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Maisa El Gamal  
*Zayed University, maisa.elgamal@zu.ac.ae*

Ameera F. Mohammad  
*United Arab Emirates University; KU Leuven*

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# Optimization and enhancement of CO<sub>2</sub> sequestration in industrial wastes for environmental applications

Maisa El Gamal<sup>a,\*</sup>, Ameera F. Mohammad<sup>b,c</sup>

<sup>a</sup> College of Natural and Health Sciences, Zayed University, Abu Dhabi, United Arab Emirates

<sup>b</sup> College of Engineering, UAE University, Al Ain, United Arab Emirates

<sup>c</sup> Chemical Engineering Department, KU Leuven, Leuven, Belgium

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## Abstract

This paper explores carbon capture and storage (CCS) through carbide lime waste (CLW), a by-product of acetylene production, under different conditions. This process is specifically designed to provide an onsite waste management solution for several industries that can easily be integrated into existing systems. In addition, the effect of the carbonation process on collected solids morphology and average particle size was studied. The structural and chemical characteristics of the carbonated carbide lime samples were investigated using X-ray diffraction, scanning electron microscopy, TGA analysis, and Raman spectroscopy. The effect of carbonation conditions on the total dissolved solids and change in pH was studied. All carbonated products exhibited a calcite crystal structure with a specific morphology at each carbonation condition. High CLW concentration helped to form singular long rods and agglomerated spheroidal particles. In contrast, low CLW concentration promoted truncated prismatic morphology. The maximum pH reduction was honored at the highest CLW to water ratio. In addition, a maximum conductivity reduction of 96.87% was obtained at pH 12.7, and a CLW to water ratio of 1:10. Raman analyzer, X-ray diffraction, and scanning electron microscopy confirmed the minimum CO<sub>2</sub> uptake value for the higher carbide lime to distilled water ratio. This is due to the increase in the concentration of calcium species in the CLW–water mixture, which will form a thin carbonation layer that is distributed among calcium species.

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**Keywords:** CO<sub>2</sub> capture; CO<sub>2</sub> uptake; Carbide lime waste; Carbonation; Calcium carbonate; Crystal morphology

## 1. Introduction

The synthesis of calcium carbonate involves double salt decomposition and carbonation processes. The carbonation process involves the reaction of lime (calcium hydroxide) with carbon dioxide gas to produce insoluble calcium carbonate solids and water. The thermodynamic analysis of this chemical reaction using HSC software by

\* Corresponding author.

E-mail address: [Maisa.Elgamal@zu.ac.ae](mailto:Maisa.Elgamal@zu.ac.ae) (M. El Gamal).

A. Roine [1] shows that the reaction is spontaneous up to 700 °C. The changes in the heat of reaction up to the same temperature are negative, which indicates that the reaction is exothermic. However, lime manufacturing is an energy-intensive process and releases carbon dioxide gas as an undesirable by-product. An alternative route is using waste material containing a high percentage of calcium hydroxide to avoid specific energy requirements and the greenhouse effects of the emitted carbon dioxide gas [2–4]. Carbide lime waste (CLW) is a by-product of acetylene synthesis via the hydrolysis of calcium carbide ( $\text{CaC}_2$ ) [5]. The CLWs are typically generated in aqueous slurry and are mainly composed of calcium hydroxide (more than 85%). The key features of CLW were similar to those of synthesized lime in chemical and mineralogical compositions except for the presence of unreacted carbon in CLW. CLW also contains small concentrations of silicates and calcium carbonate. Suwanthai et al. [6] studied the effect of lime concentration on the resulting calcium carbonate efficiency. They showed that maximum calcium extraction could be achieved using a small solid-to-liquid ratio. Huang et al. (2007) indicated that high-purity amorphous calcium carbonate could be extracted from medium- and low-grade lime by implementing an acidic cation exchange resin [7]. The carbonation of CLW has attracted considerable attention as an environmentally friendly process for producing a specific crystal morphology, particle size, and shape by controlling carbonation conditions such as CLW concentration, in addition to its high ability to capture  $\text{CO}_2$  [3]. This work will present the ability of carbide lime to capture  $\text{CO}_2$  at different CLW concentrations and will visualize the effect of carbonation conditions on the collected solids morphology and average particle size.

