

# Physical, thermal, and microstructural effect of CO<sub>2</sub> in carbonic acid on mortar

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## ARTICLE INFO

### Article history:

Received 04 February 2025

Revised 20 June 2025

Accepted 09 October 2025

Online first

Published 31 October 2025

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### Keywords:

Carbon dioxide

Calcium carbonate

Carbonation

### DOI:

10.24191/mjcet.v8i2.5012

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## ABSTRACT

Existing studies have shown current methods of carbon dioxide infusion are limited to slow carbonation rates and lack of carbon dioxide penetration. This study aims to tackle these problems by proposing a new method of infusion by adding carbon dioxide via liquid directly during mixing process. This study also investigates the thermophysical behaviour of mortar subjected to liquid-phase CO<sub>2</sub> infusion, focusing on its effects on mechanical strength, hydration, and carbonation processes. Mortar specimens were prepared and infused with carbonic acid during mixing, followed by compressive strength testing, thermogravimetric analysis (TGA), and scanning electron microscopy (SEM). The results indicate that liquid-phase CO<sub>2</sub> infusion increases calcium carbonate (CaCO<sub>3</sub>) formation, as confirmed by SEM observations of calcite crystallisation. Compressive strength measurements showed that 100% infusion CO<sub>2</sub> mortar does surpass normal mortar mixture showing potential in this method. TGA revealed that the amount of CO<sub>2</sub> retained or released is highly dependent on the mixing and curing conditions, but higher infusion does results in high amount of calcite form, with hydration dynamics significantly influencing the final strength. This is supported by SEM results showing more calcium carbonate formed for all samples but also the formation of pores due to high water content inside mortar.

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## 1. INTRODUCTION

Concrete has been a fundamental material in the construction industry for centuries. Its production involves cement as a binder that holds aggregates together to form the final structure. Portland cement is the most widely used binder in concrete, playing a crucial role in various construction sectors. Globally, concrete accounts for approximately 70% of all building and construction materials (Qian et al., 2018). However, widespread use of concrete has led to several challenges, particularly related to environmental impact, safety, and cost. During the early stages of concrete production, carbonation can increase concrete's strength by improving its resistance to stress, particularly in flexible pavements, by accelerating the carbonation process (Kamal et al., 2020). However, the timing of carbonation is critical, as its effects can vary significantly depending on the phase of concrete development. In the mature phase, carbonation may reduce pore solution, lower pH, and increase the risk of corrosion, while early-stage carbonation can enhance tensile strength and reduce permanent deformation, due to the formation of nanosized calcium carbonate (Kamal et al., 2020).

Several methods exist to introduce carbon dioxide into concrete. These include carbon-conditioning through the addition of carbon dioxide in the form of aggregates, direct injection during mixing, and carbonation curing, where concrete blocks are exposed to high carbon dioxide concentrations during curing. Studies done by Monkman & MacDonald (2017) and Zhang et al., (2017) prove that carbonation curing can improve concrete performance, boosting compressive strength by 15% to 20% after 28 days, depending on variables such as pressure, humidity, moisture content of aggregates, and CO<sub>2</sub> concentrations. However, carbonation curing faces challenges, particularly the slow diffusion of carbon dioxide into the concrete matrix during the early stages, which results in slower carbonation rates. Furthermore, Qian et al. (2018) highlighted the impracticality of injecting gas into large concrete blocks, as it requires large quantities of precast materials and cannot be applied in situ. As carbonation progresses, the outer layers of concrete become denser, obstructing carbon dioxide diffusion and limiting the depth of carbonation (Tam et al., 2021).

An alternative method is carbon-conditioning, which involves infusing carbon dioxide into concrete using solid waste materials such as recycled aggregates and steel slag (Ashraf, 2016; Lu et al., 2019). Metal oxides in these materials react with carbon dioxide to form carbonates and effectively trapping carbon dioxide within the concrete (Šavija & Luković, 2016). However, this approach depends heavily on the chemical composition and physical properties of the aggregates. Some solid wastes can cause internal sulphate attacks or leach toxic substances into the environment, presenting environmental and safety risks (Liu & Meng, 2021). Another approach involves directly dissolving carbon dioxide into the cement mixture during the mixing process to facilitate the formation of calcium carbonate. While this method can sequester a small amount of carbon dioxide (less than 0.3% of the cement mass), it faces limitations due to the low solubility of carbon dioxide in water and the slow reaction rate between carbon dioxide and alkalis in fresh concrete. Moreover, special facilities are required to prevent the release of unreacted carbon dioxide into the atmosphere during mixing (Liu & Meng, 2021; Monkman & MacDonald, 2017).

