strong inverse correlation between porosity and compressive strength observed in this study (correlation coefficient of -0.89) is consistent with the findings of previous research on high-performance cementitious composites^{53,54}. The incorporation of nGP at optimal dosages contributed to reduced porosity through multiple mechanisms: improved particle packing density, enhanced hydration product formation, and reduced water absorption capacity. These mechanisms are particularly effective at lower w/b ratios, where the reduced water content allows for better particle packing and more efficient utilization of the pozzolanic and reinforcing effects. The experimental results demonstrate that the mechanical performance of cementitious composites can be significantly enhanced by carefully optimizing the w/b ratio, silica fume content, and nGP dosage. The synergistic effects of these parameters result in a more refined, denser microstructure, thereby improving mechanical properties. The performance-dependent optimum can be explained by particle-packing and agglomeration theories. In dense matrices, nGPs fill residual voids and improve packing up to a saturation dosage; beyond this dosage, they disrupt packing^{55,56}. Above a critical dosage, van der Waals attraction causes re-agglomeration. Thus, the effective surface area and nucleation sites plateau or decline^{19,57}. A denser matrix disperses and anchors more platelets before agglomeration dominates, which raises its effective threshold. The optimum dosage is therefore the point where the packing benefit is overtaken by agglomeration, and this point shifts with the matrix performance level. The findings of this study contribute to the growing body of knowledge on the development of high-performance cementitious composites and provide valuable insights for optimizing mix designs in practical applications.

Capillarity water absorption

Capillary water absorption constitutes a fundamental transport phenomenon that critically governs the durability characteristics of cementitious materials under service conditions. The initial and secondary absorption rates provide essential insights into microstructural features, particularly pore connectivity and capillarity, which ultimately determine the material's resistance to deleterious environmental agents. The experimental investigation revealed significant variations in capillary absorption behavior across different mix

formulations, with the water-to-binder ratio emerging as a primary controlling parameter. The comparison of capillarity test results is shown in Fig. 5. Conventional mortar specimens (M series) exhibited a systematic reduction in initial absorption rates from 14×10^{-3} mm/√s for M_W0.55_N0 to 6.5×10^{-3} mm/√s for M_W0.30_N0, corresponding to a 54% decrease. This trend aligns with established cement hydration principles, wherein reduced water availability promotes denser microstructural development and diminished capillary porosity. Secondary absorption rates followed a parallel pattern, declining from 5.6×10^{-3} mm/√s to 0.9×10^{-3} mm/√s across the same w/b range. The observed correlation between w/b ratio and capillary absorption was further substantiated by porosity measurements, with M_W0.55_N0 exhibiting 18.08% porosity, compared with 12.26% for M_W0.30_N0, confirming the inverse relationship between porosity and capillary transport characteristics.

Silica fume incorporation yielded remarkable enhancements in capillary resistance, with the MS series demonstrating superior performance compared to conventional formulations. At a w/b ratio of 0.40, initial absorption rates decreased from 11.2×10^{-3} mm/√s (M_W0.40_N0) to 5.6×10^{-3} mm/√s (MS_W0.40_N0), representing a 50% improvement. Similar enhancements were observed at a w/b ratio of 0.30, with absorption rates declining from 6.5×10^{-3} mm/√s to 3.7×10^{-3} mm/√s (43% reduction). These improvements can be attributed to the pozzolanic reactivity of silica fume, which consumes portlandite and generates additional calcium-silicate-hydrate phases with refined microstructural characteristics^{49,50,58,59}. The microstructural refinement is evidenced by the substantially lower porosity values in MS formulations, with MS_W0.30_N0 exhibiting only 3.03% porosity compared to 12.26% for the corresponding M series specimen.

Graphene nanoplatelet dosage effects manifested complex behavior patterns that varied considerably with both w/b ratio and silica fume presence. Conventional mortar formulations displayed variable optimal nGP dosages depending on the w/b ratio. M_W0.55 series achieved minimum initial absorption (11.5×10^{-3} mm/√s) at 1.2% nGP content, while M_W0.40 series demonstrated optimal performance at 2.4% dosage (7.5×10^{-3} mm/√s). However, the M_W0.30 series exhibited more complex behavior, with minimum absorption occurring at 1.2% nGP content (4.15×10^{-3} mm/√s) and maximum absorption at 2.4% dosage (6.85×10^{-3} mm/√s). Silica fume-modified formulations displayed more consistent nGP responses, with MS_W0.40 series maintaining stable absorption rates (3.9 – 5.6×10^{-3} mm/√s) across all dosages, while MS_W0.30 series exhibited rates ranging from 1.8×10^{-3} mm/√s to 3.7×10^{-3} mm/√s. This consistency suggests that silica fume-nGP interactions create more predictable capillary resistance characteristics. The underlying mechanisms involve multiple complementary effects: nucleation enhancement promoting cement hydration, physical barrier formation reducing capillary connectivity, and potential agglomeration phenomena at elevated dosages that may compromise beneficial effects.

