



Synergistic effects of low-cost graphene and coal fly ash on the strength–porosity performance of cellular lightweight concrete

Yola Bertilsya Hendri¹, Ahlun Nazar¹, Geta Arianty¹, Dhina Febryza¹, Hussein A. Miran², Zainab N. Jaf², M. Mahbubur Rahman³, Hatem Taha⁴, Zhong Tao Jiang⁴, Amun Amri^{1*}

¹Department of Chemical Engineering, Universitas Riau, Indonesia

²Department of Physics, College of Education for Pure Science, Ibn Al-Haitham, University of Baghdad, Iraq

³Department of Physics, Jahangirnagar University, Bangladesh

⁴Surface Analysis and Materials Engineering Research Group, School of Engineering & Information Technology, Murdoch University, Australia

Abstract

Cellular lightweight concrete (CLC) is attractive for envelope and infill applications due to its low density and low thermal and sound insulation. However, its high pore volume often reduces compressive strength and limits wider application. Graphene-based additives and fly ash have been widely studied in cementitious composites. However, their combined effect in low-density CLC remains insufficiently understood, particularly regarding the strength-porosity balance, cellular pore stability, and microstructural mechanism. This study investigated the synergistic effect of low-cost graphene produced by turbulence-assisted shear exfoliation (TASE) and coal fly ash on CLC with a target density of approximately $1.1 \text{ g}\cdot\text{cm}^{-3}$. TASE graphene was selected because its aqueous shear-exfoliation route offered lower processing complexity, better scalability, and stronger cost potential than many commercial or oxidized graphene derivatives, making it more suitable for large-volume cementitious applications. CLC mixtures containing 0–15 wt.% graphene and 0 or 5 wt.% fly ash were cast and cured for 14 and 28 days. Compressive strength, porosity, and microstructure (SEM/TEM) were evaluated. Among the investigated mixtures, the mixture containing 10 wt.% graphene and 5 wt.% fly ash produced the best strength–porosity performance, achieving the highest 28-day compressive strength of $1.86 \pm 0.20 \text{ MPa}$ and the lowest porosity of 23.70%. Compared with the graphene-free mixture at the same fly ash level, this composition increased strength by 64.60% and reduced porosity by 14.84%. SEM/TEM observations supported a dosage-dependent mechanism involving graphene-assisted nucleation, fly ash-assisted pozzolanic densification, pore-wall refinement, and reduced reinforcement efficiency at excessive graphene loading. These findings supported the practical design of graphene–fly ash modified CLC material systems.

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Corresponding Author:

Amun Amri

Department of Chemical Engineering, Universitas Riau, Indonesia

Email:

amun.amri@eng.unri.ac.id

INTRODUCTION

Cellular lightweight concrete (CLC), also known as foamed concrete, has been widely

used for non-structural walls, envelopes, partitions, and infill applications due to its low density, high flowability, and favorable

insulating properties [1, 2, 3]. These characteristics make CLC useful for reducing dead loads and improving construction efficiency. However, the high air-void content that gives CLC its lightweight character also reduces its load-bearing capacity [4]. This limitation becomes more critical at low density because CLC strength is strongly affected by pore characteristics, including pore volume, pore connectivity, and cell-wall continuity [2, 3, 5]. Therefore, CLC requires a material strategy that improves matrix compactness and compressive strength without sacrificing its low-density and insulating advantages.

Several studies on CLC and lightweight cementitious composites focus on fiber and nano-silica reinforcement. Fibers can bridge cracks and improve flexural resistance, while micro-silica and nano-silica can refine the hydration network and reduce pore size [3, 6, 7]. Although these approaches improve mechanical performance, they also have important limitations. Fiber incorporation may reduce flowability and increase mixture complexity, especially in low-density foamed systems [8]. Silica-based nanomodification may also increase cost and be highly sensitive when the dosage is not properly controlled [6][7]. Thus, although these additives can improve strength, they do not fully address the combined challenges of strength, processability, density control, and cost-efficiency in low-density CLC.

This limitation has encouraged the use of carbon-based nanomaterials as alternative reinforcement agents for cementitious composites. Among these, graphene-based nanomaterials, including graphene oxide, graphene nanoplatelets, and few-layer graphene, have received increasing attention for their ability to promote C-S-H nucleation, pore filling, crack bridging, and nanoscale load transfer [9][10]. Graphene has a two-dimensional, atom-thick structure composed of a hexagonal network of sp^2 -hybridized carbon atoms [11]. Reddy et al. [12] reported that graphene oxide and fly ash improved the flexural, split tensile, and compressive strength of concrete by 16%, 12%, and 10%, respectively, at 28 days. Chen et al. [13] also showed that graphene-reinforced fly ash concrete had higher compressive and flexural strength than concrete without graphene. However, graphene performance remains sensitive to dosage, dispersion quality, and compatibility with the alkaline cement pore solution [14]. In addition, many previous studies used commercial graphene or oxidized

graphene derivatives, which increase processing complexity and limit large-volume applications. Therefore, turbulence-assisted shear exfoliation (TASE) graphene is relevant because it offers a scalable aqueous shear-exfoliation route with lower processing complexity and stronger potential for cost-efficient cementitious applications [15].

Although graphene offers nanoscale reinforcement, its effectiveness can be influenced by the surrounding cementitious matrix and the availability of reactive supplementary materials. In this context, fly ash is relevant because it promotes matrix densification, pore refinement, and more sustainable binder design. Coal fly ash is a widely available industrial by-product that reduces cement consumption and supports more sustainable concrete production [16, 17, 18]. In cementitious systems, fly ash acts as a fine filler and pozzolanic precursor. Its silica and alumina phases react with calcium hydroxide to form secondary C-S-H or C-A-S-H phases, thereby improving matrix densification and reducing pore connectivity [16, 17, 19]. In foamed or cellular concrete, fly ash contributes to framework densification and influences pore-structure-related performance, although its effect depends strongly on mixture composition and fresh-state stability [17, 18, 20]. However, the effect of fly ash is not always linear. Excessive fly ash may reduce early-age strength, alter slurry viscosity, affect bubble stability, or impair mechanical performance if the degree of reaction and mixture balance are not properly controlled [17, 18, 21]. Therefore, fly ash must be used at a suitable and carefully controlled dosage, especially in low-density CLC systems.

