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Early CO₂ capturing mortar by incorporating a new potential carbide lime waste under controlled CO₂ curing

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Abstract. Early-age cement paste or mortar can be enhanced by curing it in a CO₂ chamber. The study was initiated with investigation on the optimum compressive strength by replacing cement to carbide lime with 0, 5, 10, 15 and 20% of Carbide Lime Waste (CLW) content. The desired CLW mortar at 28 normal curing age was selected and the similar mix proportion was again casted and the fresh CLW mortar was cured for 1,3 and 7 days under controlled CO₂ curing. The CO₂ concentration and temperature were fixed at 20% and 60% respectively. The compressive strength and depth of carbonation of CLW mortar was investigated. In addition, their microstructural properties under TGA test was analysed at the early curing ages by using cement pastes. It was found that the mortar containing 5% of CLW had the desired optimum compressive strength and UPV under water curing condition. Overall, the strength properties of control mortar and CLW mortar significantly enhanced at early strength gained by controlled CO₂ curing as compared to the water curing technique. The used of 5% of CLW as the potential to substitute the cement portion has provide better understanding on CO₂ sequestration in cement mortar. The microstructural result shows the CaCO₃ precipitation in the CLW mortar was higher than the control mortar as CLW has Ca(OH)₂ behaviour which effectively react with CO₂ and formed stable CaCO₃ precipitation in the mortar.

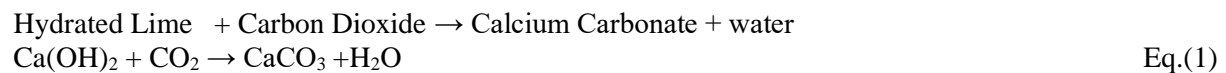
Keywords : Carbide Lime Waste, Early Strength, Controlled CO₂ Curing, Higher Temperature Curing, CaCO₃ precipitation

1. Introduction

Limestone has long been used as a primary raw material in the production of acetylene gas. However, as a byproduct of gas generation, carbide lime is formed. Acetylene gas is one of the main products that produce an industrial waste known as Carbide Lime (CL) or Ca(OH)₂. Calcium carbide waste, carbide lime or lime sludge is a by-product of acetylene production through the hydrolysis of calcium carbide. It is usually being disposed by land filling where it gives an environmental impact such as the issue on the leaching of harmful compound and alkali (pH>12) to ground water. Because there are few



commercial uses for carbide lime and producers are unwilling to pay the treatment and disposal costs for neutralising high pH, millions of tonnes of carbide lime are collected as waste in lagoons, pits, and heaps around the world, and the quantity is increasing annually [3]. Carbide Lime (CL) is the world's third-heaviest by-product waste material. According to calcium carbide industry statistics, the global production of calcium carbide is estimated to be 1370 million tonnes per year [4], [5]. CLW can be recycled and reused for a variety of purposes, including industrial processes and water treatment plants, construction engineering applications, soil stabilisation, particularly acid soil liming [5]. Since $\text{Ca}(\text{OH})_2$ is the main component of CLW, it offers a potential use in the cement industry and can be used as a cement substitute material [6]. The current research looked into a variety of materials such as steel slag, fly ash, limes and others for use in CO_2 curing. Besides, due to its high CaO content, CLW could be utilised as supplementary for cement in the making of concrete. Furthermore, the partial substitution of cement in concrete with waste will help to ease the strain on cement demand, lessen the degree of limestone reserves being depleted, and cut down on the amount of CO_2 released into the atmosphere during the cement production process [7]. However, there were lack of studies on the use of Carbide Lime Waste (CLW) on the CO_2 curing application as it is a possible material that produce thermodynamic stable calcium carbonate (CaCO_3) mineral after curing through CO_2 exposure application as shown in Equation 1.