The capillary absorption findings have profound implications for long-term durability assessment of the developed nanocomposites. The synergistic combination of a low w/b ratio, silica fume incorporation, and an optimized nGP dosage creates a multifaceted approach to capillary resistance enhancement, thereby improving resistance to chloride ingress, carbonation, and freeze-thaw cycling. MS_W0.30 series formulations, with initial absorption rates below 4×10^{-3} mm/√s, demonstrate exceptional durability potential for aggressive environmental applications. Secondary absorption rates, which reflect long-term capillary behavior, consistently remained below initial rates across all formulations, indicating the effectiveness of the developed systems in maintaining capillary resistance over extended service periods. This characteristic is particularly important for infrastructure applications, where long-term durability is a critical design consideration.

The magnitude of reduction observed in the present study (54% from w/b 0.55 to 0.30) exceeds the 30–40% reductions typically reported in conventional concrete formulations⁶⁰. The exceptional 43–50% reduction achieved through silica fume incorporation surpasses the 25–40% improvements documented in previous studies⁶¹. This superior performance can be explained by the optimized silica fume content (11%) and its interaction with the low w/b ratio, which creates exceptionally dense microstructures. The pozzolanic reaction

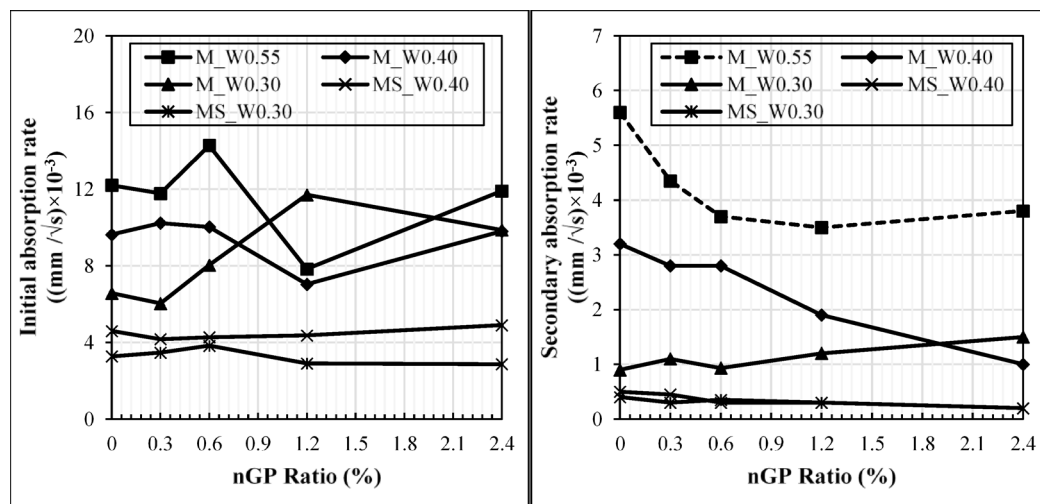


Fig. 5. Results of capillarity test on specimens: (a) Initial absorption ratio, (b) Secondary absorption rate.

generates additional C-S-H phases with reduced Ca/Si ratios, resulting in refined capillary pores and diminished connectivity. This mechanism is further enhanced by nGP's physical barrier effects, creating a multiscale pore-refinement system.

The complex nGP dosage behavior presents both similarities and differences compared to previous investigations. The optimal dosage of 1.2% for the M_W0.55 and M_W0.30 series aligns with Zhou¹⁸, who reported optimal barrier performance at 1% nGP content. However, the optimal dosage of 2.4% for the M_W0.40 series differs from other contents. This discrepancy may be due to several factors, such as silica fume-nGP interactions potentially altering the optimal dosage range, low w/b ratios promoting nGP agglomeration at high dosages, and variations in nGP surface characteristics and dispersion properties. The synergistic effect of silica fume-nano materials observed in this investigation aligns with Li et al.⁶², who documented enhanced performance when combining nano silica with supplementary cementitious materials. The mechanism involves complementary effects: silica fume refines capillary pore structure through pozzolanic reactions, while nGP provides nucleation sites and physical barriers to water transport. This combination creates hierarchical pore structures with reduced connectivity and enhanced tortuosity, significantly impeding capillary absorption. Despite the significant improvements in capillary resistance demonstrated, several factors may influence the practical implementation of these findings. nGP dispersion within cementitious matrices remains challenging, and the observed performance may prove sensitive to mixing procedures and environmental conditions. Additionally, the long-term stability of capillary resistance under various environmental exposures requires further investigation. Future research should focus on optimizing nGP dispersion techniques, investigating different nGP types and surface modifications, and evaluating performance under accelerated durability testing conditions. The exceptional capillary resistance achieved, particularly in MS_W0.30 series formulations, suggests potential applications in aggressive environments, including marine structures, wastewater treatment facilities, and infrastructure exposed to deicing salts. However, economic feasibility and scalability require careful evaluation, considering nGP costs and specialized mixing requirements. The development of cost-effective nGP production methods and optimized mixing protocols will be crucial for the widespread adoption of these nanocomposites in construction applications.

