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Mechanical and Microstructural Proxies of LC³ Mortars up to 50% Clinker Replacement Using a Southern Libyan Calcined Kaolinite Clay.

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ABSTRACT

This experimental study investigates the properties of LC³ (limestone-calcined clay) cement mortars with high clinker replacement, utilizing locally sourced kaolinite-rich clays from southern Libya. Mortar mixtures were prepared with clinker contents of 40, 50, and 60%. The mechanical performance of the LC³ blends was assessed through compressive and flexural strength tests, ultrasonic pulse velocity (UPV), porosity measurements, and workability evaluations. The results show that replacing up to 50% of cement with a limestone–calcined clay blend in mortar mixes achieves compressive and flexural strengths comparable to, or slightly exceeding, those of ordinary cement. At 28 days, mortars with 50% substitution reached the same compressive strength as CEM I (37 MPa) while exhibiting a 19.6% increase in flexural strength. Furthermore, higher replacement levels (up to 50%) improved pore structure refinement, leading to a denser microstructure. Mortars with 50% substitution demonstrated about a 9.65% reduction in porosity compared to CEM I.

KEYWORDS: LC³ blends cement, Clinker replacement, Compressive strength, Flexural strength, Workability, Porosity.

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1.INTRODUCTION

Metakaolin (MK), a highly reactive pozzolanic material, enhances both early-age strength and long-term durability in cementitious systems by refining their pore structure (Sabir et al., 2001). To reduce the high clinker content in conventional cement, supplementary cementitious materials (SCMs) are commonly employed; however, replacing more than 30% of clinker often reduces early strength. Given the limited availability of industrial by-product SCMs such as fly ash and slag, calcined clays rich in kaolinite have emerged as promising alternatives due to their ability to form reactive metakaolin upon thermal activation (Antoni et al., 2012) . Limestone calcined clay cement (LC^3), introduced in 2012, offers a sustainable solution by combining calcined clay (CC) and limestone (LS), enabling clinker replacement levels of up to 60% through the synergistic pozzolanic reaction between alumina and carbonates (Antoni et al., 2012). In addition to its technical performance, LC^3 aligns with circular economy principles by lowering CO_2 emissions and encouraging the utilization of abundant and low-cost raw materials. As the construction industry increasingly prioritizes sustainability, research has turned toward optimizing low-carbon binder systems through tailored mix design and a deeper understanding of hydration and reaction mechanisms (Snellings, 2016). Research by Hawkins et al., (2003) has shown that incorporating up to 5% limestone in ordinary Portland cement (OPC) does not compromise strength and improves energy efficiency by enhancing the grinding process. Moreover, when combined with calcined clay, limestone enhances the performance of ternary blends by participating in additional reactions (Dhandapani et al., 2021).

In the Libyan context, the industrial research centre has identified large deposits of kaolinite-rich clay in the southern region, which can be thermally activated at 700–850 °C to produce reactive metakaolin (Akasha, 2015). These local resources present a strategic opportunity to develop sustainable cementitious systems within the region. The performance of LC^3 is influenced by several interrelated factors, including the purity and mineralogical composition of raw materials, the calcination temperature, curing conditions, and overall mix design. Key parameters such as the grinding sequence, gypsum content, water-to-binder ratio, CC-to-LS ratio, and the clinker replacement level have been shown to significantly affect both mechanical strength and workability (Balasubramanian & Sarangapani, 2023). Previous studies (Lin et al., 2021, Yu et al., 2021, Balasubramanian and Sarangapani, 2023) have highlighted the sensitivity of compressive strength to these variables. Lin et al. (2021) reported that a mix composed 85% OPC, 10% calcined clay, and 5% limestone yielded the highest compressive strength, with further increases in substitution leading to a decline in performance. Likewise, Yu et al. (2021) showed that LC^3 blends with 50-70% clinker content complied with BS EN 197-1 strength standards. Balasubramanian and Sarangapani (2023) further supported these findings, noting that clinker replacement levels could vary between 30 and 70%, contingent on the quality and purity of the supplementary materials used. Kafodya et al., (2023) evaluated LC3 cement made with local Malawian materials at two clinker levels: 50% (LC3-50) and 40% (LC3-40). Using varying calcined clay-to-limestone ratios and water-to-cement ratios, results showed that LC3-50 achieved higher compressive strength (up to 39.2 MPa) at 28 days. Increased clay content reduced workability and strength due to higher water demand. LC3-50 also showed lower porosity and improved microstructure, confirming its potential as a sustainable cement alternative. Chaipanich et al., (2024) examined the replacement of 50% of OPC with varying proportions of limestone (30 - 45%) and calcined clay (5 - 20%). Their findings showed that the optimal blend (30% LS, 20% CC) achieved a compressive strength of 36 MPa at 28 days 225% higher than the blend containing 50% limestone only. The improved performance was attributed to the enhanced pozzolanic reactivity and improved pore structure associated with higher CC content. Although the strength remained below that of 100%