To meet the global goal of zero CO_2 emissions by 2050, there is a pressing need to develop and deploy practical and economically viable technologies that can significantly reduce cement use or collect CO_2 in the atmosphere [1,8]. The carbonation reaction transforms the products of the hydration reaction into C-S-H gel or stable CaCO_3 . This process will help to transform CO_2 gas into stable C-S-H gel or calcium carbonate [9, 10]. Early calcium carbonate precipitates in cement paste pores. As a result, cement-based materials gain improved pore refinement, durability and strength [11]. This type of curing process gives the benefit toward the early strength development of concrete with lesser energy consumption. Due to the low CO_2 concentration in the atmosphere, carbonation reactions of cement-based materials occur at a very slow rate in the natural environment (400 ppm or 0.04%). Higher CO_2 concentrations and a regulated atmosphere are used to achieve higher carbonation rates such as temperature, relative humidity [12].

Thus, this study has been carried out to initiate the characterization of Carbide Lime Waste (CLW) under several microstructural examination; Particle Size Analyser (PSA), X-ray Fluorescences test (XRF), X-ray diffraction test (XRD), Thermogravimetric Analysis with Differential Thermal Analysis (TGA). There were two stages involved after characterisation of CLW. At the initial stage, the determination of the most optimum CLW were carried out and followed by further studies on the cement was mixed with different replacements (0%, 5%, 10%, 15% and 20%) of CLW under water curing. In order to obtain the optimum mix proportion of CLW replacement, the test that involved were flow, strength and UPV test. After obtain the desired optimum mix, the second stage was conducted. The optimum mixes among the various replacement was tested by using controlled CO_2 curing condition and compared with the normal OPC as a control data. The tests involved were strength and carbonation depth test. The samples were tested at day 1, 3 and 7 in order to determine the early strength properties. Lastly, the precipitation of stable CaCO_3 in the control and Carbide Lime cement pastes was examined by TGA examination.

2. Materials and Methods

2.1 Materials

The Ordinary Portland Cement (OPC), fine aggregates and Carbide Lime Waste (CLW) was used in this study. The density of OPC and fine aggregate is 3150kg/m^3 and 1576kg/m^3 , respectively. Tap water with the density of 1000kg/m^3 was utilized for all the samples preparation. The raw carbide lime waste was collected in Perak. The CLW was then dried in an oven at 105°C for 24 hours. The CLW was then

grounded with a steel ball mill machine to produce fine CLW powder that passing through $45\mu\text{m}$ within 4 hours process and the result of as shown in Figure 1. Particle Size Analyser (PSA) was conducted to determine the particle size distribution and fineness of OPC and CLW. CLW had 92% while OPC had 99.73% of particle distribution with fineness greater than $45\mu\text{m}$, respectively. These data suggested that CLW has enhanced the fineness following the grinding process, indicating a comparable fineness to the OPC. Due to its comparable fineness, CLW can be used as cementitious substitute materials in this context. The CLW consist of 96% of CaO content as shown in Table 1. Particle density or specific gravity is defined as the ratio of a certain volume of sample to the weight of an equal volume of distilled water at a constant temperature. In this process, the small sample of CLW has been tested by using small pycnometer method that complied BS 1377-2:1990. The specific gravity of CLW obtained is 2.11.