When CO<sub>2</sub> is infused into water, it reacts to form carbonic acid, which shows by Eq. (1):



During the mixing process, carbonic acid can dissociate to produce hydrogen ions and bicarbonate ions, while the cation dissociates further into additional hydrogen ions and carbonate ions, as shown in Eq. (2) and (3):



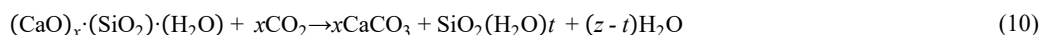
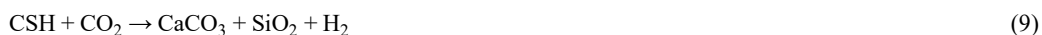
Carbon dioxide is added during the mixing process to initiate the carbonation of freshly hydrated cement.  $\text{CO}_2$  reacts with  $\text{Ca}(\text{OH})_2$  to form calcium carbonate Eq. (4). While the carbonation reaction is similar across different methods of  $\text{CO}_2$  infusion, the amount of product formed varies depending on the  $\text{CO}_2$  medium used. When  $\text{CO}_2$  is infused in gas form, one mole of  $\text{CaCO}_3$  and one mole of  $\text{H}_2\text{O}$  are produced Eq. (5). In contrast, when  $\text{CO}_2$  is introduced in the liquid phase as carbonic acid, the reaction produces a greater amount of water (Lippiatt & Ling, 2020; Vogler et al., 2022).



During the carbonation process, carbonic acid also reacts with other critical cement compounds. The primary components that usually react with carbonic acid are silicate oxide; calcium silicate and certain cement also contain magnesium compound in it. Eq. (6) until (8) show the chemical reaction that occurs when carbonic acid reacts with cement compounds in mortar.



Other studies show that increased carbonation will accelerate the reaction rate of the calcium-silicate-hydrate (C–S–H) complex. As carbonation removes calcium ions from C–S–H, calcite and amorphous silica gel are formed based on Eq. (9), with the process intensifying as the calcium-to-silicon (Ca/Si) ratio decreases. Additionally, the reaction of belite and alite with  $\text{CO}_2$  results in the formation of silica and calcite which is shown in Eq. (10). Carbonation can also affect ettringite and aluminate, leading to the production of gypsum and alumina gel (Joel André, 2017; Lippiatt & Ling, 2020; Šavija & Luković, 2016).



Chemical reactions result in the formation of nanosized  $\text{CaCO}_3$  and other carbonates, including aragonite, vaterite, and calcite (Akhila & Rao, 2023). The impact of nanosized  $\text{CaCO}_3$  has been demonstrated in previous studies. To utilise and benefit from this theory, this research aims to evaluate the effect of carbon dioxide in carbonic acid when infusing into mortar mixtures. The investigation is guided by three objectives to quantify the carbon dioxide content in the mixture by measuring its concentration within the mortar, to assess the effectiveness of the mortar in retaining carbon dioxide as a stable product

by analysing its functional groups and to examine the microstructure of the mortar formed through reactions with carbon dioxide and carbonic acid.

## 2. METHODOLOGY

### 2.1 Material and methods

The overall research workflow is illustrated in **Error! Reference source not found.** Raw materials required for both carbonic acid and mortar were first prepared and gathered prior to the casting process. During casting, carbonic acid was incorporated into the mortar mixture to facilitate the infusion process. The prepared mortar blocks were then subjected to a curing period before being tested. Three main analyses were conducted to address the research objectives which were universal strength test, thermogravimetric analysis (TGA), and scanning electron microscopy (SEM). The results obtained were subsequently compared and analysed to derive conclusions regarding the experimental findings.