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OPC, the blend demonstrated promising performance for use as a low - carbon, medium - strength alternative binder. Nararueang et al., (2024) explored the performance of various Thai calcined clays as partial replacements for OPC across six different mixtures containing fly ash (FA), MK, and regional CCs. Despite identical clinker replacement levels across the blends, those incorporating calcined clays outperformed fly ash-based mixes in terms of both compressive strength and ultrasonic pulse velocity (UPV). These improvements were attributed to the higher pozzolanic activity of calcined clays, which resulted in lower porosity and better mechanical performance. Ferrari et al., (2023) investigated how increasing CC content affects mortar workability through flow table tests performed on both mortars and pastes at water-to-binder (w/b) ratios of 0.5 and 0.4, using CEM-I and CEM-II cements. Their findings confirmed that higher CC contents significantly reduce workability due to rapid water absorption by CC, which reduces the amount of free water available for paste fluidity. This behavior necessitated greater use of superplasticizers to maintain adequate workability. Complementary to this, Takhi et al., (2024) examined the fresh properties of LC^3 systems with 50 and 70% clinker replacement, employing different CC:LS ratios (2:1 and 1:1). Compared to OPC, these mixes exhibited increased water demand and prolonged setting times with higher CC content. The reduced flowability was once again linked to the water-retaining nature of metakaolin, which imposes a need for higher superplasticizer dosages to achieve workability comparable to OPC. Shao et al., (2025) further reported that although LC^3 tends to have slightly lower compressive strength than OPC at early ages, it typically matches or surpasses OPC by 7 days, with continued strength development through 28 days. This trend consistent across multiple studies demonstrates that LC^3 can achieve comparable long-term mechanical performance with substantially lower clinker content. Furthermore, LC^3 concrete has exhibited superior flexural strength and enhanced chloride-binding capacity, broadening its applicability in durable construction. Despite these advances, research is still limited on the effects of high clinker replacement levels (≥50%) on the performance of LC^3. In particular, no previous studies have investigated high clinker replacement LC^3 produced with kaolinite-rich clays from Libya.

This experimental study examines the properties of LC^3 (limestone-calcined clay) cement mortars featuring high clinker replacement levels, incorporating locally sourced kaolinite-rich clays from southern Libya. Mortar mixtures were formulated with clinker contents of 40, 50, and 60%. The performance of the LC^3 blends was evaluated through compressive strength tests, flexural strength tests, ultrasonic pulse velocity (UPV) measurements, porosity analysis, and workability assessments.

2.EXPERIMENTAL WORK

2.1.Materials:

Portland cement CEM-I 42.5 N, sourced from the Benghazi Cement Factory, was used in this study. This cement meets the European standard (EN 197-1, 2011). The limestone was also procured from the same factory, where it was dried, ground, and sieved to pass through a 75 µm sieve. Gypsum, provided by the cement factory, was milled and sieved to achieve a particle size of less than 75 µm. The chemical and physical characteristics of the cement, limestone, and gypsum are summarized in Tables 1, while Figure 1 illustrates the limestone and gypsum before and after grinding.