Microstructural analysis

SEM analysis

Scanning electron microscopy (SEM) examinations of the fractured surfaces provide comprehensive insights into the microstructural characteristics of cementitious composites modified with nGP and silica fume. The SEM photos reveal distinct microstructural characteristics that directly correlate with the observed macroscopic performance variations across different mix formulations, establishing the foundation for understanding the synergistic effects of nanomaterial incorporation and supplementary cementitious materials. Figure 6 presents the reference specimens: M_W0.55 (a) exhibits a relatively open microstructure with visible voids and a rough surface texture characteristic of higher porosity systems, reflecting the measured porosity of 18.08%. M_W0.40; (b) shows a more compact matrix with reduced surface roughness and fewer visible voids, corresponding to the reduced porosity of 15.14%. M_W0.30; (c) shows the densest microstructure among conventional mortars. A smoother surface texture and minimal visible porosity are consistent with a reduction in measured porosity (12.26%). The silica fume-modified specimens MS_W0.40 (d) and MS_W0.30 (e) demonstrate progressively refined surface characteristics. MS_W0.30 exhibited the most homogeneous and smooth surface morphology among all reference mixtures. Corresponding to the porosity values of 2.96–3.42%.

The addition of silica fume resulted in significant microstructural changes through pozzolanic reactivity. The MS specimens exhibit clear evidence of enhanced matrix refinement, where the pozzolanic reaction consumes portlandite. It generates additional calcium-silicate-hydrate phases with refined microstructural characteristics. This pozzolanic reaction creates a more tortuous pore network and enhanced matrix density, directly supporting the observed capillary resistance, with initial absorption rates decreasing from 11.2×10^{-3} mm/ $\sqrt{s}$ to 5.6×10^{-3} mm/ $\sqrt{s}$ (50% improvement) for MS_W0.40 compared to M_W0.40. The microstructural refinement is evidenced by the substantially smoother surface characteristics and enhanced homogeneity in silica fume-modified formulations.

Figure 7 illustrates the nGP-modified specimens. M_W0.55_N1.2 (Fig. 7a) shows microstructural changes compared to its reference. The limited effectiveness of nGP at higher w/b ratios is consistent with the minimal compressive strength improvements observed (63 MPa range). M_W0.40_N0.6 (Fig. 7b) shows improved matrix density. Suggesting beneficial nGP effects at this optimal dosage level, correlating with the achieved compressive strength of 83.1 MPa. M_W0.30_N1.2 (Fig. 7c) displays a dense and smooth surface morphology, representing the most compact microstructure among conventional nGP-modified mortars. This results correspond to a maximum compressive strength of 113.2 MPa and a porosity reduction from 12.26% to 5.92%. Corresponds to the maximum compressive strength of 113.2 MPa and porosity reduction from 12.26% to 5.92%.

MS_W0.30_N1.2 (Fig. 7d) exhibits surface characteristics with a smooth and homogeneous appearance, indicating the synergistic effects of silica fume and nGP in creating a highly refined matrix. This surface quality correlates with the measured compressive strength of 118.2 MPa and minimal water absorption characteristics (2.64%), representing the peak of microstructural development achieved in this study. The combination creates a hybrid matrix where pozzolanic reaction products interact favorably with nGP, establishing a multi-scale refinement system that optimizes both chemical and physical enhancement mechanisms. The agglomeration regions identified for M_W0.40_N2.4 in Fig. 7e appear as a matrix within the hydration product. This morphology is consistent with the agglomeration characteristics reported for nano-carbon-modified cement-based composites at optimal dosages in recent studies^{63,64}. The image in Fig. 7e is also confirmed by three independent performance tests for the same mixture (see Fig. 4, Sect. 3.2, Table 4). In contrast, M_W0.40_N2.4 (Fig. 7e) shows surface irregularities and a less uniform texture compared to optimally dosed mixtures,