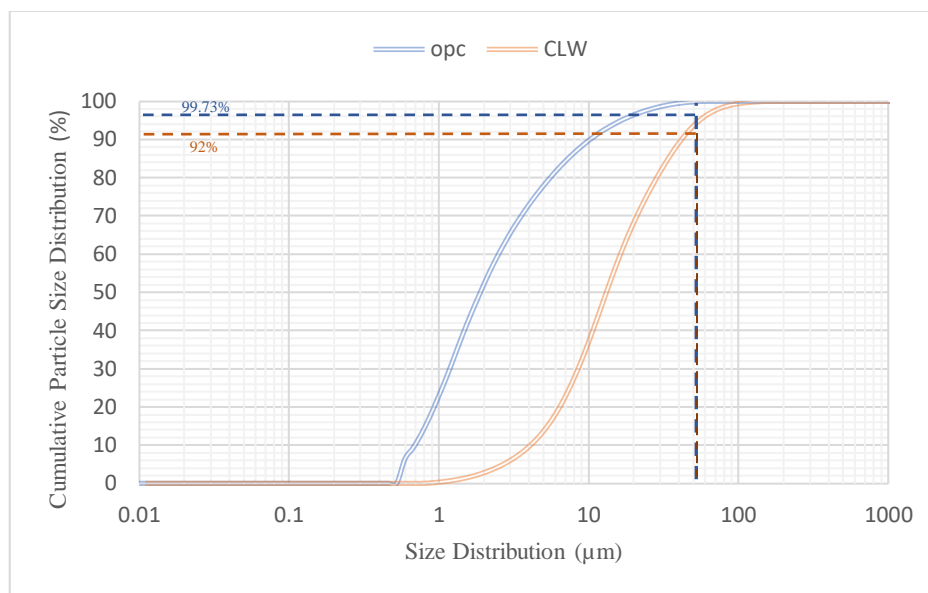


Figure 1. The particle size analysis of OPC and CLW

Table 1. The chemical composition of OPC and CLW

Component	Percentages (%)	
	OPC	CLW
CaO	60.0	96.0
SiO ₂	14.60	1.98
Al ₂ O ₃	3.34	0.495
MgO	0.62	0.392
Fe ₂ O ₃	2.20	0.302
TiO ₂	0.21	0.0252
MnO	-	0.0063
SO ₃	2.63	0.022

2.2 CLW Crystallographic Structure under X-Ray Diffraction (XRD)

XRD findings as shown in Figure 2 indicate that portlandite, calcite, and graphite are the major minerals in CLW. However, Ca(OH)₂ is the primary mineral phase of grounded CLW, while XRD has also shown some CaCO₃ and graphite. The presence of CaCO₃ in the grounded CLW is likely the result of a reaction between the primary mineral phase Ca(OH)₂ and CO₂ in the atmosphere during long-term stocking or dumping. Graphite, on the other hand, is a byproduct of the calcium carbide's manufacturing process

[15]. In addition to the XRD, thermogravimetric analysis (TGA) of CLW was performed to quantify the presence of portlandite and calcite for raw grounded CLW.

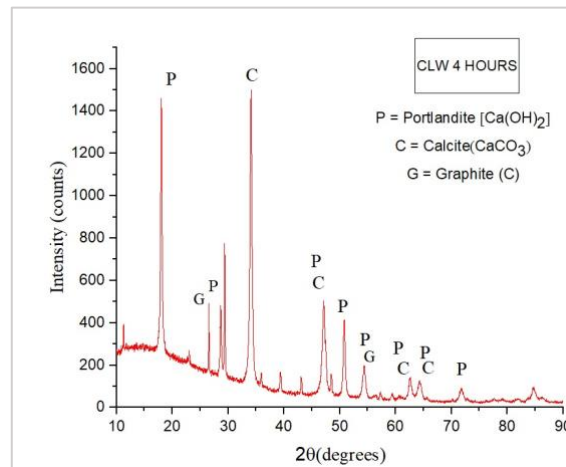


Figure 2. XRD for grounded CLW

2.3 CLW Characterisation under Thermogravimetric Analysis (TGA)

TGA as shown in Figure 3 shows the major minerals of CLW were Ca(OH)_2 and little amount of CaCO_3 . Basically, there were three different peaks that occurred in this process. The evaporation of free water and the breakdown of C-S-H and Aft can be seen within the weight loss between 30.9 to 320°C. The decomposition of Ca(OH)_2 at the second peak, from 332 to 500°C with 65.74%. This is the phase of $\text{Ca(OH)}_2 \rightarrow \text{CaO} + \text{H}_2\text{O}$. The decomposition of CaCO_3 can be seen at the third endothermic peak beyond 600 °C. The mass loss was 15.69%, attributed to the degradation of the calcium carbonate in the CLW waste.