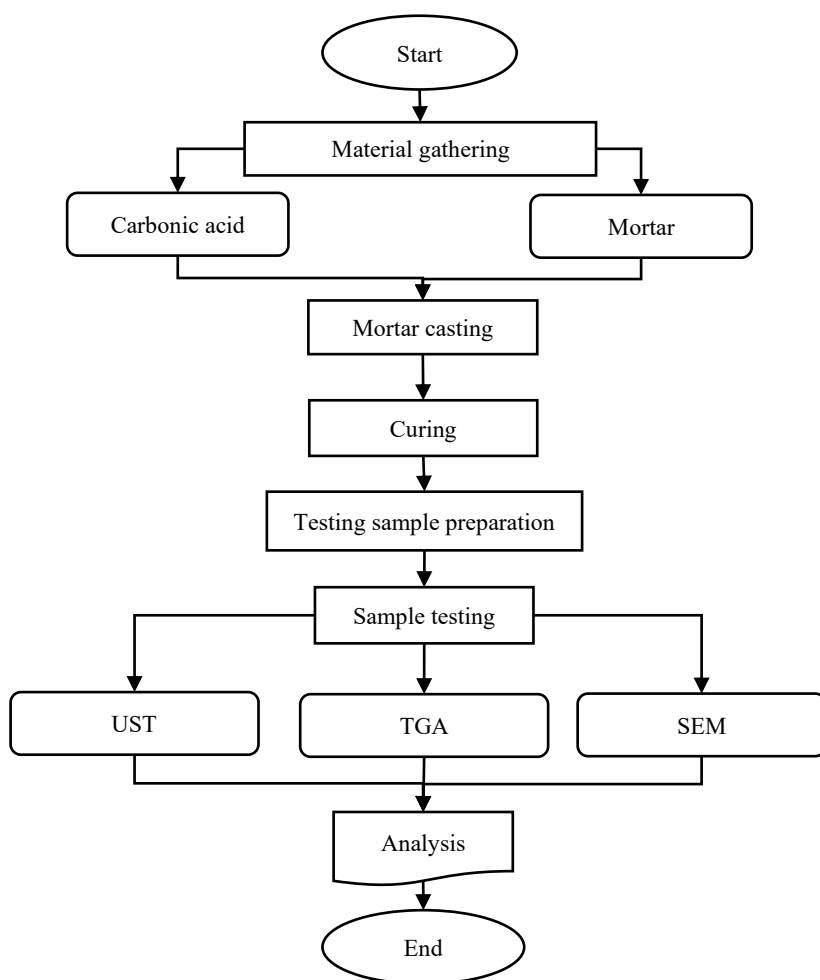


Fig. 1 Process flow diagram of the methodology

Source: Author's own data  
<https://doi.org/10.24191/mjceet.v8i2.5012>

### *Mortar cube sample preparation*

The preparation of carbonated water for the experiment began with the collection of fresh tap water on the day of testing. The water was allowed to equilibrate with ambient conditions by standing at room temperature ( $\sim 25^{\circ}\text{C}$ ) for one hour. To preserve the natural ionic composition, no further purification or deionisation was performed. The equilibrated water was then transferred into a 5 L container, where a calibrated pH electrode and temperature probe were inserted to enable real-time monitoring.

Carbon dioxide gas was introduced into the water through a submerged rubber tube at a constant flow rate of 20 L/min. The infusion continued for approximately four minutes or until the pH stabilised within the range of 4.0 to 4.2, indicating successful carbonation. Once the target pH was achieved, the carbon dioxide flow was terminated, and the container was immediately sealed to minimise degassing. The carbonated water was subsequently used without delay to ensure the retention of dissolved carbon dioxide.

The experimental investigation on mortar carbonation was carried out using ordinary Portland cement (OPC) – CEM I 42.5N from Tasek Cement. River sand was used for the fine aggregates with sizing of 3 to 4 mm. The ratio used was 1:6 which can be considered as conventional and common in mix ratio that used in research (John Adabara et al., 2019; Khalid et al., 2021; Qureshi, 2020). The mix proportions are listed in Table 1.

Table 1. Mix proportions and ratio for mortar mixture

Mixture	Carbonated to tap water Ratio		Component Ratio	
	Carbonated water	Tap water	OPC	Fine Aggregates
Mortar <sub>1</sub>	0	1	1	6
Mortar <sub>2</sub>	0.25	0.75	1	6
Mortar <sub>3</sub>	0.5	0.5	1	6
Mortar <sub>4</sub>	0.75	0.25	1	6
Mortar <sub>5</sub>	1	0	1	6

Source: Author's own data

The mortar mixtures were prepared by initially dry-mixing the cement and fine aggregates for 3 minutes to achieve uniform distribution. Water was then gradually added while mixing continued until a fully homogeneous mixture was obtained. The fresh mortar was cast into standardised 50 mm cube moulds in accordance with ASTM C109 and compacted using a tamping rod. The specimens were left undisturbed for 24 hours before being demoulded and subsequently air-cured under ambient conditions for a duration of 28 days.

### *Universal test analysis (compressive strength)*

The cube-shaped mortars were tested for compressive strength at various concentrations of carbonic acid, all while maintaining a consistent curing time of 28 days. When testing, the mortar block was positioned flat beneath a hydraulic compressor. To ensure even force distribution, a flat metal sheet was placed on top of the moulded mortar before applying the pressure. The pressure gradually increased until the mortar reached its maximum load, at which point a noticeable crack became visible. A compressive strength test is conducted to determine the compressive strength of the cured mortar, which can then be correlated to its microstructure and the amount of carbon dioxide it contains. The compressive testing was performed using a universal testing machine (plate number: UTM-1000, Jinan Chenda Testing Machine Manufacturing Co., Ltd., China). Fig.2 shows the set up of the testing unit.