## 2. Experimental methodology

### 2.1. Description of the original carbide lime samples

Samples of real CLW generated as a by-product in the acetylene manufacturing process were collected from Sharjah Oxygen Company, Dubai, UAE. The carbide sludge was dried in an oven at 110 °C for 24 h. Next, the solid samples were grounded with a mortar followed by sieving. The majority of the resultant particles (90%) were less than 100  $\mu\text{m}$ . The grounded samples were then characterized and used in the experiments. The chemical composition of CLW was determined using inductively coupled plasma atomic emission spectroscopy (ICP-AES); it was found to contain a high percentage of CaO (68%) and low contents of  $\text{MgO}$ ,  $\text{FeO}$ ,  $\text{Al}_2\text{O}_3$ , and  $\text{SiO}_2$ . The XRD analysis showed that CLW mainly comprised portlandite with unreacted carbon in the form of graphite. Thermogravimetric analysis (TGA) was conducted to examine the thermal degradation of the original, and the treated CLW at three temperature ranges, i.e., 150 °C, 400–620 °C, and 620–900 °C, which corresponded to the removal of water portlandite decomposition, and release of carbon dioxide, respectively [5]. The pH of CLW was measured to be approximately 12.27, which proves its high reactivity with acidic carbon dioxide gas [2]. In addition, the total dissolved solids and conductivity were measured to be approximately 4080 ppm and 8947  $\mu\text{S/cm}$ , respectively.

### 2.2. Carbonation reactor and system setup

As shown in Fig. 1, a Plexiglas vessel with a working volume of 1 L was fabricated for this work and operated in a semi-batch mode, in which the CLW and distilled water mixture (CLW–DW) was exposed to a continuous flow of carbon dioxide–air mixture (10%  $\text{CO}_2$  and 90% air vol.%). The inlet feed gas line was extended from the top to the vessel's bottom to ensure maximum contact between the CLW–DW and  $\text{CO}_2$  gas mixtures.

The gas flow rate was controlled using a mass flow controller connected to supervisory control and data acquisition station to monitor and control the gas flow rate during each experimental run. The treated gas left the CLW–DW compartment from the top of the vessel and passed through a moisture trap to remove any water droplets. It was then directed to a  $\text{CO}_2$  gas analyzer [Model 600 Series-Nondispersive infrared].

### 2.3. Experimental procedure

The Plexiglas vessel was filled with CLW–DW mixture (1 L) at 1:5, 1:8, 1:10, and 1:12 CLW–DW mass ratios. The  $\text{CO}_2$ –air gas mixture flow rate was controlled to be 1 L/min for all experimental runs. The initial and final CLW–DW mixture pH, total dissolved solids (ppm), and conductivity ( $\mu\text{S/cm}$ ) for the carbonation process were recorded. The total  $\text{CO}_2$  uptake for each unit volume of the CLW–DW mixture was measured by integrating the function of  $\text{CO}_2$  capture with the carbonation reaction time until the full saturation state is reached. After each run,

