

OPTIMISING THE PERFORMANCE OF CARBIDE LIME WASTE MORTAR: THE ROLE OF TEMPERATURE IN ACCELERATED CO₂ CURING

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Abstract

This study examined the effects of elevated temperatures higher than ambient temperature on the physical, mechanical, and CO₂ (carbon dioxide) capturing performance of Carbide Lime Waste (CLW) mortars with 20% cement replacement at early curing ages (1, 3 and 7 days). The CLW mortars cured under accelerated CO₂ curing (CLW_{20acc}) were compared with the control samples under ambient curing (CLW₂₀). The temperatures were set at 30°C, 40°C and 60°C with fixed 20% CO₂ concentration. Key assessments include density, compressive and flexural strengths, carbonation depth, and microstructural analyses using Thermogravimetric analysis (TGA). Results revealed that mortar densities decreased with increased temperature, but higher curing temperatures significantly enhanced the performance of CLW_{20acc} mortars over control samples. At 60°C, carbonation depth reached 8.50%, 36.18%, and 86.25% after 1, 3, and 7 days, respectively, while compressive strength increased by 51.5% at day 7 compared to day 1. TGA results demonstrated progressive CaCO₃ (calcium carbonate) precipitation from 26.6% on day 1 to 58.5% on day 7, indicating calcium silicate and portlandite conversion into carbonates. XRD study showed calcite as the main phase and vaterite to aragonite as curing temperature and age arose. Ambient curing reduced mechanical strength and carbonation performance in all control samples. These findings demonstrate that elevated temperatures expedite carbonation and enhance the mechanical properties of CLW mortars, validating the potential of accelerated CO₂ curing as a sustainable strategy for optimising lime-based materials. This approach offers a dual benefit: reducing CO₂ emissions while leveraging elevated CO₂ levels to improve construction material performance.

Keywords: accelerated CO₂ curing, accelerated temperature, carbide lime waste, compressive strength, carbonation thickness

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1. INTRODUCTION

Cement production has steadily increased from 0.94 billion tonnes in 1970 to 4.1 billion tonnes in 2018, with a projected 45% rise by 2050 [1]. This growth comes at a significant environmental cost. Current production rate is already contributing 5–7% of global carbon dioxide (CO₂) emissions, with CO₂ representing 65% of all greenhouse gases [1]. To support a more sustainable environment, it is essential to adopt alternative methods that minimise cement consumption while still achieving or maintaining the required strength [2].

In response to these challenges, researchers have increasingly explored methods for capturing and storing carbon dioxide to mitigate emissions [3]. Among these, accelerated CO₂ curing through carbonation offers a dual benefit: improving the strength and durability of mortar while simultaneously sequestering CO₂. This method promotes the rapid reaction of calcium silicates in cement-based materials, enabling faster strength gains in a shorter time frame [5, 23]. Exposing mortar to high levels of CO₂ in a specialized curing environment not only accelerates carbonation, but also supports the hardening process. Similar to how natural processes like rock weathering absorb CO₂ over time, accelerated curing takes advantage of CO₂-enriched environments to speed up these reactions artificially. This approach mirrors natural carbonation processes, but under controlled conditions that optimize the formation of calcium carbonate (CaCO₃) within cementitious matrices. By leveraging such strategies, it becomes possible to turn the challenge of elevated atmospheric CO₂ into an opportunity for sustainable material innovation.

Various industrial by-products and waste materials, such as slag, fly ash and recycled concrete aggregate (RCA) have also been studied for their effectiveness in speeding up CO₂ curing, offering potential solutions to environmental and economic challenges [6]. Besides, some studies have examined the mineral carbonation of construction and demolition waste including bricks, concrete, and tiles which consist of silicates rich in calcium and magnesium [7]. However, several factors, including CO₂ concentration, temperature, pressure, moisture content and relative humidity affect the carbonation rate of concrete under CO₂ curing [6, 7, 8]. For instance, carbonation and hydration product formation, along with microchemical property changes, are significantly affected when the CO₂ concentration differs from as low as 0.04% until 20% [11]. Research indicates that when CO₂ concentration is lower than 4%, microstructural changes are minimal, and the material primarily forms a calcium-silicate-hydrate (C-S-H) gel with a low Ca/Si ratio [12]. The degree of carbonation also depends on the CaO content in the material [10]. Due to the low atmospheric CO₂ concentration (0.04%), natural carbonation of cement-based materials occurs at slow rate in the natural environment [9,10].

Temperature is another critical factor influencing the carbonation rate. Higher temperatures enhance the CO₂ reaction rate and transmission coefficient, resulting in increased carbonation depth and degree in concrete specimens [8]. Temperature affects four key aspects - reactant solubility, ion or gas movement, change in moisture condition, and promotion in cement hydration [13]. A temperature increase of 10°C can boost carbonation rates by 1.1-1.7 times within the 10-60°C range [14]. Research generally highlighted the significant role that temperature plays in CO₂ curing [13, 14]. It is able to alter the diffusion rate of CO₂, affecting the ability of calcium ions to dissolve in concrete. High temperature may also decrease the solubility of CO₂ and calcium ions while enhancing the diffusivity of CO₂ across the carbonated layers [16]. Besides, the permeability of the material and the concentration of CO₂ in the atmosphere also influence the degree of carbonation [17].