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Table 1. Properties of cement, limestone, and gypsum.

Chemical Composition (%)	Cement	Limestone	Gypsum
CaO	62.62	52.00	51.92
SiO_2	20.65	1.65	1.74
Al_2O_3	5.17	0.34	1.20
Fe_2O_3	2.77	0.04	0.14
MgO	3.33	2.16	1.06
Na_2O	-	-	3.95
K_2O	-	0.09	0.13
Cl	-	0.05	-
SO_3	2.61	0.58	39.28
LOI	1.56	44.97	32.81
Surface area (Blaine) m^2/kg	360	-	-
Finesses (%)	3.23	-	-



Figure 1. Finely milled limestone and gypsum.

Standard sand, sourced from the Benghazi Cement Factory, met the specifications outlined in (ASTM778, 2009) was used to prepare the mortar specimens. Kaolinite-rich clays were sourced from 10 km north of Sebha in southern Libya, comprising about 95% kaolinite. This clay is classified as type A (Akasha, 2015). The raw clay was crushed into fragments ranging from 0.5 to 1.25 cm and then calcined at 800 °C for two hours in a controlled furnace (type 30400, see Figure 2). After calcination, the materials were cooled in an airtight desiccator to maintain their reactivity. The resulting calcined clays were processed using a Los Angeles abrasion machine, then a conventional mill, and finally sieved to ensure they passed through a 75 µm sieve, as shown in Figure 3. The chemical composition of the calcined clays is detailed in Table 2.



Figure 2. Type 30400 furnace used.



Figure 3. Finely milled calcined clay

Table 2. The chemical analysis of calcined clay (by XRF).

Chemical composition (%)	Sebha clay type (A)
SiO_2 56.14	
Al_2O_3	32.74
Fe_2O_3	2.41
CaO	6.86
CI	0.16
Na_2O	0.6
K_2O	-

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Chemical composition (%)	Sebha clay type (A)
SO_3	-
TiO_2	-
MnO	0.01
P_2O_5	0.07

2.2.Mixture Details:

A total of four mortar mixtures (Table 3) were designed to evaluate the influence of combined calcined clay (CC) and limestone (LS), at a fixed ratio of 2:1, on the mechanical performance of blended cement mortars. To mitigate the potential impact of sulfate interaction, the gypsum (Gyp) content was maintained at 6% in all mixtures. This approach aligns with established practices for maintaining the balance between setting time and strength development in cementitious systems, as sulfate levels can significantly influence hydration kinetics and ettringite formation (Taylor, 1997). Three LC^3-based mixes were prepared with clinker contents of 60, 50, and 40%. Each of these mixes contained 6% gypsum, with the remaining fraction composed of calcined clay (CC) and limestone (LS) at the 2:1 ratio. Additionally, control mixture was included using CEM I 42.5 with no substitution of calcined clay or limestone. The detailed composition of all mixtures is presented in Table 3, and the obtained LC^3 blends cement mixtures are given in Figure 4.

Table 3. System compositions.

Mixture ID	Clinker(%)	CC(%)	LS(%)	Gypsum(%)	Total(%)	Clinker Reduction vs CEM	CC:LL Ratio
CEM	94.00	-	-	6	100	-	-
LC^3-60	60.00	22.67	11.33	6	100	36.17	2:1
LC^3-50	50.00	29.33	14.67	6	100	46.81	2:1
LC^3-40	40.00	36.00	18.00	6	100	57.45	2:1

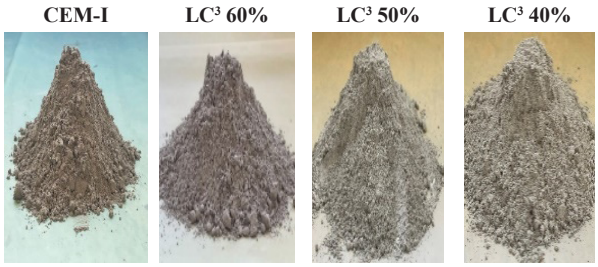


Figure 4. LC^3 blends cement mixtures.