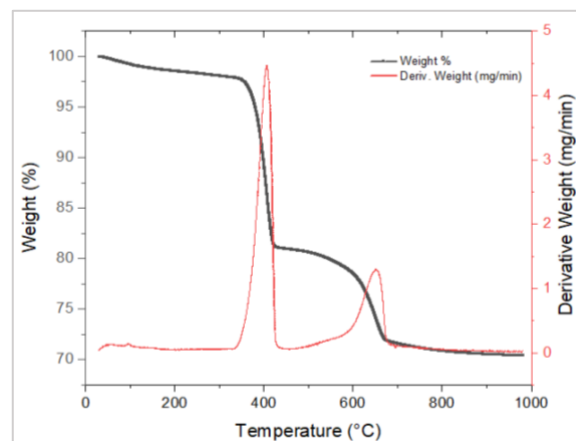


Figure 3. TGA of ground CLW

2.4 Mix Proportions

At the initial stage, five mix designs of replacement used to determine the strength of CLW and OPC mortar. One control mix mortar (without replacement) and four mixtures of CLW replacement to the OPC as shown in Table 2. Mortar that consist of CLW with OPC mixes were compared to mortars containing control OPC mixes. The control mixture is designed using the dry volume method, with a cement-to-sand ratio of 1:3 and the water cement ratio was 0.5.

Table 2. Mix design proportions

Mix Notation	OPC (%)	CLW (%)	W/C
0% CLW	100	-	0.5
5% CLW	95	5	
10% CLW	90	10	
15% CLW	85	15	
20% CLW	80	20	

2.5 Mixing Procedure

In this study, at the initial stage, the determination of the most optimum CLW mortar were carried out with different replacements (0%, 5%, 10%, 15% and 20%). Cement, CLW and sand were mixed for 2 minutes, followed by the addition of the appropriate amount of water and mixing for another 2 minutes. The mortar was placed in the moulds in two layers immediately after mixing and prodded 20 times after each layer to remove entrained air. After that, the cubic mould was vibrated for 5 seconds to ensure proper compaction. The open surfaces of the specimens will be levelled smoothly to provide homogeneous force distribution under load as to reduce strength measurement error [13]. To prevent evaporation loss, the cast specimens were wrapped. Specimens will be taken from their moulds after 24 hour and will be placed in a water tank with a temperature of 22 (± 2 °C) until the time of the test.

2.6 Water and CO₂ Curing

The samples were cured under water curing in order to determine the optimum mix proportion. Then, the desired optimum mix proportion was tested under controlled CO₂ curing condition. For CO₂ curing, the chamber was used to provide the requirement of CO₂ amount that needed. The chamber was outfitted with a pressure gauge and a safety valve, and it was connected to a CO₂ gas tank cylinder containing pure CO₂ gas with a purity of 98%. At a pressure of 15 psi, CO₂ will be injected into the chamber. The relative humidity (RH) was fixed between 65-70%. The temperature and CO₂ concentration has been fixed at 59.9°C (+1°C) and 19.9% (+0.1%).

2.7 Testing

2.7.1 CaCO₃ Precipitation under TGA analysis. The sample weight approximately 4 to 5 mg was heated at a rate of 10 °C/min from 30 to 1000 °C in a nitrogenic (Inert) atmosphere. It was conducted for cement paste to investigate their reactivity under microstructural examination for CaCO₃ precipitation under CO₂ curing.

2.7.2 Flow Water Table. Following the completion of mixing, the flow rate of the fresh mortars was determined using the flow table method in accordance with ASTM C1437 for the workability determination. The acceptable flow test range of mortar was 110 (± 5 %).

2.7.3 Compressive Strength. Three cubic samples were taken for each test to achieve the average result. A total of 60 mortar specimens with sizes of 50 x 50 x 50 mm³ were evaluated on with a load rate of 2 kN/s. The test was carried out in accordance with ASTM C207. Compressive strength test for the mortar in cubic samples with dimensions of 50x50x50 mm³ were performed using an electronic universal testing machine. After 24 hours of casting, the mortar samples were demoulded and soaked in tap water until they reached the testing age. The compressive strength of the mortars was measured at three distinct ages: 1, 3, 7, and 28 days. The optimum mix proportion under water curing was obtained from this stage. after obtained the desired optimum mix proportion, the strength test was conducted under controlled CO₂ curing.