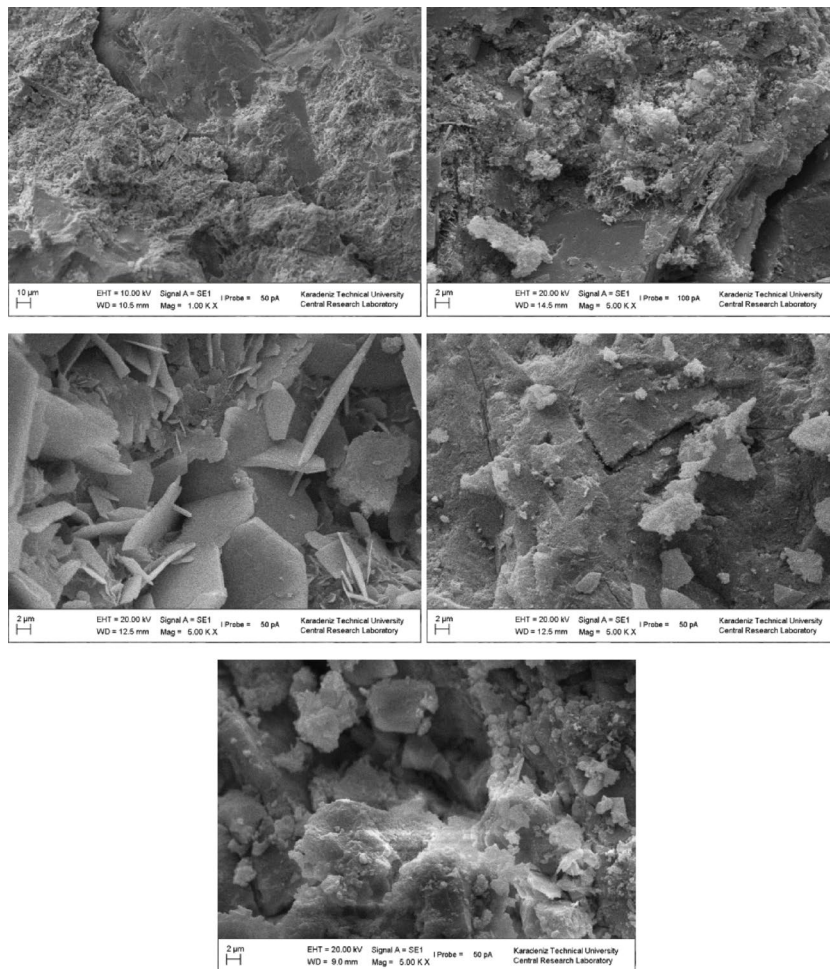


Fig. 6. SEM photographs of references (without nGP) (a) M_W0.55 (b) M_W0.40 (c) M_W0.30 (d) MS_W0.40 and (e) MS_W0.30.

suggesting potential issues related to excessive nGP content. It aligns with the observed performance loss and increased water absorption (8.77%) at this dosage level.

The comparative analysis reveals a consistent trend of improved surface smoothness and matrix density with decreasing w/b ratios and the incorporation of silica fume. The nGP effects are more effective in lower w/b mixes. The dense matrix enables more effective nanomaterial integration, supporting the observed optimal dosages of 1.2% for the M_W0.30 and MS_W0.30 systems. The microstructural refinement directly correlates with the measured physical properties. The specimens exhibiting smoother and denser surface characteristics consistently demonstrate reduced porosity (2.96–5.92% for optimal formulations), enhanced compressive strength (113.2–118.2 MPa), and superior capillary resistance ($1.8\text{--}4.15 \times 10^{-3}$ mm/ $\sqrt{s}$ initial absorption rates). These microstructural observations establish a foundation for understanding the performance enhancements achieved through the combined use of reduced w/b ratios, silica fume incorporation, and optimized nGP dosages in advanced cementitious composites.

Pore size distribution and porosity

Mercury intrusion porosimetry (MIP) analysis provides quantitative insights into the pore-structure characteristics of nGP-modified cementitious composites, revealing intricate relationships between mix design parameters and pore network refinement. Table 4 presents comprehensive pore-structure data, while Fig. 8 illustrates the pore-size distributions across different mix series, establishing correlations between microstructural modifications and macroscopic performance attributes. The conventional mortar series (M) shows a systematic refinement of the pore structure with decreasing water-to-binder ratios. The M_W0.55 series exhibits relatively high total intrusion volumes ranging from 0.0508 to 0.0614 mL/g, with corresponding porosity values of 11.22–13.09%. The median pore diameter varies considerably across this series (0.0887–0.1109 μm), indicating heterogeneous pore-size distributions that reflect the open microstructure characteristic of higher w/b ratios. Figure 8(a) reveals a broad pore size distribution with significant contributions from both capillary and gel pores, consistent with the observed high water absorption values (8.50–8.77%) and moderate compressive strengths (61.8–64.2 MPa) for this series.