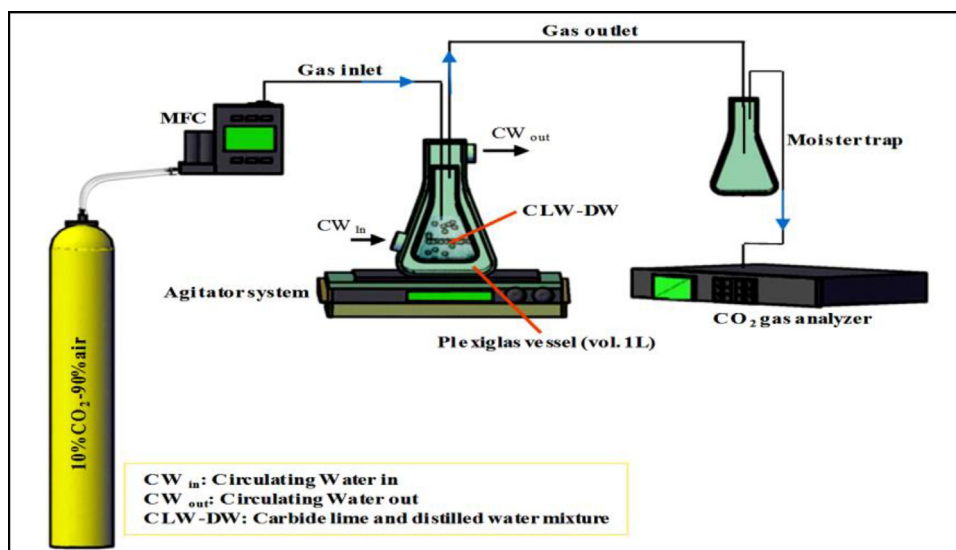


Fig. 1. Schematic of the CLW carbonation system.

the treated CLW–DW mixture samples were collected after simple filtration and then dried in the oven at 110 °C for 24 h. Powder XRD, Raman spectroscopy, scanning electron microscopy (SEM), and TGA was used to identify the effects of CLW–DW ratio on the crystalline structure and morphological characteristics of the carbonated CLW.

### 3. Results and discussion

#### 3.1. Effect of CLW–DW ratio on the structure of carbonated products

The effect of the CLW–DW ratio on the calcite structure was studied by decreasing the mass ratio from 1:5 to 1:12 to investigate the change in the morphological structure, as presented in Fig. 2. These experiments were conducted at a high pH range (12.4–12.9) and a constant carbonation temperature of 20 °C. Initially, at a CLW–DW ratio of 1:5, a mixture of singular elongated rods and agglomerated spheroidal particles with an average size of 0.32–2.08  $\mu\text{m}$  was observed. Upon decreasing the CLW–DW ratio to 1:8, a change from singular rods to submicrometric elongated agglomerates was observed. A truncated prismatic morphology was also observed at the same CLW–DW ratio (1:8). By decreasing the CLW–DW ratio from 1:10 to 1:12, the submicrometric (chain-like) elongated agglomerates and truncated prismatic morphology were transformed into a more concentrated truncated prismatic morphology. Upon increasing the CLW–DW ratio from 1:12 to 1:5, the calcite particle size decreased from 0.52–2.08 to 0.24–0.31  $\mu\text{m}$ , respectively. This can be attributed to the increase in the concentration of calcium species in the CLW–DW mixture, and accordingly, a thinner carbonation layer is distributed among calcium species [8]. These results were confirmed by the Raman analysis of these samples Fig. 3, in which a noticeable deviation in the recorded intensity was noticed upon increasing the CLW–DW ratio up to 1:5. Thus, a significant reduction in the particle size resulted in a decrease in the Raman intensities [9]. These findings were confirmed by XRD analysis Fig. 4, which indicated that the change in the CLW–DW ratio only affects the morphology and size distribution of the calcite crystals. This was suggested by the minor changes in the peak intensity of the samples with different CLW–DW ratios [10]. In addition, a reduction in the calcite degradation temperature was observed with an increase in the CLW–DW ratio from 1:12 to 1:5 in the carbonation process, Fig. 5. This can be attributed to the carbon dioxide uptake value for each mass unit of CLW. A higher concentration of the CLW–DW mixture requires a lower carbon dioxide uptake ( $\text{CO}_2$  mass: CLW mass) [11].

