# Effectiveness of Carbide Lime Waste as CO<sub>2</sub> Capturing Materials of Mortar at Early CO<sub>2</sub> Curing Age

Asyrofuddin Fadhlullah<sup>1,a</sup>, Berlianada Rizki Priandita<sup>1,b</sup> Adrina Rosseira A. Talip<sup>1,2,c</sup>, Nur Hafizah A. Khalid<sup>1,d,\*</sup>

<sup>1</sup>Faculty of Civil Engineering, Universiti Teknologi Malaysia 81310 Johor Bahru, Johor, Malaysia. <sup>2</sup>College of Engineering, School of Civil Engineering, Universiti Teknologi Mara, 40450 Shah Alam, Selangor, Malaysia.

<sup>a</sup>asyrofadhlullah@gmail.com , <sup>b</sup>rpberlianada@graduate.utm.my, <sup>c</sup>adrina9587@uitm.edu.my, <sup>d</sup>nur\_hafizah@utm.my\*

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Abstract. This study explored the use of Carbide Lime Waste (CLW) in mortar production and its exposure to controlled  $CO_2$  curing for 24 hours. The results indicate that CLW mortar under  $CO_2$  curing has improved compressive strength and early  $CO_2$  capturing performance due to the reaction between Ca (OH)<sub>2</sub> in CLW and  $CO_2$  gas. With a cement replacement rate of 20, a 60% solid carbonates precipitation was recorded after just 7 days of curing. In general, the longer the curing durations, the higher the  $CO_2$  capturing capability. Further investigations into the long-term mechanical and durability properties are recommended to assess its feasibility for practical applications. Re-utilization of waste materials like CLW can make significant strides towards more eco-friendly and sustainable practices.

# Introduction

Lime-based products are conventional cementitious material that have gained popularity in the construction industry. The lime cycle, illustrated in Figure 1, serves as the fundamental process behind these products. The cycle starts with calcination, where limestone ( $CaCO_3$ ) is heated to an extremely high temperature in kilns, producing reactive quicklime. While some quicklime is used directly, the majority is dissolved in a violent hydration reaction, generating significant heat. Carbon dioxide ( $CO_2$ ) reacts with calcium hydroxide ( $Ca(OH)_2$ ) to form calcium carbonate ( $CaCO_3$ ) and water. In recent studies on mortar and concrete curing, researchers have introduced CO<sub>2</sub> as a carbonation curing method, aiming to replicate the chemical reaction observed in the lime cycle to produce carbonate precipitation [1, 2]. The carbonation capability of Ca(OH)<sub>2</sub> allows for its use as the main binder in lime-sand mortars and plasters [3]. Despite concerns about greenhouse gas emissions and environmental pollution associated with CO<sub>2</sub>, curing under the CO<sub>2</sub> method offers the potential to convert CO<sub>2</sub>, a major greenhouse gas, into a mineral that become trapped in the mortar or concrete for the long term. This CO<sub>2</sub> curing method not only reduces carbon footprint, but also enhances compressive strength, effectively managing the environmental issues in the construction industry [4]. The carbonation curing technique presents a promising practical solution for cementbased products to effectively reduce CO<sub>2</sub> emissions, particularly those produced by industries.



Fig. 1. The lime cycle

One of the potential lime waste materials that can be utilized is Carbide Lime Waste (CLW), a high-quality hydrated lime by-product of acetylene gas production. Previous studies have identified CLW as rich in calcium hydroxide (Ca(OH)<sub>2</sub>) through microstructure examinations. Chemical analysis using X-ray Fluorescence (XRF) spectrometry shows that the chemical composition of Calcium Oxide (CaO) in CLW is approximately similar to hydrated lime (HL) [5]. X-ray Diffraction (XRD) analysis further confirms that both lime types exhibit Ca(OH)<sub>2</sub> behavior. Typically, Ca(OH)<sub>2</sub> is added to improve the plasticity and water retention of cement mortar and gypsum plasters. Given that CLW demonstrates no significant difference in flexural strength compared to conventional lime or HL, it has been considered a suitable cement replacement [6]. CLW shows potential for use in building applications, either as the main binder or as an addition to plasters and mortars, although some formulation adjustments are certainly required due to differences in physical characteristics between the waste and industrially-produced lime [5].

The CO<sub>2</sub> curing process, similar to carbonation curing, involves introducing CO<sub>2</sub> gas into a controlled curing chamber, diffusing the CO<sub>2</sub> into fresh concrete under low pressure, and transforming gaseous CO<sub>2</sub> into solid calcium carbonates (CaCO<sub>3</sub>). The CO<sub>2</sub> curing setup can be adjusted for hardened concrete, mortar, or raw aggregates, depending on their application. In the context of mortar, early carbonation from CO<sub>2</sub> curing does not negatively impact the long-term development of samples. This early age carbonation occurs concurrently with the early hydration of the cement, resulting from deliberate exposure of fresh mortar to CO<sub>2</sub> [2]. When cement is mixed with water and exposed to CO<sub>2</sub> gas, the cement compounds C<sub>3</sub>S and C<sub>2</sub>S are instantly carbonized into CaCO<sub>3</sub> and silica gel. Curing carbonation is highly exothermic, which accelerates the sample's hardening rate compared to steam curing at 75°C. Utilizing the carbonation process from CO<sub>2</sub> curing, Ca(OH)<sub>2</sub> is transformed into CaCO<sub>3</sub>, leading to the deposition of calcite crystal that fill the pores, reducing porosity and enhancing mechanical properties, such as hardening and strength [3,7].