However, the performance of low-density CLC cannot be explained only by hydration and pozzolanic reactions. Unlike dense mortar or normal concrete, CLC contains a large volume of entrained air voids, making its mechanical response closely related to foam stability, air-void distribution, and slurry rheology [2][21]. Changes in slurry characteristics can disturb air-void stability before setting, leading to connected pores or weak zones in the hardened matrix [2]. Therefore, findings from dense graphene-cement composites cannot be directly transferred to low-density CLC. In this system, graphene dispersion, fly ash reaction, and pore-structure control must be considered together.

A critical synthesis of previous studies shows that no single strategy fully addresses the strength limitation of low-density CLC. Fiber

and nano-silica reinforcement can improve mechanical performance but may affect flowability, density, cost, and dispersion stability [6, 7, 8]. Graphene-based nanomaterials offer nanoscale strengthening [9, 10], while fly ash supports pozzolanic densification and pore refinement [18, 22]. However, both approaches require careful dosage control because poor

dispersion or unbalanced mixture design can reduce their effectiveness. These findings indicate that CLC improvement requires a balanced strategy that integrates nanoscale reinforcement, pozzolanic reaction, and pore-structure control. Table 1 summarizes the most relevant studies and highlights the position of the present work.

Table 1. Comparative Summary of Previous Studies Related to Lightweight Cementitious Composites, Graphene-based Additives, and Fly Ash Systems

Ref.	Composite/System	Additive/Modifier	Investigated Factors	Conducted Tests and Sample Monitoring	Main Findings and Conclusions/Remarks
Shcherban' et al. [6]	Lightweight fiber-reinforced concrete	Micro-silica	Nanomodification effect on lightweight concrete quality	Density, strength-related properties, and constructive quality coefficient	Micro-silica improved the quality of lightweight fiber-reinforced concrete through matrix densification. However, silica-based modification requires dosage control and may increase mixture sensitivity.
Alghrairi et al. [7]	Lightweight concrete	Nano-silica	Effect of nano-silica dosage on mechanical performance	Mechanical property testing of lightweight concrete	Nano-silica improved mechanical properties by refining the cementitious matrix. The study supports the role of nanoscale additives, but dispersion and cost remain practical concerns.
Reddy et al. [12]	High-strength concrete	Graphene oxide and fly ash	Synergistic effect of graphene oxide and fly ash on concrete performance	Workability, flexural strength, split tensile strength, compressive strength, and microstructural analysis	Graphene oxide and fly ash improved flexural, split tensile, and compressive strength by 16%, 12%, and 10%, respectively. However, the system was conducted on dense concrete, and the role of CLC pore architecture was not addressed.
Chen et al. [13]	Fly ash concrete	Graphene	Effect of graphene on fly ash concrete strength and durability	Compressive strength, flexural strength, and durability-related evaluation	Graphene increased compressive and flexural strength by 12.89% and 10.78%, respectively. The study confirmed the reinforcing effect of graphene in fly ash concrete but did not evaluate low-density cellular concrete.
Liu et al. [18]	High-belite sulphoaluminate cement-based foam concrete	Fly ash	Effect of fly ash on bonding and shrinkage behavior	Bonding behavior, shrinkage behavior, and foam concrete performance monitoring	Fly ash influenced the bonding and shrinkage behavior of foam concrete. The study showed that fly ash can modify the performance of foamed concrete, but graphene reinforcement was not considered.
Liu et al. [23]	Lightweight cellular concrete	Fly ash	Strengthening mechanism of fly ash-filled cellular concrete	Mechanical testing and microstructural evaluation	Fly ash improved the framework structure of lightweight cellular concrete through hydration and pozzolanic reactions. The study supports the use of fly ash in CLC but did not examine the graphene-fly ash synergy.
Xiao et al. [2]	Foamed concrete	Slurry characteristics	Effect of slurry properties on bubble stability	Bubble stability monitoring and fresh-state behavior analysis	Bubble stability was strongly affected by slurry characteristics. This finding is important for CLC because pore structure and foam stability strongly influence its hardened performance.
Ababneh et al. [24]	Mortar containing high levels of natural pozzolan	Graphene nanoplatelets	Effect of graphene nanoplatelets on compressive strength	Compressive strength testing of pozzolan-based mortar	Graphene nanoplatelets enhanced the compressive strength in mortar. However, dense mortar differs from low-density CLC because it lacks a cellular pore architecture.

Based on the state-of-the-art comparison in Table 1, previous studies have improved cementitious composites using fibers, nano-silica, graphene-based nanomaterials, and fly ash. However, these studies have mainly focused on dense concrete, mortar, fly ash concrete, or general foamed concrete. Quantitatively, graphene oxide-fly ash concrete showed strength improvements of around 10–16% in dense concrete systems [12], while graphene-reinforced fly ash concrete increased compressive and flexural strength by 12.89% and 10.78%, respectively [13]. The present study differs from these previous works by evaluating low-density CLC, where strength development is closely linked to porosity, foam stability, and microstructural densification.

The following research gaps can therefore be identified. First, limited studies have evaluated low-cost TASE graphene in low-density CLC, despite its potential advantages in scalable production, lower processing complexity, and cost-efficient large-volume application. Second, the combined effect of TASE graphene and coal fly ash on the strength–porosity balance of CLC has not been sufficiently quantified. Third, limited evidence connects graphene dosage, fly ash addition, porosity reduction, and SEM/TEM-based microstructural observations within a single CLC system. Fourth, the effective graphene dosage range for CLC remains unclear because its effect may differ from dense cementitious systems. Fifth, a mechanistic framework linking graphene-assisted nucleation of hydration products, fly ash-assisted pozzolanic densification, cellular pore-wall continuity, and strength–porosity performance in low-density CLC has not been clearly established.