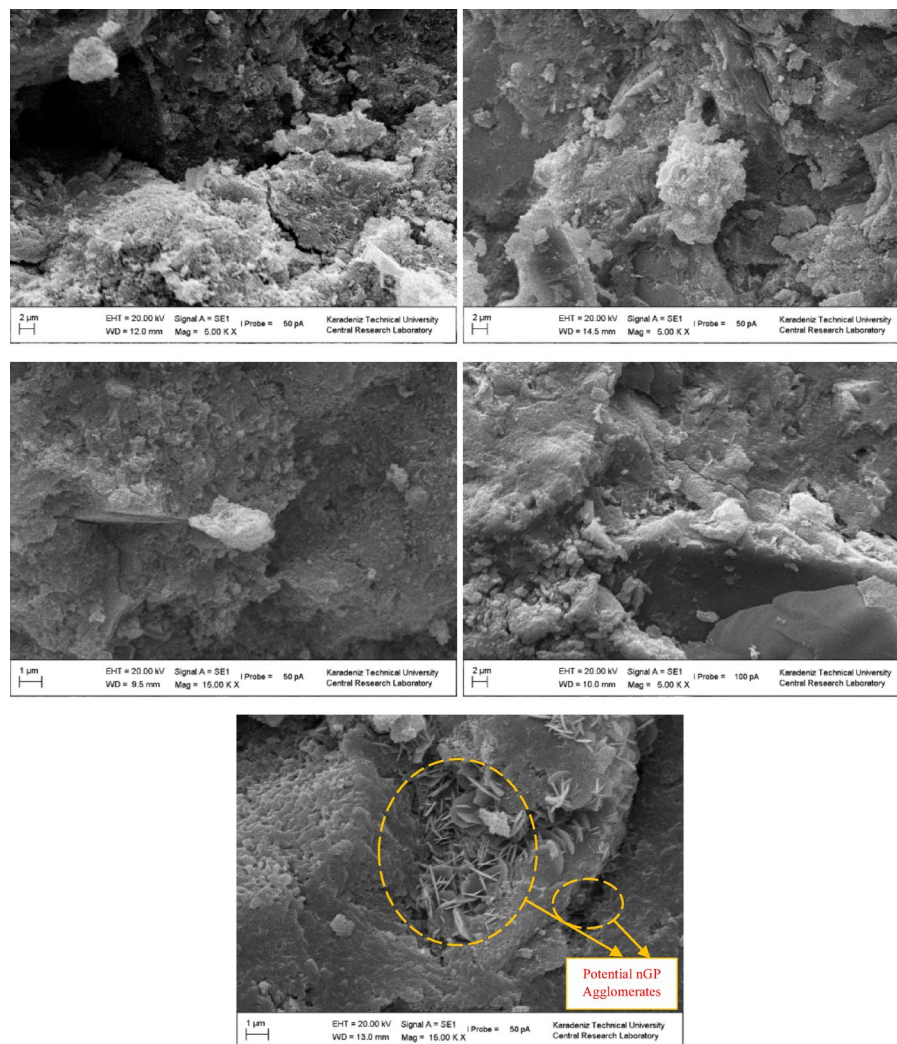


Fig. 7. SEM morphologies of (a) M_W0.55_N1.2, (b) M_W0.40_N0.6, (c) M_W0.30_N1.2 (d) MS_W0.30_N1.2, (e) M_W0.40_N2.4.

The M_W0.40 series demonstrates improved pore structure characteristics compared to M_W0.55, with total intrusion volumes decreasing to 0.0361–0.0460 mL/g and porosity values ranging from 8.07% to 10.16%. The incorporation of nGP produces variable effects depending on dosage, with optimal refinement at 1.2% nGP content, achieving the lowest total intrusion volume (0.0361 mL/g) and porosity (8.07%). Figure 8(b) illustrates a shift toward finer pore structures with nGP incorporation, particularly evident in the specimen M_W0.40_N1.2, which exhibits reduced macropore content and enhanced gel pore formation. This pore-structure refinement directly correlates with the observed compressive strength enhancement (83.1 MPa) and improved capillary resistance.

The M_W0.30 series represents the most refined pore structure among conventional mortars, with total intrusion volumes ranging from 0.0272 to 0.0425 mL/g and porosity values of 6.03–9.60%. The reference specimen (M_W0.30_N0) exhibits a porosity of 7.15%, which is further reduced to 6.03% with 0.3% nGP incorporation, demonstrating the effectiveness of nanomaterial addition in low w/b systems. However, higher nGP dosages (0.6–2.4%) result in higher porosity values (8.71–9.60%), suggesting potential agglomeration effects that compromise pore-structure refinement. Figure 8(c) reveals a predominantly fine-pore structure with median pore diameters ranging from 0.0558 to 0.0755 μm , supporting the exceptional mechanical performance (up to 113.2 MPa) and durability observed in this series. The silica fume-modified MS_W0.40 series demonstrates superior pore structure characteristics compared to corresponding conventional mortars, with total intrusion volumes consistently below 0.0397 mL/g and porosity values ranging from 7.22% to 8.76%. The pozzolanic reaction of silica fume generates additional C-S-H phases that contribute to pore refinement, as evidenced by the reduced median pore diameters (0.0220–0.0759 μm) and increased total pore areas (4.355–7.125 m^2/g). Figure 8(d) illustrates the effectiveness of silica fume in creating a more tortuous pore network with enhanced connectivity between smaller pores, directly supporting the observed 50% improvement in capillary resistance compared to conventional mortars. The MS_W0.40_N1.2 specimen achieves optimal pore structure refinement