2.3.Specimens Preparation:

Mortar mixtures were prepared for 50 mm cubes (following ASTM C109/C109M-02, 2020) and 40 × 40 × 160 mm prisms (per ASTM C348-21, 2021) to assess compressive and flexural strength, respectively. In all mixtures, a water-to-binder ratio of 0.485 and a binder-to-sand ratio of 1:2.75 (by weight) were adopted. The control mixture (CEM) was prepared with a water-to-cement ratio (w/c) of 0.485, in accordance with ASTM C109, to achieve a target mortar flow of 110 ± 5% on the flow table. For all other mixtures, the water-to-binder ratio (w/b) was consistently maintained at 0.485 as a controlled variable. As a result, the measured flow values differed based on the clinker replacement level in each formulation. Notably, no superplasticizer (SP) was needed to achieve the desired workability. Mixing was conducted with an automatic mortar mixer in compliance with (ASTM C305/C305-20, 2009). The mortar specimens were compacted, kept in molds for 24 hours, demolded, and then cured in water until testing.

2.4.Test Methods:

The hardened densities of cement mortars were measured in compliance with ASTM C188. Workability was assessed using the flow table test (ASTM C1437, 2007).

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Compressive strength was determined at different curing ages (3, 7, 28, 56 days) using 50 mm mortar cubes, following ASTM C109. Flexural strength was evaluated after 28 days using 40 × 40 × 160 mm mortar prisms as per ASTM C348-21.

Porosity was evaluated at 3, 7, and 28 days using 50 mm mortar cubes following the vacuum saturation procedure described in RILEM (1994). The open porosity of the samples was determined in accordance with RILEM Recommendation CPC 11.3, which quantifies the volume of accessible pores through vacuum saturation and subsequent mass measurements of the specimens in dry, saturated, and immersed conditions. Prior to testing, mortar samples were oven-dried at 105 ± 5 °C until a constant mass was achieved, then cooled to room temperature in a desiccator. Vacuum saturation was carried out as per CPC 11.3: a vacuum pressure of ≤ 2 kPa was maintained for 3 hours to evacuate air from the open pores. Deionized water was then gradually introduced under vacuum for an additional 1 hour to ensure full infiltration. All procedures were performed at 20 ± 2 °C to minimize temperature-related changes in water viscosity and specimen stability. After vacuum release, specimens remained fully submerged in water for 24 hours to achieve complete saturation. Three masses were subsequently recorded: the dry mass (Md) after oven drying, the saturated-surface-dry mass (Ms) after vacuum saturation and gentle blotting, and the immersed mass (Mi) while suspended in water. The accessible porosity (P) was then calculated using equation (2.4.1) below:

$$P = \left[\frac{Ms - Md}{Ms - Mi}\right] x100 \qquad (2.4.1)$$

Additionally, the ultrasonic pulse velocity (UPV) was measured on 50 mm mortar cubes at different curing ages following the procedure outlined in ASTM C597-09 (2010). The test was conducted to assess the quality, uniformity, and internal condition of the mortar specimens. A pulse velocity apparatus equipped with 54 kHz transducers was employed for all measurements. To ensure efficient signal transmission, a thin film of gel was applied between the transducers and the opposite faces of each specimen. Prior to testing, the mortar cubes were conditioned to a saturated-surface-dry (SSD) state. This state was achieved by immersing the cubes in water for 24 hours, followed by gentle wiping of any surface moisture immediately before the test. All UPV measurements were carried out at a controlled laboratory temperature of 20 ± 2 °C. The pulse velocity (V) was calculated using the equation (2.4.2) below:

$$V = \frac{L}{t} \qquad (2.4.2)$$

where L represents the travel path length (Km) and t denotes the measured travel time (s).

The reported values of compressive strength, flexural strength, and porosity represent the averages of 3, 2, and 1 test specimens per age, respectively.