This study addresses these gaps by investigating TASE graphene and 5 wt.% coal fly ash as combined modifiers for low-density CLC with a target density of approximately 1.1 g.cm⁻³. Graphene was added at 0, 5, 10, and 15 wt.%, while coal fly ash was used at 0 and 5 wt.%. The study evaluates whether TASE graphene improves compressive strength within the investigated dosage range, whether coal fly ash reduces porosity and enhances strength through pozzolanic C-S-H/C-A-S-H enrichment, and whether excessive graphene weakens the CLC matrix through reduced dispersion efficiency and local defect formation. The novelty of this work lies in integrating scalable, low-cost TASE graphene production, fly ash incorporation, strength–porosity performance, and SEM/TEM-based microstructural evidence in a single low-density CLC system.

The theoretical contribution of this study is the formulation of a strength–porosity–microstructure framework for TASE graphene–fly-ash modified CLC. The framework interprets CLC performance through three coupled mechanisms. First, graphene facilitates the nucleation of hydration products and microcrack bridging [22, 25, 26]. Second, fly ash promotes pozzolanic densification through the formation of secondary C-S-H/C-A-S-H phases [16, 17, 20, 27]. Third, cellular pore-wall continuity, governed by pore structure and foam stability, controls the strength–porosity response of low-density CLC [2, 5, 21]. Together, these mechanisms indicate that strength development in low-density CLC is governed by hydration or total porosity and by interactions among nanoscale reinforcement, secondary hydration products, and cellular pore-structure stability. Therefore, the proposed framework provides a mechanistic basis for interpreting the dosage-dependent behavior of TASE graphene–fly-ash modified CLC.

METHOD

Materials Preparation

Coal fly ash (type C) obtained from a steam power plant (PLTU) in Pekanbaru was first sun-dried to reduce its moisture content. Subsequently, the fly ash was sieved, and only the fraction that passed through a 30-mesh sieve was used. Before mixing, the sand was also sieved, and only the fraction passing through a 30-mesh sieve was used. The fly ash was subsequently mixed with the sand until a homogeneous mixture was obtained. Graphene was produced using the low-cost, simple, and environmentally friendly turbulence-assisted shear exfoliation (TASE) method, as reported in [15].

Preparation of Cellular Lightweight Concrete

Cellular Lightweight Concrete (CLC) bricks were prepared by mixing all the materials. The mixing process was conducted manually, beginning with the preparation of a homogeneous cement–water mixture, followed by the addition of sand, fly ash, and graphene. In this study, the coal fly ash and graphene dosages were expressed as wt.% relative to the cement mass. Coal fly ash was added at 0 and 5 wt.% of the cement mass, while graphene was added at 0, 5, 10, and 15 wt.% by cement mass. The mixture was then stirred until homogeneous. Subsequently, foam was added by diluting the foaming agent with water at a 1:20 volume ratio. The mixture is then stirred using a mixer until the paste expands. After mixing, the paste was

poured into metal molds shaped like cubes with dimensions of $10 \times 10 \times 10 \text{ cm}^3$. The mixture was compacted to fill the entire mold. The molded CLC specimens were then dried at room temperature for 48 hours. After 48 hours, the specimens were demolded and allowed to rest at room temperature until compressive strength measurements at 14 and 28 days.

The experimental design followed an input-mechanism-output research model. Graphene dosage, coal fly ash content, and curing age were treated as the main input variables. These variables were expected to influence the CLC matrix through C-S-H nucleation, pozzolanic reaction, pore refinement, graphene dispersion, and graphene agglomeration. At an appropriate dosage, graphene was expected to provide nucleation sites for hydration products and contribute to nanoscale crack bridging. In contrast, coal fly ash was expected to supply reactive silica and alumina species for the formation of secondary C-S-H or C-A-S-H phases. These mechanisms were evaluated using the output variables: compressive strength, porosity, and SEM/TEM-based microstructural morphology. The model assumes that graphene-modified CLC may exhibit a dosage-dependent response rather than a strictly linear

reinforcement response, as excessive graphene may agglomerate and create local defects in the cellular matrix. Figure 1 shows the flowchart for the production and characterization of CLC.

Characterization

CLC (Cellular Lightweight Concrete) aged 14 and 28 days underwent density tests to determine the lightweight brick density. This was followed by compressive strength tests in accordance with ASTM C 579-01 and porosity tests in accordance with ASTM C 642-21. In this experiment, two specimens were tested for compressive strength for each variation of fly ash (0% and 5%) and graphene (0%, 5%, 10%, and 15%), resulting in a total of 16 specimens. For porosity testing, one specimen was used for each variation of fly ash and graphene, with a total of 8 specimens. The compressive strength data were analyzed using descriptive statistics and reported as mean $\pm$ standard deviation. Porosity data were reported as descriptive values because only one specimen was tested for each mixture. Subsequently, the surface atom morphology of CLC was analyzed using scanning electron microscopy (SEM) coupled with SEM-EDX JEOL JSM-6510LA.