Mix ID	Total Intrusion Volume (mL/g)	Total Pore Area (m ² /g)	Median pore volume diameter (μm)	Median pore area diameter (μm)	Average pore diameter (μm)	Porosity (%)
M_W0.55_N0	0.0573	4.174	0.1109	0.0216	0.0549	12.1924
M_W0.55_N0.3	0.0508	3.698	0.1007	0.0299	0.0549	11.2195
M_W0.55_N0.6	0.0545	4.786	0.0887	0.0243	0.0455	11.9344
M_W0.55_N1.2	0.0614	4.705	0.0970	0.0287	0.0522	13.0884
M_W0.55_N2.4	0.0572	4.233	0.0940	0.0350	0.0541	12.3617
M_W0.40_N0	0.0377	3.802	0.9300	0.1670	0.0396	8.4494
M_W0.40_N0.3	0.0450	4.408	0.7760	0.0189	0.0408	9.9286
M_W0.40_N0.6	0.0460	3.886	0.0762	0.0317	0.0474	10.1627
M_W0.40_N1.2	0.0361	3.450	0.0613	0.0299	0.0419	8.0688
M_W0.40_N2.4	0.0404	2.700	0.0721	0.0527	0.0598	8.8905
M_W0.30_N0	0.0314	1.985	0.0558	0.0447	0.0633	7.1499
M_W0.30_N0.3	0.0272	1.222	0.0755	0.0641	0.0891	6.0250
M_W0.30_N0.6	0.0425	2.780	0.0679	0.0527	0.0612	9.5978
M_W0.30_N1.2	0.0394	2.506	0.0732	0.0507	0.0629	9.1309
M_W0.30_N2.4	0.0383	2.500	0.0754	0.0515	0.0612	8.7090
MS_W0.40_N0	0.0397	4.355	0.0759	0.0127	0.0365	8.7639
MS_W0.40_N0.3	0.0326	5.083	0.0386	0.0130	0.0257	7.2868
MS_W0.40_N0.6	0.0340	5.153	0.0479	0.0110	0.0264	7.5067
MS_W0.40_N1.2	0.0328	7.125	0.0220	0.0109	0.0184	7.2215
MS_W0.40_N2.4	0.0329	5.073	0.0441	0.0111	0.0259	7.2791
MS_W0.30_N0	0.0281	5.044	0.0376	0.0088	0.0222	6.3815
MS_W0.30_N0.3	0.0297	5.276	0.0521	0.0085	0.0225	6.7102
MS_W0.30_N0.6	0.0717	5.430	0.0535	0.0092	0.0292	14.3300
MS_W0.30_N1.2	0.0266	7.288	0.0156	0.0078	0.0146	5.9610
MS_W0.30_N2.4	0.0238	5.850	0.0270	0.0069	0.0163	5.3489

Table 4. MIP analysis of mixes.

with the smallest median pore diameter (0.0220 μm) and highest total pore area (7.125 m²/g), indicating extensive pore surface development through secondary hydration reactions.

The MS_W0.30 series exhibits the most advanced pore structure characteristics, with porosity values ranging from 5.35% to 14.33%. The exceptional performance of MS_W0.30_N2.4 (5.35% porosity) and MS_W0.30_N1.2 (5.96% porosity) demonstrates the synergistic effects of low w/b ratio, silica fume incorporation, and optimal nGP dosage in achieving unprecedented pore structure refinement. However, the MS_W0.30_N0.6 specimen exhibits anomalous behavior, with significantly increased porosity (14.33%) and total intrusion volume (0.0717 mL/g), potentially indicating non-optimal dispersion characteristics at this parameter combination. The MS_W0.30_N0.6 specimen returned an anomalous mercury intrusion porosity of 14.33% with a high intrusion volume of 0.0717 mL/g, against 5.35% to 6.71% for the rest of the series. This value is considered an outlier. Mercury intrusion uses a single small fragment. It is sensitive to local micro-cracking and the ink-bottle effect, which raises the apparent intrusion volume^{65,66}. The effective porosity from water absorption for this mix is about 3%. Its strength and capillary results match the rest of the series. The bulk material, therefore, does not show a true porosity increase. Figure 8(e) reveals highly refined pore structures in optimal formulations, with median pore diameters as low as 0.0156 μm (MS_W0.30_N1.2), resulting in tortuosity levels that effectively impede capillary transport and enhance durability.