This study explored the use of Carbide Lime Waste (CLW) as a high-potential material in the mortar mixture.  $CO_2$  was utilized as a curing agent for mortar at 24 hours of  $CO_2$  curing age, and its effectiveness was compared to air curing at the early stage. The study also evaluated the effectiveness of CLW as a  $CO_2$  material for mortar, considering compressive strength, rapid carbonation assessment using phenolphthalein indication, and  $CO_2$  capturing capabilities.

# Methodology

**Material and Sample Preparations**. Carbide Lim Waste (CLW) was utilized as a cement substitute, first oven-dried at 105°C for 24 hours. Subsequently, 500 grams of the dried CLW was weighed and placed in a ball mill with steel balls for a four-hour grinding process. The ground CLW was then stored in a plastic container with silica gel to prevent hydration. CLW was directly substituted for Ordinary Portland Cement (OPC) at replacement levels ranging from 20% to 60%. The water-to-binder ratio was fixed at 0.5, and the mortar mixture consisted of 1 part cement and 3 parts sand.

Air Curing. Mortar samples were produced following ASTM C311 guidelines, and air curing served as the control method for comparison with CO<sub>2</sub> curing. After 24 hours of hardening, the

mortars were demoulded and subjected to the designed curing periods of 1, 3, 6, 10, 13, 16, 20, and 24 hours in an open environment at a typical outdoor temperature of 30°C.

**CO<sub>2</sub> Curing**. After the 24-hour hardening period, the samples were demoulded and subjected to  $CO_2$  curing. The  $CO_2$  concentration in the chamber was set to 20% at specific temperatures [8], with a maximum temperature of 60°C. Each sample was placed in a temperature-controlled chamber for curing durations of 1, 3, 6, 10, 13, 16, 20, and 24 hours. Regular inspection of the  $CO_2$  tube was conducted to prevent leaks or blockages, and the  $CO_2$  chamber was operated within a controlled room temperature environment.

**Compression Test**. Compressive strength of the mortar samples was determined according to ASTM C109 using a universal test machine at a rate of 2 N/mm<sup>2</sup>/s, determined based on the sample's physical dimensions. The samples were tested at ages of 1, 3, 6, 10, 13, 16, 20, and 24 hours. Mortar cubes were removed from the CO<sub>2</sub> curing chamber after the specified curing times.

**Carbonation Assessment by Phenolphthalein Indication**. Cured mortar prisms underwent a three-point load test as per ASTM C140. This samples were divided into half, and one half was subjected to a rapid carbonation assessment using phenolphthalein as an indicator. After conducting the flexural strength test at 0.045 N/s for a 40 x 40 x 160 mm sample, the freshly fractured surfaces were sprayed with a 1% phenolphthalein-ethanol solution. After 60 minutes, the carbonation level was determined by measuring the distances between the colour change boundary and sample margin on each side of the sample using a digital calliper. The depth of carbonation for each sample was represented by the mean of the measured values.

**Carbonate Precipitation by Thermogravimetric Analysis**. The mortar sample with the highest strength and CO<sub>2</sub> capturing in the initial stage was selected for further analysis. The sample was reproduced and cured for 1, 3, and 7 days. Carbon sequestration was measured using the thermogravimetric analysis (TGA), which permits the identification of various hydrates and carbonates. Differential thermal analysis (DTA) measurement was utilized in this study [9] to detect heat flow associated with reaction temperatures. The cement paste sample was immersed in acetone prior to the test to prevent additional carbonation effects and cease interaction with hydrate phases. After drying in a furnace at 100°C for 24 hours, miniature samples were hand-crushed and further processed using a mortar and pestle to obtain a powder form. The sample material was heated from 35°C to 1000°C at a heating rate of 10°C/min and a nitrogen flow rate of 80 mL/min. For each measurement, 35 mg of sample material was loaded into an Al<sub>2</sub>O<sub>3</sub> crucible and inserted in the TGA system. This ensured the actual temperature measurement of the sample was independent from the ambient temperature.

#### **Results and Discussion**

**Compressive Strength.** The compressive strength of the mortar samples increased with curing age, regardless of the curing method. Notably, the graph for  $CO_2$  curing demonstrated significantly higher compressive strength values starting at 13 hours of curing time compared to air curing at 20% replacement rate, as shown in Figure 2 and Figure 3. The  $CO_2$  curing procedure led to a substantial improvement in mortar strength when compared to air curing. This enhancement is attributed to the nanosized CaCO<sub>3</sub> filling gaps within the samples [4]. However, it should be noted that mortar compositions with CLW concentrations exceeding 20% exhibited lower compressive strength compared to the control. Increasing the proportion of CLW in the mortar resulted in reduced compressive strength of the mortar [10].