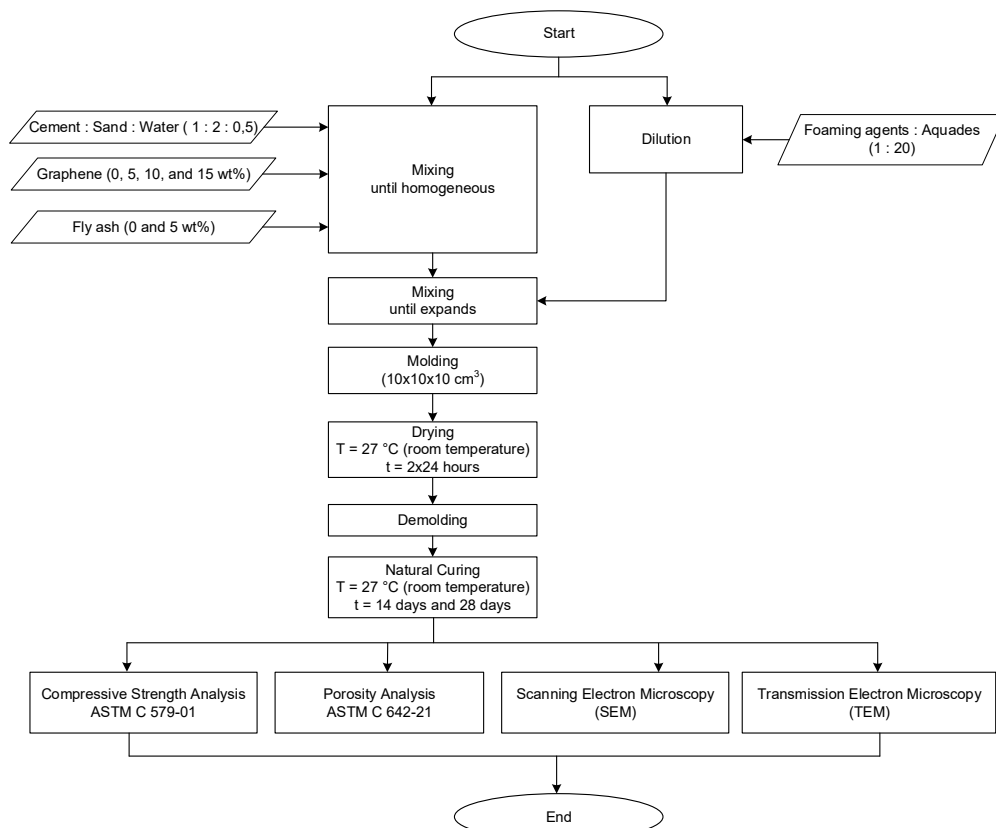


Figure 1. Experimental Workflow for The Preparation and Characterization of CLC Incorporating Low-Cost Graphene and Coal Fly Ash

The morphology of graphene formation and distribution within CLC was analyzed using transmission electron microscopy (TEM) with an HR TEM H9500 instrument.

RESULTS AND DISCUSSION

Compressive Strength Analysis

Figure 2 illustrates the compressive strength development of CLC after 14 and 28 days. The 28-day samples generally showed higher strength than the 14-day samples, indicating continued hydration and matrix development during curing. This later-age improvement is associated with the progressive formation of hydration products, especially C-S-H, which densifies the cementitious matrix and improves the load-bearing capacity of pore walls [5][28]. However, the strength response was not linear with graphene dosage. The results showed a dose-dependent pattern, with strength increasing up to 10 wt.% graphene before decreasing at 15 wt.% graphene. Since the compressive strength values were obtained from two specimens for each mixture, the results are interpreted as descriptive trends rather than statistically confirmed differences.

Figure 2(a) shows the compressive strength response of the fly ash-free CLC mixtures. At 28 days, graphene addition increased compressive strength by approximately 11.6% at 5 wt.% and 32.9% at 10 wt.% compared with the graphene-free control. The 14-day results showed a similar pattern, with strength increasing by approximately 15.3% and 43.7% at 5 and 10 wt.% graphene, respectively. These increases suggest that a moderate graphene addition improved the CLC matrix through hydration-product nucleation, pore filling, microcrack bridging, and nanoscale load transfer. This interpretation aligns with recent studies reporting that well-dispersed graphene-family materials can improve hydration behavior, matrix compactness, interfacial bonding, and mechanical performance in cementitious composites [22, 25, 26].

The decrease in strength at 15 wt.% graphene indicates that the reinforcing effect was dosage-dependent. In the fly ash-free mixtures, increasing graphene from 10 wt.% to 15 wt.% reduced compressive strength by approximately 15.3% at 28 days and 15.8% at 14 days. This decline suggests that excessive graphene promoted agglomeration, reduced dispersion uniformity, and created weak zones within the CLC matrix. This explanation aligns with Hu et al. [22] and Prudente et al. [26], who emphasized that poor dispersion of graphene-family materials

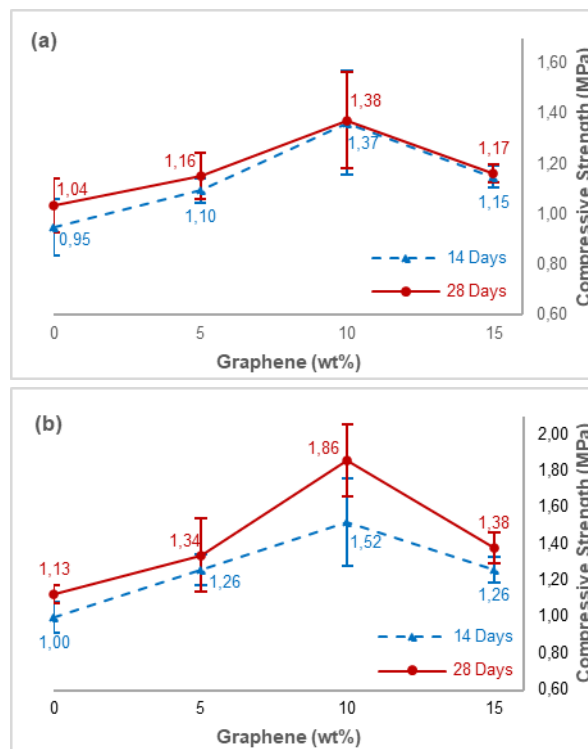


Figure 2. Compressive Strength of CLC at 14 and 28 Days With the Addition of (a) Graphene (Fly Ash-Free), (b) Graphene and 5% Fly Ash

in alkaline cementitious systems can limit reinforcement efficiency, disturb hydration-product distribution, and generate local microstructural defects.