Pore-size distribution analysis reveals three distinct mechanisms governing pore-structure modification in nGP-silica fume hybrid systems. First, the pozzolanic reaction of silica fume consumes large portlandite crystals and generates fine C-S-H products that fill capillary spaces, reducing both pore volume and median pore size⁶⁷. Second, nGP incorporation at optimal dosages (0.3–1.2%) promotes nucleation-controlled hydration, resulting in more uniform pore-size distributions and enhanced gel pore formation¹⁹. Third, the combination of reduced w/b ratio and nanomaterial addition results in improved particle packing efficiency, thereby minimizing capillary pore formation during the early hydration stages⁶⁸. The correlation between pore structure parameters and macroscopic performance shows the critical role of pore network characteristics in governing composite behavior. Pores are classified here as gel pores (below about 10 nm), capillary pores (about 10 nm to 10 micrometers), and macropores (above 10 micrometers)^{69,70}. Gel pores influence shrinkage and creep, capillary pores control sorptivity and permeability, and macropores act as strength-limiting flaws. Reading the median pore diameter in Table 4 across the five series shows a progressive shift from the large-capillary range to the fine-capillary and gel ranges as the performance level increases. The low-performance M_W0.55 series remains in the large-capillary range (about 89 to 111 nm). The intermediate M_W0.40 series refines to about 61 to 76 nm at 1.2% nGP, while the silica-fume MS_W0.40 series reaches about 22 nm. The high-performance MS_W0.30 series reaches 15.6 nm at 1.2% nGP, near the gel and fine-capillary boundary, which is the finest network in

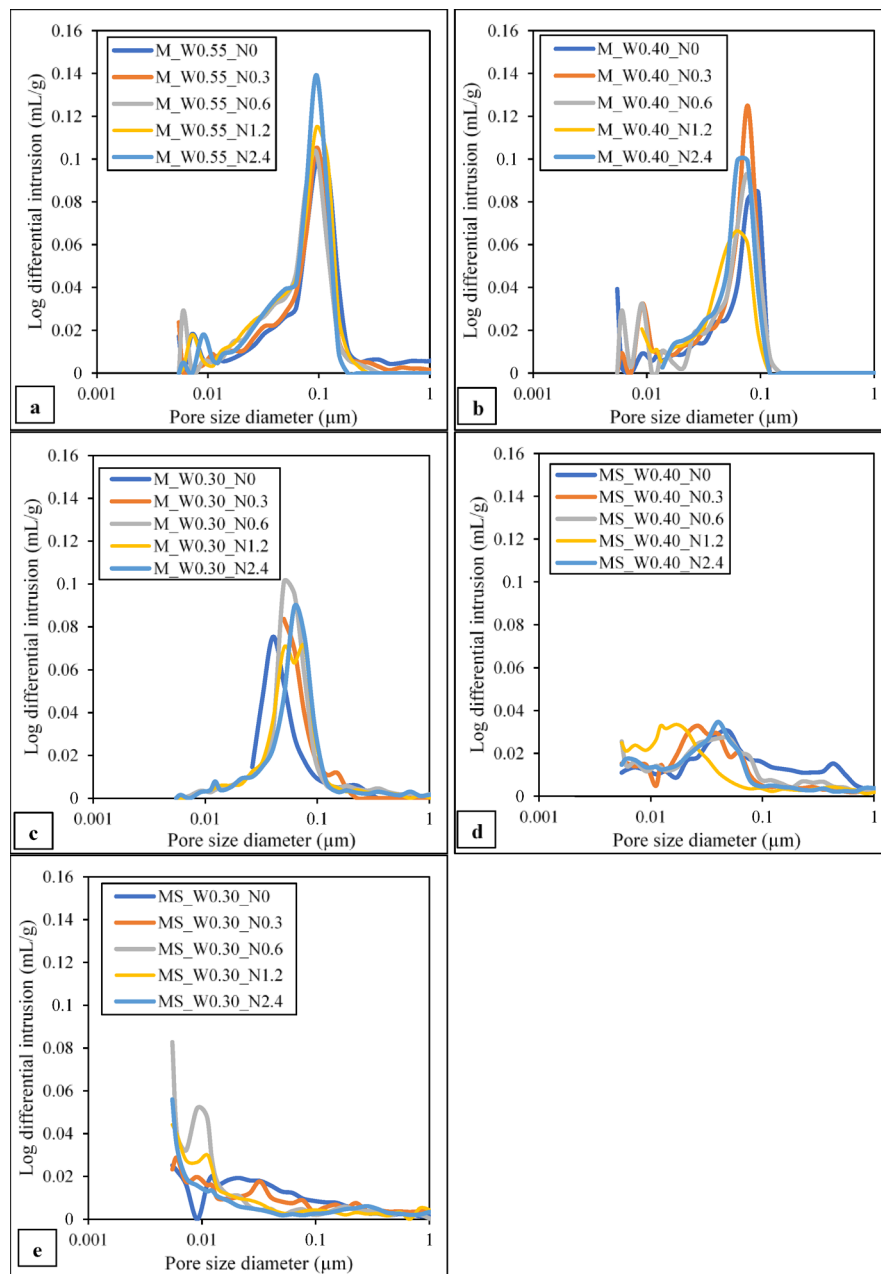


Fig. 8. Pore size distribution of (a) M_W0.55 series, (b) M_W0.40 series, (c) M_W0.30 series, (d) MS_W0.40 series, (e) MS_W0.30 series.