#### 3.2. Effect of the carbonation process on the total dissolved solids of treated CLW

The carbonation of a lime–DW mixture typically causes a reduction in the total dissolved solids [2,12]; similar behavior is expected in the carbonation of CLW. The initial pH plays a significant role in the carbonation process,

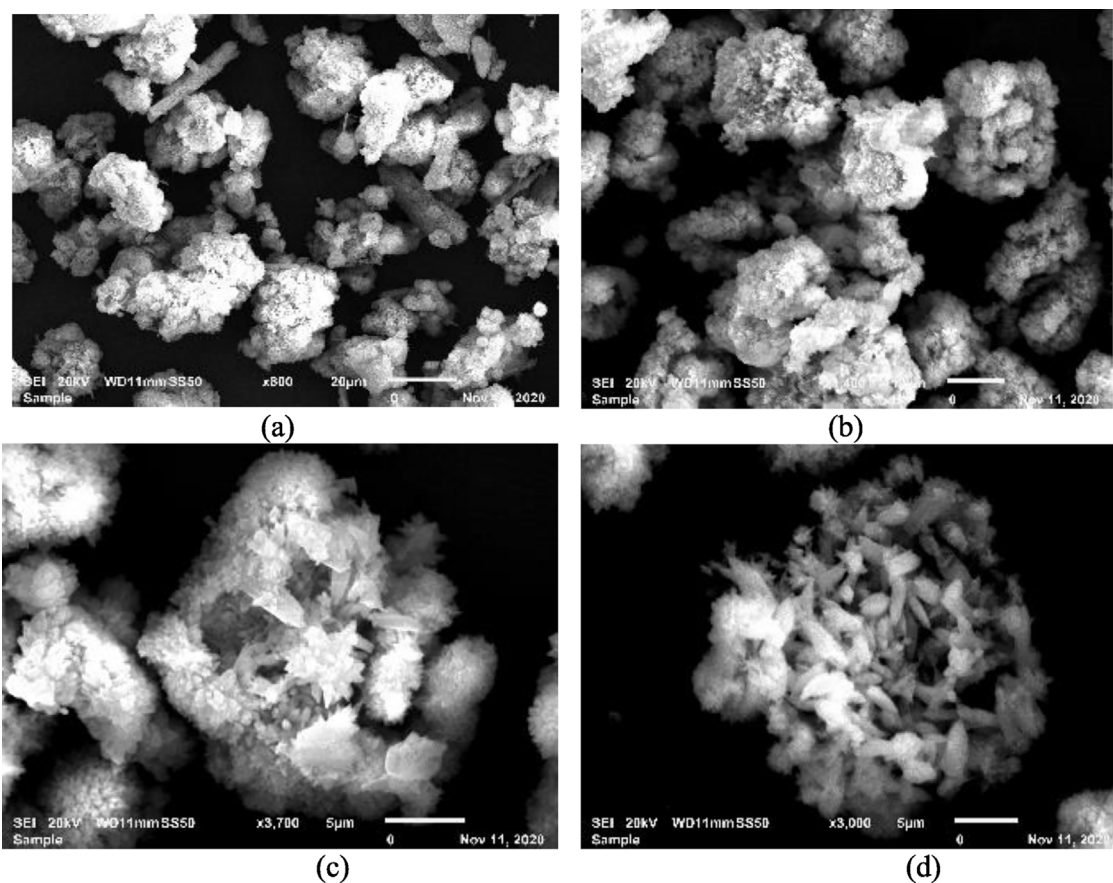


Fig. 2. Cross-sectional SEM analysis of treated CLW samples at a CLW–DW ratio of (a) 1:5, (b) 1:8, (c) 1:10, and (d) 1:12.