Among the waste materials studied for accelerated CO₂ curing, Carbide Lime Waste (CLW) stands out as a promising candidate due to its high CaO content [18]. Thus, the aim of this study is to investigate the effects of varying accelerated CO₂ curing temperatures on the mechanical properties, including compressive and flexural strengths, of CLW-based mortars. Furthermore, microstructural

analyses using thermogravimetric analysis (TGA) and X-ray Diffraction (XRD) were employed to evaluate and explain the CO₂ capture performance of CLW mortars. This helps to bridge the current knowledge gap, but most importantly, it addresses the dual challenge of mitigating CO₂ emissions while ensuring the resilience of cement-based products to the impacts of rising global temperatures. In conventional concrete, elevated temperatures often result in cracking and material deterioration [25]. Therefore, the study has been designed to be aligned with the United Nations' 13th Sustainable Development Goal (SDG), which targets combating climate change, and to offer practical solutions to promote climate resilience.

2. MATERIALS AND METHODS

2.1. Materials

The materials were Ordinary Portland Cement (OPC), fine aggregates, and Carbide Lime Waste (CLW). The density of OPC is 3150 kg/m³, fine aggregate is 1576 kg/m³ and tap water is 1000 kg/m³. These were used to produce all mortar specimens. CLW exhibits a moisture content of approximately 13%. The CLW was milled to a particle size of 45µm to ensure uniformity in particle size throughout the process [26]. Table 1 shows the chemical composition of OPC and CLW that acted as a binder.

Table 1. Chemical composition of materials

Component	CaO (%)	SiO ₂ (%)	Al ₂ O ₃ (%)	Fe ₂ O ₃ (%)	MgO (%)	TiO ₂ (%)	SO ₃ (%)
OPC	60.0	14.6	3.3	2.2	0.6	0.2	2.6
CLW	96.0	2.0	0.5	0.3	0.4	0.03	0.02

2.2. Sample preparation

The substitution of 20% CLW as an alternative material to replace cement was formulated using weight method. Two types of mortar mixtures were prepared: CLW₂₀ and CLW_{20acc}. The former has 20% cement replacement cured under ambient conditions and the latter has the same amount of cement replacement cured under accelerated CO₂ curing. Both mixtures used a cement-to-sand ratio of 1:3 and a water-to-cement ratio of 0.6. The samples preparation was accordance to ASTM C305 [27]. Table 2 provides the mix design proportions of the mortars as compared to conventional mortars with no cement replacement. The workability of fresh CLW₂₀ mortar has been evaluated as previously stated in previous studies [26].

This study involved two curing regimes: ambient curing (CLW₂₀) and accelerated CO₂ curing (CLW_{20acc}). Both curing methods were tested at early curing durations of 1, 3, and 7 days using the same mix design with 20% cement replacement, as shown in Table 3. For the conventional curing method, the demoulded samples were immersed in water. For the accelerated CO₂ curing regime, the samples were placed in a CO₂ chamber with temperature set to 30°C, 40°C and 60°C as well as a fixed CO₂ concentration of 20%. All mortars were cured according to specified durations to evaluate the influence of temperature on both the mechanical strength and CO₂ capturing performance of the CLW-based mortars.

Table 2. Mix design proportions

Percentage of CLW (%)	Quantity (kg/m ³)			
	CLW	Cement	Sand	Water
0	-	550	1650	330
20	110	440	1650	330

Table 3. Curing regimes properties

Mix Notation	OPC (%)	CLW (%)	w/c	Curing method
CLW ₂₀	80	20	0.6	Conventional (ambient curing)
CLW _{20acc}				Accelerated CO ₂ curing (CO ₂ chamber)

Notation:

CLW₂₀; CLW mortar with 20% of cement replacement under ambient curing

CLW_{20acc}; CLW mortar with 20% of cement replacement under accelerated CO₂ curing

2.3. Sample tests

The compressive strength tests were conducted on the cubic mortar samples of 50x50x50 mm by using a Universal Testing Machine (UTM) at a loading rate of 2 kN/mm as to determine the maximum compressive load of the mixture sample can resist before fracture accordance to specification [28]. The density test was obtained in accordance to standard requirement [30]. The flexural strength tests were conducted on the prism samples of 40x40x160 mm at a loading rate of 0.046 kN/s, as to determine the maximum bending capacity of the mixture samples accordance to specification [19].

The carbonation thickness of the mortar was determined using the phenolphthalein spray technique on prism samples. Each prism sample was split into two halves, and the exposed surfaces were carefully cleaned with a brush to remove dust and debris. A 1% phenolphthalein alcohol solution was sprayed on the split surfaces to identify the carbonation thickness, which was then calculated by averaging measurements taken at 10 places across the sample's cross-sections [9].