3.RESULTS AND DISCUSSION

3.1.Density of Hardened Mortar:

Figure 5 shows a comparison of density among different types of LC^3 blends cement mortars. CEM has the highest density, while LC^3-40 has the lowest. The densities of LC^3-60 and LC^3-50 fall between these two, with LC^3-60 being denser than LC^3-50. This suggests that as the LC^3 blend ratio changes, the density of the mortar decreases.

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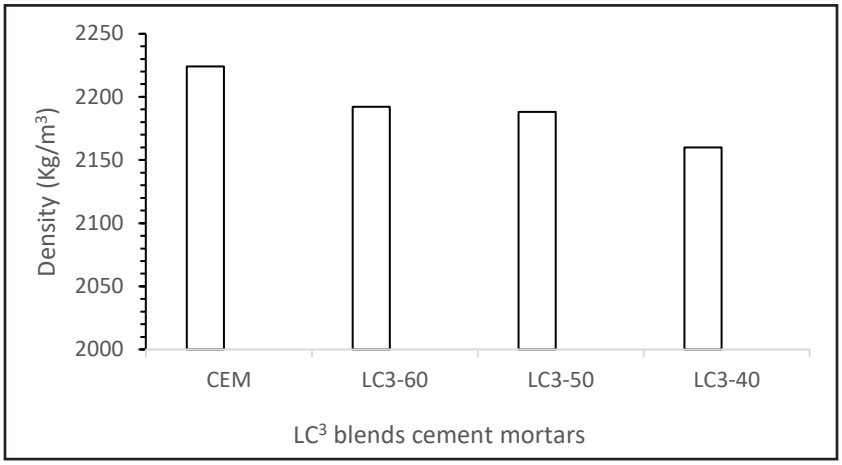


Figure 5. Hardened density of LC³ blends cement mortars.

3.2.Workability:

The results of mortar's flow are shown in Figure 6. It can be seen from the graph that workability declins with rising replacement levels, highlighting a trade-off between sustainability and fresh-state performance, consistent with findings by (Balasubramanian & Sarangapani, 2024).

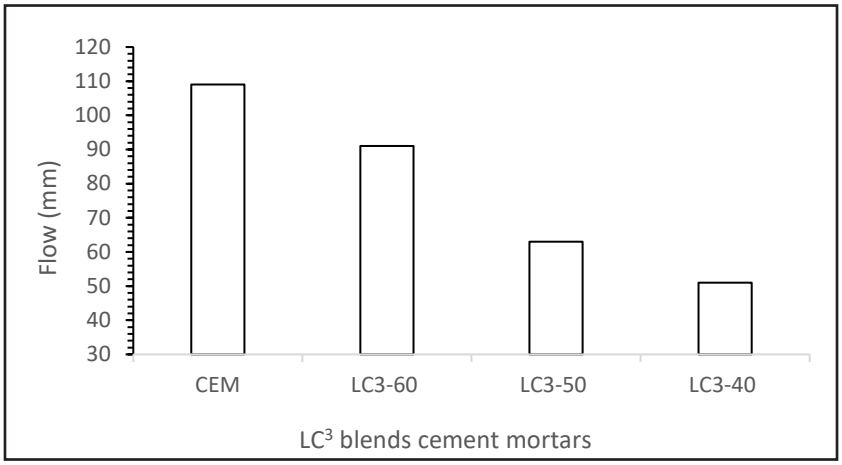


Figure 6. Flow of LC³ blends cement mortars.

3.3.Compressive Strength:

The results of compressive strength of CEM I and LC³ blends cement mortars are shown in Figure 7. All mortars gain strength with age, showing the expected trend of increasing compressive strength as hydration progresses. LC3-60 consistently demonstrates the highest compressive strength at all ages, indicating that this blend maintains considerable strength even with partial clinker substitution. LC3-50 also shows enhanced strength compared to CEM, though it is slightly lower than that of LC3-60. In contrast, LC3-40 has reduced strength, likely because the increased clinker replacement diminishes both early and long-term strength. The dissolution of reactive silica and alumina from metakaolin in hydrated cement systems provides additional components that interact with portlandite (Shi et al., 2015). When the released silica is promoted, calcium silicate hydrate (C-S-H), the main binding phase in cement paste, is cre-