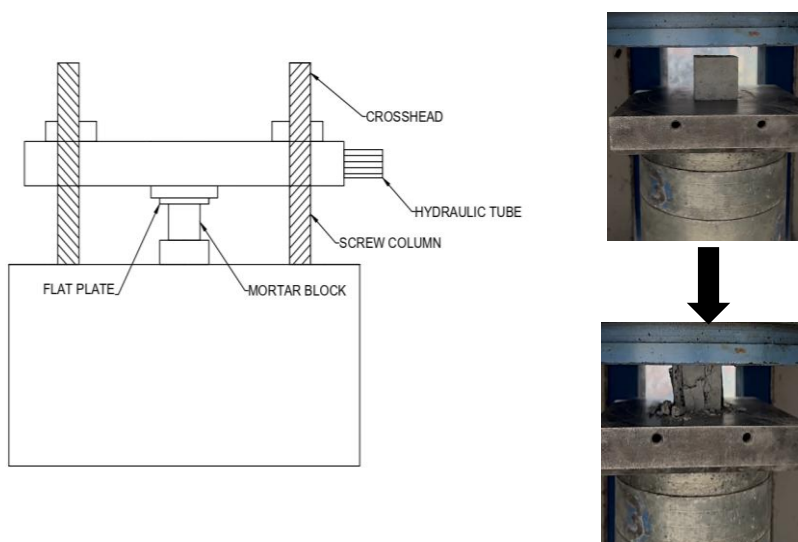


Fig. 2. Universal strength test setup and before and after test

Source: Author's own data

#### *Thermogravimetric analysis sample preparation*

Thermogravimetry involves the continuous measurement of a sample's mass under a controlled temperature increase to quantify reactions involving gaseous emissions. Thermogravimetric analysis (TGA) is an effective method for determining concrete carbonation, as it quantifies the calcium carbonate and portlandite contents in powdered mortar samples extracted from concrete, which also known as concrete mortar (Villain et al., 2007). After drying, the powder samples were prepared by profile grinding the flat surfaces of the specimens using carbide burs. The resulting mortar powders had a fine particle size, with a D50 value of  $20 \pm 4 \mu\text{m}$ . Powder extraction was conducted in three defined regions: the outer region (0–8 mm), the middle region (8–16 mm), and the core region (16–24 mm), designated as layers 1, 2, and 3, respectively (Alimi et al., 2022). The powders from each layer were individually stored in sealed plastic bags. The thermogravimetric test was conducted using a TGA/DSC (STAR<sup>®</sup> System, Mettler Toledo, United States). During the test, the samples were heated from 35 to 1000 °C at a constant rate of 10 °C per minute, with air supplied to facilitate the combustion process.

#### *Scanning electron microscopic sample preparation*

The specimen was finely ground and mixed with KBr for microstructure analysis. The device used for this analysis was the SEM (SU3500, Hitachi High Technologies, Japan) which operated at an acceleration voltage of 15 kV and were conducted within the frequency range of 500 to 4000  $\text{cm}^{-1}$ .

### 3. RESULT AND DISCUSSION

#### 3.1 Universal test analysis (Compressive strength)

The compressive strength of five mortar mixtures was evaluated after 28 days, and their performance were compared as shown in the graph in Fig. 3. A compressive strength test analysis of various carbon dioxide infusion mixtures revealed the distinct influence of carbon dioxide on the mechanical strength of

the mortar. The highest values recorded is the infusion of 100% carbonic acid, the increment in strength recorded is 18% higher compared to control samples (0% carbonic acid) with an average maximum load of 10.45 MN and an average stress of 4.18 MPa. 75% carbonic acid infusion results in similar strength to the control mixture, with an average maximum load of 8.77 MN and an average stress of 3.51 MPa. On the other hand, a significant reduction in strength can be observed on the 50% and 25% carbonic acid infusion which result in 17% and 30% reduce in strength respectively as compared to control samples.

The trend that has been observed is align very well with TGA study that confirming the involvement of calcium carbonate helps in strengthening the concrete if the concentration is sufficient to fill in the micropores. 100% carbonic acid infusion suggest that carbon dioxide concentrations may help offset the initial loss in strength which shown by 25% and 50% carbonic acid infusion. This indicates that higher concentration of carbon dioxide can progressively restore the mortar's strength.

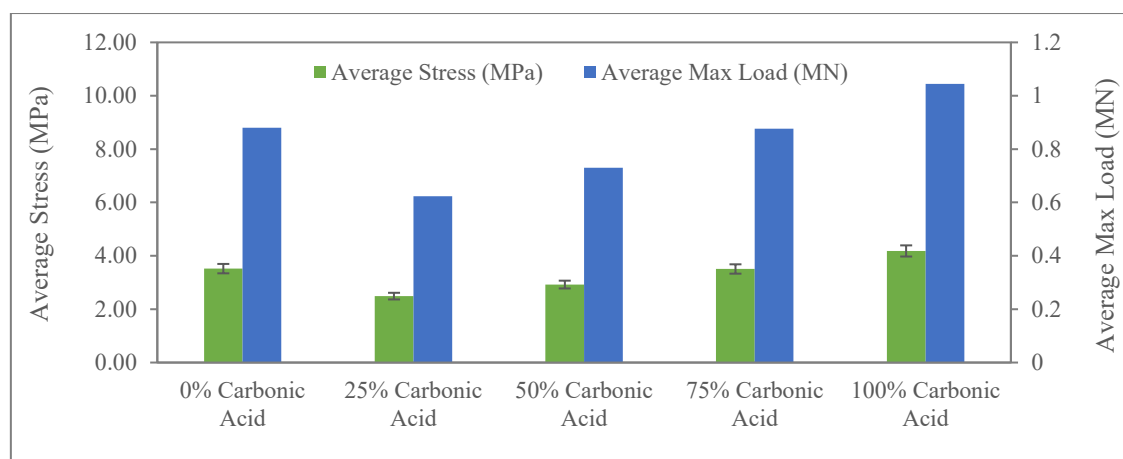


Fig. 3. Universal test analysis comparison for all ratio of carbon dioxide solution

Source: Author's own data

Beside the formation of calcite (calcium carbonate), the strength of the mortar is also influenced by the amount of water contained in the mortar will affect its porosity (Park et al., 2024). Carbonic acid reacts with components in the cement to produce new compounds such as silicon dioxide ( $\text{SiO}_2$ ), aluminium oxide ( $\text{Al}_2\text{O}_3$ ), and calcium sulphate ( $\text{CaSO}_4$ ) shown in Eq. (9). As the reaction progresses, the amount of water produced increases which are formed due to the addition of carbonic acid and formation of water when carbon dioxide reacts with C-S-H in Eq. (9) and (10).