All strength and carbonation test were conducted at curing intervals of 1, 3 and 7 days. A total of 56 cubes (compressive strength properties) and 56 prisms (carbonation thickness evaluation) were tested. For each test, data from three samples were averaged to ensure accuracy and reliability of the results. For the microstructural examination, paste samples cured for extended curing durations were evaluated using Thermogravimetric Analysis (TGA) and X-ray Diffraction (XRD) tests. The paste sample was immersed in an acetone solution to stop hydration, then oven-dried at 40°C for 24 hours. TGA-DTG was performed with approximately 15 mg of the powder sample at a heating rate of 10 °C/min, ranging from 20 °C to 1000 °C, under a nitrogen gas flow of 20 ml/min. XRD measurement was performed using a Cu α X-ray source at 40 Kv and 40 mA. The 2 θ scans were performed between 5°-80° with a step size of 0.02° and a pace of 2 °/min. Both tests were used to measure the amount of carbonated products and crystalline phases in the optimum temperature based on the highest carbonation depth, respectively. The methodologies followed were consistent with those established in previous studies [17].

3. RESULTS AND DISCUSSION

3.1 Density

Figure 1 shows a comparison of mortar density using conventional curing (CLW₂₀) compared to accelerated CO₂ curing (CLW_{20acc}). Without an improvised curing regime, the density remained constant throughout the seven curing days with an average density of 2000kg/m³. However, when subjected to accelerated CO₂ curing and elevated temperatures, the mortar's density had significantly reduced. At higher temperatures, some hydrated chemicals and particularly calcium silicate hydrates (C-S-H) would have become dehydrated. This reduces the amount of moisture, leading to increased voids and porosity. This phenomenon facilitates the penetration of CO₂ into the mortar's pores, enhancing its CO₂ capture capacity. This is further discussed in subsection 3.3.2.

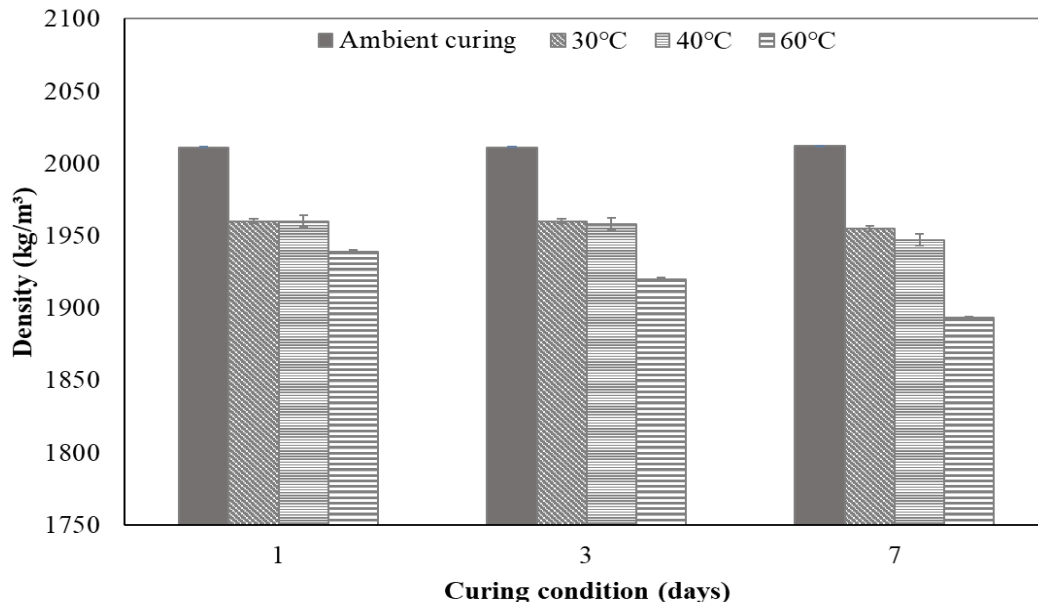


Fig. 1. Density of CLW₂₀ (ambient curing) and CLW_{20acc} mortars (accelerated CO₂ curing)

3.2 Mechanical strength properties

Figure 2(a) and 2(b) show the compressive and flexural strengths of the mortar samples tested after 1, 3 and 7 days of curing. In general, both figures showed that temperature and CO₂ carbonation age strongly influence mechanical strength properties. Under ambient temperature, the mortars exhibited the lowest compression and flexural strength at all times. It is apparent that elevated temperatures during accelerated CO₂ curing clearly resulted in significant strength gains within the first 24 hours, compared to ambient curing condition. This enhancement was similar to the flexural strength of the mortar samples. The highest flexural strength (9.56 MPa) was recorded at 60°C after seven days of accelerated CO₂ curing.