Figure 2(b) shows the compressive strength of CLC mixtures containing 5 wt.% coal fly ash. In this system, graphene produced a stronger strengthening response than in the fly ash-free mixtures. At 28 days, compressive strength increased by approximately 19.1% at 5 wt.% graphene and 65.3% at 10 wt.% graphene compared with the graphene-free fly ash mixture. At 14 days, the corresponding increases were approximately 26.0% and 52.0%. These results indicate that coal fly ash enhanced the effectiveness of graphene, particularly at 10 wt.% graphene, which was the best-performing dosage among the investigated graphene levels. This improvement can be attributed to the complementary action of graphene-assisted nucleation and fly ash-assisted pozzolanic densification, where fly ash supplies reactive silica and alumina for the formation of additional C-S-H or C-A-S-H phases [25][27]. Reactive Si-OH and Al-OH groups are generated through the dissolution of the coal fly ash structure under alkaline conditions in the presence of OH⁻ ions. These species subsequently react with calcium ions to form secondary C-S-H phases with

properties similar to those of cement hydration products. Therefore, the formation of these C-S-H compounds is a key factor in enhancing concrete quality [29][30].

The strength reduction at excessive graphene dosage was more pronounced in the fly ash-containing mixtures. Increasing graphene from 10 wt.% to 15 wt.% reduced the 28-day compressive strength by approximately 25.8%. Although the 15 wt.% graphene mixture still showed higher strength than the graphene-free fly ash mixture, the decline relative to the 10 wt.% graphene mixture indicates that excessive graphene may have disturbed the balance among nanoscale reinforcement, pozzolanic reaction, and cellular pore-wall stability. This effect is important in CLC because its strength is controlled by cementitious matrix density, pore structure, slurry behavior, and bubble stability [2]. Therefore, graphene agglomeration at high dosage may weaken pore-wall continuity and reduce the effectiveness of fly ash-induced densification.

The comparison between Figure 2(a) and Figure 2(b) further clarifies the contribution of coal fly ash. After 28 days, the addition of 5 wt.% coal fly ash increased compressive strength by approximately 8.7%, 16.0%, 35.3%, and 18.5% at 0, 5, 10, and 15 wt.% graphene, respectively, compared with the corresponding fly ash-free mixtures. The greatest improvement attributable to fly ash occurred at 10 wt.% graphene, indicating that fly ash did not act merely as a passive filler. Its contribution was greater when graphene remained sufficiently dispersed and mechanically active. After 14 days, the fly ash-related improvement was lower, ranging from approximately 5.3% to 15.1%, suggesting that fly ash contributed more strongly to later-age strength development through continued pozzolanic reaction [27].

The trends observed in this study align with previous findings on graphene- and pozzolan-modified cementitious composites. Reddy and Prasad [25] reported that graphene oxide-fly ash hybridisation improved hydration, microstructure, and mechanical characteristics of cement concrete, while Ababneh et al. [24] showed that graphene nanoplatelets enhanced the compressive strength of mortar containing high levels of natural pozzolan. Jaramillo and Kalfat [9] also reported that graphene nanoplatelets improved the mechanical performance of concrete. The present results align with these studies, showing that graphene can improve strength when used at an effective dosage. However, the strength decreased at 15 wt.% graphene also confirms that the benefit of

graphene is strongly dependent on dispersion quality and dosage control.

The mixture containing 10 wt.% graphene and 5 wt.% coal fly ash produced the highest observed strength response. Relative to the mixture without graphene and without fly ash, this composition increased 28-day compressive strength by approximately 79.7%. This improvement exceeded the response obtained from graphene alone or fly ash alone, suggesting a synergistic interaction between nanoscale reinforcement and pozzolanic densification. In this mechanism, graphene contributes to hydration-product nucleation, crack bridging, pore filling, and interfacial load transfer, while coal fly ash contributes to secondary hydration products and pore-wall densification [22, 25, 26]. This combined effect is particularly relevant for CLC because its load-bearing capacity depends strongly on the compactness and continuity of the pore walls. Overall, the results indicate that 10 wt.% graphene was the best-performing dosage among the investigated graphene levels under the present experimental conditions, especially when combined with 5 wt.% coal fly ash.

Porosity Analysis

Figure 3 presents the porosity of CLC with different graphene dosages and coal fly ash contents. Since only one specimen was tested for each porosity condition, the values are interpreted as descriptive indicators of pore-structure development rather than statistically confirmed differences. Overall, moderate graphene addition reduced porosity. However, the response was not strictly linear, indicating that pore refinement in CLC depends on dosage balance, dispersion quality, and cellular pore stability. In foamed concrete, pore development is strongly influenced by slurry characteristics, bubble stability, pore distribution, and density variation [2].

Figure 3 shows that graphene addition reduced the porosity of the fly ash-free mixtures by approximately 14.9% at 5 wt.% and 19.9% at 10 wt.% compared with the graphene-free control. This trend indicates that graphene addition up to 10 wt.% improved the pore structure of CLC by reducing accessible voids and increasing pore-wall compactness. Similar pore-refinement effects have been reported for graphene-family cementitious composites, where adequate dispersion supports hydration-product development and matrix refinement [9][22]. This interpretation is further supported by Mokhtar [31], who reported that nano-metakaolin/graphene oxide modification reduced pore size and pore volume in cement mortar.

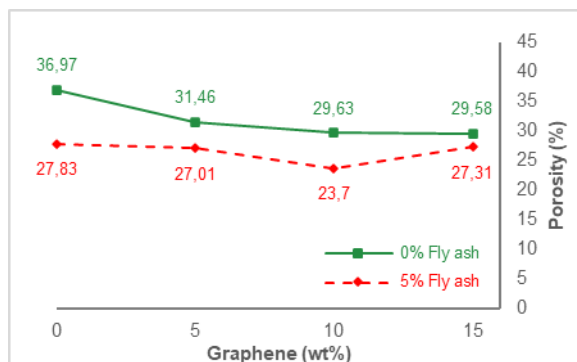


Figure 3. The Influence of Graphene and Fly Ash Addition on the Porosity of CLC Bricks

Jaramillo and Kalfat [9] also showed that graphene nanoplatelets improved compressive strength and reduced permeable voids when properly dispersed.