Overall, the results show a trend where low concentrations of carbon dioxide initially reduce mechanical strength, but as the concentrations of carbon dioxide increase, it leads to a notable improvement, culminating in superior structural performance at full carbonic acid infusion. These findings highlight the potential of full carbonic acid infusion to optimise mortar strength beyond the levels of the control mixture as several journals have shown similar results that indicated higher carbon dioxide concentrations and pressures enhanced compressive strength and carbon dioxide (Lee et al., 2017; Patil, 2021; Winnefeld et al., 2022).

### 3.2 Thermogravimetric analysis of mortar sample

Fig. 4 depicts the derivative thermogravimetric (DTG) curves of various carbonic acid infusion mortar sample at 28 days. TGA curve displays the several stages of mass loss indicating reaction that occur when the compound or component in the mortar is being decomposed or transformed. A significant mass loss occurs from 20 to 180 °C indicates the mass loss associated with moisture evaporation within the mortar sample. As the temperature increases to the range between 420 to 500 °C, a significant and sharp peaks is corresponding to the dehydroxylation of calcium hydroxide into calcium oxide and water vapor. This stage is considered important in determining the degree of hydration and availability of portlandite within the matrix. At temperature ranging between 550 and 700 °C, a substantial weight loss is observe indicating the decarbonation of calcium carbonate which decompose into calcium oxide and carbon dioxide. In this range, the graph peaks are varied in term of sharpness but maintaining the mass loss rate.

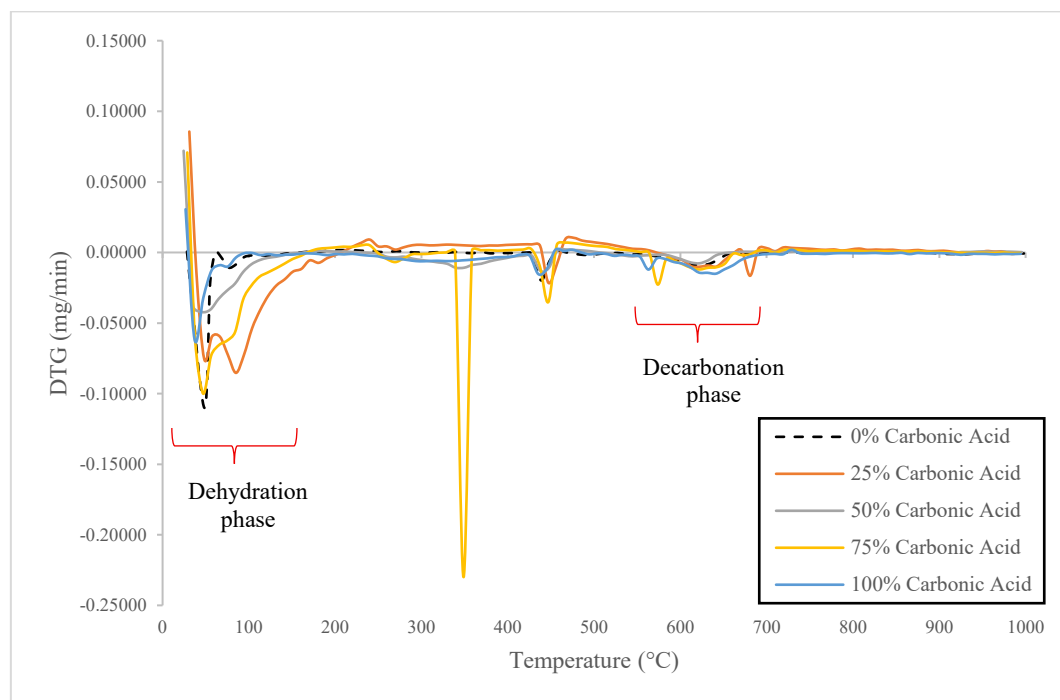


Fig. 4 Derivative thermogravimetric result of all mortar samples

Source: Author's own data

The DTG curves in Fig. 5 indicate calcium dioxide is being release at between 550 and 700 °C, corresponding to the thermal decomposition of calcium carbonate ( $\text{CaCO}_3$ ) into calcium oxide ( $\text{CaO}$ ) and carbon dioxide (Karunadasa et al., 2019). This temperature range was selected from the attributes of  $\text{CaCO}_3$  decomposition, enabling the extent of carbon dioxide release to serve as an indicator of the  $\text{CaCO}_3$  content in each sample. All samples exhibit a major decomposition peak between approximately 550 °C and 700 °C, which corresponds to the decarbonation of calcium carbonate, a common decomposition process for carbonated cementitious materials. This thermal reaction becomes more pronounced in samples treated with carbonic acid, as evidenced by the increased peak intensity (more negative rate) and sharper profiles. The 0% carbonic acid sample (control) exhibits a single broad peak between 600 and 650 °C, characteristic of conventional calcium carbonate ( $\text{CaCO}_3$ ) decomposition in non-carbonated mortar. This broadening



likely reflects the limited and heterogeneous distribution of naturally carbonated phases, which decompose over a wider temperature range due to variations in crystallinity or local bonding environments.