With accelerated CO₂ curing and at elevated temperatures, both strengths have increased, albeit at different rates. The highest increase in compressive strength was observed for mortars cured at 60°C, where it increased by 111.3% or 29.64 MPa after 7 days of curing compared to CLW_{20acc} mortars. Moreover, it can be observed that the rate of compressive strength enhancement at 30°C and 40°C of curing temperature were 54.0% and 97.15%, respectively. Even since day 1, the mortar strengths at 60°C

have been greatly enhanced by 8.3% to 59.4% under accelerated CO₂ curing, regardless of temperature. This trend continued for mortars cured for 3 and 7 days, which is a strong and positive outcome from accelerated CO₂ curing by elevated temperature to enhance the compressive strength of mortars. Previous studies have also reported that high curing temperatures enhance compressive strength, making it comparable to that of OPC mortar in the early stages [32]. The accelerated CO₂ curing of a 20% CLW mortar sample at 60°C initially exhibited optimal compressive strength; however, a strength reduction was observed on day 3, which might be due to the CaCO₃ precipitation, which hindered further CO₂ diffusion. On day 7, it was noted that the sample exhibited the highest overall performance. In contrast, the 40°C condition demonstrated more uniform improvement, albeit it attained lower peak strength. 30°C condition generated the minimal reaction rate and the most pronounced reduction on days 3 and 7, resulting in the lowest compressive strength. The findings suggest that a curing temperature of 60°C is ideal for enhancing strength.

An increase in compressive strength has been attributed by active carbonation reaction at elevated temperatures that favours the production of more calcium carbonate (CaCO₃) from the interaction between calcium hydroxide (Ca(OH)₂) in CLW material and carbon dioxide (CO₂). CaCO₃ production enhances compressive strength in lime mortar. When CLW mortars carbonation increases, more CaCO₃ crystals are produced, which refine the pores, fill the loose walls, and stimulate the matrix to become denser, enhancing the mechanical properties of CLW mortars [19]. The mechanical strength also progressively improved with increasing carbonation age, indicating that the carbonation process is time-dependent. As the duration of CO₂ exposure increases, the carbonation reaction becomes more extensive, resulting in enhanced mechanical strengths.

These results indicate that elevated temperatures significantly enhance the compressive strength. Higher curing temperatures corresponded to greater strength gains in CLW mortars subjected to accelerated CO₂ curing. As expected, longer curing durations improved the strength of all mortars, including the control samples. The strength enhancement of CLW mortars under accelerated CO₂ curing meets the standard specification for load-bearing applications according to ASTM C270, where the average compressive strength at 28 days for Type M mortar is 12.5 MPa [31]. Interestingly, based on the results, even a minimal curing temperature of 30°C could meet the average 28-day strength specified in the standard. Thus, these findings indicate that elevated temperatures not only improve early-age strength, but also reduce overall construction time. Curing regimes can be optimised in real-world applications to align with the specific demands of the construction industry.

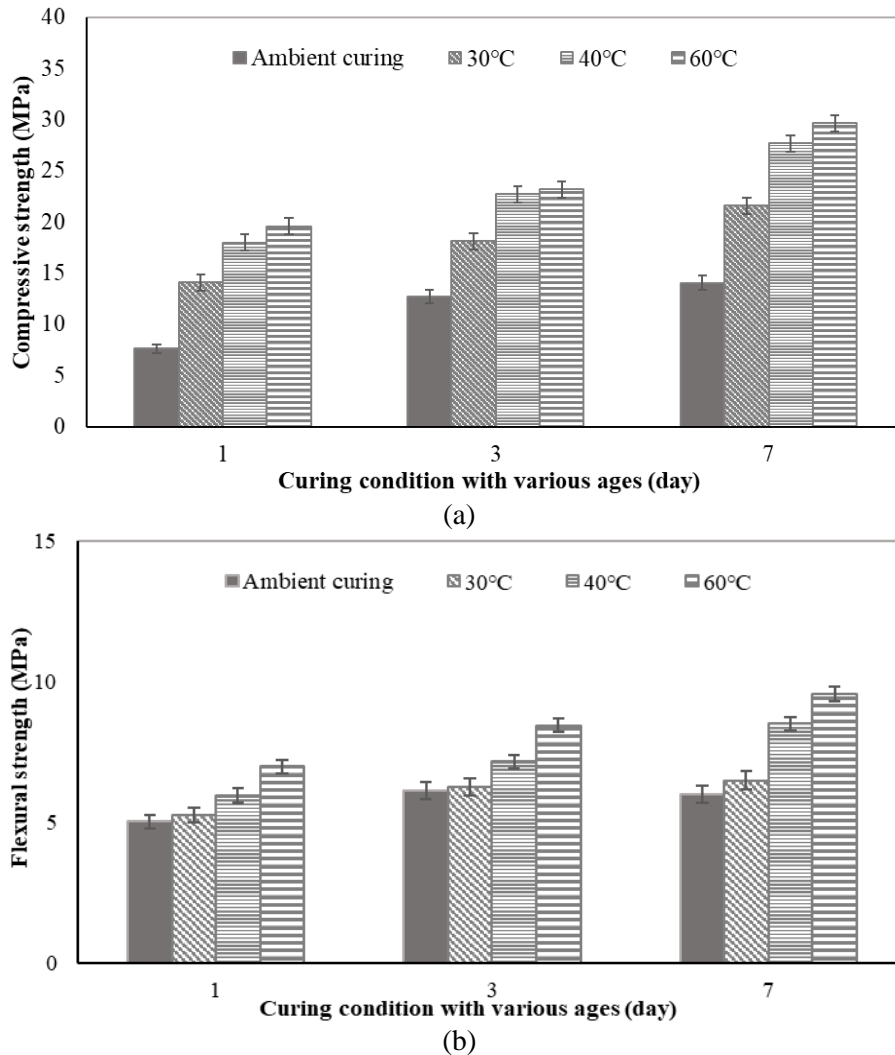


Fig. 2. Mechanical strength properties of CLW₂₀ (ambient curing) and CLW_{20acc} mortars (30°C, 40°C and 60°C): a) compressive strength and b) flexural strength

3.3 CO₂ capturing properties

The CO₂ capturing properties was evaluated based on two tests, carbonation depth by phenolphthalein spray and microstructural examination under TGA-DTG.