However, increasing graphene to 15 wt.% produced only a marginal additional porosity reduction of approximately 0.2% in the fly ash-free mixtures. At the same time, the 28-day compressive strength decreased by approximately 15.2%. This indicates that total porosity alone did not fully govern the mechanical response at excessive graphene dosage. The result suggests that pore connectivity, local defects, and graphene agglomeration may reduce the mechanical performance by limiting the effective reinforcing and pore-refining functions of graphene [22][32].

The addition of 5 wt.% coal fly ash consistently reduced porosity compared with the corresponding fly ash-free mixtures. The porosity reductions caused by fly ash addition were approximately 24.7%, 14.1%, 20.0%, and 7.7% at 0, 5, 10, and 15 wt.% graphene, respectively. The largest fly ash-related porosity reduction occurred at 10 wt.% graphene, indicating that the pore-refinement role of fly ash was most effective at the best-performing graphene level in this study. This behavior aligns with studies showing that fly ash and pozzolanic additives can improve compactness and hardened performance in

foamed and cementitious materials when used at suitable contents [20][31].

The mixture containing 10 wt.% graphene and 5 wt.% coal fly ash showed the lowest porosity, corresponding to a 35.9% reduction relative to the mixture without graphene and fly ash. This mixture also produced the highest 28-day compressive strength, representing an increase of approximately 78.8% relative to the control mixture, as summarized in Table 2. Therefore, the best strength–porosity performance was obtained when lower accessible porosity was accompanied by sufficient pore-wall continuity. This coupled change confirms that excessive graphene did not further improve the pore structure and likely introduced agglomeration-induced defects within the cellular matrix. Recent X-ray computed tomography evidence has also shown that graphene nanoplatelet re-agglomeration can reduce effective surface area and alter the reported effective dosage in hardened cement composites [32].

Table 2 presents the descriptive statistical analysis of compressive strength and porosity. Since only two specimens were tested for compressive strength and one specimen for porosity in each mixture, the analysis was limited to descriptive statistics. Therefore, the compressive strength results are reported as mean $\pm$ standard deviation and coefficient of variation (CV), while porosity is interpreted as a descriptive indicator of pore-structure development. After 28 days, compressive strength CV ranged from 3.42% to 14.93%, providing a relative measure of data variability for descriptive comparison. Among the investigated mixtures, the composition containing 10 wt.% graphene and 5 wt.% coal fly ash showed the best strength–porosity performance, with the highest 28-day compressive strength of 1.86 ± 0.20 MPa, a CV of 10.75%, and the lowest measured porosity of 23.70%.

Table 2. Descriptive Statistical Analysis of Compressive Strength and Porosity of CLC Mixtures

Fly ash (wt.%)	Graphene (wt.%)	Compressive strength (MPa)				Porosity (%)	28-day strength change vs same-fly ash control (%)	Porosity change vs same-fly ash control (%)
		14-day	CV 14-day	28-day	CV 28-day			
0	0	0.95 ± 0.11	11.58	1.04 ± 0.11	10.58	36.97	0.00	0.00
0	5	1.10 ± 0.05	4.55	1.16 ± 0.09	7.76	31.46	11.54	-14.90
0	10	1.37 ± 0.21	15.33	1.38 ± 0.19	13.77	29.63	32.69	-19.85
0	15	1.15 ± 0.04	3.48	1.17 ± 0.04	3.42	29.58	12.50	-19.99
5	0	1.00 ± 0.08	8.00	1.13 ± 0.05	4.42	27.83	0.00	0.00
5	5	1.26 ± 0.08	6.35	1.34 ± 0.20	14.93	27.01	18.58	-2.95
5	10	1.52 ± 0.24	15.79	1.86 ± 0.20	10.75	23.70	64.60	-14.84
5	15	1.26 ± 0.07	5.56	1.38 ± 0.08	5.80	27.31	22.12	-1.87

Compared with the graphene-free mixture at the same fly ash level, this composition increased the 28-day compressive strength by 64.60% and reduced porosity by 14.84%. However, when the graphene content increased to 15 wt.% in the fly ash-containing system, the 28-day compressive strength decreased to 1.38 ± 0.08 MPa, and porosity increased to 27.31%. These results indicate that 10 wt.% graphene was the best-performing dosage among the investigated graphene levels under the present experimental conditions, rather than a mathematically optimized universal dosage.

From a nanostructural perspective, the superior performance of the mixture containing 10 wt.% graphene and 5 wt.% coal fly ash can be attributed to a favorable balance among graphene-assisted nucleation of hydration products, pore-wall densification, microcrack bridging, and fly ash-assisted pozzolanic reaction. Well-dispersed graphene-family materials can provide nucleation surfaces for hydration products and improve matrix compactness. At the same time, fly ash supplies reactive silica and alumina species that support the formation of secondary C-S-H/C-A-S-H phases [25, 26, 27]. At 10 wt.% graphene, these mechanisms were likely balanced, resulting in lower porosity and higher compressive strength. However, increasing the graphene content to 15 wt.% may exceed the effective dispersion limit, increasing the tendency for sheet restacking, local agglomeration, and non-uniform pore-wall continuity. Such re-agglomeration can reduce the effective surface area of graphene, create weak regions, and reduce reinforcement efficiency in cementitious matrices [32][33]. Therefore, the reduced performance at 15 wt.% graphene is more likely related to decreased dispersion efficiency and local defect formation than to graphene content alone. This interpretation also aligns with reports that graphene can contribute to the formation of graphene-C-S-H hybrids when adequately dispersed. However, this reinforcing mechanism becomes less effective when excessive graphene promotes agglomeration [34].