In contrast, samples with higher carbon dioxide infusion (75% and 100%) display sharper and larger peaks within the same temperature range, signifying a more substantial and uniform calcium carbonate content. The increased peak intensity aligns with accelerated carbonation, where abundant carbon dioxide promotes denser calcium carbonate formation, as observed in studies of accelerated carbonation curing (Zhang et al., 2022). Notably, the 100% sample demonstrates the highest mass loss, confirming that maximal carbon dioxide exposure yields the greatest calcite precipitation. The 50% sample's unexpectedly shows low calcium carbonate content (lower than the control sample) further underscores the non-monotonic relationship between carbon dioxide concentration and carbonation efficiency.

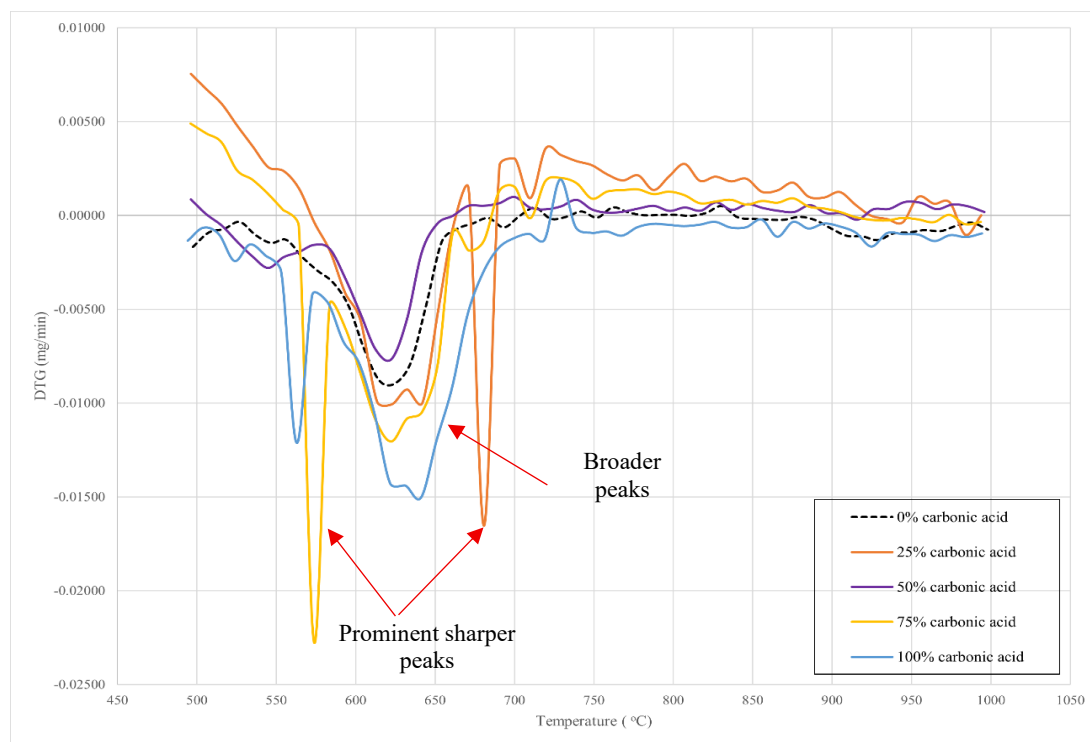


Fig. 3 DTG results on carbonation phase

Source: Author's own data

The analysis further shows that mass loss occurs within the 550 to 700 °C range, and stabilises around 800 °C. Therefore, Eq. (11) can be applied to calculate the percentage of calcite formed during the curing process, as  $\text{CaCO}_3$  decomposition primarily occurs between 500 and 900 °C (Vogler et al., 2022).

$$m_{\text{CaCO}_3} = \Delta m_{500\text{ }^{\circ}\text{C}-900\text{ }^{\circ}\text{C}} \times \frac{M_{\text{CaCO}_3}}{M_{\text{CO}_2}} \quad (11)$$

The relationship between carbon dioxide infusion and  $\text{CaCO}_3$  formation is clear, with higher carbon dioxide infusion levels generally corresponding to increased  $\text{CaCO}_3$  content, as indicated by the progressive rise in carbon dioxide release peaks from 0% to 100% of carbonic acid ratio to water. The data in Fig. 4 shows that while calcium carbonate formation in the mortar samples increases with  $\text{CO}_2$  infusion, the trend is not strictly linear. Notably, 75% carbonic acid, with the highest carbon dioxide infusion (34.09%), exhibits the greatest potential for calcite formation, followed by 100% (29.50%), 25% (15.18%), 50% (12.59%), and 0% (11.91%). These results underscore the role of carbon dioxide infusion in enhancing  $\text{CaCO}_3$  formation in mortar, highlighting its potential to improve carbonation and, consequently, the material's durability and performance. This indicates that the onset temperatures are nearly identical across all samples, suggesting that the carbonation products in the mortar may primarily consist of low-crystallinity  $\text{CaCO}_3$  (Lippiatt & Ling, 2020). The decomposition of well-crystallised calcites into  $\text{CaO}$  and carbon dioxide typically occurs at higher temperatures, representing the decarbonisation of carboaluminates and poor crystalline  $\text{CaCO}_3$  (Scrivener et al., 2016). Additionally, carbon can exist in less tightly bound forms, with amorphous calcium carbonate potentially breaking down into  $\text{CaO}$  and carbon dioxide at temperatures as low as 400 °C.