3.3.1 Carbonation depth

The results of carbonation depth are shown in Table 4. Consistent with the trends observed in mechanical strengths, the carbonation depth was significantly affected by temperature differences. Exposure to elevated temperatures under CO₂ curing markedly enhance carbonation, attributed to controlled temperature setting that promoted continuous carbonation at higher rate.


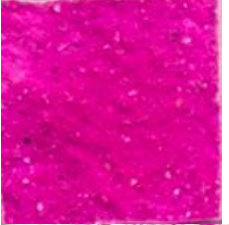
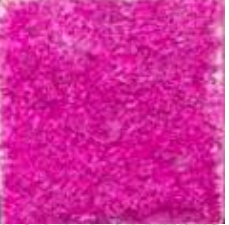


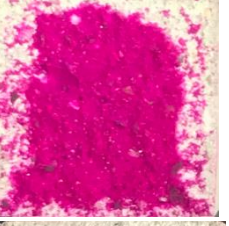






Figure 3 shows the percentages of carbonation depth for the CLW mortars subjected to accelerated CO₂ curing. At 30°C, the carbonation depth steadily increased as the carbonation age increased. After 7 days of exposure to CO₂, the carbonation depth reached about 7 mm. This

comparatively slower carbonation rate has been attributed to the reduced kinetic energy of the reacting substances and the slower diffusion of CO_2 into the mortar matrix.

In contrast, higher curing temperature at 60°C resulted in a more pronounced rise in carbonation depth, reaching 18.5 mm after seven days. The accelerated temperature significantly enhanced the carbonation reaction by improving the transmission coefficient and reaction rate of CO_2 , leading to greater carbonation degrees and depths in the samples [12, 13]. Notably, even within 24 hours of curing, the higher temperature resulted in substantial improvements in carbonation depth compared to 30°C and 40°C , with increases of approximately 50 % and 67 %, respectively. Ambient conditions were excluded from detailed analysis since no obvious changes were observed on the mortar surface.

These findings suggest that higher curing temperatures significantly accelerate the transformation of clinker phases and portlandite into CaCO_3 , highlighting the importance of temperature in optimizing the carbonation process.

Table 4. Carbonation depth of 20% of CLW mortars under natural ambient and subjected temperature

Curing regimes	Day 1	Day 3	Day 7
Natural ambient			
30°C			
40°C			
60°C			

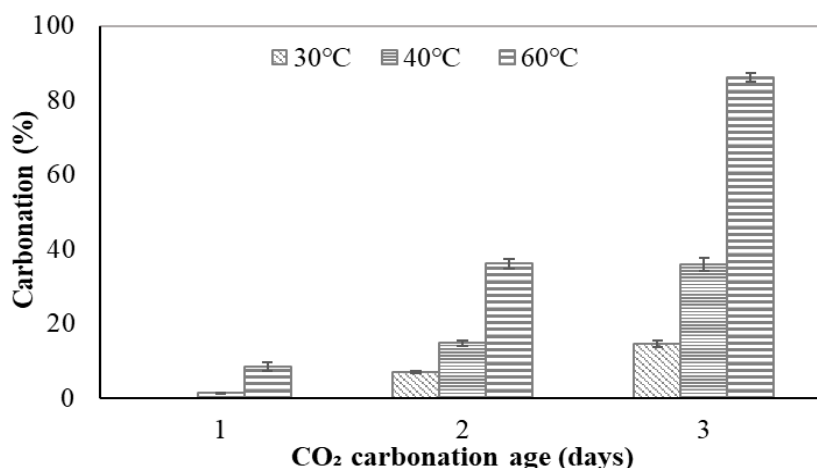


Fig. 3. CO₂ capturing performance of CLW_{20acc} mortars

3.3.2 Thermogravimetric analysis (TGA-DTG)

Figure 4(a) and 4(b) show the TGA-DTG profiles of the carbonated products for CLW mortar with 20% cement replacement. Two curing conditions were defined. First was ambient curing, where the uncarbonated sample was designated as UC-CLW₂₀. The second was accelerated CO₂ curing at 60°C for one, three and seven days.

The mass loss observed between 100°C and 200°C corresponds to the evaporation of free water within the mortar mix. The hydration phase of the UC-CLW₂₀ sample was less stable compared to that of the CLW_{20acc} sample. A more pronounced peak was observed in the CLW_{20acc} mortar at day 1, which diminished as curing progress to day 7. This reduction in mass loss reflects the transformation of C-S-H into CaCO₃ during the carbonation process.