Figure 4 clarifies the relationship between porosity and compressive strength. In general, lower porosity corresponded to higher 28-day compressive strength. Nevertheless, the fly ash-free mixtures containing 10 wt.% and 15 wt.% graphene show that nearly identical porosity values did not produce similar strength. Thus, the mechanical performance of graphene-modified CLC is controlled by total porosity, pore connec-

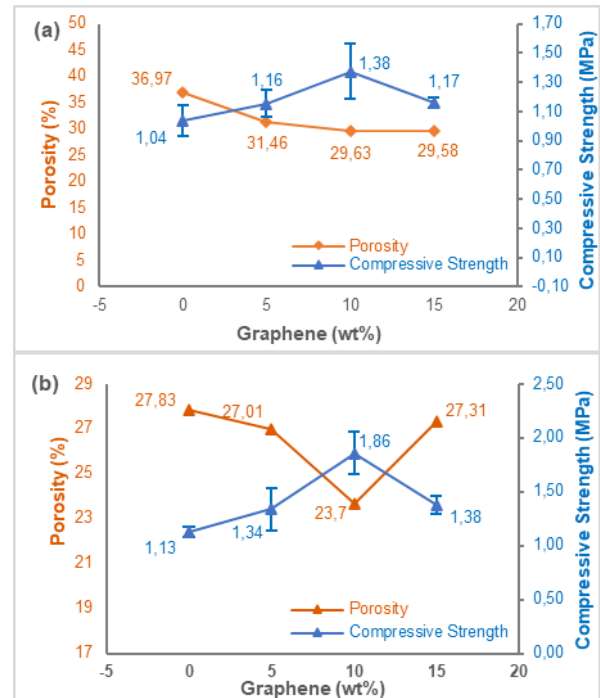


Figure 4. The Relationship Between Compressive Strength and Porosity of CLC With the Addition of (a) Graphene (Fly Ash-Free); (b) Graphene and 5% Fly Ash

tivity, pore-wall continuity, graphene dispersion, and the stability of the cellular pore system [2, 9, 32].

Compared with recent studies, the present results show a competitive porosity–strength response. Beskopylny et al. [20] reported that structural foam concrete containing 10% fly ash showed an approximately 12% increase in compressive strength. In the present study, 5 wt.% coal fly ash combined with 10 wt.% graphene increased 28-day compressive strength by approximately 34.8% compared with the corresponding fly ash-free mixture. Therefore, the porosity results support that the mixture containing 10 wt.% graphene and 5 wt.% coal fly ash achieved the best strength–porosity balance among the investigated mixtures, whereas 15 wt.% graphene reduced the overall efficiency of the system.

Scanning Electron Microscope (SEM) Analysis

Figure 5 depicts the morphological characteristics of lightweight CLC bricks obtained using a scanning electron microscope (SEM) at a magnification of 5000 times. Figure 5(a) presents the control CLC without graphene and coal fly ash. The control mixture in Figure 5(a) shows a relatively loose matrix with visible voids and less continuous pore walls. This morphology aligns

with the higher porosity and lower 28-day compressive strength of the control mixture.

Figure 5(b) presents the mixture containing 10 wt.% graphene and 5 wt.% coal fly ash. The mixture exhibited a denser and more continuous matrix. This observation aligns with the quantitative results, where this mixture showed the lowest porosity of 23.70% and the highest 28-day compressive strength of 1.86 MPa. Compared with the control mixture, porosity decreased by approximately 35.9%, while compressive strength increased by approximately 78.8%. The more compact morphology indicates improved pore-wall continuity and reduced accessible voids. This interpretation aligns with previous studies showing that adequately dispersed graphene can improve cement-based and geopolymer materials, including reducing permeable voids and enhancing matrix compactness [9].

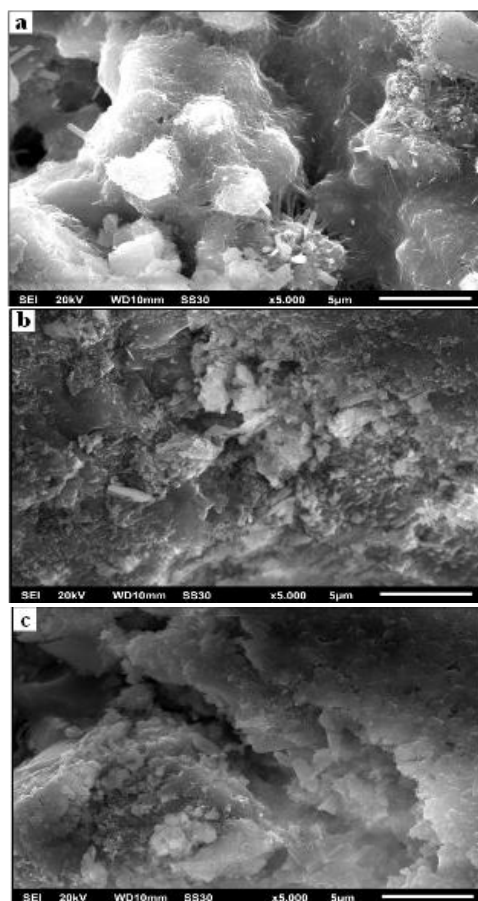


Figure 5. The SEM Analysis on CLC (a) Without the Addition of Graphene and Fly Ash, (b) With the Addition of 10% Graphene and 5% Fly Ash, and (c) With the Addition of 15% Graphene and 5% Fly Ash

In contrast, the mixture containing 15 wt.% graphene and 5 wt.% coal fly ash showed a less uniform morphology, as shown in Figure 5(c). Compared with the 10 wt.% graphene and 5 wt.% coal fly ash mixture, increasing graphene to 15 wt.% increased porosity by approximately 15.2% and reduced 28-day compressive strength by approximately 25.8%. This microstructural deterioration indicates that excessive graphene did not further densify the matrix. Instead, the higher graphene content likely promoted non-uniform dispersion and local agglomeration, interrupting pore-wall continuity and reduced the efficiency of pore refinement. Similar dosage-sensitive behavior was reported by Ahmed et al. [33], who found that higher graphene nanoplatelet aggregate content reduced compressive strength because of the agglomerated nature of the graphene-based additive.

Overall, the SEM observations support the dosage-dependent response observed in the compressive strength and porosity results. The mixture containing 10 wt.% graphene and 5 wt.% coal fly ash showed the most compact morphology and the best strength–porosity balance, whereas the 15 wt.% graphene mixture showed a less uniform microstructure and reduced performance. Therefore, the SEM results indicate that increasing graphene content does not linearly improve CLC performance and that excessive graphene addition may reduce matrix uniformity.