2.7.4 Ultrasonic Pulse Velocity (UPV). The standard test method for pulse velocity through concrete ASTM C597, was adapted for this study. One of the non-destructive tests on mortar is ultrasonic pulse

velocity, which measures the time of wave transmission through the mortar. This part made use of mortar prism sample of 40x40x160 mm³. The obtained wave speed was then divided by the distance travelled over the measured period. The UPV testing was carried out in a dry environment. As a result, the wet mortar must be dried for a period of time before testing. Before the test, the UPV testing instrument was calibrated using the reference bar to eliminate instrumental error. The UPV was determined using the direct transmission approach, which involves placing transducers on opposite surfaces of the mortar sample. To achieve good contact between the transducer and the sample surface, the end surfaces of the specimens were smoothed before testing, and a thin layer of grease was applied as coupling gel. In this study, this test was conducted for water curing in order to obtain the optimum mix proportion and the relationship between compressive strength and UPV was analysed.

2.7.5 Carbonation Depth. Prism specimens of 40x40x160 mm³ were used for the carbonation depth study (RILEM, 1988). The carbonation progression over time was examined qualitatively by spraying freshly fractured mortar surfaces of half flexural test specimens with a phenolphthalein solution (0.2%). A computerised calliper was used to measure the carbonated depth. The measurements were gathered from three mortar specimens. In this study, the testing had been conducted on two mixes with CO₂ curing method, control and the optimum CLW mortar mixes. The relationship between strength and carbonation depth was analysed between water curing and controlled CO₂ curing condition.

3. Results and Discussion

3.1 Workability

The diameter of mortar flow increased when Portland cement was replaced with 5% of CLW, and then reduced as the CLW percentages increased (from 10%, 15% and 20%) as shown in Figure 4. The increased percentage of CLW material, which is incredibly fine and has a low specific gravity, has a major impact on the rise in the amount of water needed to wet the particles [16]. The flow rate value obtained for the test is in the range of 174 mm to 211 mm. According to the ASTM C207 Specification for Hydrated Lime for Masonry Purposes, the mortar has a good workability of 100 to 115 percent. The design mix for the control mortar was within an acceptable range, with the control mix design falling between the specified ASTM range of 107.33%. The data given for the 5% and 10% of CLW mortar were still in the range of good workability, where both of these replacements were in the range of 110% ± 5%. The decreased in the flow rate was particularly noticeable with increased of the CLW percentages. This reduction could be due to the CLW grains' high absorption capacity [17]. Besides, the fineness size of CLW that grounded until 45µm was influenced the high absorption capacity.

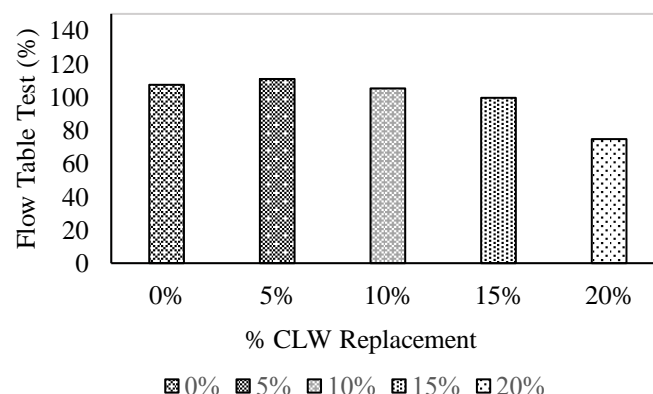


Figure 4. Workability test for tested mortar

3.2 Compressive Strength and Ultrasound Pulse Velocity (UPV) under Water Curing

The UPV used to analyse the void presence, concrete quality, crack repair effectiveness, and concrete durability [17]. In this finding, the UPV values were inconsistent within the mixes. However, as the