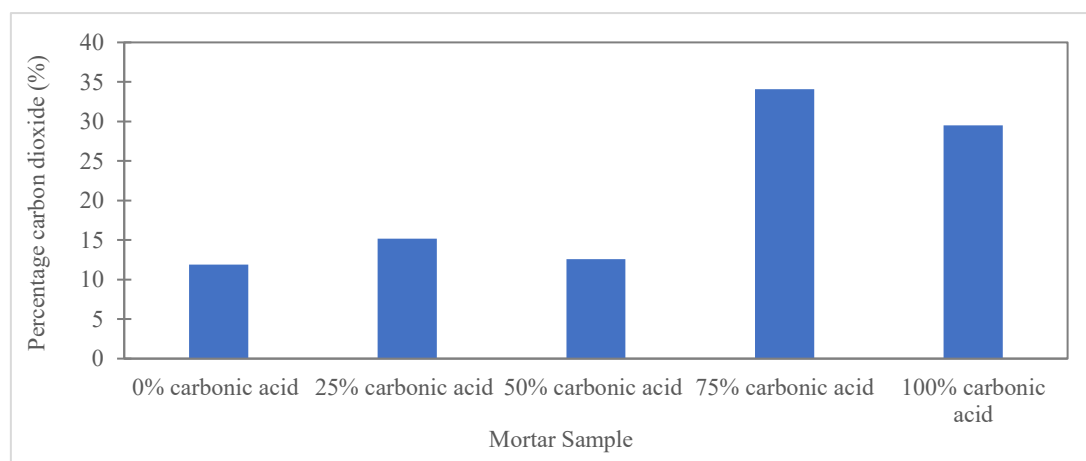


Fig. 4 Percentage of carbon dioxide released during TGA testing

Source: Author's own data

### 3.3 SEM and EDS Analysis

The evidence of carbon dioxide has been properly reacting with cement component can be detected via examination of microstructure changes of the mortar across different samples. These changes in microstructure can be analysed using scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS). As carbon dioxide absorption increases, noticeable microstructural transformations occur, as illustrated in Fig. 5, which highlights the formation of carbonation reaction products. The most prominent changes include the presence of voids and pores on the mortar surface.