Fig. 2. Compressive strength development under air curing

**Fig. 3.** Compressive strength development under CO2 curing

**Carbonation Depth.** The air curing graph for air curing, shown in Figure 4, depicted relatively low carbonation depths. In contrast,  $CO_2$ -cured mortar exhibited a consistent increase in carbonation depth, with higher values achieved after 24 hours of curing, as shown in Figure 5. Notably, the CLW mortars with a 20% replacement rate demonstrated significantly higher carbonation depth than the control mortar starting at 13 hours of curing time. This indicates that the 20% CLW mortar mix proportion is the most effective in capturing  $CO_2$ .



3.0 Control 20% 30% 2.47 Carbonation Depth (mm) 60% 40% 50% 2.38 1.63 1.03 0.5 0.0 0.0 24 10 16 20 1 6 13 Curing Ages (Hours)

Fig. 4. Carbonation depth development by air curing

**Fig. 5.** Carbonation depth development by CO2 curing

**Carbonate Precipitation**. Figure 6, 7 and 8 depicts the observations from thermogravimetric analysis (TGA) at 1, 3, and 7 days of CO<sub>2</sub> curing age, respectively. In general, the trends revealed the presence of carbonate (CaCO<sub>3</sub>) precipitation in the temperature range of 500 to 900°C, coinciding with the greatest mass loss of cement paste. CO<sub>2</sub> curing resulted in the formation of CaCO<sub>3</sub> on the mortar surface layer, reducing the sample's porosity [11]. The CaCO<sub>3</sub> content increased significantly with increasing curing ages, as depicted in Figure 9. This indicates that CO<sub>2</sub> not only reacted with Ca(OH)<sub>2</sub>, but also with the cement component such as C<sub>2</sub>S and C<sub>3</sub>S during the early stages of carbonation [11].



**Fig.6.** Typical TGA-DTA curves of cement with 20% CLW at 1 day curing age



**Fig. 7.** Typical TGA-DTA curves of cement with 20% CLW at 1 day curing age



**Fig. 8.** Typical TGA-DTA curves of cement with 20% CLW at 7 days curing age

**Fig. 9.** The effect of CO<sub>2</sub> curing at 1, 3 and 7 days curing age at content of CaCO<sub>3</sub> on the surface layers of cement paste

Table 1 summarizes the TGA results, which demonstrated that increase in the curing age has led to a considerable increase in the amount of CaCO<sub>3</sub> that precipitated in the CLW mortar. Accelerated CO<sub>2</sub> carbonation significantly sped up the carbonation process. For instance, after just 24 hours of CO<sub>2</sub> curing, approximately 26.60% of CaCO<sub>3</sub> was precipitated. Subsequently, the CaCO<sub>3</sub> content gradually increased to approximately 52.40% and 59.80%, respectively, after 3 and 7 days of curing. In other words, the longer the CO<sub>2</sub> curing period, the more CO<sub>2</sub> can be captured within the cement paste, leading to increased CaCO<sub>3</sub> precipitation. However, as the curing age increases, the effective penetration of CO<sub>2</sub> from the surrounding to deeper layers of the samples decreases due to the formation of CaCO<sub>3</sub> and the reduction of pore structure [11].

Table 1. Ca(OH)<sub>2</sub> and CaCO<sub>3</sub> precipitation measured by TGA at different curing ages

Day (s)	CO <sub>2</sub> concentration (%)	Temperature (°C)	Ca(OH) <sub>2</sub> (%)	CaCO <sub>3</sub> (%)
1			11.69	26.60
3	20	60	23.05	52.40
7			26.31	59.80

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# Conclusions

The findings demonstrated the substantial potential of Carbide Lime Waste (CLW) as a waste material for  $CO_2$  capture in mortar applications. Several important conclusions can be drawn from the findings:

1. The incorporation of 20% CLW to replace cement in mortar resulted in the highest compressive strength and superior  $CO_2$  capturing capability compared to other mix proportion after 24 hours of  $CO_2$  curing. This suggests that the 20% CLW mix shows promising performance for both structural strength and environmental impact reduction.

2. As the curing age increased up to 7 days under CO<sub>2</sub> curing, a progressive increase in CaCO<sub>3</sub> precipitation in the mortar was observed. The evidence from microstructural examination using Thermogravimetric Analysis (TGA) supported this finding, providing insights into the enhanced carbonation process in the CLW-based mortar.

Further investigations into the long-term mechanical and durability properties of CLW-based mortars are recommended to assess its feasibility for practical applications. Additionally, considering the impact of different CLW concentrations and curing ages on various mortar properties would provide valuable insights for optimizing the use of CLW as a CO2 capturing material in the construction industry. By promoting the utilization of waste materials like CLW, the construction sector can make significant strides towards more eco-friendly and sustainable practices, contributing to global efforts to mitigate climate change and enhance the industry's overall environmental footprint.

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