curing period continued, the result showed all the mixes exhibits such a good UPV. This is due to the most of the mixes pores in the mortar matrix were filled with hydration products (i.e., C-S-H), resulting in more compact and denser microstructure specimens and hence, the pulse velocity rises. [19]. Thus, the velocity of ultrasonic pulses rises as the pulses need less time to penetrate through the solid mortar matrix [22]. This study showed that CLW content and curing age have a substantial impact on the UPV and compressive strength of mortar. The linear regression analysis was plotted in order to determine the most significant R^2 value. The coefficient of determination (R^2) varies from 0.8310 to 0.9644 as shown in Table 3. It can be seen that the OPC mortar and 5% of CLW mortar showed the same highest significant R^2 value, 0.9644. This represents that the variation in compressive strength with UPV within the mixes were accounted well by linear regression relationship. Thus, 5% CLW mortar was chosen as the most optimum among the mixes based on the relationship and highest R^2 value.

Table 3. Correlation of UPV and compressive strength for various mix replacement

Mix Replacement (%)	Curing age (days)	Correlation between compressive strength and UPV	R^2
0		$y = 21.571x - 50.91$	0.9644
5	1,3,7, 28	$y = 30.028x - 82.894$	0.9644
10		$y = 24.57x - 62.458$	0.8610
15		$y = 33.962x - 96.893$	0.8398
20		$y = 17.475x - 41.533$	0.8310

3.3 CLW Mortar under Water and CO_2 Curing Condition

The difference in the carbonation depth and curing condition between control and 5% CLW mortar can be observed as shown in Figure 5. The finding proved CO_2 curing enhanced the carbonation depth compared to the normal water curing. It can be seen that the difference between Day 1 by water curing was not efficient as the Day 1 by CO_2 curing. This study can be justified with the comparison to the normal curing (with RH more than 95%), the dry curing condition was enhanced in increasing the carbonation degree. This is due to the water produced during carbonation reactions hinders CO_2 diffusion through the pores of the cement paste, thereby reducing the carbonation rate, which is defined as the depth of carbonation per exposure time [23]. As high temperatures increased the transmission coefficient and reaction rate of carbon dioxide, the carbonation degree and depth of concrete specimens increased [24]. However, the carbonation depth for 5% CLW mortar under water curing condition was enhanced compared to the control OPC mortar. This shows that the $Ca(OH)_2$ in the CLW helps to increase the penetration of the CO_2 absorption. Meanwhile, the incorporation of temperature in the CO_2 curing helps to remove the excess water. It has been demonstrated that the rate of carbonation increases as water is removed from the cement mortar pores, allowing the ingress of CO_2 . The carbonation depth for both control OPC and 5% CLW mortar were improved under controlled CO_2 curing condition. This is due to the several factors that affecting the rate of carbonation such as time, temperature and CO_2 concentration in which the carbonation reaction occurs all affect the rate of carbonation. Apart from that, the early strength of both OPC and 5% CLW mortar was enhanced by controlled CO_2 curing condition. Besides, the findings showed that there were similarities within the carbonation depth pattern between OPC mortar and 5% CLW mortar with CO_2 curing. This showed that the potential of the CLW can be further investigated to be used as a cement replacement. In term of compressive strength, Figure 6 shows the compressive strength increases with the application of CO_2 curing and increasing of CO_2 curing time respectively. It can be seen where the strength of control mixture obtained was enhanced by 10.4 %, 22% and 28% by 1 day, 3 days and 7 days of CO_2 curing. Meanwhile, 5% of CLW mortar, the strength was enhanced by CO_2 curing, 6% and 16% at day 1 and day 3. At day 7, it found that there was small enhancement between water and CO_2 curing condition, which is 6.3%. However, the strength for 5% of CLW mortar from day 3 to day 7 was increased for both curing conditions.