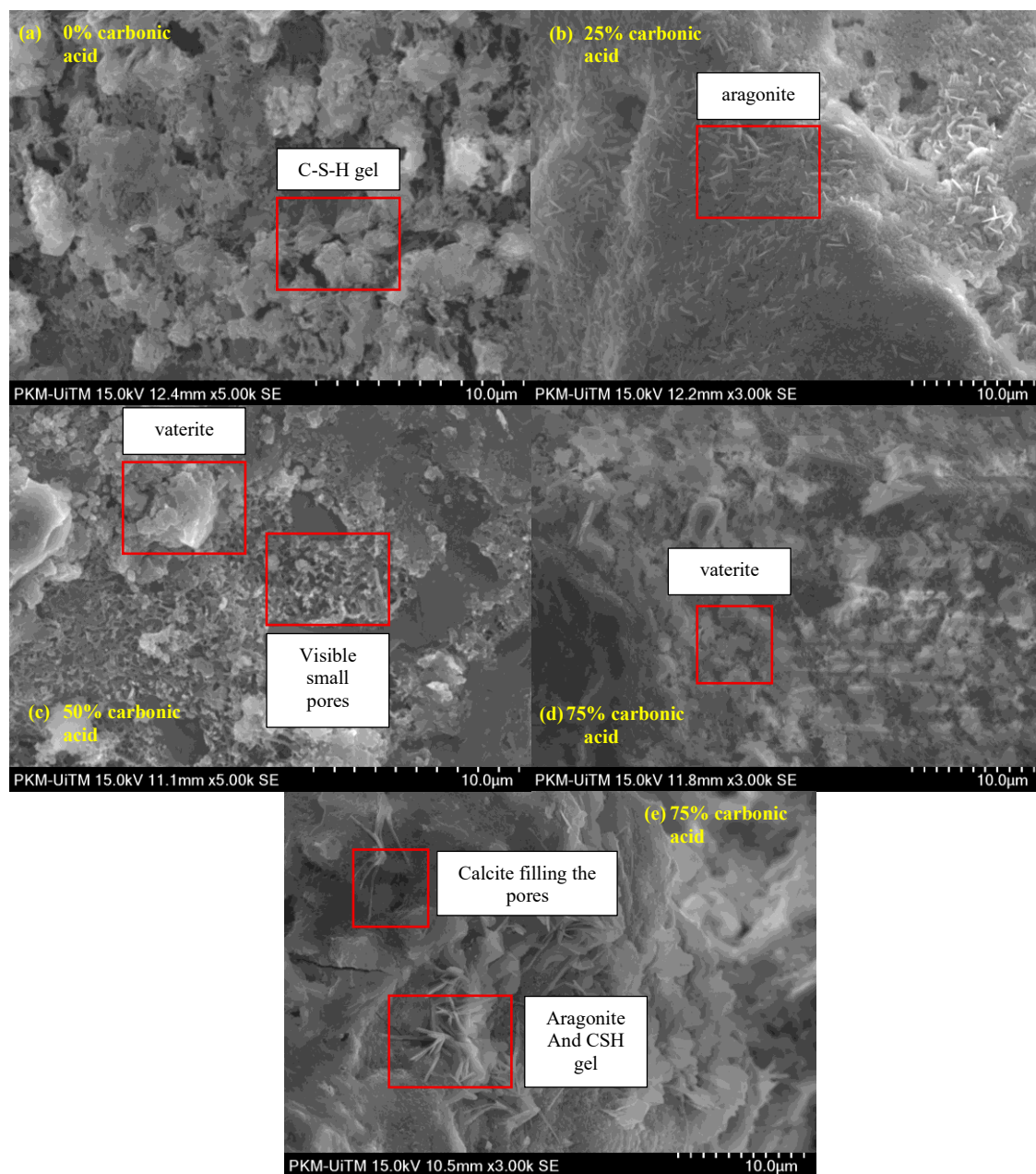


Fig. 5. Show the microstructure changes occur on the mortar at (a) 0%; (b) 25%; (c) 50%; (d) 75%; and (e) 100% carbonic acid

Source: Author's own data

In samples with 0% of carbonic acid, large voids were absent, but the formation of granular compounds was clear to be seen. As CO<sub>2</sub> infusion increases, the mortar surface becomes smoother, while more pore formations are visible. Samples of 25% and 50% carbonic acid, found to have interconnected pores, whereas mortar with 75% and 100% carbonic acid display larger and deeper pores in specific areas.

Upon examining the microstructure of mortar with 0% carbonic acid (Fig. 5(a)), numerous small pores were visible across the sample. However, beneath these pore-like structures, the surface appears dense, with no significant cracks or larger voids. The surface texture was relatively rough, which characterised by small, densely packed granular particles formation. This suggests the hydration of mortar is in controlled manner, resulting in a fine, interconnected structure typical given by fully hydrated mortar which no significant pores that indicates that the water content inside the sample is not excessive. The small crystalline formations could be calcium hydroxide, a common hydration product often appearing as hexagonal plates, along with calcium silicate hydrate (C–S–H) gel. The SEM image of the mortar infused with 25% carbonic acid reveals a dense distribution of needle-like crystalline structures across the matrix surface. These formations are indicative of carbonation products, which is aragonite based on the similarities when comparing it with Choi et al. (2017). In 50% infused carbonic acid however, vaterite can be detected as calcium carbonate instead of aragonite. Approximately, 75% infused carbonic acid produce similar product in carbonation which is vaterite and C–S–H gel however having visible micropores which may be due to high water content after curing. In 100% carbonic acid sample calcite can be detected filling in the micropores while having visible aragonite and C–S–H gel visible on the surface of the sample. The abundance and variety of calcium carbonate products suggest significant carbonation activity, leading to pore refinement and densification of the microstructure. Such microstructural modification may initially enhance the compactness of the mortar.

#### 4. CONCLUSION

The infusion of carbon dioxide through the solution method was found to influence the strength of mortar blocks, as evidenced by the universal strength test results. However, the changes in strength between 100% infused carbonic acid and control sample is not too significant. The findings indicate that higher carbon dioxide concentrations enhance compressive strength and alter the mortar's microstructure by promoting the formation of crystalline and gel-like phases. These microstructural modifications contribute to a reduction in fine pores; however, insufficient compaction prior to curing may still result in the development of larger voids. Furthermore, elevated water content can increase pore formation, thereby exerting a considerable negative effect on the overall strength of the mortar blocks.

Thermogravimetric analysis (TGA) confirms that CaCO<sub>3</sub> decomposes into carbon dioxide and CaO at approximately 600 °C, aligning with previous research findings. Additionally, the study indicates that infused carbon dioxide may escape during curing, resulting in non-linear carbon dioxide release patterns in TGA analysis. The carbonation process also contributes to the transformation of CSH gel into calcite over extended curing periods, further enhancing the mortar's strength. Future research would focus on optimizing the carbon dioxide infusion process to accurately quantify initial carbon dioxide content and analyse the degree of hydration. Establishing a clearer correlation between hydration levels and compressive strength will be crucial for improving mortar performance.

#### ACKNOWLEDGEMENTS AND FUNDING

The authors express their gratitude to Universiti Teknologi MARA for providing necessary facilities and funding through the Strategic Research Partnership (SRP) Grant (100-RMC 5/3/SRP (030/2024)) to carry out this research. They also extend their appreciation to the staff of the Faculty of Chemical and Faculty of Civil Engineering, Universiti Teknologi MARA, Shah Alam, for their assistance with the laboratory work.

## CONFLICT OF INTEREST STATEMENT

The authors agree that this research was carried out without any personal benefits, commercial or financial conflicts, and they declare no conflicts of interest with the funders.

## AUTHOR CONTRIBUTIONS

**Muhammad Irfan Khumaini Kamaruhisam:** Methodology, investigation, formal analysis, and writing-original draft; **Miradatul Najwa Muhd Rodhi:** Conceptualisation, methodology, draft manuscript preparation, validation, supervision, and writing-review & editing; **Mohd Fadzil Arshad:** Project administration and conceptualisation, supervision and validation, and writing-review; **Mohd Syazwan Mohd Syukor:** Project administration and conceptualisation and writing-review; **M Ghaddaffi M Noh:** Project administration and conceptualisation and writing-review.

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