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ated. According to Richardson (2000), the modified form created here frequently has a lower Ca/Si ratio than regular C-S-H, which is linked to greater durability and less leaching susceptibility. When alumina is added to C-S-H, its nanostructure is further altered, producing a denser microstructure that enhances performance over the long run (Scrivener et al., 2015). Additionally, in the presence of carbonates, the alumina derived from kaolinite/metakaolin helps to generate AFm phases, particularly monocarboaluminate and hemicarboaluminate (Matschei et al., 2007). These carboaluminate compounds are stabilized by the combination of the aluminates and calcium carbonate, which is obtained from limestone filler (Lothenbach et al., 2008). In addition to consuming portlandite, which lowers the possibility of efflorescence and alkali leaching, this procedure improves the cement matrix's structural stability and refines the pore network (Hooton et al., 2007).

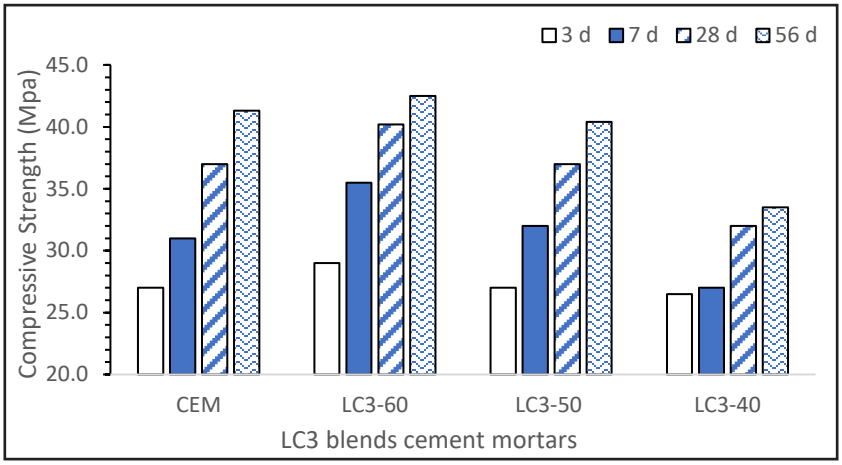


Figure 7. Compressive strengths of LC³ blends cement mortars at different ages (days).

Strength Activity Index (SAI)

The Strength Activity Index (SAI) offers a reliable indirect measure of pozzolanic performance. As per ASTM C618, an SAI of ≥75% is required for a material to be considered pozzolani. To assess the effect of calcined clay - limestone blends (2:1 ratio) on reactivity, SAI was measured at 7 and 28 days for LC³-based mixtures, as shown in Figure 8. It can be seen from the graph that all mixes exceeded the ASTM threshold. LC³-60, LC³-50 and LC³-40 blends mortars show enhancement in early-age performance with SAI values of 113.3%, 103.2%, and 87.1%, respectively. LC³-60 blend shows the highest SAI at both curing times, indicating it is the most effective in enhancing strength among the samples tested. While, LC³-50 has a moderate SAI, suggesting it provides a good balance of strength but is less effective than LC³-60. However, LC³-40 blend has the lowest SAI, indicating it may not be as effective in improving strength compared to the others.