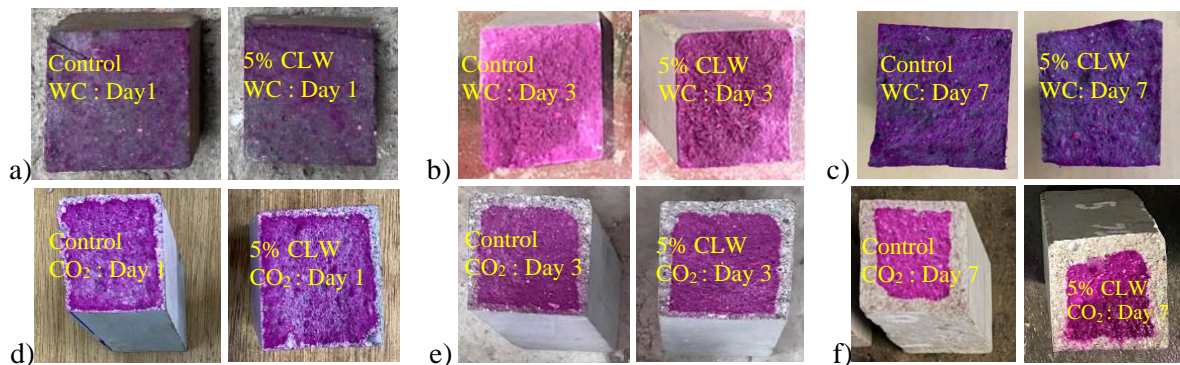


Figure 5. The carbonation depth within the mixes. a) Day 1 by water curing (WC), b) Day 3 by water curing (WC), c) Day 7 by water curing (WC), d) Day 1 by CO₂ curing, e) Day 3 by CO₂ curing and f) Day 7 by CO₂ curing

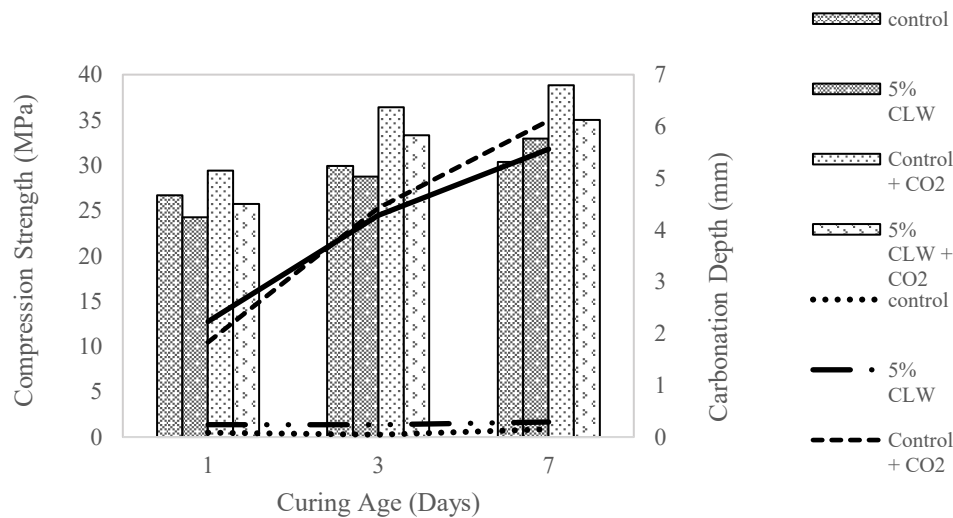


Figure 6. The relationship between compression strength, carbonation depth and curing ages between water and CO₂ curing