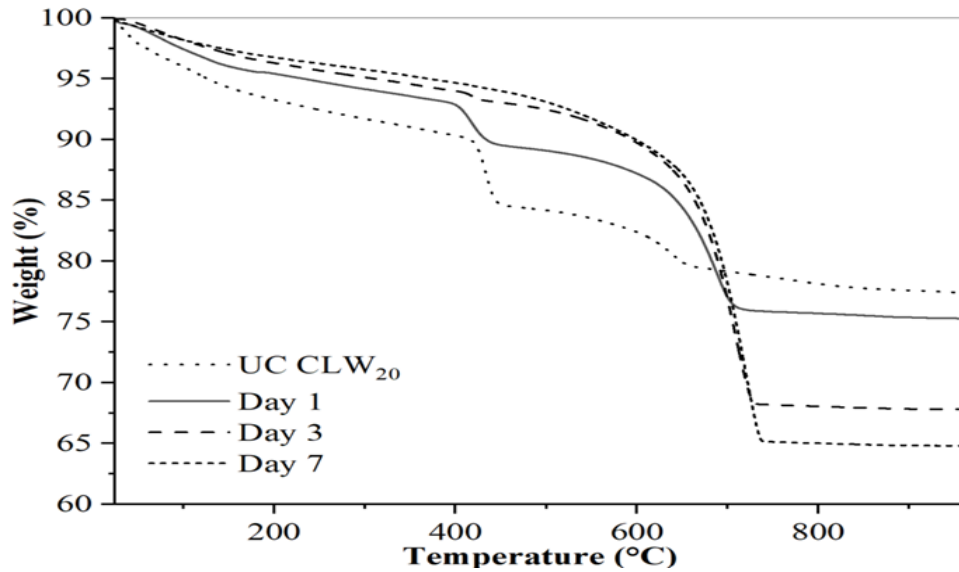
The carbonation process primarily involves the reaction of calcium silicates and portlandite. For portlandite, the mass loss due to decomposition occurred between 400°C and 500°C. The Ca(OH)₂ content increased proportionally with the replacement of CLW in the mortar. However, the mass loss associated with the portlandite phase decreased as the curing age extended, from 12% on day 1 to 2% on day 3, and further to 0.2% on day 7. This indicated progressive consumption of Ca(OH)₂ during carbonation.

Mass loss observed between 600°C and 800°C was attributed to the CaCO₃ precipitation in the mortar; which was reflected in the peak increase from day 1 until day 7. At day 1, the precipitation was 26.6%, this increased to 52% on day 3 and finally 58.5% on day 7 when the mortar was cured at 60°C.

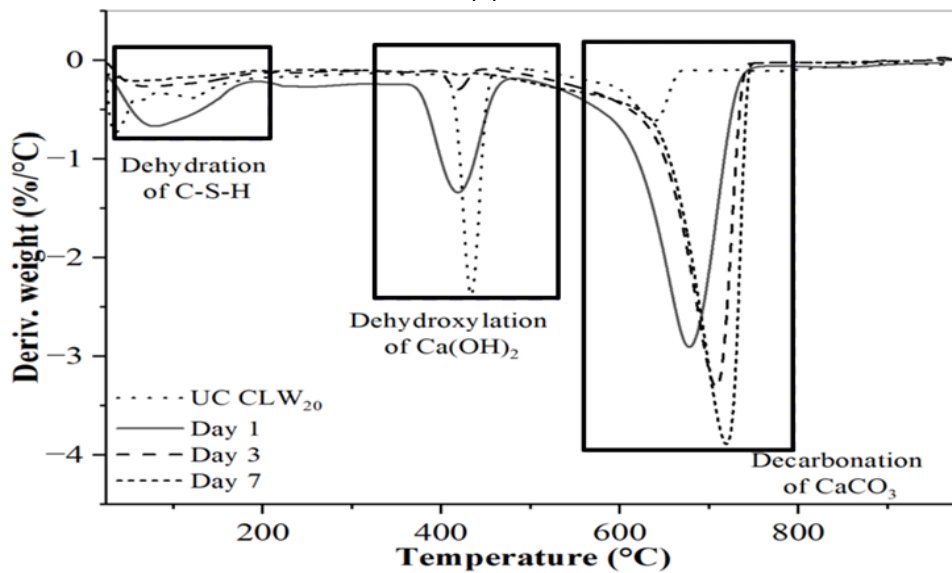
Previous research suggests that CaCO₃ exists in two distinct forms: a poor crystalline CaCO₃ structure, decomposing between 500°C and 650°C, and a well-defined crystalline CaCO₃ structure, decomposing above 650°C [16]. Figure 4(c) details the major losses within the CaCO₃ decomposition range. For the CLW mortar under ambient curing, the mass loss predominantly occurred in the poor crystalline region. However, the exposure to elevated temperatures promoted the crystallization of CaCO₃, as indicated by a shift in the decomposition peak to higher temperatures. This finding demonstrates that increased curing temperatures not only accelerate carbonation and improve mechanical properties, but also enhance the structural development of more well-defined CaCO₃ crystallisation.