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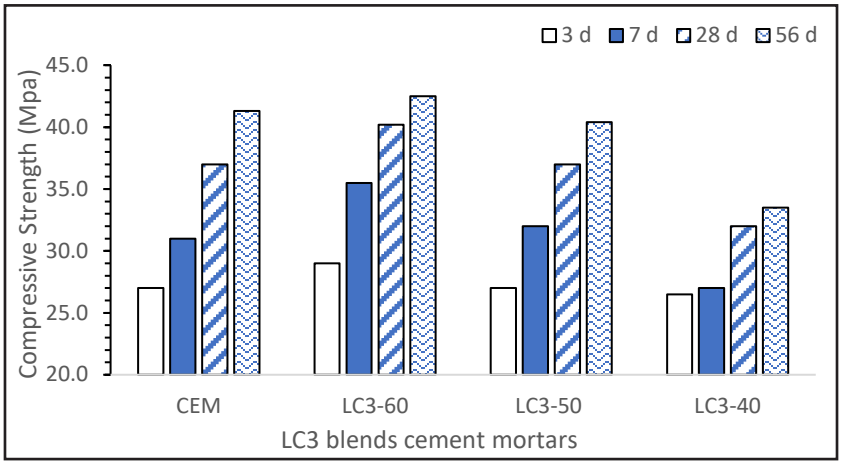


Figure 8. SAI for LC³ blends cement mortars at 7 and 28 days.

3.4.Flexural Strength:

Flexural strength results for CEM I and LC³ blends cement mortars with varying clinker replacement levels at 28 days are given in Figure 9. The Figure shows that LC³-60 blend exhibits the highest flexural strength (about 7.8 MPa), significantly outperforming the conventional CEM blend (about 5.9 MPa). LC³-50 also shows improved strength (about 7 MPa) over CEM, though slightly lower than LC³-60, indicating a potential trade-off between material proportions and performance. However, LC³-40 blend has the lowest strength of about 5.2 Mpa among the LC³ blends, even slightly below CEM.

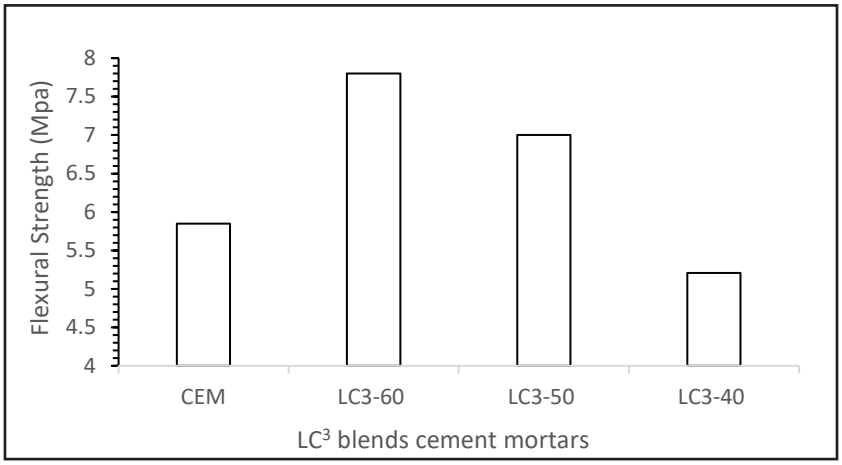


Figure 9. Flexural strengths of LC³ blends cement mortars at 28 days.

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3.5.Porosity:

As shown in Figure 10, LC^3 blends cement mortars (LC^3-60 and LC^3-50) exhibits significant reduction in porosity at all ages, with 28-day values of 9.6%, and 10.57%, respectively, lower than CEM I (11.7%). This improvement may be attributed to the high surface area of fine calcined clay particles, which enhances pozzolanic activity and promotes C-S-H formation, leading to a denser microstructure. These findings align with (Zunino & Scrivener, 2021) and (Zunino et al., 2022), who reported that the metakaolin-limestone synergy improves packing and promotes carboaluminate formation, reducing pore connectivity. However, LC^3-40 blend has the highest porosity across all curing periods, suggesting it has a lower density compared to the LC^3 blends, as Figure 5 illustrates.