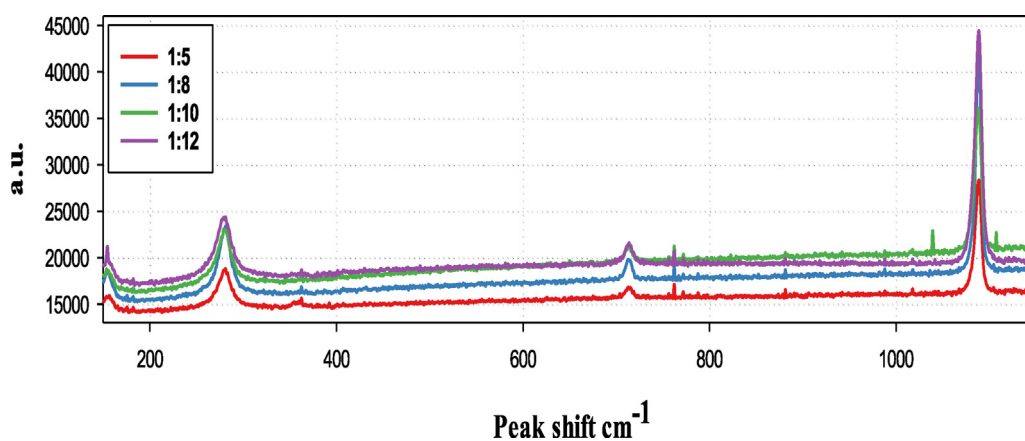


Fig. 3. Raman graphs of the treated CLW material at different CLW–DW ratios; ; 1:5, 1:8, 1:10 and 1:12.

where higher pH is associated with the maximum reduction in the total dissolved solids (Table 1); this is related to the reaction rate and acidity of the carbon dioxide gas [13].

Table 2 shows the effect of the CLW–DW ratio on the dissolved solids, which was not significant, as the pH range (12.9–12.4) for these ratios is very narrow and considered to be high. The effect of the carbonation process on the