Figure 4(d) shows the percentages of the carbonated products formed at curing ages of 1, 3 and 7 days. These results indicate that extended curing under elevated temperatures in a CO₂-rich environment

significantly promoted the formation and crystallization of CaCO_3 , supporting the efficacy of accelerated CO_2 curing at higher temperatures.



(a)



(b)

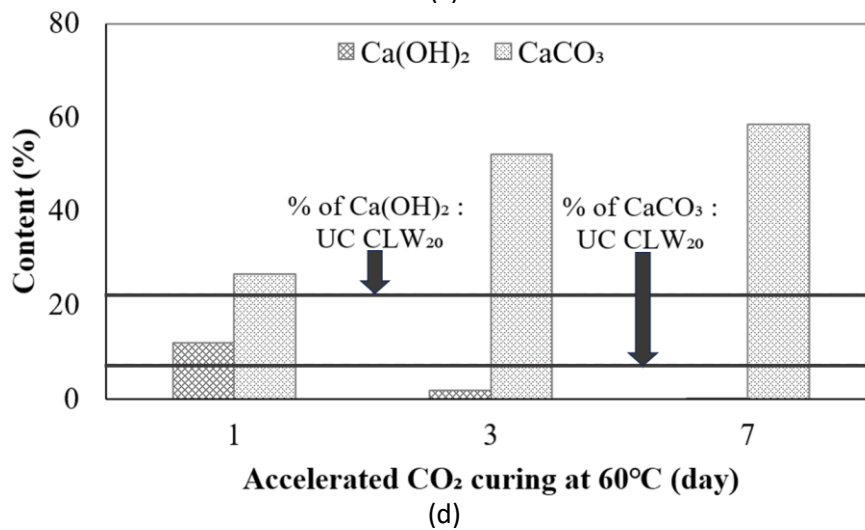
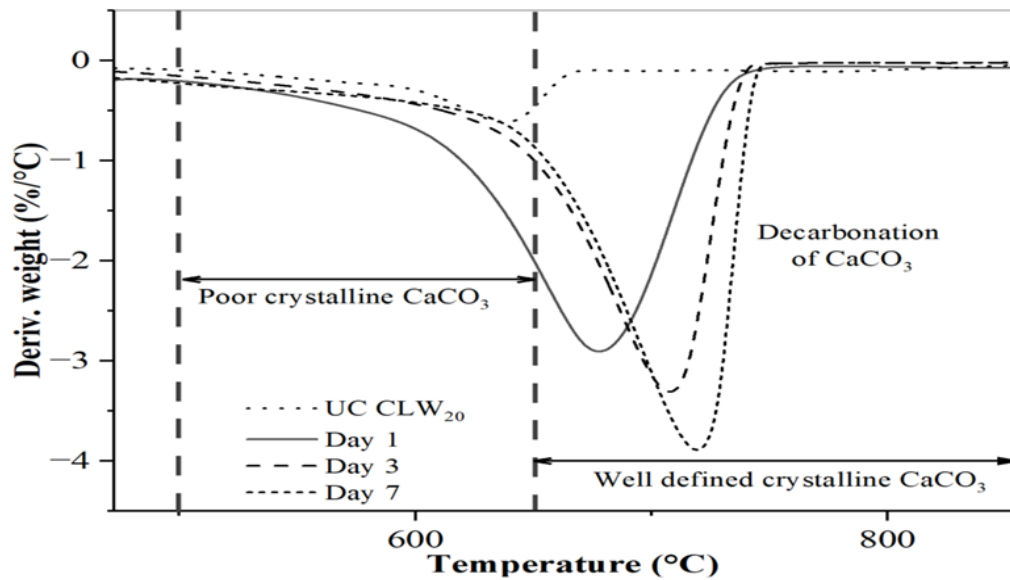


Fig. 4. Carbonated products of CLW_{20acc}: a) TG, b) DTG, c) content of carbonated products

3.3 Crystalline Identification – X-Ray Diffraction (XRD) Analysis

The crystalline phases in the CLW_{20acc} paste cured at 60°C under varying curing ages were identified using X-ray diffraction (XRD), as shown in Figure 5. The analysis reveals the presence of three CaCO₃ polymorphs - vaterite, calcite, and aragonite - with calcite being the predominant carbonate phase in all samples. The results demonstrate that increasing the curing temperature significantly enhances the intensity peak of calcite (CaCO₃), as confirmed by the quantitative XRD analysis.

Additionally, the experimental data indicate that the samples were not completely carbonated, aligning with the carbonation depth test results. This incomplete carbonation is evidenced by the presence of residual calcium silicate phases in the XRD patterns. However, as the curing age progresses, these calcium silicate peaks diminished, confirming their gradual transformation into calcite. Vaterite, a less stable phase, tends to transform into calcite at lower temperatures and into aragonite at high

temperatures [24]. This is reflected in the XRD results, where the highest peak for aragonite was observed at day 7, indicating that the conversion of vaterite to aragonite increases with extended curing duration.