3.4 Microstructural Analysis under TGA

Table 4 shows the TGA results for the normal OPC and 5% CLW cement pastes. The amount of portlandite, Ca(OH)₂ in each cement pastes was lesser due to the increasing in the carbonation rate. The corresponding peak could not show up if the carbonation process consumes the majority of the Ca(OH)₂. The strength of the weight loss peak associated with calcite, which occurs between 600-800 °C, increases as the degree of carbonation in a cement paste sample increases [25]. The amount of CaCO₃ in the normal OPC mortar was increased from day 1 to day 3. However, at day 7, the amount of CaCO₃ was started to decrease. The improvements can be seen at the early ages of CO₂ curing in comparison to other concretes because pervious concretes' porous media aids the entry of CO₂ gas. As a result, these improvements decline for the later ages [26]. Thus, normal OPC does not fulfil the CO₂ absorption at early ages. Meanwhile, surprisingly, the 5% CLW mortar shows an increment of CaCO₃ at all early ages. When compared to other samples, the calcite peak for the carbonation-cured 5% CLW sample showed the largest peak as shown in Figure 7. These proved that the CLW can be used to double up the amount of CO₂ absorption in the mortar. Apart from that, this finding shows that the potential of CLW can be further utilised as cement replacement due to its behaviour. Small amount of mass loss was observed between 200 and 450°C. This may be proof of the quick carbonation reaction kinetics and the creation of carbonates in the CO₂-cured samples prior to the formation of Ca(OH)₂ [27].

Table 4. Ca(OH)₂ and CaCO₃ from TGA analysis

Day (s)	CO ₂ concentration (%)	Temperature (°C)	Ca(OH) ₂ (%)		CaCO ₃ (%)	
			Control	5% CLW	Control	5% CLW
1			0.96	0.44	39.66	44.52
3	20	60	0.34	0.35	46.57	47.57
7			0.49	0.21	43.50	48.98

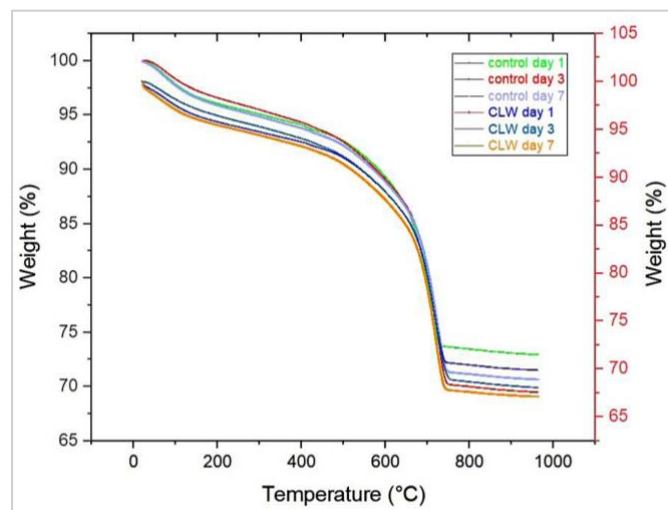


Figure 7. TGA results of normal OPC and 5% CLW cement paste samples at the early age CO₂ cured

4. Conclusions

Based on the findings that have been obtained throughout this study, the following are concluded:

1. CLW is calcium based materials which rich in CaO content and dominantly has Ca(OH)₂ behaviour with little amount of CaCO₃ (carbonated by natural ambient environment as CLW was dumped into the open landfill). The incorporation of up to 5% of CLW as a potential of cement replacement was within the acceptable range of good workability. The optimum 5% of CLW had proved the best strength, UPV and workability under water curing condition as the R² value between these two parameters has very strong relationship.
2. In term of the early strength of both OPC and 5% CLW mortar under CO₂ curing, the normal OPC mortar achieved higher strength than 5% CLW. However, the strength of 5% CLW mortar was enhanced under CO₂ curing as compared to 5% CLW mortar under water curing condition.
3. This finding proved CLW that rich in CaO content and dominantly has Ca(OH)₂ behaviour can used to double up the CO₂ absorption. TGA analysis shows that the 5% CLW cement paste under CO₂ curing has high potential to capture CO₂ as their high amount of CaCO₃ precipitation compared to control sample.

The carbonation under natural environmental conditions is unable to attain the same carbonation volume as that accomplished by controlled CO₂ curing condition. Overall, this study has added to our understanding of current construction materials. The selection of alternative material leads to the intelligent function on CO₂ capturing technology. Further to that, this research contributes to the current global agenda of reducing CO₂ emissions and greenhouse gas emissions. Thus, with the availability of highly potential of valuable waste material, this research is intended to add on the new findings in Malaysian construction industry based on local materials as to enhance current construction technology in future.

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