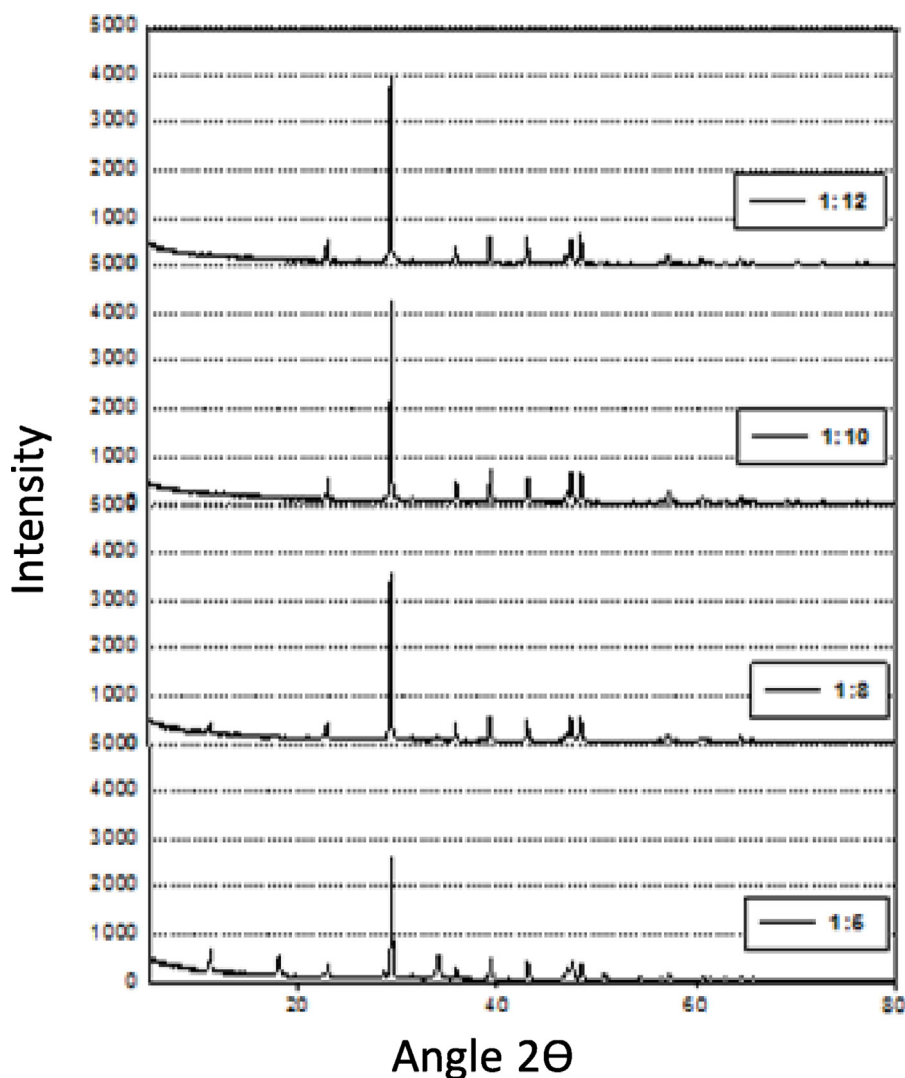


Fig. 4. X-ray analysis of treated CLW at different CLW–DW ratios; 1:5, 1:8, 1:10 and 1:12.

Table 1. Effect of initial pH on the reduction in the total dissolved solids.

pH	Initial TDS (ppm)	Final TDS (ppm)	Time of reaction (min)
10.9	248	160	15
11.75	496	383	60
12.7	4877	325	248

conductivity of the treated CLW mixture is very similar to that of the total dissolved solids. Moreover, a maximum conductivity reduction of 96.87% (from 8950.0 to 279.6  $\mu\text{S}/\text{cm}$ ) was obtained at a pH of 12.7, temperature = 20  $^{\circ}\text{C}$ , and CLW–DW ratio of 1:10.

### 3.3. Effect of the carbonation process on the pH of the treated CLW

The pH of the CLW–DW mixture was reduced through the carbonation reaction (Table 3). This can be attributed to the reduction in the hydroxide ions and the increase in acidity due to the continuous flow of carbon dioxide gas.



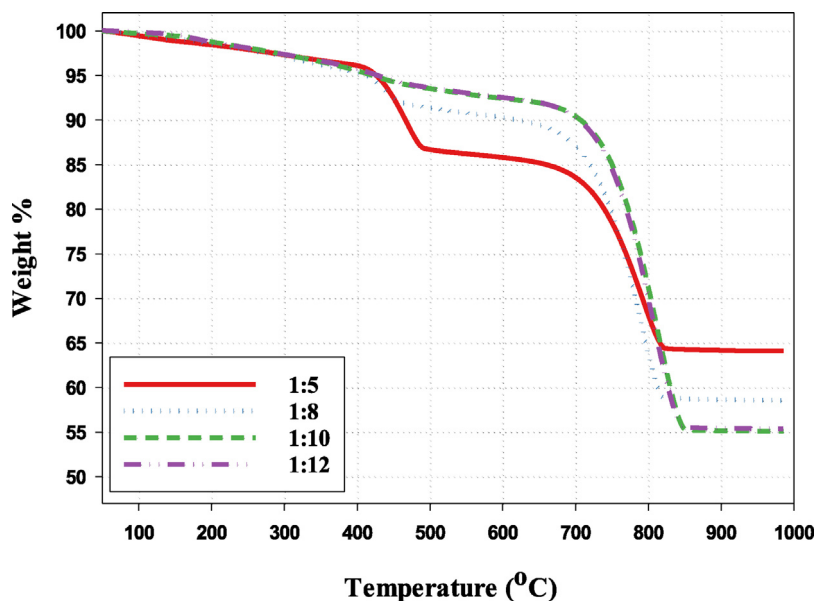


Fig. 5. TGA of treated CLW samples at different CLW–DW ratios; 1:5, 1:8, 1:10 and 1:12.