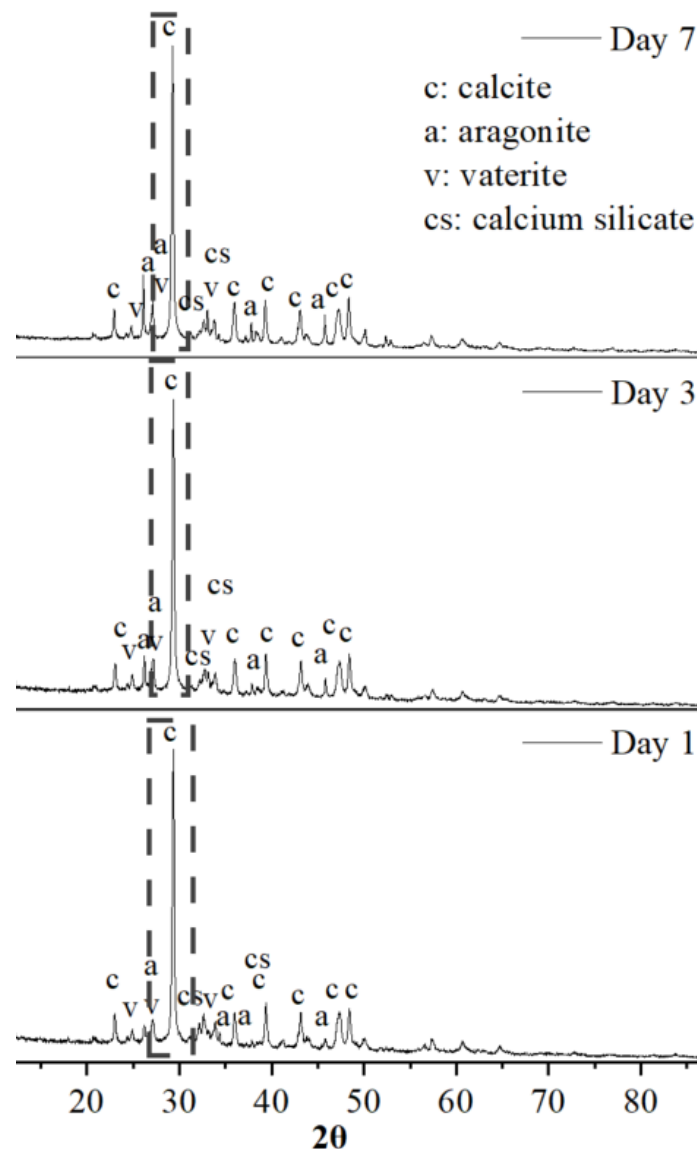


Fig. 5. XRD of CLW_{20acc} under 60°C with different curing ages

4. CONCLUSIONS

The study demonstrates that the mechanical performance and microstructural development of mortars containing Carbide Lime Waste (CLW) can be improved by accelerated CO₂ curing at high temperatures. The faster carbonation rate aids in achieving early-age strength appropriate for load-

bearing applications. TGA and XRD data show that CaCO₃ forms more prominently at higher temperatures, indicating better crystallinity and a stronger microstructure. The findings of this study lead to several conclusions and recommendation:

- i. **Density of Mortars:** The density of the CLW₂₀ mortar remained consistent across all curing ages, averaging 2000 kg/m³. For CLW_{20acc}, the density was lower than 2000 kg/m³, indicating that elevated temperatures contribute to density reduction. In practical applications, this weight reduction can enhance productivity by easing material handling and transportation, as well as reducing the overall dead load on structures, depending on the application.
- ii. **Carbonation Depth and Temperature:** The carbonation depth rate at 60°C was significantly higher compared to 30°C, highlighting the crucial role of temperature in accelerating the carbonation process in CLW mortars. Both temperature and carbonation age has a synergistic effect on the compressive strength development of the CLW_{20acc} samples.
- iii. **Mechanical Strength Development:** Higher temperatures and prolonged carbonation ages favour the carbonation process, leading to improved mechanical strength properties of the mortars. Even at day 1, the compressive strength of CLW_{20acc} mortars meets the requirements for load-bearing applications when cured at a minimum temperature of 30°C.
- iv. **Microstructural and Chemical Changes:** TGA results confirmed that elevated temperature enhanced CaCO₃, resulting in a well-defined crystallisation structure as curing age increased. XRD analysis further supported these findings.

Accelerated CO₂ curing at high temperatures requires specialised heating equipment, which may elevate costs and potentially overcome the environmental advantages attributed to decreased cement demand. In Malaysia, where this technology is still relatively new, it is essential to conduct pilot projects, life-cycle assessments, and economic viability studies to enhance industry acceptance and support the transfer from laboratory research to full-scale construction applications. Overall, the results underscore the importance of considering temperature and carbonation age in designing and optimising cementitious materials incorporating lime. Future research should explore the integration of pozzolanic materials with lime-based minerals under accelerated CO₂ curing conditions to further reduce cement usage in the construction industry.

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DISCLOSURE STATEMENT

The authors declare no competing interests.

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