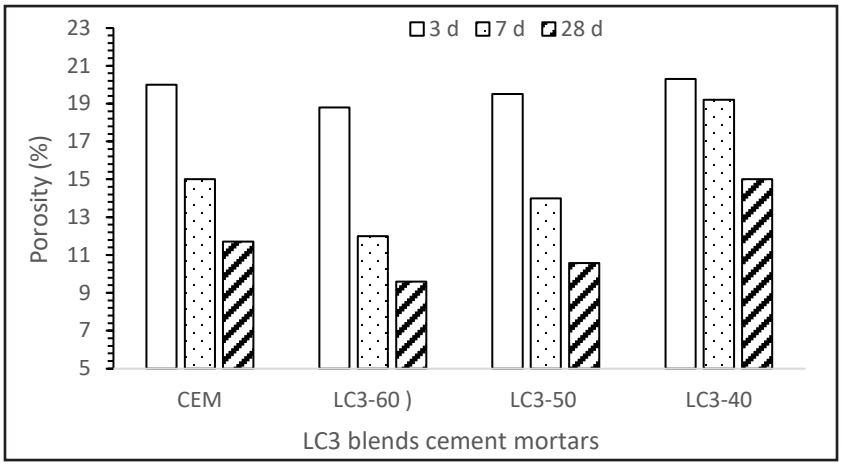


Figure 10. Porosity of LC^3 blends cement mortars at different ages.

3.6.Ultrasonic Pulse Velocity (UPV)

Figure 11 compares the ultrasonic pulse velocity (UPV) results of different cement mortar blends (CEM, LC^3-60, LC^3-50, and LC^3-40) over curing periods of 3, 7, and 28 days. As expected, UPV generally increases with curing time, indicating improved material properties as the cement hydrates and gains strength. CEM (presumably the control blend) serves as a baseline for comparing the performance of the LC^3 blends. The LC^3 blends (LC^3-60, LC^3-50, LC^3-40) show varying UPV values, which could suggest differences in their performance and hydration characteristics. Across all ages, LC^3-60 demonstrates the highest UPV values, peaking at 4.75 km/s at 28 days, signifying superior hardening and densification. While LC^3-50 shows increasing UPV, its values remain comparable to or slightly below CEM's at each stage, indicating moderate strength gain. In contrast, LC^3-40 consistently exhibits the lowest UPV, suggesting either reduced density or slower reaction kinetics relative to the other mixtures.

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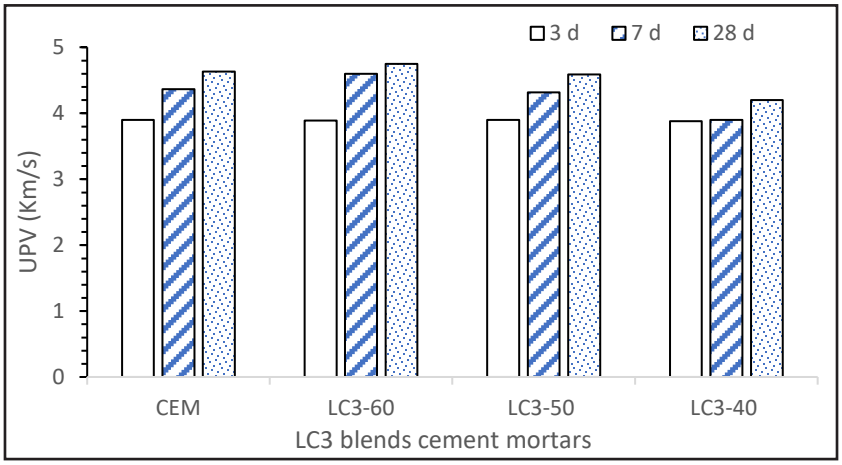


Figure 11. UPV of LC³ blends cement mortars at different ages.

5.CONCLUSIONS

Within this test program, LC³ mortars prepared with locally calcined kaolinite-rich clay and limestone achieved comparable 28-day compressive and flexural strengths to the OPC control at 50% clinker replacement, while exhibiting lower flow that will require admixture/water optimization. Mixtures at this replacement level also showed reduced accessible porosity, indicating microstructural densification. These findings suggest that 50% clinker replacement is a promising target for Southern-Libyan LC³ mortars. Further work should quantify durability (chloride ingress, carbonation, sulfate), report statistical confidence, and assess sustainability benefits with a simple CO_2/energy model.

6.ACKNOWLEDGMENT

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