Transmission Electron Microscopy (TEM) Analysis

Figure 6 shows the TEM morphology of selected CLC mixtures. The TEM observations provide qualitative evidence to support the dispersion state of graphene within the CLC matrix. The control mixture in Figure 6(a) provides the baseline morphology for comparison with the graphene-containing mixtures. Hydration products cannot be identified with certainty based solely on TEM images, as C-S-H morphology may vary with hydration age, local chemical environment, and observation scale [35].

Figure 6(b) shows graphene-related sheet-like regions within the mixture containing 10 wt.% graphene and 5 wt.% coal fly ash. The relatively distributed graphene features support the quantitative results, as this mixture produced the lowest porosity and the highest 28-day compressive strength. This result indicates that the graphene population at 10 wt.% was sufficiently effective to support matrix refinement without severely disturbing the cellular structure.

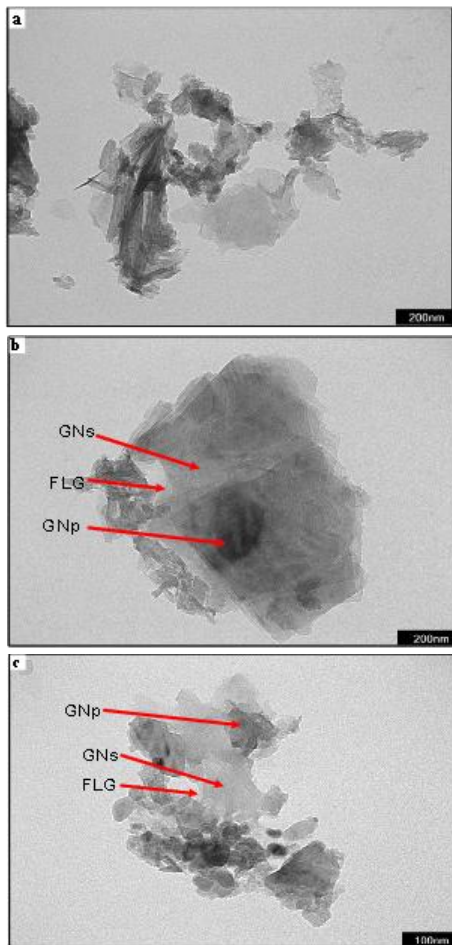


Figure 6. The TEM Analysis on CLC (a) Without the Addition of Fly Ash and Graphene, (b) With the Addition of 5% Fly Ash and 10% Graphene, and (c) With the Addition of 5% Fly Ash and 15% Graphene

Previous studies have also shown that the effectiveness of graphene in cement-based and geopolymer materials depends strongly on adequate dispersion, reducing permeable voids and improving matrix compactness [9][22]. Based on TEM contrast, the graphene-related features can be qualitatively classified into few-layer graphene (FLG, 3–6 layers), graphene nanosheets (GNs, 6–20 layers), and graphene nanoplatelets (GNp, >20 layers). Darker regions were associated with thicker multilayer domains, whereas more transparent regions were attributed to thinner graphene sheets [36][37]. However, this classification should be interpreted qualitatively because the TEM images were not used for statistical layer-number analysis.

In contrast, Figure 6(c) indicates a less favorable graphene distribution at 15 wt.% graphene. The darker and more concentrated graphene-related regions suggest a higher tendency toward multilayer stacking or local

agglomeration. Compared with the mixture containing 10 wt.% graphene and 5 wt.% coal fly ash, increasing the graphene to 15 wt.% increased porosity by approximately 15.2% and reduced 28-day compressive strength by approximately 25.8%. This confirms that excessive graphene did not further improve the CLC matrix. Instead, the higher dosage likely reduced dispersion uniformity and weakened the pore-wall structure. This interpretation aligns with recent studies showing that graphene nanoplatelet re-agglomeration and graphene aggregate formation reduce effective surface area, create weak regions, and change the dosage level required for efficient reinforcement in cementitious systems [22][33]. At an appropriate dosage, graphene acts as a template for C-S-H development, allowing hydration products to form around graphene sheets and potentially producing a stiffer graphene-C-S-H hybrid structure than C-S-H alone [34]. However, this reinforcing mechanism becomes less effective when excessive graphene promotes agglomeration. Overall, the TEM observations support the dosage-dependent behavior observed in the compressive strength, porosity, and SEM results.

CONCLUSION

This study investigates the combined effect of low-cost TASE graphene and coal fly ash on the strength–porosity performance of cellular lightweight concrete (CLC) with a target density of approximately $1.1 \text{ g}\cdot\text{cm}^{-3}$. The results showed a dose-dependent response, in which graphene improved compressive strength up to 10 wt.% among the investigated dosage levels but reduced performance at 15 wt.%. Among the tested mixtures, the composition containing 10 wt.% graphene and 5 wt.% coal fly ash showed the best strength–porosity performance, with the highest 28-day compressive strength of $1.86 \pm 0.20 \text{ MPa}$ and the lowest measured porosity of 23.70%. Compared with the graphene-free mixture at the same fly ash level, this composition increased the 28-day compressive strength by 64.60% and reduced porosity by 14.84%. Compared with the control mixture without graphene and fly ash, this composition increased compressive strength by approximately 78.8% and reduced porosity by approximately 35.9%. SEM and TEM observations corroborated the quantitative results by revealing a more compact matrix and a more favorable graphene distribution in the best-performing mixture. In contrast, increasing the graphene content to 15 wt.% reduced the system's efficiency, indicating that excessive graphene addition may promote

agglomeration and reduced pore-wall continuity. Because the present study used a limited number of specimens, the findings should be interpreted as descriptive trends rather than statistically confirmed differences. Future studies should include greater replication, statistical analysis, longer curing periods, durability evaluation, and quantitative pore-structure characterization of pore structure to better determine the effective graphene dosage range for low-density CLC.

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