this study. The higher total pore area of the silica fume series (about 5 to 7 m²/g, against 2 to 4 m²/g for the plain series) is consistent with large capillaries being converted into fine capillary and gel pores. It increases surface area while decreasing connectivity and increasing tortuosity^{71,72}. The refinement therefore concentrates in the capillary range, the scale that governs transport, while gel pores and macropores change little. In some mixes (for instance, M_W0.30 series), the total mercury intrusion porosity rises slightly with nGP dosage, while strength does not fall in step. This reflects the fact that strength is controlled by the pore-size distribution, the critical pore diameter, and the interfacial transition zone, rather than by the total intruded volume^{71,73}. nGP refines the critical pore diameter and locally densifies the interfacial zone. The rise in total porosity stems from a few larger entry pores and the ink-bottle effect of mercury intrusion^{66,74}. Furthermore, strength and durability can be improved by controlling the pore structure through the addition of fine-sized additives^{75,76}. The refined fine-pore fraction and denser interface sustain strength despite the small increase in porosity. Specimens with porosity below 7% consistently exhibit compressive strengths exceeding 100 MPa and capillary absorption rates below 4×10^{-3} mm/√s, establishing clear performance thresholds for high-performance applications. The median pore diameter emerges as a particularly sensitive parameter, with values below 0.03 μm consistently associated with superior durability, reflecting the importance of pore-size refinement in controlling transport properties.

The MIP analysis validates the microstructural observations from SEM examinations, confirming that surface smoothness and matrix density correlate directly with pore structure refinement. The exceptional pore characteristics achieved in the MS_W0.30_N1.2 and MS_W0.30_N2.4 formulations, combining porosity values below 6% with median pore diameters below 0.03 μm , establish these systems as promising candidates for aggressive environmental applications requiring superior durability. These findings provide fundamental insights into the pore-scale mechanisms governing performance enhancement in nano-modified cementitious composites and establish quantitative relationships between pore-structure parameters and engineering properties, thereby supporting the development of optimized mix designs for advanced construction applications.

Conclusions

This study systematically explored the synergistic effects of graphene nanoplatelets (nGP) and silica fume (SF) on the fresh and hardened properties of cementitious mortars across a range of water-to-binder (w/b) ratios. The integration of nGP at controlled dosages significantly influenced rheology, strength development, pore structure, and capillary transport characteristics. The key findings can be summarized as follows:

- Matrix performance level governs the optimum nGP dosage. Low-performance mortar (0.55 w/b ratio) barely responded to nGP, with compressive strength near 63 MPa at all dosages. High-performance mortar (0.30 w/b with 11% silica fume) peaked at 1.2% nGP and reached 118.2 MPa.
- Flowability fell as the nGP dosage rose. The drop was largest at low water-to-binder ratios and in silica fume mixes. Workability stayed acceptable up to 0.6% nGP with superplasticizer adjustment.
- Compressive strength scaled inversely with porosity (correlation coefficient of -0.89). The strongest mix, MS_W0.30_N1.2, combined a 0.30 w/b ratio, 1.2% nGP, and 11% silica fume.
- Capillary transport dropped sharply in dense matrices. Initial absorption fell by 54% from water-to-binder 0.55 to 0.30, and by a further 43% to 50% with silica fume. MS_W0.30 mixes reached initial absorption below $4 \times 10^{-3} \text{ mm}/\sqrt{\text{s}}$.
- Mercury intrusion and microscopy confirmed pore refinement. The median pore diameter dropped to 0.0156 micrometers in MS_W0.30_N1.2. Mixes with porosity below 7% exceeded 100 MPa.
- Excess nGP (2.4%) gave no further gain and locally raised water absorption, up to 8.77% in M_W0.40_N2.4. This is consistent with dosage-dependent re-agglomeration.
- Based on the data obtained from the study, the conclusion is that the optimal nGP dosage is not a fixed material constant. This optimal amount of nanomaterial must be adjusted according to the target performance level of the matrix.

In conclusion, the findings underscore the considerable potential of nano-engineered cementitious composites incorporating graphene nanoplatelets and silica fume. Through appropriate mix design and dosage optimization, it is possible to achieve high-performance mortars with enhanced mechanical strength and resistance to environmental degradation. These results hold significant promise for applications requiring advanced durability in aggressive service environments and contribute valuable insights toward the development of next-generation sustainable construction materials.

Data availability

The datasets used and/or analysed during the current study available from the corresponding author on reasonable request.

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Declarations

Competing interests

The authors declare no competing interests.

Informed consent

All the authors have been listed, and the authors have no objection for the authorship list.

Additional information

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