**Table 2.** Effect of CLW–DW ratio on the reduction in the total dissolved solids.

S : L ratio	Initial TDS (ppm)	Final TDS (ppm)	Time of reaction (min)
1:05	4650	290	240
1:08	4877	325	240
1:10	4879	133	240
1:12	4630	388	240

**Table 3.** Effect of CLW–DW ratio on the reduction in the pH of the treated CLW.

S : L ratio	Initial pH	Final pH	Time of reaction (min)
1:05	12.6	6.14	240
1:08	12.8	6.78	240
1:10	12.83	9.7	240
1:12	12.63	7.22	240

These results are in agreement with the findings of previous studies [2,12,14]. The maximum pH reduction was observed at the highest CLW–DW ratio (1:5) owing to the high reaction rate (maximum concentration of calcium species).

### 3.4. Effect of the CLW–DW ratio on the CO<sub>2</sub> uptake

The previous section described the effect of the carbonation conditions on the calcite particle size and morphology. These phenomena were further investigated by recording the actual CO<sub>2</sub> capture percentage overtime at different CLW–DW ratios. Moreover, the total CO<sub>2</sub> uptake per mass of the CLW was calculated at each specific CLW–DW ratio based on the curves of the CO<sub>2</sub> capture% over time [15,16]. Fig. 6 shows that the increase in the CLW–DW ratio decreases the CO<sub>2</sub> uptake per CLW mass, and accordingly, smaller particles are generated. Therefore, the carbonation conditions can be successfully used to obtain specific calcite morphology and particle size. However, further investigation and parametric analysis are required to optimize the carbonation conditions for each required calcite crystal characteristic.

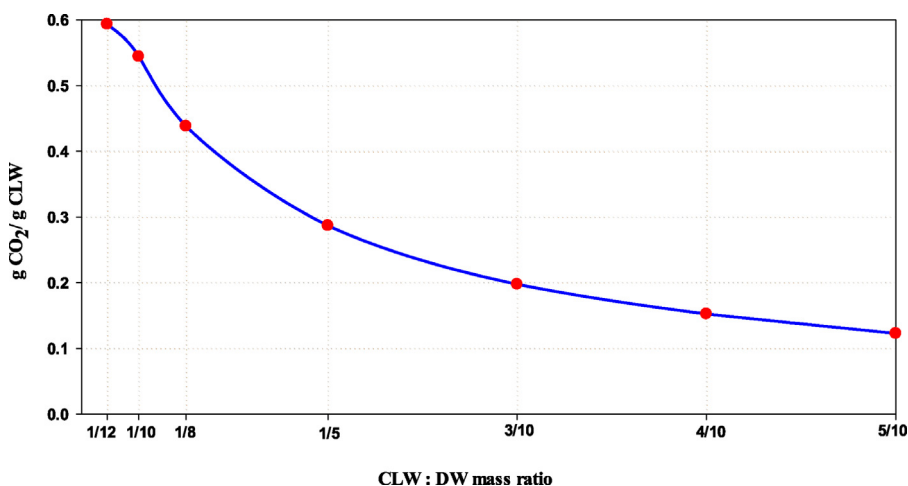


Fig. 6. CO<sub>2</sub> uptake (g CO<sub>2</sub>/g CLW) over different CLW to DW ratios.

#### 4. Conclusions

The ability of CO<sub>2</sub> capture through the carbonation process of the carbide lime waste (CLW) was tested at different CLW concentrations. It was found that the maximum CO<sub>2</sub> uptake value was reported for the minimum CLW–DW ratio. Singular long rods and agglomerated spheroidal calcium carbonate particles were produced at high CLW concentration, while truncated prismatic morphology was dominant at low concentration. The pH reduction was maximum at the highest CLW–DW ratio. Accordingly, the structural and chemical characteristics of the carbonated carbide lime could be predicted based on the used CLW concentrations.

#### Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

#### Data availability

No data was used for the research described in the article.

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